Physical quality of pelleted feed.

A feed model study

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Menno Thomas

Proefschrift

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Abstract

In this study, a review and experimental work is presented on the relation between processing conditions and functional properties of feed constituents with respect to hardness and durability characteristics of pelleted feed. In the first three chapters, a literature overview is presented which describes the methodologies used to determine hardness and durability characteristics of pelleted feed. The second article deals with the effects of processing conditions on the physical quality of pelleted feed. In the last review, article an overview is presented on the effect of raw material constituents and pellet quality.

The effects of processing conditions on changes in starch degree of gelatinization of tapioca meal and protein quality of soy grits (as two model feeds) have been studied. It was concluded that addition of steam and water had a larger effect on changes in the starch degree of gelatinization and the denaturation of protein than the amount of dissipated mechanical energy. Hardness and durability characteristics of pelleted feed were influenced by the amount of dissipated mechanical energy and the amount of steam used.

Mixture studies were conducted in which the state of the model feed constituents -starch and protein- was altered and the subsequent effects on pellet hardness and durability were studied. From these studies it was concluded that an increase in the degree of gelatinization, or an increase in the amount of native protein leads to harder and more durable pellets. Possible explanations for these effects such as the change in deformability of the particles that make up the feed mash and the ability of the starch or protein to act as a binder, are described.

PhD-thesis. Wageningen Agricultural University, Wageningen Institute of Animal Sciences, Section Animal Nutrition, Marijkeweg 40, 6709 PG Wageningen, The Netherlands.

Stellingen

1. De ontsluitingsgraad van zetmeel en de dispergeerbaarheid van eiwit in gepelleteerde diervoeders hebben geen causale relatie met de hardheid en slijtvastheid; het zijn desondanks goede indicatoren voor de 'deformatie' en het 'bindend vermogen' van de deeltjes in de pellets.

   (Dit proefschrift)

2. Een verhoging van hardheid en slijtvastheid van gepelleteerde diervoeders kan worden bereikt door vervanging van *natief* zetmeel (tapioca) door *ontsloten* zetmeel maar ook door vervanging van *gedenatureerd* eiwit (soya) door *natief* eiwit.

   (Dit proefschrift)

3. Voor het voorspellen van de fysische kwaliteit van gepelleteerde zetmeelrijke voeders is de glas-overgang in zetmeel een belangrijker kenmerk dan de ontsluitingsgraad.

   (Dit proefschrift)

4. De pelletkwaliteit in mengvoederfabrieken kan aanzienlijk beter worden voorspeld indien behalve procescondities en voersamenstelling ook PDI (Protein Dispersibility Index) en zetmeelontsluitingsgraad als kenmerken van functionele eigenschappen worden gemeten.

   (Dit proefschrift)

5. Deformeerbaarheid en arbeid benodigd voor breuk van pellets zijn van groter belang voor de voeropname door jonge varkens dan de maximale breuksterkte.

6. Het gebruik door actiegroepen van schokkende beelden in de media over misstanden in de agrarische sector draagt niet bij aan een structurele oplossing van problemen die spelen in deze sector.

7. De sterkste drijfveer voor de aanleg van natuurterreinen op uit productie genomen landbouwgrond is een romantisering van een Nederlands natuurbeeld dat nooit heeft bestaan.

8. Statistische methodieken ten behoeve van procesoptimalisering worden in technologisch onderzoek zeer slecht benut.

9. De door TNO gehanteerde slagzin 'de kracht van kennis' geeft een onjuiste weergave van de feitelijke wetenschapsbeoefening; het verdient daarom de voorkeur deze te veranderen in 'de kracht van een kennis'.

10. Goede wetenschappers zijn herkauwers.

11. Een ras is een verzameling erfelijke afwijkingen (Midas Dekkers, 1997).

12. Verscherpte regelgeving en controle leidt zowel bij justitie alsmede bij de Nederlandse mengvoederindustrie tot een cellentekort.

13. Door de toename van GMP en ISO-normering in de mengvoederindustrie gaat het gezegde 'wie het eerst komt, het eerst maalt' niet meer op.

Stellingen behorend bij het proefschrift:
'Physical quality of pelleted feed. A feed model study'.

Menno Thomas.

17 Juni 1998.

Voor mijn Ouders,
Diane

Voorwoord

Dit is 'm dan... Na op de kop af vijf jaar zwoegen, ligt nu voor U -mijn proefschrift-. En hoewel mijn naam op de voorkant prijkt, is dit werk niet tot stand gekomen zonder de hulp van velen. Een kleine (bij lange na niet complete) greep uit de verzameling:

Zonder goed materiaal en apparatuur, geen onderzoek. Daarom, bedankt, alle mensen van de werkplaats van het Biotechnion en in het bijzonder Evert Janssen en Andre Sanders. (Alle sleutels en doppensets liggen weer op de goeie plek). Van de afdeling electro; Reinoud Hummelen en Hans Meijer, bedankt. Jullie hebben er met z'n allen voor gezorgd dat de installaties draaiende bleven, ook als ik weer eens een proef had bedacht aan de grenzen van het kunnen van mens en machine.

Zonder grondstoffen geen resultaat. De CHV in Veghel ben ik zeer erkentelijk voor het ter beschikking stellen en in bewaring houden van de 40 ton tapioca, die is gebruikt in de verschillende proeven beschreven in dit proefschrift.

Zonder kennis en kunde geen thesis. Ton van Vliet en Katja Grolle van de groep levensmiddelen natuurkunde ben ik zeer dankbaar voor de mogelijkheid om allerlei metingen aan pellets uit te kunnen voeren in jullie groep. Verder ben ik zeer veel dank verschuldigd aan Ton van Vliet. Op het voor mij onbekende vakgebied van de levensmiddelenrheologie en breukgedrag ben jij mijn steun en toeverlaat geweest.

Dick van Zuilichem, Willem Stolp en alle andere mensen binnen de extrusie groep hebben op twee manieren bijgedragen aan dit onderzoek; ten eerste, doordat veel ideëen en concepten in dit proefschrift zwaar leunen op de vergaarde kennis uit de levensmiddelenextrusie en ten tweede, door de vele praktische adviezen omtrent het pelleteren van veevoeders. Zeker in de begindagen van m'n AIO-schap, toen ik nog geen pers van een koffiezetapparaat kon onderscheiden, heb ik daar zwaar op geleund. (Koffiezetten lukt inmiddels aardig...)

Een bijzonder woord van dank gaat uit naar de studenten die een bijdrage hebben geleverd aan de verschillende proeven of onderdelen daarvan. In chronologische volgorde waren dat; Ronald ten Cate, Edwin van Kol, Paul Huijnen, Kees-Jaap Hin, Arjan Wegereef en Henk Bhagwandin. Soms was het weleens moeilijk uit te maken wie nu de meester was en wie nu de leerling. Jullie allen bedankt voor jullie bijdrage.

Wetenschap kan niet gedijen als het niet is ingebed in een groep waarin samenwerking, saamhorigheid en vriendschap vanzelfsprekende onderdelen zijn. Alleen dan is het mogelijk om onbevangen elkaars ideëen te kunnen bediscussiëren en de grens van het weten weer een stukje op te schuiven. Daarom, veevoederaars, ALLEMAAL dank voor de fijne jaren binnen de groep, zowel op wetenschappelijk terrein als daarbuiten. Twee collega's wil ik echter in het bijzonder vermelden, m'n collega-BIC'ers: Jacob en Jos. Met z'n drieën ongeveer gelijk begonnen, hebben heel wat theoriën het levenslicht gezien, eerst in de kelder in het oude

gebouw en later in de nieuwe vleugel, evenzovele zijn echter ook weer snel afgebrand. Rest mij nog maar één ding te melden: Heren, de lat ligt!

Thomas, jij bent degene geweest die m'n eerste schreden op het pad van de wetenschap heeft begeleid, eerst tijdens mijn studie en daarna meer als vriend en collega dan als een 'strakke baas' tijdens de aansluitende AIO-periode. Vooral de grote mate van vrijheid bij mijn onderzoek die je, tezamen met Martin en Seerp, mij hebt gegund, heb ik in het begin wel eens moeilijk gevonden. Echter, met het toenemen van mijn onderzoekservaring heb ik dat steeds meer als plezierig ervaren. (Nu had ik willen schrijven dat deze vrijheid mij heeft gemaakt tot de onderzoeker die ik nu ben. Echter, met het oog op de stukjes na de promotie heb ik uit taktische overwegingen besloten dat toch maar niet te doen...) Daarom, Martin, Seerp en Thomas in het bijzonder, dank voor het door jullie in mij gestelde vertrouwen.

Pa, Ma, wijsheid komt met de jaren. Om in de gelegenheid te zijn om wijsheid te ontwikkelen moet de basis goed zijn. En nu ik dan wat ouder ben weet ik, dat die basis perfect is geweest!

"In bepaalde landen, met name Duitsland, wil het gebruik dat de ega van een (mannelijke) gepromoveerde met 'mevrouw Doctor' wordt aangesproken. Volgens Nederlandse maatstaven is dit 'overdreven'. Wie echter gehuwd is of samenwoont en in deze toestand een proefschrift schrijft, weet dat, als er dan toch betiteld moet worden, er wel degelijk redenen zijn om de eer op deze wijze te delen" (vrij naar H.W.J. Huiskes, stelling dissertatie Eindhoven, 1979).

Diane, al meer dan zeven jaar delen we lief en leed. En als geen ander weet jij dat het schrijven van een proefschrift soms een asociale aangelegenheid is. Diane, bedankt voor je steun en begrip. Op naar de volgende jaren.

Pa, Ma, Diane, aan jullie draag ik dan ook dit proefschrift op!

April 1998

Contents

General Introduction

M. Thomas

General Introduction

Animals require feed. Feed that has been shaped into pellets is the major subject of this thesis. High producing animals, as they are being kept in modern husbandry-systems require feed optimised to fit their nutritional needs. In addition, modern feeding systems have certain requirements with respect to the quality of the feed, in order to be able to transport and dose it without additional spoilage. A possible means to do this is to fix the diet composition to assure that every animal receives its nutrients in a the correct proportion. This is what is done in feed manufacturing by a pressure agglomeration process called pelleting.

Pelleting is one of the oldest means of feed processing. Already in 1937, Patton et al defined the nutritional superiority of pelleted poultry feeds over mashes. However, earlier records have been found and date back to the time of Napoleon where the horses in his army were fed a type of feed that was agglomerated by an expeller type of device. The feed was compressed by means of a screw through holes in a die-plate and thus agglomerates where formed without any pretreatment. With an ever increased use of pelleted feeds, pre-treatments were more and more used to condition the feed to increase its pelletability. Output of the installation increased and the hardness and durability of the feeds was positively influenced by these pre-treatments. Earlier conditioning methods may have applied only water as means of conditioning. Nowadays, the use of steam, other liquids like molasses and, recently, the introduction of equipment which imparts shear to the feed materials have turned the production of animal feed in a high capital investment industry. From this, a constant stream of research work emerged (see Chapter 2) and in the last decade(s) considerable effort has been directed towards determining the effect(s) of the different process variables on the systems parameters of the pelleting process and the effects of processing variables on pellet hardness and durability.

From this work and experience, rules of thumb were developed on how to handle feeds which were rich in specific ingredients, for instance, specific conditioning requirements were put forward for groups that are high in starch or heat sensitive (like feeds with sugars or milk incorporated) high in protein, high in fibre or mixtures containing urea or molasses (Payne, 1978; Maier and Gardecki, 1992). From this it follows that, although the only means of adjustment of the pelleting process is via changes in the processing conditions, the operators were, and still are, aware of the effect of specific diet ingredients and the effects of these raw material (properties) on pelleting ability of the feed mash. This is examplified by the large amount of research that has been conducted on the effects of the process variables, like adding more or less water or heat in the form of steam, the introduction of residence time by means of

ripening kettles or the effect of shear with respect to energy consumption and pellet quality. Recently introduced processing equipment e.g. the expander, introduced a change from the pellet type of feed towards an expandate, an agglomerate without a precisely defined shape, but with the advantages of a fixed nutrient composition. Still, pelleted feed comprises about 85 to 90 % of the feeds produced in the Netherlands.

Still, with all the means available, it is difficult to achieve a certain pellet quality, in terms of hardness and durability. Pellets may therefore have not enough structural integrity to survive the handling and transport from the factory to the farm. This may then result in segregation of the particles, with negative nutritional consequences. Dustiness may increase, which impairs health of animal and man.

Wood (1987) studied the effect of functional properties of protein and starch in a feed model system and found that large effects could be found on pellet hardness and durability of the feed, depending on the functional properties of the feed ingredients. His results showed that by gradually replacing native starch with pre-gelatinized starch, hardness and durability of the pelleted feed was improved. Likewise, the exchange of denatured protein by raw protein showed that pellet hardness and durability became higher. This study of Wood (1987) showed that large effects on pellet quality can be expected, depending on the properties of the raw materials or their constituents. His study has been the onset of the work described in this thesis.

Aim of the thesis

The aim of the thesis is to study the relation between functionality of some feed constituents, with respect to the pellet hardness and durability. Protein and starch have been chosen as subject of research. The choice has been restricted to two raw materials in which the effects of (changes in) functional properties of starch and protein on physical pellet quality were evaluated. This choice is also based on the fact that protein and starch represent a valuable portion of the nutritive value of the feed. Tapioca has been chosen for its high starch content and negligible protein content. Soy has been chosen for its high protein content and negligible amount of starch. Hence, it is possible to estimate the effect of tapioca starch and soy protein and their contribution to the physical pellet quality with minimum interference (as compared to other feedstuffs) of the non-protein components or non-starch components present.

The general hypothesis throughout this thesis will be that an increase in native protein increases pellet hardness and pellet durability over the inclusion of denatured protein. Likewise, an increase in degree of gelatinization is related to an increase in the physical quality of pelleted animal feeds. Furthermore, it is hypothesized that the state of the protein

and the state of the starch can be altered by the processing conditions, to such an extent that they provide a means of controlling the pellet hardness and durability.

In this thesis, concepts will be developed on the effects of some raw material constituents, the state of these constituents, and how they are related to pellet hardness and pellet durability. This is especially important if it is considerated that new feeding systems emerge, like choice-feeding and pelleting of diet ingredients, which both require a certain physical quality of the pellet to be met (van der Poel et al., 1997). It is hoped that the knowledge generated in this thesis may help in a further understanding on how the quality of a pellet is influenced by the properties of the feedstuffs, and in addition, that some of the methods used may be helpful for the feed manufacturer to solve problems that are associated with pellet quality, whenever they appear.

Outline of the thesis.

The thesis can be subdivided in three parts. In the first part, covered in the first three chapters, an overview is given on the state of the art concerning the manufacture of pelleted feed. The chapters 4 and 5 are two studies in which the effects of different processing conditions which respect to changes in protein quality and starch degree of gelatinization have been investigated. Thus, these chapters cover the effects of processing conditions on changes in constituents of two model feed materials and subsequent effects on hardness and durability of pelleted animal feeds. The last three chapters are concerned with the effects of changes in functionality of model feeds and their effect on pellet hardness and durability. Figure 1 gives a schematical overview of the contents of this thesis.

Review part

The chapters 1, 2 and 3 give an overview of the conditioning and pelleting process from three different perspectives. In Chapter 1 an overview is presented on the various methods used in feed manufacturing and feed science to evaluate the physical quality in terms of hardness and durability of the feed. In Chapter 2 an overview is given on the various effects of processing parameters, like water and steam addition, on the hardness and durability of the pelleted feed. In the third and last chapter of the review section, an overview is presented on the effects of raw materials and their constituents in animal feeds with respect to their pelletability.

Effects of process-conditions on functional properties

In Chapter 4, an experiment is described which relates the effect of changes in steam pressure, water addition and screw speed to changes in protein quality. Protein quality is determined by

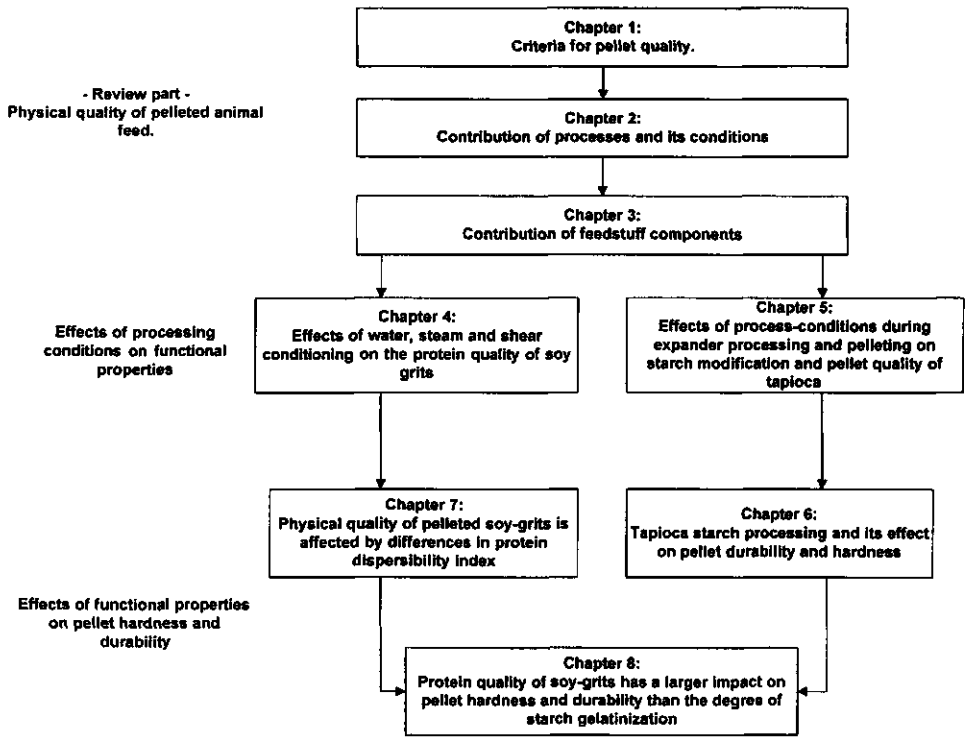

Figure 1: A schematical representation of the outline of the thesis.

the protein dispersibility index (PDI), trypsin inhibitor analysis (TIA) and nitrogen solubility index in KOH (NSIkoh). In chapter 5, tapioca was used to determine the effect of changes in steam pressure, water addition, screw speed and dissipated energy of the expander to changes in degree of gelatinization (SGD). In the last experiment the feed mash was pelleted, and the resulting pellet hardness and durability was evaluated with respect to the four factors mentioned

Effects of functional properties on pellet hardness and durability

The last three chapters are concerned with the key objective of the thesis, to determine the contribution of changes in protein quality and changes in degree of starch gelatinization to the physical quality of pelleted animal feeds. In all of the three experiments mixture designs have been used, in which low functional protein is mixed with high functional protein or low functional starch is mixed with high functional starch. Changes in the functional properties of the feeds are determined by changes in the protein quality as measured by PDI and NSIkoh and in the case of starch by using an enzymatic test (SGD) or differential scanning calorimetry

(DSC). In Chapter 6, tapioca was pre-processed to induce differences in the degree of starch gelatinization by means of expander processing. This expander processed tapioca was then mixed with unprocessed tapioca to give differences in the degree of gelatinization. These mixtures where then pelleted and pellet quality was evaluated. Likewise, in Chapter 7 soy-grits where used to evaluate pellet quality on mixtures of soy-grits with high PDI and low PDI. In this experiment, the soy-grits where not processed but obtained from a commercial supplier. In the last experiment (Chapter 8) a feed model system was used consisting of 50% soy-grits and 50% tapioca. Within each 50%, PDI or SGD were changed to obtain differences in functionality of the mixtures. These mixtures were pelleted and the pellets were evaluated for their physical quality.

In all experiments was the pellet quality evaluated using industrial standards like the Holmen durability tester, the Pfost durability tester or the Kahl hardness tester. In addition, use was made of the Kramer shear press and a general compression and tension tester.

It is hoped that with the knowledge developed in this thesis, feeds can be further optimized to suit the need of man and animal.

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1 Physical quality of pelleted animal feed. 1. Criteria for pellet quality.

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Physical quality of pelleted animal feed 1. Criteria for pellet quality.

M. Thomas and A.F.B. van der Poel

Abstract

In a series of three articles, the physical quality of pelleted animal feeds is discussed from an engineerers point of view. In this first manuscript an overview is given on the binding mechanisms in pelleted animal feeds. Principles and methods for evaluation of physical quality of pelleted animal feeds are being reviewed with respect to pellet hardness and durability. Methods are outlined with respect to quality standards both from a pragmatical and scientific point of view.

It is concluded that binding in pellets most probably is due to solubilisation and subsequent crystallisation of feedstuff components e.g. starch, sugars, fats or 'liquid necking'. Liquid necking is a binding mechanism which uses the surface tension of water, in a three phase system of air, water and particles to maintain structural integrity of the pellet. Soluble components might be introduced in the feed mash subjected to pelleting. They are either incorporated in the mixing phase or result from processing as a function of processing variables during the subsequent stages, conditioning, pelleting and cooling/drying, of the feed manufacturing process. To evaluate the physical quality of pelleted feeds, generally a subdivision is being made into tests that evaluate 'hardness' and tests evaluating 'durability' of a given pellet. Several devices measuring fragmentation strength, and devices determining abrasion strength of pellets are being discussed. It is concluded that some tests presently available, evaluate a mixture of hardness (fragmentation) and durability (abrasion) effects. The feed manufacturer or feed technologist should be aware of the reason for evaluating the pellet quality and subsequently choose the appropriate, most suitable method, since no unique test exists that covers all parameters of interest related to physical quality of pelleted animal feeds.

Introduction

Animal feed manufacturing involves the use of a variety of raw materials to produce compound feeds. The feeds are defined according to certain specifications with regard to nutritive composition based on specified descriptions for nutritional, hygienic and physical quality. Together, these specifications require knowledge of a vast number of different properties of ingredients to optimize processing while maintaining or controlling nutritional quality for a given feed form. It is therefore that concerted action by the disciplines 'nutritional science' and 'feed science and technology' may nowadays be essential for further progress in livestock production.

Animals which receive pelleted feeds generally have higher performances in terms of average daily gain and lower feed conversion compared to mash feeds as reported for pigs (Vanschoubroek *et al.*, 1971; Pond and Maner, 1984) and poultry (Calet, 1965; Quemere *et al.*, 1988; Moran, 1989). Feed processing influences rate of degradation and rate of passage of feed components of ruminants (van der Poel *et al.*, 1995). For instance, pelleting reduces the resistance of starch against ruminal degradation by about 15% (Tamminga and Goelema, 1995).

Nielsen (1994) showed that expander processing reduced the effective protein degradability (EPD) of raw materials with on average 8% units. These effects may lead to differences in milk production and composition. It can therefore be argued that nutritional value is both, influenced by the raw materials used (van Rooy, 1986) and by processing conditions during operation (Skoch *et al.*, 1981; 1983).

Different animal species require different physical properties for their respective feeds. This means that different quality standards are used. For example, for mice feed (Koopmans *et al.*, 1989a,b) or broiler feed (Moran, 1989) different qualities are required. For fish-feeds, additional pellet characteristics such as flowability, sinking velocity, water absorption and water solubility are important.

Furthermore, the hygienic quality of feeds is important. Hygienic quality involves the control of microbiological contamination of feeds based on levels of enterobacteriaceae and salmonella (McCapes *et al.*, 1989).

Feed processing includes the treatment (physical, chemical, thermal) of a feed prior to consumption by animals (Maier and Bakker-Arkema, 1992). In general, the variability in processing effects is associated with the choice of equipment, with processing conditions as well as with the processing system e.g. the combination and sequence of process equipment (Melcion and van der Poel, 1993). Therefore, processing may involve a simple process such as blending in the form of mash or processing can be much more complicated such as (double) pelleting, crumbling or when an extruder or expander is used (Pipa and Frank, 1989; Veenendaal, 1990; Van Zuilichem and Van der Poel, 1993). It should be noted however, that a specific piece of equipment per se is limited in its application and cost-effectiveness. Generally, the specific equipment is used as a component of a process system where the efficacy of this equipment can only be optimized by careful consideration (Das *et al.*, 1993; Tran *et al.*, 1991), taking into account the upstream and downstream processing.

In routine animal feed manufacturing raw materials are blended and ground to obtain mash feeds that are further subjected to some form of steam and/or water conditioning before e.g. granulation is applied. Granulation and its products (granulates, pellets) offers a product form that has many advantages over meal and that's why its application is widespread for both raw materials, feed additives and animal feeds. Some advantages (Rumpf, 1958; Friedrich and Robohm, 1969; Vanschoubroeck *et al.*, 1971) are:

- Pellets have better flow properties, necessary for good transport in conveying equipment, and (gravitational) discharging behaviour from silos, than the meal they were prepared from.
- The bulk density of pellets is generally higher than that of meal, so that more tonnage can be carried by truck.

- The composition of the pellets as obtained from carefully blending and mixing ingredients remains fixed, no segregation of e.g. additives occurs.

However, the use of shaping equipment, like pellet presses, requires additional costs in terms of energy demand and the costs for the necessity of additional equipment like; a boiler to generate steam, conditioning equipment, a pellet press and a cooler. In light of the fact that after mixing and blending already a complete diet in the form of mash exists, additional costs for investment in pelleting equipment should be considered with respect to the gain that can be achieved due to incorporation of the pelleting process.

Making up mash in the form of pellets originates from almost 60 years ago, when Patton *et al.* (1937) defined the nutritional superiority of pelleted poultry feeds over mashes. Pellets are obtained by a pressing process in animal feed manufacturing. In former times, the mash was simply pressed between two rollers or in a cake press to obtain pellets/cakes without any preliminary treatment. Relatively low pressures were used and the feed did not heat up (Calet, 1965). In modern feed mills the mash is pelleted in a so-called roller-and-die pellet-press, both vertical and horizontal. Before entering the pellet-press, the mash is subjected to some form of pre-treatment before granulation such as mixing with molasses or fats (Beumer 1978; 1980a,b,c,d), conditioning with steam (Skoch *et al.*, 1981) or the use of an expander (Veenendaal, 1990; Pickford, 1992) to increase temperature or moisture level (Friedrich and Robohm, 1969; Maier and Bakker-Arkema, 1992). When steam is used, the temperature of pellets after leaving the die is generally higher in comparison with that of the conditioned meal due to the frictional heat in the die. Finally, pellets are cooled with ambient air.

The physical quality of feed pellets is important for a number of reasons. First of all, transportation and handling in both factory and on farm situation require pellets of a certain integrity without fines produced by attrition stresses. Pellets of high physical quality must have properties which give a high nutritional quality for example in terms of higher feed intake and, perhaps, an improved nutritional value (Stevens, 1987; Skoch *et al.*, 1983; Koopmans *et al.*, 1989a,b).

Pellets also need to have a basic form of physical quality in terms of e.g. hardness and durability to withstand the rigors of transportation. Hardness is the force necessary to crush a pellet or a series of pellets at a time; durability is the amount of fines returning from pellets after being subjected to mechanical or pneumatic agitation. Such quality parameters can also be used to evaluate the effects of diet formulation, conditioning, expander treatment, pellet binders, die selection, etc. (Pfost, 1963).

For optimization of product quality in terms of physical characterization, knowledge of fundamentals for aggregating particles of different size, hardness and shape is needed. Therefore it is important to understand how particles bind and to gain insight on binding properties and

binding mechanisms in granulates or pellets and their behaviour during transportation and storage. Finally, this will reveal information that can be used as a basis for the criteria used to evaluate pellet quality.

The objective of this series of studies is to describe the contribution of causative factors that contribute to the physical quality of animal feed pellets. In this first article, criteria for pellet quality are evaluated. An overview is given of the binding mechanisms involved in pelleted feeds. In addition, different devices are discussed that are used in the compound feed industry to evaluate the hardness and durability of the pellets. Some general recommendations are given on what should be taken into account when using the different methods with which pellet quality can be evaluated.

In a second article and third article (Thomas *et al.* 1997, 1998), the physical quality of pellets is discussed in view of the contribution of raw materials and their components to the desired properties of pelleted animal feeds. Furthermore, the effect of unit operations in preparing animal feeds (conditioning, pelleting and cooling/drying) and some of their inherent process variables are discussed.

Binding of particles

Mash feeds are often subjected to granulation (pelleting) in routine feed manufacturing. The subsequent stages in the pelleting process are meal conditioning, pelleting and drying/cooling. Meal conditioning is a prerequisite for the actual compression of the meal into pellets and can be controlled by its process variables such as temperature, time and moisture level. Proper conditioning therefore brings adhesive properties on the surface of meal particles thereby improving pellet quality in terms of a harder pellet or a reduction of fines produced from pellets during additional handling (Skoch *et al.*, 1981; Friedrich, 1977).

Various hypothesis have been postulated in order to clarify the factors that determine the structural integrity in a pellet (Rumpf, 1958; Knacke and Pohl, 1959; Friedrich, 1964b).

In general, causative factors that affect pellet quality are diet ingredient composition and its properties, process technology and specific pellet binders (Table 1).

Table 1: Causative factors affecting pellet quality

Factor	Dimension	References
Diet ingredient composition		
Physical		
- part. size (distribution)		Rumpf, 1958; Stevens, 1987; Mercier and Guilbot, 1974; Payne, 1977
- specific density	kg m^{-3}	Friedrich, 1964a
- bulk density	kg m^{-3}	Friedrich, 1964a; MacMahon and Payne, 1991; Tešić, 1977
- angle of repose	°	Mohsenin, 1986; Friedrich, 1969
- surface area	m^2	Knacke and Pohl, 1959; Friedrich and Robohm, 1969
Chemical		
- moisture	g kg^{-1}	Skoch *et al.*, 1983; Knacke and Pohl, 1959
- ether extract	g kg^{-1}	Salmon, 1985; Richardson and Day, 1976, Friedrich and Robohm, 1981c
- crude fibre	g kg^{-1}	Tešić, 1977; Friedrich and Robohm, 1981a,b
- crude protein	g kg^{-1}	Friedrich and Robohm, 1981a,b; Stark, 1990
- ash	g kg^{-1}	Friedrich and Robohm, 1981a,b
Functional		
- viscosity	Pa sec	Nissinen *et al.*, 1993; Keller, 1983
- protein solubility	%	Hermansson, 1979; Kinsella, 1979; Wood, 1987
- starch gelatinization	%	Wood, 1987; Smith, 1983; Heffner and Pfost, 1973
- diet ingredients	%	MacMahon and Payne, 1991; Israelsen *et al.*, 1981; Payne, 1977
Process technology		
Conditioner		Leaver, 1984
temperature	°C	Skoch *et al.*, 1983; Winowiski, 1988; Stevens, 1987
time	sec	Israelsen *et al.*, 1981; Mercier and Guilbot, 1974; Beumer, 1978, 1980a,b,c,d
moisture		
- steam	%	Melcion *et al.*, 1974; Maier and Gardecki, 1992; Stevens, 1987;
- water	g kg^{-1}	Friedrich and Robohm, 1970
Pellet press		
die specification		Stevens, 1987; Schwanghart, 1969, 1970;
- diameter to length ratio		Tešić, 1977; Drevet, 1972; Hanrahan, 1983/84
Cooler/drier		
air speed	m sec^{-1}	Robohm, 1968
time	sec	Maier and Bakker-Arkema, 1992
air moisture level	g kg^{-1}	Schwanghart, 1970
pellet layer thickness	m	Maier and Bakker-Arkema, 1992
Pellet binders		
Adhesion and cohesion between particles.		Van Zuilichem *et al.*, 1979a,b, 1980; Winowiski, 1988 Brüggeman *et al.*, 1964; Eberhardt, 1964; Payne, 1977

More fundamentally, pellet integrity can be examined by studying the binding of particles that is accomplished through solid-solid bonds between diet ingredient particles, the use of liquids (e.g. molasses) or the use of specific pellet binders. The best hypothesis to date is probably the theory of Rumpf (1958) and extended by Friedrich (1964b, 1977), who described causative factors by which feed particles are held together by various mechanisms (Figure 1).

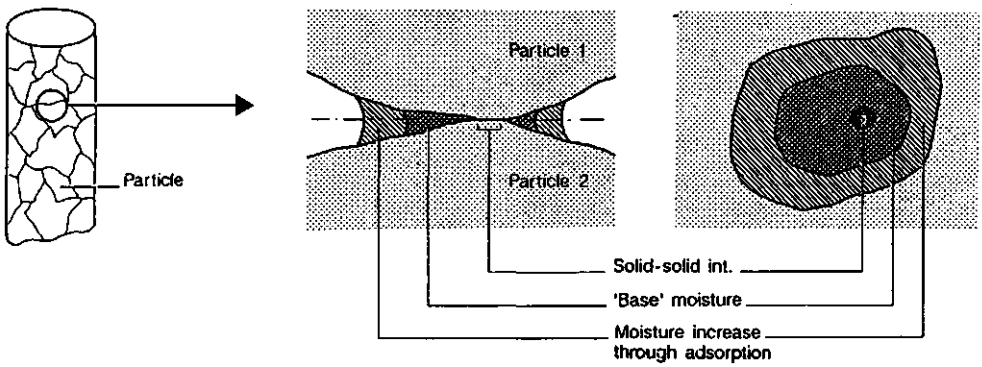

Figure 1: Model figure of general binding forces between two particles (Modified after Rumpf, 1958 and Friedrich, 1977).

The general mechanisms for binding feed particles can be divided in 'solid-solid' interactions between particles, capillary forces in a three phase system of water, air and solid material, so-called 'liquid necking', adhesive- and cohesive forces between particulates and binders and interactions between particles due to folding and plying.

'Solid-solid' interactions may consist of sintering, recrystallization or crystall growth of some ingredients, chemical reactions, melting of thermoplastic materials and solidifying afterwards into a crystalline state (Rumpf, 1958). Bonds between particles of the solid-solid interaction type are established mainly during the drying/cooling process depending on the applied conditions.

In a porous agglomerate, such as pellets, three separate phases can be distinguished: water, air and the solid material from the particles. These particles can be held together by liquid necking. The force of the bond is dependent on the surface tension (Δp) of the binding liquid γ ($N\ m^{-1}$), and the radius (r) of the neighbouring particle (m) (Rumpf, 1958). The binding pressure of the bond (N) is given by the equation of Laplace $\Delta p = 2 * (\gamma/r)$, in case the radii of the neighbouring particles is equal. In pelleted feeds this is normally not the case, since milling induces a particle size distribution (with subsequent larger and smaller particles) of the diet ingredients. From the above equation it can be easily derived that with decreasing radius of the particles, binding strength becomes higher. This is in line with the generally accepted principle

that with finer grind, better pellets are produced (Payne, 1978). The binding agent (water) between the different particles can be redistributed around the particles without loss of the established bond. Elevated temperatures cause redistribution of water via evaporation and condensation and this will increase the mobility of water between particles (Friedrich and Robohm, 1968). In the case that pellets moisture level is decreasing, then the moisture bridges will shrink and total binding forces will decrease due to a smaller number of moisture bridges, however force per bond will increase since water in the larger capillaries is evaporated first. Subsequently, it follows that the remaining water establishes bonds between smaller particles. From the equation given it follows that binding force between neighbouring particles is stronger with decreasing radius. In the case large quantities of water fill all pores, there will be a two-phase system of water and particles, with no capillary force present to maintain structure in the pellet. It is shown by Knacke and Pohl (1959) that an optimum exists in binding strength of clay agglomerates depending on the amount and surface tension of the water added.

Ingredients with (high) viscous properties will 'stick' particles together. Therefore distances between particles will decrease while at the same time the interacting surfaces between particles increases. Solid-solid interactions between different particles may come into effect when distance between particles is sufficient small, for instance, when pressure during pelleting is applied. Under such conditions, binding agents cannot be redistributed around the particles. When these bonds are present in large amounts in the feed, time will affect the binding between particulates and the structural integrity of the pellets may change with storage time (Pfost and Young, 1973).

'Van der Waals' forces may play a role in building up structural integrity in the pellet when particles are sufficient small (<50 μm, De Jong 1995; <60 μm, Rumpf, 1958; Friedrich, 1964b). In general, in animal feeds the fraction of these particles is neglectible small to substantially increase binding strength in pellets. When particle size increases, the effect of 'Van der Waals' forces diminishes and other binding mechanisms (capillary forces and binder materials) become more pronounced.

Electrostatical forces are neglectible between particles due to repulsional forces between materials. On theoretical grounds the magnitude of binding force between particles is 0.2905 smaller as with 'Van der Waals' forces (Rumpf, 1958).

Finally, particles and fibres may be folded due to the pelleting process and thus plied around each other (Rumpf, 1958). This phenomenon will also aid in building structure in the pellet. However, up till now no theoretical framework has been developed to quantify this type of binding (Rumpf, 1958). In Thomas *et al.* (1998) some of the effects of raw material components, for instance starch, protein and fibre are discussed with respect to their effects on physical pellet quality.

The above proposed mechanisms are useful to study the structural integrity of pellets. Which

of the different above mentioned effects is the most causative factor for pellet integrity, depends on the combination of raw materials used and the different operating/processing variables during pelleting, cooling and storage. All these variables affect pellet integrity, the magnitude of the different binding mechanism being dependent on the type of feed produced with its constituents and physical properties.

For quality control reasons, pellet quality devices have been developed to measure pellet integrity and - again - these devices can be further used to examine the effects of diet formulation and of process operation conditions on quality of pelleted animal feeds.

Pellet quality criteria and devices

When forces are involved that are statical or are present as a consequence of movements (dynamic forces), handling of cooled pellets causes attrition. Static forces for example may occur during bin storage while dynamic forces are present during screw or pneumatic transportation or during the filling of a bin. Attrition of feed pellets comprises two phenomena, fragmentation

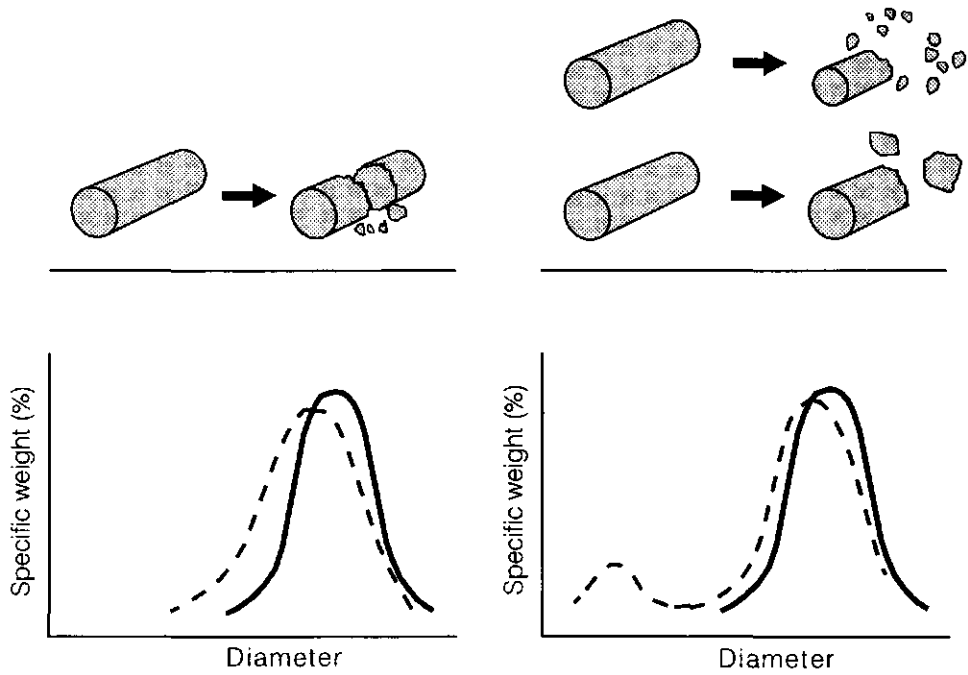

Figure 2: Theoretical effects of attrition of pellets (fragmentation and abrasion) on the particle size distribution curves (After de Jong, 1993).

and abrasion, both of which have consequences for its particle size distribution (Figure 2) and bulk density.

Fragmentation involves the fracture of pellets into smaller particles and fines at the fracture area. Fragmentation causes a small shift in the particle size distribution but will cause only a limited increase in bulk density or specific surface (de Jong, 1993). The stresses required to overcome the cohesive and adhesive forces in the cracktip and the amount of energy necessary, is dependent on the direction of the applied stresses, length of the crack and material dependent characteristics. The relation between these quantities has been described in detail by Luyten *et al.* (1992).

Abrasion involves the fracture on the edges or surface-unevenesses of particles. This type of attrition affects the particle size distribution. The fines produced are smaller than the original pellet size and form an additional peak in the particle size distribution curve (Figure 2). Depending on the amount of fines produced, bulk density may increase, since small particles occupy space in the large voids inbetween pellets, thereby decreasing pellet porosity.

Fragmentation of inhomogeneous materials always starts near the point of inhomogeneities due to the fact that local stresses and strains are always highest near such imperfections. Larger defects cause fracture to occur at lower overall stresses and strains. In homogeneous materials such imperfections can be errors in crystalline structure, small holes in or just below the surface (Luyten *et al.*, 1992; de Jong, 1993). When dealing with pellets, by nature of the material, particles of different sizes, hardnesses and various shapes are aggregated. It is at these cracktips that stresses will accumulate. When the local stress at the tip of a crack becomes higher than the cohesive or adhesive stresses, the crack (defect) in the material starts to grow, fracture starts and ultimately the material will fall apart. Fracture will propagate spontaneously as the deformation energy released is - at least - equal to the energy needed to create new surfaces.

Attrition can be brought about by tension, compression and shear stresses, compression being most important when related to animals feeds. Shear and tension tests may give much information but are often more difficult to perform (Luyten *et al.*, 1992). In practice, therefore, more emphasis has been put on compression devices. The actual assessment, however, when pellets are subjected to attrition by means of different devices are derived properties of the more fundamentally desired quantity 'energy' needed for fracture or abrasion of pellets. Actual routine assessments are often expressed just as 'kg force' after simulation of impact devices (mainly fragmentation) or as 'percent fines returned' after simulation of transport (mainly abrasion).

Several types of empirical developed devices for the routine evaluation of pellet quality have been developed (Melcion and Delort-Laval, 1981). In general, these routine devices test the hardness of pellets or their durability or resistance to attrition stresses (Pfost, 1963). Some important devices are discussed in the following paragraphs with the often used quantities

'hardness' and 'durability' as pellet quality parameters.

Hardness

Hardness is a quantity which is important for the nutrition of animals since hardness may play a role with preference of animals (Skoch *et al.*, 1983). Also, availability of nitrogenous components for intestinal absorption has been reported to be affected by hardness (Čuperlović *et al.*, 1973; Čuperlović, 1973).

In the case of statical pressure one may simulate the forces on the pellets. The statical pressure is caused for example by the weight of the pellets on top of the lower ones. In wide silo's where a part of the pressure cannot be relieved to the walls of the silo, breakage of the pellets on the bin bottom may occur.

Studies of Knacke and Pohl (1959) using clay minerals showed effects of water content on hardness of the agglomerates. The hardness of the agglomerates is proportional to the surface tension of the pelleting fluid and inversely related to the particle-size distribution. They showed that with an increase in the filling of the pores with water, an optimum value for hardness exists. This optimum is dependent on the surface tension of the pelleting fluid. Hardness is determined by using equipment which measures the force needed to fragmentate a pellet. In general, one can distinguish between tension, compression and impact based devices in which the compression component is the most important one.

Test devices for hardness have been developed for both scientific objectives and for on-line application (Anonymous, 1992) in routine animal feed manufacturing.

Early devices for testing hardness were developed by McCormick and Schellenberger (1960) and Young (1962). Young used a 'model handling system' in which pellets could be subjected to typical actions encountered in the handling of pellets. This model system was further used as a control for comparison of various other test devices (see Pfost, 1963) such as the Farmhand tester, tumbling can device and the Stokes hardness tester.

Nowadays, several devices are available for the evaluation of product hardness, each of which have different attrition or operating mechanisms (Table 2).

Table 2: Generally applied test devices for pellet hardness, their attrition mechanisms and operation.

Principal force applied at	Attrition mechanism	Operation[a]	Force	Device example	Reference
One point	Fragmentation	Compression (Axial tension)	Static	Kahl tester	Melcion and Delort-Laval, 1981
One point	Fragmentation	Compression (Axial tension)	Dynamic	Schleuniger	Beumer and Vooijs, 1993
One point or whole pellet	Fragmentation	Impact	Dynamic	Pendulum	Jindal, 1976
Variable	Fragmentation and/or shear	Compression (Radial tension)	Dynamic	Instron	Luyten, 1992
Variable	Fragmentation and/or shear	Compression/ Shear (Axial or radial tension)	Dynamic	Kramer shear press	Anonymus, 1970

[a] Depending on standard conditions

A first and common device in industry to test pellet hardness used is the '*Kahl*' device (Figure 3), analogous to the early developed, manually used Stokes tester. In the Kahl device, a pellet is inserted between two bars, and by increasing statical pressure applied by means of a spring, the force needed to crack the pellet is determined. The average of 10 measurements is referred to as the 'Kahl-hardness' of the pellet. Up-to-date devices for Kahl hardness imply the use of an automated version to generate the statical pressure.

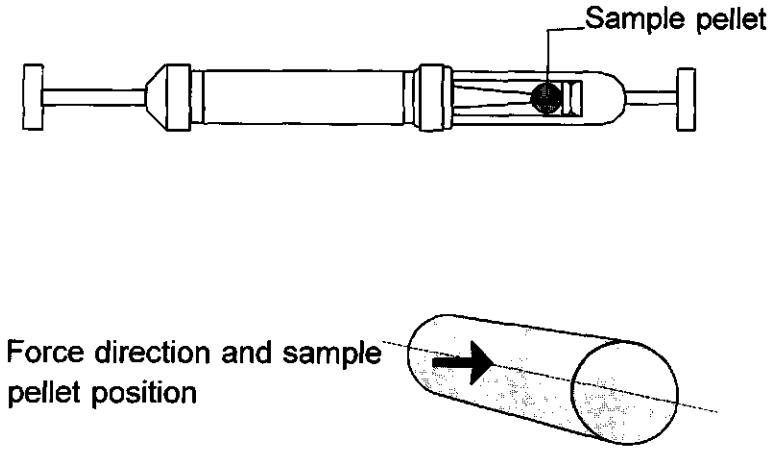

Figure 3: Kahl pellet hardness tester.

The *Schleuniger* test apparatus comprises a steel moving ram with a width of approximately 2 mm of the measuring part. Individual pellets are placed between the moving ram and a flat anvil. The moving ram will be pushed against the pellet by an electrical driven spindle with an increasing force. The force needed to fracture the pellet is recorded by a force transducer and registered. Measurement of Schleuniger hardness is normally performed in tenfold and is expressed in (kilo)pounds.

A further device which can be used to determine pellet characteristics is the *Pendulum* (Figure 4). The pendulum is an impact resistance testing device, by which the amount of energy needed to fracture a pellet can be investigated. The basic principle of a pendulum impacting device consists of a swingarm with a sample holder. From a known height this arm with the clamped on material of interest is dropped against a non-deformable and heavy block. The physical characteristics of the material under investigation that are recorded, are:

- Angular position of the pendulum arm before and after impact.
- Peak deceleration or force due to impact.
- The duration of the contact.
- The area of contact.

Using a pendulum type impacter several damage resistance characteristics of the used materials can be determined such as hardness, dynamic yield pressure and dynamic elasticity modulus (Jindal and Mohsenin, 1976). Not much research has been conducted in which physical quality of feed pellets is determined using a pendulum. Van Zuilichem and Stolp (1976) used the pendulum impact test device to asses the hardness (mechanical strength) of extruded pellets of maize grits and extrusamyl; a potato starch derivate.

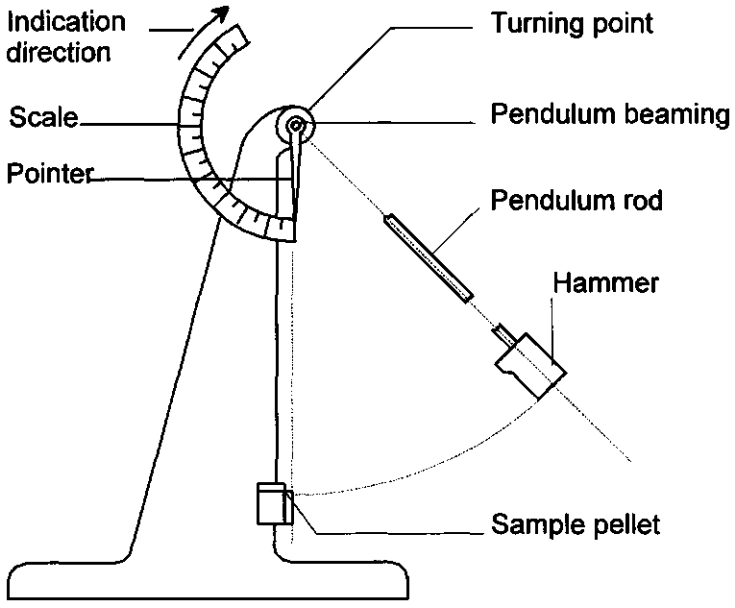

Figure 4: Pendulum pellet hardness test device.

Other devices use both compression and tension, and comprise so-called 'universal tension and compression' apparatus (*Instron; Overload dynamics*). Their main advantage is the accuracy with which the different materials can be subjected to the different tests known in the field of feed and food engineering. These devices consist of a fixed plate containing a load cell and a moving bar with variable speed. On this instrument measuring bodies of various geometry (either knife or plate for example) can be fitted and uniaxial compression, uniaxial tension, 3-point bending and cutting experiments, respectively, can be performed (Luyten *et al.*, 1992). For the testing of pellets, uniaxial testing (plate) and cutting (knife) experiments are most appropriate. The force units needed to break the pellet (or a series of pellets) is recorded as a function of time. Both single (see example Figure 5, plate) and multiple sample pellet(s) can be tested.

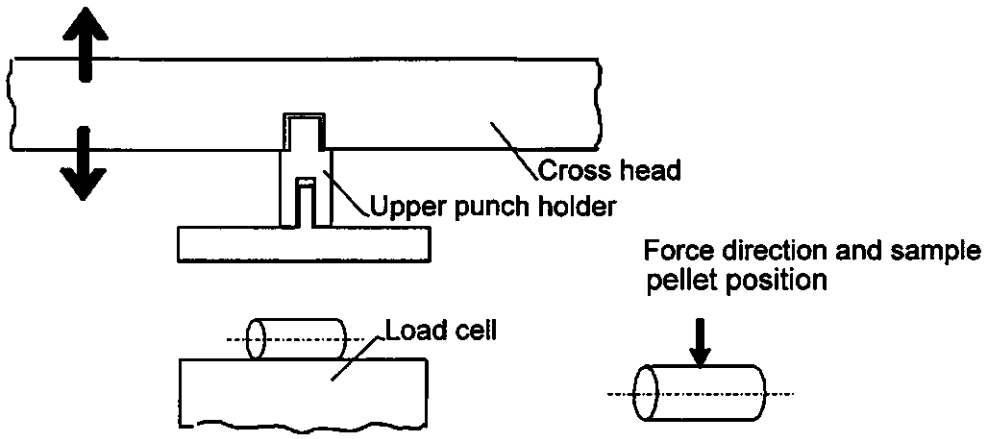

Figure 5: Universal compression test device.

The *Kramer shear press* (Anonymous, 1970) consists of a hydraulic ram on which a measuring body with various blades is mounted (Figure 6). The test material is inserted at the grid bottom of the shear box. When the ram comes down, the material is compressed and sheared between the intermeshing blades and grid. The force units are recorded as a function of time and from this

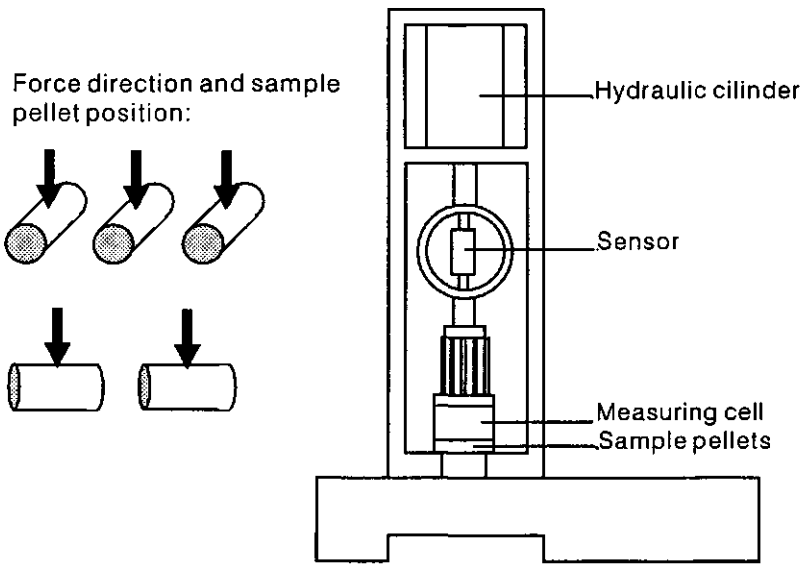

Figure 6: Kramer shear press for measurement of pellet hardness.

graph the maximum force can be read and the energy necessary for shear can be calculated. The force exerted on the material is recorded by means of a proving ring and transducer. The speed of the hydraulic ram can be adjusted over a relatively limited range and is in the same order of magnitude as with the Instron.

Table 3: Effects of processing conditions on durability and hardness of a barley pellet according to different devices (Means and coefficient of variation; Van der Poel and Thomas, 1993, unpublished results).

Temperature	Hardness			Durability	
	Kahl (kg)[a]	Instron (Newton,N)[b]	Kramer (Pounds, lb)[c]	Holmen (%)[d]	Pfost (%)[d]
*Die 5*25 mm*					
70°C	6.6 (52.7)	125 (22.9)	163 (5.8)	75.7 (1.1)	91.8_{+24} (0.2)
80°C	6.6 (21.8)	161 (6.4)	190 (5.0)	91.3 (0.2)	96.7_{+24} (0.1)
90°C	7.6 (10.8)	166 (4.6)	192 (3.1)	92.8 (0.2)	97.4_{+24} (0.0)
*Die 5*35 mm*					
50°C	7.6 (13.9)	155 (9.4)	192 (6.2)	94.1 (0.1)	97.8_{+24} (0.0)
60°C	7.8 (15.4)	164 (6.7)	206 (5.0)	94.3 (0.1)	97.9_{+24} (0.1)
70°C	7.8 (15.6)	172 (6.9)	208 (3.8)	94.0 (0.1)	98.1_{+24} (0.0)
80°C	7.8 (16.4)	175 (6.2)	203 (6.8)	94.5 (0.2)	98.2_{+24} (0.0)
90°C	8.0 (23.7)	176 (5.0)	204 (6.5)	94.0 (0.8)	98.2_{+24} (0.1)

[a] Single pellet measurement; 10 replicates.

[b] Average of 5 pellets each, 10 replicates.

[c] 15 grams of pellet sample, 5 replicates.

[d] Percentage of pellets returned, 2 replicates.

In Table 3, some data on hardness and durability of barley pellets are given as determined using different devices. Pellets of barley were manufactured using a Laboratory CPM pellet press and two dies. Different temperature conditions, by the aid of steam, were used. From these data it is clear that Kahl hardness shows considerable variation as can be gathered from the high coefficient of variation (CV). Using the Instron or Kramer shear press reduces the CV. However, with these devices more pellets are sheared during one test which will decrease the CV since it is calculated as the variation in multiple tests. It is noted that under the conditions of this experiment the CV increases when hardness is decreasing.

Durability

Durability is another physical quality parameter of feed pellets. The devices used for the determination of durability measure the amount of fines returned from a batch of feed pellets under standardized conditions.

In practical feed manufacturing, pelleted feeds are subject to shearing and abrasing actions during transportation. This induces fines in the feed. For the purpose of feeding ease, pellets need to have a certain resistance against the stresses exerted on it during transportation and distribution for the animals. In the past decennia, the mechanical transport of feed changed from relative low speeds to high speed pneumatic transport, thereby changing the order and magnitude of forces on feed pellets.

Pellets are sensitive to shearing actions at the places where they are cut off after leaving the die. Through this impact new surfaces are created which are sensitive for further deterioration. Improper cooling may increase this sensitivity to further deterioration. Pellets that are not properly cooled can have a reduced durability due to stresses in the pellet between the (cooled) outer layer and the (still) warmer center. Up to a certain level, relative cool air takes up moisture and heat from the pellets during the cooling process. In a steady state, the same amount of moisture (and latent heat) is transported through capillaries from the inner part to the surface. When air speed is increased, more water and heat will be removed from the pellet surface than can be delivered by the capillaries; a brittle outer layer emerges with physical properties differing from those of the inner kernel, the latter being warmer and more viscous. These differences in physical properties create stresses in the pellet which cause the outer layer to crack under less optimal conditions. These cracks will allow for an easier formation of fines.

Robohm and Apelt (1985, 1986) conducted a simulation experiment for pneumatic transport to elucidate the effect of air speed (m/s), air load (kg pellets/kg air) and pellet diameter on the amount of fines appearing. They found that increasing the air speed from 10 to > 30 m/s increased the amount of fines with approximately 3.5 percent. On the other hand, higher air load (more kg pellets per kg air for transport) decreased the amount of fines. Furthermore, the authors showed that pellets with small diameters (3 mm) proved to be more susceptible to breakage than larger pellet diameters (6 mm).

Several instruments are nowadays available for the evaluation of durability of pellets. In principal, these devices bring about attrition stresses that are exerted to pellets, due to either mechanical or pneumatic transportation (Table 4). Similar to hardness devices, the test devices for durability have been developed for both scientific objectives and on-line application in the factory. Already in the early sixties, a rather complete model handling system was used (Young,

1962) followed by the development of tumbling can devices (Gutekunst, 1962; Pfost and Allen, 1962).

Table 4: Generally applied test devices for pellet durability and their mechanisms.

Principal	Attrition mechanism	Operation*	Device example	References
Mechanical resistance	Abrasion	Surface flow	Pfost tumbling can	Pfost, 1963
Vibration	Abrasion/ Fragmentation	Surface flow/ Impact	Sieve	de Jong, 1993
Pneumatic resistance	Abrasion	Impact Fragmentation	Holmen Surface flow	Major, 1984

* Depending on standard conditions

According to the procedure of Pfost (Pfost and Allen, 1962; Pfost, 1963) durability is determined by inducing fines through an abrasing action of pellets shearing over each other and over the wall of drums (*Tumbling can device*: Figure 7). The procedure is standardized by using a drum with specified dimensions, in which fivehundred grams of sieved pellets are inserted. After tumbling for 10 minutes at 50 rpm, the pellets are subsequently sieved and the amount of fines passing a sieve with a grid size 'just smaller than the nominal pellet diameter' (Pfost, 1963) is determined. In practice, grid-sizes are used of 0.8 * pellet diameter. Durability is then expressed as the ratio of the weight after tumbling over the weight before tumbling, multiplied by ten. The resultant figure (between 0 and 10) should be rounded of to two significant digits. Nowadays, however, durability is normally expressed as a percentage which gives the amount of fines returned or the amount of pellets recovered. In the original description, a subscript is added depicting the time of physical analysis of pellets after their manufacturing in hours. Subscript +24 indicates durability results from pellets out of the cooler for more than one day, -1 indicates results from warm pellets. In scientific publications, however, the subscript - an important indication for comparison reasons - is not always given. Nowadays, automated devices exist to monitor on-line the durability of pellets during their manufacture (Anonymous, 1984a). Often these devices incorporate a fast cooling procedure to quickly obtain physical quality figures that can be used to optimize process parameters of that specific batch.

A *sieve* is a further device for measuring durability. Although not all standard procedures

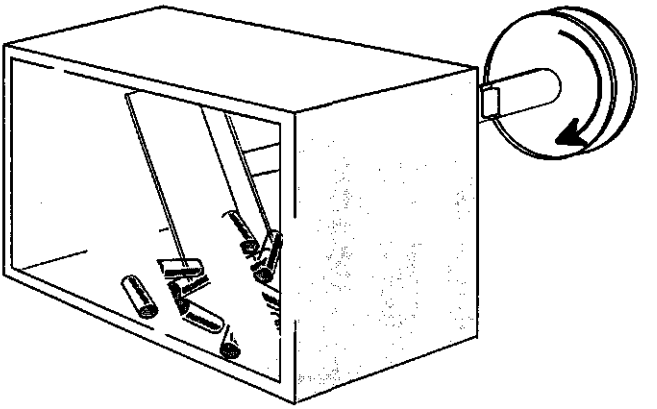

Figure 7: Pfost tumbling can device for measurement of pellet durability.

for sieving are suitable, those which comprise the use of balls between the screens can be used. By vibrating a pellet sample in a sieve apparatus, abrasion and perhaps fragmentation will occur through the action of (rubber) balls. Variables in this device are the sieve openings, the number of balls, the amplitude, screening time and eventually the use of interval sieving. Particle size

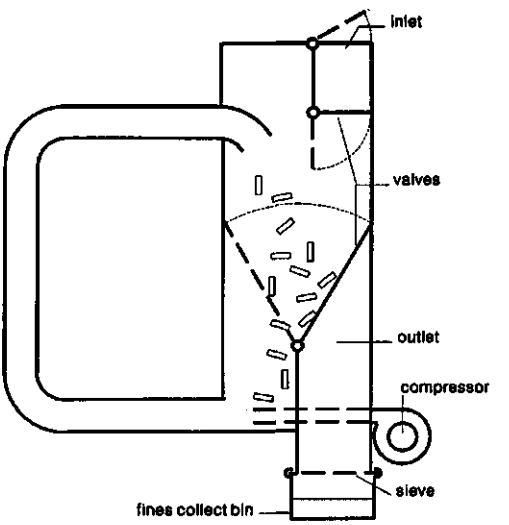

Figure 8: Holmen pellet durability tester.

distribution obtained by dry sieving may add useful information when the influence of sieving (under standardized conditions) for example is expressed in terms of the modulus of fines or the

modulus of uniformity before and after sieving (Pfost and Headley, 1976).

The '*Holmen*' pellet tester (Major, 1984; Figure 8) simulates a more rigorous treatment of pellets by pneumatic handling. In this apparatus hundred grams of sieved sample of pellets are introduced in a stream of air. For a standard time (0.5 to 2 min.) this air together with pellets is circulated through right-angled bends, impinging repeatedly on hard surfaces. Pellet attrition will then occur. After treatment, the product is sieved again using a sieve with an opening of approximately 80% of the pellet diameter. Standards have been developed for the testing time in relation to the pellet diameter under investigation. Many people refer to the Holmen pellet tester as a device for measuring durability of pellets. Closer examination of pellets under investigation reveals that fines produced by the Holmen tester comprises a 'mixture' of fines derived from both abrasion and fragmentation.

Not much information can be found on the correlation between measurements conducted on all of the types of equipment mentioned, except for the Holmen durability tester and Pfost Tumbling Can. Data from McKee (1990), indicated that for the Holmen pellet tester there was a lineair decrease in durability, ranging from approximately 95% to 60 % with testing time, up to 5 minutes. The Pfost tumbling can showed a curvilinear decrease, ranging from approximately 98 to 91% with time, up to 20 minutes. From these results it can be concluded that the Holmen pellet tester gives results in a wider range and in a shorter timespan than Pfosts tumbling can.

Wood (1987) found in his research a log-linear relationship (R=0.94) between hardness tested with the Kahl device and durability tested with a Holmen pellet tester. These relations may be found within certain diet formulations. However, because of the fact that hardness and durability are two distinct quantities, differences in relationships may be found between different diet formulations. These differences may become more distinct when new types of equipment are used to manufacture pelleted feed, e.g. expanders.

Other criteria

In the former paragraphs some of the most widespread measures of physical quality from a manufacturers point of view are evaluated. However other criteria exist to evaluate feed physical properties. These criteria are for instance water absorption index (WAI), water solubility index (WSI), and sinking velocity (SV), all criteria being useful in e.g. fish feeding. Other criteria well related to properties of the raw materials from which feeds are manufactured are the angle of repose, bulk density and specific weight. These criteria can be used to characterize the different feeds or feedstuffs. Some of these criteria can be related to complete feeds as well. For a more fundamental description of both methods and theories on physical properties related to characterizing plant and animal materials, the reader is referred to Mohsenin (1986). Since these

criteria are not specific for pellets as such, and thus not within the scope of this study, they are not discussed.

Discussion

Different methods for the evaluation of the attrition behaviour of pellets supply different information. It is obvious that it is not possible to use each type of device with every type of feed pellets. The choice for a certain method therefore is partly determined by the objective of the measurement, related to handling or nutritional purposes or to study simulated production of fines caused by either fragmentation or abrasive stresses.

The described methods for the evaluation of physical quality of pellets have been shown to be tests that use stresses brought about by bending/tension or compression. In the context of animal feed pellets, bending tests and tensile tests in general may give more information compared to compression test (Luyten *et al.*, 1992) but are of less value due to the type of attrition that feed pellets undergo between manufacturing in the feed mill and the animal feeding trough.

The compression test methods more generally employed in the routine evaluation of pellet attrition, more or less reflect a mixture of measuring fines produced by fragmentation and abrasion but what is normally distinguished by the terms 'hardness' and 'durability', respectively. There may be a relationship between hardness and durability (Wood, 1987) and between the results obtained by different devices for durability (McKee, 1990). However, these relations may only hold for a given feed composition. The relationship is strongly influenced by the diet ingredient composition of the pellet and the used pelleting conditions. With all devices applied, pellets are subjected to a change in particle size distribution. Therefore, the addressed question should not be 'what is the hardness or durability of feed pellets' but from a scientific point of view 'which combination of fragmentation or abrasion reflects at best the way feed pellets are handled, conveyed or stored'. The subsequent choice for a type of device also depends on the outline of the factory, whether mechanical or pneumatic transport is used, the form and shape of storage bins, and - last but not least - the ultimate consumers demand (whether man or animal) for a certain physical quality of pellets. For instance, Skoch *et al.* (1983) found differences in feeding preference of pigs, when feeds in different forms were given in a 'choice-feeding' experiment. Pigs preferred pellets over meal but softer pellets over harder pellets.

The large variation that exists within results of e.g. hardness devices (Table 3) is partly due to the fundamental misunderstanding on breaking behaviour of agglomerates of biological origin. Analysis of pellet hardness figures with data from five experiments confirmed this large variation (Table 5). As shown by Fell and Newton (1970), empirical modification would permit

to decrease the large variation that can be found in fragmentation-type measurements that employ diametrical compression such as the Kahl test, Schleuniger test and (partly) the Kramer shear press. Carefull application of the standard conditions under which the device is operated, is therefore recommended. It may be relevant to adjust these standard conditions in particular for those devices, that show a large variation in pellet quality results.

Table 5: Test criteria for pellet quality measuring devices.

Device	Test criteria		
	Repeatability[a]	Validity	
		Fragmentation	Abrasion
Compression devices			
Kahl	--	yes	no
Schleuniger	-/+	yes	no
Pendulum	+	yes	no
Universal tension compression[b]	+	yes	no
Kramer-shear press	+	yes	yes/no
Attrition devices			
Pfost	+++	no	yes
Sieving	?	yes/no	yes
Holmen	+++	yes	yes

[a] Repeatability on the basis of coefficient of variation (CV). The CV ranges between (M. Thomas, unpublished results): +++, 0 - 1; ++, 1 - 5; +, 5 - 10; -, 10 - 20; --, > 20.

[b] Examples: Instron and Overload dynamics

Current used routine devices for durability are a compromise between measurements that give a practical value for pellet quality (on-line level) and measurements that have scientific value. For example, durability measured according to the tumbling can method (Pfost, 1963) is an actual measurement of abrasion. The pneumatic resistance test (Holmen tester), however, is an actual measurement of both fragmentation and abrasion (Robohm, 1987).

Since transport and handling involve both fragmentation and abrasion phenomena it would therefore relate more closely to quantities measured with the Holmen pellet tester than with the tumbling can device, since the first device acts as an 'in between' simulator. For fundamental directed studies, some devices may give no valid estimate of pellet quality as a function of various processing conditions (insufficient resolution) but are still useful as a quick, routine device for both processing control and/or quality control in the factory. The latter means that the use of a certain device method appears suitable for process control within a certain factory when used under standard conditions.

In the authors point of view, it should be recognized that the time of physical quality measurement after manufacturing of pellets took place should always be indicated, as included in the original description for durability (Pfost, 1963). In fact, for each measurement it is important irrespective the used device. In addition, (specified) standard conditions need not to be similar to guideline recommendations. While the individual feed manufacturer should set standards for his own situation, science asks for a standardized approach for comparative reasons. In the case a number of pellets is examined, the CV of data is commonly lowered by excluding the highest and lowest value. This procedure is rather commonly applied by for example the Kahl and Schleuniger tests. From a scientific point of view, this procedure, however, is highly questionable.

It can be concluded that only general descriptions can be given on the magnitude of the different binding mechanisms. It appears that crystallisational processes taking place in the pellet during cooling/drying and capillary forces account for the largest part of binding particles (Schwanghardt, 1970). However, other non-discussed phenomena as for instance glass-formation may play a role as well in creating structure within agglomerates. Within the scope of animal feed manufacturing these are still poorly understood. More research is needed to gain knowledge on the effects of different processing conditions and the magnitude of the different binding mechanisms.

The devices used in animal feed manufacturing most of the time yield a figure which merely reflects a measurement which consists of a mixture of fragmentation and abrasion, for instance the Holmen pellet tester. For practical purposes the aim should be to identify the method which at best reflects the mixture of hardness and durability which is closest related to the figures derived from the specific plant at which the feeds have been produced. For this purpose empirical equipment has been developed which at present is commonly used in animal feed production: Pfost's tumbling can, the Holmen pellet tester and the Kahl hardness tester. Recently, on-line test devices have been developed for use during the manufacturing stage. Therefore variation in pellet quality during a production run can be evaluated and subsequently used to adjust pelleting conditions during manufacturing.

For scientific evaluation, equipment is needed with which measurements can be performed under standardized conditions for one unique characteristic, for instance hardness. Still, using standardized conditions, results found with 'scientific' equipment as for instance Instron, Overload dynamics or Kramer shear press, are often not better as found with empirical tests. This is due to inherent variability of the pellets produced in the factory and to limited knowledge on breaking behaviour of materials of biological origin. The main advantage yet in using 'scientific' equipment is its use via standardized conditions and with measuring one property, whether fragmentation or abrasion. This will ensure pellet quality measurements which are comparable between and within pellet manufacturing systems.

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2

Physical quality of pelleted animal feed. 2. Contribution of processes and its conditions.

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Physical quality of pelleted animal feed. 2. Contribution of processes and its conditions.

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Abstract

The effects of changes in process parameters and their effect on pellet quality in terms of hardness and durability are discussed. The pelleting process in this respect is the combination of conditioning, pelleting and cooling. The parameters discussed with respect to the conditioning process are process variables such as steam and water and system parameters such as residence time and pressure.

Parameters during the pelleting process that can be adjusted or influence pelleting properties of a feed mash are layout (e.g. flat-bed vs. ring-die pellet press) and dimensions, roller and die assembly and die velocity of the pellet press. The effect of the changes in one or more parameters and its effect on pellet quality (durability and hardness) is however often a matter of judgement and experience of the operator. For instance, a certain increase in the amount of steam added to a feed mash generally improves pellet hardness and durability. Increasing the amount of dissipated power in the feed mash, generally has a similar favorable effect on pellet quality. The extent to which pellet hardness and durability rise is however dependent on feed formulation and other parameters as temperature of the feed mash and cooling air characteristics. The latter parameters can be measured but their relation to the pelleting process is often not clear.

The cooler is a decisive factor in determining the ultimate physical pellet quality and mainly determined by the bed-height of the pellets in the cooler, pellet-size, air-flow and air-characteristics during the cooling period.

The use of modern design conditioning systems to operate at a wide range of processing and system variables means that pellet quality is dictated more by equipment than by diet formulation.
However, use of such conditioning systems must always be justifiable in terms of cost and pellet quality, rather than a result of the dictates of fashion.

It is concluded that the amount of steam is a more decisive factor than steam pressure. In addition, it seems that individual steam supply of a factory has a larger influence on the measured pellet hardness and durability than would be expected from theoretical relationships concerning steam in an ideal situation. Although water has binding properties as well, it is concluded that the use of steam over water is by far superior to produce good quality pellets. The additional heat included in the meal permits changes in physico-chemical properties which lead to more durable and hard pellets. In addition, it is concluded that equipment which incorporates some form of hold-time enhances the possibility to incorporate more liquids, without detrimental effects on pellet quality. The use of pressure to alter physico-chemical properties of the feed in combination with water and heat, and the use of pressure to pre-densify the feed mash prior to pelleting seems to be important in obtaining a good quality pellet.

Finally, pelleting is not the sum of conditioning, pelleting and cooling steps, but should be considered as an integral system which performance is dependant on interrelations between the three unit operations: conditioning, pelleting and cooling. However, these interrelations with respect to the different formulations used, are still poorly understood. The use of decision support systems and process optimization procedures may nowadays greatly enhance the opportunity to obtain the best possible quality of pelleted feeds with minimal use of labor and energy for a given feed formulation.

Introduction

In addition to functional properties of the feed ingredients, the choice of equipment and processing and system variables used in the manufacturing process determine the physical quality of a pelleted animal feed. Amongst these, the dimensions and lay-out of the equipment and the range of process variables of the used apparatus are important. These two factors determine the eventual range of operating conditions that can be reached and hence the degree of flexibility of an apparatus. For instance, a pellet press dissipates a certain amount of motor power into the feed mash. Depending on design, two-roller versus three-roller presses permit the use of a certain maximum amount of steam to facilitate pelleting. The expander also dissipates motor power in the feed mash, but its design permits a considerable higher amount of steam to be used in the pelleting process. In figure 1 a schematical overview is given on how process variables and raw material characteristics relate to the desired objectives via system variables and subsequent functional changes of the feed mash.

Conditioning requires a vast knowledge of the influence of the processing and systems parameters (e.g. water, steam and energy dissipation) and its induced changes in physico-chemical properties of a feed mash. When the requirements of the factory for a given set of processing and systems variables with respect to physical, nutritional and hygienic quality is set forward, a choice must be made from the wide range of conditioning equipment available, which meets these sets of processing and system variables. Likewise, the choice for conditioning/pelleting and cooling/drying equipment should be derived from requirements of the manufacturer for a given set of process and system variables, necessary to produce good quality feed (Fig. 1).

In this paper the effects of equipment and of differences in process and system variables are discussed and the way they affect the physical quality of pelleted animal feeds.

Conditioning

Conditioning in animal feed manufacturing can be defined as the process of converting the mixed mash with the use of heat, water, pressure and time to a physical state, that facilitates compaction of the feed mash. Conditioning will increase production capacity and simultaneously affects the physical, nutritional and hygienic quality of the produced feed (Skoch *et al.*, 1981).

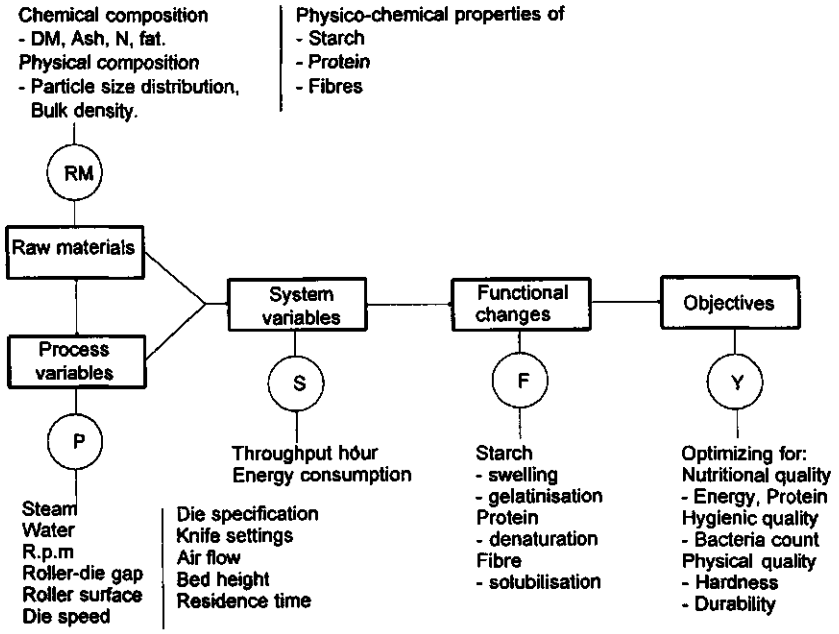

Figure 1: Schematical representation of the relation between properties of raw materials, process variables and subsequent system parameters and changes in the feed mash with respect of the objectives set forward by the manufacturer.

Heat and water added will alter components like starch and protein in the feed mash, in a way that binding properties come into effect (Thomas and van der Poel, 1996). The effect on changes in protein and starch properties are normally enhanced when some form of residence time is incorporated. Production output, energy consumption and pellet quality are related depending on the applied combination of process variables. Applying too much heat or water, however, will impair production capacity and pellet quality and may lead to plugging of the pellet press (Winowiski, 1985).

Properly conditioned feed mash will help the manufacturer in meeting the standards on pellet hardness, pellet durability and hygienic quality (Liu *et al.*, 1969; McCapes *et al.*, 1989) dictated by market and production while retaining or improving nutritional value of the feed. It should be denoted that conditioning is only one aspect in the production of pelleted feed and standards on hygienic, nutritional and physical pellet quality should always be evaluated against the background of the total manufacturing line.

The choice of conditioning equipment depends on several factors, such as the type of feed processed. Cattle feed requires a different way of processing in comparison to, for instance, pig

feed. Secondly, the choice should be based on the necessary range for processing and systems variables in the factory. When for instance cattle feeds are produced, the pelleting line can be set up to satisfactorily produce this cattle feed, given the constraints of the diet formulation such as high fiber. When a great number of different diet formulations need to be pelleted (e.g. cattle, pig, poultry and fish), the desired degree of flexibility with respect to the process and systems parameters requires conditioning equipment which includes a wide range of operating conditions. Thirdly, due to restrictions in construction height and limitations in the pelleting line itself, use of conditioning equipment which requires a huge amount of space can be limited. Within these main three criteria, the feed manufacturer has to choose equipment with its own specific advantages and disadvantages.

In addition to general outlines of conditioning equipment, the effects of the different process (e.g. steam and water) and systems (e.g. residence time and energy dissipation) variables and their effect on physical pellet quality are discussed.

Steam addition

The conditioner most widely used in animal feed manufacturing to incorporate water, steam and/or other liquids is a barrel type conditioner. It consists of a hollow shaft. Inside, paddles are mounted on a revolving axis which blend the steam and liquids with the feed mash to give the mash its desired properties. Advantages of this type of conditioner are a relative ease of use and its short residence time ranging from 20 seconds (Audet, 1995) up to 255 seconds (Oechsner de Conink and Bouvier, 1995) depending on throughput, the amount of revolutions per minute of the paddle bar and the degree of fill (Oechsner de Conink and Bouvier, 1995). Problems due to incorrect maintenance or steam supply result in decreased pelleting performance (see study of Maier and Gardecki, 1992) and blocking of the die (Winowiski, 1988). Recent versions of this type of conditioner can be equipped with twin shaft paddle bars (Oechsner de Conink and Bouvier, 1995) or can be fitted with heater jackets (Audet, 1995).

The application of steam in animal feed manufacturing has long been recognized as a means to facilitate the production of good quality pellets (Friedrich and Robohm, 1968; Skoch *et al.*, 1981, Skoch *et al.*, 1983; Maier and Gardecki, 1992). The overall effect of steam addition is an increase in moisture content and heat of the feed-mash. Because of its gas state, steam is more homogeneously dispersed through the feed mash. During condensation of the steam, a thin film of water is created around the particles, which, together with the temperature increase, facilitates binding between particles. The effect may be twofold; first, the water itself may exhibit bonds via capillary sorption between particles (Rumpf, 1958; Friedrich, 1977). Secondly, the heat and water induces a wide range of physical and chemical changes, including amongst others:

Table 2: The effect of steam inclusion level and steam pressure on pellet durability and production characteristics

Steam		Durability (%)	Conditioning Temperature (°C)	Specific[a] production (kWh ton^{-1})	Remarks on production or diet ingredients	Reference
Pressure (kPa)	Added (kg ton^{-1})					
620.7	23.0	93.5	65	13.0	Constant production rate	Skoch *et al.* (1981)
620.7	33.0	96.5	80	9.8	„	Skoch *et al.* (1981)
0.0	0.0	79.1	27	26.4	„	Skoch *et al.* (1981)
620.7	26.4	90.6	65	10.9	Maximum production rate	Skoch *et al.* (1981)
620.7	35.5	93.8	78	9.0	„	Skoch *et al.* (1981)
0.0	0.0	69.5	21	27.4	„	Skoch *et al.* (1981)
204.0	-	97.4	66 - 89	-	> 700 g kg^{-1} soybean meal	Winowiski (1985)
136.4	25[b]	91.0	65	8.6	724 g kg^{-1} wheat in diet	Stevens (1987)
544.2	25[b]	90.3	65	8.9	„	Stevens (1987)
136.4	25[b]	57.5	65	7.5	724 g kg^{-1} corn in diet	Stevens (1987)
544.2	25[b]	57.6	65	8.2	„	Stevens (1987)

[a] Power consumption for pellet press.

[b] Calculated from 2.5 % moisture added during conditioning.

- Not determined in the tests conducted

can not be deduced from this experiment. There does not seem to be a clear relation between steam pressure and pellet quality, as is the case between the amount of steam and pellet quality. It may be the case, taking into the account the results of Maier and Gardecki (1992), that the individual steam supply of a factory has a larger influence on the measured pellet hardness and durability than would be expected from theoretical relationships concerning steam in an ideal situation.

Water addition

Water is generally added by means of a conventional barrel type conditioner. One of the contributions of water to the physical quality of pellets is the possibility to provide sorption bonds as described by Rumpf (1958) and Friedrich (1977). Rumpf (1958) derived different formulas for the effect of water and salts on the hardness of agglomerates. Knacke and Pohl (1959) evaluated the contribution of water and solubilized components on the hardness of clay agglomerates under low consolidating pressure (50 N cm^{-2}). The effect of solubilized components (detergents, nitrobenzol, benzol and methylalcohol) decreased the hardness of the clay agglomerates by lowering the surface tension of the fluid. Water without solubles was found to yield highest strength in the agglomerates. However, these results should be interpreted with care since consolidating pressures used in this experiment where lower than pelleting pressures normally applied in feed industry (up to 12300 N cm^{-2}; Schwanghart, 1969a,b). Furthermore, feed components may act different from the used clay minerals, since the clay minerals may be thought inert towards the used solvent.

Feed components exhibit different behavior when water is present. Gelatinization (Collison and Chilton, 1974; Chiang and Johnson, 1977; Lund, 1984), denaturation (Ledward and Mitchell, 1986; Kinsella, 1979; Martinez, 1979) and solubilization processes are facilitated by the presence of water. Subsequently, the water in this respect changes the (surfacial) structure of other materials to such an extent that binding between the feed particles becomes possible. In order to enhance binding properties and therefore increase pellet quality characteristics, often heat is a neccesary prerequisite (Table 3). Friedrich and Robohm (1969, 1970) found an increase in pellet durability when water was exchanged for steam. These results are in line with results of Wood (1987), who also indicated that water coming from condensing steam is superior with respect to pellet hardness and durability.

Table 3: The effect of exchanging water for steam on pellet durability

Moisture addition		Pellet durability[a] (%)	Reference
Water (%)	Steam (%)		
3	0	94.3	Friedrich and Robohm (1970)[b]
1.5	1.5	95.3	Friedrich and Robohm (1970)
0	3	95.6	Friedrich and Robohm (1970)
3.5	0	$96.4_{+0.5}$[b]	Friedrich and Robohm (1969)[c]
0	3.5	$97.3_{+0.5}$	Friedrich and Robohm (1969)

[a] Pfost tumbling can

[b] Pig feed containing 520 g kg^{-1} cereals.

[c] Finisher pig feed.

Water influences compression characteristics of materials as is shown by Mohsenin and Zaske (1976) and Nathier-Dufour *et al.* (1995). Water was the main determinant of compaction behavior in wheat meal as determined from the yield point. Particle-size and chemical composition as determined with an infra-red analyzer did not significantly affect compaction behavior. Inclusion of water decreased the force corresponding to the yield point (Nathier-Dufour *et al.*, 1995). However, in this study no durability or hardness figures are given with respect to the compacted wheat meal used in the tests. Mohsenin and Zaske (1976) also showed the effect of water on compaction behavior and attributed the amount of water to stress relaxation in the feed pellet or feed wafer. Water and holding time significantly affected stress relaxation of the pellet or wafer produced. In turn, relaxation of the stress after ejection from the die determines expansion, with higher residual stresses inducing a larger degree of expansion and subsequently a lower final density and a lower durability of the wafers.

It can be concluded that water enhances structural integrity of feed pellets firstly by means of capillary sorption of particles (Rumpf, 1958; Knacke and Pohl, 1959) and secondly via modification of the compression characteristics of feed (Mohsenin and Zaske, 1976; Nathier-Dufour *et al.*, 1995). However, water added as steam is by far superior over conditioning with water alone, since the additional heat modifies physico-chemical properties (gelatinization of starch, denaturation of protein) to such an extent that binding between particles is greatly enhanced resulting in improved physical pellet quality (Table 3).

Residence time

Only a few scientific articles deal with the subject of long-time conditioning of feed mash with the aid of ripening vessels. In the technical literature available, the positive effects of ripening, with or without steam, points to better quality feed pellets. More moisture or other liquids (molasses) can be added when a ripening time of 20 min is incorporated in the process line,

which is then followed by steam-injection, with no loss in productivity and feed quality (Beumer, 1980a,b,c,d). In case of ripening without steam injection before pelleting, pellet quality was lacking (Beumer, 1978). These effects were attributed to the diffusion of the liquids in the feed particles during holding in the vessel. The relative dry surface of the particles which appears will have a negative effect on the physical quality of the pellets. From this it can be concluded that long time ripening followed by steam injection permits higher uptake of liquids and improves pellet quality.

However, modern feed manufacturing involves the use of a high number of different animal diets that have to be produced in relative short time-spans. This means that equipment which incorporates some form of hold-time will decrease flexibility of the plant with respect to the number of feed changes per time unit. Interest is, therefore, raised in equipment with short hold-times (< 2 min) with the same favorable effect on pellet and nutritional quality.

Pressure elevation

Pressurised conditioners are used to deairate the feed mash. Subsequent compaction by a pellet press may then result in a decreased energy consumption of that press. Furthermore, the decrease in porosity aids in the amounts of bonds that can be formed in the pellet and may improve pellet hardness (Rumpf, 1958; Ouchiyama and Tanaka, 1985).

Double pelleting is mostly used for manufacturing cattle feed. This feed normally includes large proportions of fiber that often exhibits a resilient character. Depending on the type of fiber, pellet quality may be decreased. A double pelleting system consists of two presses, serially connected to each other. The first press, equipped with a conventional barrel type conditioner and a relative thin die, is used to pre-compress the feed. Actual pelleting is performed by the second press, equipped with a thicker die. Results after Robohm and Apelt (1989a) indicate that for double-pelleting the decrease in energy consumption for the actual pelleting press may be between approx. 2 and 8 kWh ton^{-1}. However, total energy consumption for both the pre-densifying press and the actual pellet press is approximately 8 to 13 kWh ton^{-1} (Robohm and Apelt, 1989a) higher, when compared with conventional pelleting. In Table 4 the effects of double or single pelleting on physical quality of a swine diet is depicted with inclusion of 4 or 6 percent fat in the mash (after Robohm and Apelt, 1989a). From this table it can be derived that especially at higher fat levels, double pelleting increases pellet quality as compared to conventional (single-press) pelleting.

Table 4: Effects of pelleting and double pelleting and its effect on pellet quality, shear strength and specific power consumption (Derived from Robohm and Apelt, 1989a)

	Fat inclusion level					
	4%		6%		6%	
	Double[a] pelleting	Pelleting[a]	Double[a] pelleting	Pelleting[a]	Double[b] pelleting	pelleting[b]
Hardnes (N / pellet)	65	40	25	20	80	48
Shear-strength (N cm^{-2})	40	40	24	15	40	30
Durability (%)	95	95	93	88	96	94
Specific power consumption (kWh $tonne^{-1}$)	27[c]	13	19[c]	12	23[c]	15

[a] 5*25 is pre-compression die; 5*40 pelleting die.

[b] 5*25 is pre-compression die: 5*80 pelleting die.

[c] Pelleting press and pre-compression press.

Recently, a new piece of equipment is introduced in the animal feed industry, the so-called expander. Such an expander consists of a conveying screw with mixing bolts mounted in the barrel. This slotted screw exerts a shearing, mixing and transport action into the feed. The difference between an expander and a single screw extruder is the moving die installed at the outlet of the expander, thus creating an annular shaped die.

Table 5: The effect of pelleting and expander-pelleting on durability and specific energy consumption of the press and expander for corn and wheat (after Robohm, 1991)

Treatments[a]	Raw material	Durability[b] (%)	Energy consumption (kWh ton^{-1})		
			Expander	Press	Total
Pelleting	Corn	66	not used	11	11
Expander pelleting[c]					
20 bar	„	85	15	8	23
30 bar	„	89	22	8	30
Pelleting	Wheat	92	not used	14	14
Expander pelleting[c]					
20 bar	„	91	17	8	25
35 bar	„	93	23	7	30

Pelleting during all tests was conducted on a 4 * 15 mm flat-die press.

[a]Conditioning with 2 % steam + 2 % water, [b]Durability after Pfost, [c]Counter pressure of the cone.

The position of the cone is controlled by the power take-up of the expander drive, resulting in differences in physico-chemical properties of the feed mash. The combination of different phenomena, densifying the feed mash, shear and mixing alter the structure to such an extent that binding between feed particles in the pelleting process is enhanced. This explains the observed trend of increased pellet hardness and durability (Table 5) of expander-pelleted feeds. Especially in the case of raw materials which may impair pellet quality, the use of an expander leads to increased durability figures. In case of raw materials having good pelleting ability like wheat, the additional energy consumption during expander pelleting may not be justified. Energy consumption of the press in series connected with the expander decreases compared to energy consumption of a pellet press without expander. However, total energy consumption was significantly increased due to the use of an expander (Table 5). This is in line with results of Michaelsen and Heidenreich (1992).

Table 6: The effect of cone counter pressure in the expander on pellet durability of animal feeds after expander pelleting.

	Diet Fat level ($g\ kg^{-1}$)	Production criteria			Durability	References
		Pressure (Bar)	Temperature[d] (°C)	Rate (Tonnes h^{-1})		
Swine diet[a]	not given	0	75	5.2	97.7	Robohm, 1991
		7.5	75	5.4	97.9	„
		14.9	85	5.6	98.1	„
		18.7	110	5.6	98.6	„
		18.7	95	7.9	98.0	„
Broiler feed[b]	60	40	70	-	99.6	Peisker, 1992
	90	40	70	-	99.1	„
	120	40	70	-	98.3	„
	60	80	70	-	99.4	„
	90	80	70	-	98.8	„
	120	80	70	-	98.2	„
Broiler feed[c]	70	40	70	-	99.3	„
	100	40	70	-	98.9	„
	130	40	70	-	98.4	„
	70	80	70	-	99.2	„
	100	80	70	-	98.8	„
	130	80	70	-	98.4	„

[a] Diet contained: wheat 320 $g\ kg^{-1}$, barley 200 $g\ kg^{-1}$, corn germ meal 120 $g\ kg^{-1}$, 100 $g\ kg^{-1}$ soy bean meal.

[b] Diet contained 350 $g\ kg^{-1}$ tapioca

[c] Diet contained 440 $g\ kg^{-1}$ tapioca

[d] Expander temperature for swine diet; conditioner temperature for broiler feed.

The extent to which the feed is altered by shearing and mixing actions is dependent on the process conditions under which the expander is used (Tables 5 and 6). Increasing the pressure by decreasing the annulus, the feed mash will receive a higher shear energy and subsequently the temperature of the feed mash rises. Together with the deairating action of the expander, improved pellet durability can be found, even at high fat levels (Table 6).

Other equipment exists which combines a predensifying and shearing action to the feed mash, for instance the mix-compress or compactor type devices. The first part of this type of conditioner is the mixing chamber, that is similar in design to a conventional barrel type conditioner. In the second part, the material is compacted by means of a roller assembly and pressed through an adjustable V-shaped gap. Temperature of the material may rise to about 110°C. A slight flash-off in the order of magnitude of 1% of water may occur (Van Bruggen, 1995). The residence time within the conditioner is approximately 30 seconds, which is long enough to eliminate most types of microbial contaminants (van Bruggen, 1995). No scientific results have been published in literature about this type of conditioner.

Pelleting

In the Netherlands the amount of pelleted feed in 1993 was 88.4 % of the total amount of 16.1 million tonnes of compound feed (Anonymous, 1994). Advantages and disadvantages of this key process in animal feed manufacturing have been described by Vanschoubroeck *et al.* (1971).

In the pelleting process, conditioned feed mash is pressed through a die. A majority of the pellet presses operated in feed manufacturing are of the ring die design. Different designs exist in which usually two or three rollers are used. In most designs, the die is revolving around the fixed rollers, although alternatives exist. A minority is designed as flat-die presses in which the die is static and the horizontal rollers rotate around a vertical axis while forcing the meal through the die plate. Die sizes may differ in their length to diameter ratio, normally expressed as width of the bore times the length of the die-hole. Increasing die-thickness or decreasing bore-width will increase the amount of shear which the feed mash receives. This is limited since a too high amount of shear (thick dies or small bore holes) will block the pellet press (Pfost, 1971).

The objective of the pellet press operator is to obtain feed pellets with a quality level sufficient to withstand the rigors of transport and handling at least possible cost in terms of energy consumption and wear. In connection with the conditioning process some adjustments can be made at the pellet press.

Roller/Die dimensions

Pressure generated in the die-hole of the pellet press depends on the coefficient of surface friction between feed mash and die wall, moisture content, relaxation time of the plastic deformable portion in the mash, die temperature and compressibility of the material (Tešić, 1977). These properties are influenced by die specifications like length of the bore and the diameter of the bore hole. However, the exact relation is unknown and in most cases difficult to measure and should be subject for further research.

When feed mash enters the die (in case of a pellet press with a moving, ring shaped die), a layer of material will build up on the inside of the die. When overrolled, the material is subjected to compression. The extent of compression is dependent on the height of the layer and the gap distance between the die and the roller. When pressure is exceeding the frictional force of the material in the die hole, a layer of material is added on top of the pellet under formation. The height of the layer of material that can be pelleted is dependent on the ratio between the radius of the die, the radius of the roller and a material dependent factor; the coefficient of friction between roller surface and the feed mash (Schwanghart, 1969a). The coefficient of friction (f) is a dimensionless factor determined by the ratio between the force of friction (F) and the force normal to the surface of contact (W) in the equation: f=F/W (Mohsenin, 1986). The coefficient of friction is dependent on material properties which are highly influenced by the conditioning process.

When h_G/R is reached: see fig. 2, material starts to 'squirt' out between roller and die. Material will build up in front of the roller and eventual cause blocking of the press. From this figure it follows that the maximum pelletable height of the feed layer (and thus throughput) can be achieved when the coefficient of friction is 1 and the radius of roller over radius of the die is 0.58. This would mean however, that only presses with one roller can be used since there is no room in the die chamber for more rollers.

With increasing length to diameter ratios of the die hole, the pellet under formation encounters a larger amount of shear. With the material remaining longer in compacted state, the role of elastic components in the feed will decrease because of relaxation of stresses in the material (Mohsenin and Zaske, 1976). Binding between particles is therefore enhanced as contact surface is increasing. Resilient material, for instance fiber rich feed, expands after pelleting and therefore decreases the amount of contacting sites between particles, or, in other terms, increase porosity. According to theoretical descriptions given by Rumpf (1958) and Ouchiyama and Tanaka (1985), porosity is one of the main factors determining the hardness in agglomerates.

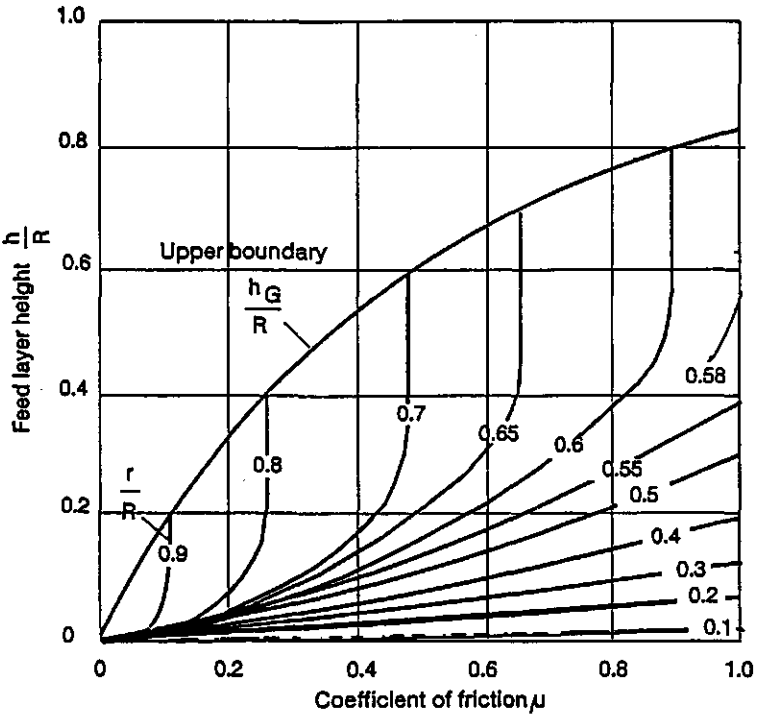

Figure 2: Theoretical height of the feed layer that can be pelleted depending on coefficient of friction and ratio between radius of the roller and radius of the die (Schwanghart, cited by Tešić, 1977). Legend: μ = coefficient of friction of feed mash between roller and die, r = radius of the roller, R = internal radius of the die. h_G/R is the maximum pelletable height with respect to the internal radius curvature of the die.

Another main parameter during pelleting is the distance between roller and die; the gap-size. Often the rollers are set to just touch the die. With increasing gap-size, pellet hardness or durability will firstly increase, thereafter pellet durability and hardness will become worse. Payne (1979) using a dairy ration, reported an increase in Kahl hardness of 5.1 to 5.3 units when rollers were set back for 1/10 inch (2.5 mm), at the same time energy consumption decreased from 220 to 210 Amp. Increasing gap-size to 3/16 inch (4.8 mm) resulted in a less smooth operation of the pelletizer and an increased energy consumption of 240 Amp. This indicates that clearance between roller and die became too large. Research conducted by Robohm and Apelt (1989b) indicates that a maximum pellet durability exists depending upon the gap-size between roller and die. Using a commercial pig-feed ration, they found an initial increase in pellet durability with the highest durability corresponding to a 2 mm clearance. A further increase in gap-size resulted in a decreased pellet durability of 97.2 % corresponding with a 4 mm clearance (Table 7). The

initial decrease in specific power consumption observed by Payne (1979) was not found by Robohm and Apelt (1989b). Differences in the composition of the used ration, dairy-ration versus pig feed, may have accounted for differences in specific power consumption. However, the same trend in pellet quality was observed in both trials. Between 2 and 2.5 mm, clearance induced an increase in hardness or durability. Upon further increasing gap-clearance, to approximately between 4 and 4.8 mm, durability dropped (Robohm and Apelt, 1989b) or energy consumption increased. The initial increase in pellet durability is attributed to a dense layer of material emerging as a result of increased shear and prolonged pre-compression. A further increase in clearance will result in decreased stability of the feed mash on the edge of the roller and die assembly, therefore leading to sideways 'leaking' of the feed mash. This in turn may account for the decrease in durability and the increase in power consumption. This is in agreement with Schwanghart (1969a) who showed that the width of the roller is one of the determining parts in the amount of material 'leaking' away.

Table 7: The effect of clearance between roller and die (gap-size) and pellet durability (After Robohm and Apelt, 1989b).

Clearance (mm)	Durability[a] (%)	Spec. energy cons. (kWh ton^{-1})	Temperature after pelleting[b] (°C)
0	96.5	10	75
1	97.5	11	80
2	97.7	16	85
3	97.5	20	90
4	97.2	26	95

[a] Pfost's tumbling can

[b] Temperature after conditioning is 68 °C.

Die velocity

The effect of die velocity on the pelleting characteristics as efficiency and production rate is not clear. Stevens (1987) conducted research on a corn or wheat formula, investigating the effects of seven different values of circumference velocity of the ring die on electrical efficiency (kWh ton^{-1}), fines production (%) and production rate (kg hour^{-1}) (Table 8). For the corn formula there was an increase in production rate with increasing die velocities, though the regression equation was not significant ($P>0.05$). Highest production rate was 1667 and 1656 Kg hour^{-1} observed at 235 and 166 m min^{-1} peripheral die velocity respectively. Pelleting efficiency (kWh ton^{-1}) increased with increasing die velocity, again, the regression equation was not significant ($P>0.05$).

Table 8: The effect of peripheral die velocity on production rate, specific power consumption and durability of two basal formulations containing 72.4% corn or wheat (Stevens, 1987).

Formula[1]	Die velocity ($m\ s^{-1}$)	Production rate ($kg\ h^{-1}$)	Efficiency ($kWh\ t^{-1}$)	Durability[2] (%)
corn	2.4	1261^{b}	17.4^{b}	91.0^{b}
corn	2.8	1656^{a}	13.4^{a}	89.9^{a}
corn	3.2	1457^{ab}	15.2^{ab}	89.6^{a}
corn	3.5	1579^{ab}	14.2^{ab}	89.4^{a}
corn	3.9	1667^{a}	12.8^{a}	89.7^{a}
corn	4.3	1462^{ab}	15.1^{ab}	89.8^{a}
wheat	2.0	1664^{ab}	13.1^{ab}	97.5^{c}
wheat	2.4	1736^{a}	12.5^{a}	97.7^{abc}
wheat	2.8	1579^{ab}	13.9^{ab}	97.8^{ab}
wheat	3.2	1522^{abc}	14.4^{b}	97.6^{bc}
wheat	3.5	1459^{bc}	14.8^{bc}	97.8^{a}
wheat	3.9	1488^{bc}	14.9^{bc}	97.7^{abc}
wheat	4.3	1339^{c}	16.4^{c}	97.7^{abc}

[abc] Means within the same formula with the same superscript differ not significantly (P>0.05).

[1] Die plugged at 2 $m\ s^{-1}$ die velocity with corn formula.

[2] Durability according to Pfost.

The wheat formulation showed an increase in production rate with decreasing die velocities, the regression equation was significant at P<0.05 level. With increasing die velocities (143 m min^{-1} and 256 m min^{-1} peripheral die velocity) the production decreased from 1736 kg $hour^{-1}$ to 1339 kg $hour^{-1}$. The efficiency for the wheat formula was highest at low die velocity, 143 m min^{-1} (12.5 kWh ton^{-1}) and lowest at 256 m min^{-1}peripheral die velocity (16.4 kWh ton^{-1}). These results indicate that large differences exist in the behavior of raw materials subjected to pelleting. However, the production of compound feed involves blending of raw materials. The combined effect of these raw materials may then result in better pelleting characteristics at low die-velocity. For pellets of 3 to 6 mm in diameter, Leaver (1982, cited by Stevens, 1987) claims that a velocity of 609 m min^{-1} is an optimum. For larger pellets and cubes Leaver recommends lower peripheral die velocities of 366 to 396 m min^{-1}.

Cooling/drying

In order to decrease moisture and latent heat, pellets need to be cooled after the conditioning and pelleting step. Pellets generally leave the die of the press at temperatures ranging between 60 to 95°C and with moisture contents of 120 to 175 g kg^{-1}. The free water content of the feed is decreased in the cooling process, making it possible to store the feed for a sufficient time period.

The amount of water and heat that is dissipated from the pellets is a function of air-flow and air-properties, raw material characteristics and pellet-size (Beumer, 1988; Maier and Bakker-Arkema, 1992).

In the compound feed industry generally two types of coolers are used; horizontal crossflow coolers and counterflow bunker coolers. Other types of coolers exist but are not often used in feed industry. During the cooling and drying stage, soluble components in the feed recrystallize and help to create bonds between feed particles. With the decrease in temperature, viscosity of some components will increase and thus aid in maintaining structural integrity in the pellets (Friedrich, 1964ab; 1977). For a good functioning of the cooler in the factory, the operator has the possibility to change the air-flow characteristics and the residence time by varying bed-height in the cooler. Research of Flores and Martinez (1993) indicates that the drying temperature seems to be the most important factor in determining 'usable pellet recovery'. The latter was determined as the amount of feed, which fell in the range of 0.373 to 2.3 mm after hammer-milling of 100 gram of extruded sample over 2, 3 and 5 mm sieves.

The air-flow in the cooler determines for a great deal the amount of water evaporating from the pellets. This water is added in the conditioning stage together with heat. The heat from the steam and additional heat generated due to friction in the die is the driving force in the evaporation process of the water in the pellets. After evaporation of the water on the pellet surface the pressure gradient and the heat at the inside of the pellet provides migration of the water from the inner pellet kernel to the outside of the pellet. When excessive air-speeds are used in the cooler, the outer layer of the pellet is drying at such a high rate that stresses are induced in the outer pellet layer; this will in turn induce cracks at the surface of the pellet which therefore becomes more susceptible to abrasion. This negatively affects pellet durability. Friedrich and Robohm (1968) conducted research on the durability of feed pellets as related to the Reynolds-figure (Re), a dimensionless number determining the air flow characteristics (laminar or turbulent) taking into account the speed of the flow, a characteristic dimension e.g. tube diameter and the viscosity of the medium. They reported that the highest pellet durabilities were obtained at Re between 30 and 40, indicating that currents were of the laminar flow type. Air velocities were approximately between 0.74 and 0.98 m sec^{-1} (recalculated; pellet diameter 5 mm). These experiments were conducted with a 3 deck horizontal cooler.

Maier and Bakker-Arkema (1992) conducted research on the counterflow bunker-cooler and found from their simulation model that pellet diameter and initial pellet temperature significantly affected the cooling rate and the moisture loss of pellets. Smaller pellet diameters had faster cooling rates than larger diameter pellets. Pellets of 3.2 mm remained within 5 °C of ambient air temperature when cooled within 3.5 min, whereas 6.4 mm pellets remained more than 5 °C above ambient air temperature. Initial moisture content, inlet air temperature and relative

humidity were varied and found not to be related to pellet cooling rate or moisture loss.

Varying residence time inevitably implies varying bed depth. With increasing residence time in the cooler more moisture and heat will be lost, which in turn affects the physical quality of the feeds. Depending on factors as bed height and the air flow characteristics this can result in higher or lower hardness or durability of pellets.

Discussion

In the previous chapters, the effects of steam addition, water addition, residence time and pressure elevation and its relationship with equipment on the one hand and changes in physico-chemical characteristics on the other hand have been discussed. The combination of the previously described unit operations (conditioning, pelleting, cooling) and process variables with their effects on the feed mash, proved to be decisive factors in determining the physical quality of the pellets. It is not always clear how a parameter change in one variable is influencing another. Therefore, rules of thumb are used in feed manufacturing to estimate the order and magnitude of changes in process variables (even maybe at subconscious level of the operator). Recently, algorithms are available which systematically investigate the parameter range of a technological process. These algorithms try to optimize the different variables present in the system given a set of objectives (Tran *et al.*, 1991). In some industries in the Netherlands decision support systems (DSS) have been developed and used in order to optimize pellet quality and pellet production rate while minimizing energy consumption. The DSS as developed by De Blank (1995) uses information of previous runs in the factory to calculate regression equations that describe the effects on energy consumption and production output. In this way a specific diet formulation can be optimized given a set of standards for pellet quality. From these regression equations a linear programming problem is formulated. Processing variables are then adjusted according to the advice generated from this problem. Table 9 shows an example of how changes in process variables affect output variables due to use of a DSS. It follows that production capacity was increased with approximately 9 percent and specific energy consumption decreased with approximately 3 percent.

Table 9: The effect of advice generated by a Decision Support System on capacity, hardness, percentages of fines and specific energy consumption of a cattle feed.

	Process variables			Output variables			
	Molassis[a] (%)	Temp.[b] (°C)	Amp.[c] (A)	Capacity (ton h^{-1})	Hardness (kg)	Fines (%)	Energy Consump. (kWh t^{-1})
Operator	0.0	30	283	8.8	8.4	3.9	31.8
Advice DSS	1.0	30	300	9.1	7.9	3.8	33.4
Result	1.0	30	305	9.6	7.7	4.2	30.8

Target in this operation was to maximize capacity with constraints on hardness and percentage fines (after De Blank, 1995).

[a] Percentage of molasses added during conditioning.

[b] Temperature in the conditioner.

[c] Amperage of the press engine.

Pellet hardness and durability decreased slightly but were still within the standards put forward by the manufacturer. Table 10 shows the overall effect when one or (successively) two advices were generated for one run. Though specific relations still need to be established in experiments, DSS may help to improve performance of the feed mill in terms of decreasing process costs and improve physical quality.

Table 10: 95% confidence intervals[a] for increase in capacity and energy-reduction with use of a DSS, with two succesive advices within a run (de Blank, 1995).

	Increase in capacity	Reduction in spec. energy consumption
Advice I	[7.8%, 15.1%]	[2.7%, 10.2%]
Advice II	[10.8%, 19.1%]	[2.9%, 10.0%]

[a]Results based on 20 testruns with cattle feed.

Research in the field of animal feed manufacturing in this respect should aim more on quantifying and integrating the upstream and downstream chain of equipment, and subsequently optimizing the various parameters involved. However, the use of decision support systems requires a vast knowledge on the relations between the different process- and systems variables. Especially the relation between raw materials and process variables is still poorly understood. It therefore seems justified to look into greater detail into the effects of processing equipment on functional changes in the components of raw materials and its blends. Therefore, with respect to the previous mentioned aspects it is necessary to obtain relationships between the effect of these processing variables and the induced changes in the feed components starch, protein and fiber. As shown by Behnke (1995), equipment with a higher degree of flexibility, for instance the

expander, does permit to deal with large variability in feed components normally found when formulating e.g. European diets. Using expanders will cause a shift to an increased contribution of the expander towards pellet quality (Fig. 3). Care should be taken that use of such equipment is justified from a process- and economic perspective.

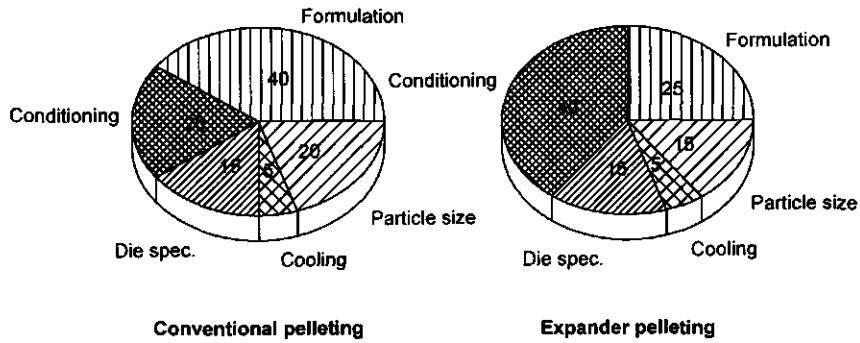

Figure 3: The contribution towards pellet durability of conventional pelleting and of expander equipment with its extended range of process variables (After Behnke, 1995).

Vitamins and other valuable heat labile components (lysine) should not be sacrificed when in need for equipment with a high degree of flexibility. This aspect should be taken into account in the design of that equipment. The techniques to incorporate heat labile components after pelleting or research into adding thermal stability to these components seems to be necessary when manufacturing equipment is not able to maintain the levels of these components during processing.

Specific knowledge of interrelations between process and systems variables on the one hand and feed components on the other hand will permit to design even better equipment for the manufacture of animal feed. Therefore, process research rather than product research in this field is recommended and justified.

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3

Physical quality of pelleted animal feed. 3. Contribution of feedstuff components.

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Abstract

An overview is given of effects of the diet ingredients and their composition (e.g. starch, protein, sugar, fat and fibre content) on production characteristics of pellets and on their physical quality (pellet hardness and pellet durability) as compound animal feeds.

Large differences exist in the effect on pellet physical quality within and between groups of diet ingredients when incorporated in pelleted animal diets. Differences in pellet quality between groups of diet ingredients, e.g grains or legume seeds, can be attributed to differences in their physico-chemical properties which in turn are primarily affected by processing history, geographical and climatical origin and cultivar. Differences in physico-chemical properties due to the latter factors are also the main causative contributors to explain differences in pelleting quality (e.g. throughput, energy consumption of the pellet press) within groups (e.g. cereals or legume seeds) and thus in the resulting pellet quality. Raw materials within one group are much more the same than between groups with respect to pelleting properties and pellet quality, since their composition varies only within a relative narrow range. Because of the inherent variability of the raw materials, the effects of its constituents were investigated with respect to pellet quality. Effects of raw material constituents, both their level and physico-chemical properties may provide more information on pelleting characteristics and pellet quality than the diet ingredient inclusion level of the raw material per se. The effects of starch (native versus gelatinized), sugar, protein (raw versus denatured) and solubility and resiliencenes of fibre are discussed with respect to pellet quality. When pellet hardness or durability is lacking, pellet binders may be used to improve pellet quality. The effects of pellet binders and their mode of action are discussed.

It is concluded that more research effort should be directed towards the effects of individual constituents and their respective properties, since the latter seems to affect to a large extent the final hardness and durability of pelleted compound feeds. Moreover, the properties of a specific constituent reflects the processing history of that specific ingredient. By relating pellet quality to physico-chemical properties, e.g. functionality of the constituent, the manufacturer of compound animal feeds will be able to decrease the variability in final pellet quality caused by differences in geographical origin and processing history of the diet ingredients. Objective criteria from animals are still necessary to evaluate pellet quality as far as nutritional quality is concerned.

Introduction

It has been shown that the physical quality of pellets (pellet durability and hardness) is influenced by the ingredient composition of the diet (van Rooy, 1986; Wilson, 1994). In general, the effect of ingredient composition upon pellet quality has been attributed to changes that occur in the ingredients when they are subjected to physical compression and shear during the pelleting process. The extent of these changes increases with conditioning of the material (Moran, 1989) prior to pelleting and by the influence of the pelleting process itself (van der Poel, 1994). In a

previous article (Thomas and van der Poel, 1996) different binding mechanisms with respect to the pelleting process have been discussed. In the present article an overview is given on the influence of diet ingredients on physical quality of feed pellets.

Diet ingredients

The use of least cost formulation to formulate diets, leads to a large number of feedstuffs incorporated at different inclusion levels. This may lead to variation in physical quality of the feeds after pelleting, although the calculated nutritional requirements are met. In addition, some pelleting parameters such as pressure (distribution) in the die hole and porosity will change during pelleting. Also, the amount of energy required to overcome friction in the die hole can only be roughly estimated rather than exactly measured and these parameters are strongly dependent on the physico-chemical properties of the diet ingredients themselves.

In order to predict pellet quality, a pragmatical approach is often used, as for example proposed by MacMahon and Payne (1991). They tried to relate different raw materials to pelleting criteria used in animal feed manufacturing. For certain raw materials a classification was made according to type of raw material e.g. cereals, oilseeds or byproducts. They estimated the effect of inclusion of a specific raw material on the physical quality of pellets, pelleting capacity of the pellet press and wear of the die. These three physical factors were scaled so that virtually all raw materials fell in a range between 0 and 10, exceptions being fat and binding agents (Table 1). These figures are estimates from literature and experience and one should carefully interpret these data since origin, storage and processing conditions may alter the raw material properties and their related binding actions. Israelsen et al. (1981) used linear regression to relate pellet quality to different diet ingredients. They estimated the effect of inclusion of different diet ingredients on pellet durability (%) and energy consumption (kWh $tonne^{-1}$) of the pelleted raw material (Table 2) by substituting barley or cotton seed meal by a number of other ingredients. From their investigation it was concluded that some byproducts alter significantly pelleting properties of the feed. The authors concluded that inclusion of about 10 percent cane- or beet molasses in combination with solid byproducts reduced the formation of fines and improved specific press capacity. Cottonseed meal gave more durable pellets compared to soyabean meal and rapeseed meal (durabilities of 97.2, 94.5 and 91.2% respectively). Specific energy consumption of the pellet press using cottonseed meal (6.5 kWh $tonne^{-1}$) was higher as compared to soyabean meal (5.6 kWh $tonne^{-1}$).

Table 1: Feedstuffs and their respective nutritional and pelleting properties (modified after MacMahon and Payne, 1991).

Raw material	Constituents[a]					Physical Factors [-][b]		
	Crude Protein ($g\ kg^{-1}$)	Crude Fat ($g\ kg^{-1}$)	Crude Fibre[c] ($g\ kg^{-1}$)	Starch[c] ($g\ kg^{-1}$)	Bulk Density ($kg\ m^{-3}$)	Pellet quality	Press capacity	Die wear
Milling by-product								
Barley meal	107	22.0	47	490	480	5	6	5
Maize meal	87	38.0	21	585	610	5	7	6
Milo meal	90	28.0	40	580*	540	4	6	7
Oat meal	112	48.0	106	378	520	2	3	7
Rice	78	20.0	88	580*	480	5	5	4
Wheat meal	119	17.0	23	555	540	8	6	3
Oilseeds and derivatives								
Coconut cake	207	82	130	5	480	7	8	6
Cotton decorticated	403	308	28	0	640	7	8	6
Cotton meal extracted	436	30	118	8	610	8	6	7
Groundnut cake dec.	469	79	54	63	620	7	8	4
Groundnut meal extracted	503	5	125	20	670	8	6	5
Linseed meal extracted	334	31	94	16	560	7	6	5
Palm kernel cake expeller	146	91	188	4	480	6	7	4
Palm kernel meal extracted	152	21	189	3	700	6	5	5
Palm kernel (whole)	93	478	102	0	750	3	8	3
Rapeseed meal extracted	343	22	114	11	510	6	6	6
Sesame meal expeller	451	114	62	14*	560	7	7	4
Soyabean meal extracted	449	18	53	8	500	4	5	4
Soyabeans full fat	356	189	53	9	480	4	8	3
Sunflower cake expeller	383	71	167	35*	560	6	6	4
Sunflower meal extracted	339	20	192	26	530	6	5	5

(Table 1 continued)

Animal by-products								
Blood meal	878	7	0	0	560	3	5	3
Fat (added at mixer)	0	1000	0	0	900	<-10	>10	0
Feather meal	824	76	0	0	400	4	5	5
Fish meal	564	116	0	0	640	4	7	5
Meat meal	569	100	22	0	620	5	7	3
Meat and bone meal	498	91	16	0	690	4	7	4
Poultry by-product meal[d]	711	133	27	0	590	3	8	4
Legumes								
Field beans	254	13	73	376*	690	7	5	5
Peas	206	11	55	410	720	6	5	5
Lentils	229	13	45	422*	800	4	4	5
Locust beans	40	0	71	0	400	4	4	6
Others								
Brewers grains, dried	252	67	126	24	320	3	4	5
Citrus pulp	61	22	118	13	330	7	3	6
Maize germ meal	142	53	69	339	480	5	8	3
Maize gluten feed	185	38	70	188	540	3	4	6
Maize gluten meal	607	37	11	156	480	4	5	5
Tapioca	24	4	43	655	640	5	3	7
Minerals	0	0	0	0	1000	2	4	10
Beet molasses	110	0	0	0	1230	7	6	0
Rice bran	129	130	110	200*	320	2	3	6
Skim milk powder	349	23	0	0	640	9	2	9
Sugar beet pulp (molassed)	n.a.	n.a.	n.a.	n.a.	240	7	3	5
Lignosulphonate	n.a.	n.a.	n.a.	n.a.	500	>10	>10	0

[a] Constituent levels derived from the Dutch CVB-table (CVB, 1994).

[b] Figures on a scale of 0 (poor contribution) to 10 (high contribution), based on pellet press operaters experience.

[c] Crude fibre as component of 'Weende analysis' ; starch content is given as determined by enzymatic determination (Anonymous, 1974) except were indicated (*), determined by polarimetric method (Anonymous, 1979).

[d] Poultry by-product meal (chemical data after El Boushy and van der Poel, 1994).

Table 2: Effect of diet ingredients on specific power consumption and durability of pellets (After Israelsen et al., 1981).

Diet ingredient	Spec. power consumption (kWh Tonne^{-1})	Durability[a] (%)
Control[b]	6.5	97.2
Grain substitute[c]		
Beet pulp, dried, pelleted	6.0	98.9*
Barley malt culms, dried	4.2*	98.3*
Citrus pulp, dried, pelleted	4.9*	97.6
Barley, ground	7.3	97.6
Coconut meal	7.1	97.4
Alfalfa meal, dried	6.8	97.2
Grass, dried, pelleted	7.6*	97.2
Wheat bran pellets	6.9	96.9
Palm kernel cake	8.1	96.8
Cotton seed substitute[d]		
Sunflower seed meal	7.0	94.9*
Soybean meal	5.6*	94.5*
Rapeseed meal	5.8	91.2*

[a] Durability determined with the Pfost tumbling can.

[b] Control consisting of 287 g kg^{-1} alkali-treated straw pellets, 287 g kg^{-1} barley, 287 g kg^{-1} cottonseed meal, 40 g kg^{-1} fat and 100 g kg^{-1} molasses.

[c] Grain substitutes replaces barley.

[d] Cotton seed substitutes replaces cottonseed meal

* Significant at $P<0.05$ different from control.

It has been stated by both MacMahon and Payne (1991) and Israelsen et al. (1981) that differences between batches and processing history greatly affect the pelleting characteristics of a given feedstuff. Friedrich and Robohm (1981), Lake (1991) and Wilson (1994) gave estimates for some raw material composition and their variability. For instance, coefficients of variation in protein content ranged between 2.4 % (mean protein content: 485 g kg^{-1}) for soyabean meal (Wilson, 1994) and 8.6 % (mean protein content: 105 g kg^{-1}) for barley (Lake, 1991) respectively. During the course of processing of the product, the physico-chemical properties of the raw material change as a function of the different processing procedures and conditions applied. This is obvious for byproducts from the food industry which have undergone several treatments prior to their inclusion in animal diets. However, also raw materials directly used for animal feed purposes may undergo processing before pelleting which alters the functionality of its constituents. For instance, country of origin and pelleting conditions influence the degree of gelatinization of tapioca starch during pelleting (Jongbloed and Smits, 1993). The resulting differences in tapioca starch may reflect differences in physical quality of pellets.

So far, not much research has been conducted on the physico-chemical properties of

feedstuff constituents with respect to physical quality of the animal feed. An outstanding example in this respect is the work of Wood (1987), who related degree of starch gelatinization and of protein denaturation towards the durability and hardness of pelleted animal feeds. His results show that there are relationships, but these are too limited to allow quantitative conclusions. Consequently, further research should aim to quantify these relations. Such a research may also provide a better understanding of the underlying fundamental processes.

Effects of raw material constituents

Ingredient constituents can be classified as starch, protein, sugar and non-starch-polysacharides (NSP), fat, fibre, inorganic matter and water. The matrix structure in which the different components are arranged is very complex (Chesson, 1987) and this complexity may prevent the expression of a single constituent on pellet quality.

A well-known example for instance, is the diet inclusion of fats. Free fats added in the mixer negatively affect pellet durability and hardness but will improve press capacity in terms of tonnes per hour produced (Friedrich and Robohm, 1981). This effect is attributed to the lubricating effect of added fat on the mash - die interface during pelleting. The same amount of naturally occuring fats enclosed in cell walls does not have such a marked effect on these parameters. Though general descriptions can be given of the functionality of feedstuff constituents, this functionality may vary clearly within for example a type of grain or between groups of grains, because of differences in grain morphology. However, common effects can be observed due to changes in certain constituents, for instance, due to gelatinization of starch, denaturation of protein, solubilization and consecutive recrystallisation of sugars and salts, all of which might affect hardness and durability of animal feeds. The contribution, either positive or negative as well as the magnitude of these effects should be subject of further research on the objectives of pellet quality and animal performance.

Starch and sugar

In human food engineering, starches are used for various purposes. According to Smith (1983) starch may function, amongst others, as an adhesive or binding agent. Starches used for these purposes have undergone a heat- or chemical treatment in which the properties of the native starch are changed. Gelatinizing of starch in the presence of water and heat, in the presence or absence of shear, is the most common way to affect functional properties of starches (Smith, 1983). Chemical reagents are used to modify starches to specific needs, an example is the various processing techniques using for example soda, borax or urea to create different adhesives for the

paper and board industry (Kennedy and Fisher, 1984). Processing of starch leads to crosslinking which may give the starch its desired properties as for instance changed rate and amount of swelling (Smith, 1983) which may be important for purposes in food applications.

Table 3: Gelatinization temperature, amylose content and granule shape of feedstuffs (Smith, 1983) and pellet quality (MacMahon and Payne, 1991).

Starch origine	Amylose[a] ($g\ kg^{-1}$)	Gelatinization temperature[b]. (°C)	Granule shape	Pellet quality[c]
Waxy Maize	0-30	74	Round polygonal	-
Tapioca	170	63	Truncated, round, oval	5
Rice	170	81	Polygonal	5
Potato	200	64	Oval	-
Maize	250	80	Round, polygonal	5
Wheat	250	77	Oval, truncated	8
Sago	270	74	Oval, truncated	-

[a] In percent of starch (Smith, 1983).

[b] 5% solution in water; gelatinization temperatures for 'raw' starches are listed covering the temperatures at which loss of birefringence is first noticed and less than 10 percent remains intact (Smith, 1983).

[c] Pellet quality; ranking figures from 0 - 10 when available (MacMahon and Payne, 1991); see Table 1.

Differences in amylose / amylopectin ratio in starches might also affect properties of the feed subjected to processing. However, only limited research seems to be undertaken to investigate the effect of variations in amylose/amylopectin ratio on hardness or durability of pellets produced. After gelatinization of the starch granule, amylose immediately forms double helices which may aggregate (hydrogen-bonds) to each other and create semi-crystalline regions. However, pellet binding occurs probably by amylopectin due to the double helices formed at the non reducing ends of this very large branched molecule which may aggregate with compatible starch or fibre surfaces on the different particles present during and after gelatinization (Moran, 1989; Schwartz and Zelinskie, 1978). The starch needs to be heated first in order to destroy its native structure and to allow reordening of the molecules which is required to provide good binding properties in tablets (Schwartz and Zelinskie, 1978). The relation between starch functionality and pellet quality is also influenced by other components in the feed which affect starch gelatinization itself. Eliasson (1981a) showed that an increasing amount of oil added to a starch suspension reduces the volume of the produced gel. However, derived thermograms from differential scanning calorimetry studies of the starch/oil suspensions did not show any significant difference in energy required for gelatinization or temperature at which gelatinization

occurs, indicating that gelatinization was not affected (Lund, 1984). Other components present might affect the contribution of amylose or amylopectin towards pellet quality. No apparent relation seems to exist between amylose content of some raw materials and pellet quality (Table 3). No literature has been found on the effect of resistant starch and pellet quality.

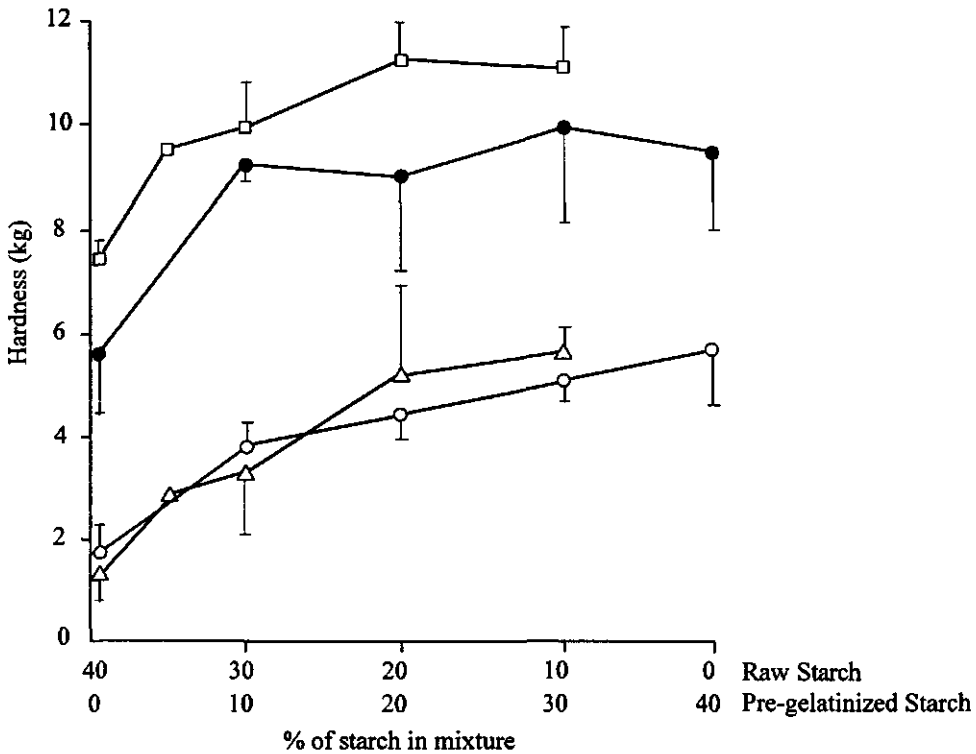

Figure 1: Effects of functional properties of starch and protein on hardness (Kahl device) of pellets after cold and steam conditioning prior to pelleting (Wood, 1987). Two replicates per treatment (means ± 1 SD).
legenda: ● Cold-conditioned raw protein. Temperature exit conditioner 22 °C.
□ Hot-conditioned raw protein. Temperature exit conditioner 59 -73 °C.
Δ Hot-conditioned denatured protein. Temperature exit conditioner 59 - 73°C.
o Cold-conditioned denatured protein. Temperature exit conditioner 20 - 22°C.

To obtain feeds with a certain specified quality standard in terms of hardness and durability, starch needs to be modified by either the feed manufacturing process itself or in a pre-processing step, since native starch in itself does not possess functionality in terms of binding or adhesion to produce durable feed pellets. Wood (1987) showed that the amount of pre-gelatinized starch

in feed is related to the physical quality of the feed pellets. Using a design in which native starch was gradually replaced by pre-gelatinized starch (feed model system containing 40 % starch), an increase was found in physical quality as determined by Kahl hardness test (Figure 1) and Holmen durability test. Within the range tested, no optimum was found indicating that pregelatinized starch included in the diets of percentages up to fourty percent might still lead to harder (Figure 1) and more durable pellets (Wood, 1987).

Heffner and Pfost (1973) found a higher degree of gelatinization of feed starch with the use of a smaller diameter of the die-hole. Gelatinization and durability of the pellets were also higher with smaller die-holes. However, ratio of length to diameter was also sligthly altered which makes it difficult to attribute higher gelatinization percentage to the smaller die diameter per sé.

The underlying mechanism of the contribution of gelatinized starch in binding properties in pellets is still not fully elucidated. The presence of water, for instance, is a prerequisite to initiate gelatinization. According to Lund (1984) a ratio of 0.3/1 (water/starch) is needed. Various authors (Eliasson, 1980; Marchant and Blanshard, 1980 and Wootton and Bamunuarachchi, 1979) state that in order to fully gelatinize starch, water is needed in a ratio of at least 1.5/1 (water/starch). This implies that during the manufacturing stage of compound animal feeds, water is a limiting factor to fully gelatinize starch. Depending on feed ration used, approximately 3 percent water in the form of steam is added. In order to act as a binder, bonds have to be formed at the contacting sites of particles, so it would be sufficient if the starch in the outer layers of the particle is gelatinized. (Thomas and van der Poel, 1996).

Inclusion of sugar increases power-requirements of the pellet mill due to higher resistance at the feed-die interface. Because of re-crystallization after cooling of solubilized sugars, binding may occur between feed particulates due to the formation of a solid bridge. Aumaitre et al. (1978) did not find significant differences in pellet durability between diets pelleted with different amounts of glucose, saccharose or molasses high in fructose content. Throughput of the pellet press increased on average with 14 percent when 10 percent molasses high in fructose was used to replace an approximate equal amount of saccharose. A low pellet hardness was obtained, this could be due to the fact that their experimental diets were pelleted without steam addition. For this reason, sugars may not have been sufficiently solubilized, since one may expect to find positive effects of sugars on pellet hardness only if sufficient water, preferably water added in the form of steam, is available. Upon cooling/drying recrystallisation or a glass transition may take place, creating fixed binding points 'solid-solid interactions' between particles. Increase in pellet durability may well be obtained by use of sugars, for instance, those originating from molasses as is shown by Friedrich and Robohm (1982). Binding properties of molasses might occur via 'solid-solid' interactions (re-crystallisation of sugars) or the formation of a glass with

its subsequent binding properties. With the incorporaton of sugars the risk of unwanted Maillard reactions, however, is greatly enhanced. Especially when moisture is present, free aldehyde and amino groups may combine to form melanoides that darken the product and become viscous, when they polymerise (Motai, 1976), they may act as a filler. This is positive for pellet-binding, however Maillard products may impair nutritional value (Van Barneveld, 1993; Hendriks et al., 1994).

Protein

Properties of proteins in foods or food-systems have been classified according to Nakai and Powrie (1981) as follows:
- Sensory and kinesthetic properties (e.g., flavor, odor, color, texture)
- Hydration, dispersibility, solubility and swelling
- Surface-active properties (required for e.g., emulsion and/or foam formation)
- Rheological properties, including gelation and texturization
- Other properties (e.g., inducing adhesion, cohesion, dough making, film and fibre formation during extrusion)

In feed manufacturing, especially the adhesive forces that proteins may exert are of interest. Protein can act as a binding agent between different feed particulates. Processing involves the combined effect of shear, heat, residence time and water resulting among others in partial denaturation of the protein in the feed. As has been shown by Wood (1987) partial denaturation during processing may positively affect the hardness and durability of the feed pellets. Denaturation involves the breakdown of the (spatial) three-dimensional structure of the proteins (the secondary, tertiary and quaternary structures) thereby changing the bio-activity of the protein (Van Barneveld, 1993). Upon cooling proteins reassociate and so bonds can be established between the different particles. No reports could be found in which the effect of processing variables in animal feed manufacturing has been described on the denaturation of protein and its consecutive effects on physical quality. Both Hermansson (1979) and Howell (1991) emphasize the lack of knowledge concerning the behaviour of proteins in mixtures with a large amount of other (non-protein) particles present, as is the case for feed mashes during the conditioning and shaping process.

Interactions which according to Howell (1991) will affect mechanical stability of proteins, and therefore may affect hardness and durability characteristics of pellet, involve covalent binding, electrostatic interactions, van der Waals-forces, hydrogen bonds and entropic factors. Conformational changes are the result of a change in the combination of all of these

forces. In some products like wheat, the presence and contribution of protein fibrils to pelletability can be the reason for differences observed in pellet quality (Moran, 1989). Fibrils formed by aggregated protein molecules are important for wheat dough structure and so for bread-making (Simmonds, 1972). Protein fibrils may act likewise in creating binding sites between particles in the pelleting process (Moran, 1989).

Wood (1987) showed that there was an effect of the inclusion of raw or denatured protein on the physical quality of feed in terms of pellet hardness and pellet durability (Kahl and Holmen pellet criteria, respectively). Physical quality was improved when incorporating 35 % raw protein rather than denatured protein (Figure 1) in a feed model system.

Processing may result in the so-called Maillard-reaction, in which many constituents of raw materials can participate and which affects many quality attributes. Reactions between reducing sugars and free amino groups from amino acids, lysine in particular, prevail (Voragen et al., 1995). Due to formation of Maillard products, the utilization of protein and perhaps carbohydrates may be reduced. This phenemenon emphasizes the conflict between physical and nutritional quality of feed after processing (Goering, 1976; van der Poel et al., 1995).

Fibre

Fibre can be classified in a water soluble part, which may cause a high viscosity, and a water insoluble part (Frølich, 1990; Lo, 1990). This classification may be usefull in explaining the effects of different fibre sources on pelleting characteristics.

Water soluble fibre like glucans, arabinoxylans and pectins strongly raise the viscosity which may affect structural integrity of the feed agglomerates. Materials with a high viscosity may act as a filler in the feed; the viscous material embeds the more coarse particles thereby reducing porosity in the feed. Subsequently, structural integrity of the feed agglomerate is higher which will result in higher durability and hardness of the pellets. During processing, process variables such as residence time, moisture content, pressure and heat, alter the state of the soluble fibre. To obtain the desired functional properties (viscosity, binding) for a pectin, the effect of time, pH and temperature are of major importance (Keller, 1983). Due to the complexity of the feed mash (blends of a number of raw materials which differ in composition and functional properties) and processing conditions (heat, pressure and time relationships), quantitative descriptions of the effect of fibres that forms with water a viscous solution/paste, on pellet quality are difficult to acquire.

The effect of not dissolved plant fibres may be twofold. Plant fibres may prove beneficial in the pelleting process since they have the opportunity of entangling and folding between different particles or strands of fibre (Rumpf, 1958). On the other hand, due to their stiffness and

elasticity they may impair problems to the pellet-press operator since resilience characteristics of the material oppose good contact between particles or fibres. Moreover, when large fibres are present within the pellet they might induce a weak spot in the pellet. Large particles may serve as inhomogeneity at which pellets are most likely to break. Increasing the residence time in the die of the pellet press would diminish the effects due to the resilience of the material. This is in line with results from Mohsenin and Zaske (1976) who found that an increase in hold-time within a wafering machine resulted in further stress decay of the material under investigation and subsequently increased durability of the formed wafer. Sihag et al. (1991) found that expansion of wheat straw blocks after compression, was lower after a longer residence time.

Chemicals and water affect resilience characteristics of plant fibres, resulting in different compaction behaviour and subsequent pellet quality. Addition of 4 % NaOH decreased post-compression expansion in wheat-straw based feed blocks as compared to a control (Sihag et al., 1991). Tešić (1977) found an increasing durability of wheat straw pellets with the addition of 0, 1, 3, 5 or 7 % of NaOH respectively. The mechanism might be that addition of NaOH separates lignin from cellulose thereby decreasing the strength and resilient character of the fibre in the pellets (Tešić, 1977) or in feed blocks (Sihag et al.; 1991). Other chemicals and reagents such as CaO or urea may likewise affect pelleting characteristics by degrading part of the cell wall structure, therefore, resilienceness is decreased and durability of the feed pellets will increase.

Results of Mohsenin and Zaske (1976) show that moisture content influences the stability of wafers made from hay, bark or sawmill waste. For compressed forage materials, moist materials (between 19 and 23 percent moisture on weight basis) resulted in more durable wafers when tumbled immediately after compaction. This was attributed to the more elastic deformable behaviour of the material which allows better withstanding of the impact loads during tumbling. Nathier-Dufour et al. (1995) studied the compaction behaviour of wheat meal and tried to relate that to its composition, as derived from near infrared reflectance spectroscopy (NIRS). A so-called yield point of the powder particles and strain attained due to a stress of 100 kN were used to asses compaction behaviour of the material. Water proved to be the main determinant of the compaction behaviour of the wheat. A slightly higher water content (range 11.0 - 12.8 % w.b.) resulted in a lower 'yield point' of the particles and so a better compaction behaviour.

It is noted that upon processing, plant cell walls or their constituent fractions may undergo the Maillard reaction (Theander, 1980). These products may affect the apparent lignin content and cell wall characteristics and may negatively influence the nutritive value (Van Soest and Mason, 1991)

Fat

Added fat in compound animal feed is known for its adverse effect on pellet hardness and pellet durability (van Vliet, 1981). Since most binding of feed particules incorporates water or, when involved, solubilized starches, proteins and fibres, fat with its hydrophobic nature may interfere with binding properties of water soluble components in the feed. Moreover, added fat (and to a lesser extent, fat enclosed in the matrix of cellwalls) acts as a lubricant between particles and between the feed mash and the die-wall and so resulting in a lower pelleting pressure. This will already cause lower pellet quality in many cases. Also as a result of fat addition various properties of a raw material constituent might be influenced. For instance, gelatinization of starch can be inhibited in the presence of lipids or delayed to higher temperatures (Eliasson, 1981b; Larsson, 1980). This may indirectly lead to weaker pellets, vulnerable to deterioration in transport systems. Salmon (1985) showed that more fat in the pellet (0, 30, 60, 90 g kg^{-1}) significantly increased the amount of fines returned; 2.7, 7.0, 10.3, 15.6% with no bentonite added, and 3.8, 5.1, 7.0, 15.3 % with 25 g kg^{-1} bentonite added. Richardson and Day (1976)

Table 4: Effect of adding fat to a chicken broiler feed before and after steam pelleting on the proportion of fines and power consumption of the pellet press (Richardson and Day, 1976).

Added fat (g kg^{-1})		Fines (%)[a]	Production (Tonnes $hour^{-1}$)	Power consumption (kWh $tonne^{-1}$)
Mixer	Post-cooling			
10.0	46.6	18.0	11.6	11.0
20.0	36.6	22.0	12.1	9.7
30.0	26.6	29.2	13.2	8.7
40.0	16.6	31.6	13.2	7.9
53.3	3.3	50.8	-	-

[a] Percentage of fines passing through a 2.36 mm screen.

showed that an increasing amount of fat added in the mixer increased the amount of fines returned from the pellet mill and decreased the amount of mechanical energy (kWh/ton) needed to convert one ton of mash into pellets (Table 4).

Fat enhances the production rates in pellet-mills, primarily because of the lubricating effect of fats between the meal and the die surface (Walter, 1990, Richardson and Day, 1976; van Vliet, 1981). Because of low friction, pressure in the die is decreased which, in turn, would result in feed pellets with lower hardness and durability. However, some authors (Von Sybel and Wittmann, 1960; Schwanghart, 1970) suggest that during blending the feed mash with steam, natural oils and waxes are released from the interior of (plant) cell walls. These oils and/or waxes would accumulate at the contacting sites of two particles and create a binding point, either solid (waxes) or via liquid necking (oil or water), between particles upon cooling (Friedrich, 1977). This would have a positive effect on pellet hardness and durability. Because of immiscibility of water and fats there would be an optimum in the amount of bonds established by waxes and fats on one side and water on the other side. The amount of bonds being dependent on the concentration in a certain not well-known range.

Oxidation and thermal degradation of lipids lead to complex chemical changes. Also, interactions of reaction products with other feed constituents may produce compounds deleterious to the physical quality and nutritional value of feed (Mawar, 1985; Voragen et al., 1995).

Other constituents

When abundant water is available, marked effects of pH, salts and chemical reagents on the functional properties of starches, proteins and fibres are found in human food engineering (Smith, 1983; Arntfield et al., 1990, 1991; Autio et al., 1992). During the feed manufacturing process, pH, salts and chemical reagents, may affect the different processes taking place during the conditioning, pelleting and cooling stage. The magnitude of these effects in feed systems still needs to be investigated, since a lot of literature concerning this subject is conducted on food systems with higher water contents as compared to those of compound feeds.

In the conditioning stage, when water is available during a sufficient period of time, some chemicals might react with raw materials, or their respective constituents, to yield different surface properties of the feed mash. This may in turn affect the interaction forces between the components formed, and so influence the stucture of the pellet. No reports have been found which explicitly determine the effects of chemical agents on the physical quality of pelleted animal feeds. However, the study of Knacke and Pohl (1959) showed that differences in surface tension of the water used, influenced binding force in clay agglomerates. Reagents affecting surface tension of water may influence structural integrity of agglomerates, if binding is through liquid necking in feed pellets, which may be the case (Thomas and van der Poel, 1996).

Pellet binders

When physical pellet quality is not sufficient to obtain a saleable product or does not meet the manufacturers quality standards, binder materials can be incorporated to increase physical quality of the pellets. For this purpose, different types of binders are available. The best known binders used in animal feed production are bentonite (a clay mineral), carboxy methyl cellulose (CMC; modified cellulose) and lignosulphonates. The effect of binder materials depends on the presence of water which is required for a binding agent to become active. The proposed mechanisms for binding particulates in general have been discussed previously (Thomas and van der Poel, 1996).

Lignosulphonates are byproducts from the paper industry. During the process of delignifying of fibres, $NaHSO_3$ and/or Na_2SO_3 is added of which the alkaline components $(SO_3)^{2-}/(HSO_3)^-$ react with chemical bonds of the lignin to form water soluble ligno sulphonates (Gellerstedt and Gustafsson, 1987; Heitner and Min, 1987). Common inclusion levels range from 0.5 to 3 percent. Several authors (Friedrich and Robohm, 1970; Van Zuilichem et al., 1979a,b; 1980) have reported an increased pellet durability and decreased energy consumption (kWh tonne^{-1}) by use of lignosulphonates.

Table 5: Effect of steam addition and bentonite inclusion level on production characteristics of a pig grower diet (After Friedrich and Robohm, 1970).

Process variables		Production characteristics	
Steam (g kg^{-1})	Bentonite (g kg^{-1})	Pellet durability (%)	Specific power consumption (kWh tonne^{-1})
0	0	94.3	22
	10	94.7	21
	20	95.5	21
	30	96.1	21
15	0	95.3	17
	10	95.6	16
	20	96.5	15
	30	97.0	15
30	0	95.6	9
	10	96.1	5
	20	97.0	3
	30	97.5	2

Bentonite is a binder used in feed technology to improve physical pellet quality. Its main target is acting as a filler thereby decreasing porosity in pelleted feed. In addition, bentonite

works as a lubricant in the die hole (Friedrich and Robohm, 1970), it decreases pressure and subsequently energy requirements of the pellet press (Table 5). Friedrich and Robohm (1970) postulated that bentonite doesn't have any binding properties in itself. Especially with voluminous feed mashes as for instance fibre rich diets, bentonite may prove advantageous. Salmon (1985) did not find a significant effect of the addition of 25 gr bentonite per kg feed mash on liveweight, feed intake or feed efficiency when fed to white turkeys. Pellet durability decreased non-linearly when fat-levels increased (0, 30, 60, 90 g kg^{-1}). Pellet durability was not affected by addition of bentonite to formulations which included 0 or 90 g of fat per kg, but it improved pellet durability at fat inclusion levels of 30 and 60 g kg^{-1} feed.

Pfost and Young (1973) found beneficial effects of the inclusion of bentonite on pellet durability as measured by a feed model handling system. No significant effect was observed of the effect of bentonite in relation to variations in grind (fine, medium, coarse) of the feed mix. Increasing amount of steam increased pellet durability for both control (mash without bentonite) and mash with bentonite. However, adding bentonite improved pellet durability on average with 5.4 percent as opposed to the control (Table 6).

Table 6: Pellet durability and specific power consumption as affected by steam inclusion, grind of the grain portion in the feed mix and bentonite inclusion (Pfost and Young, 1973).

Steam		Grind	Production characteristics[a]			
Tempera-ture rise (°C)	Moisture inclusion[b] (g kg^{-1})		Durability (%)[c]		Spec.Power (Kwh $tonne^{-1}$)	
			Control	Bentonite[d]	Control	Bentonite[d]
17	14	fine	71.3	79.8	17.5	16.6
		medium	70.8	76.6	17.8	17.9
		coarse	74.6	79.4	17.4	17.2
34	27	fine	83.5	88.8	10.9	11.8
		medium	80.5	87.0	11.4	11.3
		coarse	80.9	86.7	11.0	11.8
51	39	fine	88.3	92.2	8.5	9.7
		medium	86.9	92.1	8.9	10.2
		coarse	88.2	90.7	8.8	9.8

[a] Each value is the average of nine tests.

[b] Moisture originating from added steam.

[c] Measured by Pfost's 'feed model handling system' (Pfost and Young, 1973).

[d] Bentonite: 24 g kg^{-1}

Carboxymethylcellulose (CMC) can also be used as a binder in animal feed production. CMC gives a viscous solution/paste when mixed with water. Hydration occurs during conditioning. Subsequent compaction brings particulates close together and allows for the development of ionic attractions between the CMC and particulates.

Discussion

The manufacture of compound animal feeds involves nowadays a wide variety of ingredients, all differing in their ability to contribute to physical quality of animal feeds. Although efforts have been undertaken to quantify the effects of specific diet ingredients on pellet durability or hardness (Israelsen et al., 1981; MacMahon and Payne, 1991), responses of the pelleting process to changes in diet ingredients are often found to be inconsistent. Simple regression analysis using constituents and bulk density figures from Table 1 revealed that bulk density of the feed mash, at a first approximation, seems the main factor influencing physical pellet quality. Other factors, however, may obscure this effect. An increase in bulk density showed an increase in physical pellet quality. Press capacity seemed to be most affected by the fat content present in the raw material. Adding fat increases press capacity, which is in line with expectation. Die wear seemed to be most influenced by bulk density and fat content of the material. Increasing bulk density and fat content decreased die wear.

It should be born in mind that the large variation in nutrient functionality within raw materials also affects pellet durability and specific energy consumption as has been shown for instance by Friedrich and Robohm (1981) for the inclusion of coconut cake in dairy cattle feed originating from five different regions. The variable effects of raw materials on physical properties of pelleted feeds can therefore be attributed to differences in chemical and nutrient composition, resulting from different geographical regions where the plant crops have grown or from differences in processing conditions (Friedrich and Robohm, 1981; Lake, 1991; Wilson, 1994). The use of figures relating the composition of the diet ingredient (e.g. the content in g kg^{-1} of starch or protein) to physical pellet quality of compound animal feeds is therefore of limited value as long as the exact overall quality and processing history of the diet ingredient is not known. In addition to process parameters during pelleting, physico-chemical properties of diet ingredients may provide a worthwhile guide line to estimate physical quality of pelleted feeds.

Several constituents of diet ingredients have been reported to affect physical pellet quality. Gelatinized starches and raw protein make a positive contribution to pellet hardness and durability (Wood, 1987), whereas fats added in the mixer or conditioner impairs physical quality of pellets (van Vliet, 1981). Sugar, not in the form of molasses, incorporated in feed

mash subject to pelleting, in general, increases power requirements of the pellet mill (Aumaitre et al., 1978). Part of the sugar may solubilize during conditioning and pelleting. Subsequent drying in the cooling phase will cause sugar to recrystallise or to form a glass (van den Berg, 1992) which may act as a binder. Sugars in the form of molasses positively affect pellet hardness and durability, because a large part of its sugar is already solubilised.

Fibre has a resilient character when incorporated in livestock diets. Furthermore, large fibre strands are possible weak spots, which facilitate breaking of the pellet. However, the fibre-like structure may also prove benificial in that it may entangle other particles (Rumpf, 1958). Upon establishing bonds, this may aid in maintaining structural integrity of the pellet. However, no research has been conducted to proof if such a binding mechanism exists.

It has been pointed out that functionality of constituents of a diet ingredient is dependent on processing history and the interactions of the different constituents during the manufacture of the animal feed. 'Free' fat affects the process of gelatinization of starches (Eliasson, 1981a,b) and may therefore affect the functionality of starches during the manufacturing stage and its subsequent physical pellet quality.

From results of Mohsenin and Zaske (1976), Sihag et al. (1991) and Nathier-Dufour et al. (1995) it can be concluded that future research in pelleting behaviour of raw materials and derived physical pellet quality should focus more on the effect of composition of raw materials and of their rheological behaviour on these properties. In so far we know, the approach of rheological characterization of diet constituents and its subsequent effect on pellet physical quality has never been undertaken. Relaxation behaviour of the material subjected to compression, is depending on elasticity, viscosity and fracture properties of the feed mash. This relaxation will influence expansion and eventually durability of the pellets formed. Increasing the hold time of the material under compression increases the relaxation of stresses in the materials and so post-compression expansion of the material is decreased. This will in turn increase durability of the feed (Mohsenin and Zaske, 1976). An important factor is that rheological behaviour of ingredients may be affected by water content, even by changes over a relative small range of 11.0-12.8% units as has been shown by Nathier-Dufour et al. (1995).

This overview is primarily concerned with the understanding of physical quality. An attempt has been made to discuss factors related to feedstuff components that contribute to the physical quality of pellets in terms of durability and hardness. However, the authors are aware of the fact that changes or improvements in feed processing should be weighed against improvements in nutritive value and in animal performance.

Studies with dairy cattle, pigs and poultry have been covered in literature with respect to nutritional effects of pelleted animal feeds.

Minson (1963) and Moore (1964) reviewed the effect of pelleting or wafering forages and its effect on nutritive value. Their review shows that dry matter and crude fibre digestibility often is slightly decreased due to pelleting. A pronounced effect of fineness of grind on the digestibility of the structural components (crude fibre and cellulose), with finer grind inducing lower digestibility coefficients was found by Blaxter and Graham (1956). Digestibility of cell constituents was also decreased but less severe (Blaxter and Graham, 1956). In reviewing nutritive value of pelleting of forage for beef cattle and sheep, Beardsley (1964) concluded that animal performance may be increased by grinding and pelleting. Feed intake may be increased by as much as 25%, daily gain by 100% and feed efficiency by 35%. Recent studies from in situ experiments indicates that processing (grinding, mixing and pelleting) affects the disappearance of amino acid nitrogen (van Straalen et al., 1997). The authors attributed this effect to a higher disappearance of nutrients from the nylon bags, due to the decrease in particle size mediated by the processing steps.

In pigs, pelleting generally results in a decreased feed intake of 2 percent, an increase in weight gain of about 7 percent and an improved feed utilisation of about 8 percent. However, a higher incidence of ulcers and stomach erosion is noted, most probably due to the smaller particle size induced by the processing steps (Vanschoubroeck et al., 1971) .

Poultry benefices from the pelleting process as has been reviewed by Moran (1989). One of the determinant factors in the preference of poultry of pelleted feeds over mashes is the easier prehension of the feed. The size of the pellet should fit the size of the oral cavity to minimize the work involved in prehension, time spent at the feeder and competition for food (Reddy et al., 1962; Jensen et al., 1962; Savory, 1974)

There may be definite conflicts between physical, hygienic and nutritional quality of pelleted animal feed. If pelleting, irrespective the type of conditioning, results in very hard or low durable pellets, feed intake by livestock may be reduced and utilization of nutrients may be reduced due to undesired chemical reactions. However, routine processing conditions for the single pelleting process generally are not referred to as rigorous conditions. For consequences of technological treatments on the nutritional value the reader is referred to recent articles on this subject (Van der Poel et al., 1995; Voragen et al., 1995). Formation of Maillard products for example do influence the nutritional properties of proteins, carbohydrates, lipids and fibres (Van Soest and Mason, 1991; Voragen et al., 1995). It is therefore that concerted research by the disciplines nutrition and feed technology is essential for further progress in feed processing techniques.

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4 Effects of water, steam and shear conditioning on the protein quality of soy grits.

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Effects of water, steam and shear conditioning on the protein quality of soy grits.

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Abstract

The effect of adding steam (at 0.6 and 1.2 bar) and water (1.9% and 64% of the flux of soy grits) in a conventional barrel type conditioner and its effects on protein dispersibility index (PDI), nitrogen solubility in 0.2% KOH and trypsin inhibitor activity (TIA) of commercial soy grits was studied. In addition, conditioned soy grits were subjected to expander treatment at two different screw speeds (2 and 3 rps) and PDI, NSI and TIA was measured. Analysis of variance was used to determine the effects of steam pressure, water addition and screw speed on the mentioned protein quality parameters. In addition, resultant temperature from steam addition was used in an analysis of covariance to determine the effects of temperature as a covariate, water addition and screw speed on protein quality parameters.

A significant interaction for steam pressure and water addition was found on all parameters. The highest denaturation or inactivation of the protein was found at the combination incorporating high steam and high water addition. Screw speed during expander treatment did not significantly affect protein quality parameters.

It was concluded both from this study and from literature that PDI is a better parameter to evaluate technological treatments when low amounts of motor power are dissipated (<110 kJ kg^{-1}) and that NSI in 0.2% KOH is a better parameter to discriminate between technological treatments when moderate to high amounts of motor power are dissipated in soy grits. TIA decreased with increasing water or steam content. The effect of trypsine inactivation in soy grits was highest at higher temperatures and was not affected by screw speed of the expander.

Introduction

The potential utilization as feeds for farm animals of plant materials which have been subjected to thermal processing, requires knowledge on their chemical as well as nutritional consequences, particularly of proteins. Thermal processing of soybean meal has proved valuable to desolventize and to improve the flavour of soybean meal or flour (Kinsella 1979) and to eliminate enzyme inhibitors (Liener 1980). The commercial preparation of soybean meal for use in livestock diets induces physico-chemical changes and the extent of these changes varies with the applied process variables such as product temperature, moisture content and equipment used (Thomas *et al.* 1997). Moist heat, for example, rapidly insolubilizes soybean proteins (Smith and Circle 1978).

Expander processing has been introduced as a new unit operation in the animal compound feed industry (Veenendaal 1990; Pipa and Frank 1989). Expanders are mainly used as preconditioners prior to the actual shaping process of feed pellets. They consist of a barrel in which a slotted screw and mixing bolts impart a mixing and shearing action towards the processed feed mash. Part of the motor power of an expander is transferred to the feed mash, which results in an increase in product temperature (Pipa and Frank, 1989) and alters physical

and chemical properties of this feed mash (Thomas *et al.* 1998).

The effects of thermal processing on the elimination of proteinaceous antinutritional factors in beans have been described by Rackis *et al.* (1986) and Van der Poel *et al.* (1990). Literature on the influence of process variables such as steam or water addition on the changes in functional properties is scarce, especially related to the newly introduced expander. Extruders normally employ a fixed die, whereas expanders have an adjustable annular gap. In construction, expanders and extruders are largely similar except for this outlet. Experiments conducted with single screw extruders are therefore relevant to this study.

In this study the effects of steam and water conditioning and shear conditioning (expander processing) of commercially available soy grits were investigated. Protein dispersibility (PDI) and solubility (NSI in 0.2%KOH) and the level of trypsin inhibitor (TIA) were used as the parameters to describe protein quality.

Materials and methods

Untoasted soy grits were purchased from Cargill BV, Amsterdam, The Netherlands. The batch soy grits was characterized by its mean particle size and chemical composition.
The soy grits were used to evaluate the effect of adding water (WA); 1.9 % and 64% tapwater (at a flow rate of 250 kg hour^{-1} of the feed mash) and steam (ST; 0.6 and 1.2 bar) via a conventional barrel type conditioner on its protein quality. After conditioning, the material was subjected to expander treatment at two different screw speeds (SS; 120 and 180 rpm). Since the heat- and water content of steam is also dependent on the used equipment (Maier and Gardecki 1992), the resultant temperatures of the feed mash, instead of steam pressure, were also used to study its effect on protein quality.

Soy oil (1.5%) was added to the material in the conditioner in order to improve flow behaviour of the material, since preliminary tests showed difficulties when high PDI soy grits were subjected to expander processing. Fat content of the processed material was analysed as a check on irregularities during processing.

The facilities of the Wageningen Feed Processing Centre (Figure 1) were used to conduct the experiment. The processing line consists of a storage bin, a continuous variable feeder unit, a conditioner with possibilities to add water, steam and liquids and an expander (150 mm ϕ; Almex BV Zutphen, The Netherlands) fitted with a 22 kW engine. After processing the material was cooled in a Robinson counterflow bunkercooler. Specific mechanical energy (SME) was calculated as the net amount (corrected for power consumption when running idle) of expander motor power that is dissipated in 1 kg of feed mash during expander treatment.

Product temperatures after conditioning were measured using a thermos flask.

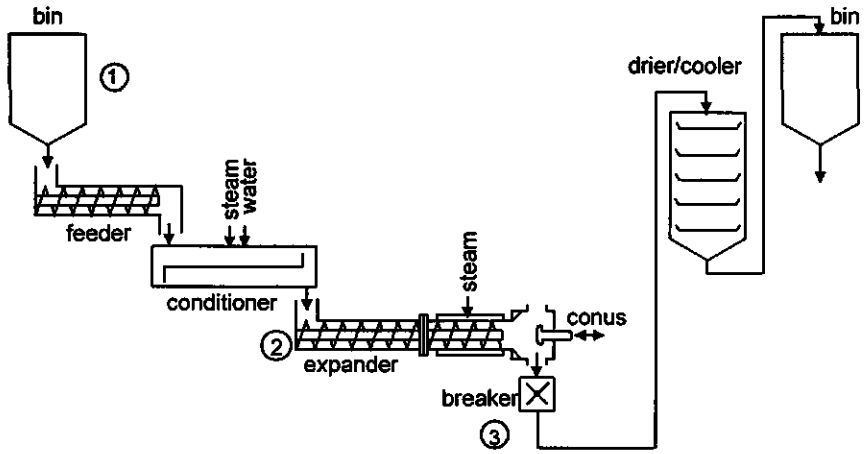

Figure 1: Flow sheet of the conditioner and expander line used. Encircled numbers indicate spots were samples were taken.

A thermocouple (Tempcontrol, Voorburg, The Netherlands) was fitted into the last mixing bolt just before the exit of the expander. This temperature was regarded as the exit temperature of the expander. A modified parabolic-shaped expander cone was used with no slots.

During every run the mass flow of the soy grits through the installation was measured; for one minute the flow of the meal was redirected into a tared container, weighed and recalculated to kg hour^{-1}.

Samples of the raw materials (sample spot 1; Fig. 1) and of the processed materials, after arriving at steady state conditions, were taken. Samples of the processed soy grits were taken after conditioning (sample spot 2; Fig. 1) and after the expander treatment (sample spot 3; Fig. 1).

Air dry matter of all samples was determined by drying at 50°C for 24 hours and afterwards acclimatizing to air for two hours. All samples were sequentially ground in a Retsch ZM1 centrifugal mill with no sieve, a 1 mm sieve and a 0.5 mm sieve respectively, to ensure that material did not heat up during the grinding procedure. The material was kept in plastic bottles and stored at 4°C until further analysis.

A Fritsch Analysette 3 equipped with six sieves (2500, 1250, 630, 315, 160 and 71 μm) and a pan (<71 μm) was used to determine particle size and standard deviation of the soy grits (Table 1). The sieve analysis was performed in duplo. Mean particle size, modulus of fineness and modulus of uniformity were calculated according to Waldo *et al.* (1971) and after Stevens (1961). Modulus of uniformity was determined by pooling the material retained on sieves 1 and 2 (2500, 1250 μm), 3 and 4 (630, 315 μm) and the last two sieves and the pan (160, 71 and <71

μm). Modulus of uniformity is then expressed as the fractional weight of the pooled material multiplied by ten and rounded off to the nearest whole number. The sum of the three numbers should be 10. These numbers represent the coarse, medium and fine fraction respectively. Chemical composition and physical characteristics of the soy grits are shown in Table 1.

Table 1: Proximate composition (g kg^{-1} dry matter), protein quality parameters and particle size distribution of the used soy grits.

Composition	Values
Chemical composition	
Moisture[a]	45.1
Crude protein	558.0
Crude fiber	39.2
Crude fat	21.4
Protein Dispersibility Index (%)	74.6
Nitrogen Solubility Index [0.2% KOH] (%)	98.1
Trypsin Inhibitor Activity [mg g^{-1}]	26.9
Sieve analysis	
Particle size (Mean ± SD)	619 ± 360 μm
Modulus of Fineness	3.8
Modulus of Uniformity	1:9:0

[a] g kg^{-1} raw material

Samples were analyzed for dry matter by oven drying during four hours at 103 °C. Crude protein was analyzed using standard Kjeldahl procedure. Crude fat was analyzed according to ISO-CD6492 (1995). Crude fibre was determined using NEN 5417 (1988).

Protein Dispersibility Index (PDI) was determined according to a modified AACC 46 - 24 procedure. One-hundred (100) ml of distilled water is brought in a Waring blendercup, 20 ± 0.1 gr of (un)processed soy grits are added. After stirring, 200 ml of aqua dest. is added with which the stirring rod is cleaned. The cup is then placed on the blender and the cooling and the motor are mounted. Temperature is held constant at 25 °C, speed of the mixer is held constant at 8500 rpm. After 10 min. the suspension is poured in a beaker. After sedimentation the upper layer is poured into a 80 ml centrifuge tube and centrifuged (r.c.f. = 1400 g) for 10 minutes. N-content is determined according to standard Kjeldahl procedure. The PDI (%) is then expressed as: (N in supernatant [g kg^{-1}]) / (N of the sample [g kg^{-1}]) * 100.

Nitrogen Solubility Index (NSI) was determined according to a modified procedure of the AOCS. Five gr (± 0.01 gr) of sample is weighed and put in a 400 ml beaker. Two-hundred (200) ml of a 0.2% KOH solution (30 °C) is added and the mixture is stirred with a magnetic stirrer for 90 minutes, at such a speed that no air is incorporated and the sample does not settle.

After stirring, the slurry is quantitatively put in a measuring flask of 250 ml. Distilled water is added up to 250 ml. The slurry is allowed to stand for a few minutes until the coarse material has settled. Forty (40) ml of the liquid is decanted into a centrifuge tube and centrifuged for 10 minutes at 340 g. The supernatant is filtered over a Schleicher & Schuell folded filter 595½, ϕ150 mm. Twenty-five (25) ml of the filtraat is used for the determination of crude protein according to standard Kjeldahl procedure. NSI (%) is expressed as: (N-content of supernatant [g kg^{-1}]) / (N-content of sample [g kg^{-1}]) x 100.

Trypsin inhibitor activity (TIA) was analysed according to NEN 3575 (1994). Trypsin inhibitors are extracted from the sample at a pH of 9.5. An amount of trypsin inhibitor from the sample is added to a standard solution of trypsin. After mixing, the residual trypsin activity is measured by determining the amount of p-nitroaniline formed from the (added) substrate benzoyl L-arginine p-nitro-anilide (L-BAPA) using a spectrofotometer. Results (mg g^{-1} product) are expressed on dry matter basis.

Treatments were arranged according to a 2*2*2 factorial design with 4 replicates after expander treatment. Treatments consisted of adding steam at two steam pressure levels (0.6 and 1.2 bar), addition of water at two levels (1.9 and 64% of the flow of the soy grits) and two screw speeds at the expander (120 and 180 rpm). Statistical analysis was performed by using steam pressure and water addition after conditioning (Model 1; 8 replicates per treatment) or by using steam pressure, water addition and screw speed after combined conditioner and expander treatment (Model 2: 4 replicates per treatment). Non-significant interactions were removed in the order of least-significant interactions first. All analysis were conducted using the SAS-package (SAS Institute Inc 1990).

Model 1: $Y_{ijl} = \mu + WA_i + ST_j + Interactions_{ij} + error_{ijl}$

Model 2: $Y_{ijkl} = \mu + WA_i + ST_j + SS_k + Interactions_{ijk} + error_{ijkl}$

With μ=general mean; i = 1 .. 2, levels of water (WA); j = 1..2 levels of steam (ST); k = 1..2, levels of screw speed (SS); l = number of replicates. When significant interactions were present, differences between the combinations were tested using the scheffe procedure (SAS Institute Inc 1990).

The effect of steam pressure and its associated quality on physical and chemical properties of feed raw materials is to a large extent dependent on machine properties (Maier and Gardecki 1992). Therefore, the effect of temperature as the resultant of steam-, and water addition on protein quality parameters was analysed using analysis of variance with temperature as a covariate. The same approach as previously described in the text was followed in which the

effect of temperature and water on protein quality parameters was analysed after conditioning (Model 3) and the combined effect of conditioner and expander treatment after the expander (Model 4).

Model 3 $$Y_{ij} = \mu + WA_i + \beta_1(x_i - \bar{x}) + \beta_{2i}(x_i - \bar{x}) + error_{ij}$$

Model 4 $$Y_{ijk} = \mu + WA_i + \beta_1(x_i - \bar{x}) + \beta_{2i}(x_i - \bar{x}) + SS_j + error_{ijk}$$

With μ=general mean; i = 1 .. 2; β_1 = the average slope; β_{2i} = the slope on the two different water levels. $\bar{x}$ = mean temperature at the exit of the conditioner (model 3) or mean temperature at the exit of the expander (model 4). x_i = temperature at high (WA_H) or low (WA_L) water level. For abbreviations see Table 2.

Table 2: Levels of the different factors employed in the experiment. Steam and water were added in the conditioner; screw speed was varied during expander processing.

Factor	Abbreviation	Level	
		Low (Abbr. $_L$)	High (Abbr. $_H$)
Steam pressure	ST	0.6 bar	1.2 bar
Water addition[1]	WA	1.9 %	64%
Screw speed expander	SS	2 rps	3 rps

[1] Amount of water added to the stream of soy grits. On a v/w basis; flow of soy-grits is 250 kg/hour.

An extra replication was performed at the low water, high steam and low screw speed level of the experiment. This additional replicate is incorporated in the analysis of variance. Since no temperature was available it is excluded from the regression analysis.

Results

The unprocessed soy grits had a PDI-value of 74.6 %, an NSI-value of 98.1 % and TIA amounted 26.9 mg g^{-1} product (Table 1). These values indicate that a large portion of the protein was present in the native form.

Table 3: The effect of steam level and water addition on TIA, PDI and NSI at the exit of the conditioner.

	Treatments[1]				sem[2]	Probability levels of the effect[3]		
	ST_LWA_L	ST_LWA_H	ST_HWA_L	ST_HWA_H		Water	Steam	Interaction
Protein quality parameters:								
PDI	43.5[a]	37.8[a]	40.6[a]	22.5[b]	3.1	***	***	**
NSI	94.5[a]	85.8[b]	95.1[a]	78.4[c]	0.9	***	**	***
TIA	11.1[a]	5.8[b]	10.2[a]	2.1[c]	0.7	***	**	#
Processing parameters:								
Temperature exit conditioner [°C]	95.5[a]	78.6[b]	97.8[a]	89.5[a]	2.1	***	**	*

Different superscripts per row indicate significant differences at $p<0.05$.

[1] For abbreviations see table 2.

[2] Pooled standard error of the LSmean.

[3] ns = $p>0.1$; # = $p =< 0.1$; * = $p < 0.05$; ** = $p < 0.01$; *** = $p < 0.001$.

Effects after conditioning

Both water-, and steam addition decrease protein quality parameters when measured as PDI, NSI and TIA (Table 3). At high steam level and at high water level more protein of the soy grits was denatured than at the low water and steam levels as can be derived from the values of PDI, NSI and TIA in Table 3. The interaction between steam and water was significant at $p<0.05$ with respect to protein quality parameters and temperature at the exit of the expander. The lowest protein quality parameter figures were found at the combination of high steam and high water; PDI 22.5 %, NSI 78.4% and a TIA of 2.1 mg g^{-1} (Table 3). The highest protein quality parameter values were found at the low water level; PDI was 43.5% at low steam level, NSI was 95.1 at the high steam level and TIA was 11.1 at the low steam level. Addition of tapwater during conditioning affected temperature; temperatures were lower at the high water level. Temperatures were higher when more steam was incorporated. The lowest temperature (78.6°C) was reached at the combination high water and low steam. The highest temperature (97.8°C) was reached at the combination of high steam and low water.

Effects after expander processing

In all cases water- and steam addition affected the protein quality parameters. Moreover, for all protein quality parameters tested, interactions between effects of steam and water were found. Screw speed of the expander did not significantly affect the protein quality parameters (Table 4). From table 4 it follows that PDI values at the lower water level (33.6 and 30.1%) are in between the PDI values found at high water level and this accounts for the non-significant main-effect for water. A high PDI value (36.9 %) was found at ST_LWA_H and the lowest PDI value (23.6%) was found at ST_HWA_H. NSI showed the highest values on the low water level (94.8% and 94.4% at ST_LWA_L and ST_HWA_L respectively). NSI-values declined on the high water level with the decrease in NSI being most pronounced at the high steam level (78.7%). The lowest amount of TIA (2.9 mg g^{-1}) was found at the ST_HWA_H treatment. The highest amount of TIA (10.0 mg g^{-1}) was found at the ST_LWA_L treatment, although not significantly different from the figures found at ST_LWA_H and ST_HWA_L. In all cases, the lowest values for the protein quality parameters are on the combination of high level of steam pressure and high level of water addition (ST_HWA_H). This treatment has therefore the highest potential to denature or inactivate proteins.

Specific Mechanical Energy (SME: kJ kg^{-1}) is influenced by water and by screw speed and their interaction. High energy consumption is associated with high screw speed and low water content. It was observed, during the experiment, that on the high water level, the feed mash flowed very easy along the cone. Therefore, not enough motor energy could be dissipated into

Table 4: The effects and probabilities of steam level, water addition and screw speed of the expander on trypsin inhibitor activity (TIA) [mg g^{-1}], protein dispersibility index (PDI)[%], nitrogen solubility index in 0.2 %KOH (NSI) [%], temperature at the exit of the expander [°C] and specific mechanical energy (SME) [kJ kg^{-1}]. Figures are LSmeans.

									Probability levels of the effect[3]				
Treatments[1]:	SS_L	SS_H	sem[2]	ST_LWA_L	ST_LWA_H	ST_HWA_L	ST_HWA_H	sem[2]	SS	WA	ST	ST*WA	SS*WA
PDI	32.0	30.1	1.8	33.6^{ab}	36.9^{a}	30.1^{ab}	23.6^{b}	2.5	ns	ns	**	#	-
NSI	88.9	87.5	0.7	94.8^{a}	84.9^{b}	94.4^{a}	78.7^{c}	1.0	ns	***	**	**	-
TIA	7.7	7.4	0.6	10.0^{a}	7.5^{a}	9.7^{a}	2.9^{b}	0.8	ns	***	**	*	-
Temp. exit Expander [°C]	101.3	102.5	1.0	110.6^{a}	88.8^{b}	111.5^{a}	96.8^{c}	1.4	ns	***	**	*	-
Treatments:	ST_L	ST_H		SS_LWA_L	SS_LWA_H	SS_HWA_L	SS_HWA_H						
SME[4]	50.8	57.4		76.0	8.0	110.0	22.5	4.3	***	***	ns	-	#

Different superscripts per row and per interaction indicate significant differences at $p<0.05$.

[1] See table 2 on abbreviation of treatments.

[2] Pooled Standard error of the LSmeans.

[3] See table 3 for significance levels.

[4] Specific Mechanical Energy [kJ kg^{-1}].

the soy grits.

Effect of temperature as a covariate on protein quality.

Temperature affected protein quality parameters both after conditioning and after expander treatment (Table 5). The slope, averaged over water levels, for all dependent variables deviated from zero. The probability of the interaction (Table 5) was never significant, indicating that the slopes associated with different water levels were similar. Regression coefficients were negative indicating that with an increase in temperature, a decrease in PDI, NSI and TIA was observed. The effect of water addition on the protein quality parameters was tested at the mean temperature after each processing step; this means at 90 °C at the exit of the conditioner and 102 °C at the exit of the expander. Differences between water levels were significant in all cases. From figure 2 it follows that the treatments with low water content had higher temperatures than the treatments with high water content. This can also be observed from table 4 were low water level has a higher temperature (111.1°C) than high water level (92.8 °C); figures are means averaged over steam level. As a check, multiple linear regression was performed with both absolute water

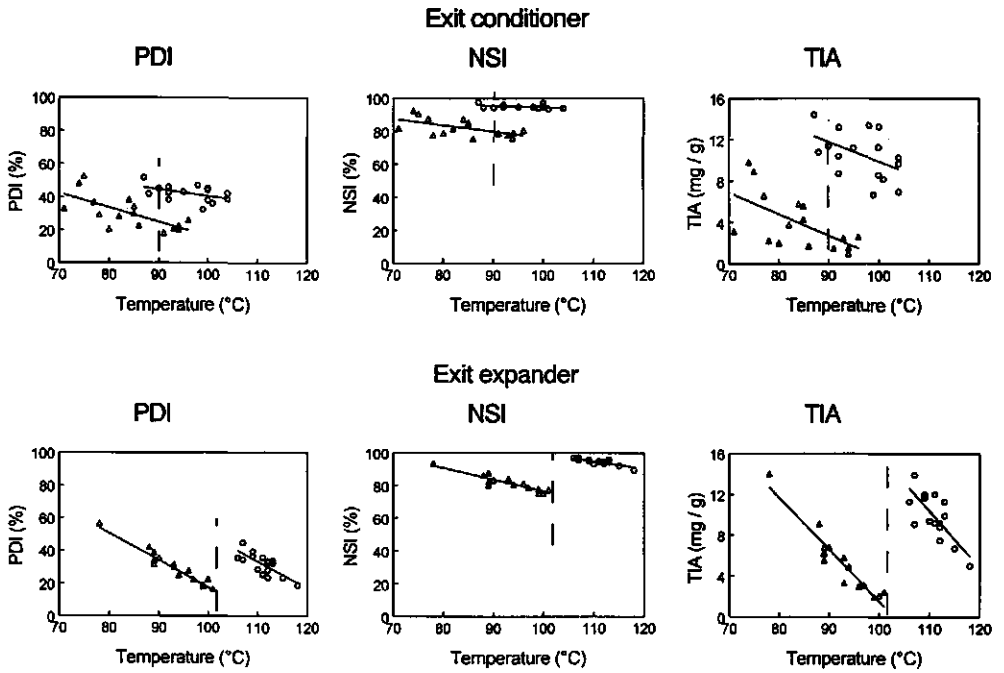

Figure 2: Effect of temperature on PDI (%), NSI (%) and TIA (mg g^{-1}) after conditioning and after expander treatment. o indicates low water level, Δ indicates high water level. Dashed vertical lines indicate where the two regression slopes (at 90 and 102 °C) have been compared.

content and temperature. The resultant response surface showed no interaction between water content and temperature. The directions of the slopes of the curves for temperature did not drastically change (<15%). Furthermore, water contents differed greatly which may violate the assumption of a linear relation between water content and temperature. Therefore, the described approach of analysis of covariance seems the most appropriate.

Discussion

The denaturation of proteins requires water and heat (Smith and Circle 1978). This is also shown by the data in this study: higher water contents and an increasing amount of added steam facilitate loss of dispersibility and solubility of protein. Moreover, interactions exist between steam and water addition with respect to protein denaturation both after conditioner and after expander treatment. Literature on extrusion of full fat soy flour from Mustakas *et al.* (1970) shows that there is a pronounced effect of the interaction between moisture and temperature on trypsin inhibitor activity and NSI. This was also found in the present study (Table 5). The lowest observed temperature in their experiment was approximately 121°C, which was higher than the maximum temperature reached in this study (119°C). Water content in the study of Mustakas *et al.* (1970) was lower (maximum 30%; wet-basis) than the highest water content in this study (64% v/w basis). The effect of moisture level on NSI after extrusion was also found by Marsman *et al.* (1993): extrusion of untoasted soybean meal with 35% moisture gave lower NSI values when compared to extrusion of soybean meal with 26% moisture.

In this study no effect is found of screw speed of the expander on protein quality parameters. Available literature on extrusion technology suggests that, depending on feed raw material conditions, the effects of screw speed may counteract each other. High screw speeds, in general, leads to shorter residence times (Mustakas *et al.* 1970; Van Zuilichem 1992) and higher amounts of shear, whereas low screw speeds increase residence time and lower the amount of shear received by the material. High levels of water may act as a lubricant prohibiting denaturation by shearing. From table 4 it can be seen that at the high water level, specific mechanical energy (SME) was considerable lower compared to the low water level. The effect of moisture on protein quality parameters may have been mediated by the higher thermal conductivity (Wallapapan *et al.* 1984) and water diffusivity (Hsu, 1983) values of soy flour found at high(er) moisture contents. Prolonged processing times (in the conditioner stand-alone or during conditioning and subsequent expander processing) then facilitates diffusion of water. This will affect the denaturation process. Although differences in protein quality parameters between conditioner and expander were not present (except PDI in the case of low water level; see later) it is observed from figure 2 that the spread around the regression lines becomes less and the slope of the regression lines becomes more negative after expander processing (Table 5). TIA was inactivated by conditioning and expander processing. Both steam and water are causative factors to facilitate TIA inactivation in soy grits under the conditions of this study. Product temperature

Table 5: Effect of temperature as a covariate at the exit of the conditioner and at the exit of the expander on PDI, NSI and TIA.

Effect:	Water addition[1]				Screw speed				Temp.	Interaction		
	WA_L	P WA_L=WA_H[2]	WA_H	sem[4]	SS_L	P SS_L=SS_H[3]	SS_H	sem[4]	P[5]	WA_L	P WA_L=WA_H	WA_H
Exit Conditioner												
PDI	44.5	***	24.7	2.1					***	-0.41	ns	-0.87
NSI	95.3	***	79.7	1.1					*	-0.09	ns	-0.38
TIA	11.7	***	2.6	0.8					**	-0.19	ns	-0.21
Exit Expander												
PDI	46.7	***	15.1	2.6	30.7	ns	31.1	1.9	***	-1.65	ns	-1.66
NSI	98.7	***	75.4	1.2	87.4	ns	86.7	0.8	***	-0.46	ns	-0.70
TIA	14.9	***	0.6	0.9	7.5	ns	8.0	0.7	***	-0.56	ns	-0.51

[1] Values represent protein quality parameters at the average temperature exit conditioner (90 °C) or at the exit of the expander (102 °C).

[2] Probability for the test of effect of water addition (see table 3 for significance levels).

[3] Probability for the test of effect of screw speed.

[4] Pooled standard error of the mean.

[5] Probability for the test if the regression coefficient, averaged over water levels = 0.

[6] Probability for the test if the regression coefficients, associated with water levels, are equal for WA_L and WA_H.

is related to inactivation of TIA (Fig. 2), with increasing temperature leading to decreasing TIA-levels in expander processing. According to the European Federation of Feed Manufacturers (Monari, 1993) the threshold value for feeding soy-products is 4 mg g^{-1} for residual TIA. Values of 4 mg g^{-1} TIA and lower, are found in this experiment only on the high water level and at temperatures above 95°C. However, the large amount of water used (64% on a v/w basis) makes the feed prone to microbial or fungal deterioration. Assuming a linear relation and extrapolating beyond the experimental range, temperatures of at least 123°C are necessary to lower TIA below threshold levels of 4 mg g^{-1} on the low water level (1.9% added water) used in this experiment.

From table 4 it follows that SME is low (< 110 kJ kg^{-1}) when compared to values of 1000 - 1700 kJ kg^{-1}, at 28 % moisture, found by Marsman *et al.* (1995) for single screw extrusion of soybean meal. Using these high amounts of SME, PDI was found not to be a suitable parameter to discriminate between processing conditions, since values on all treatments remained almost constant (between 6.6 and 8.0%). NSI values at this level of SME could be used to discriminate between treatments and NSI was considered a more appropriate parameter to evaluate processing conditions (Marsman *et al.* 1995).

Using multiple t-tests, differences between the interaction treatments for steam and water between conditioner and expander were tested. Significant differences were only found for PDI-values for the ST_LWA_L and ST_HWA_L treatments ($p<0.01$) between conditioner and expander (probabilities of other treatment combinations >0.1). SME values for expander processing at these treatments were also slightly higher, which indicates that the higher energy consumption may have accounted for the decrease in PDI-values. NSI was not affected by the increase in SME since SME values found in this study were to low to induce lower NSI-values, compared to those in the study of Marsman *et al.* (1995). Overall, the effect of water and steam addition resulted in the largest decrease in protein quality parameters, with the largest effect on changes in protein quality parameters found directly after conditioner treatment with respect to the starting material. Subsequent expander treatment did not significantly affect protein quality parameters except in the case of PDI.

Conclusions

1) Protein quality parameters during combined conditioning and expander treatment are only affected by the temperature and moisture levels and only slightly to neglectibly by differences in screw speed at low amounts of dissipated motor energy.

2) The difference in levels between PDI and NSI (%) found in this study and from literature indicate that PDI is a better parameter to discriminate between technological treatments when low to intermediate amounts of SME are used (< 110 kJ kg^{-1}). NSI is a better parameter to evaluate technological treatments when moderate to high levels of SME are used.

3) The protein quality parameters, PDI, NSI and TIA all decrease with increasing water content and raising temperature.

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5 Effects of process-conditions during expander processing and pelleting on starch modification and pellet quality of tapioca.

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Effects of process-conditions during expander processing and pelleting on starch modification and pellet quality of tapioca.

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Abstract

In this study the effect of processing conditions during the manufacture of pelleted animal feed is related to the degree of gelatinization of tapioca starch as measured by the Amyloglucosidase method (SGD_{AGS}) and Differential Scanning Calorimetry (DSC). The process-conditions were related to some physical quality characteristics of the pelleted feeds as well. Physical pellet quality was evaluated for hardness and durability which incorporated tests that are also used in the feed industry. The processing line used in the experiment consisted of a conventional barrel type conditioner where steam pressure (100 - 180 kPa) and tap water addition were varied (0 - 52 g kg^{-1} of the feed debit). Subsequently, expander processing was carried out and the screw speed of the expander was varied (60 - 140 rpm) as well as the amount of dissipated (expander) motor power (1.3 - 5.7 kW). Response surface regression methodology was used to assess the direction and relative magnitude of changes in processing conditions on starch modification, physical pellet quality and some system variables.

The results show that no combination can be found that satisfies one common maximum or one common minimum value for all of the dependent variables tested. Hardness and durability values of the pellets were most affected by steam pressure and the amount of used expander motor power, whereas starch modification was mainly affected by steam pressure and water addition.

Introduction

Starch is, apart from cellulose, the most abundant carbohydrate present in plants. The starch is stored in granules and acts as a energy reserve. Starches can be present in seeds; e.g. corn, wheat and rice; in tubers as like potato and tapioca, and in stalks; e.g. sago. Starches have traditionally been used in the food industry for their nutritive value and for the functional properties they have in food systems. For instance, they may provide texture, enhance thickening of liquids and keep solids suspensed (Smith, 1984). Starches are also used in non-food applications as, for instance, adhesives for board, paper or etiquettes in the paper industry. Different processes have been developed to create a starch(-derivate) that incorporates the desired properties for a specific adhesive (Kennedy and Fisher, 1984).

Many literature sources describe the interrelations between processing equipment, e.g. extrusion equipment and functional properties of starches (Lund 1984; Camire *et al.* 1990; Eliasson and Gudmundsson 1996). However, most of the research conducted on starches is

carried out at relatively high moisture conditions (>30% water) and the question arises to what extent these results are comparable to low moisture processing conditions as used during the manufacture of pelleted animal feed. Heffner and Pfost (1973) and Skoch *et al.* (1981) investigated the effects of conditioning and pelleting equipment on the degree of gelatinization of starches. In the study of Heffner and Pfost (1973), an increase in the degree of gelatinization after conditioning was found, whilst Skoch *et al.* (1981) did not find any differences in degree of gelatinization after conditioner treatment. Pelleting the feed mash raised the degree of gelatinization to approximately 25% in both studies. Moisture content in both of the studies was between 10 and 15 % during the different processing stages.

Physical quality of pellets is affected by the level of pre-gelatinized starch present in the feed material. High levels of (pre-) gelatinized starch increase the pellet hardness and the pellet durability (Wood 1987). Apart from the study of Wood (1987) it appears that little systematic research has been undertaken to evaluate the influence of a certain raw material or one of it's components to the physical quality of the derived pellets. MacMahon and Payne (1994) gave an overview, based on empirical data, of the effects on pelletability and pellet quality of a compound animal feed, due to incorporation of a certain raw material. However, large variations in pellet quality can be found since raw materials used in feed manufacturing can vary considerably in composition, depending on origin and previous processing steps (Wilson 1994; Lake 1991).

To gelatinize starches the following factors are important (Lund 1984; Remsen and Clark 1978; Smith 1984; Bhattacharya and Hanna 1987).

- water: necessary for swelling and weakening of the different bonds in the starch kernel.
- heat: facilitates entry of water and causes part of the granule (amylose) to solubilize. In the presence of enough water, it induces melting of crystalline regions.
- shear: physical force is neccesary for disruption of the kernel, thereby facilitating the entry of water and aiding in swelling and solubilization of the kernel and starch respectively.
- residence time: prolonged periods of time increases the combined effects of shear, water and heat on the degree of starch gelatinization.

However, it should be stressed that under low water conditions prevailing in the pelleting of animal feeds, differences in gelatinization behavior can be expected as opposed to the higher (>30%) moisture content in, for instance, extrusion applications (Harper 1981; Eliasson and Gudmundsson, 1996). Recent work of Keetels (1995) suggests that in concentrated starch systems amylose in combination with friction forces are the predominant factors in adhering

starch particles. The friction force between (swollen) particles is further enhanced due to their irregular shape. Although water content was still higher than used in routine feed manufacturing (often 30% w/w concentrations of starch were used), her results may provide guidelines on binding mechanisms and consequences for the physical properties in feed mashes, high in starch content.

Various authors have argued that the gelatinization behaviour of starch is a multi-factorial process (Remsen en Clark 1978; Lund 1984; Eliasson and Gudmundsson 1996). Within the scope of this study no attempt has been made to discriminate between the various stages that may be observed during modification of starch. The general term '(degree of) gelatinization' has been adopted to relate the various effects of starch modification in any sense to the observed phenomena. Two methods have been chosen to evaluate the starch modification; an enzymatic method (SGD_{AGS}) and Differential Scanning Calorimetry (DSC). SGD_{AGS} is used in feed manufacturing to determine the amount of readily available starch for the metabolism of either the animal or microflora. The second method (DSC) is used in food technology to evaluate the state of the materials and their relation to functional properties of the (food) system under study.

The present study aims to quantify the effects of different processing conditions of conditioning and pelleting equipment on the degree of gelatinization of tapioca starch. In a conventional barrel type conditioner, the temperature was varied by changing the steam pressure and moisture content was changed by adding tap-water. This is done, since temperature, as well as water contents, affects the gelatinization of starch. In addition, in the expander the amount of dissipated energy was varied as well as the speed of the expander screw. Mechanical energy dissipation during extrusion processing affects gelatinization (van Zuilichem and Van der Poel 1986). Furthermore, changes in screw speed affect the residence time during expander processing (Bhattacharya and Hanna 1987; Chiang and Johnson 1976; van Zuilichem 1992).

In this study, the effects of the different processing conditions were related to the physical quality of pellets. It is hypothesized that - given a set of manufacturing equipment - the degree of gelatinization affects the physical quality of expanded and/or pelleted animal feeds, with higher levels of gelatinized starch inducing harder and more durable pellets.

Materials and Methods

Ground tapioca-chips were obtained from CHV, Veghel, the Netherlands. The ground chips were characterised by its mean particle size (Waldo *et al.* 1971) and chemical composition (Table 1).

Table 1: Composition of the milled tapioca chips.

Chemical composition		
Dry matter	889	g kg^{-1}
Total starch[a]	768	g kg^{-1}
Sugars[a]	~0	g kg^{-1}
Gelatinized starch[a]	79	g kg^{-1}
Degree of gelatinization	10.3	%
Physical characteristics		
Enthalpy of gelatinization	7.22 (±0.64) J g^{-1} DM	
Mean particle size[b]	201.6 µm[b] ± 376.5 µm	

[a] Based on Amylo-glucosidase test (in DM)

[b] Mean ± SD (Waldo *et al.* 1971).

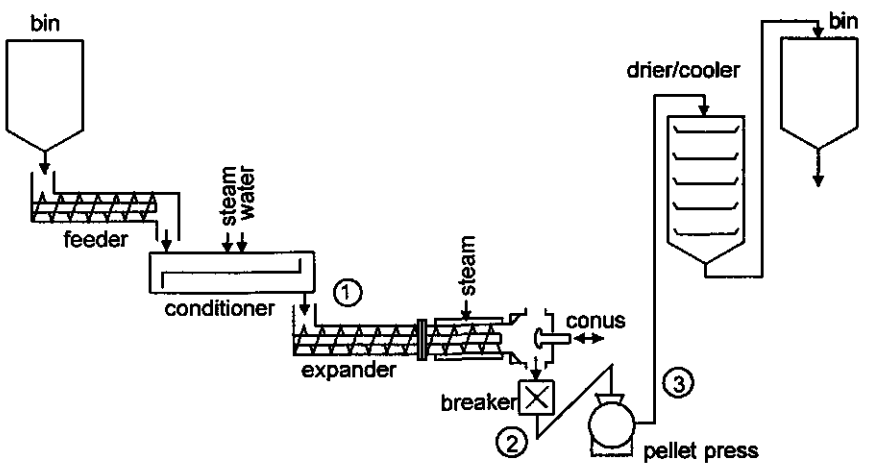

Figure 1: Flow sheet of the processing line used. Encircled numbers indicate were samples were taken.

The tapioca meal was used to evaluate the effects of water and steam addition on the degree of gelatinization of the starch fraction in a conventional barrel type conditioner. The conditioned (tap-water and steam) feed mash was subsequently processed with an annular gap expander (Contivar AL-150, Almex BV, Zutphen, the Netherlands). Tap-water was added using a volumetric flow meter. The amount of added tap-water was calculated from the feed-debit. The screw speed of the expander and motor power dissipated in the mash were varied during expander processing. The conditioned and expander treated mash was subsequently pelleted in a modified Simon Heessen pellet press of the ring-die design with two rollers using a 13*80 mm die. Pellets were cooled in a 2-deck Robinson counterflow bunker cooler. Total transit time for the pellets in the cooler was approximately 8 min 30s. Data on power consumption of the expander and pellet press were collected using a μ-mac 6000 data-logger. Power consumption values of the expander and the pellet press are corrected for idle running. The experiments were conducted using the facilities of the Wageningen Feed Processing Centre (Fig 1). Soy oil (1.5%) was added to the material in order to improve flow behaviour. Fat content of the samples was analysed as a check on irregularities during processing. Samples of approximately 1 kg each were taken after conditioning (sample spot 1: Fig 1), after expander treatment (sample spot 2; Fig 1) and after pelleting (sample spot 3; Fig 1). Samples were collected in 1 liter plastic flasks. Temperatures were measured using thermocouples (Tempcontrol, Voorburg, the Netherlands) at the beginning of the feed zone and at the annulus of the expander. The thermocouples were fitted in the tip of the mixing pins. The temperature in the first part of the feeding zone of the expander was regarded to equalize the exit-conditioner temperature. The temperature of the pellets was measured immediately after pelleting using the thermosflask method. Flow of the feed mash was held constant throughout the experiment and was, at intervals, measured by collecting the flow for one minute in a tared container. During charging of the feed bin, samples from the ground tapioca-chips were taken at random and stored for further analysis.

Air dry matter of all samples was determined by drying at 50 °C for 16 hours. Afterwards material was acclimatised for a period of one hour. All samples were ground in a Retsch ZM1 centrifugal mill in two steps: first, without sieve, then grinding over a 1 mm screen to prevent overheating. Material was kept in plastic flasks and stored at 4°C until further analysis. Dry matter of the samples was then determined by oven drying at 103 °C for four hours. The amount of moisture in the feed is calculated from both air dry matter and dry matter determinations.

To evaluate the effects of the different treatments, the degree of starch gelatinization was measured using the enzymatic amylo glucosidase test (SGD_{AGS}) and differential scanning

calorimetry (DSC). The degree of gelatinization and the DSC-values were determined after conditioning, after expander treatment and after pelleting (Fig 1: sample spots 1 to 3).

SGDags was determined in three steps according to the NIKO method (Brunt, 1992). Total starch is analysed by extracting the lower sugars with a 40% ethanol solution, followed by autoclaving for 3 h at 130°C and enzymatic breakdown (1 h at 60°C, pH 5) to glucose, using an enzyme cocktail containing amyloglucosidase, α-amylase and pullulanase (A). Glucose is subsequently determined using hexokinase and G6P-dehydrogenase. For the determination of the degree of gelatinization of starch, two additional analysis were conducted: starch is analysed as above, but without the ethanol extraction (B) to quantify the amount of starch and lower sugars. Finally, the sample was hydrolysed with amyloglucosidase (60 U g^{-1} sample) for 75 min at 50°C (pH 4.8) to determine gelatinized starch and lower sugars (C). The SGDags was calculated as a percentage of total starch after correcting for lower sugars according to: SGDags = 100 * [C-(B-A))]/A.

A Mettler-Toledo DSC12-E was used to determine the residual enthalpy of the samples after treatment. Between 15 and 20 mg of sample was weighed into a medium pressure crucible (Mettler-Toledo). Approximately 60 mg of demineralised water was added. The crucibles were sealed and left to equilibrate for one hour at room temperature to allow the water to fully hydrate the sample. The sample was then subjected to a controlled temperature program in which the sample was held isothermal for 5 minutes at 20°C and then subjected to a linear temperature rise at a rate of 5°C min^{-1}. The measured range was from 20°C to 120°C. A crucible filled with 90 mg of aluminium foil was used as a reference. The residual enthalpy is a measure for the amount of ungelatinized starch present in the sample.

The temperature at the head of the expander, the net motor power consumption of the pellet press, temperature of the pellets after pelleting and physical quality measurements were further used as dependent variables.

Physical quality determinations of pellets involved durability according to Pfost and Allen (1962) and a combination of abrasion and hardness of the pellets using the Holmen pellet-tester (Major 1984). Sieves used in both durability and hardness tests had a mesh-width of 4 mm. Hardness-tests of the pellets were conducted using the Kahl pellet tester and a general purpose compression and tension apparatus (Overload Dynamics series 100, fitted with a remote control series 900 and a 2000 N load cell). Pellets were crushed in axial direction. The length of 10 pellets was measured using vernier callipers. One pellet at a time was crushed between flat perspex platens with the speed of the moving bar set to 10 mm s^{-1}. Crushing strength of the pellet was expressed as the force at which breakage occured divided by the

length of the pellet. Mean of the 10 values is taken as the crushing strength of the pellets. For a more thorough overview of the used tests see Thomas and Van der Poel (1996 a).

Treatments were arranged according to a Central Composite Design (CCD) with each factor (water addition, steam pressure, screwspeed and net motor power) initially set at five levels (Table 2).

Table 2: Treatment combinations in the experiment. Values of the factor levels are rounded to one decimal.

Conditioner		Expander		Replications
Steam pressure (SP)	Water additon (WA)	Screw speed (SS)	Motor power (MP)	
[kPa]	[g kg^{-1}]	[rpm]	[kW]	
100	25.9	100	3.5	1
120	12.9	80	2.0	1
120	12.9	80	3.7	1
120	38.8	80	2.0	1
120	38.8	80	3.7	1
120	12.9	120	2.9	1
120	12.9	120	5.5	1
120	38.8	120	2.9	1
120	38.8	120	5.5	1
140	25.9	60	2.2	1
140	0	100	3.5	1
140	25.9	100	1.3	1
140	25.9	100	3.5	12
140	25.9	100	5.7	1
140	51.7	100	3.5	1
140	25.9	140	4.9	1
160	12.9	80	2.0	1
160	12.9	80	3.7	1
160	38.8	80	2.0	1
160	38.8	80	3.7	1
160	12.9	120	2.9	1
160	12.9	120	5.5	1
160	38.8	120	2.9	1
160	38.8	120	5.5	1
180	25.9	100	3.5	1

A CCD is a first order factorial design augmented with certain additional points to be able to estimate the coefficients of a second order surface. The points that are determined by the 2^i factorial are called cube-points. The additional points are called star-points when they are located on the axis of the factors used. The mean value for all levels of the factors is called the

center point since this point is central in the region of experimentation. The stationary point is the point in space at which all partial derivatives of the fitted regression equation with respect to x_i are zero (i is the number of factors used; 1 - 4). In addition, the second order fitted regression equation can be rewritten such that the nature of the stationary point and the entire response system becomes more clear. It involves a translation from the origin of the axis to the stationary point and a rotation of the axis system into the direction of principal orientation of the response surface. This modified form is called the canonical form of the equation. The model used (second order polynomial) is not likely to give thrusthworthy results when extended outside the region of experimentation. Thus, in cases where the stationary point is relatively far from the region of experimentation an analysis of the ridge was used. This analysis yields the line of steepest ascent or steepest descent within the region of experimentation. Its use is to find the combination of factors that maximize or minimize the response variable as a function of the distance from the centerpoint of the design to the boundary of the experimental region (Myers, 1976).

In the ridge-plots (figures 3 to 7), all factors are scaled between -1 and 1 and depicted on the left Y-axis. With use of table 3 the corresponding (unscaled) values for the four factors can be recalculated. On the right Y-axis the value of the dependent variable is given. Thus, effectively the ridge plots give the lines of steepest ascent or descent as a function of the (coded) center point of the design. Reported maximum or minimum values are estimates from the regression equation at points with (coded) radius 1 from the centerpoint (Myers, 1976).

Table 3: Coding formulas to standardise the factors to -1 and 1 for use in the ridge plots.

(SP - 140) / 40	(WA - 25.862) / 25.862
(MP - 3.512) / 2.2	(SS - 100) / 40

SP is steam pressure (kPa), WA is water addition (g kg^{-1} feed debit), MP is motorpower of the expander (kW) and SS is screw speed of the expander screw (rpm).

The general formula used is x_i - mean(highest - lowest value) / (0.5*(highest - lowest value)), i = 1 to 4. Highest and lowest value are the highest and lowest level of the i-th factor in the design. Given equations permit to recalculate the originial values from the coded variables in the ridge-plot(s). Example: See figure 3a: SGD_{AGS} after expander treatment. When an SGD_{AGS} of 30 % is wanted, the neccesary factor level(s) can be calculated as follows: draw a perpendicular line from the right Y-axis to the line representing SGD_{AGS}. From the SGD_{AGS}-line draw a line perpendicular to the X-axis. At the intersection with the line representing a scaled factor, draw a line perpendicular to the left Y-axis to obtain the scaled factor setting. From this scaled factor setting calculate the original levels. For instance, to obtain the amount of water necessary to obtain 30 % SGD_{AGS} use the line-method and obtain 0.08 scaled units from the left Y-axis. With use of the formula above:

$$0.08 = (WA - 25.862) / 25.862 \rightarrow WA \text{ is } 27.9 \text{ g } kg^{-1}$$

The other levels can be calculated likewise using the above formulas.

The arrangement of the different levels of the experiment was calculated using the CADEMO-package; Computer Aided DEsign of experiments and MOdelling (Rasch *et al.* 1992). Initially, all levels were constructed, in which steam pressure (SP) was varied between 100 and 180 kPa (absolute) pressure, water (WA) added was 0 to 5% from the feed flow, screw speed (SS) was 30 to 70% of the maximum screw speed (200 rpm) and motor power (MP) was in the range of 30 to 70% of maximum emissible power of the expander engine (22 kW). Since the expander motor was of the direct current type, maximum emissible power output is dependent on the rotational screw speed of the expander. For this reason the design was not orthogonal and rotatable as in the initial construction. Table 3 lists the used treatment combinations.

The initial conditions used in the experiment were transformed to g kg^{-1} water added to the feed mash, velocity of the screw of the expander in rotations per minute, and motorload was converted to the net motor power consumed. All data was fitted to the general quadratic model using proc RSREG from the SAS-system (SAS, 1990):

$$\hat{Y}_u = \beta_u + \sum_{i=1}^{k} \beta_i x_{iu} + \sum_{i=1}^{k} \beta_{ii} x_{iu}^2 + \sum_{i<j} \beta_{ij} x_{iu} x_{ju} + \varepsilon_u$$

in which k is the number of factors in the experiment, i = the factor of the experiment being either 1 (steam pressure), 2 (water addition), 3 (expander motor power) or 4 (expander screw speed), u is 1 to 36 representing the u-th observation in the experiment. Subsequently, x_{iu} represents the i-th factor in the u-th observation. j is a factor not equal to i.

Data were analysed in two steps. The effects of water (1) and steam pressure (2) on degree of gelatinization and residual enthalpy during conditioning were determined using samples taken directly after conditioning (Fig 1; sample spot 1). Since this is an abstract of the full design (no screw speed and no motor power) it consists of 16 experiments at the centerpoint, 4 experiments at each cube point and 1 experiment at each star point. The effect of water- (WA), steam pressure (SP), screw speed (SS) and motor power (MP) on SGD_{AGS} and DSC-values after expander treatment of the material was determined using samples obtained from sample spot 2 (Fig 1.) The latter (full) experimental design consisted of 12 replications at the center point to obtain an estimate for the experimental error. One experiment was performed at each cube point in the design and 1 experiment at each star point. Whenever the stationary point was within or close to the region of experimentation, the canonical form of the equation was used to assess the relative magnitude (eigenvalues) and the direction (eigenvectors) of the effects of the factors. Tests on significance of a factor is conducted using all the occurences of

the parameter for that factor in the equation. Thus, a test on the significance of for instance the factor steam pressure is a test on the hypothesis that all of the parameters involving steam pressure are zero. The test on lack-of-fit yields, whether or not, the second order polynoom is a reasonable model for the data. Response surface regression, canonical and ridge analysis were performed using the RSREG procedure from the SAS package (SAS, 1990).

In addition, the effects of the processing units (conditioner, expander and pellet press) on SGD_{AGS} and DSC were tested using the centerpoints of the design. One-way analysis of variance using the centerpoints was used to determine whether differences existed between the processing apparatus. Differences between conditioner and expander and between expander and pellet press were tested using contrasts. The following model was used:

$$Y_{ij} = \mu + \beta i + \epsilon_{ij}$$

in which i = conditioner, expander or pellet press and j is the number of replications per treatment; either 16 or 12. Y_{ij} is either SGD_{AGS} or DSC from the j-th replication of treatment i.

Results

Although the statistical design was not exact orthogonal and rotatable, exploration of the design indicated that there still was a 'broad base' to support the response surface. Calculated variance inflation factors (VIF) were all below 4, which is smaller than the threshold level of 10 suggested by Glantz and Slinker (1990) indicating that multicollinearity was neglectible. Thus, the design still permits a solid base for the analysis of the response surfaces.

The canonical analyses indicated that no minimum or maximum is found for all of the dependent variables tested within the experimental range. Consequently, higher or lower values for the dependent variables are only limited by the physical boundaries imposed by the processing equipment or the dependent variable itself. For instance, power consumption of an expander cannot take negative values and 'degree of gelatinization' as dependent variable implies no values over 100 percent.

Conditioner treatment

The regression equation relating SGD_{AGS} to SP and WA was significant ($p<0.001$) as is shown by table 4. SP was the major contributor to changes in SGD_{AGS} ($p<0.001$) after conditioning, whereas water did not contribute to changes in SGD_{AGS} ($p>0.1$). The lack of fit for the model used is nearly significant ($p<0.1$) indicating that other factors, not considered in the

experimental design, should be considered as well. Table 4 lists the coefficients of the model as obtained from the fitting procedure.

Table 4: Model regression coefficients and analysis of variance of the SGD_{AGS} and DSC values after conditioning, expander processing and pelleting.

Factors[b]	After conditioning		After expander		After pelleting	
	SGD_{AGS} (%)	DSC ($J\ g^{-1}$)	SGD_{AGS} (%)	DSC ($J\ g^{-1}$)	SGD_{AGS} (%)	DSC ($J\ g^{-1}$)
Intercept	2.2854	3.0825	-53.1668	8.1212	-87.3974	11.6518*
SP	0.0952	0.0988	0.7185	0.0383	2.1581**	-0.1258**
WA	-0.6622	0.0727	-1.2540	0.1320	-0.6299	0.0112
MP	--	--	-2.4701	0.0458	-1.9171	0.1356
SS	--	--	0.4281	-0.0654	-0.0586	0.0212
ST^2	-0.0003	-0.0003	-0.0002	-0.0003	-0.0071**	0.0004*
WA^2	-0.0010	0.0015*	-0.0127*	0.0021**	0.0007	-0.0007*
MP^2	--[c]	--	0.7648	-0.0256	1.3609*	-0.0867#
SS^2	--	--	-0.0002	0.0002	0.0058*	-0.0005*
ST*WA	0.0057*	-0.0011	0.0048	-0.0006	0.0068	0.25E-6
ST*MP	--	--	-0.0095	0.0015	0.0656	-0.0063#
ST*SS	--	--	-0.0042	0.0002	-0.0041	0.0003
WA*MP	--	--	0.1273	-0.0178	0.0426	-0.0011
WA*SS	--	--	0.0087	-0.0010	-0.0044	0.0002
MP*SS	--	--	-0.0267	0.0023	-0.1524*	0.0121*
R^2	0.55	0.29	0.67	0.64	0.67	0.71
Regression[dc]	***	#	*	*	**	**
Lack of fit	#	**	#	ns	ns	ns
Root MSE	3.01	0.70	6.48	0.67	4.76	0.31
SP	***	#	**	*	**	**
WA	ns	ns	ns	*	ns	ns
MP	-	-	ns	ns	*	*
SS	-	-	ns	ns	ns	*

For abbreviations see table 3. *ns=p>0.1; #=p<0.1; *= p<0.05; **=p<0.01;***=p<0.001.

[b] regression coefficients: (p>t). [c] -- factors not incorporated in the regression equation. [d] regression: (p>F).

SGD_{AGS} of the starting material in this experiment was 10.3%. SGD_{AGS} values of the samples taken after the conditioner indicate that with an increasing amount of water added and an increase in steam pressure, SGD_{AGS} becomes higher. Highest value as estimated from the response surface equation (RS-equation) was 22.0 % (se ± 2.24) at a steam pressure of 173 kPa and 40.2 gr water added per kg feed mash. The lowest value estimated from the RS-equation (Table 4) was 5.1 % (se ± 2.24) at 103 kPa steam pressure and 35.6 gr added water per kg feed mash. The largest eigenvalue in magnitude is -3.47, and its eigenvector is associated with water. Smallest eigenvalue is 2.38 and its eigenvector is associated with steam. From figure 2a, a rising ridge can be observed in the direction of higher steam pressure and higher water content. Figure 2a shows that a decreasing SGD_{AGS} (below 10.3% of the untreated material) is favored by lower steam pressures, whereas adding more water affects both increasing and decreasing SGD_{AGS} values. At low steam pressures (<130 kPa) SGD_{AGS} of the treated material was lower than the SGD_{AGS} of the untreated feed mash. Approximate temperatures (at SP ~ 130 kPa) estimated from temperatures at the beginning of the expander indicate that feed mash temperatures were between 66°C and 72°C, which is approximately the gelatinization temperature of tapioca starch in abundant water (Cooke and Gidley 1992).

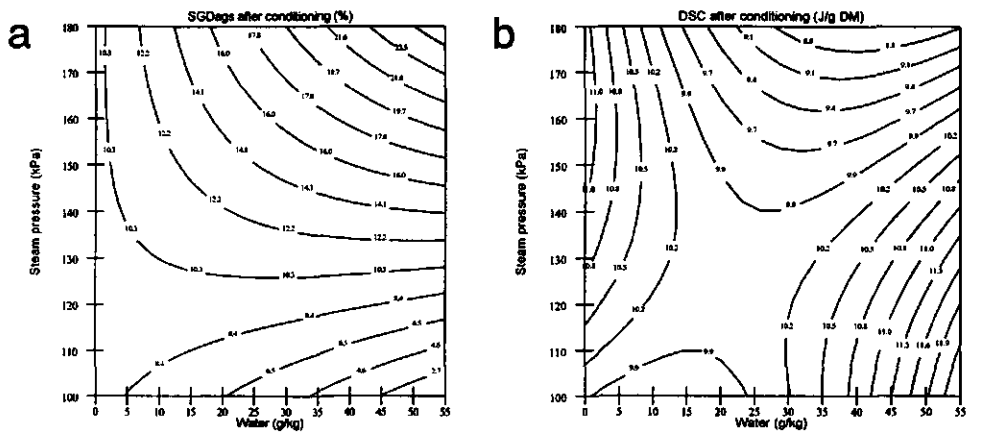

Figure 2: 2a) Contour graph of the Starch Gelatinization Degree (SGD_{AGS}) after conditioner treatment. 2b) Contour graph of the Differential Scanning Calorimetry (DSC) values of the starch fraction after conditioner treatment. SGD_{AGS} of the starting material is 10.3 %. DSC value of the starting material is 7.22 J g^{-1} DM.

DSC-values show that the response surface after conditioning is saddle-shaped (figure 2b). The stationary point is within the boundaries of experimentation. The principal orientation of

the surface is in the direction of water with an eigenvalue of 1.20, the eigenvalue for steam pressure is -0.67. With increasing water content the residual enthalpies become higher indicating that a higher proportion of the starch remains in native state after conditioner treatment. Higher steam pressures induce lower residual enthalpy, hence, indicating an higher degree of gelatinization. Lack of fit is, however, significant ($p<0.01$; Table 4) indicating that the response curve is not adequatly represented by a second order polynomial.

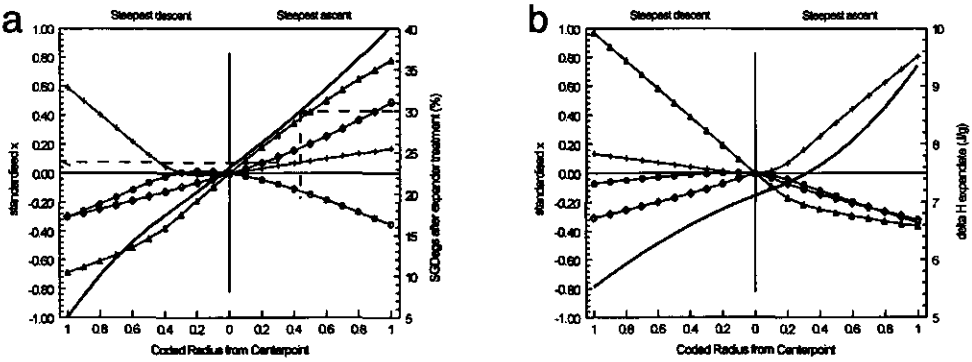

Figure 3: 3a) Ridge plot of SGD_{AGS} after expander treatment. 3b) Ridge plot of DSC-values after expander treatment. Legenda of factors: WA = +, SP = △, MP = ◇, SS = ○. The left Y-axis represent the scaled factors. The thick vertical line represents the dependent variable on the right Y-axis. Values of the independent variables at the centerpoint (coded radius 0) are: SP, 140 kPa; WA, 25.9 g kg^{-1}; MP, 3.51 kW and SS is 100 rpm. Dashed lines in the figure are explained in detail in table 3.

Expander treatment

Table 4 lists the coefficients of the model for degree of gelatinization and residual enthalpy as obtained from fitting the full response surface model. SP was the major contributor to changes in SGD_{AGS} ($p<0.01$) after expander processing, whereas WA, MP and SS did not contribute ($p>0.1$) to changes in SGD_{AGS}. The lack of fit for the model used is near significant ($p<0.1$) indicating that other factors could be considered.

Maximum SGD_{AGS}-values were higher after expander treatment than after conditioner treatment. Highest SGD_{AGS}-value estimated from the response curve or ridge plot (Fig 3a) is 40.2 % (± 6.3) at SP = 170 kPa, WA = 30.2 g kg^{-1}, MP = 4.6 kW (~11.3 kWh ton^{-1}; based on the average mass flow of 406 kg feed $hour^{-1}$ used in this study) and SS = 86 rpm. The lowest SGD_{AGS} value was comparable to that after conditioning; 5.0 % (± 4.6). Factor levels at the

lowest SGD_{AGS} were: SP = 110 kPa, WA = 41.1 g kg^{-1}, MP = 2.9 kW and SS = 88 rpm. From figure 3 it follows that, when moving away from the centerpoint of the design (SP = 140 kPa, WA= 25.9 g kg^{-1}, MP = 3.51 kW and SS = 100 rpm), higher values of SP, MP, WA and lower values of SS lead to an increase in SGD_{AGS}. Minimizing SGD_{AGS} is achieved by increasing WA and lowering SP, MP, and SS relative to the centerpoint of the design.

WA and SP were the factors predominantly affecting DSC-values ($p<0.05$; Table 4). Residual enthalpies from the DSC analysis showed the same tendency, although inversely, as the SGD_{AGS} values after expander treatment (Fig. 3b). Residual enthalpy was lowest, 5.54 (± 0.52) J g^{-1} dry matter at SP is 178 kPa, WA is 29.3 g kg^{-1}, MP is 3.9 kW and SS at 97 rpm. The highest residual enthalpy, 9.35 (± 0.47) J g^{-1} dry matter was found at the factor levels: SP is 126 kPa, WA is 46.9 g kg^{-1}, MP is 2.8 kW and SS is 86 rpm.

The highest measured temperature at the head of the expander was 101 °C (± 3.7) at the factor levels: SP, 165 kPa; WA, 44.0 g kg^{-1} ; MP, 4.1 kW and SS at 107 rpm. The lowest temperature was 43 °C (±3.9) at the factor levels: SP, 100 kPa; WA, 28.7 g kg^{-1}; MP, 3.5 kW and SS at 99 rpm (Figure 4a). Temperature was most affected by steam pressure ($p<0.001$). The stationary point of the RS-equation for temperature at the head of the expander was within the experimental region. Temperature at the stationary point was 99.7 °C with the factors set at: SP, 175 kPa; WA, 31.7 g kg^{-1}; MP, 5.1 kW and SS at 65 rpm. The canonical analysis showed that the largest eigenvalue was -18.3. Its eigenvector showed that the response surface was mostly aligned with SP. The second largest eigenvalue was 5.9, its associated eigenvector showed that it was mostly aligned with WA. The remaining two eigenvalues were -2.9 and 1.7 and were associated with MP and SS, respectively.

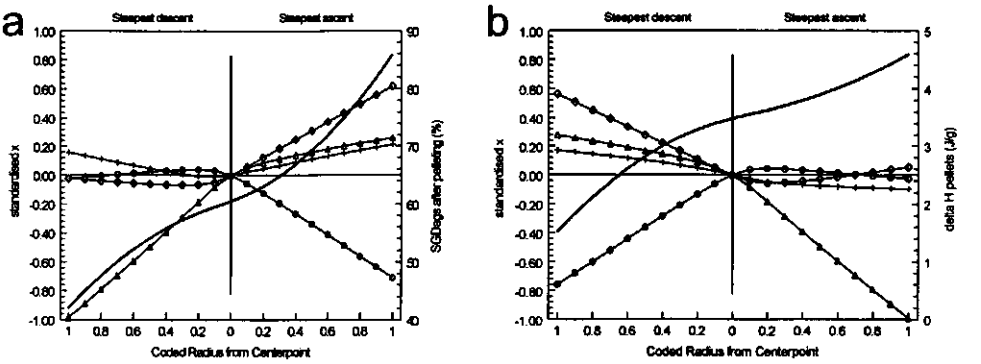

Figure 4: 4a) Ridge plot of SGD_{AGS} after pelleting. 4b) Ridge plot of the DSC-values after pelleting. Legenda of factors: WA = +, SP = △, MP = ◇, SS = ○. The left Y-axis represent the scaled factors. The thick vertical line represents the dependent variable on the right Y-axis. See also figure 3.

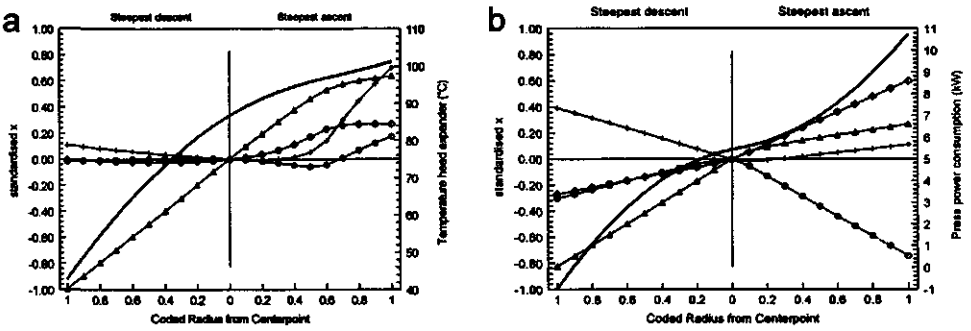

Figure 5: 5a) Ridge plot of temperature at the head of the expander. 5b) Ridge plot of the net power consumption of the pellet press. Legenda of factors: WA = +, SP = ▵, MP = ◇, SS = ○. The left Y-axis represent the scaled factors. The thick vertical line represents the dependent variable on the right Y-axis. See also figure 3.

Pelletizer treatment

The results from Pfost durability and the kahl hardness test have in one case been set to 'missing' since no data was present for that case.

The response surface of SGD_{AGS} after pelleting was adequately represented by the fitted equation; lack of fit was non-significant. Table 4 shows that SP and MP of the expander were the two factors affecting ($p<0.05$) SGD_{AGS} values after pelleting. The minimum SGD_{AGS} value from the RS-equation after pelleting was 42 % (±3.6), at the factor levels: SP = 100 kPa, WA = 29.9 g kg^{-1}, MP = 3.5 kW and SS = 99 rpm. Maximum value for SGD_{AGS} found from the RS-equation was 85.9 % (±6.6) at the factor levels: SP = 150 kPa, WA = 31.4 g kg^{-1}, MP = 4.9 kW and SS = 71 rpm. From figure 5a it follows that increasing MP and decreasing SS leads to higher values of SGD_{AGS}. Lowering SGD_{AGS} is affected most by decreasing SP.

Residual enthalpies (DSC) after pelleting also showed that the degree of starch gelatinization increased after pelleting compared to expander processing. Except for water addition, all the factors contributed significantly ($p<0.05$) in explaining the observed variation. Minimum residual enthalpy was 1.52 (±0.43) J g^{-1} DM at the factor levels SP = 151 kPa, WA = 30.2 g kg^{-1}, MP = 4.8 kW and SS = 70 rpm. Maximum residual enthalpy was 4.59 (±0.23) J g^{-1} DM at the factor levels SP = 100 kPa, WA = 23.4 g kg^{-1}, MP = 3.6 kW and SS = 99 rpm (Fig 5b). The canonical analysis showed that the stationary point was near to the experimental region. The factors SP, WA and SS were within the experimental region, their values being respectively 145 kPa, 19.7 g kg^{-1} and 83 rpm. The factor MP was slightly out of the experimental region with a value of 1.18 kW. The predicted value at the stationary point

was 3.61 J g^{-1} DM. Although observed eigenvalues were not large in magnitude (<1.5), eigenvectors and eigenvalues showed that the response was most sensitive to changes in screw speed of the expander.

Table 5 shows that the power consumption of the pelletizer was affected most by steam pressure ($p<0.001$) and water addition ($p<0.05$). Maximum power consumption of the pelletizer according to the RS-equation was 10.8 kW (±2.04) at the factor levels 151 kPa SP, 28.7 g kg^{-1} WA, 4.8 kW MP and 70 rpm SS. Minimum power consumption of the pellet press, as indicated by the RS-equation, was -0.99 kW (± 1.04) at the factor levels SP = 107 kPa, WA = 36.1 g kg^{-1}, MP = 2.9 kW and SS = 88 rpm. The stationary point was in the region of experimentation. The estimated power consumption at the stationary point was 5.53 kW at the factor levels SP = 142 kPa, WA = 24.8 g kg^{-1}, MP = 3.8 kW and SS = 108 rpm. The canonical analysis showed that the eigenvalue largest in magnitude (-4.9) was aligned with steam. The second largest eigenvalue (4.5) was most aligned with SS. From figure 4b it follows that the path of steepest ascent for maximum power consumption of the pelletizer is influenced by lowering SS and increasing MP. A lower power consumption of the pellet press is obtained by decreasing SP and increasing WA.

Temperature of the pellets showed a considerable amount of scatter as indicated by the low R^2 of 0.45 (Table 5). The fitted regression equation was not significant.

The values obtained from the Holmen pellet tester were significantly affected by SP, WA and MP ($p<0.01$). Lack of fit was not significant. Highest Holmen value was 117.4 % (±13.3) at the factor levels SP = 171 kPa, WA = 36.6 g kg^{-1}, MP = 4.4 kW and SS = 88 rpm. The lowest value was -8.8 (±11.3) at the factor levels SP = 145 kPa, WA = 14.7 g kg^{-1}, MP = 1.5 kW and SS = 100 rpm. Both calculated maximum and minimum values are higher and lower than the possible physical boundaries imposed by the test (0 - 100 %). In this case, the direction of the factor settings with respect to the centerpoint of the design, indicates on how improved Holmen values can be found under practical circumstances. Lower values of MP and water lead to lower Holmen values (Fig. 6a). Increasing Holmen values are mostly affected by increasing SP, MP and WA and lowering SS.

The regression for durability according to Pfost was significant (Table 6). Durability was most affected by SP ($p<0.05$) and MP of the expander ($p<0.01$). Lack of fit of the equation was not significant ($p = 0.89$). Maximum value for Pfost durability was 93.2 % (±4.7) and occured at the factor levels SP =167 kPa, WA = 44.9 g kg^{-1}, MP = 4.2 kW and SS = 110 rpm

Table 5: Model regression coefficients and analysis of variance of selected parameters during processing.

Factors	Temperature at the head of the expander (°C)	Net Power consumption of the pelletizer (kW)	Temperature of the pellets after pelleting (°C)
Intercept	-168.7582*	-55.0580*	-47.9158
SP	3.5831***	0.7241**	1.9683**
WA	-1.5566#	-0.8925**	-0.7883
MP	-5.1351	4.4824	2.1125
SS	-0.2358	0.2193	-0.0747
ST^2	-0.0110***	-0.0018*	-0.0062**
WA^2	0.0068	0.0011	-0.0018
MP^2	-0.2326	0.3236	0.5066
SS^2	0.0004	0.0014	0.0018
ST*WA	0.0060	0.0043**	0.0043
ST*MP	0.0330	-0.0147	-0.0048
ST*SS	-0.0012	-0.0025*	-0.0022
WA*MP	-0.0470	0.0324	-0.0412
WA*SS	0.0047	0.0010	0.0034
MP*SS	0.0490	-0.0522*	-0.0352
R^2	0.90	0.69	0.45
Regression	***	**	ns
Lack of fit	ns	#	ns
Root MSE	5.08	1.45	4.91
SP	***	***	*
WA	ns	*	ns
MP	ns	ns	ns
SS	ns	#	ns

For abbreviations see table 3.

For probability levels see table 4.

Table 6: Model regression coefficients and analysis of variance of the physical quality parameters of the pellets.

Factors	Pfost	Holmen	Kahl	Compression test
Intercept	17.53	309.2435	-8.2753	95.7189#
SP	0.9168	-3.6002#	0.4732	-1.0617*
WA	-1.6251	-5.9162*	-0.6743	-0.0660
MP	-1.7192	-15.8276	-7.7161	2.8382
SS	-0.6054	-0.2101	-0.0936	-0.3599
ST^2	-0.0022	0.0126#	-0.0019	0.0034*
WA^2	0.0095	0.0238	-0.0000	0.0091*
MP^2	-1.4402	-0.4036	0.5699	0.0838
SS^2	0.0027	0.0111	0.0017	0.0029
ST*WA	0.0060	0.0348*	0.0040	-0.0025
ST*MP	0.0948	0.3903*	0.0637#	0.0551
ST*SS	0.0034	-0.0128	-0.0013	-0.0004
WA*MP	-0.1359	-0.1173	0.0056	-0.0483
WA*SS	0.0098	0.0103	0.0015	0.0028
MP*SS	0.0692	-0.2121	-0.0364	-0.0789
R^2	0.81	0.80	0.63	0.61
Regression[c]	***	***	*	*
Lack of fit	ns	ns	ns	***
Root MSE	5.74	14.80	2.93	3.47
SP	***	***	**	ns
WA	#	**	ns	*
MP	**	**	*	#
SS	ns	ns	ns	ns

For abbreviations see table 3.

For probability levels see table 4.

(Fig 6b). The minimum value was 48.5% (±5.37) at the factor levels SP = 146 kPa, WA = 16.8 g kg^{-1}, MP = 1.6 kW and SS = 111 rpm. Increased Pfost durability figures are obtained with an increasing amount of added water (WA) and higher steam pressures (SP). Decreasing Pfost durability is mainly governed by a decrease in MP.

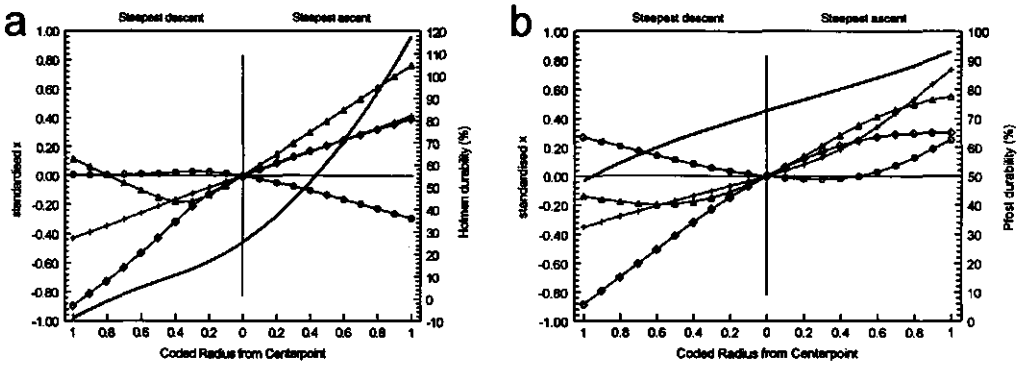

Figure 6: 6a) Ridge plot of Holmen durability values (%) of the pellets. 6b) Ridge plot of Pfost durability values (%) of the pellets. Legenda of factors: WA = + , SP = ▵, MP = ◇, SS = ○. The left Y-axis represent the scaled factors. The thick vertical line represents the dependent variable on the right Y-axis. See also figure 3.

Kahl hardness of the pellets was affected by SP and MP ($p<0.05$). Maximum value of Kahl hardness was 21.8 kgf (±3.7) at the factor settings SP = 162 kPa, WA = 31.0 g kg^{-1}, MP = 5.2 kW and SS = 82 rpm. Minimum value for Kahl hardness was 5.6 kgf (±2.69) at the factor settings SP = 165 kPa, WA = 9.3 g kg^{-1}, MP = 2.3 kW and SS = 107 rpm (Fig 7a). Increasing values for SP, WA and MP and lower SS induce harder pellets, whilst lowering SP from 140 to 100 kPa leads to lower hardness of the pellets.

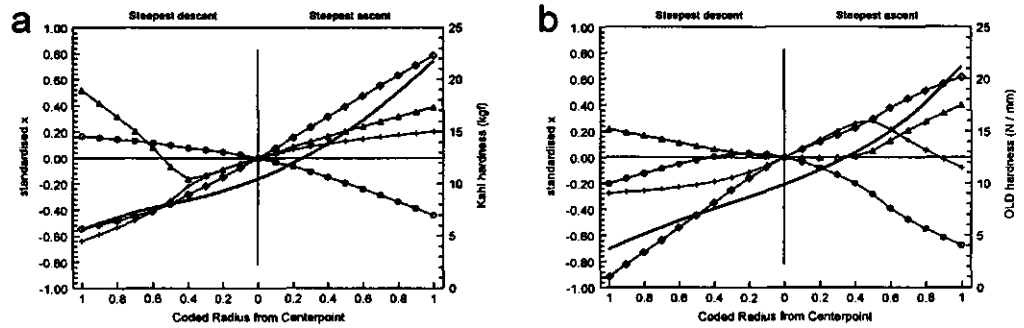

Figure 7: 7a) Ridge plot of Kahl hardness (kgf). 7b) Ridge plot of the values derived from the compression test (N mm^{-1}). Legenda of factors: WA = + , SP = ▵, MP = ◇, SS = ○. The left Y-axis represent the scaled factors. The thick vertical line represents the dependent variable on the right Y-axis. See also figure 3.

Values for crushing strength of the pellets showed that lack of fit was highly significant ($p<0.001$). Values for crushing strength were most affected by WA ($p<0.05$) and MP ($p<0.1$). The stationary point was found within the region of experimentation. Predicted value at the stationary point was 9.34 N mm^{-1} at the factor levels SP = 131 kPa, WA = 14.1 g kg^{-1}, MP = 4.6 kW and SS = 128 rpm. However, since lack of fit is highly significant, it indicates that other unknown factors, most probably particle size and its distribution, strongly influence crushing strength of the pellets. Figure 7b indicates that low values for crushing strength are induced by decreasing values for MP. Low screw speeds of the expander and increasing MP and SP favor higher values for crushing strength.

SGD_{AGS} levels after the different processing steps were significantly ($p<0.0001$) different, as indicated by the mean values at the centerpoints. Mean value at the centerpoint after the conditioner was 12.6 % (std. err. ± 0.96), after the expander was 23.1 % (std. err. ± 1.11) and after the pellet press 60.6 % (std. err. ± 1.11). Both, the contrast between conditioner and expander and between expander and pellet press were significant ($p<0.0001$).

DSC values after the different processing steps were significantly ($p < 0.0001$) different. Mean value at the centerpoint after the conditioner was 9.8 J g^{-1} dry matter (std. err. ± 0.14), after the expander it was 7.14 J g^{-1} dry matter (std. err. ± 0.16) and after the pellet press 3.49 J g^{-1} dry matter (std. err. ± 0.16). Both, the contrast between conditioner and expander and between expander and pellet press were significant ($p<0.0001$).

Discussion

In this study, tapioca meal has been used to investigate the differences in the degree of gelatinization and residual enthalpy after processing, with respect to the physical quality of derived pellets and some system parameters. The choice to use tapioca meal instead of some other starch rich feed material was based on the fact that tapioca contains relatively low amounts of protein. Therefore, to ensure that, at least, interactions with protein are absent, tapioca has been used as a model feed.

Conditioner treatment

To gelatinize starch, heat and water are a necessary prerequisite (Lund 1984; Camire *et al.* 1990). For steam pressures higher than 130 kPa, with associated temperatures of the feed mash of 70 °C or higher, this is in line with our results. Results from literature indicate that the gelatinization temperature of tapioca starch in abundant (>75%) water is approximately

69.9-71.5 °C (Cooke and Gidley 1992). Although, the amount of added water due to WA and SP are by far lower as the above mentioned figure, it can not be excluded that at a local scale (inbetween or around particles) the amount of water is high enough to induce gelatinization at approximately 70° C. As can be seen from figure 2a, degree of gelatiniziation may also become lower after processing relative to the untreated material. A possible explanation can be that during the air-drying process or subseqent storage reordering of starch has taken place. At temperatures close to (but not above) the gelatinisation temperatures, reordering may take place which involves part of the amorphous starch fraction. This process is governed by the duration at which this temperature is present and the moisture contents of the sample (Keetels 1995; Eliasson and Gudmundsson 1996). Both SGD_{AGS} and DSC values after conditioning show a saddle-type response surface. It should be noted however that DSC measurements on the conditioner samples were conducted at a much later stage in the project and if reordering is important during storage, these DSC-figures and SGD_{AGS} values cannot be compared for the conditioning process. Whether or not reordering has taken place during storage or during the cooling and air-drying period cannot be concluded from this experiment. Lack of fit and the low R^2 for the conditioning samples is significant (Table 4), which might be indicative for reordering taking place and affecting DSC and SGD_{AGS} values.

Expander treatment

Steam is the most predominant factor contributing to changes in starch properties. Increasing steam pressure leads to an increase in SGD_{AGS} values and decreasing DSC-values. Lowering screwspeed leads to an increase in residence time and a lower shear rate (Bhattacharya and Hanna 1987; Chiang and Johnson 1976; van Zuilichem 1992). Prolonged residence times and mixing will lead to better absorption of moisture and an increase in starch particle size due to swelling. This in turn will facilitate disruption of the particles by shearing action against screw and barrel wall. At high screw speed, residence times will be shorter with reduced swelling of the starch particles. These two effects tend to be counteractive (Bhattacharya and Hanna 1987). In this study, although the effect of SS was not significant (Table 4), the path of steepest ascent showed that lowering screw speed tended to lead to a higher degree of gelatinization when jointly SP, MP and WA were increased. DSC-measurements follow the same (inverse) pattern as SGD_{AGS}. Our results are in line with results from Chiang and Johnson (1977) who found that lowering screw speed in their extrusion experiment lead to a higher degree of gelatinization. This was attributed to the prolonged residence time of the material caused by the lower screw speed.

MP in this study did not significantly contribute to changes in degree of gelatinization of the tapioca starch. Specific mechanical energy (SME) calculated as the net amount of expander motor power was between 11.5 and 50.5 kJ kg^{-1}. The maximum degree of gelatinization, as estimated from this study was 40.2%. By comparison, to obtain fully gelatinized starchy materials using single screw extruders, values of 350 and 700 kJ kg^{-1} (van Zuilichem and van der Poel 1986) and between 300 and 900 kJ kg^{-1} with use of twin-screw extruders (Della Valle *et al.* 1989) were reported.

Linko *et al.* (1983) reported high degrees of gelatinization during extrusion cooking at moisture contents of about 20 %. Typically, temperatures used in the described experiment were well above 120 °C, all leading to complete loss of organised crystalline structure. In the present study the moisture content, originating from added water, steam and initial feed moisture, at the entrance of the expander was between 13 - 21.5%. Degree of gelatinization (SGD_{AGS}), however, does not exceed 50%, likewise DSC measurements show that minimum residual enthalpy is 5.54 J g^{-1} dry matter. These figures indicate that the starch is only partially gelatinized. In this study, temperatures at the head of the expander did not exceed 101°C which is lower than the observed values of Linko *et al.* (1983).

In the present study, neither the amount of specific mechanical energy (350 - 700 kJ kg^{-1}; van Zuilichem and van der Poel, 1996) nor the necessary temperatures as mentioned by Linko *et al.* (1983) have been reached. Energy dissipation of the expander motor has an neglectible effect on SGD_{AGS} as is shown from the non-significant contribution. Steam incorporates heat and water in the feed mash. Since steam pressure is the most important factor in increasing temperature it can be stated that gelatinization during expander treatment is predominantly water (from steam) and heat driven. Addition of free water does not seem to have such a large impact on starch properties as would be considered from the above conditions. The beneficial effect of steam over water as a means to improve physical pellet quality has long been recognized in the manufacturing of pelleted animal feed (Friedrich and Robohm, 1968; 1969). They attributed this effect to the better dispersion of steam through the feed particles, due to the higher viscosity of water at room temperature as opposed to the viscosity of steam. Furthermore, for starchy materials the ratio between water and heat diffusion indicates that heat penetrates the feed material much faster than water. Thermal diffusivity is approximately 10^{-7} m^2 s^{-1}, whilst water diffusivity is approximately 10^{-9} m^2 s^{-1} (Oechsner de Coninck and Bouvier 1995). For the above mentioned reasons it seems plausible to assume that inclusion of steam or water will lead to relative high water concentrations at the exterior of the feed particles. This in turn will lead to a higher degree of starch gelatinization of the exterior of the particle relative to the interior, if temperature is high enough. In order to obtain a relative high degree of gelatinization using expander equipment, the direction of research should be to

improve the mixing behaviour (better dispersion of added water through the feed mix) of the expander. Moreover, since MP does not significantly affect degree of starch gelatinization or DSC-values after expander processing, MP can be set at a low value to decrease energy costs, when only a high degree of starch gelatinization after expander processing is wanted.

Pelletizer treatment

The degree of starch gelatinization increased after pelleting. Furthermore, in this study it was observed that the pellets which had the highest degree of gelatinization after pelleting started to develop exit-die defects (sharkskin). Deformability of starch granules, the main component in tapioca, is dependent on the degree of gelatinization (Eliasson and Bohlin 1982; Eliasson and Gudmundsson 1996). Surface defects of the pellets may have been introduced by the relative increase in storage (elastic) modulus over loss (plastic) modulus at the highest degrees of gelatinization which leads to elongational stresses which in turn may induce sharkskin-defects (Venet and Vergnes 1996). As a consequence, physical quality determinations for pellets with a high degree of starch gelatinization will lead to different values than values derived from symmetrical pellets.

MP does not significantly affect SGD_{AGS} and DSC values after expander treatment, but does affect SGD_{AGS} and DSC values after pelleting. Therefore it can be assumed that an intermediate parameter is involved. A possible explanation could be the densifying effect, due to the screw action of the expander, on the feed mash. The higher density should then result, through shear at the exterior of the pellet, in an increased amount of gelatinized starch. This is supported by the data from the present study. The same factor levels that maximize power consumption of the pellet press (SP = 150 kPa, WA = 31.4 g kg^{-1}, MP = 4.9 kW and SS = 71 rpm) also maximize SGD-values and minimize DSC-values, both indicative for a high amount of gelatinized starch. If specific mechanical energy (SME; kJ kg^{-1}) is a main contributor to the degree of starch gelatinization during pelleting, then an SME of between 350 and 700 kJ kg^{-1} seems a prerequisite for complete gelatinization (van Zuilichem and van der Poel 1986). In this study the maximum power consumption of the press is approximately 95 kJ kg^{-1}. As can be seen from figure 3a and 4a, degree of starch gelatinization increases with respect to expander processing. Since it may be assumed that mixing within the die hole does not occur, the amount of feed mash that receives this shear energy will be at the exterior of the pellet. Hence, the outside of the pellet will have a higher degree of gelatinization than the pellet interior. Evidence for this in the case of corn meal is given by Stevens (1987). Furthermore, preliminary investigations of some pellets using a polarization microscope revealed that

indeed differences existed between interior and exterior of the pellet with respect to loss of birefringence.

All tests for physical quality showed that SP and MP at least, affected hardness and durability. Steam incorporates heat and this is a known aid in obtaining good quality pellets. Heat induces thermal softening of the feed (Rao and Lund 1986) which make particles more pliable. In the presence of a slight amount of water, soluble components like sugars can solubilize and upon recrystallisation form bonds between particles (Rumpf 1958; Pietsch 1990). In high-starch rations, the glass transition may also play a role in acting as a 'solid' continuous phase in which the feed particles become attached. However, the extent and possibility of such a binding mechanism in compound feed has never been investigated. The action of MP is most probably a densification of the feed mash. Thomas *et al.* (1998) using data from MacMahon and Payne (1994) indicated that apparent density of feedstuffs affects physical quality. Denser feeds lead to better pellet quality, most probably because the press does not have to spend energy on deairating the feed mash.

From table 6 it becomes apparent that the widely used tests, Pfost, Holmen and Kahl yield a better fit, than the more controlled compression test. The relative contribution of tensile, compression and shear stresses towards breaking of the pellet in bulk is generally not known and depends on speed of travel, applied load and direction of load.

Fell and Newton (1970) conducted diametral compression tests of lactose tablets on an Instron compression and tension instrument and found that when padding material was used to obtain an ideal loading position, tensile failure would occur in the tablets. Tabil (1996) used three layers of blotting paper to obtain an ideal loading of alfalfa pellets and was able to fracture pellets in tension. Values thus obtained have a lower variation than when more modes of fracture are present. Due to sharking of the pellets in the present study it was not possible to obtain even loading on the pellets. Therefore crushing strength of the pellets is determined as the force at which failure of the pellets occurs.

The effect of inhomogeneities, for instance large particles, may become apparant when using the Kahl pellet tester. In this tester, a load is locally applied to the pellet and may be more sensitive to the presence of large particles in the pellet at the place where the force is applied. It is assumed that the large particles in this case act as inhomogeneities and breaking is more likely to occur at these places. Since in the compression test the applied load is extended over the whole length of the pellet, the effect of inhomogeneities due to large particles may be even more pronounced and thus leading to figures, less accurate predicting the effects of the experimental factors. This may be an explanation for the highly significant lack of fit found for the physical quality in the compression test. The Holmen pellet tester

will, due to the higher applied speeds of travel, evaluate the impact behavior of the pellets due to impingment against the wall and bends of the instrument. The Pfost pellet tester mainly tests the friability of the pellets when sheared. The surface defects (exit-die) present in the pellets provide for inhomogeneities which in turn affects all these measurements. However, these exit-die defects occured at relatively high levels of gelatinization, which also inherently reflects a change in the properties of the pelleted material. Therefore, the interaction between these two effects on physical pellet quality can not be elucidated from this experiment and should be further investigated.

Correlations between SDG_{AGS} and DSC were -0.15 (ns), -0.72 ($p<0.0001$) and -0.86 ($p<0.0001$) after conditioning, expander treatment and pelleting respectively. Overall correlation between SGD_{AGS} and DSC was -0.94 ($p<0.0001$). Figure 8 shows a scatter plot of DSC values against SGD_{AGS}. It should however be emphasized that DSC measurements on the samples derived after conditioning were done in a later stage of the project. Therefore, reordering of the starch during storage may have lead to discrepancies between SDG_{AGS} and DSC after conditioning. Samples taken after conditioning may show a larger amount of scatter than subsequent values found after expander treatment (Thomas *et al.*, 1997). Taken into consideration the values for water and heat diffusivity as given by Oechsner de Coninck and

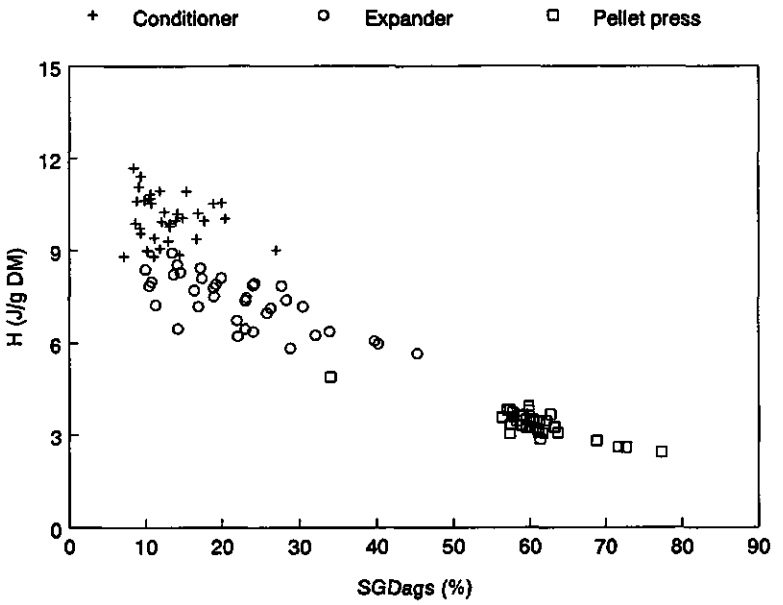

Figure 8: Scatterplot of the SGDAGS and DSC values after the different processing steps. + = after conditioner treatment, o = after expander treatment and □ = pelleting.

Bouvier (1995) it can also be argued that residence time in the conditioner is to short (approx. 30 to 60 s) to reach homogeneous mixing throughout the feed mash. The longer total Bouvier (1995) it can also be argued that residence time in the conditioner is to short (approx. 30 to 60 s) to reach homogeneous mixing throughout the feed mash. The longer total processing residence time as induced by incorporating an extra process (expander-treatment) may provide for the additional amount of time (approx. 10 to 15 s) plus thorough mixing, which may disperse more evenly the amount of water present. The fact that only steam and water are determining factors in explaining the amount of gelatinized starch after expander treatment provides further evidence of such an existing mechanism. This study, however, cannot provide enequivocal evidence to accept or abandon this mechanism since scatter after conditioning may well have originated from the difference in storage time of the samples, with which reordering of starch may have interfered. In addition, the amount of shear the feed mash receives from the three operations (conditioning, expander-treatment and pelleting) may also affect reordering characteristics of the starch fraction as well.

From this study it is concluded that partial gelatinization of starch is predominantly induced by the moisture and heat present during conditioning and expander processing. The effect of shear is not clear since reduction of SS may reduce shear but also increases residence time within the equipment. Reduction of shear will leave more of the granules intact. In contrast, prolonged residence times may lead to a better absorption of water in the particles which in turn facilitates swelling and susceptibility to shear. The two effects may therefore be counteractive and not contribute to the regression equation for SGD_{AGS} and DSC (Table 4).

MP does not affect gelatinization behaviour over the range investigated. The full specific mechanical energy calculated from MP and feed flux indicates that it is to low to induce considerable gelatinization of the starch fraction. Moreover, temperatures which are reported in literature at which complete gelatinization occurs at the low water contents involved, are not reached in this study. Studies which examined the effect of SME on starch degree of gelatinization should preferably extend the applied range of SME to approximately 1000 kJ kg^{-1}.

If degree of gelatinization is an important factor with respect to nutritional parameters or affects systems parameters further downstream the expander, the most fruitful directions for optimization as presented in this study are changes in steam pressure (SP) and water addition (WA). Motor power of the expander (MP) and to a lesser extent screw speed (SS), mainly determine the physical pellet quality characteristics and energy consumption of the pellet press as is evidenced by table 6.

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Tapioca starch processing and its effect on pellet durability and hardness.

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Abstract

In a dose-response design the effects of an increase in the amount of gelatinized starch in a model feed mash was investigated on hardness (Kahl, compression test and Kramer shear press) and durability (Holmen and Pfost) characteristics of the pelleted feed. Model feeds were either steam pelleted (SP) or cold pelleted (CP). It was hypothesized that an increase in the amount of gelatinized starch present, would lead to harder and more durable pellets. Results showed that, due to a pre-processing step, the effects of particle size of the feed mash (109 μm to 315 μm) and water content (12.8 % to 16.9 %) were confounded with the degree of gelatinization (ranging from 11.5% to 53.1%) of the starch fraction, as measured with the amyloglucosidase method (SGDags). Broken line regression models were used to summarize the data. A decrease in hardness and durability of the pellets was found with increasing SGDags, up to an intersection point, after which a rapid incline in hardness and durability was observed with further increasing SGDags values. The appearance of exit-die defects, past these intersection point(s), associated with tough and durable pellets, led to the conclusion that part of the starch fraction underwent a glass transition from a brittle to a rubbery state. This may have accounted for the different rheological properties, which in turn induced exit-die defects. It is concluded that, in order for these exit-die defects to appear, two requirements should be met. First, the temperature at which the glass transition takes place must be below the processing temperature. Secondly, the amount of amorphous starch, which can exhibit this glass transition, must be large enough to become the dominant phase that determines the rheological behavior of the feed mash. However, since the effects of particle size and moisture content are confounded with SGDags it is not possible to unequivocally determine the absolute magnitude of the different factors with respect to the measured characteristics. No differences were found between CP and SP before the intersection point on most hardness tests. No difference was found in energy consumption, mass debit and the appearance of exit-die defects between CP and SP. After the intersection point, SP led to higher pellet hardness and pellet durability when compared to CP.

It is concluded that in experiments evaluating the effect of gelatinized starch on hardness and durability, particle size and moisture content should remain equal for all batches.

Introduction

Due to differences in geography, climate and processing history, the variability within feed ingredients can be large (Wilson, 1994). The inclusion of a large number of feed ingredients in a wide variety of animal feeds can make it difficult to maintain technical quality standards for pellet hardness and durability (Thomas and Van der Poel, 1996). For a feed manufacturer, only few means are available to influence or alter this variability in the feedstuffs, although these changes may affect the outcome of the pelleting process. In order to maintain technical

quality standards, changes in the processing variables during manufacture are used. Various authors in literature have described the effects of process and equipment variables on pellet hardness and durability (Maier and Gardecki, 1992, Friedrich and Robohm, 1969, 1970; Thomas *et al.*, 1997). Variables that have been studied included; the addition of steam, water or other liquids, the lay-out of the equipment as well as the order of processing, for instance the use of compaction equipment (expander or double pelleting) prior to the actual shaping process (Robohm, 1991; Peisker, 1992). For the pellet press operator, the only means of adjustment is through changes in the processing variables, often with inconsistent results with respect to the pellet quality (Winowiski, 1988). With respect to the physico-chemical characteristics of the raw materials and its effect on the pelleting process (e.g. degree of gelatinization or the amount of denatured protein), and for instance the resultant pellet quality, only one study seems to have been conducted.

Wood (1987) showed that a gradual replacement of native starch by pre-gelatinized starch led to an increase in pellet hardness and durability. Furthermore, it was shown that the state in which the protein was present in the feed, was a further causative factor. Native (soy) proteins lead to harder and more durable pellets, whilst denatured proteins lead to softer and less durable pellets. However, the effects of protein and starch have never been studied seperately.

Due to the processing steps in the manufacture of pelleted feeds, changes may occur in the physico-chemical properties of the feedstuffs, sometimes with contradictory results. For example, Heffner and Pfost (1973) and Skoch *et al.* (1983a), studied the effects of conditioning and pelleting on the degree of gelatinization of starches. In the study of Heffner and Pfost (1973), an increase in the degree of gelatinization after conditioning was found, whilst Skoch *et al.* (1983b) did not find any differences in degree of gelatinization after conditioner treatment with respect to the starting material. Pelleting the feed mash raised the degree of gelatinization to approximately 25% in both studies. Total moisture content in both studies was between 10 and 15 % during the different processing stages. Both studies were conducted with the same installation. More studies exist, but these two examplify that during feed manufacturing changes in the feed may occur. Even when manufacturing feeds at the same installation, the obtained results may differ (although processing conditions in the above study also slightly differed). The state of the feed constituents (for instance; protein or starch) is, however, one of the causative factors in the ultimate hardness and durability of pelleted feed, as has been shown by Wood (1987).

Although rules of thumb exist for the effects of changes in process variables and its effect on changes in the feed mash, the predictive capability of such rules on, for instance, the hardness and durability of the pellets, is limited. Part of this may also be attributable to the

lack of knowledge on how pelleted feed fractures, and how this fracture behaviour is related to the process variables and (changes in) physico-chemical properties of the feedstuffs. Rumpf (1958) studied the binding mechanisms that are primarily responsible for maintaining structural integrity of the pellet. To date, only in simple model systems it is possible to predict some of the physical characteristics of an agglomerate (Pietsch, 1991). One of the key determinants responsible in determining the strength of agglomerates (and therefore pellets) is porosity. Hence, in this study an attempt was made to determine the pellet porosity.

The focus of this study is on the effect of the functional properties of the feed ingredients, rather than to study the effects of changes in processing conditions on the physical quality of pelleted feed. The aim of the present study is to determine the effect of a gradual replacement of tapioca meal, used as a model feed, containing native starch with tapioca meal containing (pre-)gelatinized starch, on pellet hardness and durability. It is hypothesized that an increase in the amount of gelatinized starch will lead to harder and more durable pellets. In addition, the effect of inclusion of (pre-)gelatinized starch and pellet porosity will be studied.

Materials and Methods

Tapioca meal is used as a model feed. The choice for tapioca meal originates from the high starch content of the material.

Pre-processing step.

A commercial batch of ground tapioca meal was obtained from the CHV, Veghel, The Netherlands. This batch was divided in two portions. One portion remained unprocessed, the other was processed using an expander (AL-150 Contivar, Almex b.v., Zutphen, The Netherlands) to partly gelatinize the starch in the tapioca. Processing conditions during this pre-gelatinization phase were a steam pressure of 180 kPa, water addition of 20.5 ltr $hour^{-1}$ with a feed flux of 398 kg $hour^{-1}$ and a net power consumption during expander processing of 42 kJ kg^{-1} feed. The expander processed tapioca was subsequently cooled in a Robinson counter-flow bunker cooler with two decks with a total cooling time of 8 min 30 sec. The coarse expandate was then ground in a Wijnveen hammermill with a screen size of 2.25 mm. The expander processed and the ground material was blend into five batches with different proportions of pre-gelatinized starch, using a ribbon mixer. These batches are referred to as B0, B25, B50, B75 and B100, the code representing the percentage of processed tapioca present in the batch. Batches where dispatched to the Wageningen Feed Processing Centre

(WFPC) in paper bags, each containing approximately 25 kg of feed. Figure 1 gives an overview of the used processing scheme.

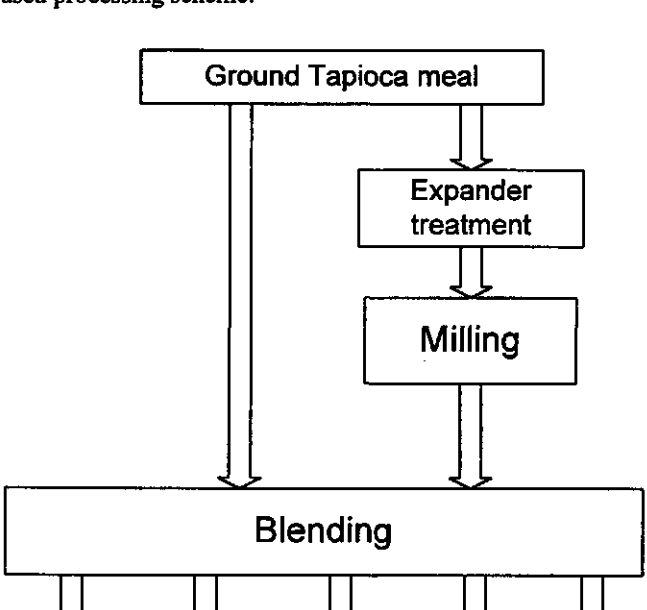


Figure 1: Processing scheme used to obtain the batches differing in degree of gelatinization. Subscript denotes the percentage of expander treated tapioca in the batches.

Pelleting trial

Batches were pelleted with (steam pelleting: SP) and without steam (cold pelleting: CP) at the WFPC. A conventional barrel type conditioner was used to add steam. Steam pressure was adjusted to obtain a feed mash temperature entering the pellet press of approximately 70 °C. Subsequently, steam was turned off and the remainder of the batch was pelleted without steam. Soy-oil (1.5% of the feed debit) was added to the feed material during the conditioning phase to improve flow behavior of the tapioca meal. The pellet press was a modified Simon Heessen V3-30 fitted with a 22 kW engine. The pellet press was equipped with two rollers. In all runs, a warm die of 13*80 mm (bore*die-thickness) was used. During pelleting the mass debit was adjusted to obtain constant load on the pellet press (17 kW). From the mass debit and the amount of power consumed by the pellet press (corrected for idle running), the amount of specific mechanical energy (SME) was calculated in kJ kg^{-1}. Samples were taken in

quadruplicate and temperatures were measured after the process had reached steady state. During processing, data were recorded using a μ-mac 6000 datalogger with PC-read out. This was determined from the logged data and graphs plotted on a PC-screen during processing. Temperatures were recorded at the end of the expander using a thermocouple. This was regarded as the entrance temperature of the feed mash at the pelleting stage. Pellet temperature of the pellets leaving the die, was determined using the thermosflask method.

Sampling

Samples were taken of each bag from a batch during charging of the bin and pooled within a run. In addition, sampling was carried out before the feed material entered the pellet press (sample spot 1: figure 2), after leaving the pellet press (sample spot 2: figure 2) and after cooling (sample spot 3: figure 2), these samples were at least taken in duplo. All samples were collected in plastic flasks of 1 liter. Samples were air-dried at 50°C for 16 hours. Subsequently, dry matter was determined in air-dry matter samples by oven drying for four hours at 103°C. Moisture contents was calculated using both air-dry matter and dry matter determinations. Samples (~25 kg) used for evaluating the physical pellet quality were collected after cooling and stored in plastic bags at 4°C. The samples used for evaluation of the physical pellet quality were not subjected to drying treatments.

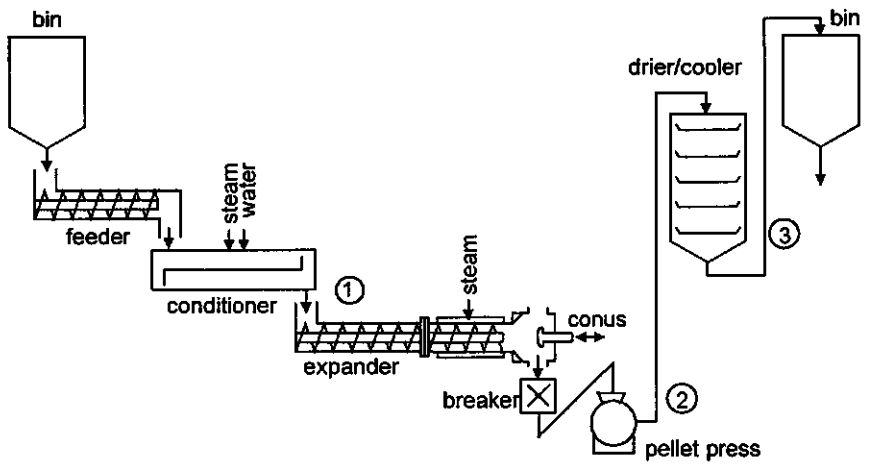


Figure 2: Schematical drawing of the pelleting line used. Encircled numbers indicate spots were samples were taken for analysis.

Physical and chemical analysis

A dry sieve analysis was used to determine the particle size and particle size distribution of the five different batches. A Fritsch Analysette 3 was used which was equipped with six sieves (2500, 1250, 630, 315, 160 and 71 μm) and a pan (<71 μm). Particle size distribution of the tapioca was calculated according to Waldo *et al.* (1971). Reported values are means of duplo sieve analysis.

To evaluate the effects of the conditioning and pelleting treatments, the degree of starch gelatinization was measured using the enzymatic amyloglucosidase test (SGDags) or using differential scanning calorimetry (DSC). SGDags was determined in three steps according to the NIKO method (Brunt, 1992). Total starch was analysed by extracting the lower sugars with a 40% ethanol solution, followed by autoclaving for 3 h at 130°C and enzymatic breakdown (1 h at 60°C, pH 5) to glucose, using an enzyme cocktail containing amyloglucosidase, α-amylase and pullulanase (A). Glucose is subsequently determined using hexokinase and G6P-dehydrogenase. For the determination of the degree of gelatinization of starch, two additional analysis were conducted: starch is analysed as above, but without the ethanol extraction (B) to quantify the amount of starch and lower sugars. Finally, the sample was hydrolysed with amyloglucosidase (60 U g^{-1} sample) for 75 min at 50°C (pH 4.8) to determine gelatinized starch and lower sugars (C). The SGDags was calculated as a percentage of total starch after correcting for lower sugars according to: SGDags = 100 * [C-(B-A))]/A.

A Mettler-Toledo DSC12-E was used to determine the residual enthalpy in the samples. Between 15 and 20 mg of sample was weighed into a 120 μl medium pressure crucible (Mettler-Toledo). Approximately 60 mg of demineralised water was added. The crucibles were sealed and left to equilibrate for one hour at room temperature to allow water to fully hydrate the sample. The sample was then subjected to a controlled temperature program in which the sample was held isothermal for 5 minutes at 20°C and then subjected to a linear temperature increase at a rate of 5°K min^{-1}. The measured range was from 20°C to 120°C. A crucible filled with 90 mg of aluminium foil was used as a reference. The residual enthalpy as measured with the DSC is a measure for the amount of ungelatinized starch present in the sample. In addition, some samples (see later) where scanned as-is (without the water), to determine the presence of a glass transition. Moisture content in these latter samples was determined after DSC analysis by punching a hole in the lid of the crucible and drying for 4 hours at 103°C, after correction for crucible weight.

Hardness, durability and porosity determinations

Pellet porosity was calculated from the difference between density determinations with and without the void fraction present in the pellet. Density of the pellets without the void fraction, was determined using a Beckman air-comparison pycnometer (Model 930). The air-comparison pycnometer uses two gas-tight cylinders; a reference cylinder and a measuring cylinder which are connected by a differential pressure indicator and a valve. After insertion of the pellet sample in the measuring cylinder, the change in pressure due to the displaced volume in the cylinders is balanced by adjusting a piston on the reference cylinder. The displaced volume is read from a calibrated scale. Approximately 15 gr pellets were inserted in the measuring cylinder and the volume determined. From the weight and displaced volume the density of the pellets (without the void fraction) is calculated as: pellet weight / pellet volume (kg m^{-3}). All measurements were done in quadruplicate.

Determinations of the pellet volume (including void fraction) was done by submersion in peanut-oil. Approximately 50 gr of pellets were weighed and inserted in a wire-frame cage. This wire-frame cage was mounted to an electronic Mettler Toledo scale. The scale and mounted wire frame cage were placed above an open container containing ~15 ltr of peanut oil. The empty wire frame cage is lowered in the oil and the scale is tared. After insertion of the pellet sample in the cage, the weight of the submersed sample is recorded every second for 200 sec using a PC and commercial spreadsheet package (Lotus 123) and the data read-out option of the scale. The obtained data was graphically analyzed and the weight of the pellets at the time of submersion (t_0) was determined. Apparent density of the pellets (kg m^{-3}) is calculated with Archimeds law using the density of the peanut oil, the initial weight in air and weight of the pellets at t_0. Density of the peanut-oil was determined with an aerometer. The porosity of the pellets was then calculated as: (true density pellets - apparent density pellets) / true density pellets.

Physical quality determinations of pellets comprised the following tests for hardness: Kahl, a general compression and tension apparatus and the Kramer shear press (Anonymous, 1970). Durability tests were conducted using the Pfost and Holmen test apparatus. Kahl hardness was tested by inserting a pellet in the device and the force necessary to crush the pellet was recorded. Reported values are means of 10 measurements. Hardness was tested in a general compression and tension test (Overload Dynamics series 100, fitted with a remote control series 900 and a 2000 N load cell) by crushing the pellet between two flat platens. The speed of the moving plate was fixed at 10 mm min^{-1}. A pair of vernier calipers is used to determine the length of each pellet. Reported figures are an average of 10 measurements. Results are reported as maximum force necessary to crush the pellets per mm pellet length. Tests made

with the Kramer shear press were done as follows: approximately 10 gram of pellets (4 pellets) were inserted in the shear box, half of the pellets in axial direction and the other half in radial direction to the grid of the shear box. The shear press was modified to obtain shearing forces in Newtons instead of pounds. The force neccesary to shear the pellets was recorded and maximum force necessary was calculated as N g^{-1} pellet material used. Durability tests were done according to the description given in Thomas *et al.* (1996). In the Holmen test, the circulation time was 1 minute. A 5 mm sieve was used. Reported figures are means of 3 measurements. In the Pfost durability test a 3 mm sieve was used. Reported figureas are means of duplo measurements. Details are described in Thomas *et al.* (1996).

It was observed that some of the pellets showed development of a fir-tree like appearance. The amount of pellets affected by these 'exit-die defects' or 'sharkskin' was determined by taking 100 pellets at random and determine visually whether or not these defects were present. Results are expressed as the percentage pellets which showed exit-die defects.

Statistical analysis

The results of the measurements of the pellet hardness and durability, porosity, SME and the results of the determination of the amount of pellets which showed exit-die defects have been fitted to the following starting model (1):

$$Y = a + b_1 \cdot x - (b_1 - b_2) \cdot r \cdot \ln[1 + \exp^{(\frac{x-c}{r})}] \qquad \text{Model 1}$$

This model describes the transition from one linear line segment to another linear line segment. a is the intercept with the y-axis at x=0, b1 is slope of the first line segment and b2 is the slope of the second line segment. c is the point where the two lines intersect. x= the degree of gelatinization (SGDags, %) of the batches. The equations of the two lines takes the following form:

for $x<c$: $y= a + b_1 \cdot x$

$x>c$: $y= (a+b_1 \cdot c - b_2 \cdot c) + b_2 \cdot x$

$x=c$: $y= a+b_1 \cdot c-(b_1-b_2) \cdot r \cdot \ln(2)$

The r-parameter regulates the smoothness of the transition from one line segment to the other; when r becomes large, the transition becomes smoother whereas at small values for r, the transition becomes less smooth. This model is used to estimate the effect of several

characteristics, as indicated above, as a function of SGDags in treatment CP. The starting model can be rewritten by use of indicator variables (Neter *et al.*, 1990) to estimate the slopes and intersection point in treatment SP by substituting the parameters as follows:

$$a=a_0 + \alpha \cdot z$$

$$b_1=b_{01} + \beta_1 \cdot z$$

$$b_2=b_{02} + \beta_2 \cdot z$$

$$c=c_0 + \gamma \cdot z$$

In these set of equations, z is an indicator variable which is 0 for CP and 1 for SP. a_0, b_{01}, b_{02} and c_0 are the estimates of the parameters for treatment CP and a, b_1, b_2 and c are the estimates obtained for treatment SP. In addition, the parameters α, β_1, β_2 and γ give the difference in estimates between the intercept, slope at the first line segment, slope of the second line segment and the intersection point, respectively. From preliminary curve-fittings it was observed that, due to the few points in the experimental region, r could not be estimated properly. From the fits obtained it appeared that 2 was a reasonable value, hence r was fixed at 2 for all calculated regressions. From the porosity determinations it appeared that three linear segments were involved. An extension of model 1 is used in which one linear segment was added (three segments per treatment were used). Details on the extension of the model with more than two linear segments can be found in Koops and Grossman (1993). The same procedure as described above is used to obtain the differences in slopes (extended with b_3 and β_3) and intersection points between treatment CP and SP.

Mass debit through the pelleting line was analysed using model 2. The same methodology as described in model 1 was used for this analysis. On the basis of the information derived from figure 4c and the appearance of exit-die defects around 42% SGDags (see later) an indicator variable (z_1) was formed which divided the data on feed flux in two groups. The starting model was:

$$Y = a_1 + b_1 \cdot x + (a_2 + b_2 \cdot x) \cdot z_1 \qquad \text{Model 2}$$

z_1 was coded 0 in group I and 1 in group II. Model 2 describes therefore two lines: $a_1 + b_1 \cdot x$ in group 1 and $(a_1+a_2) + (b_1+b_2) \cdot x$ in group II. A further refinement of model 2 by using the indicator variable z, and the same methodology as described in the previous paragraph, showed that CP and SP did not differ in feed flux. Hence, these figures are not reported in the text.

Model 3 was used to study the effect of conditioning on differences in SGDags of the feed mash. It was also used to study the effect of conditioning and pelleting-cooling on the

differences in SGDags induced by these operations. In addition, the effect of the pellet press was studied by comparing the SGDags values before the pelletizer with the SGDags values in the pellets.

$$Y_{ij} = \beta_0 + Treatment_i + \beta_1 * (x_j - \bar{x}) + \beta_{1i} * (Treatment * (x_j - \bar{x})) + e_{ij}$$

Treatment is used as a class variable (either CP or SP). β_0 is the intercept at mean value of the independent variable ($\bar{x}$). Treatment$_i$ is the difference in intercept between CP and SP at mean value of the independent variable. β_1 gives the slope of the regression line for Treatment i and β_{1i} gives the difference in slopes for the two regression lines. In case of an absence of an interaction between the two lines (β_{1i}=0) the slopes of the lines have been compared at the mean value of the independent variable. If an interaction of SGDags with a unit-operation is present then β_1 differs from one.

All data was analysed using NLREG 3.5 (Sherrod, 1996), except the data on mass debit (model 2) and the data on differences between SGDags over unit-operations (model 3) which were analysed with the SAS-package (SAS, 1990).

Results

The characteristics of the used tapioca is given in table 1. The chemical and physical characteristics of the five test batches before pelleting are given in table 2. The particle size distribution showed the presence of a bi-modal distribution in each of the five batches B0 to B100. This bi-modal distribution originates from the distinct particle size- and distribution between the native and expander processed and reground material. The fractions of meal retained on the respective sieve diameters (large to small sieve-opening respectively) for B0 were; 0.4%, 0.7%, 9.8%, 14.6%, 12.6%, 18.8% and 43.2%. For B100 the following weight fractions were obtained: 0.3%, 7.1%, 34.7%, 23.7%, 12.8%, 7.2%, 14.2%. Due to the pre-processing step, the amount of water in the batches ranged from 128 to 169 gr kg^{-1} feed. Hence, mean particle size and moisture content are confounded with SGDags.

Table 1: Characterization of the tapioca.

Chemical composition	
Dry matter	873 g kg^{-1}
Total starch[a]	728.5 g kg^{-1}
Sugars[a]	~0 g kg^{-1}
Gelatinized starch[a]	94.1 g kg^{-1}
Physical characteristics	
Enthalpy of gelatinization[b]	7.65 J g^{-1} DM

[a] Based on Amylo-glucosidase test (in DM)

[b] Based on Differential Scanning Calorimetry

One of the replicates of treatment B50 could not be evaluated for its effect on physical pellet quality because no pellets were formed. It is not clear why no pellets were formed, since the replicate treatment was pelletable. Therefore, 19 samples were used in the analysis.

Table 2: Composition of the 5 starting batches before pelleting with different inclusion levels of pre-gelatinized tapioca.

Batch ID	SGDags (%)	Enthalpy (J g^{-1})	SGDdsc[1] (%)	x_{50} (µm)	Moisture content (%)
B0	11.5	7.65	0	109	12.8
B25	22.8	6.01	21.4	184	14.4
B50	34.2	5.72	25.2	208	14.9
B75	42.0	4.58	40.1	248	16.4
B100	53.1	3.84	49.8	315	16.9

[1] SGDdsc is expressed relative to the enthalpy of B0.

Temperatures reached after conditioning were 67.6 °C (± 3.8) at the SP treatment and 44.1°C (± 3.1) at the CP treatment. Due to the design of the experiment in which motorload was held constant, feed flux ranged between 235 kg h^{-1} (± 6.4) at B100 and 497 kg h^{-1} (± 37.2) at B25 in the CP treatment and between 291 kg h^{-1} (± 37.1) at B100 and 531 kg h^{-1} at B50 in the SP treatment.

The results of the hardness, durability, SME and exit-die defects test were fitted using model 1 using two connected linear segments. The porosity figures were fitted to model 1 using three connected linear segments. It was noted during the physical quality tests that some of the pellets did not break but yielded under the load. Physical quality of pellets which did not break or crush during the measurement, or which strength exceeded the limits of the tester,

were set to missing values and as such treated in the analysis. Thus, the following tests incorporated missing values: Kahl hardness; one replicate from B100. The compression test had 4 missing values (all treatments B100) due to yielding of the pellets instead of crushing. Past the intersection point(s), all pellets tested within the limits of the apparatus showed no clear breaking (crushing) behaviour, but yielded under the load. Furthermore, all hardness and durability values showed the presence of a minimum, which was in contrast with what was hypothesized. The first segments of the curves of the hardness, durability and SME slopes (b_{01} or b_1) were either zero or negative. Past the intersection point (γ) an increase in strength or energy consumption was observed at higher SGDags values. Regression coefficients are given in table 3. No differences were found between CP and SP before the intersection point of most hardness tests. No difference was found in energy consumption, feed flux and the appearance of exit-die defects between CP and SP.

The Holmen durability test showed no difference in slope and intercept for the first line segments (b_{01} and b_1) between the CP and the SP treatment. The slope was negative in this part of the curve indicating a decreasing durability with increasing SGDags. The intersection points differed (γ; $p<0.05$) between the CP and the SP treatment with 3.2 units. On the CP treatment the intersection point was found at 32.4 % (±2.33) SGDags, at the SP treatment the intersection point was found at 29.2 % (±2.05) SGDags. Slopes were similar between the line segments in the second part (b_{02} and b_2) of the curve. The slope of the curve was positive (3.18 ±0.40), indicating that, whenever past the intersection point, an increase in the amount of SGDags improved pellet durability (Figure 3a).

The Pfost durability test showed a difference in slope (β_1; $p<0.01$) between the CP and the SP treatments for the first line segment. Both slopes were negative (-1.01 ± 0.30 for the CP treatment and -0.70 ± 0.28 for the SP treatment, respectively). The intercept at SGDags=0 between the CP and SP treatment did not differ. No difference in intersection point (γ) was observed between CP and SP. The intersection point was estimated at 31.2 % SGDags (± 2.53). The slopes of the line of CP and SP in the second part (b_{02} and b_2) of the curve were equal. The slope was positive (1.52 ± 0.21) indicating an increasing durability past the intersection point with an increase in SGDags (Figure 3b).

Table 3: Regression coefficients of the curve-fits obtained from model 1 (For explanation see text).

Dependent variable	Intercept			Slope first line segment			Slope second line segment			intersection point			Model characteristics	
	a_0	a	α	b_{01}	b_1	β_1	b_{02}	b_2	β_2	c_0	c	γ	MSE	Prob. regr.
Holmen (%)	84.00***		-	-2.26**		-	3.18***		-	32.4***	29.2***	-3.2*	79.63	***
Pfost (%)	85.32***		-	-1.01**	-0.70*	0.31**	1.52***		-	31.24***		-	20.56	***
Kahl (kgf)	9.86*		-	-0.15ns		-	2.09***		-	41.4***	36.7***	-4.77*	21.32	***
Kramer (N mm^{-1})	79.46ns		-	-0.22ns		-	35.75***		-	41.8***	36.8***	-5.06**	4371.8	***
OLD (N g^{-1})	19.82***	9.60**	-10.22*	-0.63*	-0.12ns	0.51#	0.016ns		-	24.2**		-	1.78	**
SME (kJ kg^{-1})	96.37***		-	-1.81#		-	5.26***		-	35.80***		-	361.5	***
Sharkskin (-)	0.21ns		-	-0.01ns		-	8.24***		-	41.96***		-	78.22	***

ns = $p>0.1$; # = $p =< 0.1$; * = $p < 0.05$; ** = $p < 0.01$; *** = $p < 0.001$.

Smoothness parameter was fixed to a value of 2, differences in smoothness parameter between CP and SP were set to zero.

a_0 is the intercept for treatment CP at SGDags = 0.

a is the intercept for treatment SP at SGDags = 0.

α is the difference in intercept between CP and SP ($a-a_0$).

b_{01} is the slope of the first line segment for treatment CP.

b_1 is the slope of the first line segment for treatment SP.

β_1 is the difference in slope between treatment CP and SP (b_1-b_{01}).

b_{02} is the slope of the second line segment for treatment CP.

b_2 is the slope of the second line segment for treatment SP.

β_2 is the difference in slope between treatment CP and SP (b_2-b_{02}).

c_0 is the intersection point of the two line segments for treatment CP.

c is the intersection point of the two line segments for treatment SP.

γ is the difference in intersection point between treatment CP and SP ($c-c_0$).

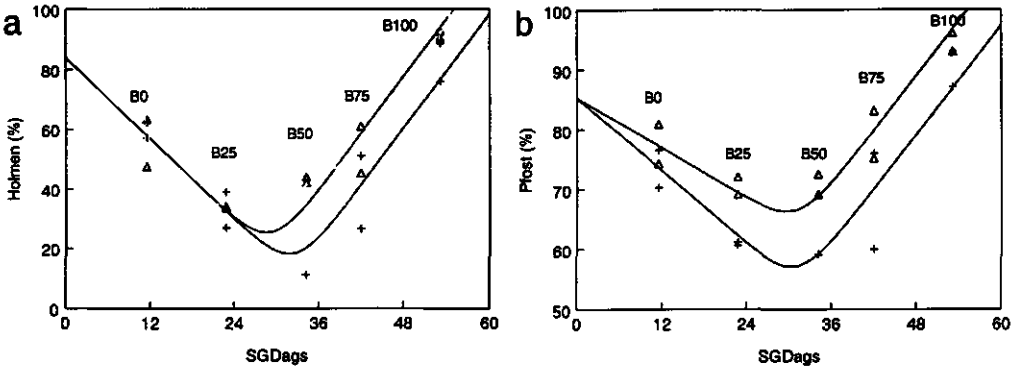


Figure 3a) pellet durability as tested with the Holmen pellet tester. 3b) Pellet durability as tested with the Pfost pellet tester. Solid line represents the fitted model (see text). + is CP treatment, ▲ is SP treatment.

The Kahl hardness test showed no difference in slope and intercept in the first line segments (α_0 and β_1; $p>0.1$) between CP and SP. The slope was negative, but not different from zero (-0.15 ± 0.18). The intersection point differed (γ; $p<0.05$) between CP and SP with 4.8 units. On treatment CP the intersection point (c_0) was found at 41.4% SGDags (± 3.49), at SP the intersection point (c) was found at 36.7% (± 3.15) SGDags. No differences in slope were observed between the line segments in the second part (β_2; $p>0.1$) of the curve. The slope of the curve was positive (2.09; ± 0.41), indicating that, whenever past the intersection point, an increase in the amount of SGDags improved Kahl hardness (Figure 4a).

The fit for the curve of the compression and tension test (OLD) showed differences in the slope (β_1; $p<0.1$) and the intercept (α; $p<0.05$) in the first line segments between CP and SP. The slopes (b_1 and b_{01}) were both negative in this part of the curve indicating a decrease in crushing strength with an increase in SGDags. The slope was -0.63 (± 0.20) at the CP treatment and -0.12 (±0.11) at the SP treatment. The intersection points for the two curves were not different (γ; $p>0.1$). The intersection point (c) was found at 24.2% (± 5.42) SGDags. No differences in slope was observed between the line segments in the second part (β_2; $p>0.1$) of the curve. The slope of the curve in the second line segment was not different from zero (0.016 ±0.097), indicating that, whenever past the intersection point, no change in crushing strength was obtained with an increase in SGDags (Figure 4b). The fact that no effect was present of an increase in SGDags on the compression test values indicate that, taking into account the relative low speed of the moving plate in the test (10 mm min^{-1}), that the pellets had enough time for relaxation. Hence, it is argued that these values are due to yielding and not fracture of the pellets.

The fit for the curve of the values derived from the Kramer Shear Press (figure 4c) showed no differences in slope and intercept of the first line segments (α and β_1; $p>0.1$) between CP and SP. The slope was negative but not different from zero (-0.22 ± 2.44). The difference in intersection point (γ) was significant ($p<0.01$) between CP and SP with 5.06 (± 1.57) units. On treatment CP, the intersection point (c_0) was found at 41.8% SGDags (± 3.04) at SP the intersection point (c) was found at 36.8% (± 2.83) SGDags. No differences in slope were observed between the CP and the SP treatment in the second part (b_2 and b_{02}) of the curve. The slope of the curve was positive (35.75; ± 5.95), indicating that, whenever past the intersection point, an increase in the amount of SGDags improved the shearing strength as measured with the Kramer Shear Press.

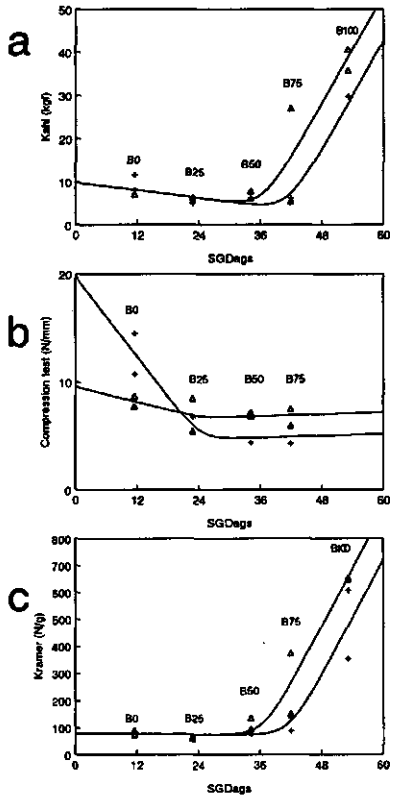


Figure 4a) Kahl hardness as a function of SGDAGS; 4b) Crushing strength of pellets as tested between flat platens; 4c) Shearing strength of the pellets as determined with the Kramer Shear Press. Solid lines represent the fitted model (see text). + is the CP treatment, ▵ is the SP treatment.

Specific Mechanical Energy (SME) did not differ between CP and SP. The fitted curve showed a decrease in the first line segment (b_1) which tended to differ from zero (-1.81 ± 0.86). The intersection point (c) was found at 35.8% (± 3.77). The second line segment (b_2) had a positive slope (5.26 ± 1.27) (straight line, Figure 4a). Since the amount of SME on the B50 treatment was low, an analysis was performed which excluded this treatment (dotted line). This resulted in a slope of the first line segment (b_1) which did not differ from zero (-1.11 ± 0.68). The intersection point increased 1.9 percent units ($p>0.1$) to 37.7% (± 3.57) SGDags. The slope of the second (dotted) line (b_2) segment was 5.07 (± 1.18), which was not different from the slope of the 'straight' line ($p>0.1$) (Figure 5a).

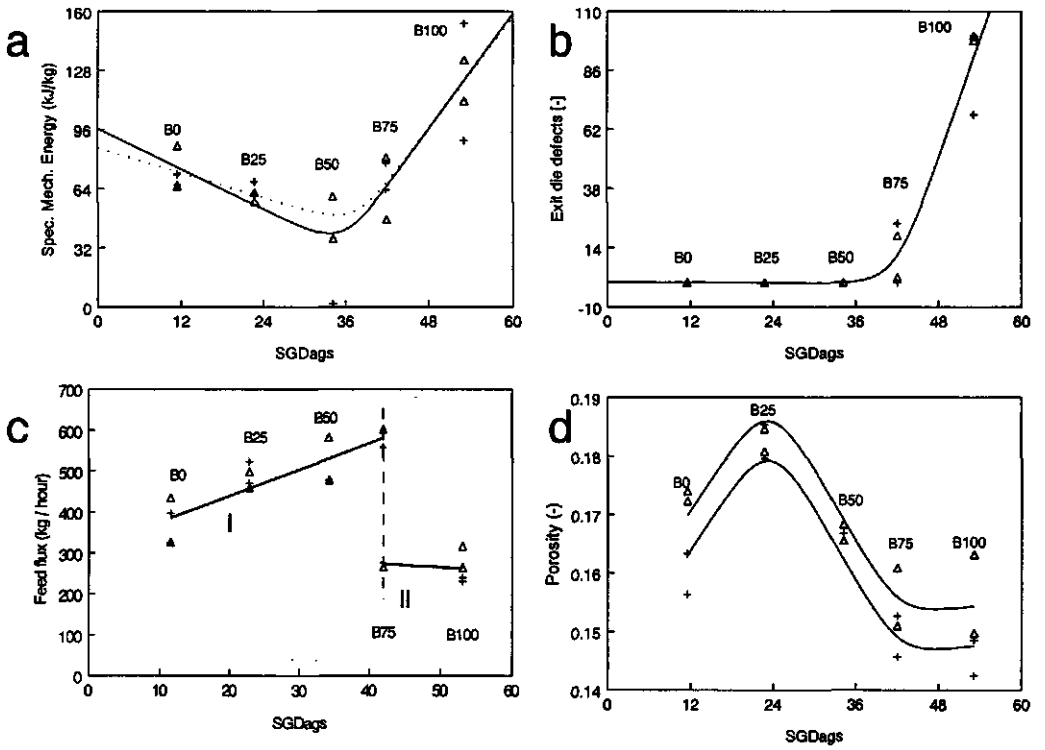
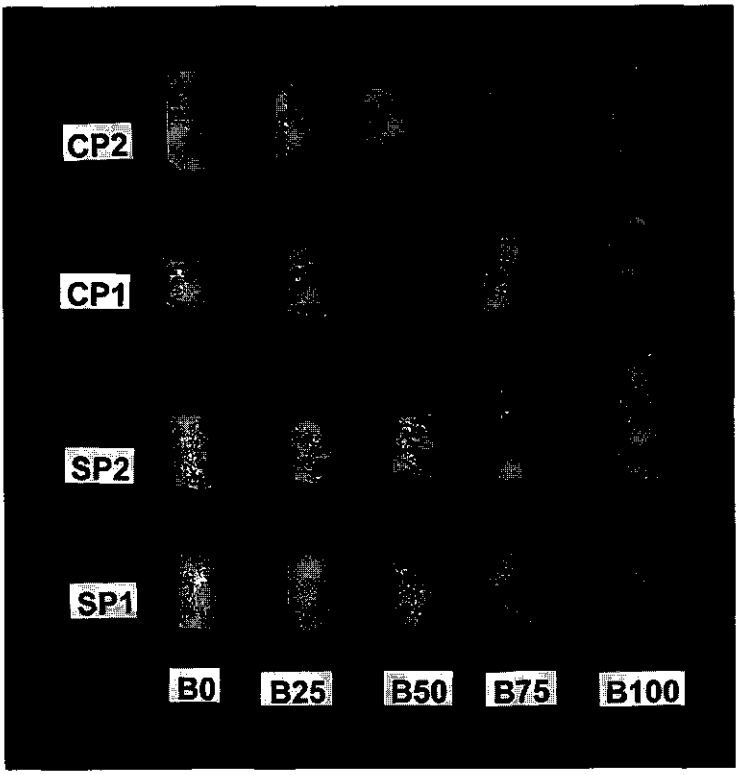


Figure 5a) Specific mechanical energy consumption of the pellet press (kJ kg^{-1}); 5b) Percentage pellets showing exit-die defects; 5c) Feed flux as a function of SGDags. I and II represents arbitrarily formed groups at which data has been divided (see text for explanation); 5d) Pellet porosity as a function of SGDags. + is the CP treatment, ▵ is the SP treatment.

Exit die defects appeared in both the CP and SP treatment (figure 5b). No differences were observed in the slopes between the line segments (β_1, β_2) or in the intersection points (γ). Hence, one regression line was fitted to the exit die defects. The first line segment of the curve showed no difference between CP and SP. Neither the intercept nor the slope was different from zero (intercept: 0.21 ± 7.12, p>0.1; slope: -0.02 ± 0.31, p>0.1). The intersection point was found at 42.0 % (± 2.32). The slope of the second line segment was positive; 8.24 % (± 1.38) SGDags (Figure 5b). Picture 1 shows the pellets that were formed. The exit-die defects are present in the B75 (CP1 and SP2; the numbers denote the replicates) and B100 (all).



Picture 1: Photograph of the pellets obtained in this study. Note the exit-die defects on the pellets derived from the B100 batches and B75 (SP2 and CP2).

Table 4 lists the regression coefficients for the regression of porosity on SGDags. The fit for porosity was extended to include three line segments. The second intersection was fixed at 42% SGDags in order to avoid mutual dependencies between the slope of the second line segment (b_2), the second intersection point and the third line segment (b_3).

Table 4: Regression coefficients from the regression of porosity on SGDags. The regression coefficients are obtained after reduction of the model (see also figure 5d).

	CP	SP	α_0
a Intercept	0.142 (± 0.010) ***	0.148 (± 0.010) ***	0.0060 ± 0.0022 *
b_1 (slope first line segment)	16.07E-4 (± 7.83E-4) #		-
b_2 (slope second line segment)	-18.72E-4 (± 6.37E-4) *		-
b_3 (slope third line segment)	1.21E-4 (± 4.05E-4) ns		-
c1 (first intersection point)	23.5% SGDags ***		
c2 (fixed second intersection point)	42.0% SGDags		

Mean Square Error of the model: 2.296E-5.

Asymptotic probability of the regression: prob(F) < 0.001

This procedure seems reasonable since from the exit-die defects tests it followed that at approximately 42% SGDags changes in the relation between physical pellet quality and system parameters occur. For all the three line-segments, the differences between the slopes of the respective segments were not significant ($p>0.1$) between the CP and SP treatment. A difference in intercept was present between CP and SP ($p<0.05$), with the CP treatment having the intercept at 0.142 (± 0.0102) and SP at 0.148 (±0.010). The slope of the first line segment is positive (b_1; 0.0016 ± 0.0008). No differences between CP and SP were observed in the intersection point between the first and second line segment (γ_1; $p>0.1$). The intersection point was estimated at 23.5% SGDags (± 4.89). The slope in the second part of the curve (b_2) was negative (-0.0019; ± 0.0006), indicating a decrease in porosity with an increase in SGDags between 23.5% (estimated) and 42% (fixed) SGDags. The slope (b_3) at the third part of the curve was positive (0.00012 ± 0.0004), but not different from zero (Figure 5d).

The analysis of the feed flux as a function of SGDags was further refined by inclusion, in model 2, of an indicator variable (z), coding for CP and SP, as used in model 1. By use of the same methodology as described in the text for model 1, differences between the CP and SP treatment were tested. The analysis showed that no differences were present between the CP and the SP treatment, therefore, these results are not included in the text. An increase in feed flux with increasing SGDags in group I was observed. In group II, the slope of the curve did not differ from zero, hence no change in feed flux was observed with increasing SGDags in group II. No differences were observed in intercept between the lines of group I and group II. The regression equation was: 312.3 (± 28.9) + 6.42 (± 1.06) * SGDags for the line representing group I. For group II the regression equation was: 312.3 (± 28.9) - 0.93 (± 0.68) * SGDags. At 42.0% SGDags, the estimated feed flux in group I was 582 kg hour^{-1} (± 21.8 std.err.) and the estimated feed flux in group II was 273 kg hour^{-1} (± 15.5 std.err).

Original samples (not dried) were only present of the B0 and B100 treatments (pellets and mash), only these were evaluated to check for the appearance of glass transitions and moisture content. Since all experiments were done with pure non-pretreated (B0) or partly pre-gelatinized (B100) tapioca meal or mixtures thereof, it is assumed that mixtures of these treatments have their glass transition points somewhere inbetween, depending on the different inclusion percentages. Although the DSC-measurements on the samples to evaluate whether or not glass transitions were present, were done at a much later stage in the project, these may be indicative for what may have happened during the processing stage and as such provide a causative reason for the observed exit-die defects and large increase in pellet hardness and durability figures. The DSC-measurements show the presence of a glass transition in the B0 feed mash at approximately 42 °C and 11.8 % moisture. In the feed mash of the B100 treatment a glass transition was found at 32 °C and 18.4 % moisture content. In the pellets from the B100 treatment the glass transition was found at approximately 40°C and 18.6 %

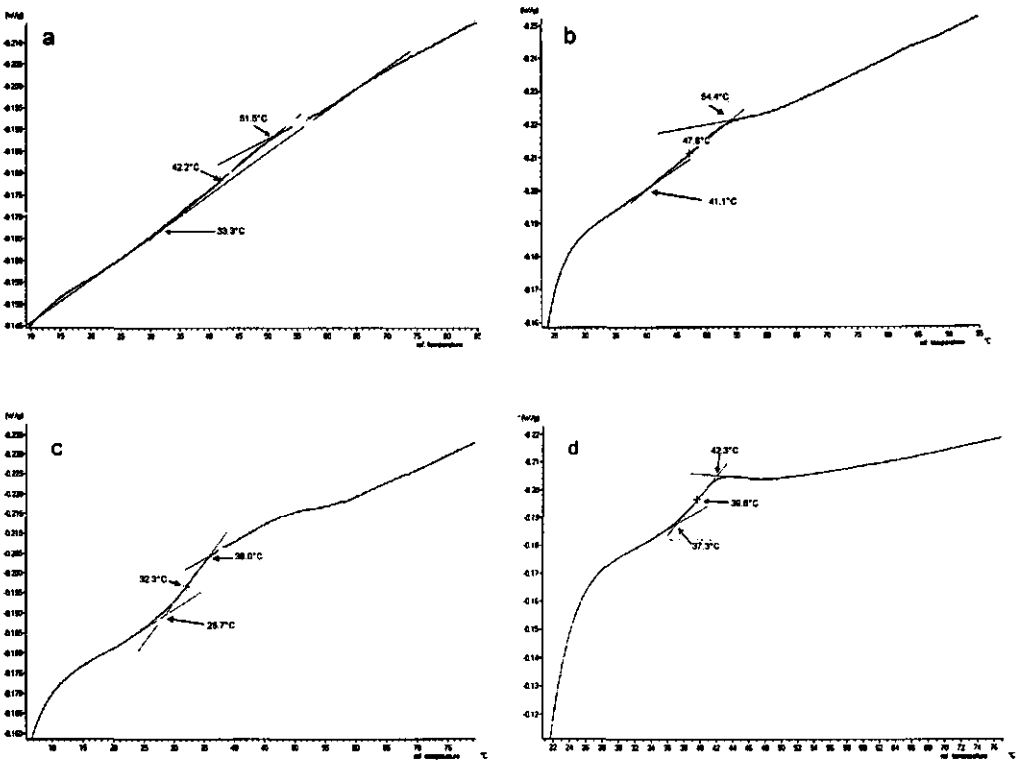


Figure 6: Glass transitions in: 6a) the starting material of B0; 6b) the pellets of B0; 6c) the starting material of B100 and 6d) in the pellets of B100.

Table 5: Model fits and estimates of the differences in degree of starch gelatinization (SGDags) induced by the unit-operations.

Dependent Variable	Intercept at treatment means ($\bar{x}$)			Slope at the two temperature levels			Model Characteristics.			
	CP[a]	SP[a]	Diff[ab]	CP[cd]	SP[cd]	β_{1i}[a].	R^2	MSE	Prob. Regression	Range of values (lowest - highest)
Batches -> conditioner[e]	30.0***	30.7***	0.058ns	0.97ns	1.03ns	0.059ns	0.99	3.50	***	8.1 - 54.2[f]
Batches -> pelleting[e]	64.5***	70.6***	16.86***	0.76*	0.43***	0.326*	0.88	15.70	***	43.1 - 84.7[g]
Conditioner -> pelleting	64.7***	70.7***	16.54***	0.77*	0.42***	0.357**	0.89	14.41	***	Idem

For probability levels see table 3.

[a] Probability indicated is the probability of the LSmean or parameter differing from zero (H_0: parameter=0).

[b] Diff. is the difference in intercept at treatment means or the difference in slopes at the two temperature levels.

[c] All slopes were different from zero at P<0.01 level.

[d] Probability indicated is the probability of the parameter differing from one (H_0: parameter=1)

[e] Mean SGDags value of the Batches was 32.6%.

[f] Highest and lowest SGDags value after the conditioner

[g] Highest and lowest SGDags value after pelleting cooling.

[h] Mean SGDags value of the conditioned feed mash was 30.4%.

moisture. The pellets from the B0 treatment showed a glass transition at 48°C and 13.0 % moisture (see figures 6a, b, c and d). The glass transition is found in or below the region of processing temperatures during experimentation, and therefore indicates that a transition from hard (cristallyne) material to a rubbery-like material may have occured.

Changes in physico-chemical characteristics after the unit-operations.

Conditioner treatment did not have a large effect on the SGDags values. Pelleting-cooling had an effect on the SGDags values compared to the values after the conditioner, these became higher, indicating that pelleting-cooling increased the degree of gelatinization of the starch fraction (Table 5). Results from the analysis of the SGDags values after the conditioner showed that the slopes of the regression of SGDags after the conditioner on the SGDags values of the starting batches were not different from 1. In addition, no interaction was present between the two slopes (Diff), hence the intercepts at mean value of the independent variable were compared. These were 30.0% (± 0.6 stderr) and 30.7% (± 0.6 stderr) for CP and SP respectively. Values after the conditioner were lower ($p<0.05$) as compared to the mean value of the starting batches; 32.6% SGDags (figure 7a). It can therefore be concluded that all values were equally affected and became approximately 2% units lower.

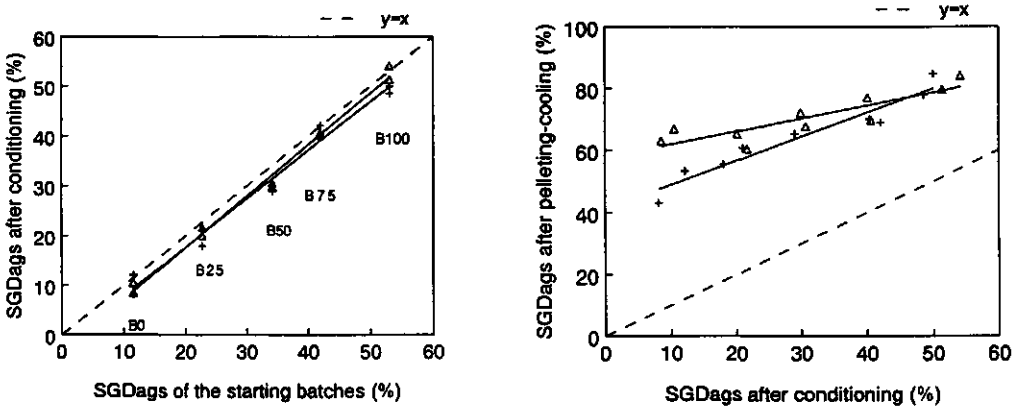


Figure 7: 7a) Comparison of SGDags values after conditioning as a function of SGDags values of the starting batches. 7b) Comparison of SGDags values after pelleting-cooling as a function of SGDags values after the conditioner. (See text).

From table 5 it follows that the results of the regression of the SGDags values after the pelleting-cooling step as a function of the SGDags values of the starting batches are virtually the same as the results of the regression of the SGDags values after pelleting-cooling on the SGDags value after the conditioner. Therefore, only the results of the regression of the SGDags values after pelleting-cooling on the SGDags values after conditioner will be discussed. An interaction existed between the two treatments CP and SP, as can be seen from

the significant difference in slope between CP and SP (β_{1i}; 0.357±0.116). Both slopes were significantly differing from 1 indicating that an effect is present of the pelleting-cooling step on the degree of gelatinization of the starch fraction. An interaction was present between the two slopes, hence the comparison of the two intercepts at mean value of the SGDags values after the conditioner is ambiguous. SGDags values increased due to the pelleting process as can be observed from figure 7b. All values were considerably higher after pelleting-cooling than after condtioning; all values were above the line y=x.

The correlation between SGDags and the enthalpy values for the 19 measurements of the mashes was -0.98 ($P<0.0001$). Correlation between the SGDags and enthalpy values of the pellets for the 19 measurements was -0.86 ($P<0.0001$). Enthalpy-values and SGDags values of calculated percentages starch gelatinization from these DSC figures are given in table 2. SGDags values after the conditioning and the pelleting-cooling step are given in table 5. The range of values of the residual enthalpy values derived from the DSC determinations are inbetween 1.35 J g^{-1} and 3.45 J g^{-1} DM.

Discussion

From table 2 it can be observed that the effects of SGDags, particle size and moisture content are not independent of each other. These confounding factors make it difficult to attribute the measured traits uniquely to SGDags. Part of the discussion will therefore focus on the known effects from literature, on the effect of particle size on agglomerate strength. Furthermore, the effect of moisture on physical and chemical properties of starch and possible interactions with the formation of pellets will be discussed.

Although initially the feed flux seemed to increase with an increasing amount of gelatinized starch present in the batches, the lowest feed flux was found in the batch with the highest inclusion level of gelatinized starch. The feed flux was adjusted to obain constant load on the pellet press. The effect of an increasing amount of gelatinized starch may therefore be twofold, at low concentrations it may act as a lubricant, whereas at high levels of pre-gelatinized starch, the rheological properties of the feed mash are modified to such an extent that feed flux had to be lowered. The change in rheological properties is further evidenced by the appearance of exit-die defects. The high temperature for the feed mash at the CP treatment resulted from the residual heat in the different processing apparatus, due to previous SP treatments.

The effect of particle size and porosity.

Smaller particles induce higher agglomerate strength as opposed to larger particles (Rumpf, 1958; Shotton, 1979; Pietsch, 1991) due to the fact that more contacting points between particles in a unit volume of the agglomerate are present. Therefore, when particle size is the only factor, the highest strength is expected in B0 with mean particle size being 109 μm. Consequently, a lower strength is expected in B100 with a mean particle size of 315 μm. Indeed, in most of the curves on hardness and durability figures an initial decline or a trend to a decline in the measured hardness and durability values is observed (Figures 3a, 3b and 4a, 4b and 4c), which may be attributable to particle size. In addition, porosity increased when going from B0 to B25 with a concomittant decrease in hardness and durability values, most probable, due to a decrease in the amount of contacting points between particles in a unit volume of pellet (Rumpf, 1958; Ouchiyama and Tanaka, 1985; Pietsch, 1991). However, a further increase in SGDags, shows decreasing porosities, which do not closely match with increased hardness and durability values in the pellets. For instance, in the pellets derived from batch B75 and B100, porosity values do not change as a function of the SGDags values (b_3=0; table 4), whereas a tremendous increase in hardness values is observed.

Functional properties of starch.

The feed mash in this study is mainly composed of starch (>70%). Starch consists of a molecularly ordered and partly cristallyne part and an amorphous fraction (Cooke and Gidley, 1992). With progressively increasing SGDags, a larger part of the starch consists of an amorphous fraction which may exhibit a glass transition. A larger proportion of this amorphous starch makes the behavior of the feed mash progressively dependent on the glass transition properties of this fraction. When the amorphous fraction passes the glass transition, than the (sudden) differences in behaviour may lead to large differences in observed physical properties of the agglomerate. Before the glass transition the amorphous starch fraction may exhibit the same properties as the crystalline fraction (relatively hard and brittle). Past the glass transition, the amorphous fraction will behave elastically. When the amount of amorphous starch is large enough and it is in the rubbery state, then this may induce a different phase behavior. This transition of one phase to another may lead to drastically altered physical behaviour as for instance has been shown for extruded potato-starch soya-protein mixtures by Zasypkin (1992) and is discussed by Eliasson and Bohlin (1982). The results obtained from the DSC-analysis show that glass transitions appeared in the feed mash of the batches B0 and B100 and in the pellets derived thereof. These glass transitions appeared at temperatures below the processing temperatures during experimentation.

The physical properties of starch are, amongst others, modified by the amount of moisture present. Table 2 indicates the increasing amount of moisture present in the batches B0 to B100, due to the mixing step. Water incorporated in the feed material may act as a plasticizer

thus altering the characteristics of the feed material as readily has been shown in extrusion experiments (Remsen and Clark, 1978; Harper, 1981; van Soest *et al.*, 1996). A further consequence of the presence of water is that the glass transition of the starches moves to lower temperatures (Zeleznak and Hoseney, 1987; Keetels, 1995). This, in turn, affects the rheological behavior of the starch. Kalichevsky *et al.* (1992) show that the Young's modulus of waxy maize starch at room temperature decreases as a function of water content (between 15 - 25% w/w) due to the transition from a glassy to a rubbery state. Venet and Vergnes (1996) showed that exit-die defects are related to the viscoelastic properties of polymers. Therefore, a transition from solid-like to rubbery-like in the region under investigation may have altered the properties of the tapioca mash during pelleting in such a way that exit-die defects occured due to the changed rheological properties. According to Kurtz (1992) stretching is a necessary prerequisite for the induction of exit die defects. Due to this stretching, the porosity of the pellets will change. It appears that higher temperatures (as was present in the SP treatment) induces a higher amount of stretching, since porosity values of the SP treatment induce a higher porosity compared to the (colder) CP-treatment. As can be observed from figure 5d and table 4, the necessary addition of a third line segment, in the range of SGDags values were exit-die defects occured, may therefore provide further evidence for the proposed concept. Van Soest *et al.* (1996) found a sharp decrease in E-modulus when passing the glass transition point. These change in E-modulus was accompanied by a sharp increase in elongation and tearing energy as measured with an Instron Universal Testing machine.

Hardness tests

The results of the compression test show, in contrast to the Kahl and Kramer shear press data, no increase in strength past the intersection point. The low values for the compression test may have originated from the low speed of travel of the moving bar (10 mm min^{-1}), giving the material enough time for relaxation. The high hardness values found with the Kramer shear press may originiate from the fact that speed of travel of the measuring body is much larger (~10 mm s^{-1}), thus providing not enough time for the pellets to relax. The Kramer shear press and the Kahl hardness test showed a sharp increase in crushing strength after 41 % SGDags. In the first part of the curve, no differences between the CP and SP treatments were observed. The slopes in the hardness tests ($<\gamma$), were not differing from zero, except for the hardness values in the CP treatment derived from the compression test. This slope was negative and different from zero. Hardness values according to Kahl in the first part of the curve, indicate levels which are normally encountered in feed manufacturing practice (<10 kgf). Past the intersection point, crushing strength properties were at a much higher level than normally encountered, up to ~ 40 kgf.

Durability tests

Both Holmen and Pfost data for durability show a decrease in durability before the intersection point(s) were reached. This may be partly attributed to the increased particle size and concomitant increase in porosity. Although exit-die defects led to a rough and open structure of the pellets in treatments B75 and B100, this was not reflected in lower durability figures. Moreover, durability in the B100 treatments was higher as opposed to the other treatments. This is in line with literature data of Van Soest *et al.* (1996), who show that tearing energy of the material past the glass transition may rapidly increase and, therefore, makes the material less friabel.

Feed flux

Although the division of the data on feed flux into two groups is rather arbitrarily, it should be noted that the two replications on the B75 treatment in group II (figure 5c) showed clear signs of exit-die defects (19 % and 24 %) and the other two replications (in group I) had zero or 2 % exit die defects in the pellets. The appearance of exit die defects and sudden decrease in flow rate seem therefore related. This is in agreement with results of Vergnes *et al.* (1992) who found that flow rate in a ethylene propylene diene monomer and the appearance of exit die defects were related.

The results from this experiment indicate that the glass transition in the starch is a causative factor in determining hardness and durability of the pellets and observed characteristics as, for instance, changes in the feed flux. It is argued that, the results of the hardness and durability values and the sudden changes in them are not related to the degree of gelatinization of the starch fraction *per se*. The degree of gelatinization determines the amount of amorphous starch, but does not detect changes in the state of this amorphous fraction (as is possible with DSC).

Eliasson and Bohlin (1982) attributed changes in the observed rheological behavior (relaxation) to gelatinization of starch. Their measurements were conducted with a water content of at least 30 % (w/w water to starch ratio). Their results indicate that during gelatinization, granules become softer. It is argued that this softening can not provide an explanation for the results found in this experiment, since water content in the current experiment was too low to pass the gelatinization point at the temperatures prevailing. Colonna *et al.* (1992) found that for various starch sources, gelatinization temperatures at water contents < 20 % w/w exceeded 100 °C, with lower moisture content inducing higher temperatures at which gelatinization of the starch granules occurs. In addition, when granules soften, packing density is most likely to increase, which would lead to lower porosities. From figure 4d it can be observed that this is not the case. In addition, the results of the analysis of the changes in SGDags-values as a function of the different unit operations, do not show any evidence for a certain drastic change in the degree of gelatinization which then could explain

the rather sudden changes in the hardness, durability values and feed flux as observed in this experiment.

From the DSC-determinations it can be observed that all glass transitions took place in a temperature region just below, or close (to the temperature in the CP treatment), to the processing temperatures. This means that the amorphous starch fraction is in a rubbery-like state. Not all pellets showed, however, exit-die defects. It is therefore proposed that at least two requirements should be met, in order to induce the appearance of exit-die defects and its associated impact on hardness, durability values and feed flux: First, the processing temperature should be above the glass transition temperature and secondly, the amount of amorphous starch should be high enough to become the pre-dominant 'phase' in the feed material. From this study it appears that this amount of amorphous starch should be approximately 42% SGDags (appearance of exit-die defects) * 729 g kg^{-1} (starch content) which is approximately 300 g kg^{-1} or 30 % of the feed mash.

Conclusions

Due to the confounding factors, water, particle size and SGDags, which from literature appear to be important, it is difficult to determine what the most causative factors are in explaining pellet hardness, durability values, porosity, SME and the exit-die defects observed. In contrast to the hypothesis, no continous increase in hardness and durability parameters was found with a progressive increase in SGDags in the feed. It is concluded from this study, that steam pelleting (SP) yields harder and more durable pellets when compared to cold pelleting (CP) whenever past the intersection point. From the observed data it becomes clear that, at least, two counteractive effects must have been present in the data. One which induces lower pellet hardness and durability values with an increase in SGDags and one which, after a certain SGDags value, induces higher pellet hardness and durability values with further increasing SGDags values. The initial decrease in pellet hardness and durability values was attributed to the increase in particle size, whereas the large increase in pellet hardness and durability values after 42% SGDags was attributed to the glass transition in the amorphous part of the starch fraction. In order for the amorphous starch fraction to induce the observed changes in feed flux and the appearance of exit-die defects, two requirements are necessary, the first requirement is that the processing conditions during manufacturing should be above the temperature at which the glass transition appears, the second requirement is that enough amorphous starch is present to become the dominant phase in the feed mash. From this study, this appears to be approximately 30%. In this study, porosity was not found to be a causal factor in determining the physical quality of the pelleted feed. However, it may be usefull as an indicator to obtain additional information on the behaviour of starch rich feed materials and its effect on pellet quality.

In order to be able to determine the effect of raw material properties on hardness and durability traits of pelleted animal feeds it is therefore recommended to use in future experiments, batches which are equal in particle size and have an equal moisture content. Further investigations with respect to properties of animal feeds should preferably include also a form of nutritional evaluation.

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Physical quality of pelleted soy-grits is affected by differences in protein dispersibility index.

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Abstract

Model feeds of soy-grits which differed in the degree of denaturation of the protein were pelleted. It is hypothesized that at a gradual replacement of denatured protein by native protein, as determined by its Protein Dispersibility Index (PDI) will lead to harder and more durable pellets. In addition, the effect of mash temperature before entering the pellet press (65°C and 85°C) was examined on pelleting properties of the model feeds. Non-linear regression was used to evaluate the effects of PDI on hardness and durability of the pelleted feeds. Holmen and Pfost durability (%), feed flux (kg h^{-1}) and specific mechanical energy (SME; kJ kg^{-1}) were found to follow a sigmoidal relationship with PDI. Hardness parameters; Kahl (kgf), Kramer Shear Press (N per gr pellet) and Compression tests (N per mm pellet length) were found to follow an exponential relationship as a function of PDI. Pellets were harder and more durable with increasing PDI. Feed flux and SME were found to decrease with increasing PDI. Porosity of the pellets was determined and found to be linearly related with the hardness of the produced feed pellets. Pellet porosity ranged between 0.088 and 0.136. Lower porosity correlates with higher pellet strength and vice versa. The 85°C temperature level led to harder and more durable pellets in all cases, as opposed to the 65°C temperature level. NSIkoh values were also determined as a second indicator for the amount of denatured protein present; and these values, however, were found to increase after processing, in contrast to what was expected.

Introduction

Most feedstuffs that are used in pelleted animal feeds have a processing history which may affect their nutritional and functional properties. Nutritional changes associated with the feed manufacturing process, especially pelleting, have been reported in literature (Vanschoubroeck *et al.* 1971). These literature findings are not allways consistent with respect to the effects of feed processing and performance of animals. Skoch *et al.* (1983a) did not find an improved performance (average daily gain, feed intake, feed conversion and energy digestibility) of weanling pigs fed a pelleted corn-soybean meal diet over the diet fed as a mash, whereas Skoch *et al.* (1983b) found an improvement in animal performance (feed conversion) when feeding a pelleted diet over a feed mash containing wheat middlings.

A large number of parameters have been used in feed manufacturing to obtain an indicator of protein quality and its associated nutritional value. Parsons *et al.* (1991) studied the relation between soy protein solubility in KOH (NSI_{KOH}) and performance characteristics in chicks and pigs. They concluded that the NSI_{KOH} was a good indicator of *in vivo* soybean protein quality. Bookwalter *et al.* (1971) evaluated the extrusion process of full-fat soy flour using various

methods, all aiming at the quantification of the baking effects of soyflour in bread. Among the tests used were determination of color values and nutritional value by protein efficiency ratios (PER) and lysine availability, antioxidant tests and protein solubility (NSI in water). Enzymatic tests have been used by Björck *et al.* (1985) to assess protein nutritional value of cereal based extrusion products. They concluded that the *in-vitro* method used in their work correlated well with results obtained from balance experiments with rats.

According to Kinsella (1979) and Hermansson (1979), the protein fraction of soy is one of the main contributors to the functionality of the soy in its wide-spread applications. Different methods and principles are applied to determine soy-protein functionality in feed, or food products (Hermansson, 1979). In food and feed manufacturing, a few (empirical) tests have been developed to relate the state of the protein to, for instance, nutritional properties of the protein or functional properties in relation with the texture of the commodity. The protein dispersibility index (PDI) has traditionally been used to characterize the protein quality of the soy (Horan, 1974). In feed manufacturing, commercially obtained batches are often purchased with PDI-value used as an indicator of the protein quality. In addition, PDI and NSI_{KOH} have been used to determine the effects of processing conditions on changes in the state of the protein. Marsman *et al.* (1995) concluded from their study with single screw extrusion of toasted and untoasted soy bean meal that PDI was not a suitable parameter to determine differences in protein quality when materials were processed under moderate and severe specific mechanical energy (SME) conditions (up to ~2300 kJ kg^{-1}). The NSI_{KOH} was able to discriminate between protein qualities under moderate to severe conditions of processing. Thomas *et al.* (1997a) concluded from expander processing studies that PDI was a good parameter to discriminate between processing conditions when low amounts of SME were used (<110 kJ kg^{-1}). Hence, in this study PDI is used as primary determinant of protein quality, since the amount of dissipated energy in feed manufacturing usually does not exceed 110 kJ kg^{-1}. In addition, NSI_{KOH} has been used as a reference test for the protein quality of the used soy-grits.

Although nutritional effects of processing will remain the main focus for the feed manufacturing industry, the quality of production (in its broadest sense) has become a further important aspect of feed manufacturing as well. Awareness of the quality issue and subsequent action of the feed industry can simply be determined by the increased number of ISO-certified and according to GMP-codes producing feed companies. A study of Wood (1987) showed the large effects of functionality of feedstuff constituents, particularly protein and starch, on pellet hardness and durability. In the study of Wood (1987) the inclusion of raw protein led to better pellet hardness and durability than the use of denatured protein. In addition, a gradual replacement of native starch by pre-gelatinized starch led to an increase in pellet hardness and

durability. It was concluded that the functionality of the protein was one of the main contributors to pellet quality.

The objective of this experiment is to relate physical quality characteristics (hardness and durability) to the protein quality of soy-grits. It is hypothesized that a gradual replacement of denatured protein by native protein will lead to harder and more durable pellets.

Materials and methods

Two batches of soy-grits (Cargill b.v. Amsterdam, The Netherlands) differing in protein dispersibility index (PDI) were used. PDI of batch A was 25.4 % and the PDI of batch B was 86.9% (Table 1). These two batches were mixed in a Nauta-mixer to obtain five different model feed mashes differing in PDI (refered to as B1 to B5). Moisture content of the batches was equalized to a standard moisture content of 72.0 (stderr ± 1.97) g kg^{-1} by adding tap water during mixing. In a previous experiment (Thomas *et al.* 1997b) particle size and moisture content were found to be causative factors for physical pellet quality. Therefore, care was taken to ensure almost similar particle size in batch A and batch B. A Fritsch Analysette 3 equipped with six sieves (2500, 1250, 630, 315, 160 and 71 μm) and a pan (<71 μm) was used to determine particle size. The sieve analysis was performed in duplo. Mean particle size was 652.5 μm in batch A and 625.5 μm in batch B, calculated according to Waldo *et al.* (1971).

Table 1: Chemical composition and some physical properties of the used soy grits.

Constituent	Batch A	Batch B
Moisture (g kg^{-1})	70.4	71.5
Anorganic matter (g kg^{-1})	66.3	63.4
Crude Fat (g kg^{-1})	22.8	23.4
Crude Fiber (g kg^{-1})	35.1	28.4
Crude Protein (g kg^{-1})	524.3	542.6
Nitrogen Free Extract (g kg^{-1})	281.1	270.7
Geometric Mean Diameter[a] (μm) (± variance)	652.4 (± 327.9)	625.5 (± 397.6)
Protein Dispersibility Index (%)	25.4	86.9
Nitrogen Solubility Index (KOH) (%)	86.4	97.6

[a] Calculated according to Waldo *et al.* (1971).

A 2 * 5 pelleting study was conducted to investigate the effect of protein quality (5 levels), measured with PDI, and temperature (2 levels) after conditioning on hardness and durability characteristics of pelleted animal feed. Each of the five obtained model feed mashes (B1 to B5) was splitted in four lots of 350 kg each. Pelleting studies were conducted at two temperature levels; 65°C and 85°C, with two replicates per treatment. Batches were randomly pelleted within temperature level.

Process description

Pelleting was done, using the facilities of the Wageningen Feed Processing Center (WFPC). A conventional barrel type conditoner was used to blend steam into the feed mash to obtain the required conditioning temperatures. Subsequently, the material was conveyed through the expander section and pelleted in a Simon Heessen V3-30 pellet press with two rollers. Die dimensions were 5*45 mm and a warm die was used for each run. The pellets were cooled in a Robinson counter flow bunker cooler with an eight and a halve minutes residence time. During processing, temperatures of the mash entering the pellet press and pellet temperature were measured using the thermosflask method. A μ-mac 6000 process control and data logging computer was used to monitor the process during experimentation. Energy consumption of the pellet press was calculated from the data that were recorded during each run. Reported energy consumption values of the pellet press are corrected for idle running. Samples were taken immediately after conditioning and after cooling. Samples taken after the conditioner were air-dried at 50 °C for 16 hours. Samples for PDI and NSI_{KOH} determinations were milled in two steps (3 and 1 mm screen, respectively) in a Retsch ZM1 centrifugal mill. The material was kept in plastic bottles and stored at 4°C until further analysis. Samples were analysed for dry matter by oven drying during 4 hours at 103°C. Protein Dispersibility Index and Nitrogen Solubility Index were determined as described previously (Thomas *et al.*, 1997a).

Physical quality of the pellets.

Hardness (Kahl, Kramer shear press and a general compression test) and durability (Holmen, Pfost) tests were done according to the prescriptions in Thomas *et al.* (1996) and Thomas *et al.* (1997b).

For porosity determinations, true density of the model feed mashes was determined with a pycnometer using alcohol as immersing fluid. Apparent density of the pellets was determined using a 500 ml measuring cylinder and glassbeads (Fisher Scientific, 560-800 μm). Preliminary tests to determine pellet volume involved lacquering of the pellet with different sprays and immersing in fluids, differing in viscosity. The eventual choice to use glassbeads as immersion

material arised after unsatisfactory results from these tests. The measuring cylinder is filled to 150 ml with glassbeads and vibrated two times for 10 seconds on the plateau of a sieve-apparatus to obtain even packing of the beads. This procedure is repeated twice, in between each step 150 ml of glassbeads is added. The last 50 ml is added and used to level the surface at 500 ml. All glasswork used is calibrated with water. Temperature of water and associated changes in volume are taken into account. Porosity of the pellets is calculated as: (True density of the feed mash - Apparent density of the pellets)/True density of the feed mash.

Statistics

After graphical analysis and preliminary curve fitting to the growth-model of Pruitt *et al.* (1979), two empirical non-linear models were chosen to fit the data; the first is a sigmoidal model, comprehensively described by Groot *et al.* (1996), the second model is the two-parameter exponential curve. The sigmoidal model was used to fit the data for pellet durability as a function of PDI. In addition, this equation was used to describe the relation between PDI in the soy grits, Specific Mechanical Energy (kJ kg^{-1}) and feed flux (kg $hour^{-1}$) as a function of PDI.

Model 1 is:

$$Y = \frac{[A+(\Delta A \cdot z)]}{1+\left[\frac{[C+(\Delta C \cdot z)]}{PDI}\right]^{[B+(\Delta B \cdot z)]}} \qquad \text{Model 1.}$$

in which A is the asymptote for the unrealistic case that PDI→∞ and B>0; if B<0 then A is the asymptote for the unrealistic case that PDI→-∞. B and C together determine the curvature and thereby the position of the point of inflexion. B is a steepness parameter determining the speed of transition. For B→∞ a step function is obtained (Groot *et al.* 1996). C is the value of the independent variable where half the asymptotic value of Y is reached. z is a dummy regression variable coded 0 at temperature level 65°C and 1 at 85°C. Thus by incorporating ΔA, ΔB and ΔC, the difference in parameters for the two temperature levels can be estimated together with their (approximate) F-values (Neter *et al.* 1990).

A 2-parameter exponential model was used to fit pellet hardness as a function of PDI (model 2).

$$Y = (A+\Delta A \cdot z) \cdot \exp^{[(B+\Delta B \cdot z) \cdot PDI]} \qquad \text{Model 2}$$

in which A is the intercept at temperature level 65°C and ΔA is the difference in intercept between the two temperature levels at PDI=0. B is the proportionality factor and ΔB the difference in proportionality factor between the two temperature levels. z is a dummy regression variable coded 0 at temperature level 65°C and 1 at 85°C. Thus by incorporating ΔA and ΔB, the difference in parameters for the two temperature levels can be estimated together with their (approximate) F-values (Neter *et al.* 1990). Parameters for model 1 and 2 were estimated using the NLREG program (Sherrod, 1996). Due to the nature of non-linear regression, standard errors for the parameter estimates given in the text for the models 1 and 2 are approximate.

Model 3 was used in two different cases: First, it was used to describe the effect of pellet porosity as a function of PDI, since it follows from literature that porosity is one of the key determinants for the hardness of agglomerates (Rumpf, 1958; Pietsch 1990).

Secondly, model 3 was used to test if differences were present in the protein quality characteristics before and after conditioning and before and after pelleting. The protein quality parameters (PDI or NSIkoh) after the condioner (dependent variable) were regressed on the protein quality parameters before conditioning (independent variable) with temperature as a class variable. Likewise, the protein quality parameters PDI and NSIkoh after pelleting and cooling (dependent variable) were regressed on the protein quality before pelleting (independent variable). If no interaction is present of the conditoning or pelleting step with respect to the denaturation of protein, then the regression of protein quality parameters PDI and NSI_{KOH} before and after conditioning and before and after pelleting would lead to a straight line with regression coefficient one. With use of the 'test'-statement (Proc Reg; SAS, 1990) it was checked whether or not these estimated slopes are one. Still, the PDI or NSIkoh values after conditioning or pelleting can differ from the values before conditioning or pelleting (additive effects), this has been tested by comparing the intercepts at the mean value of the PDI or NSIkoh before conditioning or pelleting with the mean value of PDI or NSIkoh after conditioning or pelleting.

In all cases it was tested whether the slopes of the regression coefficients for the two temperature levels differed. If this is the case than the use of the unit operation does not yield the same values for the protein quality parameters at different temperatures. Hence, an interaction is present between temperature and unit operation for the respective protein quality parameters. A comparison is made between the two temperature levels at the mean value of the independent variable. When no interaction is present between the slopes (β_{1i}=0), the lines have been compared at mean value of the independent variable. All analysis were conducted with the SAS-package (SAS, 1990). Data was fitted to model 3:

$$Y_{ij} = \beta_0 + \text{Temperature}_i + \beta_1*(x_j - \bar{x}) + \beta_{1i}(\text{Temperature}_i*(x_j - \bar{x})) + \text{Error}_{ij} \quad \text{Model 3.}$$

Temperature is used as a class variabele with two levels (65°C and 85°C) β_0 is the intercept at mean value of the independent variable ($\bar{x}$). Temperature_i is the difference in intercept between the two temperature levels at mean value of the independent variable. β_1 yields the slope of the regression line at temperature level i and β_{1i} gives the difference in slopes for the two regression lines.

Results

Temperature of the mash feeds at the entrance of the pellet press was 66.4 °C (std error: 1.21) at the low temperature level and 84.4 °C (std error: 0.87) at the high temperature level. Idle energy consumption of the pellet press was 7.43 kW which was subtracted from the energy consumption values found in the experiment.

Physical quality as a function of PDI.

The sigmoidal model seems to adequately describe, within the experimental range, the relation between Holmen, Pfost, Feed flux and SME as a function of protein dispersibility index, as judged by the pseudo F-tests of the regressions (table 2) and figure 1. In figure 1 the resultant curves for the dependent variables fitted with model 1 are given. No attempt has been made in this study to use transformations of the data derived from the durability tests, since this may lead to relations which are difficult to interpret. Figure 2 gives the resultant curves for the dependent values fitted with model 2.

Holmen durability values were higher at 85°C than at the 65°C (figure 1a). The asymptotic value (the 'A'-parameter) for the dependent variable was not restricted to 100% (the maximum possible durability value). At the 65°C temperature level this lead to an higher estimate than is possible in reality, although the estimate did not significantly differ from 100%. Its value was 125.0% (±30.7 std. error). At the 85°C temperature level the asymptote approximated 100%; 94.5% (±4.6 std. error). The difference in asymptotes (ΔA) between the temperature levels was not significant (Table 2). The steepness parameter ('B') for the Holmen durability test is for both temperature levels well above 1, indicating that a point of inflexion for the curves exists. The PDI value at which half the asymptotic value of the Holmen durability is reached (the 'C'-parameter) differed for the two temperature levels (ΔC: $p<0.001$). At 65°C half the asymptotic Holmen

Table 2: Curve fits for Holmen durability (%), Pfost durability (%), feed flux (kg h^{-1}) and Specific Mechanical Energy (kJ kg^{-1}) as a function of PDI, with associated probabilities according to model 1. Subscripts at parameter values indicate the temperature level to which they apply.

Dependent variable	Asymptote - A			Steepness parameter - B			PDI at half asymptotic value - C			Model characteristics	
	A_{65}	A_{85}	ΔA^a	B_{65}	B_{85}	ΔB^a	C_{65}	C_{85}	ΔC^a	MSE^b	Pseudo F-test of the regression
Holmen durab. (%)	125.0 ***[c]	94.5 ***[c]	-30.5 ns[d]	4.06 ***[c]	2.47 *[c]	-1.59 ns[d]	73.7 ***[c]	18.82 ***[c]	-54.9 ***[d]	18.1	***
Pfost durability (%)	129.6 ***	98.9 ***	-30.7 ns	0.82 **	1.08 ns	0.26 ns	24.5 *	2.06 ns	-22.4 #	1.9	***
Feed flux (kg uur^{-1})	644.4 ***	685.7 ***	41.3 ns	-3.42 *	-0.84 ns	2.58 ns	163.3 ***	1097.4 ns	934.1 ns	97.2	***
SME (kJ kg^{-1})[e]	60.3 ***	50.1 ***	-10.2 *	-2.34 ns	-3.94 *	-1.60 ns	158.5 *	98.1 ***	-60.5 ns	10.2	***

ns = P>0.1; #=P=<0.1; * = P < 0.05; ** = P<0.01; *** = P<0.001.

[a] Calculated as difference of parameter at 65°C level and the parameter at 85°C.

[b] Mean Square Error of the model.

[c] Probability of the parameter being zero.

[d] Probability of the difference in parameters between the two temperature levels is zero.

[e] Specific Mechanical Energy (kJ kg^{-1})

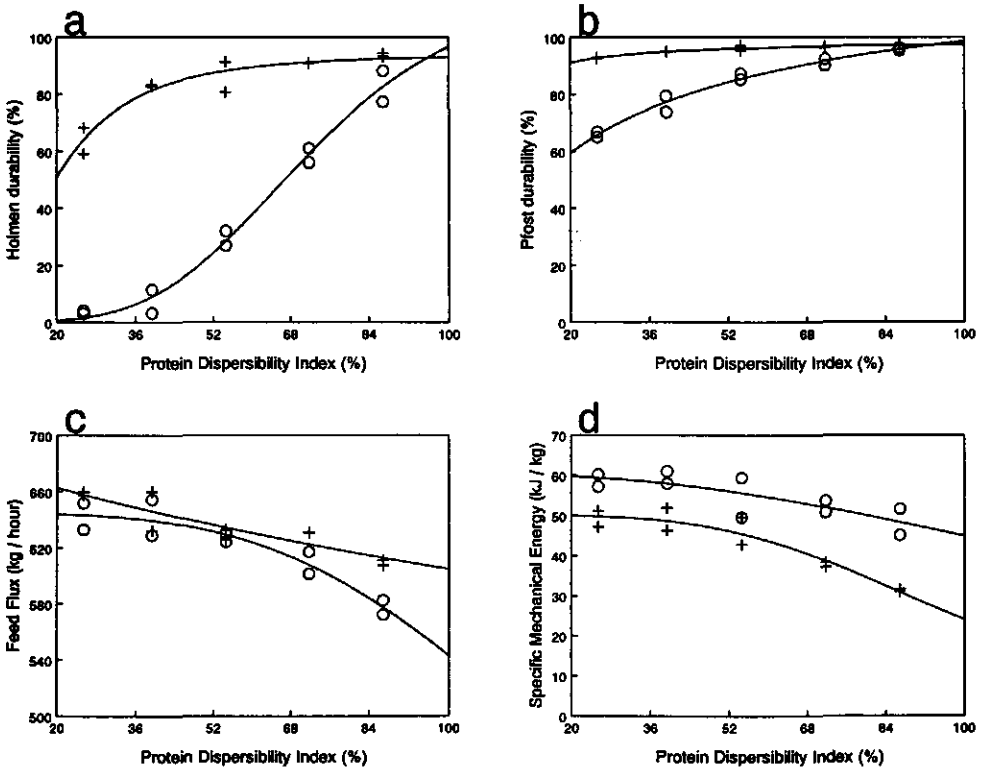

Figure 1a: Holmen durability (%); 1b: Pfost durability (%); 1c: Feed flux (kg h^{-1}); 1d: Specific Mechanical Energy (SME) consumption (kJ kg^{-1}). Curves are calculated as a function of the PDI. Open rounds (o) indicate the 65°C temperature level. 'Plus' (+) indicates the 85°C temperature level. Regressions are calculated according to model 1 (see text).

durability is found at 73.7 % PDI and at the 85°C level, half the asymptotic durability is found at 18.8 % PDI (Table 2).

Pfost durability figures were higher at 85°C than at 65°C (figure 1b). As in the case of Holmen durability the 'A'-parameter for the dependent variable was not restricted to 100% (the maximum possible durability value). At 65°C, this led to an estimation which was above the maximum value possible in this test. The asymptotic value ('A'-parameter) at 65°C was 129.6 % (±21.3 std. error) and not significantly different from 100%. At the 85°C temperature level the asymptote approximated 100%; 98.9% (±5.5 std. error). The difference in asymptotes (ΔA; 30.4%) between the temperature levels was not significant (Table 2). At 65°C the steepness parameter ('B') for the Pfost test is smaller than 1. Hence, no point of inflexion exists. The value at which half the asymptotic strength is reached, trended to be different (ΔC: $p<0.1$) for the two temperature

levels. At the 65°C level, half the asymptotic value is found at 24.5% PDI and at the 85°C level this value is found at 2.1 % PDI (Table 2).

Figure 1c and 1d show the fitted curves of the feed flux and the specific mechanical energy consumption, according to model 1. From the negative sign of the steepness parameter, it follows that an increase in PDI decreases the feed flux and SME. From table 2 it follows that the differences between the parameters in model 1 (ΔA, ΔB and ΔC) for feed flux are not significantly differing from zero. Hence, no difference can be found between the feed fluxes at the two temperature levels used in this experiment. However, the curves show that at high PDI levels a trend seems to appear for the two curves to diverge from one another, with a higher feed flux at a higher temperature level. This is also reflected by the values of the PDI values where half of the asymptotic value of feed flux is reached. Although both values are not realistic ('C' for the 65° is found at PDI is 163% and the 'C'-parameter at the 85°C level is found at PDI is 1097%) they are indicative that such a trend might be present. The lowest feed flux (means of the 4 replicates per treatment) was 594 kg hour^{-1} (std dev. 18.8 kg hour^{-1}), obtained during processing of batch B5 (at 86.9 % PDI). The highest feed flux was 651 kg hour^{-1} (std dev. 12.1 kg hour^{-1}) at the low PDI level (25.4%). The total model gives a significant (asymptotic) F-value indicating that an effect of PDI (or associated with PDI) exists on feed flux. The steepness parameters at both temperature levels were negative, although at 85°C it was not different from zero. Hence, in this experiment is the feed flux decreasing with increasing PDI (see table 2 and figure 1c).

The asymptotic values ('A'-parameter) were different for SME-values at the two temperature levels (Table 2). The value for the asymptote at the 65°C temperature level is 60.3 kJ kg^{-1} (± 3.7), in the unrealistic event that PDI$\rightarrow -\infty$. This values is higher than the 50.1 kJ kg^{-1} (± 2.2) found for the asymptote at 85°C. The highest measured SME found from the data was 59.6 kJ kg^{-1} (±2.1 std dev.) at PDI is 39.4% and 65°C temperature level. At the 85°C temperature level, the highest SME was 49.2 kJ kg^{-1} (±3.9 std dev.) at a PDI-value of 25.4%. The lowest SME-values were measured at PDI 86.9% with values of 48.5 kJ kg^{-1} (±4.6 std dev.) and 31.3 kJ kg^{-1} (±0.4 std dev.) on the 65°C and 85°C temperature level, respectively. The steepness parameters at both temperature levels were negative (although at 65°C it was not different from zero). Hence, in this experiment a decrease in SME is found with increasing PDI (see table 2 and figure 1d). This is also reflected by the values where half the asymptotic SME-values were reached (the 'C'-parameters). These trended to be different for the two temperature levels ($p<0.1$). At 65°C, the (unrealistic) value for 'C' is found at a PDI value of 159%, at 85°C this value is found at the (possible) PDI value of 98.1%. These data and figure 1d show that the SME is higher at the lower temperature level (65°C).

Table 3: Curve fits for Kahl (kgf), Shear- (N g^{-1}) and compression test (N mm^{-1}) hardness. Results as a function of PDI according to model 2. Subscripts at the parameters indicate the temperature level (see text).

Dependent variable	Intercept - A			Proportionality factor - B			Model characteristics	
	A_{65}	A_{85}	ΔA [b]	B_{65}	B_{85}	ΔB [b]	MSE[d]	Pseudo F-test of the regression
Kahl	2.77 ***[a]	7.38 ***[a]	4.61 ***[c]	0.018 ***	0.0140 ***	-0.0042 **[c]	0.52	***
Shear press	82.88 ***	189.38 ***	106.49 ***	0.013 ***	0.0073 ***	-0.0057 *	506.52	***
Compr. test	4.22 ***	7.39 ***	3.16 **	0.011 ***	0.0076 ***	-0.0035 ns	1.47	***

for probability levels see table 2.

[a] Calculated as the difference of the parameter at 65°C level and the parameter at 85°C.

[b] Mean Square Error of the model.

[c] Probability of the parameter being zero.

[d] Probability of the difference between the parameters for the two temperature levels being zero.

The Kahl, Kramer shear test and Compression test (figure 2a, 2b and 2c) showed that the difference in intercept and proportionality factor was different from zero (table 3). In the case of the compression test the difference between the proportionality factors for the two temperature levels was not significant.

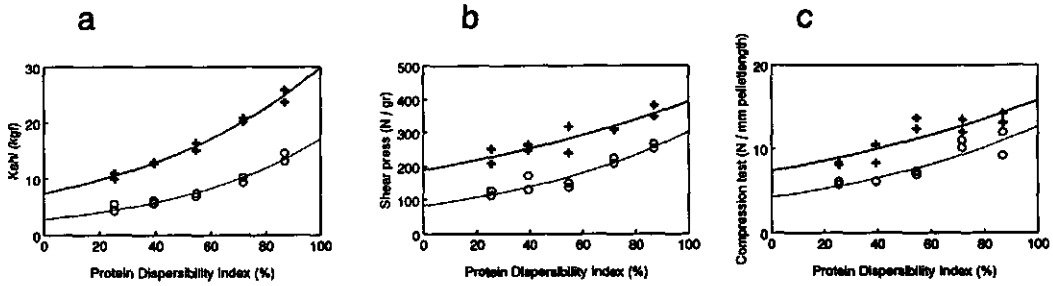

Figure 2a: Kahl hardness (kgf), 2b: Kramer shear press values, 2c: Compression test values. Hardness values are calculated as a function of the PDI. Open rounds (o) indicate the 65°C temperature level. 'Plus' (+) indicates the 85°C temperature level. Regressions are calculated according to model 1 (see text).

The graph of Kahl hardness versus PDI is given in figure 2a. Values from the Kahl hardness test ranged between a measured value of 4.8 kgf (± 0.9) at a PDI level of 25.4 % (B1) and 13.8 kgf (± 1.0) at a PDI value of 86.9 % (B5) on the 65°C temperature level. The range of Kahl values at the 85°C temperature level was between 10.4 kgf (± 0.7) at PDI 25.4 % (B1) and 24.8 kgf (± 1.6) at a 86.9 % PDI (B5). Values in brackets denote the standard deviation of the treatment mean.

The graph of the Kramer shear hardness test versus PDI is given in figure 2b. Values from the Kramer shear test ranged between a measured value of 120.4 N g^{-1} (± 10.1) at a PDI value of 25.4 % (B1) and 260.1 N g^{-1} (± 9.6) at a PDI value of 86.9% (B5) on the 65°C temperature level. The range of values from the Kramer shear test at the high temperature level (85°C) was between 230.5 N g^{-1} (± 30.7) at PDI 25.4% (B1) and 364.8 N g^{-1} (± 23.9) at 86.9 % PDI (B5). Values in brackets denote the standard deviation of the treatment mean.

The graph of the compression test values versus PDI is given in figure 2c. Values for the compression test ranged between a measured value of 5.9 N mm^{-1} (± 0.3) at a PDI level of 25.4% (B1) and 10.6 N mm^{-1} (± 2.0) at a PDI value of 86.9% (B5) on the 65°C temperature level. The range of Compression test values at the high temperature level (85°C) was between 8.22 N mm^{-1} (± 0.2) at PDI 25.4 % (B1) and 13.7 N mm^{-1} (± 0.8) at a 86.9 % PDI (B5). Values in brackets denote the standard deviation of the treatment mean.

PDI and physical characteristics as a function of porosity.

Model 3 was used to determine the relationship between PDI (independent variable) and porosity (dependent variable). The slope of the line at 65°C was -0.00044 and differed from zero ($p<0.01$). The slope at the 85°C line was -0.00026 ($p<0.10$). No interaction between temperature levels existed ($p=0.295$). Intercepts for the two lines at mean PDI (55.6%) were 0.101 at 85°C level and 0.117 at the 65°C level and were different from each other ($p<0.001$) (Figure 3a). Porosity ranged between 0.100 and 0.136 on the 65°C level and between 0.088 and 0.126 on the 85°C level. It is concluded that porosity is lower at the high temperature level and that porosity decreases with increasing PDI.

Table 4 lists the coefficients from the physical quality parameters as a function of porosity. Model 3 was used for fitting of the coefficients. From the slopes of the regression equations it follows that an increase in the porosity of the pellet is associated with lower values for all physical quality parameters. It follows from literature (Rumpf, 1958; Pietsch, 1990) that porosity is one of the properties that affects physical quality of agglomerated materials. Hence, on the basis of literature it seems justified to use porosity as an independent variable. Results for the hardness-tests derived from the Kahl, Kramer shear test and Compression test show that differences in slope between temperature levels were not significantly different (Table 4). Hence, a comparison of the two temperature levels has been conducted at mean porosity 0.1088 (Figures 3d, 3e and 3f). Only in the case of the values derived from the Kramer shear press a difference at mean porosity was found between the two temperature levels (213.7 N g^{-1} at 65°C vs. 270.4 N g^{-1} at 85°C). The difference in slope associated with the Holmen and Pfost test showed that an interaction was present for the two temperature levels, and hence the difference between the two temperature levels as indicated in table 4 is without meaning (Figure 3b and 3c).

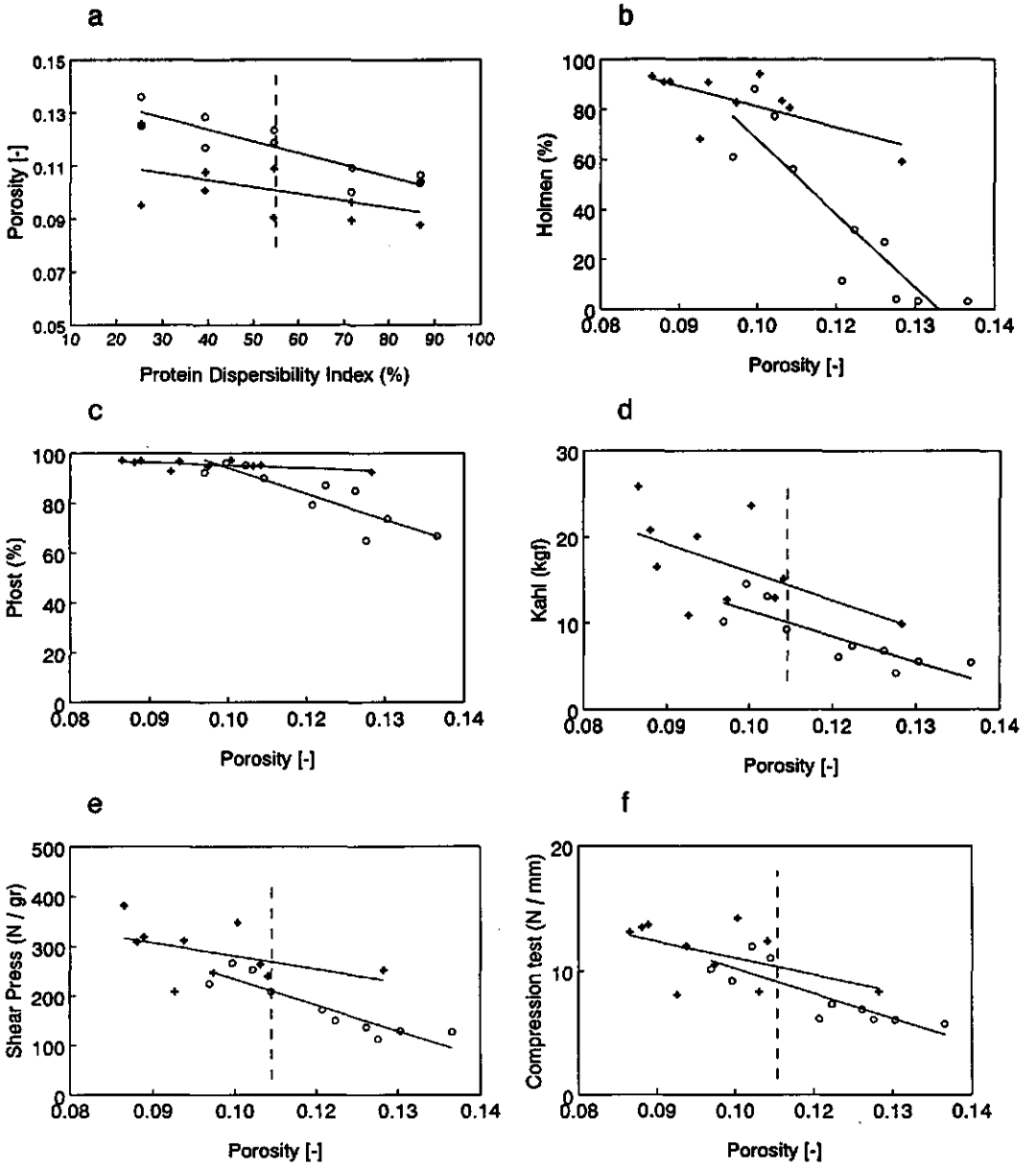

Figure 3a: Porosity as a function of PDI (%) (see text), 3b: Holmen durability (%), 3c: Pfost durability (%), 3d: Kahl hardness (kgf), 3e: Kramer Shear Press values (N g^{-1}), 3f: Compression test values (N mm^{-1}). All panels, except 3a, give hardness and durability values as a function of porosity. Open rounds (o) indicate the 65°C temperature level. 'Plus' (+) indicates the 85°C temperature level. All regression are calculated according to model 3 (see text).

Table 4: Curve fits of pellet hardness and durability as a function of porosity according to model 3.

Dependent Variable	Intercept at mean porosity (0.1088)			Slope at the two temperature levels			Model Characteristics.		
	65° C[a]	85° C[a]	diff. [b]	65° C[a]	85°C[a]	diff. [b]	R^2	MSE[c]	Prob. regression
Holmen (%)	56.2 ***	77.9 ***	21.7 **	-2466 ***	-688 #	1778 **	0.88	157.6	***
Pfost (%)	90.1 ***	94.8 ***	4.7 #	-859 ***	-93 ns	766 ***	0.86	17.8	***
Kahl (kgf)	10.3 ***	14.6 ***	4.4 ns	-247 *	-273 *	26 ns	0.71	13.4	***
Shear (N g^{-1})	213.7 ***	270.4 ***	56.7 *	-4360 **	-2204 #	2156 ns	0.78	1590	***
Compr. test (N g^{-1})	9.4 ***	10.5 ***	1.1 ns	-167 **	-110 *	56 ns	0.67	3.3	***

For probability levels see table 2.

[a] Probability of the parameter being zero.

[b] Probability of the difference between the parameters for the two temperature levels being zero.

[c] Mean Square Error

Differences in protein quality induced by the processing units

Table 5 lists the regression coefficients found after fitting the data to model 3. Figure 4 shows the effect of temperature and sampling place on PDI and NSI_{KOH} values. Regression coefficients of the slopes significantly larger or smaller than one, indicate that an interaction exists between temperature (levels) and the processing apparatus. A significant difference between the slopes of the temperature levels indicates an interaction between the two temperatue levels. In case of additivity of the slopes, the intercepts at the means of the independent variables have been compared. All the regression equations were significant at the $p<0.001$ level.

PDI changes during conditioning. At 65°C the regression coefficient did not differ from 1. The mean value after conditioning (52.0 %) was lower ($p<0.05$, table 5) than the mean PDI value (55.6%) before conditioning. Thus, at 65°C and in the range covered in this study, PDI decreases with (55.6-52.0) 3.6% units. At 85°C the regression coefficient differed from 1, hence, an interaction exists between the 85°C level and the conditioner. The range of differences in PDI values found was between 3.4% units and 19.2% units, with high PDI inducing the largest decrease. Furthermore, an interaction was present between the temperature levels ($\beta_{1i} = 0.23$: $p<0.001$). At high PDI, before conditioning, the difference in PDI between the temperature levels after conditioning was higher than at low PDI values before conditioning (see figure 4a).

PDI changes during pelleting. An interaction was present between the pelletizer and the 65°C temperature level. An interaction was also present between the 85°C temperature level and the pelletizer. The difference between the two slopes was not significant ($\beta_{1i} = 0.04$: $p>0.1$; Table 5). Therefore, a comparison between the intercepts at mean PDI is possible. The comparison of the intercepts at mean PDI after pelleting showed a tendency to a difference between the intercepts with 2.8% PDI units (46.6 vs 43.9: $p<0.1$). The PDI value after pelleting at the 65°C level trended to be different ($p<0.1$) from the mean PDI value (48.9%) before pelleting. The PDI value after pelleting at the 85°C level differed from the mean PDI value (48.9%) before pelleting (see figure 4b).

NSI_{KOH} changes during conditioning. For both temperature levels, the regression coefficient was smaller than 1 ($p<0.1$), indicating a trend to interaction between conditioning and temperature. No interaction was present between the temperature levels ($\beta_{1i} = 0.02$: $p>0.1$; Table 5). A comparison of the differences at mean NSI_{KOH} for the two temperature levels after conditioning showed that these values (93.3% at 65°C and 92.1% at 85°C) were different ($p<0.01$). A comparison of the mean NSIkoh values after conditioning with the mean value of NSIkoh before conditioning (91.8%) showed that NSIkoh increased. This increase was significant for the 65°C level (93.3% vs 91.8%) but did not differ for the 85°C level (92.1% vs 91.8%). See figure 4c.

Table 5: Curve fits of PDI and NSI_{KOH} after conditioning or pelleting, as a function of PDI and NSI_{KOH} before conditioning or pelleting. Slopes and LSmeans according to model 3.

Dependent Variable	Intercept at means ($\bar{x}$) [a]			Slope at the two temperature levels			Model Characteristics.		
	65°C [a]	85°C [a]	diff. [ab]	65°C [c]	85°C [c]	diff [ab]	R^2	MSE	Prob. regression
PDI after conditioner [e]	52.0 ***	45.7 ***	6.4 ***	1.01 ns [d]	0.78 *** [d]	0.23 ***	0.99	4.76	***
PDI after pelleting [f]	46.6 ***	43.9 ***	2.8 #	0.88 * [d]	0.84 * [d]	0.04 ns	0.97	11.23	***
NSI_{KOH} after conditioner [g]	93.3 ***	92.1 ***	1.2 **	0.88 # [d]	0.86 # [d]	0.02 ns	0.96	0.74	***
NSI_{KOH} after pelleting [h]	95.0 ***	94.9 ***	0.1 ns	0.80 # [d]	0.69 ** [d]	0.11 ns	0.88	1.29	***

For probability levels see table 2.

[a] Probability indicated is the probability of the Lsmean or parameter differing from zero (H_0: parameter=0).

[b] *diff. is the difference between the Lsmean or slope at the two temperature levels.*

[c] All slopes were different from zero at P<0.001 level.

[d] Probability indicated is the probability of the parameter differing from one (H_0: parameter=1)

[e] Mean PDI before conditioning was 55.6 %.

[f] Mean PDI before pelleting was 48.9 %.

[g] Mean NSI_{KOH} before conditioning was 91.8 %.

[h] Mean NSI_{KOH} before pelleting was 92.7 %.

NSI_{KOH} changes during pelleting. At 65°C temperature level the regression coefficient trended to be smaller than 1, indicating a trend towards interaction between this temperature level and the pelletizer. At the 85°C temperature level the regression coefficient was smaller than 1, indicating that an interaction existed between this temperature level and the pelletizer. No interaction was present between the temperature levels (β_{li} = 0.011: p>0.1; Table 5). Mean NSI_{KOH} values after pelleting were not different (95.0 % at 65°C and 94.9 % at 85°C). A comparison of the mean NSIkoh values after pelleting with the mean value of NSIkoh before pelleting (92.7%) showed that NSIkoh increased. This increase was significant (p<0.001) for both the 65°C level (95.0% vs 92.7%) and the 85°C level (94.9% vs 92.7%). See figure 4d.

It should be noted that the measurements on protein quality after the pelletizer involved the cooling step as well. This may obscure direct effects of the pellet press, if an effect of the cooler is present. This cannot be elucidated from this study.

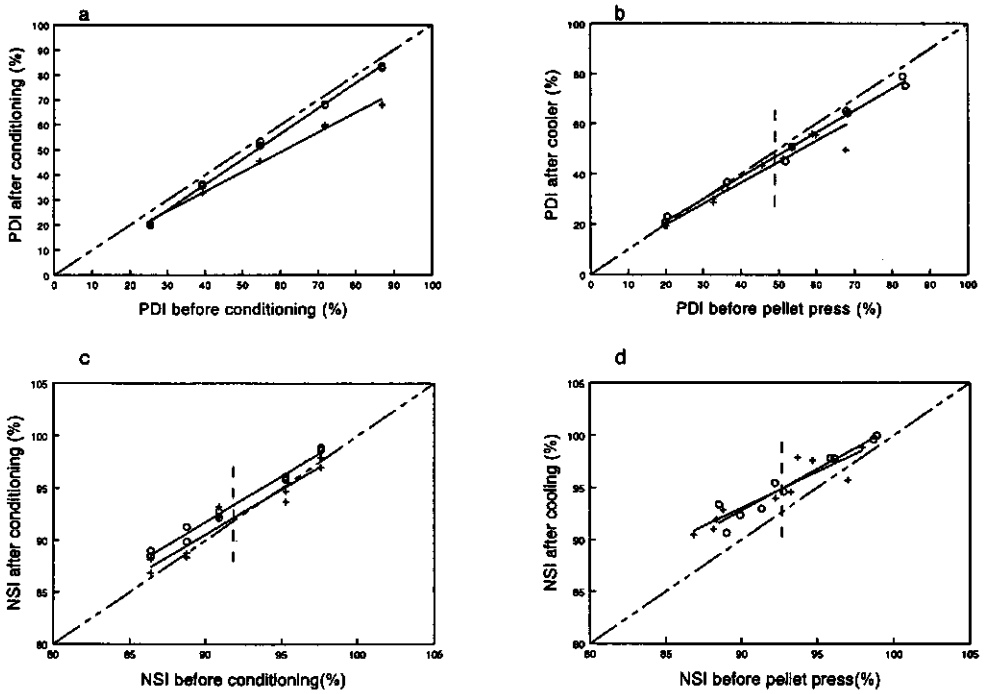

Figure 4a: Protein Dispersibility Index (PDI; %) after conditioning as a function of PDI before conditioning treatment. 4b: PDI after the cooler as a function of the PDI before pelleting. 4c: NSI_{KOH} after conditioning as a function of NSI_{KOH} before the conditioning treatment. 4d: NSI_{KOH} after the cooler as a function of NSI_{KOH} before pelleting. Open rounds (o) indicate the 65°C temperature level. 'Plus' (+) indicates the 85°C temperature level. Dashed lines in the figures indicate where the two temperature levels have been compared. Dash-dotted line is the line y=x.

Discussion

The choice of models 1 and 2 is based on preliminary regression analysis with the data in this study, according to the model proposed by Pruitt *et al.* (1979). This model incorporates a large number of growth functions. Based on interpretation of the two 'shape'-parameters present in this model, sigmoïdal and exponential curves were chosen as the most optimal candidates for fitting the data. The eventual choice for the sigmoïdal model after Groot *et al.* (1996), stems from the fact that it is more flexible than the sigmoïdal model in the model of Pruitt *et al.* (1979) while its parameters remain interpretable. Although many growth models are derived based upon assumptions of the underlying process, the use of PDI and NSI_{KOH} as independent variables cannot be judged as fundamental measures of the quality of the protein. However, since they are widely used as descriptors for the processing history of the protein in a feed commodity, its use as such in this experiment seems justified (Hermansson, 1979; Horan, 1974). The proposed models 1 and 2 are purely used as an empirical description of the data. Especially, since in model 1, parameters appear which only have a meaning for the inpendent variable going to either plus or minus infinity, which in not case for the protein quality test used ($0 \leq PDI \leq 100$). In addition, durability as a dependent variable is also limited in the range from 0% to 100%. Hence, the models should only be used for interpolation within the boundaries of the tests used. Further investigation into suitable models is encouraged on a more fundamental level with respect to the relation between the changes in protein quality (and/or associated effects) on hardness and durability parameters of pelleted animal feeds.

Physical quality as a function of PDI.

The results in this study show that an increase in the PDI of soy-grits leads to harder and more durable pellets. In addition, at conditioning temperatures of 85°C these pellets are harder and more durable than pellets produced at a conditioning temperature of 65°C. The durability tests show that sigmoïdal curves, or curves with a 'diminishing return' shape (in this study the upper part of a sigmoïdal curve) are obtained with increasing PDI in the soy-grits. The various hardness tests show curves with an exponential increase for the hardness values with increasing PDI.

Results of the different test methods to determine hardness and durability are generally not comparable, since differences exist in geometry of the measuring equipment, application of load and loading rate. The results from the hardness tests showed however, that in all cases, hardness values were higher at the 85°C level than at the 65°C level. Higher temperatures may lead to softening of food or feed commodities (Rao and Lund, 1986) and most likely this occured with

the soy-grits in this experiment as well. The lower porosity at the higher temperature level and the lower amount of SME consumed on the high temperature level may, therefore, be indicative for a higher deformability of the soy-grits.

Differences in feed flux were shown not to be attributable to differences in the temperature level. In fact, differences in feed flux were not expected, since the setting of the flow regulator was not altered. Therefore, differences found in feed flux must be related to properties of the different batches. Flow of particular materials is amongst others affected by surface roughness, geometrical properties and stress strain properties of the particles (Barbosa Canovas *et al.*, 1987; van der Kraan, 1996). Sheard *et al.* (1986) found, as in this experiment, a negative correlation between the protein solubility and the flow rate during extrusion. They attributed this effect to the higher agglomeration tendency of material containing more soluble protein. In addition, this reduction in feed flux through the pelleting line will lead to a longer hold-up time in the different processing units. This prolonged hold-up time may lead to a higher amount of solubilised protein, which can act as binder material. Since feed flux decreased with an increase in PDI in the batches, and feed flux was lower at the 85°C level, more time has been available for the protein to solubilise. This may then offer an explanation for the observed interactions found at the high temperature level for the decrease in PDI during conditioning and the interactions between both temperature levels during pelleting-cooling. Furthermore, a longer hold-up in the die of the pelletizer may lead to a larger stress decay in the material when pelleted. In this case, the lower resilience of the material after ejection from the die, would induce a lower porosity and hence a harder and more durable pellet (Mohsenin and Zaske, 1976; Hiestand *et al.* 1977; Sihag *et al.* 1991). Indeed, from figure 3a it can be observed that porosity decreases with an increase in PDI and that porosity is lower at the higher temperature level. The effect of a decrease in energy consumption of the pellet press with increasing temperatures of the conditioned feed mash has also been reported in literature (Skoch *et al.* 1983a; 1983b). The exact relation between heating by steam and decrease in power consumption during pelleting remains unclear. It is suggested that the condensed water acts as a lubricant during extrusion in the die (Skoch *et al.* 1983a, 1983b). It cannot be concluded from this study if solubilisation of the protein or deformability of the particles is the most important factor. If they are both causative factors in determining the hardness and durability of feed, then the synergistic effect of these two factors may have caused the exponential increase in pellet hardness with increasing PDI as observed from figure 2a, 2b and 2c.

Hardness and durability versus porosity.

Literature (Rumpf, 1958; Pietsch, 1990) shows that strength of agglomerates is amongst others affected by porosity. Additional properties are the amount of binder at a contacting point between the particles, the type of bond that can occur (e.g. liquid necking or recrystallisation) and properties associated with the particle itself, like geometry, size, elasticity and plasticity. For this reason the physical quality parameters have been regressed on porosity. Slopes of the regression line(s) close to the upper or lower boundary, of the durability tests (0% or 100%), will be strongly influenced by these boundaries. Hence, an interaction between the two temperature levels for the durability tests is found which arises as an artefact of the representation of the data, since durability is limited in the range 0% - 100%.

Differences in protein quality induced by the processing units

Although a linear model was fitted to the data on protein quality before and after a certain processing unit, analysis of the residuals learned that non-linear trends were present. These were not always consistent with respect to direction and magnitude. A variety of non-linear models was employed, however, none of the models gave consistent results. Hence, for simplicity reasons a linear model (model 3) was chosen.

According to Visser and Thomas (1987), high solubility data can sometimes be obtained for completely denatured proteins. A completely denatured protein may still exhibit good functional properties in terms of fat- and water binding capacity. In this study, as judged by both PDI and NSI_{KOH} values it is unlikely that the protein has been denatured completely. Therefore, a large amount of these functionalities may still exist (Hermansson, 1979).

No explanation is found is in this study for the fact that the NSIkoh values become higher after the different processing steps. Under the alkaline conditions in the 0.2% KOH solution (pH ~ 12.5), solubility of proteins generally increases (Hermansson, 1979). This may be the reason that NSIkoh values found in this experiment are higher than PDI values of the same samples. However, this can not be the explanation for the higher values found after the different processing steps (see figure 4c and 4d), since PDI values decrease as a function of processing and the NSIkoh values increase. Although NSIkoh values are not expected to decrease, due to the low SME-values of the pelletizer (Marsman *et al.* 1995; Thomas *et al.* 1997a), it may be the case that due to the processing steps, the particles become more accesible for the solvent and therefore lead to increased solubilities. Such an hypothesis remains to be investigated.

The results derived from this study, confirm the conclusions of Wood (1987) that raw (high PDI) protein contributes to pellet hardness and durability as compared to denatured (low PDI) soy-protein. Further work with practical feed rations instead of model feeds, remains necessary to determine the extent of which functional properties of feed materials can be used to optimise the pellet quality to manufacturing standards.

Conclusions

From the present study the following conclusions are drawn:

1) An increase in protein dispersibility index (PDI) in soy-grits leads to an increase in pellet hardness and durability.
2) The relation between PDI and hardness and durability of pellets is curvi-linear.
3) A temperature of 85°C after conditioning leads to harder and more durable pellets than a temperature of 65°C.
4) Increasing PDI values and a temperature of 85°C compared to 65°C leads to lower porosity in pelleted feeds.
5) With respect to the protein quality parameters PDI and NSI_{KOH}, interactions exist between these protein quality tests before conditioning and after conditioning and before pelleting-cooling and after pelleting-cooling, which are most likely attributable to changes in the feed flux in the processing-line and hence changes in hold-up time in the various parts of the unit-operations.
6) The cause, most likely responsible for the exponential increase in pellet hardness found in this experiment is due to the synergistic effect of an increase in soluble protein and higher deformability of the particles due to the processing conditions in this study.

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8

Protein quality of soy-grits has a larger impact on pellet hardness and durability than degree of starch gelatinization.

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Abstract

A feed model system (50% soy-grits / 50% tapioca w/w) was used to determine the effect of protein quality and starch degree of gelatinization on the physical quality of manufactured pellets in terms of hardness and durability. Protein quality was determined by the protein dispersibility index (PDI) and differences in the degree of starch gelatinization were determined from the enzymatic amyloglucosidase test (SGDags). In addition, correlations were calculated for additional tests as differential scanning calorimetry (DSC), and nitrogen solubility in 0.2% KOH (NSIkoh) for the starch/protein and protein properties, respectively. It was hypothesized that a high amount of dispersible protein and a high amount of gelatinized starch positively affects hardness and durability properties of pelleted feeds. A number of physical quality tests as used in the feed industry (Holmen and Pfost durability and Kahl hardness) as well as some other testing devices (Kramer shear press and a general compression and tension tester) were used to determine hardness and durability values of the manufactured pellets. Results were analyzed using response surface methodology. In addition, an apparent modulus was calculated from the values derived from the compression test. Percentage strain before fracture of the pellets was calculated from the values of the compression test.

SGDags and DSC values for the starch properties were negative and highly correlated (<-0.80). Correlations between PDI and NSIkoh were high (>0.87; $p<0.001$) but correlations between these protein quality parameters and DSC were low (<0.35; $p>0.05$).

It was concluded that the apparent modulus of the tested pellets was related to hardness properties of the pellets and fracture strain was related to friability properties of the pellets. A maximum was found within the limits of the experimental conditions for the Pfost and Holmen durability and fracture strain values. Porosity, fracture strain and Holmen and Pfost durability were most sensitive to changes in PDI values of the feed mash, whereas specific mechanical energy (kJ kg^{-1}) of the pelletizer and capacity of the installation was most sensitive to changes in SGDags values. In all cases, the hardest pellets, and highest apparent modulus, was found at the maximum inclusion level of native protein (high PDI) and gelatinized starch (high SGDags). Minimum values, within the experimental limits, were found for porosity and Specific Mechanical Energy values. It is concluded from this study that no interactions are present between PDI and SGDags.

This study shows that large effects can be expected depending on the state of the protein and starch fractions in the raw materials used.

Introduction

In modern animal farming, a substantial amount of feed is given in the form of concentrates. Although a vast quantity of feed is given in the form of mash, the largest amount of concentrate feed is pelleted (>80% in the Netherlands) and thus fed to animals. Mash feeds in itself can be already complete feeds, made by milling and blending the raw materials and other ingredients like vitamins and minerals into nutritionally balanced diets. However, in this form the feed is prone to segregation, which may lead to nutritional imbalance in the feeding

trough. To overcome this problem, feeds are agglomerated into discrete rods by means of a pressure agglomeration process. Advantages of using an agglomerated product over mash feeds are the better flow properties, reduced dustiness and the larger bulk density that can be reached (Pietsch, 1991). Further advantages may be found in the improved nutritional properties of the diets leading to better growth and feed conversion figures.

There may be definite conflicts between physical, hygienic and nutritional quality of pelleted animal feed. If pelleting, irrespective the type of conditioning, results in very hard or low durable pellets, feed intake by livestock may be reduced (Skoch *et al.*, 1983) and even utilization of nutrients may be reduced due to undesired chemical reactions (Voragen *et al.*, 1995). In this study, emphasis is directed towards the relation of the raw material properties and its relation with the physical quality of pelleted feed. Numerous literature has been published on the effects of the different processing conditions during manufacture of the pelleted feed and its effect on the hardness and durability values of pelleted feeds. A recent overview on these processing conditions and the relation with pellet quality has been given by Thomas *et al.* (1997). From this overview it follows that, although rules of thumb exist on the effects of the different processing conditions, the ability to predict a certain pellet quality is still low. This is mainly due to the large variability in raw materials and its functional properties which contribute to the ultimate physical quality of the pelleted feeds (Friedrich and Robohm, 1981; Lake, 1991; Wilson, 1994).

This study was designed to investigate the changes in protein quality as measured by PDI and degree of starch gelatinization (SGDags) in model feeds and its effect on physical quality characteristics. The choice was made for a two-way feed model system (50% soy-grits / 50% tapioca w/w) in order to be able to avoid as much as possible interactions with other feed ingredients (e.g. fibre, fat) normally encountered in animal feeds. It is hypothesized that an increase in the amount of raw protein present (high PDI) and an increase in the amount of pre-gelatinized starch present in the feed mash will lead to harder and more durable pellets.

Materials and Methods

Raw materials were obtained from commercial suppliers. 5000 kg of tapioca was obtained from the Cehave, Veghel, The Netherlands and two batches, 2300 kg each, of soy-grits characterised by a high (PDI-80) and low Protein Dispersibility Index (PDI-20) were obtained from Cargill b.v., Amsterdam, The Netherlands. The initial design of the experiment consisted of the 16 feed mixtures, derived from soy-grits and tapioca in different inclusion levels. Two replications for each treatment were used. The treatments 17 and 20 were later added to this design to obtain a better estimate of the response surface (Table 1).

Table 1: Composition of the mixtures used in the pelleting trial.

Pelleting mixture[a]	Tapioca		Soya	
	Pre-processed (%).	Native (%).	PDI 20 (%).	PDI 80 (%).
1	0	50	50	0
2	12.5	37.5	50	0
3	25	25	50	0
4	37.5	12.5	50	0
5	50	0	50	0
6	0	50	0	50
7	12.5	37.5	0	50
8	25	25	0	50
9	37.5	12.5	0	50
10	50	0	0	50
11	0	50	37.5	12.5
12	0	50	25	25
13	0	50	12.5	37.5
14	50	0	37.5	12.5
15	50	0	25	25
16	50	0	12.5	37.5
17	16.7	33.3	16.7	33.3
18	33.3	16.7	16.7	33.3
19	16.7	33.3	33.3	16.7
20	33.3	16.7	33.3	16.7

[a] Pelleting runs 1 to 16 were done in duplicate, treatmens 17 to 20 were done once.

Pre-processing

Half of the tapioca was pre-processed to gelatinize the starch fraction of the tapioca meal as much as possible. The other half used was used and referred to as the native tapioca. Pre-gelatinized tapioca-starch was obtained by expander processing (AL150, Almex BV, Zutphen, The Netherlands) and subsequent re-milling to approach the mean particle size of the native tapioca. Expander processing was done with the facilities of the Wageningen Feed Processing Centre, Wageningen, The Netherlands. The expander processed tapioca was subsequently cooled in a Robinson counter-flow bunker cooler with two decks for 8 min 30 sec. Subsequent re-milling was done with a roller mill (Weiherhammer) with two rollers using various settings of the distance between the rollers and multiple passes of the tapioca.

The raw materials were mixed in a cone mixer according to the proportions given in table 1. Tap water was added during mixing to obtain an equal moisture content in all the batches of 98.9 gr tap water kg^{-1} material, except in the treatments 17 to 20 where the moisture contents was 102.7 gr tap water kg^{-1} material.

Pelleting trial

Thirty-six pelleting runs were carried out (Table 1; treatments 1 to 16 in duplicate). Prior to pelleting, steam was added in a conventional barrel type conditoner to obtain a conditioning temperature of 70°C. Subsequently, the material was conveyed through the expander section and pelleted in a Simon Heessen V3-30 pellet press with two rollers. Die dimensions were 5*45 mm and a warm die was used before each run.The pellets were cooled in a Robinson counter flow bunker cooler with an eight and a halve minutes residence time. During processing, temperatures of the mash entering the pellet press and pellet temperature of the pellets leaving the die, were measured using the thermosflask method. Energy consumption of the pellet press was calculated from the sheets that were used to record the data during each run. The μ-mac 6000 process control and data logging computer was used to monitor the process during experimentation. Reported energy consumption values of the pellet press are corrected for idle running. Samples were taken after conditioning (conditioned meal) and after the cooler (pellets). Samples taken after the conditioner were air-dried at 50°C for 16 hours.

Samples for PDI and NSIkoh were milled in two steps in a Retsch ZM1 centrifugal mill. First, with a 3 mm screen and subsequently with a 1 mm screen. The material was kept in plastic bottles and stored at 4°C until further analysis. Samples were analysed for dry matter by oven drying during 4 hours at 103°C. Pellet samples were taken after the cooling and stored at 4°C until physical quality tests were done.

Chemical analysis

Protein Dispersibility Index (PDI) was determined according to a modified AACC 46 - 24 procedure. One-hundred (100) ml of distilled water was brought in a Waring blendercup, 20 ± 0.1 gr of (un)processed soy grits were added. After stirring, 200 ml of aqua dest. was added with which the stirring rod was cleaned. The cup was then placed on the blender and the cooling and the motor were mounted. Temperature was held constant at 25°C, speed of the mixer was held constant at 8500 rpm. After 10 min. the suspension was poured in a beaker. After sedimentation the upper layer was poured into a 80 ml centrifuge tube and centrifuged (r.c.f. = 1400 g) for 10 minutes. N-content was determined according to standard Kjeldahl procedure. The PDI (%) was then expressed as: (N in supernatant [g kg^{-1}]) / (N of the sample [g kg^{-1}]) * 100.

Nitrogen Solubility Index (NSI) was determined according to a modified procedure of the AOCS. Five gr (± 0.01 gr) of sample was weighed and put in a 400 ml beaker. Two-hundred (200) ml of a 0.2% KOH solution (30°C) was added and the mixture was stirred with a magnetic stirrer for 90 minutes, at such a speed that no air was incorporated and the sample

did not settle. After stirring, the slurry was quantitatively put in a measuring flask of 250 ml. Distilled water was added up to 250 ml. The slurry was allowed to stand for a few minutes until the coarse material had settled. Forty (40) ml of the liquid was decanted into a centrifuge tube and centrifuged for 10 minutes at 340 g. The supernatant was filtered over a Schleicher & Schuell folded filter 595½, ϕ150 mm. Twenty-five (25) ml of the filtraat was used for the determination of crude protein according to standard Kjeldahl procedure. NSI (%) was expressed as: (N-content of supernatant [g kg^{-1}]) / (N-content of sample [g kg^{-1}]) x 100.

SGDags was determined in three steps according to the NIKO method (Brunt, 1992). Total starch was analysed by extracting the lower sugars with a 40% ethanol solution, followed by autoclaving for 3 h at 130°C and enzymatic breakdown (1 h at 60°C, pH 5) to glucose, using an enzyme cocktail containing amyloglucosidase, α-amylase and pullulanase (A). Glucose was subsequently determined using hexokinase and G6P-dehydrogenase. For the determination of the degree of gelatinization of starch, two additional analysis were conducted: starch was analysed as above, but without the ethanol extraction (B) to quantify the amount of starch and lower sugars. Finally, the sample was hydrolysed with amyloglucosidase (60 U g^{-1} sample) for 75 min at 50°C (pH 4.8) to determine gelatinized starch and lower sugars (C). The SGDags was calculated as a percentage of total starch after correcting for lower sugars according to: SGDags = 100 * [C-(B-A))]/A.

DSC-measurements were conducted with a Mettler Toledo TA12E heat-flow calorimeter. Between 15 and 20 mg of homogenized sample was weighed into a medium pressure crucible (Mettler-Toledo). Approximately 60 mg of demineralised water was added. The crucibles were sealed and left to equilibrate for one hour at room temperature to allow water to fully hydrate the sample. The sample was then subjected to a controlled temperature program in which the sample was held isothermal for 5 minutes at 20°C and then subjected to a linear temperature rise at a rate of 5°K min^{-1}. The measured range was from 20°C to 120°C. A crucible filled with 90 mg of aluminum foil was used as a reference. From the thermograms, the residual enthalpy associated with the starch fraction and the residual enthalpy associated with the protein fraction was determined. Peak areas where determined by taking the peak associated with starch gelatinization of tapioca starch in abundant water, around 71°C (Wolters and Cone, 1992) or inbetween 66°C and 72°C (Cooke and Gidley, 1992). For the protein fraction, the denaturation temperature of β-conglycin in soya was taken to start around 80-88.8°C and for glycinin around 100-104.2°C (Sheard *et al.*, 1986; Wright and Boulter, 1980). The residual enthalpy determined was a measure for the amount of ungelatinized (native) starch or not denatured (soluble) protein present in the sample. Values given are in J g^{-1} dry matter content of the sample.

Physical quality tests

Physical quality determinations of pellets comprised the following tests for hardness: Kahl, a general compression and tension apparatus and the Kramer shear press (Anonymous, 1970). Durability tests were conducted using the Pfost and Holmen test apparatus. Kahl hardness was tested by inserting a pellet in the device and the force necessary to crush the pellet was recorded. Reported values are means of 10 measurements.

Crushing strength (fracture stress) was tested in a general compression and tension apparatus (Overload Dynamics) by crushing the pellet between two flat platens. The speed of the moving plate was fixed at 10 mm min^{-1}. A pair of vernier calipers is used to determine the length of each pellet. Reported values are an average of 10 measurements. Results are reported as maximum force necessary to crush the pellets per mm pellet length. From the traces obtained with the compression and tension tester, the amount of strain up until fracture of the pellet, was calculated. Fracture strain is reported as percentage of deformation of the initial diameter (which was 5 mm for all pellets) according to (deformation [mm] / pellet diameter (5 mm))*100. In addition, an apparent modulus was calculated from the data generated by the compression tester as follows: apparent modulus = $(MaxForce/(\pi r^2))/\epsilon$. MaxForce is the mean force for pellet fracture per treatment, r=2.5E-3 m and is assumed constant. ϵ=the relative deformation (-) of the pellet at fracture (initial pellet diameter is fixed at 5E-3m).

Tests made with the Kramer shear press were done as follows: approximately 10 gram of pellets (4 pellets) was inserted in the shear box, half of the pellets in axial direction and the other half in radial direction to the grid of the shear box. The shear press was modified to obtain shearing forces in Newtons instead of pounds. The force neccesary to shear the pellets was recorded and maximum force necessary was calculated as N g^{-1} pellet material used. Durability tests were done according to the description in Thomas *et al.* (1996). In the Holmen test, the circulation time was 1 minute. A 5 mm sieve was used. Reported values are means of 3 measurements. In the Pfost durability test a 3 mm sieve was used. Reported values are means of duplo measurements. Details are described in Thomas *et al.* (1996).

For porosity determinations, true density of the model feed mashes was determined with a pycnometer using alcohol as immersing fluid. Apparent density of the pellets was determined using a 500 ml measuring cylinder and glassbeads (Fisher, 560-800 μm). The measuring cylinder was filled to 150 ml with glassbeads and vibrated two times for 10 seconds on the plateau of a sieve-apparatus to obtain even packing of the beads. This procedure was repeated twice, in between each step 150 ml of glassbeads was added. The last 50 ml was added and used to level the surface at 500 ml. All glasswork used was calibrated with water and

corrected for temperature. Porosity of the pellets was calculated as: (True density of the feed mash - Apparent density of the pellets)/True density of the feed mash.

Statistical analysis

Correlations are calculated between DSC, NSIkoh, SGDags and PDI values for the samples obtained after the different processing steps. PDI, NSIkoh and DSC correlations are given for protein properties and correlations between DSC and SGDags values are given for starch properties. The variables Holmen, Pfost, Kahl, Kramer shear press, compression test, apparent modulus, fracture strain and porosity of the pellets were fitted to the full quadratic polynomial. In addition, the system parameters: specific mechanical energy (SME: kJ kg^{-1}) and capacity (kg $hour^{-1}$) were fitted to the same equation. PDI and SGDags in the feed mash were used as the independent variables.

$$Y_i = \beta_0 + \beta_1*PDI + \beta_2*SGDags + \beta_3*PDI^2 + \beta_4*SGDags^2 + \beta_5*PDI*SGDags + error_i$$

With Y_i the various dependent variables, i the number of experimental units: i=1..36, PDI the protein dispersibility index of the model feeds and SGDags the degree of gelatinization of the model feeds. The design of the experiment is given in table 1. A lack of fit test was conducted on the basis of the experiment-wise error obtained from duplicate treatments. In addition, the second order fitted regression equation can be rewritten in a way that the nature of the stationary point and the entire response system becomes more clear. It involves a translation from the origin of the axis to the stationary point and a rotation of the axis system into the direction of principal orientation of the response surface. This modified form is called the canonical form of the equation. Where appropriate, the canonical form of the equation was used to determine the factor with the largest impact on the dependent variables (Meyers, 1976). All analysis were conducted using various procedures of the SAS-system (1990).

Results

Pre-processing

Processing conditions during the expander treatment to pre-gelatinize the tapioca starch were a steam pressure of 180 kPa, water addition of 4.14 ltr h^{-1} with a feed flux of 525 kg h^{-1} and a net power consumption during expander processing of approximately 45 kJ kg^{-1} feed. Temperature of the expandate was 112°C, as determined from a thermocouple fitted in the last mixing bolt before the annular gap. The temperature after expander processing was 92°C (thermos-flask method). After the pre-processing step, the SGDags of the starch fraction was

58.8 % (Table 2).

Table 2: Chemical composition and some physical characteristics of the used soya and tapioca before blending.

Component	Tapioca		Soya	
	Native	Pre-processed	PDI 20	PDI 80
Moisture [g kg^{-1}] before blending	121.2	131.0	60.3	64.3
Crude Fiber [g kg^{-1}]	30.5	33.8	47.1	40.9
Crude Fat [g kg^{-1}]	5.2	3.7	11.0	8.3
Anorganic Matter [g kg^{-1}]	34.4	35.0	67.8	67.6
Crude Protein (N*6.25) [g kg^{-1}]	34.6	31.6	528.8	534.8
Starch [g kg^{-1}]	779.9	778.3	27.3	26.8
Protein Dispersibility Index [%]	-	-	25.9	85.1
Nitrogen Solubility Index [%]	-	-	87.6	98.0
Degree of gelatinization [%]	9.4	58.8	-	-
Enthalpy [J g^{-1}][a]	9.49	4.29	3.33	3.52
Mean particle size ($x_{50;\ \mu m}$)	202	334	516	472

Composition in g kg^{-1} dry matter except moisture, which is in g kg^{-1} material before blending.

[a] Enthalpy values based on, at least, three replicates.

Correlations between DSC-values, NSIkoh, PDI and SGDags.

Correlations between the various dependent variables are given in table 3 for the starch fraction and in table 4 for the protein fraction of the samples. Correlations between the starch and protein fractions were also calculated and most of them where found non-significant (not shown). Two measurements for the DSC-values, one replicate from treatment 12 and one replicate from treatment 14 were excluded from the correlation analysis after plots had shown that these points were highly influential and would lead to erroneous results.

In the samples obtained from the mixtures only the peak of the glycinin (11S) fraction (around 100°C) is used in the calculations, since in the samples of the soya/tapioca mixtures, part (or all) of the curve for the β-conglycin (7S) fraction is obscured by the larger enthalpic values of the starch fraction (see figure 1). Correlations between the DSC-values of the starch fraction and SGDags were high with p-values smaller than 0.001 (Table 3). Correlations between these tests were inversily related and higher than 0.77. High SGDags values will lead to low DSC-values and vice versa, hence the inverse relation. Correlations within SGDags and

Table 4: Pearsons correlation coefficients between PDI, NSIkoh and DSC values for the protein fraction.

	Protein							Range of values	
	PDI mash	PDI conditioner	PDI pelletizer	NSIkoh mash	NSIkoh conditioner	NSIkoh pelletizer	DSC conditioner	Minimum value	Maximum value
PDI conditioner	0.99***	-						14.1 [%]	70.0 [%]
PDI pelletizer	0.96***	0.97***	-					13.2 [%]	54.1 [%]
NSIkoh mash	0.93***	0.93***	0.89***	-				82.2 [%]	98.9 [%]
NSIkoh conditioner	0.91***	0.91***	0.87***	0.87***	-			75.0 [%]	97.0 [%]
NSIkoh pelletizer	0.93***	0.93***	0.95***	0.91***	0.91***	-		85.9 [%]	99.9 [%]
DSC conditioner	0.23 ns	0.27 ns	0.28 ns	0.28 ns	0.34 ns	0.22 ns	-	1.09 [J g^{-1}][a]	2.20 [J g^{-1}][a]
DSC pelletizer	0.15 ns	0.17 ns	0.15 ns	0.09 ns	0.30 #	0.24 ns	0.10 ns	1.01 [J g^{-1}][a]	1.63 [J g^{-1}][a]

For probability levels see table 3.

[a] DSC-values recalculated to dry matter content of the sample.

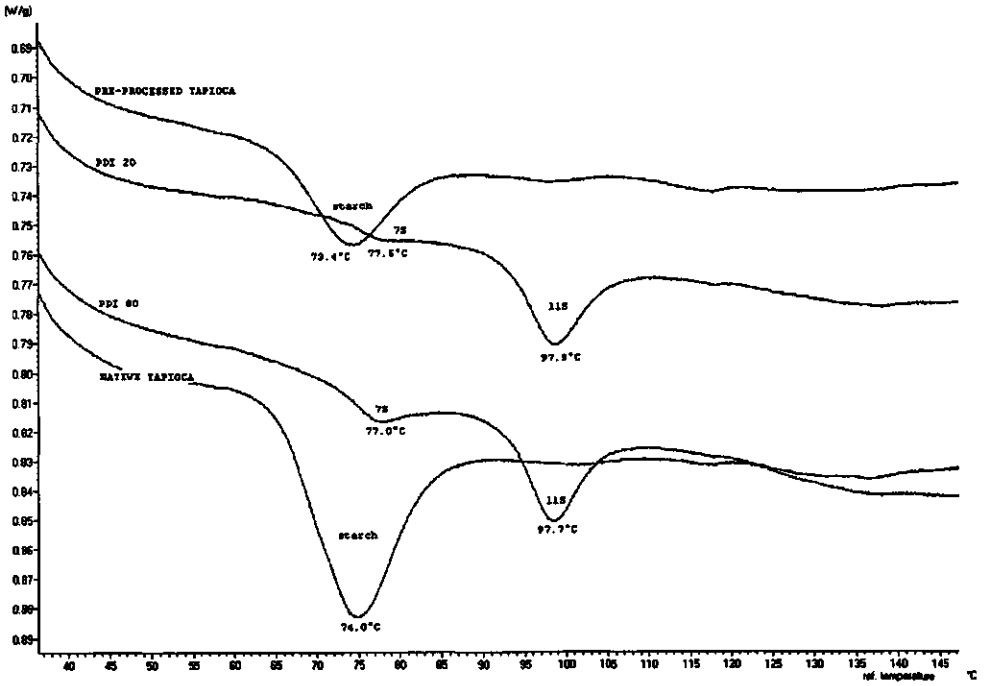

Figure 1: DSC-thermograms of the pre-processed and the native tapioca, and of the soya PDI 20 and PDI 80 used in the preparation of the mixtures. The x-axis denotes oven temperature. On the y-axis is the heat flow given (endothermic heatflow is downward). References to the fractions are obtained from literature (See text)

Table 3: Pearsons correlation coefficients between DSC and SGDags for the starch containing fraction of the model feeds.

	Starch				Range of values	
	SGDags mash	SGDags conditioner	SGDags pelletizer	DSC conditioner	Minimum value	Maximum value
SGDags conditioner	0.98***	-			9.0 [%]	60.7 [%]
SGDags pelletizer	0.94***	0.94***	-		46.4 [%]	76.1 [%]
DSC conditioner	-0.93***	-0.93***	-0.90***	-	1.97 [J g^{-1}]^a	5.34 [J g^{-1}]^a
DSC pelletizer	-0.77***	-0.79***	-0.84***	0.78***	1.35 [J g^{-1}]^a	2.79 [J g^{-1}]^a

ns = p>0.1; # = p =< 0.1; * = p < 0.05; ** = p < 0.01; *** = p < 0.001.

^{a} DSC-values recalculated to dry matter content of the sample.

DSC, but over the processes (e.g. SGDags after conditioning compared to SGDags after pelleting), were all better than 0.78 ($p<0.001$). Pelleting lead to a further increase in SGDags and a lowering of enthalpy, compared to conditioning. The SGDags values after conditioning (range 9.0% - 60.7%) was not different from the SGDags values of the feed mashes (range: 12.1% - 60.8%).

Correlations between the DSC-values of the protein fraction and PDI and NSIkoh are given in table 4. The correlations between PDI and NSIkoh were all above 0.87 ($p<0.001$). The correlation between DSC-values on the one hand and PDI and NSIkoh values on the other hand were small not significant at the $p>0.05$ level. PDI values decreased after conditioning and pelleting. PDI values of the feed mash was in the range between 18.3% and 83.3%. These values decreased after conditioning (14.1% - 70.0%) and after pelleting (13.2% - 54.1%). The NSIkoh range of values decreased after conditioning (75% - 97%) compared to the feed mashes (82.2 - 98.9%), and increased again after pelleting (85.9% - 99.9%).

Pelleting trial

Mean temperature of the pellets leaving the die was 87.8°C (std dev ± 3.15 °C). No effects were present of PDI or SGDags in the feed on pellet temperature. No effects were found of PDI and SGDags on the capacity of the installation.

The results for the fits of the curves are given in tables 5 and 6. In all cases, the joint effect of PDI on physical quality traits was significant. SGDags contributed in all cases except pellet porosity to pellet hardness and durability. The quadratic polynomial was not a suitable model in the case of Kahl hardness and pellet porosity as determined from the lack-of-fit test.

The figures 2, 3 and 4 give the contour plots obtained from this experiment. In figure 2a, 2b and 2c the contour plots are given for Holmen and Pfost durability and fracture strain, respectively.

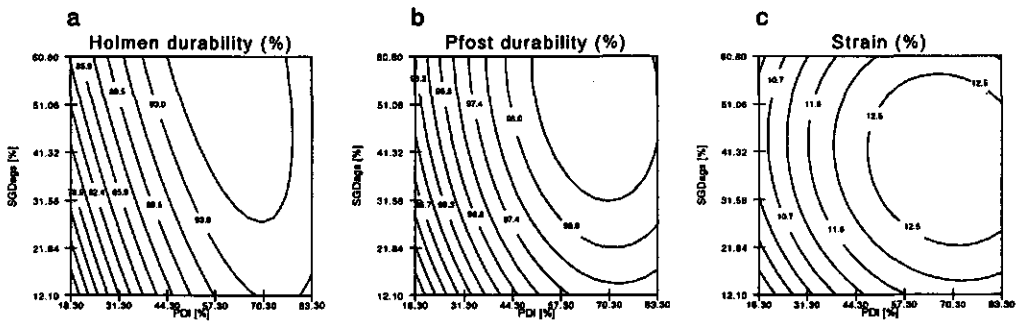

Figure 2: Contour plots of: a) Holmen durability (%); b) Pfost durability (%); c) Fracture strain (%). PDI (%) and SGDags (%) on the x-, and y- axis as determined from the mixtures.

Table 5: Regression coefficients of the pellet hardness and durability tests (obtained with model 1).

	Holmen [%]	Pfost [%]	Kahl [kgf]	Kramer shear press [N g^{-1}]	Compression tester [N mm^{-1}]
Regression coefficients[a]					
Intercept	50.24 ***	91.35 ***	12.48 ***	410.75 ***	8.87 ***
PDI	1.07 ***	0.15 ***	0.27 *	6.28 **	0.18 **
SGDags	0.443 *	0.08 **	0.003 ns	-2.24 ns	0.063 ns
PDI^2	-0.0069 ***	-0.001 ***	-0.0014 ns	-0.0486 *	-0.0011*
SGDags^2	-0.00155 ns	-0.00055 ns	0.00174 ns	0.0191 ns	-0.00044 ns
PDI*SGDags	-0.0039 **	-0.0003 ns	15.3E-6 ns	0.0551 **	0.0002 ns
Probability of the effects					
PDI[b]	***	***	***	***	***
SGDags[b]	***	***	***	***	*
Linear	***	***	***	***	***
Quadratic	***	***	ns	*	#
Crossproduct	**	ns	ns	**	ns
Lack of fit	ns	ns	**	ns	#
Total Regr.	***	***	***	***	***
R^2	0.85	0.88	0.80	0.83	0.75
MSE[c]	9.614	0.170	4.96	2223.8	1.55

For probability levels see table 3.

[a] Test for H_0: regression coefficient = 0.

[b] Test for the joint significance for the specific parameter involving all factors incorporating that parameter.

[c] Mean Square Error.

Table 6: Regression coefficients of pellet porosity, apparent modulus, fracture strain, SME and Capacity (obtained with model 1).

	Pellet porosity [-]	apparent modulus [N m^{-2}]	Fracture strain [%]	SME [kJ kg^{-1}]	Capacity [kg h^{-1}]
Regression coefficients[a]					
Intercept	0.218 ***	1.49E8 ***	4.97 **	64.9 ***	628.2 ***
PDI	-14.38E-4 **	76.2E4 ns	0.15 **	-0.06 ns	-0.58 ns
SGDags	-3.11E-4 ns	6.4E4 ns	0.142 *	-0.58 *	-1.01 ns
PDI^2	9.83E-6 *	-0.4E4 ns	-0.001 *	0.0021	0.0061 ns
$SGDags^2$	5.36E-6 ns	0.4E4 ns	-0.00153 #	0.0075 *	0.0079 ns
PDI*SGDags	3.51E-6 ns	0.2E4 ns	-0.0003 ns	-0.0024 ns	-0.0055 ns
Probability of the effects					
PDI[b]	***	*	***	*	ns
SGDags[b]	#	ns	#	**	***
Linear	***	**	***	***	***
Quadratic	#	ns	*	#	ns
Crossproduct	ns	ns	ns	ns	ns
Lack of fit	**	*	ns	ns	ns
Total Regr.	***	*	***	**	***
R^2	0.51	0.32	0.62	0.47	0.50
MSE[c]	10.4E-5	4.50E14	0.889	16.6	222.7

ns = p>0.1; # = p =< 0.1; * = p < 0.05; ** = p < 0.01; *** = p < 0.001.

[a] Test for H_0: regression coefficient = 0.

[b] Test for the joint significance for the specific parameter involving all factors incorporating that parameter.

[c] Mean Square Error.

In figure 3a, 3b, 3c and 3d the contour plots of Kahl, Kramer shear press, the compression test and the apparent modulus are given, respectively. In figure 4a, 4b and 4c the contour plots for porosity, capacity and SME are given, respectively. From these curves the range of values covered by the dependent variable can be determined. The dependent variables Pfost durability, fracture strain and SME had stationary values which where within the boundary limits of the experiment. The stationary point is the point in space at which all partial derivatives of the fitted regression equation with respect to PDI and SGDags are zero. The variables Holmen durability and porosity had optimum values which were close to, but outside the region of experimentation as determined from the canonical analysis.

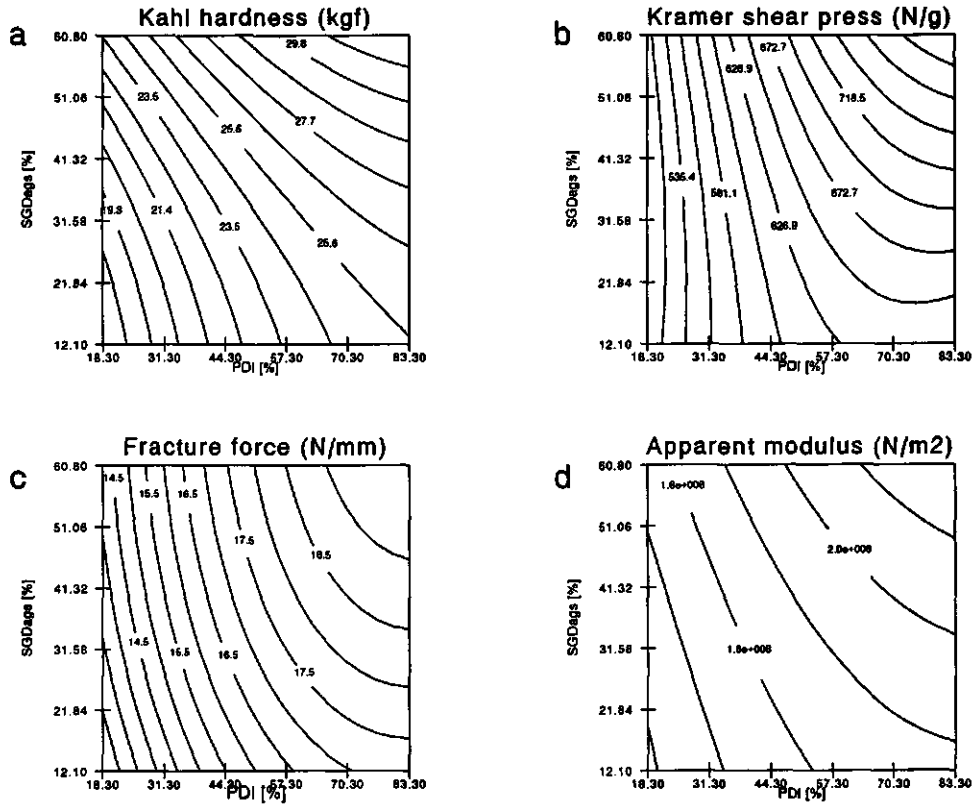

Figure 3: Contour plot of: a) Kahl hardness (kgf); b) Kramer shear press (N g^{-1}); c) Compression test (N mm^{-1}); d) Apparent modulus (N m^{-2}). PDI (%) and SGDags (%) on the x- and y- axis as determined from the mixtures.

The maximum Holmen durability estimated from the response curve was 96.7% at 57.7% PDI and 69.4% SGDags. The eigenvectors from the canonical analysis showed that the

response surface was most sensitive to changes in the PDI of the feed mash (eigenvalue -7.68) compared to SGDags (eigenvalue -0.55). See figure 2a.

The response of Pfost durability towards PDI and SGDags showed a maximum, which was found within the region of experimentation. Maximum Pfost durability was 98.3% at a PDI value of 64.7% and a SGDags value of 55.9%. The eigenvectors from the canonical analysis showed that the response surface was more sensitive to changes in the PDI of the feed mash (eigenvalue -1.09) compared to SGDags (eigenvalue -0.55). See figure 2b.

The stationary points for the Kahl hardness test, the values derived from the Kramer shear press and the fracture stress derived form the compression test were found outside the region of experimentation, hence no conclusions are drawn with respect to sensitivity of Kahl hardness values, Kramer shear press values and fracture stress towards PDI and SGDags.

The response surface for the Kahl hardness test was convex with respect to PDI and concave with respect to SGDags. Maximum value was 31.9 kgf at 83.3% PDI and 60.8% SGDags. The minimum value was 17.2 kgf, found at 18.3% PDI and 12.1% SGDags. See figure 3a. The quadratic polynomial model does not give an adequate description of the data found in this experiment as determined from the lack of fit test. Higher order terms such as cubic effect seem to be present in the data. It is not clear whether these effects should be associated with the (functional) properties of the raw materials or are related to the test method itself.

The response surface for the values derived from the Kramer shear press was convex with respect to PDI and concave with respect to SGDags. Maximum value was 810.1 N g^{-1} at 83.3% PDI and 60.8% SGDags. The minimum value was 490.0 N g^{-1}, found at 18.3% PDI and 32.2% SGDags. See figure 3b.

The response surface of the fracture stress values derived from the compression test was convex with respect to both PDI and SGDags. The maximum value was 19.4 N mm^{-1} at 83.3% PDI and 60.8% SGDags. The minimum value was 12.5%, found at 18.3% PDI and 12.1% SGDags. See figure 3c.

The stationary point for pellet porosity was found outside, but close to the region of experimentation. The stationary point, a minimum, was found at 72.1% PDI and 5.4% SGDags, its value was 0.166 [-]. Maximum porosity was 0.200 [-] at a PDI value of 18.3% and a SGDags value of 60.8%. Minimum porosity as determined from the graph was 0.166 [-] at 71.0% PDI and 12.1% SGDags. The eigenvectors from the canonical analysis showed that the response surface was more sensitive to changes in the PDI of the feed mash (eigenvalue 0.011) compared to SGDags (eigenvalue 0.003). See figure 4a. Care should be taken in the interpretation of these figures since the lack of fit test showed that the quadratic polynomial was not an adequate description of the experimental data. Moreover, the contribution of

quadratic terms to the equation is only marginal ($p<0.1$).

Although the regression equation for the calculated apparent modulus was significant at the $p<0.05$ level, the overall predictability of the equation was low. PDI was the largest contributing factor to the apparent modulus, as can be observed from the joint probability of the regression coefficients. Lowest value for the apparent modulus was 1.63E8 (N m^{-2}) found at 18.3% PDI and 12.1% SGDags. The highest value for the apparent modulus was found at 2.11E8 (N m^{-2}). See fig. 3d.

Fracture strain had a maximum value within the boundaries of the experimental range. Maximum fracture strain was 13.1 % at 68.6% PDI and 40.6% SGDags. The minimum fracture strain within the experimental range was 8.8 % and was found at 18.3% PDI and 12.1% SGDags. The eigenvectors and eigenvalues showed that the response curve was more sensitive to changes in PDI (eigenvalue -1.14) as compared to SGDags (-0.86). See fig. 2c

The highest specific mechanical energy consumption was 66.0 kJ kg^{-1} and was found at 83.3% PDI and 12.1% SGDags. The minimum value was 50.6 kJ kg^{-1} and was found at 39.3% PDI and 45.0% SGDags, within the experimental range. The eigenvectors and eigenvalues showed that the curve was more sensitive to changes in SGDags (eigenvalue: 4.79) and less sensitive to PDI (eigenvalue 1.85). See figure 4c.

The capacity of the installation changed due to the effect of SGDags. The highest capacity was found at low SGDags. The maximum value was 607.3 kg $hour^{-1}$ at 18.3% PDI and 12.1% SGDags. The minimum value was 561.6 kg $hour^{-1}$ and was found at 75.0% PDI and 60.8% SGDags. The curve was concave in both the directions (PDI as for SGDags). See figure 4b.

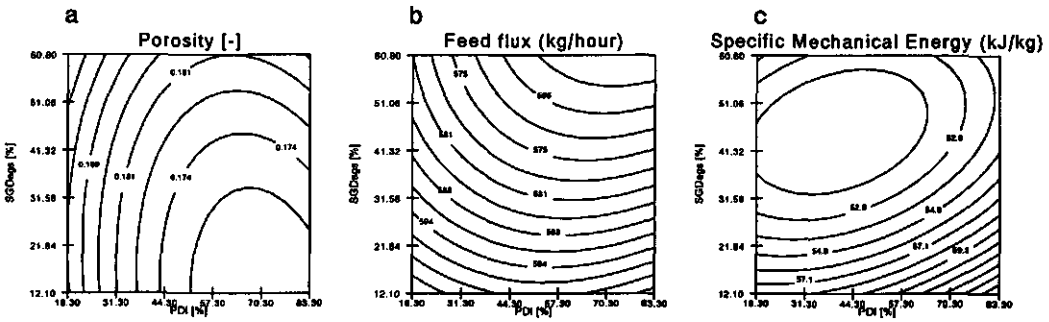

Figure 4: Contour plot of: a) Porosity (-); b) Capacity (kg $hour^{-1}$); c) Specific Mechanical Energy (kJ kg^{-1}). PDI (%) and SGDags (%) on the x- and y- axis as determined from the mixtures.

Discussion

The values obtained from the DSC measurements show a high correlation with the values obtained from the SGDags analysis. As can be observed from figure 1 a small part of the heat denaturation peak of the protein fraction is denatured in the same temperature region as where starch gelatinizes. Since these two peaks cannot be disentangled, this small portion of soya protein is included in the results for the DSC-values given for starch. This leads to an overestimation of the residual enthalpy values for starch. In addition, the conditions used in determining the residual enthalpy values of the starch and protein are done in the presence of an abundant amount of water (at least a 3:1 water/sample ratio). However, during the pelleting process only limited amounts of water are available (max. ~13% after conditioning), originating from the basal moisture in the feed or added by means of steam conditioning. It has been discussed in literature that low amounts of water causes shifts towards higher temperatures of gelatinization of the starch (Keetels, 1995; Eliasson and Gudmundsson, 1996) or denaturation of the proteins (Sheard *et al.*, 1986). Hence, the overlap as determined from the DSC-thermograms between starch gelatinization and the (7S) peak of protein denaturation, does not neccesarily have to be related to observed properties in the dependent characteristics. Although a good correlation of various physical properties of the pellets exists with both SGDags and DSC-values, it might be that these properties are not directly related to the residual enthalpy or degree of gelatinization as such, but that they are related to phenomena at a lower level. For instance, deformability of the starch granule may change upon gelatinization (Eliasson and Bohlin, 1982). In addition, the amorphous starch fraction may undergo the so-called glass transition which may have a large impact on the physical properties of the material under study. This glass transition is highly dependent on temperature and moisture content of the material subjected to treatment or testing. Above this glass transition the starch is in a rubbery state, in which it is deformable and can therefore be easily moulded during compression in the die and increases the probability of adhering particles together by means of 'viscous' bonding (Pietsch, 1991), this may occur during the processing stage where temperature can be above the glass transition temperature. Upon cooling the amorphous portion may change from a rubbery-like to a brittle type of material, thus acting as a hardening binder between particles. From literature it is known that the glass transition affects the textural properties of the system in which it is incorporated (Keetels, 1995; van Soest, 1996). In addition, indications are found that this affects the physical quality characteristics of pellets to a large extent (Thomas *et al.*, 1998). Additional DSC-measurements (not shown) indicate that a glass transition could be observed in the pre-

processed starch which in all cases was below the processing temperature of 70°C.

Various authors in literature describe the effects of soy protein and other components in relation to compressibility and density characteristics of these mixtures (Malave *et al.*, 1985; Barbosa Canovas *et al.*, 1987). In the present study a first attempt has been made to determine an apparent modulus and fracture strain properties of pellets. No comparison with literature is possible, since it appears that such studies with pellets have not been previously conducted. Although the modulus calculated in this study was obtained under conditions of fracture of the material, instead of linear elastic deformation, it might provide a first guideline on the order of magnitude of these figures. It seems however worthwhile to put more research effort in this direction, since it may provide more fundamental figures on the behavior of pelleted material when subjected to various forces.

Some reports have been published about effects of mixtures of starch with different degrees of gelatinization on tabletting properties (Schwartz and Zelinskie, 1978). However, in only one study (Wood, 1987) the effects of changes in protein quality (as measured with PDI) and in the degree of gelatinization of the starch fraction, as measured by an enzymatic method, have been studied with respect to pellet quality. Wood (1987) concluded that raw, defatted soybean meal had a larger impact on physical quality of pellets than denatured, defatted soybean meal. Inclusion of raw protein lead to harder and more durable pellets. The progressive substitution of native starch by gelatinized starch in his model feeds showed a curvi-linear increase in hardness and durability properties of the pellets. These results are partly confirmed by the data found in this experiment. An increase in protein dispersibility (PDI) and in increase in degree of starch gelatinization (SGDags) is associated with harder pellets. However, durability values from the pellets, manufactured in this experiment seemed to have a maximum value, after which the material becomes less durable again.

It is difficult to explain the observed phenomena in this experiment, since from literature various aspects are mentioned which affect compressibility of mixtures of particular materials and hence the fracture stress or hardness of the derived pellets. Amongst others, these are moisture content, particle size and deformation characteristics which lead to a behaviour of the material which cannot be deduced from the individual components alone (Barbosa Canovas *et al.*, 1987). Furthermore, it is difficult to determine which type of bond is formed between the various raw materials. When larger amounts of water are available, as for instance during extrusion, protein and starch are generally not compatible when present in the fully denatured or gelatinized state (Yurjev *et al.*, 1989; Zasypkin, 1992). This leads to extrudates which properties are predominantly described by the properties of only one of the components. This would be in line with the fact that almost no interactions are present between the PDI and SGDags values for the various measured characteristics. However,

studies conducted with extruders might be of limited value for feed engineering scientists since the amount of water used in the pelleting of animal feeds is limited and it is more likely that the observed effects are better explained by the paradigma found in powder technology than by the paradigma of 'melt-behaviour' of materials as is used in extrusion technology (Ledward and Mitchell, 1986). If solubility in water is taken as a criterion, than it might be reasoned that high PDI values and high SGDags values, which both implicate that a high affinity for water exists, may show 'compatibility' for each other and hence, may form strong agglomerates. In the study of Wood (1987) it was mentioned that raw soy and pre-gelatinized starch formed cohesive mixtures when blended together with water, with the rate of water absorption being dependent on the protein fraction. In this study, as in the study of Wood (1987), durability was more dependent on the protein than the starch fraction. It is not clear whether this protein (PDI) dependent durability, fracture strain and porosity is due to texturization of the protein. In that case the protein is a continous phase, with starch acting as a filler material, or that the deformability of the soy-grit particles is modified to such an extent that this deformability becomes the predominant controlling factor and starch acting as the binder material.

Although in the case of Holmen durability and Kahl hardness, an interaction between PDI and SGDags was observed, such an interaction was absent in all other measured characteristics. Moreover, an interaction between PDI and SGDags was absent in all of the other physical quality tests (Pfost, Kramer shear press and the compression test). In addition to this, no interaction was found between pellet porosity, apparent modulus, fracture strain, SME and capacity of the installation. Hence, it seems justified to conclude that, although the interaction between PDI and SGDags in some cases appears to be (numerically) significant, based on the other characteristics measured, it may be concluded that PDI and SGDags are additive.

The contour plots of the durability tests, fracture strain and porosity show that these characteristics are more sensitive to changes in PDI than changes in SGDags, as is observed from the canonical analysis. High PDI values favor high durability and low porosity. Such an observation may indicate that the high PDI material is more easily deformable than the low PDI material. The particle size of the soy grits is larger than of the used tapioca (see table 2), hence, less room would be available for these particles to rearrange upon compression. The smaller starch particles have more room for movement and hence may flow more easily in the larger voids whenever present. Furthermore, it should be taken into account that the energy required for compression (as determined from the SME figures) is not dependent on the PDI values but seems to decrease with increasing SGDags. This may be indicative for gelatinized starch acting as a lubricant. That gelatinized starch may act as a lubricant is also supported by

the study of Wood (1987) who found that durability and hardness where largely affected by the protein fraction and that SME-values and capacity were affected by the amount of gelatinized starch included. However, although SME values in the present study and the study of Wood (1987) are in line with each other, the feed flux is not. In the present study a decrease in feed flux was observed, whereas Wood (1987) noticed an increase in feed flux with increasing levels of pre-gelatinized starch. In the present study, the most likely explanation for the decrease in feed flux may be a change in flow behavior of the feed mash. An increase in the amount of gelatinized starch may lead to a decrease in outflow from the discharge bin. However, flowing properties of the feed mash have not been studied, therefore this cannot be a decisive conclusion.

From the foregoing it can be concluded that it is not clear what factors in the protein are responsible for the observed effects on the pellet durability, fracture strain measured and porosity. Circumstantial evidence indicates that it is the deformability of the protein containing soy-grits that primarily determines the observed physical characteristics. From the present study it can however not be ruled out that some form of texturisation and re-association of the protein fraction exists, which could induce the observed effects. However, due to the low quantity of water present in this study (~13%), as opposed to (texturizing) extrusion experiments (≥30% moisture; Yurjev *et al.* 1989; Zasypkin, 1992), the relative low maximum temperatures at which this experiment is conducted (87.8°C pellet temperature after leaving the die) versus maximum temperatures ≥150°C in the extrusion experiments, it is concluded that deformability seems the most likely cause. Further research is however necessary to fully elucidate the mechanism(s) by which soy protein and tapioca starch affect pellet quality and related characteristics.

Conclusions

Pellet quality is affected by the state in which the protein and starch fraction occur within the feed. An increase in the amount of dispersible protein (as measured with the PDI-test) and an increase in the starch degree of gelatinization (SGDags) as measured enzymatically (amyloglucosidase) or by differential scanning calorimetry, leads to harder and more durable pellets. From this experiment it is concluded that a high apparent modulus (as defined by the conditions in this experiment) in the pellets is associated with harder pellets as determined from various (empirical) tests. It was found from the statistical analyses that Holmen and Pfost durability,fracture strain of the pellets and porosity were more sensitive to changes in PDI, whereas SME was more sensitive to changes in SGDags, as was the feed flux of the installation. Fracture strain of the pellets is related to durability properties of the pellets. The

different effects found were explained by a concept in which the larger soya particles (protein fraction) are associated with the deformability of the feed mash and the tapioca (starch fraction) acts as both filler and glue between these particles. As a glue, the amorphous part of the starch may appear in viscous form, when during manufacturing, the processing conditions exceed the glass transition point of the starch and, after cooling, this amorphous starch may act as a hardening binder (below the glass transition point). In addition, 'texturization' of the protein fraction and subsequent effects on pellet quality parameters was considered, yet seemed to be a less likely cause for the observed effects. More research on the effects of protein and starch, and the state in which they appear should be conducted to be able to abandon or adopt the proposed mechanism.

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General Discussion

M. Thomas

Introduction

The focus of this thesis is to further develop understanding about the functionality of raw material constituents and to determine what their relation is with pellet hardness and durability. To do so, model feeds (soy-grits and tapioca meal) have been used throughout the various studies described in this thesis. As functional constituents, the choice has been restricted to the protein fraction and the starch fraction of these two feedstuffs.

Pelleting is an additional process to the milling and blending of the feed material to obtain a form which does not segregate during transport and therefore assures a fixed feed composition in the diet. Although it can be thought of as an additional process in the manufacture of animal feed (since a nutritionally balanced feed is already present after the milling and blending stage), a large proportion of the animal feeds are fed in pelleted or expander pelleted form. In 1995, in the Netherlands, about 15.6 million tonnes of animal feeds were produced (Anonymous, 1996) of which between 85% and 90% is in pelleted or expander-pelleted form. The used amount of energy in the preparation of these animal feeds can be divided over the following processes that occur during feed manufacturing: approximately 7% of the energy consumed in pelleting is for reception and storage of the raw materials, 16% is used in grinding and mixing, an additional 16% is used for transport in the factory, heating and light, 1% is for additional packing and the largest energy consumption in feed manufacture is made up by the conditioning, pelleting and cooling process: 60%. The total amount of energy used in feed manufacturing to convert one ton of raw material to a pelleted product is between 420 and 630 MJ tonnes^{-1} (Beumer, 1981). Its high energy consumption is one of the disadvantages of the pelleting process.

Since a large portion of animal feeds is pelleted, there should be some distinct advantages of pelleting feed over the feeding of mashes to the different animal groups. These can be divided in factors associated with the feed form and factors associated with the nutritional value. Some of the nutritional effects, both positive and negative have been discussed in Chapter 3. Advantages associated with the physical form of the feed that are related to feeding of animals are:

- No segregation of the constituents occurs. In addition, no segregation of co-agglomerated products exists which is a technology, increasingly applied in the production of pelleted animal feeds, for instance, the coating of liquid additives on the exterior of the pellets.
- A free flowing product is obtained, which improves storing and handling characteristics of the feed.
- The bulk density of the feed is normally increased (although, expander-processed animal

feeds may have a relative large bulk volume).
- Product appearance is improved, which leads to higher sales value.
- A reduction in dust is obtained, which is both beneficial in terms of feed losses and health perspective for the farmer and the animals.

From the chapters 1 to 3 it becomes apparent that, although a lot of research has been directed towards the effects of changes in processing parameters during manufacture (Chapter 2) and the role of certain components present in the feed mash before pelleting (Chapter 3), not much research has been directed towards the properties of these components and their contribution to the physical pellet quality. It is this latter field, the functionality of feedstuff components, how it is influenced by processing parameters and how it affects the hardness and durability of the pelleted feeds that has been the major subject of this thesis.

Initial hypothesis

The work of Wood (1987) has been the onset of this thesis work. In his work, a direct relation was found between functionality of the feed ingredients and physical pellet quality. The degree of starch gelatinization and solubility of the protein fraction were found to have a major impact on the pellet hardness and pellet durability. Substitution of native starch by pre-gelatinized starch led to harder and more durable pellets. In addition, the use of soluble protein over denatured protein further improved pellet hardness and durability (See Chapter 3, figure 1). At the onset of this study, it was decided to study the starch and protein fraction, since practical experiences showed that effects may be expected of both the protein and starch fraction and these two raw material constituents represent a valuable portion of the nutritive value of feeds. The eventual choice for soy (grits) and milled tapioca-chips was made on the basis of 'mutual exlusion'; in tapioca almost no protein (<26 g kg^{-1} product) is present and soy contains hardly any starch (<10 g kg^{-1} product) (anonymous, 1997). For the above mentioned reasons soy-grits and tapioca chips have been chosen as the subject of study.

The main hypothesis throughout this thesis has been that an increase in the amount of dispersible/soluble protein and an increase in the amount of gelatinized starch present would lead to harder and more durable pellets. Furthermore, in this general discussion, some questions will be addressed related to the work in this thesis.

Functional properties

What are functional properties of raw materials or their constituents with respect to pellet hardness and durability?

Throughout the thesis, the effects on the physical quality of the pellets have been attributed to the protein fraction in the soy and to the starch fraction of the tapioca. Such a choice, inevitably, possesses some limitations, because soy-grits also contain a non-protein portion and tapioca contains a non-starch portion. It can, therefore, be asked whether or not the functionality of the protein and starch fraction *per se* is contributing to the physical quality characteristics or that the functionality of these fractions should be related to the outcome of both interactions between the protein and non-protein fraction in the soy, and the interaction between the starch and non-starch components in the tapioca. For soy, it is generally accepted that the proteins are the principal functional components (Kinsella, 1979), but other fractions may play an important role in the control of the protein with respect to its target in a food or feed system. For instance, Sheard *et al.* (1984) showed that the carbohydrate components present in soy flour aid in stabilizing extrudates by minimizing hydratability and carbohydrate solubility when compared to soy isolates.

According to Hermansson (1979) the following definition is appropriate: 'functional properties are physico-chemical properties which give information on how a protein will behave in a food system'. This is in close agreement with the statement of Kinsella (1979) that functional properties are '...the intrinsic physico-chemical characteristics which affect the behavior of protein in food systems during processing, manufacturing, storage and preparation...'. These physico-chemical characteristics can be, for instance, the solubility or swelling properties of the protein. With respect to the current study, these functional properties must be related to the ability of the protein to act as a binding element and the ability to deform during pelleting, and as such contribute to the structural integrity of the formed pellets. Then a contact area between the particles will be established, large enough to help maintaining structural integrity.

In a recent overview of Eliasson and Gudmundsson (1996) gelatinization is described as '...a collective term to describe a range of irreversible events occurring when starch is heated in water...'. Functionality of starches is therefore closely related to the amount of water present during processing. Upon heating in the presence of water, starch granules start to swell and disruption of the molecular and crystalline regions occurs. Through the accompanying change in volume, these regions become more accessible to water, which further decreases

molecular and crystalline order (Cooke and Gidley, 1992). A vast amount of literature exists in which the properties of starch are investigated with respect to rheological changes as for instance shear and elasticity modulus (Eliasson and Bohlin, 1982; Eliasson and Gudmundsson, 1996; van Soest *et al*, 1996). Although this literature may provide insight in the different processes which can be exhibited by starch, they may be of little value in explaining effects of inclusion of starch in pelleted animal feeds, since these feeds ordinarily have low moisture contents (<18%) as opposed to the conditions often encountered in literature: >50% moisture (Harper, 1981). However, at the exterior of the particles, (partly) gelatinized starch may be found which can act as a binder between feed particles. In addition, it has been shown in literature, that upon gelatinization the granules soften, thus leading to better deformability of the granule (Eliasson and Bohlin, 1982), which will further enhance pelleting ability of the feed mash.

Can these functional properties be measured directly?

Most of the functional properties are determined by the balance between the forces underlying protein-protein and protein-solvent interactions. Measurements of solubility (Nitrogen Solubility Indices (NSI) in various solutions) or colloidal stability (Protein Dispersibility Index; PDI) have therefore often been used as indicators for the functionality of the protein fraction (Hermansson, 1979). It is not clear from this study, how on molecular level these factors have an impact on pellet quality. Taking into account the foregoing discussion it seems, however, justified to use PDI or NSIkoh as an indicator for the functionality of protein. Primary emphasis in this thesis was, however, on PDI.

Numerous methods exist which describe one or more aspects related to changes in starch properties. These methods may rank from the determination of X-ray diffraction patterns to determine crystall types present in the starch, to measurements of viscosity in starch/water solutions. Microscopical examinations are being used to relate changes in the starch fraction to function-structure relations (Gallant *et al*, 1992). Thermal analysis, especially differential scanning calorimetry (DSC) has gained increasing popularity as a means to investigate transitions taking place in the starch material (Hendrickx *et al*, 1987). Often through combinations of methods, an increase in understanding of the causal relations underlying starch modifications is reached (Roulet *et al*. 1987). Overall, gelatinization of starch, as is protein denaturation, is not a single process, but merely a number of related processes, which, depending on the specific contributions of certain conditions specified in the process or experiment, contributes differently to the functionality of the starch. Based upon this, the degree of gelatinization as measured with an enzymatic method or the use of differential

scanning calorimetry might well be correlated to those processes occuring in starch which impart functionality in the pellet, without necessarily providing a causal mechanism for the observed effects.

Is there an interaction between the functionality of feedstuffs?

From table 1 and 2 it appears that PDI values are more affected by the conditioning process than by the pelleting process. For SGD the opposite seems to be the case; the effect of pelleting is more pronounced than the effect of conditioning and expander processing alone.

Table 1: Ranges of values found for PDI and SGD values after the conditioner or after expander-processing[a].

Experiment described in:	Unit operation	Raw material: test	Starting material	After conditio-ning	Additional processing parameters (Range)		
			[%]	[%]	Mass debit [kg h^{-1}]	Temperature [°C]	Water [g kg^{-1}]
Chapter 4	Conditioner	soy-grits: PDI	74.6 %	22.5-43.5	250-410	78.6-97.8	19-640 added water
	Expander	soy-grits: PDI		23.6-36.9		88.8-111.5	
Chapter 5	Conditioner	tapioca: SGD	10.3	5.1-22	406	66-72	35.6-40.2
	Expander	tapioca: SGD		5.0-40.2		43-101	30.2-41.1 added water
Chapter 6	Conditioner	tapioca: SGD	11.5 - 53.1	8.1-54.2	235-531	44.1-67.6	12.8-16.9 added water
Chapter 7	Conditioner	soy-grits: PDI	25.4 - 86.9	19.7-83.5	594-651	66.4-84.4	72 total water
Chapter 8	Conditioner	soy-grits: PDI	18.3 - 83.3	14.1-70	525	70	98.9-102.7 total water
		tapioca: SGD	12.1 - 60.8	9.0-60.7			

[a] Processing parameters given, denote the highest and the lowest values found (estimated with the models used or abstracted from the raw data) in the respective experiments. Values given are only indicative. For details on the experiments see the respective chapters. In all cases conditioner treatment preceeds expander processing, hence values found after the conditioner are input values for the expander.

Hence, the soy grits and tapioca mash react different to the different unit operations.

From Chapter 8, in which mixtures of soy and tapioca were used, it was shown that no interactions were present between the PDI and SGD with respect to the paramaters: Pfost durability, Kramer shear press and compression test values, pellet porosity, apparant modulus, strain, specific mechanical energy and capacity. Although for the Holmen and Kahl test an interaction was found between PDI and SGDags, these was not consistent with the other tests used. It was, therefore, concluded that PDI and SGD values are additive and act independently under the conditions of the study presented in Chapter 8.

What is actually tested in hardness and durability tests?

Pellet quality in the feed manufacturing industry is expressed in terms of its hardness and durability. Hence, throughout the thesis, these terms have been applied for the several tests conducted on the pellets, although in Chapter 1, in reviewing the available testing methods used in industry, already precautions are given in order to avoid a too straightforward interpretation of 'hardness' and 'durability' characteristics of the pelleted feeds. These tests used in practice, comprise both elements in the values they give. For instance, the Holmen pellet test measures a mixture of hardness and durability properties of the feed (Chapter 1), but is always referred to as Holmen durability. The tests used in this thesis comprise the Holmen durability test, the Pfost durability test and the Kahl hardness test. In addition use has been made of the Kramer shear press and an overload compression and tension tester. A description with advantages and disadvantages has been given of these test methods in Chapter 1. No preference was developed for one testing method over another.

Tests to determine fracture phenomena in food or feed material can be divided according to the type of deformation that is applied to the material: all-sided compression, simple shear and uniaxial extension or compression (Luyten *et al.*, 1992). Testing methods to determine hardness in pelleted feeds often use uni-axial or diametral compression of a pellet or a series of pellets. In principle two modes of failure may occur: failure in tension (compression) and failure in shear. When working with brittle, inhomogeneous materials like pelleted feed, these modes of fracture are often not observed. Fragmentation occurs, in which the pellet falls apart in pieces of various sizes and geometry.

Fracture behaviour of feed materials is a very complex subject. Due to the relative inhomogeneities introduced by the various sizes and geometry of the particles present, it is difficult, if not impossible, to determine one unambiguous value for the strength of pellets. Apart from this, the measured fracture forces and stresses are time-dependent, which means that the determined hardness and durability values varies according to the test methods used.

Apparant relaxation times measured of pellets with an compression and tension tester show that these values may range over 2 orders of magnitude (0.3 s for soy pellets and 30 s for wheat or barley pellets. Thomas *et al.*, 1996; unpublished results). However, such results should be treated with caution since it is not clear whether these relaxation times should be attributed to energy dissipation due to frictional forces between the particles upon deformation or energy dissipation due to viscous flow (Van Vliet *et al.*, 1993) of certain parts (or the whole) of the pellet. It is known that the speed of deformation affects the observed fracture stress (Van Vliet *et al.*, 1993). Differences in values found between the various 'hardness' tests used in this thesis may therefore, apart from the differences in measuring geometry, be related to the difference(s) in speed of deformation. For instance, in Chapter 6, no apparent change in 'hardness' is observed for the pellets derived from batch B25, B50 and B75. (B100 was not tested for fracture stess, because the material deformed but did not break), whereas a tremendous increase is found in 'hardness' when pellets from the treatments B50, B75 and B100 were tested with the Kramer shear press. The speed of deformation used in the compression test was 10 mm min^{-1}, whereas the speed of the moving ram of the Kramer shear press is approximately one order of magnitude larger. Hence, marked differences can be observed in fracture stress ('hardness') depending on the test used. Similar phenomena are present within the durability tests as well, since speed of travel of the pellets differs considerable between the Holmen pellet tester (high speed of travel) and the Pfost durability tester (low speed of travel). Within the scope of this thesis it is, therefore, not tried to give a rigourous treatment on the fracturing behavior of the pellets. Empirical and semi-empirical tests have been used to evaluate the 'hardness' and 'durability' of the pellets. Further research in this area is strongly encouraged, since such studies will provide more knowledge on the type of binding mechanisms present in pellets. This may then be combined with knowledge on functional behavior of feed constituents to further optimise the manufacture of pelleted feeds.

Can the functional properties of the raw materials or their constituents be manipulated, and to what extend can they be controlled?

The functionality of a protein in its application may be chemically modified by numerous other factors. This includes, for instance, the presence of salts, changes in pH, water or other solvents, or the presence of carbohydrates in, for instance, soy. From Chapter 4 and Chapter 5 it follows that changing the processing conditions can alter the PDI, NSI (Chapter 4) or SGD (Chapter 5), as well. By use of response surface methodology (RSM) in Chapter 5, it was shown that SGD can be controlled as a function of the processing variables used in that study.

Table 1 and table 2 give an overview on the extent of which PDI or SGDags values changes as a function of processing conditions and unit operations as found in the various chapters of this thesis. More details can be found in the respective chapters. With use of RSM or other optimization algorithms, the processing conditions to set a certain (functionality) parameter can be calculated. This will then aid in reaching the determined objectives of the feed manufacturer. Although in Chapter 5, only processing conditions are given to obtain a specific set-point for one dependent variable at a time (for instance, SGD or energy consumption of the pellet press), the use of optimization techniques permits to take more objectives into account, for instance, maintaining pellet hardness at a certain minimum level and minimizing energy consumption (De Blank *et al.*, 1997). It seems, therefore, that by manipulating SGD and/or PDI via changes in processing conditions, pellet quality can be controlled.

Table 2: Ranges of values found for PDI and SGD values after the pellet press compared to the values found after conditioning. For feed flux and water added, see table 2.

Experiment described in:	Raw material: test (PDI or SGD)	After *conditioning* [%]	After the *pelletizer* [%]	Temperature pellets [°C]
Chapter 5	Tapioca: SGD	5.1-22	34.0 - 77.3	63.5 - 90.7
Chapter 6	Tapioca: SGD	8.1-54.2	43.1 -84.7	62.0 - 87.0
Chapter 7	Soy-grits: PDI	19.7 - 83.5	19.03 - 79.0	82.0 - 97.3
Chapter 8	Soy-grits: PDI (mixture)	14.1 - 70	13.2 - 54.1	88
	Tapioca: SGD (mixture)	9.0 - 60.7	46.4 - 76.1	

Indications for the extent to which PDI and SGD can be controlled might be obtained from the various statistics given in Chapter 4 and 5 on the spread of the data values after correction for the various factors studied in the respective experiments. For example, standard error of the mean for PDI values after the conditioner was 3.1, with the highest and the lowest values of the treatment means being 43.5% and 22.5% (Chapter 4; Table 3). After expander processing this was 2.5, with the highest and lowest values of the treatment means being 36.9% and 23.6% (Chapter 4; Table 4). In Chapter 5, indications can be obtained from the root mean squared error (RMSE) and the R^2 given. The RMSE for SGD after the conditioner is 3.01 and the range of values found (Table 1) ranges from 5% to 22% with a R^2 of 0.55. RMSE after expander processing was 6.48 with an R^2 of 0.64 and a range of 5% and 40.% SGD. It should, however, be emphasized that controllability of the process can not be judged by these figures alone, but should be valued with respect to the objectives set forth by the user. By use of the factors described in this thesis it can be concluded that they provide a means for controlling

the functionality of the feed constituents. Other factors, such as residence time, which have not been studied within this thesis may further enhance predictability and controllability with respect to the functional properties of the feed constituents and the related pellet quality.

Is it possible to develop a model, from which predictions on pellet quality can be derived?

One of the initial hypotheses was, that a suitable model could be found within the class of monotonically increasing models that could describe the effect of the inclusion of PDI or SGD on the physical quality of the pelleted feed. Especially in Chapter 6 and 7 it was tried to find a model which would fit in this initial hypothesis. However, as can be concluded from Chapter 6, physical characteristics like particle size and moisture content are also very important when considering functionality of starch (by SGD) and pellet quality. In Chapter 7, PDI was changed in the feed mash and the two factors particle size and moisture content where kept constant. Under these conditions, pellet hardness and durability, where found to follow a sigmoïdal or (increasing) exponential model. Although the models used may give an adequate description of the data, they only do so within the experimental region.

In Chapter 5 and Chapter 8, use was made of response surface methodology (RSM). Although the quadratical polynomial used in this analysis is not related in a causal manner to the underlying process, derived statistics and parameters provide very useful information about change in response as a function of PDI and SGD. Especially in Chapter 5, use of RSM proved to be useful in determining the relative contribution of the effects of processing conditions (addition of water, steam and changes in power consumption and screw speed of the expander) on energy consumption of the pellet press, pellet hardness and durability figures and to changes in the SGD after conditioning, expander processing and pelleting. The main conclusions were that a change in SGD of the feed is primarily induced by the factors water addition and steam pressure, whereas pellet hardness and durability are more affected by the factors steam pressure and power consumption of the expander. In Chapter 8, RSM was useful in determining the relative sensitivity of the contribution of PDI and SGD with respect to pellet hardness and durability. In the latter study it was concluded that pellet hardness and durability is more sensitive to changes in PDI than to changes in SGD. The capacity of the installation and energy consumption were more related to SGD. This methodology may prove to be very fruitful in practical feed manufacturing as well, since it posesses the possibility to provide guidelines on how to optimize processing conditions in relation to energy consumption and pellet quality.

The results from Chapter 7 and Chapter 8 indicate that increasing PDI and higher SGD values induce a higher pellet hardness and durability. The results of Chapter 6, however,

indicate that although the effect of higher SGD and higher PDI may give higher pellet durability and hardness, particle size, conditioning temperature and moisture content can mediate these effects to such an extent that, initially, decreases in strength may be observed with increasing SGD and PDI values. When *ceteris paribus* only PDI and/or SGD are increased then also an increase in hardness and durability can be expected as is evidenced by the results from Chapter 7 and 8. It is therefore concluded that, although a family of models may exist which will give an adequate description of the relation between PDI and SGD, this may prove of little value for practical feed manufacturing situations, if, at least, the changes in particle size, conditioning temperature and moistue content are not taken into account as well.

In addition, it should be mentioned that other constituents may have functional properties which affect the physical quality of pelleted feeds, as well. For instance, the effect of inclusion of fat is very large with respect to pellet hardness and durability. Therefore, research on functional properties of other feed constituents is strongly encouraged. This knowledge of functional properties of feed constituents may then be combined with research conducted on process equipment and processing conditions, to develop (numerical) models which will help us to understand how a certain pellet hardness and durability can be achieved.

A conceptual model

In figure 1 a schematical representation is given of the pelleting proces. The conditioning, pelleting and cooler parts are given. Square boxes denote unit operations like the conditioning equipment (conditioner or expander), the pelletizer and the cooler. Solid lines indicate mass flow to and from the unit operation. Rounded boxes are in- or outputs. Dashed squared boxes outside the unit operations boxes represent parameters or processes that have a major influence on processes and parameters in the unit operation box, yet are not studied as a primary factor in this thesis. Triangles denote where adjustment to the process can be made.

Feed mash enters the conditioner and is mixed with steam and water. Depending on the conditioning unit used, e.g. expander or compactor type of devices, shear may also play an important role. The deformability of the material is most likely to increase due to thermal softening (Rao and Lund, 1986). Some components, like sugars and salts will be solubilised (see Chapter 1). Feed components like starch and protein will be altered by these conditions present, the rate and extent of which is governed by the residence time, speed of diffusion of heat and water and degree of mixing and shearing (Oechsner de Koninck and Bouvier, 1995). In this phase, the relative contribution of each bonding type in the pellet to be formed is determined to a large extent. Table 1 presents an overview of the contribution of the conditioning process to changes in the PDI and SGD values of the soy and tapioca used in this

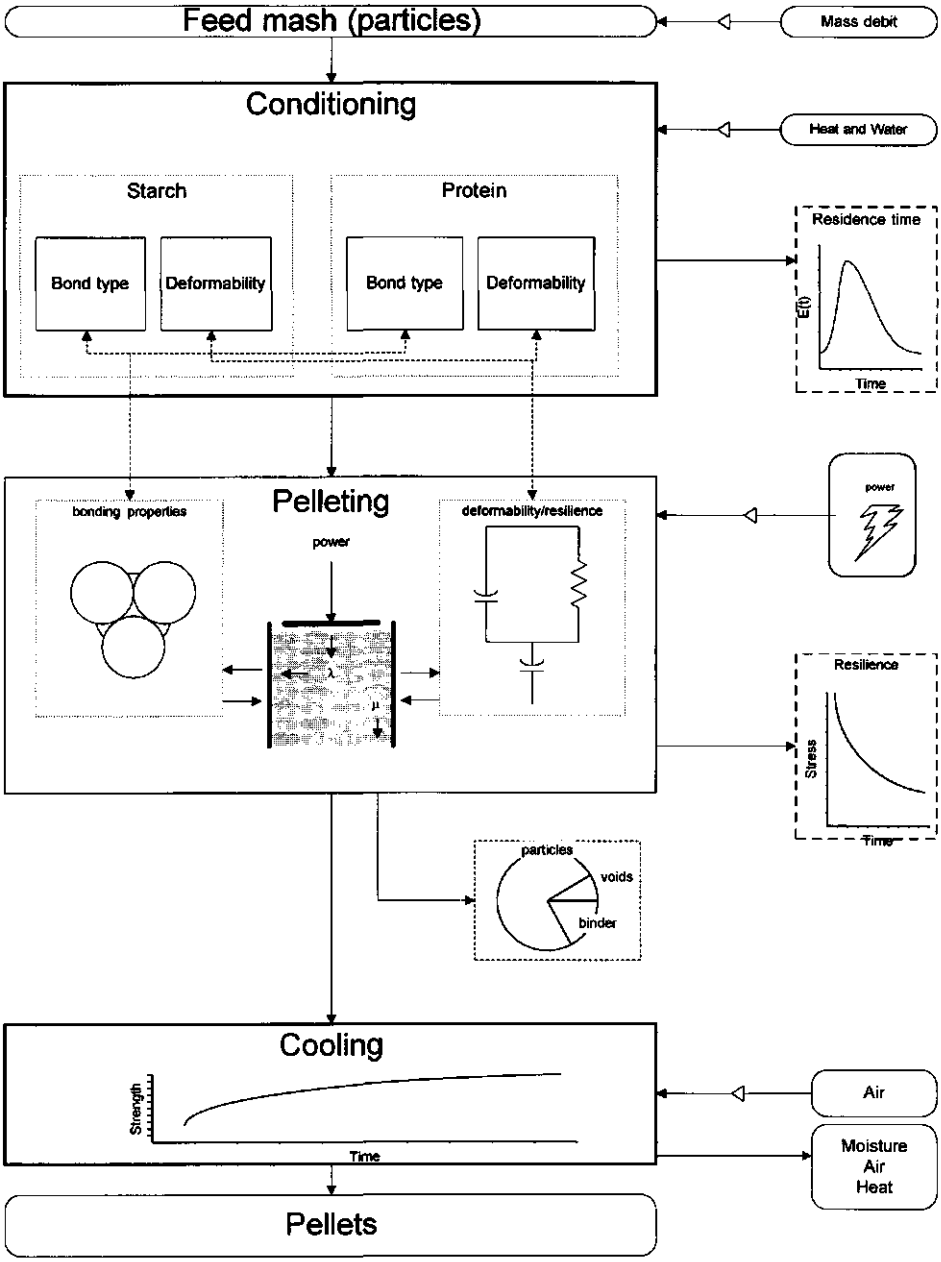

Figure 1: Schematical concept of the conditioning, pelleting and cooling process (see text).

study. It can be observed that when comparing PDI values of the results obtained from Chapter 4 with the results of Chapter 7 and 8 that the PDI of the soy grits decreases with increasing mass debit. The high mass debit (410 kg h^{-1}) found in the experiment described in Chapter 4 is due to the large amount of tap water included in the experiment. Mass debit of the soy grits in that experiment was 250 kg h^{-1}. Expander processing induces the largest changes in PDI (Chapter 4) and SGD (Chapter 5).

Although the effect of residence time was not determined in this study, this is the most likely explanation for the decrease in PDI at lower mass debits. Additional arguments for this are found in the Chapters 4 and 5 where it was concluded that the amount of specific mechanical energy is too low to induce changes in PDI and SGD, and hence can be eliminated as a main causative factor. The lower PDI-values and higher SGD values could than be interpreted in terms of an increase in residence time due to the additional time in the expander. Based on these reasons, it can be concluded that residence time as a parameter should be included in future research.

In all experiments, the lowest value for SGD found after conditioning is lower than the value of the starting material. The most likely explanation for this is that during conditioning in the barrel type conditioner, certain processing conditions were not severe enough to induce gelatinization of the starch fraction but may have led to annealing of the starch (a process that improves crystallinity; Eliasson and Gudmundsson, 1996), which in turn may have resulted in the lower SGD values found. It is unlikely that the lower values are associated with recrystallisation (retrogradation) of starch, since this would require that (part of) the starch is gelatinized to an amorphous form. If recrystallization upon storage would have occured, then it is not likely to be to such an extent that crystallinity improves beyond the initial level. As can be seen from figure 2a of Chapter 5, SGD is lower than the initial level of 10.3% SGD. With increasing water content SGD decreases, when steam pressure remains below 130 kPa. At this steam pressure, feed mash temperature after the conditioner was approximately 70°C which is approximately the gelatinization temperature of tapioca starch in abundant water (Cooke and Gidley, 1992). Although the above proposed concept is heavily relying on circumstantial evidence, and not tested as such, it seems to be in line with the results obtained from the various studies conducted with starch (Table 1) in this thesis. More research is, however, necessary to adopt or abandon the proposed concept as described above.

After the conditioning steps, the material is subjected to compression in the pellet press. Here, a distinction is made between the properties that affect pelleting behaviour due to the deformability and resilience characteristics of the particles (which have been altered in the previous conditioning process) and the type of bond that can be formed between the particles. Deformability and resilience have been shown in literature to affect the durability of formed

wafers (Mohsenin and Zaske, 1976). A theoretical framework on the relation between compaction, deformability and resilience has been put forward by Hiestand *et al.* (1977).

In Chapter 1, paragraph 2, the different types of bonds are described. According to calculations of Rumpf (1958) for idealised spherular particle systems and taking into account the mean particle size of animal feeds (Fig. 2), it can be expected that the predominant types of bonds (in cooled pellets) are formed by crystalline material, sinter bridges in the presence of appropriate materials and liquid necking. After leaving the die, the warm pellets can be deformed without loosing their structural integrity, for this reason the bond type in the warm pellets must be either liquid necking or induced by viscous material. These viscous material may have been added during the blending stage or emerged as a result of changed material properties due to the conditioning process.

The amount of compression reached in the die will be a function of the axial stress to the radial stress (λ), the coefficient of friction between the die wall and the feed mash (μ) and the length of the die. These stresses are in turn a function of the deformability, the packing properties (Barbosa-Canovas *et al.*, 1987) and the binder material of the feed mash. In figure 1, deformability and resilience is schematically depicted by some combination of a spring and damper (generalized Maxwell-model). The bond type is schematically represented by the three spheres with bonding material inbetween.

Calculations with respect to flow behaviour and amount of compression in the die are, to date, not possible. In systems with an open die, such as a pelletizer, the amount of compression is dependent on the material properties, the latter being changed as a result of the shearing action of the feed mash against the die wall. PDI and SGD after pelleting change with respect to their values after conditioning (Table 2). Moreover, the extrusion of material in a pellet press is a discontinuous process governed by the (angular) velocity of the die, the amount of rollers present and the feed flux. Under these conditions of slip-stick, chaotic behaviour may emerge, as recently has been discussed by Feeney and Liang (1997). The observed exit-die defects observed in the Chapters 5 and 6 may perhaps be related to this phenomenon. According to Kurtz (1992) exit die defects occur during stretching of the material when leaving the die land. The fracture observed (see photo 1, Chapter 6) is a function of the state of the material. It remains to be investigated if it is the initial condition of the material when entering the die or that the changing conditions during passage in the die are pre-dominant factors in inducing exit-die defects. Changes in SGD values during transit in the die are given in table 2.

The outcome of the pelleting process is the warm pellet which can be divided in three major components: the particle-, the binder-, and the void-fraction. The deformability behaviour of the particles has been altered due to the conditioning process, part of the particle

material has undergone such a change that it becomes available as binder material. Dependent on the amount of resilience and compressibility of the material, the warm pellet stretches after leaving the die, inducing a certain void volume (porosity) in the pellet. Upon entering the cooler, heat and moisture are removed from the pellet and the binder type in the pellet, most probably, changes from primarily liquid necking and viscous bonding to a more crystalline or glass type of bond in the case of starch rich materials. The exact proportion and contribution of each bond type cannot be deduced from the data in this thesis and remains to be investigated. In figure 2, some model calculations on particle size, binder type and strength, based on Rumpf (1958) and Pietsch (1991) are shown.

In table 3 the range of hardness and durability values is given for the experiments conducted in this thesis. In the experiments in which tapioca as a single ingredient was used, a die with dimensions 12*45 mm was used wheras in the experiments with soy grits and the mixture of soy and tapioca experiment a die with dimensions 5*45 mm was used. The choice of die-size was determined by practical considerations. For the tapioca a low length to diameter ratio of the die had to be used, otherwise no pellets could be formed.

Table 3: Hardness and durability values found in the different quality tests between the experiments described in the chapters 5, 6, 7 and 8.

Chapter	Raw material	Die size [mm]	Durability tests		Hardness tests		
			Holmen [%]	Pfost [%]	Kahl [kgf]	Kramer shear press [N gr^{-1}]	Compression test [N mm^{-1}]
Chapter 5	Tapioca	12*45	1.7 - 98.3	49.6 - 86.8	6.0 - 20.5	n.d.	6.8 - 24.5
Chapter 6	Tapioca	12*45	11.2 - 92.8	59.2 - 96.1	4.9 - 40.6	58.9 - 654.0	4.3 - 14.5*
Chapter 7	Soy-grits	5*45	3.1 - 94.3	65.1 - 97.4	4.2 - 25.9	113.2 - 381.7	5.8 - 14.2
Chapter 8	Soy-grits Tapioca	5*45	64.2 - 96.0	93.8 - 98.4	16.2-32.7	410.6 - 827.9	12.3 - 21.2

n.d. is not determined.

* Due to yielding; not fracture, pellet quality was not tested at maximum inclusion level of SGD.

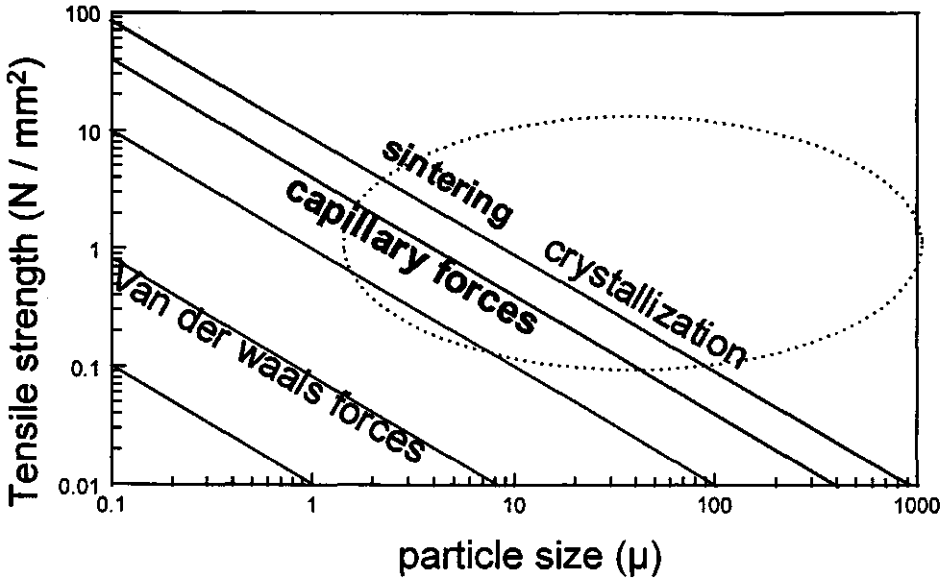

Figure 2: Binder types as a function of particle size. Dashed area indicates bonding types predominantly found in pelleted feed. (After Rumpf, 1958; Pietsch, 1991).

Practical implications.

The results from this thesis show that a large effect is to be expected from the changes in starch and protein components of feed materials on both pellet quality characteristics and systems parameters like energy consumption. Although the used feed model systems are not comparable to feeds encountered in practice, the effects found warrant further investigation on the functionality of ingredients and its effect on the physical characteristics of the pellets. To do this, further research should be conducted on the role of other feed materials which incorporate constituents with that have functional properties related to physical quality of pelleted feeds. For instance, fiber, fat and minerals are constituents with a pronounced influence on the hardness and durability of the derived pellets. In addition, this type of research should be conducted in an area in which the composition of the feeds closely matches that of animal feeds used in practice.

Recent results of Van der Poel *et al.* (1997) show that a considerable effect can be expected on behavior and feed intake pattern in pigs when a choice-feeding system was applied. In this choice-feeding system, pellets where fed which where manufactured from raw materials

which were classified according to three groups; rich in carbohydrates, rich in proteins and a rest group containing the additives and pre-mix. The study of Van der Poel *et al.* (1997) showed that grouping raw materials in for instance groups rich in carbohydrates, protein and mineral rich groups may lead to different pellet hardness and durability. The physical quality of these feed pellets then becomes increasingly susceptible to the functional properties of the main constituents. In order to avoid too extreme differences in pellet hardness and durability, the feeds should be optimized with respect to each group. Especially with the development of new feeding systems, demands on pellet quality will be more strict in terms of a certain hardness or durability that has to be met.

Relation to nutritional effects.

All the chapters have made some mention, although not in detail, of the nutritional consequences of pelleting and/or processing feeds (for example, see Chapter 3). There are *in vitro* techniques which can be used for quick screening of substrates, which can give indications of the most fruitful directions to optimize the feed manufacturing process. In the following example, use was made of the cumulative gas production technique, (see for background on the used methodology; Theodorou *et al.* 1994). In the experiment, described in Chapter 6, unusual phenomena, not commonly observed in feed manufacturing practice had emerged. The appearance of exit-die defects led to the conclusion that a subdivision of starch should be made in native cristallyne starch and an amorphous fraction (gelatinized starch). It is in this amorphous fraction that a glass-transition can be found, which, depending on the moisture conditions and temperatures prevailing during processing, and the absolute amount of amorphous starch present, may lead to the observed exit-die defects as discussed in Chapter 6. These exit-die defects are associated with the altered rheological properties of the material during the processing step itself. It is, however, also known that the amorphous starch fraction may undergo recrystallization, the extent and speed of which is governed by the moisture content and temperatures prevailing during storage (Eliasson and Gudmundsson, 1996). It was therefore hypothesized that during storage, part of the amorphous starch fraction was recrystallized into a form which was either less fermentable, or unavailable, for microorganisms.

Compared to an untreated, native starch control, such recrystallization should lead to an initial increase in speed of degradation due to the higher amount of amorphous starch in the processed starch compared to the control containing ungelatinized (native) starch. Amorphous starch is generally digested faster than the native, more crystalline starch (Gallant *et al.*, 1992). Furthermore, it is assumed that the ungelatinized 'native starch' portion of the

processed material degrades at the same rate as the native starch in the control. Depending on the extent of recrystallization, an undegradable or slowly degradable fraction could be formed which would result in less gas produced or a higher undegradable fraction. It should be re-emphasized that the amount of resistant starch formed is a function of storage conditions and moisture content during storage conditions. How much amorphous starch is formed, depends on the processing conditions.

To test the above hypothesis, two samples of pellets, from treatments 0SP and 100SP (see Chapter 6) which had been in storage (at 4°C) for at least two years, were tested for their fermentability according to the cumulative gas production. Cumulative gas production was measured according to the *in vitro* fermentation method of Theodorou *et al.* (1993, 1994) and modified as described by Williams *et al.* (1995). The data for cumulative gas production (as ml of gas accumulated with time) were fitted iteratively to the model described by Groot *et al.* (1996). There were significant differences between substrates according to the cumulative amount of gas produced per gram dry matter incubated (DMCV), the total amount of gas produced per gram dry matter disappeared (Y_{DM}) and the time at which maximum rate of gas production occurs (t_{RM}) (Table 4). The unprocessed starch led to the production of more gas, in addition the t_{RM} occured later than for the processed material, which may be indicative for the lower amount of amorphous starch present. There was no difference in the maximum rate of degradation nor in the difference in percentage dry matter loss. Some dry matter loss may however have occured since the pore-size of the filter crucibles was between 40 and 100 μm. Although differences were present between the total amount of volatile fatty acids (VFA) and the total amount of VFA produced per gram dry matter disappeared, this was not reflected in the composition of the VFA's (not shown).

Enough circumstantial evidence has been obtained to warrant further investigation into the effects of recristallyzed starch formation as a function of processing- and storage conditions. It should be emphasized that neither of the two samples were optimised to obtain large differences in resistant starch. With use of the concepts proposed by Keetels (1995) and by Eliasson and Gudmundsson (1996) it may be possible to pre-determine the amount of recrystallised starch, and hence to influence the amount of resistant starch formed. Further experiments are required to abandon or adopt the proposed mechanism. In these experiments use should be made of X-ray diffraction, DSC, *in vitro* tests and an *in vivo* evaluation to further test this theory.

Table 4: Cumulative gas parameters and volatile fatty acids produced *in vitro*. For conditions under which the samples have been derived see Chapter 6. (each sample: n=5)

Parameter	Un-processed starch (0SPII) value (±std. dev)	Processed starch (100SPII) value (±std. dev)	Probability Processed vs. Unprocessed
DMCV[a] [ml g^{-1} DM]	413.2 (±6.12)	391.3 (±8.96)	**
Y_{DM}[b] [ml g^{-1} DM disappeared]	459.2 (±11.23)	438.9 (±13.09)	*
$t_{½}$[c] [hours]	5.48 (± 0.37)	4.99 (± 0.31)	#
t_{RM}[d] [hours]	6.60 (±0.51)	5.81 (±0.47)	*
DM loss [%]	90.0 (±1.12)	89.2 (±0.72)	ns
Total VFA[e]	10.8 (±0.19)	9.82 (±0.49)	**
Yield VFA[f]	12.05 (±0.11)	11.02 (±0.55)	**

ns = P>0.1; #=P=<0.1; * = P < 0.05; ** = P<0.01; *** = P<0.001.

[a] Cumulative volume of gas produced per gram dry matter incubated.

[b] Total gas produced per gram dry matter disappeared.

[c] Time at which half the asymptotic amount of gas is produced.

[d] Time at which maximum gas production rate occurs: $B*(C-1)^{1/C}$ (Groot *et al.* 1996).

[e] Total VFA per gram DM.

[f] Total VFA per gram DM disappeared.

Conclusions

From the various chapters in this thesis and the general discussion, the following general conclusions can be adopted:

1) Hardness and durability of pelleted feeds are to a large extent determined by the functionality of the feed ingredients.
2) PDI and the degree of starch gelatinization as determined with an enzymatic method (SGD) are good indicators for the functionality of a diet ingredient with respect to hardness and durability values of the feed when the feed is subjected to pelleting.
3) Changes in PDI and SGD due to feed processing are more affected by the amount of water and steam added in the manufacturing process than by the amount of dissipated mechanical power or shear in the equipment.
4) Pellets become harder and more durable when soy with a high PDI is incorporated in the feed.
5) Pellets become harder and more durable when tapioca with a high degree of gelatinization is incorporated in the feed.

6) No uniqe model, or family of models, seems to exist by which through simple manipulation of the parameters PDI or SGD, a prediction of the physical quality can be made.
7) No interactions exist between the PDI and SGD with respect to pellet quality parameters and system parameters given.
8) In order for a model to be succesfull in predicting the physical quality of feed, at least the following aspects should be incorporated:

- particle size and particle size distibution
- deformability of the particles
- the effect of water, heat, shear and residence time in the various unit operations on the deformability of particles and the amount of binder material formed
- determination of the type of bond formed as a function of the above mentioned parameters
- determination of the strength of the bond formed as a function of the above mentioned parameters.

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Summary

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Introduction

The aim of the feed manufacturing industry is to provide a safe, hygienic and nutritionally balanced feed for the different categories or different species of animals. A large part of this feed is fed to these animals in pelleted form. In order to do so, various unit operations are used to obtain the quality standards adopted by farmers and animals. Although the last decade has shown a rise in different types of feed forms used, like expandates and crumbled feed, it is still common practice to make use of pelleting equipment. The advantage of using agglomerated feeds is that it fixes nutrient composition so that every animal receives its nutrients in the correct proportion. In addition, handling and storage of the feed are greatly enhanced when pelleted feeds is used. Apart from the advantages of an additional pelleting or agglomeration process there are also disadvantages associated with the use of pelleting equipment. First of all, pelleting is an additional process, since in terms of nutrient composition, already a complete feed exists after milling and blending of the raw materials. The conditioning, pelleting and cooling line consumes about 60% of the total energy consumption in a feed factory. In addition, due to the thermal load on the feed, valuable heat-labile components like some additives, vitamines or added enzyme's may be inactivated. Still, there seems to be more advantages associated with the pelleting process than disadvantages since the largest portion (between 85 and 90%) of the feed is sold in pelleted form.

In order to withstand the rigors of handling, pellets need to have a certain structural integrity. A lot of work has been focussing on the effect of the different processing variables and their relation with hardness and durability of the pelleted feeds. Although pellet press operators are aware of the effects of raw materials and their effect on the physical quality of pellets, means of influencing these raw materials are very limited. In addition, feed composition is dictated by economic factors and nutritional requirements, which may lead to unpelletable feed rations. This study has investigated the effect of changes in the raw materials and the pellet quality of model feeds derived from these raw materials. Specifically, changes in degree of gelatinizaton of the starch fraction and dispersibility of the protein have been used as indicators for functionality and their effect on pellet hardness and durability.

The study can be subdivided in three parts. In the first part, the Chapters 1, 2 and 3, an overview is given on the methods to determine pellet quality and what causative factors are contributing to the structural integrity of pellets (Chapter 1). What the contribution is of processes and processing conditions with respect to pellet quality is discussed in Chapter 2. In the last chapter of the overview, the effects of raw materials and raw material constituents with respect to pellet quality are discussed (Chapter 3).

In the next two chapters; Chapter 4 and Chapter 5, two experimental studies are described in which the effect of processing conditions on changes in functionality parameters of protein and starch have been studied. In the last three chapters (Chapters 6, 7 and 8), experimental studies are described in which the effect of functional changes in the protein and starch fraction have been related to pellet hardness and durability characteristics. It is concluded that changes in protein dispersibility and the degree of gelatinization correlate well with the ultimate pellet quality. Increasing the amount of gelatinized starch and dispersible protein leads to harder and more durable pellets, although large differences may occur due to the important other factors as for instance particle size and moisture content.

Physical quality of pelleted animal feeds. An overview from literature.

From Chapter 1 it is concluded that the pre-dominant type of bonding in pellets is due to solubilisation and subsequent crystallisation of feedstuff components e.g. starch, sugars, fats or 'liquid necking'. Liquid necking is a binding mechanism which uses the surface tension of water, in a three phase system of air, water and particles to maintain structural integrity of the pellet. Soluble components might be introduced in the feed mash before pelleting in the mixing phase or result from the processing stages, conditioning and pelleting.

To evaluate hardness and durability of pelleted feeds, generally a subdivision is being made into tests that evaluate 'hardness' and tests evaluating 'durability' of a given (set of) pellet(s). Several devices measuring fragmentation strength, and devices determining abrasion strength of pellets have been discussed in Chapter 1. The most important conclusion was that most tests presently available, and used in feed manufacturing, evaluate a mixture of hardness (fragmentation) and durability (abrasion) effects. Therefore, the feed manufacturer or feed technologist should be aware of the physical requirements for the pellets, and based on these requirements, a choice must be made for the most suitable testing method, since no unique test exists that covers all parameters of interest related to the physical quality of pelleted animal feeds.

In Chapter 2, the effects of changes in process parameters and their effect on pellet quality in terms of hardness and durability have been discussed. The parameters discussed with respect to the conditioning process, are process variables such as steam and water and system parameters such as residence time and pressure. Parameters during the pelleting process that can be adjusted or influence pelleting properties of a feed mash are layout (e.g. flat-bed vs. ring-die pellet press) and dimensions, roller and die assembly and die velocity of the pellet press. It is concluded that the effect of the changes in one or more parameters and its effect on pellet quality (durability and hardness) is often a matter of judgement and experience of the

operator. It is concluded that the amount of steam is a more decisive factor than steam pressure. In addition, it seems that individual steam supply of a factory has a larger influence on the measured pellet hardness and durability than would be expected from theoretical relationships concerning steam in an ideal situation. Although water has binding properties as well, it is concluded that the use of steam over water is by far superior to produce good quality pellets. The additional heat included in the meal permits changes in physico-chemical properties which lead to more durable and hard pellets. In addition, it is concluded that equipment which incorporates some form of hold-time enhances the possibility to incorporate more liquids, without detrimental effects on pellet quality. The use of pressure to alter physico-chemical properties of the feed in combination with water and heat, and the use of pressure to pre-densify the feed mash prior to pelleting seems to be important in obtaining a good quality pellet.

In Chapter 3 an overview is given of effects of diet ingredients and their composition (e.g. starch, protein, sugar, fat and fibre content) on production characteristics of pellets and on their effects on physical pellet quality: pellet hardness and pellet durability.

Within and between groups of diet ingredients, large differences exist in their effect on physical quality of pellets, when incorporated in animal diets. Differences in pellet quality between groups of diet ingredients, e.g grains or legume seeds, can be attributed to differences in their physico-chemical properties. These are in turn primarily affected by processing history, geographical and climatical origin and cultivar. Differences in physico-chemical properties due to the latter factors are also the main causative contributors to explain differences in pelleting quality (e.g. throughput, energy consumption of the pellet press) within groups (e.g. cereals or legume seeds) and thus in the resulting pellet quality. Effects of raw material constituents, both their level and physico-chemical properties may provide more information on pelleting characteristics and pellet quality than the diet ingredient inclusion level of the raw material per se. The effects of starch (native versus gelatinized), sugar, protein (raw versus denatured) and solubility and resiliencenes of fibre are discussed with respect to pellet quality. When pellet hardness or durability is lacking, pellet binders may be used to improve pellet quality. The effects of pellet binders and their mode of action are discussed.

It is concluded that more research effort should be directed towards the effects of individual constituents and their respective properties, since the latter seems to affect to a large extent the final hardness and durability of pelleted compound feeds. Moreover, the properties of a specific constituent reflect the processing history of that specific ingredient. By relating pellet quality to physico-chemical properties, e.g. functionality of the constituent, the manufacturer of compound animal feeds will be able to decrease the variability in final pellet quality caused by differences in geographical origin and processing history of the diet

ingredients. Objective criteria from animals are still necessary to evaluate pellet quality as far as nutritional quality is concerned.

Processing conditions affect functional properties

In Chapter 4, the effect of adding steam (at 0.6 and 1.2 bar) and water (1.9% and 64% of the flux of soy grits) in a conventional barrel type conditioner and its effects on protein dispersibility index (PDI), nitrogen solubility in 0.2% KOH and trypsin inhibitor activity (TIA) of commercial soy grits was studied. In addition, conditioned soy grits were subjected to expander treatment at two different screw speeds (2 and 3 rps) and PDI, NSI and TIA was measured. Analysis of variance was used to determine the effects of steam pressure, water addition and screw speed on the mentioned protein quality parameters. The resultant temperature from steam addition was used in an analysis of covariance to determine the effects of temperature as a covariate, water addition and screw speed on protein quality parameters. A significant interaction for steam pressure and water addition was found on all parameters. The highest denaturation or inactivation of the protein was found at the combination incorporating high steam and high water addition. Screw speed during expander treatment did not significantly affect the protein quality parameters. It was concluded both from this study and from literature that PDI is a good parameter to evaluate technological treatments when low amounts of motor power (<110 kJ kg^{-1}) are dissipated. NSI in 0.2% KOH is a better parameter to discriminate between technological treatments when moderate to high amounts of motor power are dissipated in soy grits.

In the study described in Chapter 5, the effect of processing conditions during the manufacture of pelleted animal feed was related to the degree of gelatinization of tapioca starch as measured by the Amyloglucosidase method (SGD) and Differential Scanning Calorimetry (DSC). The used process-conditions were related to some physical quality characteristics of the pelleted feeds as well. Physical pellet quality was evaluated for hardness and durability which incorporated tests that are used in the feed industry. The processing line used in the experiment, consisted of a conventional barrel type conditioner where steam pressure (100 - 180 kPa) and tap water addition were varied (0 - 52 g kg^{-1} of the feed debit). Subsequently, expander processing was carried out and the screw speed of the expander was varied (60 - 140 rpm) as well as the amount of dissipated (expander) motor power (1.3 - 5.7 kW). Response surface regression methodology was used to assess the directions and relative magnitude of changes in processing conditions on starch modification, physical pellet quality and some variables like energy consumption of the pelletizer. The results show that, with the factors used in this study, no combination can be found that satisfies one common maximum

or one common minimum value for all of the dependent variables tested. Hardness and durability values of the pellets are most affected by steam pressure and the amount of expander motor power used, whereas starch modification was most affected by steam pressure and water addition.

Functional properties of starch and protein affect hardness and durability of pelleted feed.

In a dose-response design, described in Chapter 6, the effects of an increase in the amount of gelatinized starch in a model feed mash was investigated on hardness (Kahl device, compression test and Kramer shear press) and durability (Holmen and Pfost device) characteristics of the pelleted feed. Model feeds were either steam pelleted (SP) or cold pelleted (CP). It was hypothesized that an increase in the amount of gelatinized starch present, would lead to harder and more durable pellets. Results showed that, due to a pre-processing step, the effects of particle size of the feed mash (109 μm to 315 μm) and water content (12.8 % to 16.9 %) were confounded with the degree of gelatinization (ranging from 11.5% to 53.1%) of the starch fraction, as measured with the amyloglucosidase method (SGD). These factors affected the measured characteristics, as was deduced from literature. Broken line regression models were used to summarize the data. Initially, a decrease in hardness and durability of the pellets was found with increasing SGD, up to an intersection point, after which a rapid incline in hardness and durability was observed with further increasing SGD values. The appearance of exit-die defects, past these intersection point(s), associated with tough and durable pellets, led to the conclusion that part of the starch fraction had undergone a glass transition from a brittle to a rubbery state. This may have accounted for the different rheological properties, which in turn induced exit-die defects. It is concluded that, in order for these exit-die defects to appear, two requirements should be met. First, the temperature at which the glass transition takes place must be below the processing temperature. Secondly, the amount of amorphous starch, which can exhibit this glass transition, must be large enough to become the predominant phase in terms of rheological behavior of the feed mash. However, since the effects of particle size and moisture content are confounded with SGD it is not possible to unequivocally determine the absolute magnitude of the different factors with respect to the measured characteristics. No differences were found between CP and SP before the intersection point on most hardness tests. No difference was found in energy consumption, mass debit and the appearance of exit-die defects between CP and SP. After the intesection point, SP led to higher pellet hardness and pellet durability when compared to CP. It is concluded that in experiments evaluating the effect of gelatinized starch on hardness and

durability, particle size and moisture content should remain equal for all batches.

In Chapter 7 the effect of differences in protein dispersibility index (PDI) in model feeds were evaluated with respect to pellet hardness and pellet durability at two temperature levels. It was hypothesized that a gradual replacement of denatured protein by native protein will lead to harder and more durable pellets. In addition, the effect of temperature before entering the pellet press (65°C and 85°C) was determined on pelleting properties of the model feeds. Non-linear regression was used to evaluate the effects of PDI on hardness and durability of the pelleted feeds. Durability (%), feed flux (kg h^{-1}) and Specific Mechanical Energy (SME; kJ kg^{-1}) were found to follow a curvi-linear (sigmoidal) relationship with PDI. Hardness parameters, Kahl (kgf), Kramer Shear Press (N gr^{-1} pellet) and Compression tests (N mm^{-1} pellet length) were found to follow an exponential relationship as a function of PDI. Pellets were harder and more durable with increasing PDI. Feed flux and SME were found to decrease with increasing PDI. Porosity of the pellets was determined and found to be linearly related with strength of the produced feed pellets. Pellet porosity ranged between 0.088 and 0.136. Lower porosity correlates with higher strength and vice versa. The 85°C temperature level led to harder and more durable pellets in all cases, as opposed to the 65°C temperature level. NSIkoh values were also determined as a second indicator of protein quality and these values became higher after processing, in contrast to what is expected.

In Chapter 8, a model feed system (50% soy / 50% tapioca w/w) was used to determine whether or not changes in protein quality and starch degree of gelatinization affected the physical quality in terms of hardness and durability of the manufactured pellets. Protein quality was determined by the protein dispersibility index (PDI). Degree of starch gelatinization was determined from the enzymatic amyloglucosidase test (SGD). In addition, correlations are given for additional tests as differential scanning calorimetry (DSC), and nitrogen solubility in 0.2% KOH (NSIkoh) for the starch/protein and protein properties, respectively. SGD and DSC values for the starch properties were negative and highly correlated (<-0.80). Correlations between PDI and NSIkoh were high (>0.87; $p<0.001$) and correlations between these protein quality parameters and DSC were low (<0.35; $p>0.05$). The low correlations between the protein quality test and the DSC-values can be partly attributed to the smaller peaks, emerging from the protein. These peaks are near the limit of detection for the DSC and hence induce a large spread in values. In addition, part of the protein denatures in the same range as starch gelatinizes. Hence, this protein peak is obscured by the larger peak found for starch. It is hypothesized that a high amount of soluble protein and a high amount of gelatinized starch positively affects hardness and durability properties of the pelleted feed. A number of physical quality tests as used in the feed industry (Holmen and Pfost durability and Kahl hardness) as well as some other testing devices (Kramer shear press and a general

compression and tension tester) were used to determine hardness and durability values of the manufactured pellets. Results were analyzed using response surface methodology. In addition, an apparent modulus was calculated from the values derived from the compression test. Percentage strain before fracture of the pellets was calculated from the values of the compression test. It was concluded that the apparent modulus of the tested pellets was closely related to hardness properties of the pellets and strain was related to friability properties of the pellets. A maximum was found within the limits of the experimental conditions for the Pfost and Holmen durability and strain values. Porosity, strain, Holmen- and Pfost durability was most sensitive to changes in PDI values of the feed mash, whereas specific mechanical energy (kJ kg^{-1}) of the pelletizer and capacity of the installation was most sensitive to changes in SGD values. In all cases, the hardest pellets, and highest apparent modulus, was found at the maximum inclusion level of native protein (high PDI) and gelatinized starch (high SGD). Decreasing values for PDI and SGD led to lower values for the hardness parameters and a lower apparent modulus. Minimum values, within the experimental limits, were found for porosity and Specific Mechanical Energy values. This study shows that large effects can be expected depending on the state of the protein and starch fractions in the raw materials used and this study is therefore relevant to practical feed manufacturing.

General remarks

Overall, from the results in this thesis it can be concluded that the functional properties of the protein fraction and the starch fraction make a significant contribution towards the pellet hardness and pellet durability. The methods, primarily used in this study (Protein Dispersibility Index (PDI), enzymatically determined degree of gelatinization (SGD), are good indicators for the functionality of the protein and starch fraction in the feed, respectively. It should be noted that these methods may not give an adequate prediction in all circumstances, since this study also suggests that PDI and SGD is the sum of a certain number of processes in the protein and starch fraction that relate very well to deformability and binding properties of the feed particles. These latter factors eventually may be the causative factors determining the pellet hardness and durability.

The protein and starch fractions themselves can be altered during feed processing. The extent of alterations of these fractions seems to be governed by the amount of water and steam added, rather than by dissipation of mechanical energy or shear. However, pellet hardness and durability not only depend on the functional properties of the feed constituents, but are also affected by steam added and the amount of dissipated mechanical energy. The extent to which they can be controlled in the different unit operations will eventually determine the use of the

concept of functional properties in the feed manufacturing industry. The extent to which they must be controlled, will be very much dependent on the objectives put forth by the feed manufacturer. From the Chapters 4 and 5, it can be concluded that the factors studied, comprise a large proportion of the domain over which the functionality of the constituents can be altered. By including other factors, such as residence time, controllability of the functional properties of the feed mash may be further enhanced. This requires further research since, for instance, residence time as such has not been studied in this thesis.

Pellet hardness and durability increases when soy high in dispersible protein is added. Although not explicitly tested in this work, this seems to be largely associated with a better deformability of the particles containing native protein over denatured protein.

Pellet hardness and durability were also improved when starch is used which is (pre-) gelatinized. As with protein, although not explicitly tested, this seems due to the starch containing particles becoming better deformable. Moreover, (pre-) gelatinized starch may act as a binder. The appearance of so-called exit-die defects is associated with the possibility of the amorphous fraction of the starch to undergo the so-called glass-transition in which the amorphous fraction goes from a brittle and relative hard material to a rubbery-like state. When enough of this amorphous starch is present (approximately 30% according to results from this thesis), and the processing temperatures are above the glass transition temperature, then these exit-die defects may occur.

One of the first hypothesis in the thesis was that an increase in dispersible protein and gelatinized starch is associated with harder and more durable pellets. Although enough evidence has been obtained that this is the case in situations where particle sizes and moisture content of experimental feeds are similar (Chapter 7 and 8), as soon as particle size and moisture content are not equal, these effects tremendously influence the outcome of the physical quality characteristics (Chapter 6). From the various results of this thesis and literature data available it is concluded that no uniqe model, or family of models, seems to exists by which through simple manipulation of the parameters PDI or SGD, a prediction of the physical quality can be made. Furthermore it can be concluded that: in order for a model to be succesfull in predicting the physical quality of feed, at least the following aspects should be incorporated in such a model (as derived from this thesis):

- particle size and particle size distibution,
- deformability of the particles,
- the effect of water, heat, shear and residence time in the various unit operations on the deformability of particles and the amount of binder material formed,
- determination of the type of bond formed as a function of the above mentioned parameters,
- determination of the strength of the bond formed as a function of the above mentioned parameters.

Samenvatting

M. Thomas

Inleiding

Een van de doelstellingen van de mengvoeder industrie is het produceren van een veilig, hygiënisch en nutritioneel gebalanceerd diervoeder. Om aan deze doelstellingen én de door de agrarisch ondernemer en het dier gestelde kwaliteitseisen te kunnen voldoen, wordt een heel scala van bewerkingen op de grondstoffen uitgevoerd. In het laatste decennium zien we, dat behalve deze eisen ook een grotere verscheidenheid aan voedervormen ontstaan, zoals expandaten en kruimelvoeders, maar ook nieuwe voersystemen zoals het deelpellets systeem en keuze-voederingssystemen. Een groot deel van het mengvoeder wordt echter nog verstrekt in gepelleteerde vorm. Eén van de voordelen in het gebruik van ge-agglomereerde voeders (zoals pellets) boven het gebruik van melen is dat in een ge-agglomeerd voeder de verhouding tussen de verschillende nutriënten wordt gefixeerd, waardoor elk dier een gebalanceerde hoeveelheid nutriënten binnenkrijgt. Verder worden de hanteerbaarheid en stromingseigenschappen in silo's gunstig beïnvloed door het gebruik van ge-agglomereerde voeders. Er zijn echter ook nadelen verbonden aan het pelleteren van diervoerders. Ten eerste, pelleteren is een additioneel proces, aangezien er na het proces van malen en mengen al een volledig diervoeder is geproduceerd. Echter het conditioneer-, pelleteer- en koelproces neemt wel ongeveer 60% van de energie consumptie in een mengvoederfabriek voor zijn rekening. Verder kan de thermische belasting in de diverse onderdelen van de installatie een negatieve invloed hebben op de nutritionele kwaliteit van het voer. Waardevolle componenten zoals vitaminen of toegevoegde enzymen kunnen ten dele worden geïnactiveerd. Maillard reacties kunnen ontstaan met negatieve gevolgen voor de voederwaarde. Er zijn echter nog steeds meer voordelen dan nadelen verbonden aan het pelleteerproces, aangezien het aandeel gepelleteerd voeder in de totale verkoop van mengvoeders in Nederland al jaren tussen de 85 en 90 % schommelt.

Om het voortijdig uiteenvallen van de pellets tijdens transport en hantering te voorkomen moeten deze een zekere mate van sterkte bezitten. Veel studie is dan ook gedaan naar het effect van diverse procesvariabelen en de relatie hiervan met de hardheid en slijtvastheid van pellets. Alhoewel pers-operators bekend zijn met het soms grote effect van grondstoffen op de fysische kwaliteit van de pellets zijn hun middelen om invloed uit te oefenen op de grondstofeigenschappen beperkt. In alle gevallen wordt de samenstelling gedicteerd door economische en nutritionele factoren. Dit leidt ertoe dat in sommige gevallen de korrelkwaliteit van de diervoeders niet kan worden gewaarborgd.

In dit proefschrift wordt aandacht besteed aan het effect van grondstofeigenschappen en de veranderingen die hierin kunnen optreden, met name in eiwit en zetmeel, en de relatie ervan met de fysische kwaliteit van de geproduceerde pellets.

Het proefschrift kan in drie stukken worden onderverdeeld. In de hoofdstukken 1, 2 en 3 is een overzicht beschreven van de literatuur die is verschenen omtrent het pelleteren van diervoeders. In het eerste hoofdstuk wordt een overzicht gegeven van de beschikbare meetmethodieken voor de evaluatie van de fysische kwaliteit van diervoeders en wordt een overzicht geschetst van de verschillende bindingtypen tussen voerdeeltjes die een rol spelen bij het totstandkomen van een zekere hardheid en slijtvastheid van het voer. In het tweede hoofdstuk wordt een overzicht geschetst van de bijdrage van de verschillende processen en procescondities aan de fysische kwaliteit van pellets. In het derde hoofdstuk worden de effecten van grondstoffen en grondstofcomponenten en hun bijdrage aan de fysische kwaliteit van gepelleteerde voeders besproken.

In de volgende twee hoofdstukken, 4 en 5 zijn twee experimenten beschreven waarin het effect van proces condities op veranderingen in functionele eigenschappen (in dit geval die eigenschappen in grondstoffen die een bijdrage leveren aan de totstandkoming van een zekere fysische korrelkwaliteit) van eiwit en zetmeel zijn bestudeerd.

In de laatste drie hoofstukken zijn drie experimenten beschreven waarin het effect van functionele veranderingen in de eiwit- en zetmeel fractie zijn bestudeerd in relatie tot de hardheid en slijtvastheid van pellets. Uit deze hoofdstukken is geconcludeerd dat veranderingen in de dispergeerbaarheid van het eiwit en veranderingen in de mate van ontsluiting van het zetmeel gerelateerd zijn aan de uiteindelijke hardheid en slijtvastheid van pellets. Een toename in de mate van dispergeerbaar eiwit en ontsloten zetmeel leidt tot een verhoogde pellethardheid en slijtvastheid. Echter, de effecten van bijvoorbeeld deeltjesgrootte en vochtgehalte in het voer zijn dermate groot dat deze er toe kunnen leiden dat het effect van eiwit dispergeerbaarheid en ontsluitingsgraad van zetmeel wordt gemaskeerd.

De fysische kwaliteit van gepelleteerde diervoeders. Een literatuur overzicht.

Uit hoofdstuk 1 is geconcludeerd dat de bindingstypen in gepelleteerde diervoeders overwegend het gevolg zijn van oplosbaarheids- en herkristallisatie eigenschappen van grondstofcomponenten, zoals bijvoorbeeld zetmeel, suikers, vetten of het gevolg zijn van vochtnekjes. Vochtnekjes is een bindingstype waarin, binnen een drie fasen systeem van vocht, water en deeltjes, door middel van de oppervlaktespanning van het water, de deeltjes worden bijeengehouden en ze een bijdrage leveren aan de sterkte van de pellets. Oplosbare materialen kunnen worden ingemengd in het voer of ontstaan ten gevolge van de verschillende processtadia, die het voer doorloopt.

Om de fysische kwaliteit van gepelleteerde diervoeders te evalueren wordt in de mengvoeder industrie in het algemeen een onderverdeling gemaakt in hardheidstesten en slijtvastheids testen. Een aantal verschillende toestellen die hardheids- en

slijtvastheidskarakteristieken weergeven, zijn beschreven in hoofdstuk 1. De voornaamste conclusies uit dit literatuur onderzoek is dat de meeste testen die worden gebruikt in de veevoederindustrie een mengeling meten van fragmentatie en afslijting. Verder is duidelijk geworden dat er niet één methode bestaat waarbinnen alle parameters van belang voor de fysische kwaliteit van pellets, kan worden gemeten. Daarom moet een mengvoederfabrikant of voertechnoloog een keuze maken uit een aantal testen, die het beste die parameter(s) weergeven waarin hij of zij is geïnteresseerd.

In hoofdstuk 2 worden de effecten van de proces-parameters tijdens conditioneren en pelleteren beschreven. Onder andere worden de effecten van stoom en water, alsmede verblijftijd en druk tijdens conditioneren beschreven. Bediscussieerde parameters die een rol spelen bij het pelleteren zijn onder andere perstype (met vlakke plaat matrijs of ring-matrijs), matrijs en rol-samenstelling en matrijs-snelheid. Er is geconcludeerd dat de hoeveelheid stoom een meer belangrijke maat lijkt te zijn dan de stoomdruk. Met het gebruik van stoom wordt ook water geïntroduceerd in het persmeel. Alhoewel water als zodanig ook bindende eigenschappen kan introduceren in mengvoeders is geconcludeerd dat het gebruik van stoom tot een betere hardheid en slijtvastheid van de pellets leidt. De reden hiervoor is gelegen in het feit dat met stoom ook warmte wordt toegevoegd aan het mengsel, waardoor de functionele eigenschappen van het voer dusdanig veranderen dat de pelletkwaliteit positief wordt beïnvloed. Een verhoging van de verblijftijd in de conditioneerfase of het gebruik van druk en afschuiving leidt in het algemeen ook tot hardere en meer slijtvaste pellets.

In hoofdstuk 3 wordt een overzicht gegeven van het effect van verschillende grondstoffen en hun componenten (bijvoorbeeld zetmeel, eiwit, vezels en vetten) op hardheids- en slijtvastheidskenmerken van pellets. Er bestaan grote verschillen tussen groepen en binnen groepen van grondstoffen (tussen groepen bijvoorbeeld grondstoffen van plantaardige oorsprong of grondstoffen van dierlijke oorsprong, binnen groepen, bijv. tussen tarwe en gerst). Verschillen in korrelkwaliteit tussen groepen kunnen voornamelijk worden toegeschreven aan verschillen in fysisch-chemische eigenschappen. Deze fysisch-chemische eigenschappen op hun beurt ontstaan als gevolg van de verschillende procesbewerkingen die deze grondstoffen hebben ondergaan, geografische en klimatologische omstandigheden en ras-eigenschappen. Verschillen in fysisch-chemische eigenschappen als gevolg van deze factoren zijn dan ook de voornaamste redenen voor geïnduceerde verschillen in pers eigenschappen (doorzet, energie-consumptie) binnen groepen (bijvoorbeeld granen en peulvruchten) en hardheid en slijtvastheid van de pellets.

Geconcludeerd is dat de samenstelling van een grondstof in termen van eiwit, zetmeel en vezel én de fysisch-chemische eigenschappen van deze componenten een grotere bijdrage kan leveren in de kennis van het pelleteergedrag en de ontstane fysische pellet kwaliteit, dan het inmengings percentage van deze grondstof in het mengvoer op zich. Een beschrijving is dan

ook gegeven van het effect van zetmeel (natief en ontsloten), eiwit (natief en gedenatureerd), suiker en oplosbaarheids- en veerkrachtigheid van vezel, met betrekking tot de korrelkwaliteit. Aansluitend is de rol van bindmiddelen en hun werkingsmechanisme besproken. Er is geconcludeerd dat het gerechtvaardigd is om meer aandacht te besteden aan de functionele eigenschappen van individuele grondstof-componenten, aangezien deze voor een groot deel de uiteindelijke fysische kwaliteit van het gepelleteerde mengvoer verklaren. Door tijdens het samenstellen van voeders rekening te houden met deze eigenschappen, kan de variatie in de korrelkwaliteit worden verkleind. Objectieve criteria uit dierproeven zijn nodig om de nutritionele kwaliteit van de geproduceerde diervoeders en de relatie met fysische kwaliteit te evalueren.

Functionele eigenschappen worden beïnvloedt door proces omstandigheden.

In hoofdstuk 4 worden de effecten van stoom- (160 en 220 kPa) en water-toevoeging in een conditioner (1.9% en 64% van de massa-stroom soja-grits) bestudeerd op de eiwit dispergeerbaarheids index (Protein Dispersibility Index, PDI [%]), stikstof oplosbaarheids index in kaliloog (Nitrogen Solubility Index in 0.2% KOH, NSI [%]) en trypsine remmer activiteit (Trypsin Inhibitir Activity, TIA [mg g^{-1} droge stof]). Aansluitend zijn de geconditioneerde soja-grits onderworpen aan een expander behandeling waarbij twee verschillend schroefsnelheden zijn gebruikt (120 en 180 omwentelingen per minuut) waarna opnieuw PDI, NSI en TIA zijn bepaald. Variantie analyse is gebruikt om de effecten van schroefsnelheid, stoomdruk en watertoevoeging te analyseren. In een co-variantie analyse is het effect van temperatuur als co-variabele, water toevoeging en stoomdruk gemeten op de eiwitkwaliteit parameters. Er bestonden interacties tussen stoomdruk en watertoevoeging voor alle eiwitkwaliteit parameters. De hoogste mate van denaturatie of inactivatie werd gevonden bij de combinatie stoom, 1.2 bar en water 64%. De schroefsnelheid tijdens de expanderbehandeling levert geen bijdrage aan het verklaren van de eiwitkwaliteit parameters. Uit deze studie en uit literatuurgegevens is geconcludeerd dat PDI een betere parameter is om technologische behandelingen te evalueren wanneer lage hoeveelheden mechanische energie wordt opgenomen (<110 kJ kg^{-1}) en de NSI is een betere parameter wanneer matige tot hoge hoeveelheden mechanische energie wordt opgenomen.

In de studie beschreven in hoofdstuk 5, worden de effecten van de proces condities stoom druk (in de range van 100 - 180 kPa) en water toevoeging in de conditioner (in de range van 0 - 52 g kg^{-1} van de massastroom) bestudeerd op de mate van ontsluiting van tapiocameel. Aansluitend is het geconditioneerde tapioca meel onderworpen aan een expander behandeling waarbij de schroefsnelheid van de expander was gevarieerd (in de range van 60 - 140 omwentelingen per minuut) alsmede de hoeveelheid opgenomen mechanisch vermogen (in de

range van 1.3 - 5.7 kW) zijn gevarieerd. De gebruikte meetmethodieken ter bepaling van de ontsluitingsgraad van het zetmeel waren een enzymatisch methode, waarbij gebruik is gemaakt van het enzym amyloglucosidase, en Differential Scanning Calorimetry (DSC). De gebruikte procescondities zijn gerelateerd aan enige fysische kwaliteitskenmerken gemeten door middel van in de mengvoederindustrie gebruikelijke testen als de Holmen en Pfost slijtvastheids test en de Kahl hardheids test. Verder is gebruik gemaakt van een druk- en trekbank ter bepaling van de breuksterkte van de pellets. Response surface analyse is gebruikt om de richtingen en relatieve sterkte van de procescondities op de ontsluitingsgraad van zetmeel, hardheid en slijtvastheid en energie consumptie van de pers vast te stellen. De resultaten laten zien dat er geen uniek minimum of maximum kan worden gevonden voor de vier factoren. De gebruikte factoren leiden in alle gevallen tot zgn. zadelcurves. Geconcludeerd is dat de hardheid en slijtvastheid van de pellets het sterkst afhankelijk zijn van de stoomdruk en gebruikte hoeveelheid motorvermogen van de expander, terwijl zetmeel ontsluitingsgraad het meest wordt beïnvloedt door stoomdruk en water toevoeging.

Functionele eigenschappen van zetmeel en eiwit beïnvloeden de hardheid- en slijtvastheid van pellets.

In een dosis-respons proef, beschreven in hoofdstuk 6, zijn de effecten van een toename in de hoeveelheid ontsloten zetmeel op de hardheid- (Kahl hardheid en breuksterkte gemeten met behulp van de duw en trekbank en de Kramer shear press) en de slijtvastheidskenmerken (Holmen en Pfost slijtvastheid) van pellets beschreven. Hierbij is een model voersysteem gebruikt bestaande uit mengsels van voorbehandelde en natieve tapioca. Deze model voeders zijn met en zonder stoom geconditioneerd (respectievelijk genaamd SP en CP) en aansluitend gepelleteerd. De hypothese luidde dat een toename in de hoeveelheid ontsloten zetmeel leidt tot hardere en slijtvastere pellets. De resultaten lieten echter zien dat door de voorbehandeling ter ontsluiting van het zetmeel, het effect van deeltjesgrootte (109 - 315 μm) en vocht hoeveelheid (12.8% - 16.9%) verstrengeld waren met de ontsluitingsgraad van het zetmeel gemeten volgens de amyloglucosidase methode (SGD). Dit beïnvloedde de gemeten karakteristieken in hoge mate, zoals uit de data en uit literatuurgegevens is geconcludeerd. Gebroken lijnen regressie modellen werden gebruikt om de data samen te vatten. Initieel werd, met een toename in SGD, een verlaging gevonden van de hardheid en slijtvastheids kenmerken. In alle gemeten pellet karakteristieken werd één of meerdere snijpunten gevonden waarna de hardheid en slijtvastheid zeer sterk toenamen. Het verschijnen van zogenaamde 'exit-die defects' leidde tot zeer taaie en slijtvaste pellets op basis waarvan werd geconcludeerd dat een gedeelte van het amorfe zetmeel een glas-overgang heeft ondergaan van een broze naar een elastische toestand. De resultaten uit deze studie suggereren dat

wanneer voldoende amorf zetmeel aanwezig is, en de temperatuur waarbij de behandelingen plaats vindt boven de temperatuur ligt van de glas-overgang, dit kan leiden tot een fase-omkering in het materiaal waarbij de rheologische eigenschappen van het materiaal zeer sterk veranderen. Echter, aangezien de deeltjesgrootte en vochtgehalte verstrengeld waren met de ontsluitingsgraad van het zetmeel is het niet goed mogelijk om de afzonderlijke bijdrage van de factoren aan de gemeten karakteristieken (hardheid, slijtvastheid, porositeit en percentage pellets wat 'exit-die defects' vertoont) te bepalen. Geen verschillen werden waargenomen in hardheid voor de meeste testen, vóór het bereiken van het snijpunt tussen CP en SP; na dit snijpunt wel. Er zijn geen verschillen gevonden in energie-verbruik van de korrelpers, in de massa-stroom en het verschijnen van 'exit-die defects' tussen CP en SP. Uit dit experiment is naar voren gekomen dat bij het bestuderen van het effect van ontsloten zetmeel op de hardheid en slijtvastheid van pellets, rekening moet worden gehouden met de deeltjesgrootte en het vochtgehalte van de model voeders waarmee deze proeven worden uitgevoerd. Dit moet zoveel mogelijk gelijk worden gehouden.

In hoofdstuk 7 zijn de effecten van verschillen in eiwit dispergeerbaarheid (PDI) in model voeders onderzocht in relatie tot hun hardheid en slijtvastheid. Dit is gedaan bij twee temperatuurniveau's, namelijk 65°C en 85°C. De hypothese luidde dat een graduele vervanging van de hoeveelheid gedenatureerd eiwit (lage PDI) door eiwit dat beter dispergeerbaar is in water (hoge PDI) leidt tot een hardere en slijtvastere pellet. Verder is verondersteld dat bij een hogere temperatuur (85°C) hardere en slijtvastere pellets ontstaan dan bij een lagere temperatuur van 65°C. Niet-lineare regressie werd gebruikt om de effecten van PDI op hardheid en slijtvastheid van de pellets te onderzoeken. Onderzochte hardheidsparameters waren Kahl (kgf), breukkracht bij gebruik van de Kramer shear press (N g^{-1} pellet) and breeksterkte bij gebruik van duw- en trekbank (N mm^{-1} pelletlengte). Deze parameters volgden een stijgend exponentiëel verband als functie van de PDI. Holmen en Pfost slijtvastheid (%), massa-stroom (kg uur^{-1}) en de hoeveelheid specifieke mechanische energy (kJ kg^{-1}) volgden een sigmoïdaal verband met PDI. De massastroom en de specifieke mechanische energie daalden met een toename in PDI. De porositeit van de pellets was lineair gerelateerd aan de sterkte van de geproduceerde pellets. De pellet porositeit lag tussen de 0.088 en 0.136. Lage porositeiten correleren met een hogere sterkte en omgekeerd. De NSI waarden werden bepaald en in tegenstelling tot de PDI waarden werden deze hoger. Dit was in tegenstelling tot de verwachting.

In hoofdstuk 8 is een model voersysteem gebruikt bestaande uit 50% soja en 50% tapioca waarbinnen de mate van eiwit denaturatie en zetmeelontsluitingsgraad is gevarieerd. De hypothese bij dit experiment was dat een hogere mate van zetmeelontsluiting en een grotere hoeveelheid niet-gedenatureerd eiwit leidden tot een hogere slijtvastheid en hardheid van de pellets. De eiwit kwaliteit werd bepaald door middel van de PDI. De zetmeelontsluitingsgraad

is bepaald door middel van de enzymatische amyloglucosidase test (SGD). De berekende correlaties tussen PDI and NSI waren hoog (>0.87: $p<0.001$) en de correlaties tussen PDI en NSI enerzijds en DSC-waarnemingen anderzijds waren laag (<0.35; $p>0.05$). De lage correlaties tussen DSC en de eiwitkwaliteits testen zijn voor een deel te wijten aan de (te lage) resolutie van de DSC bij de kleinere eiwitpieken en ten dele doordat een gedeelte van de eiwit en zetmeelpieken overlappen, en het eiwit niet als zodanig kan worden onderscheiden van de grotere zetmeelpiek. De fysische kwaliteit werd getest met dezelfde methoden als in hoofdstuk 7. Verder is een schijnbare modulus (N m^{-2}) en het percentage vervorming van de pellets (%) berekend uit de cijfers van de duw en trekbank. De schijnbare modulus was gerelateerd aan de resultaten van de hardheidstesten terwijl het percentage vervorming gerelateerd was aan de resultaten van de gebruikte slijtvastheidstesten. De resultaten zijn geanalyseerd door gebruik te maken van response surface analyse. De resultaten lieten zien dat een maximum kon worden gevonden binnen de limieten van het experimentele bereik voor de Holmen en Pfost slijtvastheids testen en het percentage vervorming. De pellet porositeit, percentage vervorming en de Holmen en Pfost slijtvastheid waren het meest gevoelig voor veranderingen in PDI van de modelvoeders. De hoeveelheid specifieke mechanische energy (kJ kg^{-1}) en capaciteit van de installatie waren het meest gevoelig voor veranderingen in de zetmeelontsluitingsgraad. De hardste pellets (volgens de gebruikte testen) en de hoogste schijnbare modulus, werden gevonden op het maximale insluitingsniveau van niet-gedenatureerd eiwit (hoge PDI) en maximale ontsluitingsgraad (hoog SGD). Bij verlaging van de PDI en de SGD daalde de hardheid en de schijnbare modulus. Binnen het proefbereik werd een globaal minimum werd gevonden voor porositeit en de hoeveelheid specifieke mechanische energie (kJ kg^{-1}). De resultaten van deze studie laten zien dat er grote effecten zijn op de fysische kwaliteit en systeem parameters van de functionele eigenschappen van de grondstofcomponenten eiwit en zetmeel en is daardoor relevant voor de praktische mengvoeder fabricage.

Algemene opmerkingen

Uit de resultaten van dit proefschrift kan worden geconcludeerd dat de functionele eigenschappen van de eiwit- en zetmeel fractie een belangrijke bijdrage leveren aan de fysische kwaliteit van de geproduceerde pellets. De twee methoden PDI en SGD zijn goede indicatoren voor deze functionaliteit van de eiwit- en zetmeelfractie. Er dient echter opgemerkt te worden dat deze methoden niet in alle gevallen een duidelijk beeld zullen geven van de korrelkwaliteit, aangezien de resultaten uit deze studie suggereren dat de, met behulp van PDI en SGD gemeten eigenschappen een optelling zijn van een aantal sub-processen op een lager niveau. Deze factoren zijn waarschijnlijk sterk gerelateerd aan de deformatie- en

bindingseigenschappen van de voer deeltjes.

De eiwit- en zetmeelfracties kunnen zelf veranderingen ondergaan gedurende de diverse bewerkingen. De mate waarin deze veranderingen optreden lijkt meer afhankelijk te zijn van de hoeveelheid warmte (toegevoegd in de vorm van stoom) en water dan van de toegevoegde hoeveelheid mechanische energie en afschuiving. Korrelhardheid en -slijtvastheid nemen toe naarmate een hoger aandeel van het eiwit bestaat uit niet-gedenatureerd eiwit. En, hoewel niet expliciet getest in dit onderzoek, lijkt dit voornamelijk te zijn geassocieerd met een grotere mate van deformatie van de deeltjes waarvan dit eiwit deel uitmaakt. Evenzo worden grotere pellet hardheden en slijtvastheden bereikt indien (voor-) ontsloten zetmeel wordt gebruikt in de (model)voeders. Enerzijds zal dit komen door een vermindering van de kristalliniteit van het zetmeel waardoor het makkelijker deformeerbaar wordt. Anderzijds kan (voor-)ontsloten zetmeel dienst doen als bindmiddel waardoor de verschillende deeltjes aan elkaar worden geplakt.

Een van de basis-hypothesen bij aanvang van dit onderzoek was dat een verhoging van de hoeveelheid dispergeerbaar eiwit en ontsloten zetmeel in (model)voeders leidt tot een hogere sterkte en slijtvastere pellets. Alhoewel is aangetoond dat dit inderdaad het geval lijkt te zijn, moet rekening worden gehouden met overige factoren zoals de deeltjes-grootte (vedeling) en het vochtgehalte van de uitgangsmaterialen, aangezien deze een zeer grote invloed kunnen uitoefenen (zowel in positieve als in negatieve zin) op de uiteindelijke sterkte van de geproduceerde pellets. Eén van de doelstellingen van dit proefschrift was het ontwikkelen van een model waarmee door middel van simpele manipulatie van parameters in termen van PDI of SGD, een voorspelling zou kunnen worden gedaan voor de fysische kwaliteit van gepelleteerde (model) voeders. De resultaten uit dit proefschrift geven echter aan dat, indien zo één model kan worden gevonden, dit model slechts geldigheid zal hebben binnen een gebied waarbinnen op z'n minst de deeltjesgrootte en het vochtgehalte gelijk moeten zijn. Het gebruik van numerieke modellen kan hierbij behulpzaam zijn. Uit de resultaten van dit proefschrift kan worden opgemaakt dat in ieder geval de volgende aspecten ook in dit model moeten worden meegenomen:

- deeltjesgrootte en deeltjesgrootte verdeling,
- deformeerbaarheid van de deeltjes waaruit het voer is samengesteld,
- het effect van water, warmte, afschuiving en verblijftijd in de verschillende proces-apparaten en hun effect op deformeerbaarheid en de hoeveelheid bindmiddel gevormd van het betreffende (model) voeder,
- bepaling van het type bindingsmechanisme als een functie van de boven genoemde parameters,
- bepaling van de sterkte van de binding tussen twee deeltjes als functie van de bovengenoemde parameters.

Curriculum Vitae

Meine (Menno) Thomas werd geboren op 4 oktober 1968 te Meppel. Zijn jeugd bracht hij door tussen de koeien op het ouderlijk melkveehouderij bedrijf in Oldemarkt. In 1986 behaalde hij het HAVO-diploma aan het Gelderingen-college in Steenwijkerwold. Aansluitend werd begonnen met een studie Nederlandse landbouw aan de toenmalige Agrarische Hogeschool Friesland (AHoF). Na deze studie in 1990 te hebben afgesloten, werd begonnen aan het doorstroomprogramma Veevoeding aan de Landbouwuniversiteit in Wageningen. In januari 1993 werd deze studie afgesloten met als afstudeervakken mengvoedertechnologie en ethologie. Vanaf die periode is hij werkzaam geweest als Assistent in Opleiding aan de vakgroep Veevoeding, waar tot februari 1998 het onderzoek werd uitgevoerd dat is beschreven in dit proefschrift.

Sinds 16 maart 1998 is hij werkzaam als wetenschappelijk onderzoeker bij het IMAG-DLO (Instituut voor Milieu- en Agritechniek) in Wageningen, alwaar hij zich bezig houdt met het ontwerpen van landbouwsystemen met minimale hoeveelheden reststromen.