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STRAW UTILIZATION DEPARTMENT
GRONINGEN - THE NETHERLANDS

REPORT

SUBJECT	: The manufacturing of light-coloured cardboard on laboratory scale from acidified sulphite pulp obtained from Jordan wheat straw.
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ANNEXES	: 3 tables 1 appendix 1 graph.

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SUMMARY

It was well-known to us that when neutral straw sulphite pulp is acidified its colour becomes brighter.

A quantitative investigation of this effect was made with neutral sulphite pulp from Jordan wheat straw.

The aim was to ascertain to what extent acidification of the pulp can be a substitute for bleaching, as acidification is much simpler than bleaching; moreover if acidification would be feasible it would not be necessary to invest in a bleachery.

In order to determine the influence of the cooking conditions and of the pH on the brightness of the cardboard eight digestions were run with Jordan wheat straw.

From these pulps handsheets were made out of fibre suspensions with a different pH in the acid range.

Finally one of the pulps was in the paper laboratory worked up into handsheets at pH = 5 (weakly acid medium). A stronger acid medium was not thought justified, for reasons of increased corrosion by the acidified pulp and higher cost of the required acid supply.

It was found that:

- cardboard formed at pH 5 was brighter by 5 - 10% G.E. than cardboard made from non-acidified pulp,
- by acidification to pH 5 a brightness could be achieved of 40 - 50% G.E..

  This is, however, a good deal lower than the approximate value of 60% G.E. for the brightness of the inside duplex cardboard, which is now used in Jordan for making cigarette boxes,
- the strength properties of neutral sulphite cardboard formed at pH 5 are equal to or even better than those of duplex cardboard of the same basis weight.

INTRODUCTION

In planning the building of a paper mill in Jordan a bleachery was originally envisaged for the manufacture of cardboard from Jordan straw (for use in cigarette boxes).

Apart from the fact that this would entail a higher capital outlay, it would also highly complicate the manufacturing process. It was for this reason that we suggested to go into the possibility of making a light-coloured pulp from Jordan wheat straw without bleaching. We knew that by acidification of neutral straw sulphite pulp a product with a light colour could be obtained. The fact is that it is much simpler to acidify the pulp than to bleach it. Moreover no extra capital investment is required.

Accordingly, the aim of the investigations was to check whether under practical conditions cardboard of a sufficiently light colour could be obtained by acidifying instead of by bleaching the neutral sulphite pulp.

Besides, a number of cardboard handsheets were to be made for the Board of the Jordan Paper Industries Ltd. as a material to show to future customers, interested in making cigarette boxes from this type of cardboard.

I. SET UP OF EXPERIMENTS

The following considerations were a guide in planning the digestions of Jordan wheat straw to a light-coloured pulp:

- In the "Proposal for a Cardboard and Kraft Factory with a Capacity of 15 tons per day Finished Product" from Bish and Partners (early 1963), related to the manufacture of cardboard from Jordan straw, it was suggested to remove the cooked mass from the digester by blowing off while depressurising the digester. This would result in a noticeable increase of the beating degree of the pulp.
  For this reason cooking conditions had to be chosen such that the beating degree of the pulp after blowing off would be much lower than 45^{o} S.R., thus making it possible to employ a sufficient amount of mechanical beating on this pulp to develop the strength properties.

For, by beating to 45° S.R. a pulp is obtained from which cardboard can be made of good strength properties at a reasonable dewatering time.

- The colour of the cardboard at a pH 5 of the pulp had to be as bright as possible. Experiments had shown that at a pH lower than 5 there was still a noticeable increase in brightness of the pulp (See Appendix). For practical reasons, such as the higher cost of a greater acid supply, pH 5 was chosen as the minimum in these investigations.
- The black liquor had to have a pH of approximately 8 after digestion to expect good strength properties of the pulp.
- The consumption of chemicals was to be at a minimum. The experience in neutral sulphite digestions of Dutch straw was the starting point in drawing up the first cooking prescription for the digestion of Jordan wheat straw. With the results of the first test, the cooking prescription was adapted to fit the next batch of straw, and so on.

The principal criteria for appraising the results were:

- degree of beating of the blown off sulphite pulp (determined with the Schopper-Riegler device for evaluating the beating degree),
- brightness of the blown off neutral sulphite pulp at pH 5 and 7 (determined in cardboard made in the laboratory sheetformer with the General Electric Brightness-Tester),
- pH of the black liquor.

At a later stage of the experiments also the black liquor composition was determined.
The pulp from the last digestion was worked up into cardboard sheets of 300 g/m^2 and from these some strength properties were evaluated.

II. THE EXPERIMENTS

A. The straw

March 1963 we received a consignment of about 100 kg of Jordan wheat straw.

This straw was equal in quality to a lot that we examined in August 1962. The straw was not chopped, but the stalks were splintered, evidently due to the threshing method applied.

This threshing had led to considerable damage of the straw and, just as was found in the earlier examined lot, part of the stalks were considerably shortened and even further reduced to powder. Moreover, the straw appeared to be contaminated by sand and stone-dust and stones of some size were even found. All through the processing only the coarse impurities could be removed from the straw.

B. The digestions

The various digestions comprised almost the same operations. These will be discussed sub. 1.

Details of the eight digestions that were made to collect sufficient data to predict optimum cooking conditions (see Chapter I), are dicussed sub. 2.

The results of the digestions are included in Table 1.

1. General

For all the cookings a 100 liter rotary digester was filled in the same way; 10 kg air-dry (= approx. 9 kg bone dry) straw together with an amount of cooking liquor of approx. 30 liter were introduced into the rotary digester. Care was taken that the coarse impurities had been removed.

During the first few digestions the contents were at first heated to 125 °C (pressure 4 kg/cm^2), which temperature was maintained for 20 minutes (impregnation of the straw). The temperature was then raised to 170 °C (pressure 8 kg/cm^2), and the digestion was at this temperature continued for 25 minutes.

To later digestions (starting with No. 6) no impregnation was applied, but the digester right away heated to cooking temperature.

When a digestion was finished, part of the pulp was blown off under pressure through a plug valve. After depressurising the digester the lid was opened and the remaining part of the pulp discharged. Out of a part of the pulp a sample of black liquor was pressed. The pH of this sample was determined, and starting with digestion No. 5, also the percentages of Na_2O and SO_2.

A sample was then taken from either pulp and these samples washed twice with tap water. Of part of this washed pulp the degree of beating was determined with the Schopper-Riegler apparatus. Another part was disintegrated in the laboratory disintegrator and subsequently beaten in the laboratory Hollander to 45-50 °S.R.

The beaten pulp was converted into laboratory cardboard sheets, the fibre suspension being acidified with sulphuric acid to the required pH before the sheets were formed (See Appendix).

After drying of the sheets their brightness was determined with the General Electric Brightness Tester. In Table 1. brightness values are included obtained from the sheets formed at a pH of about 7 and of about 5.

2. Review of the digestions.

Digestion No. 1. Chemical dosage was: 14.0% Na_2SO_3 and 5.0% Na_2CO_3 (on dry matter). A pulp was obtained of which the tipped part had a degree of beating of 27.5 °S.R., whereas the blown-off pulp had already been fiberized up to 46.5 °S.R. Such a difference in degree of beating between the two parts of the same digestion was also found in the pulps produced in the following cooking.

The brightness of the handsheets formed from a neutral suspension was 43% G.E. ("general electric"); to this first pulp no acid treatment was applied. The pH of the pulp was 8.0.

Digestion No. 2. The sulphite dosage was again 14.0%, but by increasing the sodium carbonate dosage to 5.5% it was tried to finish the cooking at a slightly higher pH, as the experience with Dutch straw had learned that as a rule the strength properties of neutral sulphite pulp were the most satisfactory when the end pH was about 8.0 to 8.5.

Although the pH of pulp No. 2 was higher indeed, viz. 8.4, there was practically no further difference. Degree of beating, of the tipped as well as of the blown off part of the pulp were about the same as found for the first pulp. The brightness after washing to neutral reaction was again 43% G.E. The pulp was acidified to pH 5 with sulphuric acid when the sheet was formed; the handsheets thus obtained had a brightness of 54% G.E.

Digestion No. 3. In this cooking the effect of a drastic reduction of the sulphite dosage, down to 8.0%, was investigated. At pH 7.6 at the end of the cooking the pulp appeared to be more uncooked, and the degree of beating of the pulp, both of the part that had been blown off and the part left behind, was lower than of pulps with a pH of 8.0 - 8.5.

This in itself was favourable as it allowed of a longer beating time to arrive at 45 to 50 °S.R., but the brightness of the samples, also after acidification to pH = 5, was poor.

Digestion No. 4. The amount of sulphite, being much too low in the previous cooking, was raised to 11.0% and 0.5% more sodium carbonate was added hence raised to 6.0%. The result was almost equal to that of digestion No. 2. As in digestion No. 4 both the amount of Na_2O and the amount of SO_2 were smaller than in digestion No. 2 (by about 10 and 20% respectively) the first one is to be preferred because of the savings on chemicals.

Digestion No. 5. This cooking was made to examine the effect of the cooking time on pulp brightness. For this purpose the same chemical dosage was chosen as used in digestion No. 2, the difference with the latter cooking being that after 12 minutes already part of the pulp was blown off (5a).

The digestion was then continued; 6 minutes later a further part of the pulp was blown off (5b). Finally, after a total of 25 minutes cooking time, still another part of the pulp was blown off and the remainder removed by tipping it out of the digester (5c).

It appeared that after 12 minutes of digesting things did not change appreciably. The mass was not digested further and there was no change in composition of the remaining black liquor, which indicated that after the first twelve minutes already the digestion was complete. However, the brightness of the hand-formed sheets at pH 5 was found to decrease considerably with increase in cooking time.

With these results in hand, it was decided to adopt another cooking scheme for further experiments.

Digestion No. 6. Chemical dosage was the same as in digestion No. 2. Cooking time was 25 minutes. No previous impregnation was applied.

The result of this cooking (pH 8.2 at end of cooking) was a pulp of 41 °S.R. beating degree (after blowing off) and reasonable brightness, viz. 48% G.E. measured on cardboard sheets formed at pH 5.

Digestion No. 7. Digestion No. 6 had shown that, so far as the degree of beating was concerned, cooking could be finished when the pulp was slightly underdone.

For digestion No. 7 a much lower amount of chemicals was used and the cooking was carried out at a considerable lower temperature, as it was supposed that also under thse new conditions the digestion would be sufficient.

The pulp was much less done indeed; the degree of beating of the tipped part was 11.5 °S.R., of the blown off part 26 °S.R.
Brightness had dropped somewhat as compared with digestion No. 6, but it was expected that a small increase in chemical dosage would be sufficiently effective in restoring the brightness.
Digestion No. 8. This cooking was made with a dose of 11.0% Na_2SO_3 and 5.5% Na_2CO_3 at 150 °C cooking temperature and 25 minutes cooking time. As it was the intention to process all the pulp into cardboard sheets losses had to be prevented and therefore the pulp was completely tipped from the digester after depressurising, instead of removing part of it first by blowing off. However, it may be assumed that also when the pulp had been removed by blowing off its degree of beating would have been so low that sufficient mechanical beating to reach approx. 45 °S.R. could have been applied to develop the strength properties. The pulp had a beating degree of 17 °S.R.

The volume of black liquor after digesting was of the same order as the supply of freh cooking liquor, and it may therefore be concluded that for the last three cookings the consumption of chemicals was about the same (see Table 1., columns 5 - 6 and 12 - 13).

III. CONVERSION OF DIGESTION NO. 8 PULP

Pulp No. 8 was worked up into cardboard sheets of 300 g/m^2 as follows. The pulp was beaten to 45 °S.R. in the laboratory Hollander for 50 minutes, and then sorted out on a screen with 0.4 mm slots. The coarse particles were sorted out, but it was impossible to remove all the impurities in this way. The samples made from this pulp therefore contained a good many of the impurities orginally present in the straw. A method for pulp purification applied on a larger than pilot-plant scale, using hydrocyclones (Vortraps, Centricleaners and the like), may be more suitable to meet this difficulty than our way of classification.

A great number of sheets were formed from the classified pulp; the sheets having a basis weight of 300 g/m^2. The brightness of these samples when formed in neutral medium was 36 °G.E., rising to 42 °G.E. when formed at pH = 5. Some of the sheets were covered with mat white paper of 60 to 70 g/m^2 and some with glossy white paper of almost equal square metre weight. This increased of course the brightness of the samples to about 80% G.E. Finally, some strength properties were determined for the three kinds of cardboard. These are summarized in Table 2.

IV. DISCUSSION OF RESULTS

As to the brightness of the samples the following remarks can be made. Although in previous digestions higher brightness values had been obtained, pulp from cooking No. 8 was used for preparing cardboard samples and for determination of the quality.

It was thus prevented that, basing ourselves on the brightness of the sheets we would have arrived at too favourable or unjustified conclusions. The brightness attained with this digestion can certainly be realized under practical conditions.

With regard to the strength properties the following may be remarked.

It is obvious to compare the strength of the neutral sulphite cardboard sheets we prepared in our paper testing laboratory with that of machine-prepared cardboard imported in Jordan.
For this purpose some strength properties of the two kinds of cardboard are compiled in Table 3. This table also includes some properties of partly bleached neutral sulphite pulp from Jordan straw obtained during the previous investigations with Jordan wheat straw (see report; "Investigations into the suitability of Jordan wheat straw for processing to board", by Th.H. Asselman (August 1962)).

In comparising these figures it should be remarked, for the sake of completeness, that in hand-formed cardboard sheets there is no or hardly any orientation of the fibres, while in cardboard made on a paper machine there is. This implies that for machine board the strength properties are determined in machine direction (M) and in cross direction (C).
This is obviously not necessary for hand-formed sheets.

From Table 3. may be concluded that the strength of cardboard sheets formed at pH 5 from neutral sulphite pulp of Jordan wheat straw is at least equal to that of machine-prepared cardboard imported in Jordan.

CONCLUSIONS

1. It is possible to achieve a noticeable increase in brightness of a neutral sulphite pulp by acidifying it (see appendix).

2. With a pH about 5 of the pulp, as conveyed to the paper machine a final product is obtained that is by 5 - 10% G.E. brighter than the product made from pulp that is not reduced in pH. (A still lower pH of the pulp is not be recommended, because of increasing corrosion and the higher cost of sulphuric acid addition).

3. Cardboard made from unbleached neutral sulphite pulp at pH 5 has a lower brightness than cardboard made from neutral sulphite pulp bleached with hypochlorite.

4. The samples prepared are of a lower brightness than may be expected to be achieved in the factory.

5. The strength of neutral sulphite cardboard at pH 5 is at least as good as that of the machine-made woodpulp cardboard that is imported in Jordan.

DIGESTIONS OF JORDAN WHEAT STRAW WITH NEUTRAL SULPHITE AND SODA

SURVEY OF THE DIGESTION SCHEMES AND THE RESULTS

DIGESTION NUMBER	AMOUNT OF CHEMICALS ON DRY STRAW			COMPOSITION OF COOKING LIQUOR		IMPREGNATION		DIGESTION		COMPOSITION OF BLACK LIQUOR			PULP DATA			
	Na_2SO_3 %	Na_2CO_3 %	dry matter liquor ratio	Na_2O g/l	SO_2 g/l	time min.	temp. °C	time min.	temp. °C	pH	Na_2O g/l	SO_2 g/l	degree of beating		brightness of "blown off pulp"	
													°S.R.-bulk	°S.R.-blown off part	% G.E.-pH 7	% G.E.-pH 5
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
1	14.0	5.0	1/3	29.6	21.4	20	120	25	170	8.0	-	-	27.5	46.5	43	-
2	14.0	5.5	1/3	30.5	21.4	20	120	25	170	8.4	-	-	25	46	43	54
3	8.0	5.5	1/3	22.1	12.5	20	120	25	170	7.6	-	-	23	35	34	39
4	11.0	6.0	1/3	27.3	17.2	20	120	25	170	8.4	-	-	25	45	45	55
5[a]	14.0	5.5	1/3	30.5	21.4	20	120	12	170	8.2	15.5	9.7	-	49.5	35	48
5[b]	-	-	-	-	-	20	120	18	170	8.3	15.3	10.0	-	51	33	44
5[c]	-	-	-	-	-	20	120	25	170	8.4	15.4	9.8	27	47.5	36	41
6	14.0	5.5	1/3	30.5	21.4	-	-	25	160	8.2	18.6	11.1	31	41	39	48
7	10.0	5.0	1/3	24.3	15.7	-	-	25	150	7.6	13.3	6.2	11.5	26	35	43
8	11.0	5.5	1/3	26.4	17.2	-	-	25	150	7.9	15.8	7.8	17	-	36 *	42 *

* Brightness of "Bulk Pulp"

PROPERTIES OF HANDSHEETS MADE FROM NEUTRAL SULPHITE JORDAN WHEAT STRAWPULP AT pH = 5.

PROPERTIES	DIMENSION	HANDSHEETS MADE FROM STRAWPULP OF DIGESTION NO. 8 BEATEN TO 45 °S.R.		
		unlined	"dull" white lined	"bright" white lined
BASIS WEIGHT	g/m^2	299	372	378
BULK	$mm/100g/m^2$	0.15	0.14	0.14
TENSILE STRENGTH	kg	58.5	63.5	70.9
STRETCH	%	2.6	3.6	4.0
FLEXING STRENGTH	kg	0.6	2.0	1.5
BURSTING STRENGTH	kg/cm^2	6.4	7.8	8.9
BRIGHTNESS	% G.E.	42	79	77

Table 3.

COMPARISON OF PROPERTIES OF SAMPLES OF DIFFERENT MATERIALS

PROPERTIES	DIMENSION	HANDSHEETS MADE FROM NEUTRAL SULPHITE JORDAN WHEAT STRAWPULP (UNLINED)		IMPORTED MACHINE CARDBOARD (LINED)
		unbleached at pH = 5 (digestion no. 8)	partly bleached	
BASIS WEIGHT	g/m^2	299	296	324
BULK	$mm/100g/m^2$	0.15	0.19	0.15
* TENSILE STRENGTH	kg	58.7	57.1	
M				54.9
C				24.8
* STRETCH	%	2.6	2.8	
M				2.0
C				3.0
* FLEXING STRENGTH	kg	0.6	0.3	
M				1.5
C				0.5
BURSTING STRENGTH	kg/cm^2	6.4	9.0	4.5
BRIGHTNESS	% G.E.	42	61	62

* Calculated at 300 g/m^2 basis weight

M = Machine direction

C = Cross direction

APPENDIX

As already discussed earlier in this report, the brightness of unbleached neutral sulphite pulp from straw increases with increasing acidity of the fibre suspension.

For ease of survey the methods applied to this acidification and the results obtained are not included in the report itself, but described in this appendix.

PROCEDURES

A. Method 1.

1. Description.

After digestion the unbleached neutral sulphite straw pulp is washed with water and defibrated during five minutes, the fibre concentration being 4 g/l.

Then the amount of fibre suspension required for formation of a board sample is acidified to the desired pH by means of 25% sulfuric acid solution.

Finally the suspension is poured into the sheet mold and the board sample formed by dewatering. After couching the sample is dried and the brightness measured.

This is done by means of a Photovolt Meter (make General Electric) with a standard light source. The ratio of the amount of reflected light from a sample and from a standard white surface of magnesium oxide is expressed in percent General Electric (% G.E.).

2. Results.

The method described in A.1. was used for six of the eight digestions. The results are compiled in figure 1. As shown the brightness of the sheets increases with decreasing pH. However the width of the shaded area is fairly large. This is partly due to the difficulties encountered when trying to reproduce the digestions. On an average the brightness increases 3% G.E. per unit of pH. The brightness of the pulp at pH = 5 varies from 40 - 50% G.E.

B. Method 2.

1. Description.

The washed unbleached neutral sulphite pulp is defibrated during 5 minutes at pH = 5, the fibre concentration being about 4 g/l.

Two liters of this fibre suspension are poured into the sheet mold, the pH is controlled and adjusted to 5, and then the sheets are formed.

2. Results.

In this way several board samples of about 250 g/m^2 were formed from the pulp of digestion number 8.

During the defibration the pH changed from 5 to about 6. This fibre suspension was brought back to pH = 5 at the sheet-former by adding a small amount of sulfuric acid.

The brightness of these board samples was 41 - 44% G.E.