

PRIVATE & CONFIDENTIAL

Bi-monthly Report - November 1959

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1. CANADIAN PCL

A. Binder Preparation.

Initially a standard VS process was applied to the Canadian stem. This yielded a binder which was dark in colour but very easily homogenised. The weight yield was 38%. Although the cooled stem was soft the W.R.V. was only 20.5.

The dark colour of the binder may have been due to two effects, either it was insufficiently extracted or the last stage of pressure cooking had brought about an oxidative degradation. The next stage was therefore continuously to extract with an AS process. A batch of stem was soaked for 24 hours in cold water, drained and then was boiled five times for  $\frac{1}{2}$  hour, using fresh water each time. It homogenised easily to give a light binder of W.R.V. 24, but again only a 33% yield. (C.286).

Table I shows that the major part of the soluble material was removed in the initial soak so that the next move was to substitute a short pressure cook for the atmospheric boils in order to complete the extraction and soften the stem.

Table I

	38% removed as solubles			
24 hr. soak in 12 gall. water,	7%	"	"	"
1st boil in 6 gall. water,	4%	"	"	"
2nd boil in 6 gall. water,	3%	"	"	"
3rd boil in 6 gall. water,	2%	"	"	"
4th boil in 6 gall. water,	1%	"	"	"
5th boil in 6 gall. water,				

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Before the pressure cooking experiment was carried out we showed that centrifuging the stem after each cook doubled the efficiency of the cook in removing solubles. This effect was due to the removal of solubles retained by the liquor within the swollen stem.

C.301 was therefore prepared by soaking the stem in cold water for 16 hours, centrifuging and pressure cooking at 30 psig for  $\frac{1}{2}$  hour, centrifuging and then homogenising with fresh water. The binder was light in colour with a W.R.V. of 25. (Yield 38%). The homogenisation went very easily.

Although this appeared to be a most satisfactory process, from the point of view of plant design the Canadians did not want any pressurised vessels. We therefore reverted to the purely AS type. A close study of the extraction process showed us that in terms of electrical conductivity and total dissolved solids there are two phases of extraction when dealing with threshed stem. In the first there is a rapid leaching of ionic material and a slighter less rapid leaching of non-ionic soluble organic material and in the second a constant slow extraction of soluble material, both ionic and non-ionic. The extraction of nicotine follows a similar pattern.

Another feature which was observed while the stem was in contact with boiling water was that at first the stem is light in colour and floats on the water but later it sinks and also becomes very much darker. These two phases did not appear, however, to be coincident with the extraction phases described above

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The conclusions reached at this stage on threshed stem were

- i) A yield of 38% only may be obtained
- ii) At least 4 atmospheric boils are required to produce a light binder, unless the stem is presoaked
- iii) The cooks do not need to exceed 40 minutes each
- iv) Centrifuging the cooked stem aids the complete extraction of soluble material

Our Canadian friends had had success with flaked stem and at this point a differential roller was made available to us with which we were able to roll the stem out into small flakes. This enabled very rapid and complete extraction to take place. Studies on the conductivity, total dissolved solids and nicotine content of the liquor showed that in 2 minutes we had extracted as much as would have been extracted in 2 hours from ordinary threshed stem.

The process was now reduced to a single cook eg. 7.7 lbs. of flaked stem were boiled in 6 gallons water for 1 hour. The liquor was centrifuged off and found to contain 3.8 lb. dissolved solids (49%). The residue was washed in 6 gallons of water for 10 minutes at 60°C and the liquor centrifuged off. This contained 0.4 lb. (5%). The residue was homogenise to give a light coloured binder (C.310) of W.R.V. 19 and yield 40%.

A programme is now in hand for studying the flaking process.

#### B. FCL Preparation.

The samples of FCL which our Canadian friends brought over with them were very much superior to our sheet in some respects. Their sheet was thinner and smoother and had a much higher flexibility. The reverse side

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ie. that in contact with the band was remarkably smooth and did not appear to have been torn from the band by the doctor blade which is what happens to our material. There are a number of possible reasons for this which are being investigated.

- i) Homogenisation. We homogenise all our binder at 2000 p.s.i. whereas at Louisville 5000 p.s.i. is now standard. We have had a Manton-Gaulin homogeniser available for some months which would work at 5000 p.s.i. but we have not been permitted to use it until recently. This extra homogenisation might well give a less fibrous binder which would offer no resistance to the doctor blade.
- ii) We have carried out some experiments on the Canadian AS/FCL and these are detailed below. By visual inspection of the Canadian flour ground at Joliett they appear to obtain more fine material than we do. We have no means, as yet, of actually measuring the particle size distribution.
- iii) Doctoring. Mr. Willis is investigating different materials and methods of doctoring.
- iv) Lesser effects might be produced by variations in time, temperature and mixing of binder with flour.

The following experiments are related to the points discussed above.

C294/S359 Variation in flour loading

Loading	Casting consistency	I.S. (g.mm <sup>-2</sup> )	f <sub>100</sub>	Colour
3:1 Ajax I	7.5%	110	107	1
4:1 "	8.8%	109	80	3
5:1 "	9.2%	133	34	2
6:1 "	9.6%	78	19	4
7:1 "	9.8%	82	11	5

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The casting consistency was kept constant since above 10-11% we find that we get an uneven and pinholed sheet. It will be seen that the tensile strength does not alter very much but that the flexural strength shows a marked decrease with increased loading. The colour has been judged visually, increasing from 1 to 5. The loading of 5:1 was considered to be the maximum for manufacturability.

C295/S360 Effect of particle size

Flour	Loading	T.S. (g.mm <sup>-2</sup> )	f <sub>100</sub>	
Unsieved Ajax I	4:1	143	36	} light in colour
"	5:1	116	33	
-100 mesh Ajax I	4:1	135	55	} dark in colour
"	5:1	101	32	

By leaving in the material > 100 mesh a lighter sheet was obtained without greatly altering the physical strength.

C310/S.375 Effect of particle size

Flour	T.S. (g.mm <sup>-2</sup> )	f <sub>100</sub>	Colour
5:1 Ajax II	98	19	} All very similar
5:1 Joliette ground Ajax II	108	21	
5:1 Ajax II + 10% micronised Ajax II	122	27	

This shows a steady increase in the tensile and flexural strengths as the percentage of finely ground flour is increased.

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C305/5370 Detailed comparison Joliett ground Ajax II and  
R. & D. ground Ajax II

(all loaded 3:1, 3% glycerol and mixed 15 minutes)

Binder	Flour	Mixing Temp.	T.S. (g.mm <sup>-2</sup> )	f <sub>100</sub>	Colour <sup>M</sup>
A. 2 passes at 2,000 psi WRV 18	R & D	41°C	78	37	1
	J	40°C	106	37	1
	R & D	82°C	106	9	1
	J	82°C	137	40	1
B. 6 passes at 2,000 psi WRV 21	R & D	41°C	101	88	1
	J	44°C	106	79	2
	R & D	82°C	111	50	2
	J	82°C	150	64	3

<sup>M</sup> increasing dark colour from 1 - 3.

From these results we can pick out the two trends which previous results had suggested. Firstly the Joliett ground flour gives a higher product tensile strength and secondly also gives a darker sheet. Both these effects are consistent with a smaller particle size flour which permits more complete leaching.

C. Decolourising experiments. (C.231/5356).

Since the yield from the Canadian stem is consistently about 38% it was felt worthwhile to try and decolourise the liquors and then return them to the binder. This was carried out quite successfully with activated charcoal to give a total yield of nearly 90%. Unfortunately the soluble material does not have the flexibility of the ordinary binder.

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eg.	Loading	T.S. ( $\text{g}\cdot\text{mm}^{-2}$ )	$\text{f}_{100}$
	2:1	224	121
	3:1	198	27
	4:1	137	24
	5:1	145	15

In spite of the high tensile strength the maximum loading for manufacturability would be about 2.5:1 which means that we have gained very little for the cost of decolourising. This line has been dropped for the time being.

## 2. SOUTH AFRICAN PCL

Burley stem from South Africa has been flaked and used for a standard VS process. The cooked stem was extremely soft but remained a dark colour. It was made into PCL with recipe A flour at two loadings.

Loading	T.S.	$\text{f}_{100}$
3:1	78	18
5:1	75	5

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