Biotrem

Process and Product innovation of bran-based material

OPD 02/056/May 2003

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Introduction
Biotrem produces containers from bran-based material. The current production rate is 10 million articles annually, which as of today is produced in Poland solely. Biotrem aims at producing and trading 200 million articles annually. This could be realised by modernising the current production factory, building a new production line based on the currently applied processing principles, and by building a new production line based on the adapted processing principles.
Within the timeframe of 7 months ATO has performed research and development activities to support these aims. Initially the research programme was based on the following questions:

1. *How should the process look like to make it as production efficient as possible? There is a need for reconsideration and the testing of some new ideas.*
2. *How can the toughness of the articles be improved. The larger the article, the larger the brittleness problem becomes. Perhaps some additives are necessary.*

During the first month research has focussed on becoming familiar with the currently applied production process, translate these to the lab scale hot-press processing conditions at ATO and detecting the most critical processing parameters. In addition initial lab scale experiments with additional, natural binders were carried out. Based on the first results and combined with the findings from both the coating research and Markhorst, the programme was extended into three directions: processing research (a), material improvement (b) and product improvement by developing a water-resistant top layer (c).
Concerning (a), with lab scale experiments focus was on reducing cycle time by uncovering the weaknesses of the current production process and by evaluating the use of a mechanical pre-processing unit; the follow issues were addressed:

- Determine standard process (pressing time, pressure, number of cycles).
- What is the influence of the amount of pressure and the amount of moisture added on the properties of the produced plates?
- How does milling/extruding of the bran influence the properties of the produced plates?

Concerning (b), focus was on the materials' toughness increase by evaluating the use of additional binders. On lab scale, both natural and synthetic binders were tested on the ease of mixing, its influence on the water-management during processing and its contribution towards the materials' toughness increase. In addition other parameters like stiffness, strength and water retention were tested.
Concerning (c), focus was on evaluating the potency of lamination technologies as an alternative to the parallel running programme on spray-coating (reference is made to the previous technical details of the latter; for the sake of completeness an elaborate outline of those results is included in this summary). Key-issues were laminate-bran compatibility, water resistance and cost reduction.

Current production process
Up to now articles produced at Biotrem do not show uniform product behaviour. Significant differences between stiffness, strength, impact, water adsorption and density is found between typical samples being produced at the production site. The differences are due to different processing parameters on the presses and the different moulds.
A minimum amount of water inside the bran is needed to obtain flow during processing. Extrusion of the material increases the flow, therefore less pressure or lower water content can be applied. Based on mechanical properties it is advised that an initial water content of

around 15 wt.% is applied for articles, which require high stiffness, whereas an initial water content of around 18 wt.% is applied for articles, which require high strength.
Theoretical analysis shows that several pressing stages are necessary to reduce the amount of water present in the bran to the desired equilibrium water content of about 11%. Depending on the temperature of the mould and the heat exchange rate, the products will be either, to dry, to moist or precisely at the equilibrium water content of the product after storage at normal conditions. Increasing the temperature of the mould results in less pressing stages needed; the model shows that, ideally, only two stages are needed to obtain the desired water content.
Concerning time reduction of the first pressing stage an optimum is found for duration of 1.5 seconds. Optimising the process by adjusting the duration of each stage to the maximum duration that does not create an explosion does not result in optimal mechanical performance of the product. Optimising the process parameters should be done by evaluating on optical and mechanical performance.
Regarding density, it is shown that during the final stages of the process the density increases. Assuming that a product of good quality can only be achieved if the density is about 1200 kg/m^3, most likely, under the currently applied processing conditions, 5 stages are necessary to produce end products of sufficient quality. This can only be reduced by process- and material adaptations (see below).

Extruder pre-processing
It has been shown that using an extruder as bran pre-processing device, results in both technical and economical advantages compared with the currently applied production process. Plates produced at Biotrem from mildly extruded bran show a significant increase in strength and stiffness relative to its reference. With severe extrusion this behaviour is not observed. Concerning the water-retention, the Cobb-value of the extruded plates is higher than the Cobb-value of the normal plates, suggesting that extruder pre-processed plates have a higher water-retention relative to the reference product. Most likely this is caused by the smaller particle size of the bran. An additional top layer is necessary for applications with a critical water retention requirement. Using extruded bran makes the process less sensitive to changes in moisture content as far as the formation of bubbles is concerned, thereby making it a less critical processing parameter. This can result in higher productivity.
The adhesion of coatings improved when extruded bran was used as raw material for the products.
Concerning the estimation of the costs for extrusion, the extra costs based on a 10 year depreciation and an energy price of 0.04€/kWH is estimated at 6.25 €/ton bran. For a process that uses 100 grams of bran to produce an item the costs are 0.000625 €/item.
Regarding the estimation of capacity increase, the current process consists of 5 pressing stages; using extruded bran this process can be reduced to 3 processing stages. Using the current presses a reduction of cycle time from 32.27 to 27.04 would be achieved, increasing the production rate from 1.86 to 2.22.

Based on the gained knowledge plates were produced at Biotrem combining mildly extruded bran with a lower moisture content and a three stage process. Moisture content was reduced to 15,8% which resulted in a flow of the extruded bran comparable with the flow of normal bran at a moisture content of 18.5%. Visual differences between the five and three stage products of extruded bran were only observed by experts, the normal consumer will not see the difference. Mechanical properties of the extruded bran plates are excellent.

Additional binders

Thermoplastic binders can be added during pre-treatment of the bran or just before moulding of the products (binders can be added as powder, films or fibres). Up till now addition of thermoplastic binders in powder form just before moulding results in the best properties. In this way a doubling in toughness value and significant decrease in Cobb value can be realised by adding 5 -10 % binder, preferably 7.5 %. Proven suitable thermoplastic binders for the actual process are Ecoflex MFI 10 and Eastar Bio GP. Since these binders have a flexible character, the stiffness of the bran/binder compositions decreases in comparison with pure bran materials. By using binders with a high melt flow, however, a tough material can be realised, which has a slightly lower stiffness in comparison with pure bran products. Adding a binder to the bran results in additional flow of the bran/binder composition. This can be used for realising thinner products, bigger products and reduction of process cycle time. In addition, adding binders hardly influences on the total density.

Usage of water-soluble binders is another promising dosing technique. Up till now only an increase of water-resistance can be realised by adding these type of binders. Moreover, no additional value has been found for the use of natural proteins.

Binder processing

Concerning compression moulding, thermoplastic binders can be added during extrusion pre-treatment of the bran or just before moulding of the products (in this way binders can be added as powder, films or fibres). Up till now addition of thermoplastic binders in powder form just before moulding results in the best properties. Regarding compression moulding, properties of plates made at Biotrem Poland and samples made at ATO Wageningen indicate that the procedure used at both plants is comparable. Although the absolute value for toughness is not the same - toughness of articles made in Poland are lower in comparison with articles made in Holland - both processing routes show a doubling in toughness value by adding suitable binders. As to injection moulding, this is another possible technology to make bran based products. However, a minimum amount of 25% binder is needed, which raises the question to what extent the end product can compete economical.

Spray-coating development

To enhance the properties, like water resistance, of bran-based articles three different types of coating material have been applied:

- polyester (Ecoflex)
- polyacrylates (Epotal D700, Epotal DS 2343, UKAPHOB L496)
- polyvinylacetates (Craymul 2376, Craymul 2323)

During the first period of the project the spraying conditions have been studied extensively for Ecoflex solutions. The thickness of the coating is mainly determined by the speed of the conveyer belt and the spraying pressure. Increasing the pressure from 12 to 24 bar also improved the properties of the coatings. The spraying temperature influenced the coating thickness and film quality as shown by SEM pictures. If the temperature is too high, the solvents in the coating formulation evaporate very quickly which results in very porous coatings.

Ecoflex is soluble in a mixture of toluene and ethanol. For environmental reasons it is aimed to use a high amount of ethanol, but there are restrictions. The amount of ethanol must lie in the range of 5-25% in order to have a high solubility of Ecoflex. Viscosity of the coating formulation is also related to the toluene/ethanol ratio. A high amount of ethanol (e.g. 25%) gives a more viscous solution than a 5% ethanol-formulation. Lower viscosity means an increase in the Ecoflex content and a reduced drying time of the coating. It is demonstrated that the coating properties are most optimal when a ratio of 85/15 toluene/ethanol is applied.

With Ecoflex, smooth and attractive coatings can be applied onto the bran articles. The highest Ecoflex concentration that can still be sprayed at room temperature lies in-between 8.2 and 10 wt.%. The 10 wt.% formulation used here cannot be processed at room temperature anymore, and pre-heating of the formulation is required. If pre-heating in explosion-free tanks is an option, then the Ecoflex concentration can probably be increased up to the maximum solubility of Ecoflex in the toluene/ethanol mixture. This will lie between 18 and 20 wt.% of Ecoflex. A higher Ecoflex concentration means shorter drying times, less emission of toluene and ethanol, reduced costs of the solvent, and higher speed of the conveyer belt. The water resistance of bran-based products that are coated with Ecoflex is very good; contact with water for 5 min did not result in leakage and water uptake by the bran. Even very thin coatings applied on these plates were very water-resistant. Adhesion of the Ecoflex to the bran is relatively good; a minor point remains the moderate scratch resistance of the coating.

Polyacrylate coatings are hard and strong, and the adhesion to the bran plates is good. The appearance of the coatings is somewhat rough due to water uptake of the bran fibers. With these water-borne formulations, the dry-matter content is very important. The best results are obtained with the formulations containing a high percentage of solids. Water is withdrawn by the bran fibers very quickly after contact with the coating formulation. This might disturb the film forming- and drying process of the coating. The minimum film forming temperature and the drying conditions are also very important. The quality of the coating is often better at higher drying temperatures. BASF advises to dry the Epotal coatings with IR, and this is in accordance with our results, as the best results are obtained for the coatings dried with IR at 70°C.

Preliminary experiment was carried out with polyvinylacetate. It can be concluded that it is possible to apply polyvinylacetate formulations onto bran articles by spray coating. The formulation with the higher dry matter content and lower MFFT (i.e. high viscosity) provided better quality coatings than the low viscosity formulation. A clear positive influence of increased spraying temperature and pressure was observed. A distinct effect of drying temperature on the coating quality was not observed, although it is expected that this is a very important parameter in coating bran articles with water-based formulations. Appearance of the current polyvinylacetate coatings is somewhat rough, probably due to sub-optimal drying conditions. The water resistance is fairly good, and the adhesion and scratch resistance is very good. Overall, polyvinylacetate coatings have good perspectives as coating material for bran-based articles. A possible problem might be the fact that polyvinylacetate is not (fully) biodegradable.

Alternative top layer technologies
Concerning thermoforming, direct adhesion between bran and Ecoflex is not sufficient. Therefore adhesion improvers like Ecoflex binder are necessary. In addition coating of products with Ecoflex film by thermoforming is quite suitable process. It can perfectly added into the carrousel principle suggested by Markhorst. Time to laminate one Biotrem item, on a fully automatic and optimised thermoform machine, is less than 20 seconds and even less than 15 seconds. Additional material costs to coat one 230 mm plate of a bran/Ecoflex mixture with 20 μm Ecoflex film are € 0.015 for the Ecoflex binder and € 0.004 for the Ecoflex coating
Regarding compression moulding, this is a possible other technique to laminate Biotrem products. No other type of machinery has to be added to the production facilities, but the processing temperature, needed to press the laminate onto the bran-surface, needs to be lowered.

1 Introduction

Biotrem has developed a process to manufacture products based on bran material. Plates, trays and bowls are some products, which are made by a compression moulding technique. ATO has done research to improve the properties of bran-based materials and suggested solutions on how to make the process become more economic.

This report is based on both process and product research. The process part mainly focusses on optimising the process in order to reduce costs. Typical parameters like time, temperature and pressure have been critically evaluated on their contribution to the price/performance ratio of the end product. In addition other parameters like moisture content and bran particle size, which are indirectly connected to the process optimisation, have been evaluated on their contribution.
Product research has been done to improve the properties of bran-based materials. This part is based on adding a binder, which can create a higher toughness of the bran-based end product.
One weakness of bran-based product is its water-permeability. The water-resistance can be improved by adding a coating to the product.

Methods to analyse the properties of bran-based products and process conditions are described in chapter 2. Chapter 3 describes the applied processing technologies as well as the different binders. Also the set-up of the theoretically optimised process and argumentation is given in this chapter. Results and discussion are described in chapter 4. The investigation of three different coating processes, a possible new technique to make bran-based products and results of the visits to the Biotrem production plant complete this chapter. In chapter 5 the economical considerations concerning the applied processes are discussed. Conclusions and recommendations for further research are described in chapter 6 and 7.

2 Methods

2.1 Moisture analyses

The moisture content of standard bran was determined using a Satorius MA-40 infrared dryer. During measurement 2.0 g ± 10% of bran was weighed continuously during a heating cycle at 95°C for 4 minutes. The moisture content was calculated by the formula mentioned below and expressed in percentage.

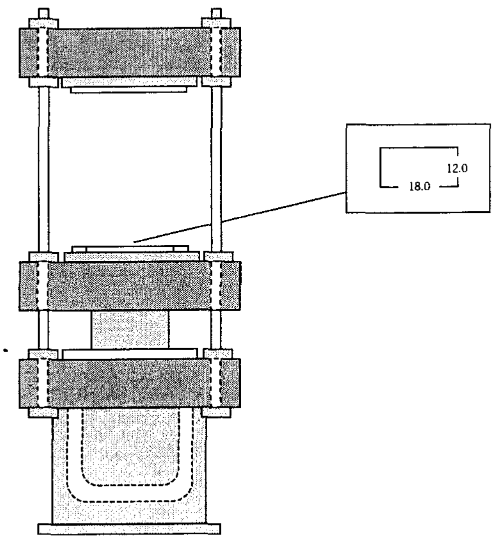
$$\text{Moisture content (0-100\%)} = \frac{\text{Initial weight} - \text{final weight}}{\text{Initial weight}} \times 100$$

2.2 Compression moulding

Compression moulding was performed using a PHI-press (Serial no: 93-007-010; Model no: 75U12095-2JCS-Y2-S5-7).
For the experiments the following process conditions were used: the temperatures of both hot-press plates were set between 140°C – 160°C, with a pressure of 30 tons for 5 times 5 seconds.
Press cycle time at ATO:

Stage 1:	0-5 sec
Stage 2:	18-23 sec
Stage 3:	36-41 sec
Stage 4:	54-59 sec
Stage 5:	72-77 sec

The big release time of the press resulted in a total process time of one experiment of approx. 83 seconds.



Two types of moulds were used during the experiments:

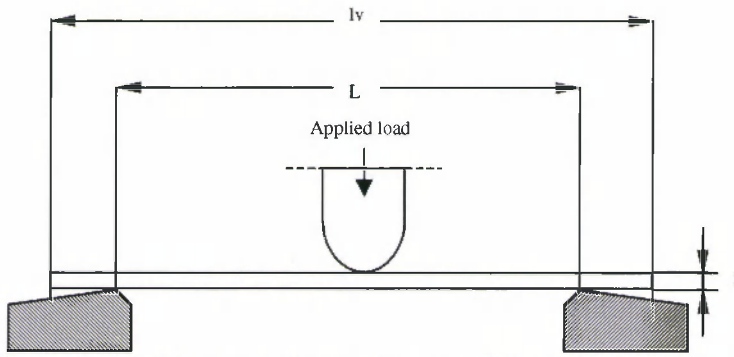
- An open mould consisting of two flat plates, in which the bran was placed in the middle of the mould on a surface of 18*12 cm. This resulted in free flow of the bran, during the experiment
- A closed mould with an open area of 18.0 cm by 12.0 cm and a thickness of 0.1 cm. The closed mould is fixed to the top plate. On the bottom we placed a closed mobile plate. The flow of the bran is limited, thereby plates were produced with comparable thickness.

The open mould method was used to research the current process, the closed mould was used to investigate the addition of binders.

The samples were cut into flexural testbars of 25.0 ± 1.0 mm width. The thickness of five separate samples was measured with an accuracy of 0.005 mm. Before measuring the mechanical properties the samples were conditioned at 55% relative humidity at 20°C for at least 7 days

2.3 Flexural stiffness and strength tests

The mechanical properties were tested according ISO 178 using a ZWICK Z1445. The distance between the two supports (L) of the testing machine is calculated by multiplying the mean thickness (h) with 35. The mechanical properties were measured with a 1 KN load-cell. The pre-load was set at 0.200 N with a speed of 2 mm/min. The test-speed was set at 10 mm/min. The measurement ended after the force dropped to 50% of the maximum force.



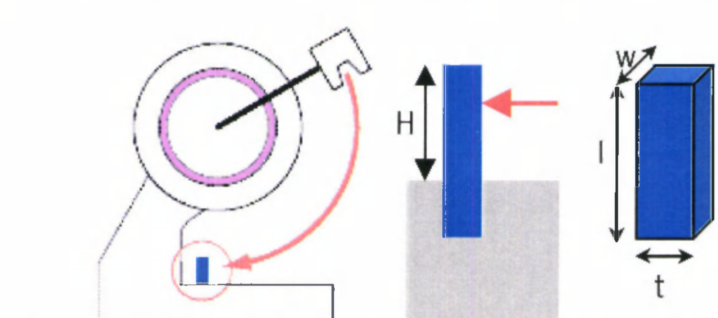
The flexural stiffness (E-modulus), maximum stress and the maximum force were determined.

2.4 Toughness tests

Materials toughness, expressed in Izod impact strength was measured with a Ceast Resil 50, using a 4J hammer, type 6547.946 with a speed of 3.46 m/s as described in ASTM D256. The impact strength indicates the resistance of a material against load. The testing machine is capable of measuring the impact energy, E, absorbed by breaking a test specimen. The Izod impact strength, a_i, is calculated using the formula $a_i = \frac{E}{w * t} * 10^3$ and expressed in kilojoules per square metre.

Where

E = the corrected energy, in joules, absorbed by breaking the test specimen
t = is the thickness, in millimetres, of the test specimen
w = is the width, in millimetres, of the test specimen; (25.0 ± 1.0 mm).
H = the length of the specimen above the clamps; (30.0 ± 1.0 mm).



2.5 Determination of water absorption using the Cobb method

The ISO 535 standard describes a method to determine the water absorptiveness of board under standard conditions. The principle of this International Standard is used to calculate the Cobb value. The Cobb value is the calculated mass of water absorbed in a specified time by 1 m^2 of a bran plate. A test piece is weighed immediately before and immediately after exposure for 120 seconds of one surface to water. The result of the increase in mass is expressed in grams per square metre according the next equation:

$$Cobb_{120} = (m_2 - m_1)F$$

$Cobb_{120}$ = water absorptiveness after exposure for 120 sec to water (g/m^2)
m_1 = the dry mass of the test piece (g)
m_2 = the wet mass of the test piece (g)
F = 10.000/test area (g/cm^2 to g/m^2)

2.6 Density

The density of the Biotrem samples were measured according the next mentioned method; the length (l) and the width (b) were measured with an accuracy of 0.05 mm and the thickness (h) was measured with a an accuracy of 0.005 mm. The weight (m) was measured on an analytic balance with an accuracy of 0.001 g.

The density was calculated according the formula $\rho = \frac{m}{l*b*h}$ and expressed in grams per cubic centimetres.

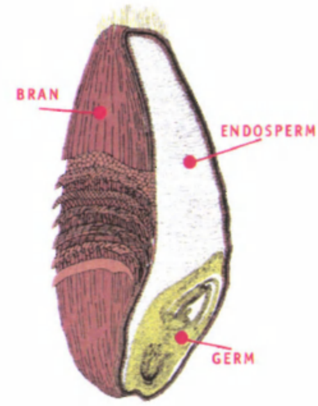
2.7 Extrusion

Extrusion experiments were performed using a Clextral BC45, co-rotating and intermeshing twin screw extruder with a diameter of 56 mm and L/D = 28. Extrusion was performed without a pre-die. Technical data is described in Appendix I.
The extruded bran-binder mixture was subsequently milled to homogenise the composition.

3 Materials & Process

3.1 Wheat Bran

During the milling process, about 70 to 75% of grain becomes flour, and the remaining 25 to 30% is available as wheat by-product. Wheat bran is one of the by-products of flour production, which predominantly consists of husks and variable portions of the endosperm.



The average wheat kernel consist of

- endosperm (75-85%) source of white flour
- bran (5 -15%) layered outer coat
- germ (0-5%) plant embryo

The bran is made up from seven different layers, each layer consist of different formed cells. The overall direction of the cells is different in every layer.

The composition of commercial wheat bran varies due to the settings of the mill. From Bergmans [2002] the following overview is given:

- Starch
  The starch (around 10-20%) originates from residual endosperm attached to the bran. It is present in two types of granules, the small spherical and the large lenticular. A quarter of the starch is present as Amylose, the remaining starch is amylopectine.

- Non starch polysaccharides
  The total content in industrial wheat bran is about 46%.
  - Arabino-xylan (around 70%)
  - Cellulose (around 24%)
  - 1-3,1-4β glucan (around 6%)

  Minute amounts of
  - Glucomannan (aleuerone and endospermcells)
  - Arabinogalactan (aleuerone and endospermcells)
  - Xyloglucan (pericarp tissue)
  -

- Protein
  The protein (15-22%) originates partly from adhering endoperm (albumins, globulins, prolamins and glutelins), most of the protein originates from the aleurone layer which contains approximately 30% of protein.

- Lignin
  The lignin content of wheat bran ranges from 4 to 8 %.

- Minor constituents
  The crude fat content is about 5%.The total free sugar content of bran is approximately 6%, the main free sugars are succrose and raffinose. The ash content (an indication of the amount of minerals) ranges from 4 to 6%.

3.2 Process

The process currently used by Biotrem aims to produce a moulded article from a mixture of water and bran. A specific pressure and temperature regime activates glueing components inside the bran, which results in a strengthened an stiffened product. In general the process can be described as five sequential pressing stages with a short opening of the mould in-between to release steam. The starting material is bran with a moisture content of 17-19%.

Although the moisture content of the bran at the beginning of the process and the applied temperature and pressure regime are thought to be very critical for the process, the process is currently running on six different presses, all working at different processing parameters. Besides the differences between the process on the six presses, on every press the process parameters are being changed in time, without a visible effect on the produced articles.

3.2.1 Description of the process

A report on the press performance by Markhorst describes in detail the used processing parameters on several presses on August 14 and 15 2002. The details from the process with the shortest cycle time are mentioned here:

	Press B
Product	Plate 200
Cycle time (sec)	32
Press time (sec)	15
Temp. Top (°C)	124
Temp. Bottom (°C)	132
Pressure stroke 1 (MPa)	27
Pressure stroke 2 (MPa)	26
Pressure stroke 3 (MPa)	10
Pressure stroke 4 (MPa)	10
Pressure stroke 5 (MPa)	10

The five stages start after introducing the bran in the mould.

Stage	Press Down (sec)	Pressure on the bran (sec)	Press Up (sec)	Steam release[1] (sec)
1	2.57	**3.36**	1.04	**2.32**
2	1.26	**1.80**	1.04	**2.44**
3	1.40	**1.30**	0.96	**2.36**
4	1.40	**0.17**	0.80	**2.03**
5	1.23	**0.20**	1.40	∞

[1] This is the sum of Press down + Press Up.

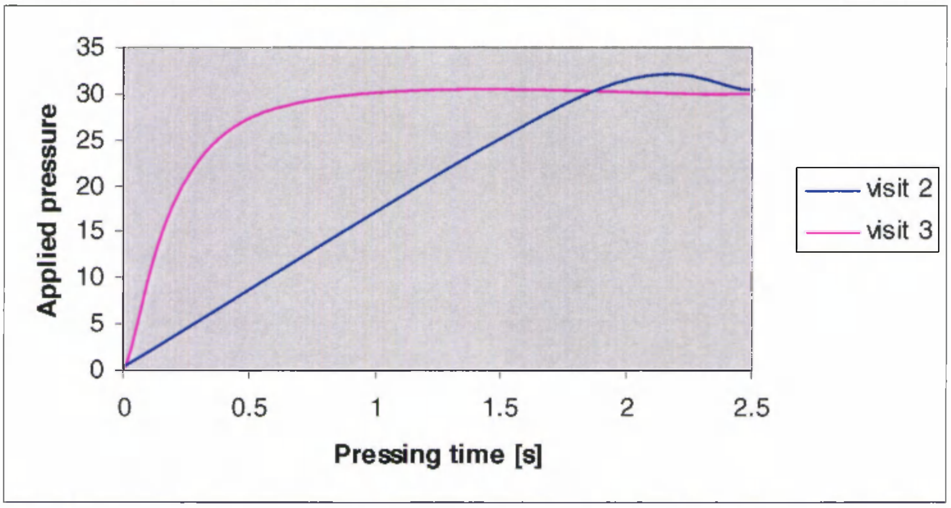
3.2.2 Moisture content

The amount of moisture in the bran before pressing is adjusted up to about 18-19%. The water in the bran influences different steps in the process:

- The flowability of the material during the first pressing stage (rheologic component).
- The heat transfer from the outside of the bran plate to the inside (thermal component).
- The reaction that is strengthening the bran plate (physical/chemical component).

3.2.3 Applied pressure

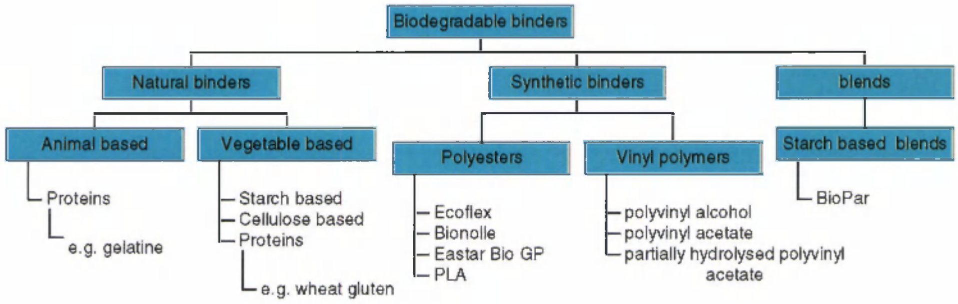
During a processing stage the pressure on the bran increases, both because of thickening of the bran layer and because of the downward speed of the press. Two different pressing regimes are applied. Most of the times the pressure is released once the maximum pressure is achieved, however sometimes the pressure is kept at maximum for a short period.



Different pressing regimes result in end products with different properties (see next chapter for detailed information). This should be kept in mind when properties of the materials, as produced under experimental conditions, are compared with the 'industrial reference'. In fact, typical industrially produced products mutually varied such in properties, that one can hardly speak of a reference material.

3.3 Binders

Biodegradable mouldings made according to the Biotrem process as described in patent WO 01/39612 have relatively weak toughness properties. This behaviour is caused by the weak adhesion between the bran particles. If these articles have to be used as packaging materials the toughness of these products have to be improved. One option to improve the mechanical properties of the bran-based articles is to add a binder. In this research we concentrated on biodegradable (and compostable) binders. In an active microbial environment such as a commercial composting site, the certified biodegradable materials becomes invisible to the unaided eye within 12 weeks. Within a maximum of 6 months, these materials will completely biodegrade into CO_2, H_2O, and biomass. The extent and rate of biodegradation depend on several factors in addition to the inherent biodegradability of the material itself. These factors include environmental conditions such as moisture and temperature, surface area, and manufacturing method of the finished product.
Biodegradable binders can be divided in materials based on renewable raw materials and non-renewable raw materials. Examples of the first group are protein, cellulose and carbohydrate based materials. Moreover, a large number of non-renewable, biodegradable materials (e.g. poly-esters) exists. We focused on relative cheap binders with a biodegradable and compostable character. Furthermore we focused on binder types with high water resistance properties which have good interaction with bran and coating materials. The binders used can be classified in different groups of basic materials. A simplified overview is given below:



3.3.1 Thermoplastic binders

Most thermoplastic binders are delivered as polymer granulates. However during research it appeared that this physical state is not appropriate for this moulding process. We need another physical state. During this project we tested the following things:

- Powder: Binders can be added as a powder to the bran. Before adding the binder to the bran, the granulate is milled with liquid nitrogen and fractionated by sieving. The particle size fraction smaller than 1.0 mm was used in this study. Mixing this powder with bran resulted in a homogeous bran-binder composition. This mixture can be moulded .
- Film: binders can be added to the bran as thin films or sheets. If the compression moulding process is started from a multilayer system composing out of alternating layers of bran and binder products with good properties can be realised.
- Fibres: most binders can be converted into (thin) fibres. These fibres can be mixed very well with the bran material. This mixture is fed to the moulding process.

During this research we started from granulated binders if we preprocess the bran with extrusion. In this case, the extruder has been fed with bran and granulated binders.
In the next paragraphs, binders as used in this project are described.

3.3.1.1 Bionolle

Bionolle materials are a range of non renewable, biodegradable, thermoplastic polyesters. Bionolle is produced via the process of polycondensation of polyols with aliphatic dicarboxylic acids. The Bionolle 3000-grades as used in this project start from succinic acid, adipic acid and 1,4-butanediol as raw materials. Bionolle is marketed by Showa Highpolymer Co. Ltd.

Under normal conditions, Bionolle is a stable material. It is insensitive for hydrolysis and has a good resistance to water. However, under composting conditions the material is biodegradable. Melting point is about 100 °C. Prices of this material are in the range of € 5,-/kg.

Bionolle has been taken as a model binder, because this material can be delivered in a wide range of viscosities. Material parameters as well as process parameters are studied in this model.

In our first experiments the moisture content is the parameter varied. It is assumed that the equilibrium moisture content lies between 10.5-12.0%. With this moisture content as starting point, four other batches were made; respectively 6.0%, 9.0%, 15.0% and 18.0%.

For binder materials three important variables can be varied: temperature of the moulding process, melt flow rate (of the binder) and the amount of binder (added to the bran). The melt flow rate (MFR or MFI) of Bionolle is usually expressed by X gram per 10 minutes at 190°C with load of 2.16 kilograms. Bionolle is available in grades with various MFR's.. Three different types of Bionolle; #3001, #3010 and #3020 were used. The standard temperature of the mould (both upper and bottom plates) during processing is 160°C. The amount of binder, the third parameter, is based on a total amount of 60.0 g. Percentages of 0, 5, 10 and 20% based on the total mass were used.

3.3.1.2 Ecoflex

Ecoflex is a non-renewable aliphatic-aromatic copolyester based on terephthalic acid, adipic acid and 1,4-butanediol made by BASF AG. Ecoflex is a certified biodegradable polymer material especially suitable for films and wrappings. In a composting system, Ecoflex is decomposed by microorganisms. Only natural residues such as water, carbon dioxide, and biomass are left.
Packaging producers have already used environmentally friendly Ecoflex sheeting to coat external and internal surfaces of disposable plates and cups. Ecoflex material is also used for production of bags for organic waste, in agricultural sheeting and for hygiene films. Ecoflex complies with the food legislation requirements of EC 90/128.
Ecoflex has a melting point between 110-115°C. Film types of Ecoflex are commercially available with different viscosities. Standard Melt Flow Index (MFI) is 3 gram/10 min. At special request other Ecoflex grades with different MFI's can be bought. In our research materials with MFI of 3 and 10 gram/10 min have been used. Ecoflex can be bought for about € 3-3.5,-/kg.

3.3.1.3 Eastar Bio

Eastar Bio copolyester as produced by Eastman is a similar material as Ecoflex. It consists out of a family of resins and additives that performs for the useful life of the product and then biodegrades under the right conditions, leaving no environmental harmfull substances. These co-polyesters meet FDA and EU requirements for food contact.
Eastar Bio co-polyester is a patented product family produced from (at this moment) non renewable conventional diacids and glycols (more specific terephthalic acid, adipic acid and 1,4-butanediol). Its chemistry was designed to combine physical strength, extended shelf life, and processability with the unique feature of complete biodegradability.
Eastar Bio has a melting point of 108 °C. Similar to Ecoflex, Eastar types can be delivered with range of viscosities. However, in general viscosities of Eastar are somewhat lower than viscosities of Ecoflex. For this research Eastar Bio GP has been used. Eastar Bio can be bought for similar prices as Ecoflex (about € 3-3.5,-/kg).

3.3.1.4 BioPar

BioPar is the name of a range of products from BIOP Biopolymer Technologies AG. BioPar is a blend of renewable, thermoplastic starch and various biodegradable poly-esters (these can be non-renewable). It is made with help of reactive extrusion. BioPar is completely biodegradable under composting conditions. BioPar has a melting range of 77-125°C. Depending on the type of starch and poly-ester that is used various viscosities can be realised. The price of Biopar is about € 2.5 - 3.5 / kg.

3.3.1.5 Natureworks PLA Polymers

Naturworks PLA Polymers are products of Cargill Dow LLC. Poly Lactic Acid (PLA) is a highly versatile aliphatic, compostable polymer derived from 100% annually renewable resources. PLA is available in pellet and fibre form and is developed for food contact products. A moisture content less than 0.025% during processing is recommended to prevent degradation (so materials have to be dried before processing). In comparison with the other poly-esters, the mechanical properties of PLA polymers can be characterised as stiff. In this study we used Natureworks PLA Polymer 4042 D. It has a melting temperature of approx. 135°C. Due to this high melting temperature processing temperatures are rather high (160 - 200 °C). Natureworks PLA Polymers can be bought at prices of € 3 - 3.5 / kg.
Cellulose Cellulose is a main carbohydrate produced by plants. It is the most abundant carbohydrate available from agricultural raw materials (so cellulose is a renewable material). Cellulose acetates are made by chemically modifying cellulose. Cellulose used is from wood or cotton but cellulose obtained from flax or old paper can also be used. . The degree of modification is given by its degree of substitution (DS), which can be maximal 3. Depending on this degree of substitution cellulose acetate can be considered as biodegradable. When DS is lower or equal than 2, it is general accepted (and proven) that these modified celluloses are biodegradable. Bioceta is a commercially available grade of Cellulose diacetate (CD) and is marketed by Acetati S.p.A., Italy. CD is an amourphous and transparent polymer. CD itself has a glass transition temperature around 200°C which can be lowered by the plasticiser content. Tri-ethyl Citrate (TEC) is an example of a plasticiser, which can be used (and has been used in this study). Plasticised CD is water-resistant. The price of CD is about € 3,-/kg

CD has to be dried for 4 hours at 50°C before use. The plasticiser content used in this study is varied between 30 and 50% TEC. Using a plasticizer content of 30% TEC lowers the Tg to approx. 100°C. So standard conditions of compression moulding of Biotrem disposables can be used.

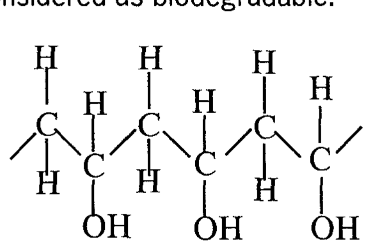
For this study cellulose di-acetate with 40% tri-ethyl citrate granulate was made on a Berstorff extruder. For a several experiments, in a second step a percentage of 10% plasticised CD granulate was mixed with the bran on the Clextral BC 45 extruder. The technical data are described in appendix I.

3.3.2 Water soluble binders

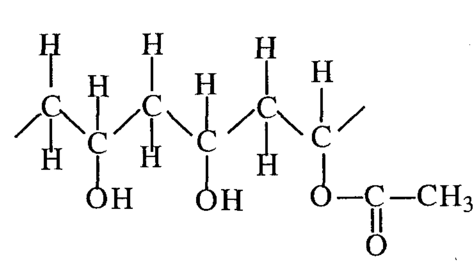
The production of standard Biotrem plates needs bran material with a moisture content of approx. 18.0%. Standard bran, which can be bought from milling companies, has a moisture content range of 10.5-12.0%. This higher moisture content during moulding is necessary for improving the flowabillity of the bran.
Therefore one of the first steps during production of Biotrem disposables is adding water to the bran material. By dissolving a polymer (which can be a binder) in water at relatively high temperatures (e.g. 90°C), adding this to the bran (till the moisture content is 18.0±1.0%) and subsequently moulding this mixture, a more or less water-resistant product can be created.

3.3.2.1 Poly Vinyl Alcohol/acetates

PolyVinyl Alcohols are polymers of vinyl alcohol. However, the monomer vinyl alcohol does not exist in the free state ; Therefore polyvinylalcohol is manufactured by polymerisation and modification of vinyl acetate. First polyvinylacetate is produced from vinylacetate. In a second step, this polymer is (partially) saponified. Hydrogen bonding, resulting from the high hydroxyl group concentration and its ability to crystallise largely control the properties of PVOH. Depending on the degree of saponification, i.e. the number of unhydrolysed acetate groups left in the PVOH molecule, a range of materials can be produced, varying from partially to fully saponified, with differing hydrogen bonding abilities and degree of crystallisation and consequently different property combinations. Physical properties such as the strength, water solubility, gas permeability, and thermal characteristics can be manipulated by adjusting these factors. In general, polyvinylacetate material needs a degree of saponification of at least 30 % to be considered as biodegradable.



Fully hydrolysed



Partially hydrolysed

In the experiments with water soluble binders two different types of polyvinyl alcohol/acetate materials are used:

1. Mowiol is a trade name of the polyvinyl alcohols marketed by Clariant. By varying the degree of polymerization of the polyvinyl acetate and its degree of hydrolysis two grades

are chosen: Mowiol 3-83 and 3-98. The first number in the grade indicates the viscosity of a solution of this material, while the second number is a indication for the degree of hydrolysis. Mowiol is a technical-grade product and therefore not recommended for use in foodstuff. However, Mowiol can be used for contact with foodstuffs, depending on quantities and viscosity. Both partially and fully hydrolysed are biodegradable in the right conditions. Mowiol is especially suitable as component is solution systems.

2. PVAXX materials are a range of biocompostable polymers, designed and marketed by PVAXX. This material is, by adding plasticizers to the system, a thermoplastic material. The key property of all PVAXX polymers is this product's unique capacity to biodegrade into non-toxic residues under both aerobic and non-aerobic conditions. PVAXX have received European and FDA safety approval for food contact use. The used grade in these expeiments, PVAXX W20 is a warm water soluble blown film grade

4 Results and discussion

4.1 The normal process: Current products

4.1.1 Current products

Samples of the current products were send to ATO and tested. The age of the products (time between production and testing) is not determined.

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
Misha ∅ 13	1265	175	8.8	0.5	6.2	0.4	0.84	0.09	176	1.21
Misha ∅ 16	1124	176	8.7	0.6	6.1	0.4	0.98	0.19	113	1.22
Misha ∅ 20	1192	144	6.7	0.3	5.4	0.3	0.75	0.11	134	1.08
Misha ∅ 23	1242	127	6.9	0.5	5.5	0.4	0.25	0.03	221	1.19
Talerz ∅ 16	976	80	7.5	0.6	4.3	0.4	0.71	0.15	245	1.16
Talerz ∅ 20	1004	102	7.9	1.1	4.0	0.5	0.76	0.13	142	1.13
Talerz ∅ 23	775	51	5.7	0.8	3.8	0.5	0.78	0.05	266	1.20

Significant differences between stiffness, maximum stress, impact, water adsorption and density is found between the different samples. The differences between the products are due to different processing parameters on the presses and the different moulds . The processing parameters of each press should be set based on both optical and mechanical evaluation of the product. Besides the inherent differences in material properties produced with the current process, ageing of the material might play a crucial role.
Of significant influence on the water absorption is also the moisture content of the products directly after processing. It was found that, using the current process, plates are produced that will either take up water or dry during conditioning.

4.1.2 Comparison with Polystyrene

Currently polystyrene plates are applied as pizza underlay. To determine whether bran plates are strong enough to replace polystyrene, the mechanical properties of bran and polystyrene plates are tested.

Raw material	Stiffness (Mpa)	Std	Force max (N)	Std	Thickness (mm)	Density (g/cm³)
Bran (normal)	1176	109	3.0	0.4	1.7	1200
Bran (up side down)	1596	322	2.3	0.3	1.7	1200
Polystyrene	90	7	4.1	0.2	4.1	56

Support length = 98 mm

From this data it can be calculated how much a plate will bent at a given loading. For ease of calculation it is assumed that the plates are square. A tree point bending is assumed, with the load in the middle. The size of the plates is 23 cm. The bending is given for different loadings.

Raw material	**100 gram**	**400 gram**	
Bran (normal)	13	51	mm
Bran (up side down)	14	54	mm
Polystyrene	12	50	mm

There is little difference in the bending between the plates. However, the bran plate will break at a lower loading because of the lower maximum force.

4.2 The normal process: The influence of the process parameters

4.2.1 Density during processing stages

The normal process consists of five sequential pressing stages. The density of the plate after each pressing stage will change because of the applied pressure and the drying of the material. The thickness of the plate will change because of the flow of the material and the density. The density and thickness of boards produced at Biotrem are shown below:

Processing stage	**Thickness (mm)**	**Density (g/cm³)**
1	1.10	1032
2	1.43	1034
3	1.32	1033
5	1.19	1200

The measurements of the density are performed after conditioning of the plates. It is shown that during the final stages of the process the density increases. It is likely that a product of good quality can only be achieved if the density is about 1200 kg/m^3. Most likely, under the currently applied processing conditions, 5 stages are necessary to produce end products of sufficient quality.

4.2.2 Over and under loading the mould

The volume inside the mould, together with the density of the final product determines the necessary amount of starting material. Ideally the excess bran will flow out of the mould, however the current process reacts differently on to much or to little starting material.

Bran amount	Thickness (mm)	Density (g/cm³)
Half	0.79	1187
Normal	1.19	1200
Double	1.99	1078

If only half the amount of bran is introduced the thickness of the plate is less, no bran is flowing out of the mould. The density of the plate is comparable to the density of the normal plate. At the normal amount, part of the bran is flowing out of the mould. When twice the amount of bran is introduced the thickness of the plate is almost doubled, but the density is less, indicating a product with minor mechanical properties.

An optimal correlation between the amount of bran introduced and the size of the mould is necessary to obtain an optimal product.

4.2.3 Estimation of the minimum number of stages

After the bran is introduced in the mould, the pressure on the bran is increased up to the maximum pressure in the process. During closing off the mould a contact trigger is passed which triggers a timing device. After a determined amount of time the first stage ends. This pressure induces flow of the material inside the mould. A little amount of bran is pressed out of the mould. The bran material is heated by several mechanisms, mainly by conduction of heat from the mould to the bran, and by evaporation of the water at the outside of the bran and consecutive condensation of the created steam inside the bran. At the end of the first stage the average temperature of the bran is at most only slightly above 100 C. Therefore only a minor amount of steam is created when the pressure on the bran is released.
The current process consists of five consecutive pressing stages. During the process the moisture content of the bran decreases from about 18.5 to10%. Because of the closed mould the water is physically removed from the bran during the opening of the mould. A simple model can be used to estimate the number of pressing stages necessary to remove the water.

The model is based on the following assumptions:

- During each pressing stage the bran/water mixture is heated up to the temperature of the mould
- The driving force for evaporating of the water during the opening of the mould is the temperature difference between the boiling point of water and the temperature of the bran/water mixture.

The model neglects the facts that

- Water is evaporating below 100 °C.
- The bran is heated from the bottom part of the mould during the opening of the mould.
- The duration of the pressing and opening stages are limited.

The simple model consists of an energy balance:

The amount of energy needed to evaporate a certain amount of water equals the difference between the amount of energy of the pressed water/bran mixture at mould temperature and the amount of energy of the pressed water/bran mixture at the boiling temperature of water.

The actual calculation is not given in this report.

	Water	Bran	
Heat capacity	4.2	1.4	kJ/kgK
Heat of evaporation	2200	-	kJ/kg
Temperature after opening of the mould	100	100	°C

Pressing stage	Moisture content [%]		
Mould temperature	120°C	130°C	140°C
start	18.5	18.5	18.5
1	16.7	15.9	14.8
2	14.9	13.0	**11.2**
3	13.1	**10.4**	7.6
4	**11.3**	7.7	4.1
5	9.6	5.1	0.7

From this simple model it can be seen that several stages are necessary to reduce the amount of water present in the bran to the desired equilibrium moisture content of about 11%. Depending on the temperature of the mould and the heat exchange rate, the products will be either, to dry, to moist or precisely at the equilibrium moisture content of the product after storage at normal conditions.

Increasing the temperature of the mould results in less pressing stages needed; the model shows that, ideally, only two stages are needed to obtain the desired moisture content. It should be noted however, that this model does not take the final product properties into account.
The amount of steam formed increases with increasing mould temperature at given duration of the pressing stage due to an increased heat transfer rate.

The current process uses an open mould, however no steam can escape during a pressing stage. Adapting the mould to make evaporation during pressing possible will result in a quicker process, with less pressing stages.

4.2.4 Flowability of the bran

During the first pressing stage the bran is flowing inside the mould. The semi-open mould is filled and the excess amount of bran is flowing out of the mould. For normal processing conditions no flow is occurring after the first pressing stage. The actual flow of the bran inside the mould is determined by several parameters:

- The applied pressure versus time schedule
- The viscosity of the bran/water mixture, which is determined by
  - The amount of water
  - The structure of the bran (extrusion/milling)
  - The addition of a binder
- The initial position of the bran inside the mould
- The shape of the mould
- The surface-material of the mould

The choice of the different parameters mentioned above will have a direct correlation with several other processing parameters and product properties:

The necessary time for opening of the mould at the beginning of a new pressing cycle can be reduced when the bran layer introduced is as low as possible: The bran is evenly distributed over the whole surface of the mould. The flow of the bran should then be limited, else most of the bran will flow out of the mould. A new mould design, a reduction of the moisture content, a reduction of the used pressure may be necessary.
The strength of the product will most likely increase if a certain amount of flow during production of the product is created. The flow will create a certain structure in the bran and the bran will be kneaded, which in bread is known to give a strengthening effect.
Flow of the bran is needed to fill the whole mould, without flow cracks are introduced in the product.

The flow of the bran in the mould was simulated between two flat surfaces. The bran was distributed on a surface of 13*18 cm. Using a five stage pressing process, different moisture content and different pressures were used. The final size of the bran plates was measured, resulting in an indication of the flow of the bran.

Moisture content	Size increase Length (%)	Size increase Width (%)	Pressure initial (Mpa)	Presure Final (MPa)
10	0.0	0.0	17.5	17.5
15	18.8	42.9	17.5	10.3
18	34.4	61.9	17.5	8.1
20	37.5	81.0	17.5	7.0
25	50.0	90.5	17.5	6.1

From this data it can be concluded that a minimum amount of moisture inside the bran is needed to obtain flow during processing. At higher moisture content the flow is linearly increasing with the amount of moisture. The difference in size increase in the length and the width of the plates indicates that the free flow of the material at the side of the mould is larger than the obstructed flow in the middle.

Applied pressure (ton)	Size increase Length (%)	Size increase Width (%)	Pressure initial (Mpa)	Presure final (MPa)
20	12.5	23.8	11.7	8.4
30	34.4	61.9	17.5	8.1
40	43.8	81.0	23.4	9.0
50	50.0	95.2	29.2	10.0
60	59.4	100.0	35.0	11.0

Using higher pressure increases the flow of the bran. A change in moisture content has more effect on the flow than a comparable change in pressure.

The shape and composition of the bran influences the flow of the material.

Raw material	Size increase Length (%)	Size increase Width (%)	Pressure initial (Mpa)	Presure final (MPa)
Polish Bran	19.4	41.7	8.1	17.5
Middlings	27.8	70.8	6.2	17.5
Extrusion mild	25.0	54.2	7.1	17.5
Extrusion middle	36.1	70.8	5.9	17.5
Extrusion severe	38.9	75.0	5.6	17.5

Extrusion of the material increases the flow of the bran. Less pressure or lower moisture content can be applied to obtain the usual amount of flow. The middlings also show increased flow, most likely because of the higher starch content. Wheat bran without starch does not flow.

4.2.5 The effect of initial moisture content

Experiments to test the effect of the initial moisture content on the mechanical properties of the product were done both at ATO and at the mill.

Moisture	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Density (g/cm³)
15%	1885	297	8.0	0.7	7.5	0.7	0.73	0.19	1.29
20%	1518	406	9.5	1.7	5.2	1.0	0.58	0.17	1.24
25%	1454	452	10.9	2.5	0.8	0.2	0.53	0.22	1.25

The test at ATO show a decrease in flexural stiffness and an increase in flexural strength as result of an increase in moisture content. However the thickness of the produced plates decreased as a result of the increased flow of the bran.

Moisture	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
17.0 %	1421	247	7.4	1.0	3.4	0.5	0.69	0.18	97	1.13
17.2 %	1160	146	7.4	0.8	3.4	0.4	0.68	0.02	82	1.14
17.4 %	1058	130	7.2	0.7	3.2	0.3	0.70	0.06	74	1.17
17.6 %	1368	170	7.8	0.9	3.3	0.4	0.63	0.05	211	1.13
17.8 %	1276	199	8.7	0.6	3.4	0.2	0.65	0.10	100	1.11
18.0 %	933	126	7.3	0.8	2.8	0.3	0.57	0.11	84	1.17
18.2 %	1041	102	8.6	0.8	3.2	0.3	0.76	0.07	68	1.13
18.4 %	1066	241	7.7	1.8	3.1	0.7	0.60	0.10	182	1.12
18.6 %	1064	131	8.0	0.6	3.3	0.2	0.75	0.00	97	1.09
18.8 %	1025	172	7.8	1.0	3.1	0.4	0.64	0.09	92	1.11
19.0 %	1037	95	8.1	0.8	3.2	0.3	0.55	0.13	118	1.14

The results of the test at the mill are less clear, most likely due to the poorer mixing and less accurate measurement of the initial moisture content. Overall a slight decrease of flexural stiffness with increasing moisture content and an slight increase in flexural strength can be observed. No clear effect on Cobb-value is found.

Based on mechanical properties it is advised that an initial moisture content of around 15 wt.% is applied for articles, which require a high stiffness, whereas an initial moisture content of around 18 wt.% is applied for articles, which require a high strength.

4.2.6 Duration of first pressing stage

The effect of a longer or shorter first pressing stages was tested at the mill. During normal processing the duration of the first pressing stage is as long as possible without destroying the plate by a steam explosion. The duration of the first pressing stage also determines the amount of flow of the material.

Duration first pressing stage	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
0.5 sec	1205	207	6.5	0.7	3.1	0.3	0.62	0.05	116	1.08
1.0 sec	1294	135	7.9	0.5	3.3	0.2	0.36	0.10	121	1.13
1.5 sec	1557	30	8.7	0.7	3.8	0.3	0.75	0.13	66	1.17
2.0 sec	1514	187	8.6	1.1	3.7	0.5	0.70	0.08	68	1.14
2.3 sec (normal)	1299	106	8.7	0.9	3.5	0.3	0.52	0.08	97	1.13
3.0 sec[1]							0.59	0.06	116	1.17
4.0 sec[2]							0.62	0.09	63	1.23

1 produced plate to thin to be tested on mechanical strength.
2 plates explode after first stage, pieces to small to be tested on mechanical strength.

An optimum is found for a duration of 1.5 seconds of the first stage. It can be concluded that optimising the process by adjusting the duration of each stage to the maximum duration that does not create an explosion, does not result in optimal mechanical performance of the product. Optimising the process parameters should be done by evaluating on optical and mechanical performance. In this tests the largest stiffness was found for a first stage that was 0.8 seconds shorter than the duration of the first stage in the normal process.

4.2.7 The effect of pressure on the mechanical properties

The effect of pressure on the mechanical properties was tested both at ATO and at the mill. The experiments at ATO show no significant differences in flexural strength and stiffness, Toughness decreases with increasing pressure, possibly due to the relatively small thickness of the plates produced at higher pressures .

Maximum pressure	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Density (g/cm³)
20 bar	1673	217	7.5	0.6	0.6	0.1	1.06	0.21	1.25
40 bar	1840	714	10.6	2.0	0.8	0.2	0.24	0.04	1.29
50 bar	1890	746	9.5	4.0	0.6	0.1	0.48	0.07	1.29
60 bar	1530	641	11.2	4.2	0.9	0.2	0.31	0.06	1.29

The experiments at the mill show a decrease in flexural stiffness and strength. Cobb-value increases with decreasing pressure.

Maximum pressure	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
Max 25 MPa	1299	106	8.7	0.9	3.5	0.3	0.52	0.08	97	1.13
Max 17 MPa	857	162	6.9	0.2	3.6	0.1	0.56	0.02	147	1.12
Max 13 MPa	1086	89	6.4	0.4	3.9	0.2	0.58	0.04	208	1.11

4.2.8 The effect of extrusion on the mechanical properties

Bran was extruded at ATO. Three different batches were produced, using increasing amounts of energy. With increasing energy the size of the produced bran particles decreased.

Raw material	Stiffness (Mpa)	Std	Strength (Mpa)	Std	Density (g/cm³)
Polish Bran	2248	653	11.7	2.3	1285
Middlings	2228	442	11.4	1.8	1280
Extrusion mild	1995	707	12.3	2.7	1272
Extrusion middle	2130	731	13.4	3.5	1266
Extrusion severe	2385	639	16.8	5.8	1334

The results at ATO show no significant effect on mechanical properties, although an increase in stiffness and strength with increasing severity of extrusion was expected.

Extrusion	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
Normal	1299	106	8.7	0.9	3.5	0.3	0.52	0.08	97	1.13
Mild extrusion	1661	93	10.6	0.7	4.0	0.3	0.60	0.07	137	1.21
Severe extrusion	908	210	7.2	0.5	2.9	0.2	0.49	0.10	245	1.20
Mild extrusion[1]	1794	414	10.0	1.4	5.3	0.8	0.7	0.1	258	1.2

[1]. Second batch

Plates produced at Biotrem from mildly extruded bran show a significant increase in strength and stiffness relative to its reference. The cobb-value of the extruded plates is higher than the cobb-value of the normal plates. Most likely this is caused by the smaller particle size of the bran. Smaller particles correlates with a larger amount of cracks around the particles, through which water can enter the product.
Severe extrusion does not result in a product with higher mechanical performance, however producing a product with an equal thickness as the normal plate was impossible because of the higher flow of severely extruded bran.

4.2.9 The addition of starch and proteins

During the production of a bran plate, the bran particles are glued together. Inside the bran there are three materials: starch, protein and sugar with glueing potential. Tests have been performed to determine which material is activated as glueing agent during the process. First the bran was treated with different processes to remove specific components:

Milling and sieving (250 μm)

	Yield [%]	Protein [%]	Starch [%]
Bran	-	16.7	11.0
Bran after milling and sieving	95	16.6	10.6
Powder	5	18.7	25.3

It is shown that by milling and sieving a powder can be produced that is rich in protein and starch.

Starch extraction and enzyme treatment

The extraction was done by six sequential washing stages. Enzyme treatment was performed using different enzymes (Alcalase, Rohalase, Fungamyl, BAN)

	Yield [%]	Protein [%]	Starch [%]
Bran	-	16.7	11.0
Bran extracted at room temperature	54	12.8	1.3
Bran extracted at 85°C	52	12.5	1.0
Bran treated with enzymes	46	10.9	0.0

It was not possible to remove the starch or protein selectively using the above mentioned methods. It should be kept in mind that the third glueing component Arabino-xylan is also removed as can be seen from the yield of the processes. During the tests at ATO it was concluded that starch is a vital component to the process because of the effect on the flow of the bran. Without starch the flow of the material is zero. The addition of starch results in an increased flow of the bran during the process. At ATO test plates were produced with different proteins and starch added.

Additives	**Dosage (%)**	**Stiffness (Mpa)**	**Std**	**Strength (Mpa)**	**Std**
Blank		1467	141	9.8	0.9
Rape Protein	7.7	1922	360	14.1	1.7
Soy Protein	14.3	2129	580	16.3	2.3
Casein	7.7	1605	261	12.2	2.3
Sodium casein	7.7	2072	817	19.4	4.6
Wheat flour	7.7	1751	390	12.2	1.5

On labscale the mechanical performance did improve by addition of protein and starch. A further improvement was found by raising the added amount of protein. These results could not be achieved in the normal process at Biotrem. Two explanations for the differences between the results on labscale and the normal process are the formation of bubbles in the product in the process at biotrem that did not occur at ATO (possibly due to the longer pressing time at ATO) and the attraction to water of the protein. (the flow of the bran decreased by adding protein most likely because the moisture content of the bran decreased).

Additives	Dosage (%)	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m^2)	Std	Cobb120 (g/m^2)	Density (g/cm^3)
Blank		1299	106	8.7	0.9	3.5	0.3	0.52	0.08	97	1.13
Sodium cassein	14	1291	288	11.3	1.4	4.9	0.6	0.69	0.09	124	0.97
Soy protein	14	1019	101	7.0	0.8	3.3	0.4	0.65	0.10	145	1.12
Rape protein	14	1258	172	7.3	0.4	3.4	0.2	0.54	0.15	111	1.10
Wheat Flour	14	1036	187	8.5	1.0	3.9	0.5	0.65	0.12	129	1.10
Cassein	14	1343	94	7.7	0.9	3.9	0.4	0.56	0.08	126	1.16

It can be concluded that by removal of starch the flow of the bran is reduced. The glueing component was not determined, however overall experience leads to the conclusion that starch, protein and arabino-xylan all participate in the strengthening of the bran product.

4.2.10 Optimal process on existing equipment

Based on the gained knowledge in March 2003 plates were produced combining mildly extruded bran with a lower moisture content and a three stage process. Moisture content was reduced to 15.8% which resulted in a flow of the extruded bran comparable with the flow of normal bran at a moisture content of 18.5%. Visual differences between the five and three stage products of extruded bran were only observed by experts, the normal consumer will not see the difference. Mechanical properties of the extruded bran plates are excellent.

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m^2)	Std	Cobb120 (g/m^2)	Density (g/cm^3)
Blank plate	1183	47	7.4	0.8	5.3	0.6	0.7	0.1	224	1.2
Extruded plate	1794	414	10.0	1.4	5.3	0.8	0.7	0.1	258	1.2

From processing side of view the extruded bran appeared to be less sensitive to changes in processing parameters.

4.2.11 Optimal process

Based upon the results from the experiments and the experience gained during the visits to Biotrem an optimal process for the production of 100% bran based articles will consist of three stages.

The first stage will be a mild extrusion stage where the bran particles are mixed with the water, extruded and pre-heated. The second stage will be a pressing stage in a open mould, comparable to the mould of the current process, in this stage the bran is flowing inside the mould, part of the water is evaporated directly after opening the mould. The third stage is a consolidating stage, the bran product is kept at a certain pressure inside a closed mould, however steam is allowed to escape through the mould. The duration of the last stage will be set based on the optimal moisture content of the product.

4.3 Binders

4.3.1 Bionolle

Results of experiments in which Bionolle has been tested as binder material are summarized in appendix.II. The first 3 series experiments has been based on bran with a moisture content of approx. 12,0%. Bran with three different types of Bionolle (main difference is melt flow index) were moulded at respectively 140°C, 160°C and 180°C. The results displayed at 140°C are unreliable, because the Bionolle isn't completely melted at this temperature.
The flexural stiffness at 160°C and 180°C decreases with increasing MFR. An increase of MFR resulted in a lower viscosity, so a better flow of the binder, which directly resulted in a decrease of flexural stiffness. Remarkable is the decrease in Cobb-value at 180°C. Unfortunately a temperature of 180°C is not preferred in practice.

In a fourth testserie Bionolle #3020 has been used as binder material. The parameter varied is the moisture content. Remarkable is the decrease in Cobb-value at increasing moisture content.
In the last serie the percentage Bionolle is varied. The decrease in flexural stiffness and Cobb-value with increasing percentage binder as can be seen from the results is according expectations. .

It can be concluded that Bionolle is useful as binder material. An increase in moisture content and percentage binder with a minimal temperature of 160°C gives a positive effect on the product properties. A disadvantage of using Bionolle is the relatively high processing temperature of 160°C in stead of the normal process conditions with a temperature of 140°C.

4.3.2 Ecoflex

Both the results of Ecoflex MFI 3 and Ecoflex MFI 10 show a decrease in flexural stiffness with increasing percentage Ecoflex. The flexural stiffness of bran/binder mixtures with Ecoflex MFI 3 and MFI 10 are comparable regarding the fault margin. As expected Ecoflex MFI 10 has a higher impact strength and lower Cobb-value compared with Ecoflex MFI 3. The lower viscosity of MFI 10 resulted in a higher/better flow through the bran.
Comparison of the results of extruded bran/Ecoflex mixtures with ATO blank shows a drastic reduction of the flexural stiffness, similar impact strength, but a relatively low Cobb-value.

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
ATO Blank	1206	426	8.1	2.3	6.4	1.8	1.0	0.1	329	1.2
5% Ecoflex MFI 3	923	106	9.8	1.3	7.1	1.0	1.5	0.3	103	1.2
10% Ecoflex MFI 3	748	128	7.1	1.0	4.6	0.7	1.4	0.4	126	1.1
20 % Ecoflex MFI 3	355	110	5.2	0.8	3.1	0.5	1.2	0.4	71	1.1
5% Ecoflex MFI 10	1449	502	11.2	1.9	10.6	1.8	1.5	0.1	42	1.1
10% Ecoflex MFI 10	967	351	8.6	2.1	8.2	1.2	1.8	0.1	66	1.1
20 % Ecoflex MFI 10	545	210	5.8	0.8	4.1	0.5	1.9	0.2	62	1.1
10% Ecoflex extruded	665	142	4.8	0.7	4.0	0.6	1.2	0.2	50	1.2
20 % Ecoflex extruded	733	156	5.3	0.4	4.6	0.3	1.0	0.1	66	1.3

It can be concluded that Ecoflex MFI 10 is a good option to use as binder material. Also the good adhesion of the bran-based product (improved with Ecoflex binder) (See 4.4) to a coating to be applied is an important reason to choose Ecoflex as binder material. The adhesion of Ecoflex to the mould materials has to be optimized.

4.3.3 Eastar BIO GP

The usage of Eastar Bio GP as binder material has positive effects on the mechanical properties. A minimum amount of 5% percent binder is needed to improve the mechanical properties. With a higher percentage binder relatively high impact strength can be reached. Mechanical properties and density of Eastar/bran combinations are comparable with materials with Ecoflex MFI 10 as binder.
Only the Cobb-values of Ecoflex MFI 10 is significantly lower compared to Eastar Bio GP.

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
ATO Blank	1206	426	8.1	2.3	6.4	1.8	1.0	0.1	329	1.2
5% Eastar BIO GP	1290	280	8.5	1.9	7.0	1.5	1.5	0.2	184	1.1
10% Eastar BIO GP	994	86	8.4	1.2	6.6	0.9	1.9	0.3	121	1.2
20% Eastar BIO GP	508	65	6.0	1.1	4.7	0.8	2.2	0.3	71	1.1

In addition to Ecoflex MFI 10, Eastar Bio GP is a promising biodegradable binder as well. Samples made with Eastar Bio look even better than with Ecoflex MFI 10.
A positive property of Eastar Bio is that it melts at lower temperatures as Ecoflex.

4.3.4 Biopar

The properties of moulded plates out of bran combined with Biopar binders are not significantly better compared with the ATO blank. An exception is the Cobb-value of the moulded plates.
A possible reason that the mechanical properties of Biopar are not significantly better compared with the blank is caused by the material composition of Biopar. The composition of Biopar must be and can be further optimized before it is suitable as binder material in bran based products (ATO performs a lot of development work on this specific material). As can be seen from the Cobb-values, Biopar can be a promising material. The relative low price and renewable character of this material confirm this.

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
ATO Blank	1206	426	8.1	2.3	6.4	1.8	1.0	0.1	329	1.2
5% Biopar	1464	268	9.8	1.5	6.8	1.1	1.2	0.3	118	1.2
10% Biopar	1390	183	8.3	0.6	5.4	0.4	1.0	0.2	97	1.2
20% Biopar	714	163	7.8	1.2	4.4	0.7	1.3	0.2	87	1.2

It can can be concluded that Biopar compositions must be further optimized before this material can be used as binder.

4.3.5 Natureworks PLA 4042 D

The properties of moulded plates out of bran combined with tested PLA binders are not significantly better compared with the ATO blank. One reason that the properties of PLA bran combinations are not significantly better than the blank can be referred to as the effect of moisture. While the bran must have a moisture content of at least 10% to flow, this moisture content has negative effect on the properties of PLA itself. This resulted in samples with many holes, therefore the measured density is relatively low. Moreoverthe melt temperature of PLA (190°C) is considerably higher than the desired processing temperature (140°C).

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
ATO Blank	1206	426	8.1	2.3	6.4	1.8	1.0	0.1	329	1.2
5% PLA 4042D	976	308	11.2	0.4	9.7	0.3	1.0	0.1	166	1.0
10% PLA 4042D	1111	242	9.6	1.8	9.4	1.7	1.1	0.1	216	0.9
20% PLA 4042D	1450	284	12.2	1.8	11.8	1.7	1.2	0.3	179	0.9

It can be concluded that Natureworks PLA 4042D as such is not suitable as binder material in combination with bran.

4.3.6 Cellulose di-acetate

Three series of experiments with bran/CD combinations have been performed. Results are shown below. The first series shows that different percentages of not pre-extruded plasticized CD, made of CD powder and liquid plasticizer, has no positive effect on all properties.
In the second series plasticized CD, made by extrusion, milled and added as powder to the bran was tested. This shows significantly better results. Both mechanical properties and Cobb-value are promising properties.
In the last experiment described the complete CD/bran composition was made with help of extrusion. Bran and plasticized CD have been extruded to realise a homogeneous composition. Moulding of these compositions results in products with high Cobb values, low toughness and low stiffness.

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
ATO Blank	1206	426	8.1	2.3	6.4	1.8	1.0	0.1	329	1.2
5% (CD + 20% TEC)	1341	214	8.2	1.4	9.4	1.6	1.0	0.1	311	1.1
10% (CD + 20% TEC)	925	169	9.0	2.1	9.7	2.3	1.4	0.2	226	1.2

20% (CD + 20% TEC)	958	246	10.1	2.2	11.2	2.4	1.3	0.2	400	1.2
5% (CD + 30% TEC)	1215	5	8.8	1.2	9.0	1.3	1.1	0.3	505	1.3
10% (CD + 30% TEC)	989	189	13.9	2.8	13.0	2.6	1.4	0.1	316	1.1
20% (CD + 30% TEC)	1034	215	10.5	2.0	10.5	2.0	1.4	0.2	371	1.1
5% (CD + 40% TEC)	1372	277	9.4	1.0	9.6	1.0	1.1	0.1	389	1.1
10% (CD + 40% TEC)	943	218	7.8	1.3	7.9	1.3	1.2	0.1	205	1.0
20% (CD + 40% TEC)	1095	261	8.8	1.3	8.2	1.3	1.6	0.2	313	1.1
5% Plasticised CD Powder (extruded) + bran	385	89	5.0	0.7	4.7	0.6	1.2	0.1	116	1.1
10% Plasticised CD Powder (extruded) + bran	441	71	4.6	0.7	3.8	0.5	1.7	0.1	58	1.3
10% Plasticised CD material + bran (extruded).	579	45	3.6	0.6	3.1	0.5	0.6	0.1	432	1.2

Up till now plasticized CD that is added as powder to the bran material has proven to be suitable as binder material..

4.3.7 Water soluble binders

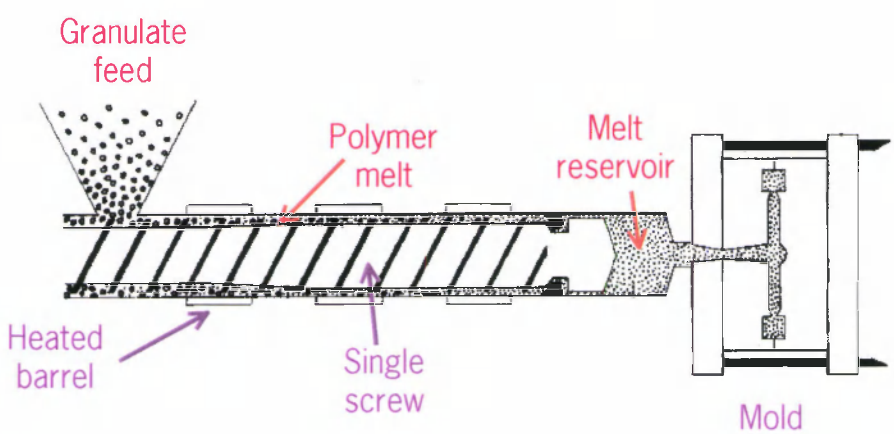
The mechanical properties of polyvinyl alcohol/acetate as water-soluble binder are not significantly better compared with the ATO blank. An exception is the Cobb-value of these binders.

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
ATO Blank	1206	426	8.1	2.3	6.4	1.8	1.0	0.1	329	1.2
10% Mowiol 3-98	1038	310	8.7	1.6	7.5	1.4	1.1	0.2	87	1.2
10% Mowiol 3-83	1145	233	9.0	1.7	7.7	1.5	1.0	0.1	79	1.2
10% PVAXX W20	1142	297	7.8	1.5	7.2	1.4	1.0	0.1	74	1.2

However, adding water-soluble binders during the process is a relatively easy and lead to more water-resistant products, so more research is adviced.

4.4 Injection moulding

Besides compression moulding, injection moulding is a possible other moulding technique to make bran-based products. Injection moulding is a process in which melted compositions are injected into a thermostated mould at high pressures. The composition is fed into a heated barrel in which a screw transports the material.. Subsequently the material is injected into the mould at high pressures. The product is cooled under pressure. When the product has cooled down, the mould is opened and the product can be taken away. A great advantage of this technique is the relatively short processing times and good reproducebility of the process.



In a feasibility study injection moulding bars were made based on bran with a thermoplastic binder. Experimental bars are made acording to ISO 294 on a DEMAG D60 NC IIIK injection moulding machine.
The mechanical properties are tested on a Zwick Z010 testing machine. Properties were measured according ISO 527 with a testing speed of 10 mm/min. Mechanical properties of the injection moulding bars based on bran with thermoplastic binders are given in table below.

Composition	Stiffness (Mpa)	Strenght (Mpa)	Strain at fraction (%)
50% (Ecoflex/PLA) blend	985[241]	9.5[0.5]	3.8[0.5]
40% (Ecoflex/PLA) blend	1058[136]	8.6[1.6]	2.6[0.6]
30% (Ecoflex/PLA) blend	1043[54]	5.3[0.2]	1.3[0.2]
25% (Ecoflex/PLA) blend	943[65]	4.3[0.5]	1.0[0.5]
33% (Biopar) 135°C	1055[29]	7.2[1.2]	1.0[0.3]
33% (Biopar) 155°C	1342[193]	7.5[0.8]	0.8[0.3]

Samples made with the injection moulding technique didn't show significantly better properties compared to samples made with compression moulding.
Injection moulding is a possible technology to make bran based material. The first experiments showed that a minimal amount of 25% binder is needed. Further research must be done to reduce the amount of binder.

4.5 Test at Biotrem

To compare the process and material conditions of ATO bran plates were produced at Biotrem

At Biotrem Poland some important material parameters were varied. Experiments with extruded bran, extruded bran/binder mixtures with different (percentage) binder and bran mixtures with powdered binder were performed. The moisture content and the temperature were varied until the product looks well .
Some plates were tested on various properties given in table below.

Composition	Stiffness (Mpa)	Std	Strenght (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
Blank plate	1183	47	7.4	0.8	5.3	0.6	0.7	0.1	224	1.2
Extruded plate	1794	414	10.0	1.4	5.3	0.8	0.7	0.1	258	1.2
10% EF powder plate	430	58	3.9	0.6	2.9	0.5	1.3	0.2	76	1.1
10% EF powder plate + EF sheet (thermo formed)	409	53	3.8	0.5	2.8	0.4	1.4	0.2	5	1.1
10% extruded CD	600	273	3.2	1.2	2.7	1.0	0.5	0.1	311	1.2

Remarkable is the significant difference of the flexural stiffness between the different materials. Extra Ecoflex powder as binder added doubles the impact strength compared with the standard plate. An extra coating of Ecoflex reduces the Cobb-value to a minimum.

Considering the properties of 10% extruded CD, indicates that the procedure used at ATO is comparable with the procedure used at Biotrem.

Composition	Stiffness (Mpa)	Std	Strenght (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
10% extruded CD made at ATO	579	45	3.6	0.6	3.1	0.5	0.6	0.1	432	1.2
10% extruded CD made at BIOTREM	600	273	3.2	1.2	2.7	1.0	0.5	0.1	311	1.2

4.6 Coating techniques

4.6.1 Lamination coating process by thermoforming

Introduction

Thermoforming is a general term encompassing many techniques for producing useful plastic articles from flat sheet or film (thickness sheet > 1.0 mm; thickness film <1.0 mm). Thermoforming is draping a temporarily softened film over a mould. In case of vacuum forming is the required force to shape a film in the right form delivered by placing vacuum below the film. One advantage of this forming technique is the relatively low pressure necessary for shaping films. The film keeps untouched, while heated to form the end product.

The experiments on a vacuum thermoforming machine to provide a Biotrem products with a film are described below.
During experiments different products were tested:

I. The standard Biotrem products (no binder present); plates ∅ 23 cm and samples (10 x 10 cm) cut out of these plates.
II. Samples (18 x 12 cm) made by compression moulding at ATO. The ATO samples contain 10% Ecoflex MFI 10.
III. A Biotrem product with 10% Ecoflex; plates ∅ 23 cm, produced at Poland.

The samples mentioned above are provided with a singular layer Ecoflex film 50 ± 5 μm made at ATO (internal code 550).
The experiments are carried out at a Mealux 50H, number 460. Some specifications of the Mealux 50H thermoform machine are described in appendix X. The experiments were carried out on a flat mould without plug-assisted mode.

The experiments, which are performed, are described in the table below. All samples, except sample E and L, are made at ATO. At ATO bran, with a moisture content of 18%, is compression moulded at standard conditions (140°C, 5 times 5 sec, 30 tons) with 10% Ecoflex powder (< 0.5 mm).

The term 'pre-heat' in the table indicates that the mould and sample are heated before the film is placed above the sample and becomes vacuum formed. The heating elements are placed above the mould and moved back when the desirable pre-heating temperature is reached. The plate is heated by infrared elements which can reach a maximum temperature of 500°C. Time and temperature of pre-heating depends on the temperature to be reached.
The gauze plate was necessary because of a technical deficiency of the Mealux 50H. This gauze plate resulted in a more homogeneous vacuum around the sample.
The time showed in table 1, is the time of heating the film. The time starts when the heating element is moved above the film and stops when the film becomes vacuum formed.

Sample	**Material**	**Pre-heat**	**Gauze-plate**	**Temperature [°C]**	**Time [s]**
A[1)]	10% EF	-	-	-	-
B[2)]	10% EF	-	-	-	-
C	10% EF		No	500	5 – 10
D	10% EF		Yes	320	25 - 30
E	Standard		Yes	320	25 - 30
F	10% EF	60°C	Yes	320	25 - 30
G	10% EF	90°C	Yes	320	25 - 30
H	10% EF	80°C	Yes	400	10 – 15
I	10% EF	80°C	Yes	400	10 – 15
J[3)]	10% EF	115°C 80°C	Yes	400	10 – 15
K[3)]	10% EF	80°C; 80°C	Yes	400	10 – 15
L	10% EF	90°C	Yes	350	20 – 25

1) Blank sample, not laminated
2) Blank sample, film sealed by hot-press at 140°C, 10 tons, 10 seconds.
3) Samples J and K are sealed at both sides.

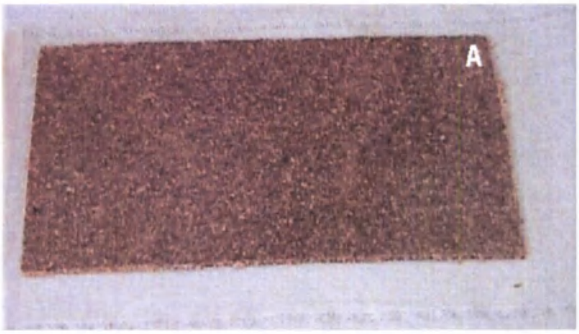
Biotrem plates produced at Poland, are coated in a second visit to the supplier of the thermoforming machinery. A simple ring was made to support the edge of the plate. Some small holes were made in the Biotrem plate to realize vacuum below the film to be thermoformed.

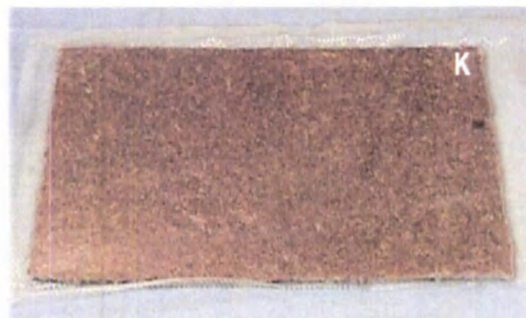
Sample A is a blank made at ATO and is not provided with a film. All ATO samples are compression moulded under the same conditions : a temperature of 140°C and a pressure of 30 ton for 5 times 5 seconds was used.
Sample B is sealed with an Ecoflex film by compression moulding by 140°C for 10 seconds at 10 ton. The film is sealed well, but the sample is distorted by the extra temperature and pressure step.

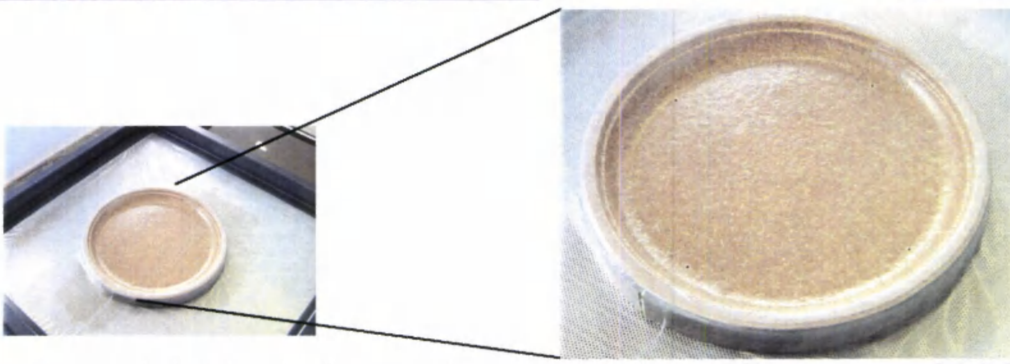
Sample	**Film sealed**	**Sample curved**	**General remarks**
A		+	Blank
B	+ +	+	Sample flowed out during extra heating step used during coating
C	- -	- -	Vacuum is not homogenous. Ecoflex in the sample is not melted; moderate adhesion.
D	- -	+	Vacuum is homogenous because of extra gauze plate. Ecoflex in the sample is not melted; moderate adhesion.
E	- -	+/-	No adhesion between bran based product and Ecoflex film
F	-	-	Moderate adhesion. Ecoflex in the sample is not melted by pre-heating to 60°C.
G	+	+/-	Time for pre-heating is too long (about 1 minute)
H	+	+/-	Sample seems to be fairly correct.
I	+	+/-	Duplicate of sample H
J	+	- -	Sample is curved. Temp. of pre-heating of one side of the sample is too high.
K	+	+ +	Adhesion is at one side of the sample very well (other side is a bit less)
L	++	- -	Optimized product, see test results

Sample C was the first experiment on thermoforming machinery. The forming area of the vacuum machine was too big for these samples. Also the vacuum holes of this machine were too big. An extra gauze plate was needed on the vacuum form machine to overcome this technical defect on the Mealux 50H. This gauze plate resulted in a homogeneous vacuum around the samples.
Probably due to no melting of the Ecoflex in the compression moulded bran samples there was no adhesion of the films of samples C and D. The lower temperature of sample D compared with sample C resulted in a more controllable softening of the Ecoflex film.
As we already know, see the results of sample E, there was no adhesion between the film and the sample on a standard Biotrem product without any Ecoflex in it.
Pre-heating the sample up to 60°C resulted in an improved adhesion of the film but was not optimal. A pre-heating temperature of 90°C by sample G resulted in a slightly melted Ecoflex film before it is vacuum formed. The pre-heating temperature was slightly too high. The temperature of 320°C of both heating elements by samples F and G resulted in a too long pre-heating phase. The samples H and I have a good adhesion at a pre-heating temperature of 80°C. Also the time of pre-heating is acceptable. The sample J and K (see figure 2) are sealed

on both sides. Because of the high pre-heating temperature of one side(115°C) of sample J, the sample is curved. During the latest visit five products made in Poland were coated. The coating adheres well when a binder is added to the product, preferably Ecoflex or Eastar Bio GP.







It can be concluded that the first experiments on the Mealux 50H are successful. It is possible to adhere an Ecoflex film to a sample with approx. 10% Ecoflex. Due to the simplified version of the thermoform machine there must be further research to optimise product and process.
In figure above is a Biotrem plate sealed with an Ecoflex film. With this experiment it is shown that you can provide plate like products with a film with the right equipment and materials.

The used equipment to laminate a Biotrem product has some limitations. The Biotrem plates must be pre-heated to heat the binder. On the used equipment it takes 30-50 sec, depending on the temperature, to pre-heat the binder up to 90°C. Second, in the used set up film to be laminated must be manually applied When the right equipment is used, this pre-heating time can be reduced till a few seconds. An average time to laminate one Biotrem item, on a fully automatic thermoform machine, is less than 20 seconds or even less than 15 seconds.

4.6.2 Lamination coating process by compression moulding

A possible other technique to laminate the Biotrem products is by compression moulding. One route, which can be followed, is based on the carrousel principle described in report Markhorst Aug-Sept 2002.

The production process starts with moulding of a bran/binder mixture (preferably Ecoflex or Eastar is used as binder material). After the normal production steps (five compression moulding stages), the still hot product is removed and placed in a next press. At that press the Ecoflex sheet is heated just above Tg, and sealed under pressure to the products. A temperature of approx. 110 °C is expected for maximum 10 seconds (time, temperature and pressure are some variables). The product will move to a cooling station, following by a cutting and ejection stationLamination coating process by spray coating

5 Economic considerations

An economical evaluation of the different processes to produce bran-based materials will be necessary to evaluate the possibilities of each bran product for a given market and will differ according to the place where the factory will be positioned. An estimation of the production costs will comprise the different cost items as given in the table. In this report no full estimation of the production costs of bran products is presented. However, because in this report new processing stages are presented, to get an idea about the costs of these stages, some costs related to these stages are estimated: the costs of the machines necessary for these stages, the costs of the different binders and coatings and the costs of the solvents. Other costs to be considered in these stages are briefly addressed below.

Cost Item	Examples	
Personnel	• Production • Maintenance • Supervision •	• Staff • Others
Energy	• Pressing • Heating • Extrusion •	• Vacuum • Process water • Other
Chemicals	• Coatings • Binders •	• Solvents
Raw Material	• Bran	• Water
Other Costs	• Supplies • Maintenance materials • Tax and insurance	• Patents • Packaging material
Capital Charge • Machinery	ISBL • Presses • Conveyer Belts • Spray-coating • Vacuum coating • Extruder • Packaging • Others • Electricity • Hoods	OSBL • Toluene/ethanol burner
Capital Charge • Buildings	ISBL • Production building • Energy	OSBL • Lab, offices, stores • Energy • Technical services

In the following paragraphs an estimation of the costs introduced by adding new processing stages in the process are given.

5.1 Production of bran plates using extruded bran

Before the current production process a new processing stage is introduced. In this stage the bran is extruded. This stage will consists of a co-rotating twin screw extruder, preferably placed nearby the presses. The introduction of this stage will result in an increase in production capacity of the current process.

Estimation of the costs for extrusion

The largest extruders available at the moment will cost about 0.85 M€. Based on the experiments done ATO a rough estimate of the production capacity of such an extruder (e.g. Clextral KRO 200) is 20 ktonne/year. The extrusion energy is estimated at 50 kWh/ton. Therefore the extra costs for extrusion based on a 10 year depreciation and an energy price of 0.04€/kWH is 6.25 €/ton bran. For a process that uses 100 grams of bran to produce an item the costs are 0.000625 €/item.

Estimation of capacity increase

The current process consists of 5 pressing stages, using extruded bran this process can be reduced to 3 processing stages. Using the current presses a reduction of cycle time from 32.27 to 27.04 would be achieved, increasing the production rate from 1.86 to 2.22.

5.2 Production of bran plates using thermoforming

One way to laminate bran-based products with a film is by thermoforming. A fully developed thermoform machine can provide a large amount of products per cycle. The estimation of the cost described below is based on thermoform machine that can provide 4 products per cycle.

Estimation of the cost for lamination with thermoforming.

An estimation of the costs for a thermoform machine based on 4 products per cycle is about 100 K€. Based on a cycle of 4 products per 20 sec, a production of 576 ton/year can be reached. The energy is estimated at 0.87 KWh/ton. The extra costs of lamination, based on an Ecoflex film of 25 μm with a lamination efficiency of 90%, and a depreciation of 10 year is about 0.0075 €/item.

A (Ecoflex) film only seals on bran products based on extra binder. An estimation of the extra costs of 7.5% Ecoflex binder is about 0.0225 €/item
Total extra costs for thermoforming an ecoflex film on an product containing ecoflex are about 0.03€/item.

The total costs depending on layer thickness and amount of additional binder:

Total costs €/item			Film thickness [micrometers]		
Binder percentage [%]	**10**	**20**	**30**	**40**	**50**
2.5	0.010	0.012	0.014	0.016	0.018
5	0.017	0.019	0.021	0.023	0.025
7.5	0.025	0.027	0.029	0.031	0.033
10	0.032	0.034	0.036	0.038	0.040

The thermoform process can be optimised by using different stages; first stage to supply the product and a film, next stage to heat and seal the film on the product and a last stage to cool and cut the products. In an optimised process with 12 products process times of at least 10 seconds can be reached.

Adding some other additives or binders can reduce the costs of a binder. Prices of 2.0 €/kg can be reached, while using 5% binder.

Total additional costs for the optimised product can be about 0.015€/item

5.3 Production of bran plates using spray-coating

The extra costs for spray coating are partly described in the Markhorst report: Coating Line concept.

Estimation of the costs for spray-coating

- Assuming a 23cm product the total material costs necessary per product using a rotating product per item will be € 0.0029.
- The costs for a machine capable of spray-coating 5 million items a year is estimated at 60 k€. Using a 10 year depreciation the costs of spray coating machinery is €0.0012 per item.
- Energy consumption is estimated at 2.5 kWh/1000 items at 0.04€/kWH. Energy costs per item is €0.0001.
- Additional costs for removal of toluene and ethanol are not included.
- Costs of additional equipment and space for drying of the coating is not included.

5.4 Overview

The additional costs for the three different process stages that can be added to the current process are estimated. An item produced from 100 grams of bran is assumed.

Cost item [€]	Extrusion	Thermoforming[1]	Spray coating[2]
Material	-	0.0058	0.0029
Process			
• Depreciation	0.000425	0.0017	0.0012
• Energy	0.0002	~ 0	0.0001
• Drying equipment			?
Extra			
• Binder	-	0.0225[3]	0.0225[3]
• Gas removal	-		?

[1] Coating layer of 25 μm
[2] Coating layer of 10 μm
[3] Coating adhesion to a plate containing binder is preferred.

- Additional costs for extrusion are limited, because of the large capacity of an extruder.
- A fair comparison between thermoforming and spray coating is not possible based on a simple calculation of the additional costs. A large storage place is necessary to deal with the long drying time of spray coated plates, the costs for removal of the toluene and ethanol are not included, however if such an industry would be set up in the Netherlands a sufficient solution will have to be found.

6 Conclusions

6.1 Current production process

- Concerning the plates produced at Biotrem, significant differences between stiffness, maximum stress, impact, water adsorption and density is found between typical samples being produced at the production site. The differences between the products are due to different processing parameters on the presses and the different moulds .
- An optimal correlation between the amount of bran introduced (not too thriftily not too generously) and the size of the mould is necessary to obtain an optimal product.
- A minimum amount of water inside the bran is needed to obtain flow during processing. Extrusion of the material increases the flow, therefore less pressure or lower water content can be applied. Based on mechanical properties It is advised that an initial water content of around 15 wt.% is applied for articles, which require high stiffness, whereas an initial water content of around 18 wt.% is applied for articles, which require high strength.
- Theoretical analysis shows that several pressing stages are necessary to reduce the amount of water present in the bran to the desired equilibrium water content of about 11%. Depending on the temperature of the mould and the heat exchange rate, the products will be either, to dry, to moist or precisely at the equilibrium water content of the product after storage at normal conditions. Increasing the temperature of the mould results in less pressing stages needed; the model shows that, ideally, only two stages are needed to obtain the desired water content.
- Concerning time reduction of the first pressing stage an optimum is found for a duration of 1.5 seconds. Optimising the process by adjusting the duration of each stage to the maximum duration that does not create an explosion, does not result in optimal mechanical performance of the product. Optimising the process parameters should be done by evaluating on optical and mechanical performance.
- Concerning density, it is shown that during the final stages of the process the density increases. It Assuming that a product of good quality can only be achieved if the density is about 1200 kg/m^3, most likely, under the currently applied processing conditions, 5 stages are necessary to produce end products of sufficient quality. This can only be reduced by proces- and material adaptations (see below)

6.2 Extruder pre-processing

- Plates produced at Biotrem from *mildly* extruded bran show a significant increase in strength and stiffness relative to its reference. With severe extrusion this behaviour is not observed.
- The cobb-value of the extruded plates is higher than the cobb-value of the normal plates. Most likely this is caused by the smaller particle size of the bran. An additional top layer is necessary for applications with a critical water retention requirement.
- Using extruded bran the process is less sensitive to changes in moisture content concerning the formation of bubbles. Less critical processing parameters will result in higher productivity.
- The adhesion of coatings improved when extruded bran was used as raw material for the products.

- Concerning the estimation of the costs for extrusion, the extra costs based on a 10 year depreciation and an energy price of 0.04€/kWH is 6.25 €/ton bran. For a process that uses 100 grams of bran to produce an item the costs are 0.000625 €/item.
- Concerning the estimation of capacity increase, the current process consists of 5 pressing stages; using extruded bran this process can be reduced to 3 processing stages. Using the current presses a reduction of cycle time from 32.27 to 27.04 would be achieved, increasing the production rate from 1.86 to 2.22.

Based on the gained knowledge plates were produced at Biotrem combining mildly extruded bran with a lower moisture content and a three stage process. Moisture content was reduced to 15,8% which resulted in a flow of the extruded bran comparable with the flow of normal bran at a moisture content of 18.5%. Visual differences between the five and three stage products of extruded bran were only observed by experts, the normal consumer will not see the difference. Mechanical properties of the extruded bran plates are excellent.

6.3 Additional binders

Conclusions on the material properties

- Thermoplastic binders can be added during pretreatment of the bran or just before moulding of the products (binders can be added as powder, films or fibres). Up till now addition of thermoplastic binders in powder form just before moulding results in the best properties. In this way an increase of 100 % in toughness and significant decrease in Cobb value can be realised by adding 5 -10 % binder, preferably 7.5 %. Proven suitable thermoplastic binders for the actual process (± 140°C) are Ecoflex MFI 10 and Eastar Bio GP. These binders have a flexible character. So the stiffness of the bran/binder compositions decrease in comparison with pure bran materials.
- Melt flow index is an important property of the binder system. By using binders with a high MFI (about 10-20 gram/10 min measured at 190 °C and 2.16 kg) a tough material could be realised. This effect can be explained from viscosity effects. Depending on stiffness of the binder various stiffnesses of the bran/binder system can be realised.
- Adding a binder to the bran results in additional flow (due to its low viscosity) of the bran/binder composition. Additional flow can be used for realising thinner products, bigger products (increased flow lengths) and reduction of process cycle time (lower moisture contents can be started with). In addition, adding binders hardly influences on the total density.
- Overview of Thermoplastic binders

Binder	Renewable	Price €/kg	Toughness	Processability	Possibilities in future
Bionolle	-	5.0-5.5	+	0	-
Ecoflex	-	3.0-3.5	+	+	+
Eastar Bio	-	3.0-3.5	+	+	+
BioPar	0	2.5-3.0	0	+	++
Natureworks PLA	+	3.0-3.5	0	-	0
Cellulose di-acetate	+	3.0-3.5	0	+	0

- Usage of water soluble binders is another promising dosing technique. Up till now only an increase of water-resistance can be realised by adding these type of binders.
- Up till now no additional value has been proven on actual scale of addition of natural proteins as binder to the bran material.

Conclusions on binder processing

- Compression moulding: thermoplastic binders can be added during pretreatment of the bran (extrusion) or just before moulding of the products (in this way binders can be added as powder, films or fibres). Up till now addition of thermoplastic binders in powder form just before moulding results in the best properties
- Compression moulding: properties of plates made at Biotrem Poland and samples made at ATO Wageningen indicate that the procedure used at both plants is comparable. Although the absolute value for toughness is not the same (toughness of articles made in Poland are lower in comparison with articles made in Holland) both results show an increase of 100 % in toughness by adding suitable binders.
- Injection moulding: Injection moulding is another, possible technology to make bran based products. However, a minimum amount of 25% binder is needed.

Conclusions on coating by thermo forming

- Direct adhesion between bran and Ecoflex is not well. Adhesion improvers are necessary. One possibility is adding Ecoflex binder to the bran material.
- Coating of products with Ecoflex film by thermoforming is quite suitable process. It can perfectly added into the carrousel principle suggested by Markhorst.
- Time to laminate one Biotrem item, on a fully automatic and optimised thermoform machine, is less than 20 seconds and even less than 15 seconds.
- Costs: Material costs of Ecoflex to coat one plate ∅ 230 mm of a bran/Ecoflex mixture (5 % Ecoflex binder is added) with 20 μm Ecoflex film are
  - Costs Ecoflex binder: € 0.015
  - Costs Ecoflex coating: € 0.004

Conclusions on coating by compression moulding

- Compression moulding is a possible other technique to laminate Biotrem products. No other type of machinery has to be added to the production facilities.
- In comparison with compression moulding of bran/binder mixtures, process temperature of coating by compression moulding has to be significantly lowered (from about 140 to about 110 °C).

7 Further research

7.1 Current production process

From the results of this research project two directions for further research concerning the optimisation of the current production process can be established:

- Determination of the effect of the age of the products on the mechanical performance.
  - What is the change in mechanical properties in time.
  - What is the change in water adsorption in time.

- Determination of the processing parameters under controlled conditions

From the four visits it is clear that the processing parameters and the properties of the products are changed continuously. It is unclear to what extent the different parameters are contributing to the differences in product properties. Undoubtedly improving the quality of the presses and the moulds will decrease the differences in the product properties, however the product properties are also influenced by other processing parameters, especially by

- The temperature and humidity of the processing environment and the storage facilities.
- The quality of the mixing between water and bran.
- The duration of the time between mixing the water and bran and the actual pressing of the product.
- The quality of the used bran (quality of the wheat)

For the production at large scale, understanding and controlling the above mentioned parameters will be necessary to obtain an expectable productivity rate. Ideally this research should be performed at ATO on an press equivalent to the presses to be used at Biotrem, enabling the measurement of all processing and product parameters under monitored conditions.

7.2 Extrusion pre-processing

From this research it can be concluded that the addition of a extrusion stage prior to the pressing stages is a promising change in the process. The main advantages are a quicker and less sensitive process, a better adhesion to coatings and improved mechanical properties. Further development should concentrate on a process where extrusion is directly followed by pressing. By doing so the pre-heating effect of the extruder is fully used, enabling an increase in production (items/min).
For the production at large scale full control and understanding of the combination of extrusion and pressing is necessary.
The main focus should be on the process described in chapter 4, consisting of three processing stages: extrusion, pressing and consolidation.

7.3 Binder systems

From this research it can be concluded that the addition of binders to the bran results in an enormous increase in toughness. However, these binders costs money. To minimize the costs of these binders the following research is needed (the following questions have to be answered):

- Can the weight of the bran based products be reduced (or can the thickness of the compression products be reduced) ? If this weight can be reduced the absolute weight of binder to be added can be reduced too. As is shown in paragraph 5.2 the binder system (Ecoflex or Eastar Bio) is the most expensive part of a water resistant, tough bran based product.
- Optimisation of the stiffness of the bran based products. It is shown that when Ecoflex or Eastar Bio is used, tough products can be realised. However, the stiffness of these materials decreases too. This is due to the very flexible character of Ecoflex and Eastar Bio. By material modification these stiffnesses can be increased.
- Usage of renewable materials as binder system. Ecoflex and Eastar Bio are both examples of non-renewable, biodegradable materials. Up till now, tests with renewable binders didn't result in good properties. For sure, there are many possibilities with these renewable binder systems (e.g. Biopar material). More research is needed.
- Binder systems comprising out of water soluble materials. Addition of this kind of binder systems can easily be realised in the current process (in the current process water has to be added to the bran). We have to look for water soluble binder systems that result in the same mechanical improvement as the thermoplastic binder systems.
- Scale up problems with binder systems. The system which has proven to give the best results starts from powdered binders. To get powder from flexible systems as Ecoflex or Eastar is not trivial. Together with an industrial partner a scale up program has to be designed.

7.4 Coating by thermoforming or compression moulding

From the research as described in this report it can be concluded that for relative thick coatings (thickness > 20 micron) coating by thermoforming or compression moulding can be an economical feasible way compared to spray coating to coat a bran based product. Especially when articles have to withstand hot materials there are more possibilities with these coating techniques. Moreover, when multilayer systems are needed, these coating techniques are favourable too. In the research described here the principle has been proven. It has to be worked out in more detail to realise a production line with this process.

7.5 Spray-coating

Bran-based articles that are coated with Ecoflex show very good water resistance (Cobb value zero). Formulations of Ecoflex dispersions and spraying conditions have been thoroughly studied. Therefore, probably no further research is necessary on this system.

Although a Ecoflex coating is very water resistant, a major disadvantage is the use of toluene as cosolvent. For safety reasons, environmental reasons and the goal of a "green" and food contact approved coating, future research should focus on water-born systems. In the previous period preliminary experiments were carried out with *copolymers of polyvinylalcohol and polyvinylacetate*. Free standing films did not dissolve in water. Dispersions were easy to process and the water resistance was in some cases fairly good. As was noticed, the fibers of the bran absorbed very fast water causing little holes in the coating. Coatings that were based on dispersions with the higher dry matter content, had less holes compared to coatings derived from the dispersions with a low dry matter content. More research is necessary to define and improve the properties of these coatings.
Research topics:

- Investigating different copolymers with e.g. variation in alcohol and acetate content
- Effect of dry matter content and viscosity
- Effect of spraying conditions on coating properties
- To prevent water uptake by bran fibres, drying conditions have to be optimised. Also, infra red drying could be an interesting option.
- Effect of hydrophobic clay in the formulation.

Preliminary experiments have been carried out with *zein*, a very hydrophobic protein. Coating quality was good although the dispersions were applied using a hand sprayer. Since zein is soluble in 85% ethanol, problems with water uptake by bran fibres, could possible be solved.
Research topics:

- Formulation of dispersions (alcohol content and dry matter content)
- Effect of spraying and drying conditions on film quality

Thirdly, part of research could attributed to *other materials*. In literature materials are described that are used as water barriers. However, mechanical properties of these coatings, prices and rheological behaviour have to be clarified.
Materials of possible interest:

- Paraffin and other waxes
- Modified linseed oil
- Shellac
- Hydroxypropylcellulose

Appendix I. Extrusion

Extrusion experiments were performed using a twin screw, co-rotating extruder, Clextral BC 45. Premix consisting of bran-binder was fed at the inlet of the extruder.
The extruded bran-binder composition was milled (Retsch) by a 2.0 mm sieve to homogenise the composition.

Technical data is describe in table below:

Composition	**10%EF[1)]**	**20%EF[1)]**	**10%CD[2)]**
Back pressure (Bar)	3-7	3-5	3-7
Speed (RPM)	40	50	40
Current (A)	69	50-60	34-39
Throughput (kg/h)	34.6	36.0	35
Temperature (°C)			
Zone 1 – 20°C	15	14	15
Zone 2 – 140°C	131	134	140
Zone 3 – 140°C	117	116	116
Zone 4 – 140°C	143	143	140
Zone 5 – 140°C	98	100	98

[1)] Ecoflex granulate is delivered by BASF
[2)] Cellulose di-acetate with 40% tri-ethyl-citrate as plasticizer was first made on a Berstorff ZE 25 extruder at ATO. The screw configuration and special data is described in an internal report.

Appendix II. Model study of Bionolle

Code (#)	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
10% #3001 140°C 12% MC	756	342	7.4	1.1	8.4	1.2	1.5	0.2	118	1.1
10% #3010 140°C 12% MC	1047	122	7.7	1.7	9.6	2.1	1.8	0.2	111	1.1
10% #3020 140°C 12% MC	634	426	7.5	2.1	8.6	2.4	1.7	0.2	136	1.1
10% #3001 160°C 12% MC	1310	286	10.6	0.7	12.3	0.8	1.4	0.3	132	1.1
10% #3010 160°C 12% MC	1178	162	8.7	1.2	10.4	1.5	1.9	0.4	125	1.1
10% #3020 160°C 12% MC	936	142	7.3	1.0	8.7	1.2	2.0	0.4	118	1.1
10% #3001 180°C 12% MC	1311	149	10.2	0.5	12.6	0.6	1.5	0.1	89	1.1
10% #3010 180°C 12% MC	1487	155	12.3	1.8	14.3	2.1	1.7	0.1	71	1.1
10% #3020 180°C 12% MC	1055	297	8.9	1.3	9.7	1.4	1.7	0.1	68	1.1
10% #3020 160°C 6% MC	856	71	5.6	0.6	6.6	0.7	1.4	0.2	146	1.3
10% #3020 160°C 9% MC	1133	67	6.7	0.4	7.9	0.5	1.5	0.1	139	1.3
10% #3020 160°C 12% MC	936	142	7.3	1.0	8.7	1.2	1.4	0.3	118	1.1
10% #3020 160°C 15% MC	977	125	7.8	1.1	9.1	1.2	1.1	0.2	95	1.0
10% #3020 160°C 18% MC	840	81	5.3	0.9	6.7	1.1	1.1	0.2	72	1.2
5% #3020 160°C 12% MC	1282	149	8.3	0.4	10.0	0.5	1.3	0.3	157	1.1
10% #3020 160°C 12% MC	936	142	7.3	1.0	8.7	1.2	1.4	0.3	118	1.1
20% #3020 160°C 12% MC	852	202	10.1	0.7	10.8	0.8	1.9	0.2	79	1.1

Appendix III. Results of thermoplastic binders

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
ATO Blank	**1206**	**426**	**8.1**	**2.3**	**6.4**	**1.8**	**1.0**	**0.1**	**329**	**1.2**
5% Ecoflex MFI 3	923	106	9.8	1.3	7.1	1.0	1.5	0.3	103	1.2
10% Ecoflex MFI 3	748	128	7.1	1.0	4.6	0.7	1.4	0.4	126	1.1
20 % Ecoflex MFI 3	355	110	5.2	0.8	3.1	0.5	1.2	0.4	71	1.1
5% Ecoflex MFI 10	1449	502	11.2	1.9	10.6	1.8	1.5	0.1	42	1.1
10% Ecoflex MFI 10	967	351	8.6	2.1	8.2	1.2	1.8	0.1	66	1.1
20 % Ecoflex MFI 10	545	210	5.8	0.8	4.1	0.5	1.9	0.2	62	1.1
10% Ecoflex extruded	665	142	4.8	0.7	4.0	0.6	1.2	0.2	50	1.2
20 % Ecoflex extruded	733	156	5.3	0.4	4.6	0.3	1.0	0.1	66	1.3
5% Eastar BIO GP	1290	280	8.5	1.9	7.0	1.5	1.5	0.2	184	1.1
10% Eastar BIO GP	994	86	8.4	1.2	6.6	0.9	1.9	0.3	121	1.2
20% Eastar BIO GP	508	65	6.0	1.1	4.7	0.8	2.2	0.3	71	1.1
5% Biopar	1464	268	9.8	1.5	6.8	1.1	1.2	0.3	118	1.2
10% Biopar	1390	183	8.3	0.6	5.4	0.4	1.0	0.2	97	1.2
20% Biopar	714	163	7.8	1.2	4.4	0.7	1.3	0.2	87	1.2
5% PLA 4042D	976	308	11.2	0.4	9.7	0.3	1.0	0.1	166	1.0
10% PLA 4042D	1111	242	9.6	1.8	9.4	1.7	1.1	0.1	216	0.9
20% PLA 4042D	1450	284	12.2	1.8	11.8	1.7	1.2	0.3	179	0.9

Appendix IV. Results plasticized cellulose di-acetate

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
ATO Blank	1206	426	8.1	2.3	6.4	1.8	1.0	0.1	329	1.2
5% (CD + 20% TEC)	1341	214	8.2	1.4	9.4	1.6	1.0	0.1	311	1.1
10% (CD + 20% TEC)	925	169	9.0	2.1	9.7	2.3	1.4	0.2	226	1.2
20% (CD + 20% TEC)	958	246	10.1	2.2	11.2	2.4	1.3	0.2	400	1.2
5% (CD + 30% TEC)	1215	5	8.8	1.2	9.0	1.3	1.1	0.3	505	1.3
10% (CD + 30% TEC)	989	189	13.9	2.8	13.0	2.6	1.4	0.1	316	1.1
20% (CD + 30% TEC)	1034	215	10.5	2.0	10.5	2.0	1.4	0.2	371	1.1
5% (CD + 40% TEC)	1372	277	9.4	1.0	9.6	1.0	1.1	0.1	389	1.1
10% (CD + 40% TEC)	943	218	7.8	1.3	7.9	1.3	1.2	0.1	205	1.0
20% (CD + 40% TEC)	1095	261	8.8	1.3	8.2	1.3	1.6	0.2	313	1.1
5% extr. powder	385	89	5.0	0.7	4.7	0.6	1.2	0.1	116	1.1
10% extr. powder	441	71	4.6	0.7	3.8	0.5	1.7	0.1	58	1.3
10% extr. blend	579	45	3.6	0.6	3.1	0.5	0.6	0.1	432	1.2

Appendix V. Results water soluble binders

Composition	Stiffness (Mpa)	Std	Strength (Mpa)	Std	F-max (N)	Std	Toughness (KJ/m²)	Std	Cobb120 (g/m²)	Density (g/cm³)
10% Mowiol 3-98	1038	310	8.7	1.6	7.5	1.4	1.1	0.2	87	1.2
10% Mowiol 3-83	1145	233	9.0	1.7	7.7	1.5	1.0	0.1	79	1.2
10% Craymul 2323	906	141	6.4	1.0	5.4	0.8	1.1	0.2	89	1.3
10% PVAXX W20	1142	297	7.8	1.5	7.2	1.4	1.0	0.1	74	1.2

Appendix IX. Results visit Poland

Code (#)	Materials	Moisture content (%)	Temperature (°C)
I	Standard bran	18.0	114/114
II	Extruded bran	15.6	114/119
III a	Extruded bran with 10%(CD+40%TEC)	18.0	114/119
III b	Extruded bran with 10%(CD+40%TEC)	13.9	114/119
IV a	Standard bran with 5% powder (CD+40%TEC)	13.9	115/115
IV b	Standard bran with 5% powder (CD+40%TEC)	13.9	120/120
IV c	Standard bran with 5% powder (CD+40%TEC)	13.9	125/125
V a	Extruded bran with 20% Ecoflex	18.0	118/124
V b	Extruded bran with 20% Ecoflex	12.0	118/124
V c	Extruded bran with 20% Ecoflex	8.3	118/124
VI a	Extruded bran with 10% Ecoflex	10.0	118/124
VI b	Extruded bran with 10% Ecoflex	8.5	118/124
VII a	Standard bran with 5% Ecoflex powder	18.9	114/114
VII b	Standard bran with 5% Ecoflex powder	13.7	114/114
VII c	Standard bran with 5% Ecoflex powder	13.7	120/120
VIII a	Standard bran with 10% Ecoflex powder	13.7	125/128
VIII b	Standard bran with 10% Ecoflex powder	13.7	140/140
VIII c	Standard bran with 10% Ecoflex powder	13.7	114/114

Remarks:
- Weight bran inserted during process: ± 100 g; weight plate ∅ 230 mm: ± 80 g.
- Surrounding temperature; 12°C and relative humidity 38%
- Moisture content measured with Draminski moisture meter.

Process times

1st pressing step: 14 s:	0-14 seconds
2nd pressing step: 8 s:	14-22 seconds
3rd pressing step: 3 s:	22-25 seconds
4th pressing step: 2 s:	25-27 seconds
5th pressing step: 1 s:	27-28 seconds
Opening press: 5 s:	28-33 seconds

With the right moisture content and the binder percentage can you reduce the process time cycle.

Appendix X Mealux 50 H

Type	Mealux 50H
Sheet dimension	500 x 500 mm
Forming area	450 x 450 mm
Forming depth	300 mm
Sheet thickness	0.1 - 3 mm
Heating capacity	7 kW
Heating zones	2
Heater type	Quarz heaters
Capacity vacuumpump	10 m³/hr
Voltage	400V 3ph, 50Hz,
Area l x b	1500 x 700 cm
Total height	1350 mm

Price of thermoform machine Mealux 50 useful for Biotrem products: € 20.000,-

Appendix XI Material comparison

Materials	Properties - process	Availability	Price €/Kg
Bionolle	Medium	Granulate	5.0-5.5
Ecoflex	Good	Granulate	3.0-3.5
Eastar BIO GP	Good	Granulate / Fibres	3.0-3.5
BioPar	Medium	Granulate	2.5-3.0
PLA	Low	Granulate / Fibres	3.0-3.5
Plasticised CD	Low	Granulate	3.0-3.5
PVOH/PVAc - Mowiol		Powder	3.0
PVOH/PVAc – PVAXX W20	Medium	Granulate	2.5-3.0
PVOH/PVAc - Craymul		Dispersion	