Production of paper grade pulp from bagasse by a novel pulping process

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SUMMARY

A novel pulping process using formic acid/acetic acid/water to selectively separate cellulose, hemicelluloses and lignin from bagasse was optimised and the bleaching response and pulp properties were determined. The process is suitable for use with high silicon containing raw materials because the silicon predominantly stays with the pulp and does not complicate chemical recovery. Key pulping variables were the percentage of formic acid and water in the pulping liquor, the liquor to fibre ratio and pulping time. This cooking method produced unbleached paper pulp at atmospheric pressure. High quality bleached pulp was produced using a TCF sequence. This process has the potential to be very environmentally effective. Pilot scale evaluation is planned.

Keywords
Formic acid, acetic acid, water, pulping, bleaching, TCF sequence, bagasse

Sugar cane residue is a raw material available in large quantities from sugar extraction plants, and offers an interesting opportunity to create agro-industrial complexes based on adding value to each major constituent of the vegetable matter, i.e. sugar, cellulose fibres and lignin.

The papermaking evaluation procedure of sugar cane bagasse that we propose in this article falls within this framework. It furthers the work of Delmas et al. (*1-8*) relating to the breakdown of plant matter in a formic acid/acetic acid/water environment. Moreover, this method of refining plant matter, which does not use any inorganic compound, retains the silicon in the unbleached pulp that is initially present in the raw material. The level of pollution generated by this method is potentially lower than with other existing methods.

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EXPERIMENTAL

Vegetable matter

The raw material used in this study was bagasse (*S. officinarum*) obtained from the sugar extracting plant in Viet-tri, a town situated 50 kilometres north of Hanoi, Vietnam. The pith, which is mainly composed of parenchyma cells, contained very few cellulosic fibres (15%) and it was removed before pulping the bagasse. The moisture content of the depithed bagasse was 10%, and its chemical composition as follows: 41.5% cellulose, 19.6% lignin, 28.4% pentosan, 2.9% ash and 1.3% silicon (based on dry matter).

Cooking

Cooking in an organic acid medium was done in a one-litre glass reaction vessel at atmospheric pressure. The bagasse (30 g o.d.), with the pith removed and cut approximately into 3 cm lengths, was first impregnated with the cooking liquor at 50°C for 30 minutes and thoroughly mixed by mechanical shaking. The rise to temperature was set at 30 minutes. After impregnation, cooking was done under various conditions as discussed later.

The pulp thus obtained was first filtered, pressed and washed twice with fresh organic acid solution, then with hot water. The fibres were then separated in a pulper (defibrator), carefully washed with cold water, and screened on 65 and 100 mesh sieves using pressurised water. The undercooked pulp (rejects) was collected on the 65-mesh sieve, while the 100-mesh sieve collected the accept fibre, and both were dried and analysed.

Pulp was refined in a Valley beater; handsheets were made using a Frank apparatus with two dryers (AFNOR standards NF Q50-002) and tested for mechanical properties.

The chemical and mechanical characteristics of the pulp were determined in accordance with the following standards:

Kappa number	AFNOR NF T 12-018,
Mechanical properties	AFNOR NF Q 03-004, AFNOR NF Q 03-053, AFNOR NF Q 03-001,
Whitenes index	AFNOR NF T 12-030
Ash/silicon content	ChineseStandard G.B 2677.3-81.

To determine the viscosity (expressed in cm^3/g) the unbleached pulp was first transformed into holocellulose using a solution of sodium chlorite in a buffered medium (acetic acid and soda; pH 4.9). The viscosity of the holocellulose was measured in accordance with AFNOR NF T 12-005.

RESULTS AND DISCUSSION

Pulp delignification and characterisation

A series of formic acid/acetic acid pulping trials were carried out to study the influence of the following factors on the delignification rate and chemical characteristics of the pulp:

- The percentage of formic acid in the cooking solution.
- Cooking time.
- The liquor/dry matter ratio (L/M).
- The formic acid/acetic acid/water ratio. This ratio is determined taking into account the amount of water initially present in the plant matter.

Effect of formic acid content and cooking time on pulp properties

Previous studies showed that increasing the percentage of formic acid in the cooking liquor and increasing cooking time greatly improved the delignification rate when implementing this process with different plant matter such as rice straw, sugar sorghum and triticale straw (*5-8*). Controlling these factors is therefore very important when cooking takes place in a formic acid/acetic acid/water (fa/aa/water) environment. Based on these earlier studies the following levels were chosen for study: formic acid in the cooking liquor, 20, 30 and 40% v/v; cooking time, 2, 3, 4 and 5 h; water in the cooking liquor, 20% v/v; temperature, 107°C; and the liquor/dry matter ratio, 10/1. The results are shown in Table 1.

Table 1 shows that increasing the cooking time from 2 to 5 h, for 20, 30 or 40 % formic acid in the cooking liquor, considerably

Table 1
Sample characteristics for the old (80 t/d) deinking line.

Sample no*.	ERIC (ppm)	Flakes (mm^2/kg pulp)	Brightness (ISO%)	Tensile index (N.m/g)
1Aa	627	65735	50.74	20.93
1Ba	384	39623	53.88	25.09
1Ca	327	17191	56.71	25.22
1Da	267	7822	57.38	29.24
1Ab	620	20846	52.75	27.33
1Bb	334	10684	56.52	30.27
1Cb	296	5427	58.59	30.16
1Db	264	2028	59.16	31.91
1Ac	151	10946	58.52	25.50
1Bc	148	9036	58.33	25.38
1Cc	136	4292	60.44	26.44
1Dc	84	720	61.90	31.27

* '1' represents the 80 t/d deinking line; 'A', 'B', 'C' and 'D' represent sampling points defined in the text; 'a' represents pulps that had ink 'fixed' with alum, 'b' represents untreated pulps (no alum fixing or washing), and 'c' represents pulps after hyperwashing.

Table 2
Sample characteristics for the new (250 t/d) deinking line.

Sample no*.	ERIC (ppm)	Flakes (mm^2/kg pulp)	Brightness (ISO%)	Tensile index (N.m/g)
2Aa	668	61058	54.76	21.48
2Ba	336	38000	58.86	23.47
2Ca	281	11101	59.37	26.63
2Da	213	5510	59.99	28.27
2Ab	688	31595	53.84	28.41
2Bb	336	14822	58.21	28.66
2Cb	209	4007	58.89	29.52
2Db	197	1312	59.47	30.39
2Ac	206	9742	53.46	21.22
2Bc	174	6574	58.14	22.46
2Cc	172	1893	58.90	23.58
2Dc	148	987	60.69	26.71

* '2' represents the 250 t/d deinking line. The other symbols are as defined in Table 1.

F_2^{th}(B-C)T.R. means total theoretical cleaning efficiency of the 2nd stage on total removable flakes (%) in the 2nd line.

F_2^{ac}(B-C)S.E. means actual cleaning rate of the 2nd stage on stage entrance flakes (%) in the 2nd line.

F_2^{ac}(B-C)T.E. means total actual cleaning rate of the 2nd stage on total entrance flakes (%) in the 2nd line.

Calculated results of cleaning efficiency and cleaning rate for the two deinking lines are given in Tables 5 and 6.

Tables 5 and 6 show that the theoretical cleaning efficiency and actual cleaning rate of the 2nd stage are higher than for the other stages, and are higher for the 2nd stage in the 2nd line than in the 1st line. This is because a smaller slot width is used in the fine screen in the 2nd line (0.15 mm) than in the 1st line (0.30 mm). Tables 5 and 6 also show that the actual and total actual cleaning rates more clearly show performance differences than the theoretical and total theoretical cleaning efficiencies. This suggests that the actual cleaning rate and total actual cleaning rate are more useful tools to evaluate the cleaning efficiency. Also flakes determination is much more convenient in a mill environment on virgin pulp than on alum-fixed pulp and hyperwashed pulp.

Tables 3 to 6 show that the actual deinking rate and total actual deinking rate, and the actual cleaning rate and total actual cleaning rate are higher in the 1st stage than with other stages. This confirms the overriding importance of the first stage in a deinking line.

Table 3
Theoretical deinking efficiency of the two deinking lines.

Stage	Theoretical deinking efficiency based on the stage deinkable ink (%)	Stage	Total theoretical deinking efficiency based on the total deinkable ink (%)
E_1^{th}(A-B)S.D.	50.7	E_1^{th}(A-B)T.D.	44.8
E_1^{th}(B-C)S.D.	23.0	E_1^{th}(B-C)T.D.	10.5
E_1^{th}(C-D)S.D.	24.7	E_1^{th}(C-D)T.D.	11.0
E_1^{th}(A-D)S.D.	66.3	E_1^{th}(A-D)T.D.	66.3
E_2^{th}(A-B)S.D.	67.2	E_2^{th}(A-B)T.D.	63.8
E_2^{th}(B-C)S.D.	33.5	E_2^{th}(B-C)T.D.	10.6
E_2^{th}(C-D)S.D.	51.1	E_2^{th}(C-D)T.D.	13.1
E_2^{th}(A-D)S.D.	87.5	E_2^{th}(A-D)T.D.	87.5

Note: S.D. represents – 'Stage Deinkable Ink'
T.D. represents – 'Total Deinkable Ink'

Table 4
Actual deinking rate of the two deinking lines.

Stage	Actual deinking rate based on ink entering the stage (%)	Stage	Total actual deinking rate based on the total ink entering (%)
E_1^{ac}(A-B)S.E.	46.1	E1ac(A-B)T.E.	46.1
E_1^{ac}(B-C)S.E	11.4	E1ac(B-C)T.E.	6.1
E_1^{ac}(C-D)S.E.	10.8	E1ac(C-D)T.E.	5.2
E_1^{ac}(A-D)S.E.	57.4	E1ac(A-D)T.E.	57.4
E_2^{ac}(A-B)S.E.	51.2	E2ac(A-B)T.E.	51.2
E_2^{ac}(B-C)S.E.	37.8	E2ac(B-C)T.E.	18.5
E_2^{ac}(C-D)S.E.	5.7	E2ac(C-D)T.E.	1.7
E_2^{ac}(A-D)S.E.	71.4	E2ac(A-D)T.E.	71.4

Note: S.E. – 'Stage Entering Ink'
T.E. – 'Total Entering Ink'

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Table 1
The effect of formic acid percentage in the cooking solution and cooking time on unbleached pulp properties.

af/aa/water (% v/v)	Time (h)	Yield (%)	Rejects (%)	Kappa number	Viscosity (cm3/g)	DPv
20/60/20	2	28.5	19.7	68.0	900	1338
	3	37.0	17.9	57.7	954	1427
	4	48.3	3.2	52.8	1012	1523
	5	50.3	–	48.1	1022	1540
30/50/20	2	45.1	6.3	47.7	972	1457
	3	50.0	–	37.6	1055	1595
	4	45.7	–	34.1	1026	1546
	5	46.9	–	30.4	990	1486
40/40/20	2	46.8	–	37.0	1022	1540
	3	45.7	–	32.0	1007	1515
	4	41.3	–	28.5	892	1325
	5	38.6	–	25.0	851	1258

improved delignification with a progressive decrease in rejects and Kappa number. Pulping was not sufficient when the percentage of formic acid in the cooking liquor was limited to 20%: a relatively long cooking time was required (5 hours) to reduce the rejects to zero. However, the level of residual lignin (Kappa 48.1) in the pulp remained high. An increase in the percentage of formic acid in cooking liquor (up to 30 or 40%) is therefore necessary for satisfactory delignification of bagasse.

Delignification of bagasse in a fa/aa/water environment can therefore be greatly improved by increasing the cooking time, the percentage of formic acid in the cooking liquor or by increasing both these factors.

The yield obtained when the cooking time increases from 2 to 5 hours trends in different ways according to the percentage of formic acid in the liquor. For cooking trials with a 20/60/20% fa/aa/water ratio, pulp yield increases progressively from 28.5% after 2 hours cooking to reach 50.3% with 5 hours cooking. This increase is essentially due to the decrease in the level of rejects. When the fa/aa/water ratio is 30/50/20, the yield reaches a maximum when cooked for 3 hours, whereas when cooking with a fa/aa/water ratio of 40/40/20%, the yield systematically decreases as the cooking time increases.

The pulp viscosity trends with cooking time and the percentage of formic acid in a similar way to the yield (Fig. 1).

The progressive increase of pulp viscosity with cooking time, observed when the fa/aa/water ratio is 20/60/20% v/v, can be attributed to loss of pentosans (2) with minimum degradation of cellulose. With a fa/aa/water ratio of 40/40/20, viscosity systematically decreases with cooking time because formic acid decomposes cellulose and this decomposition becomes greater as the cooking time increases. As a result, the pulp yield also decreases (Table 1). Cooking with a fa/aa/water ratio of 30/50/20 for 3 hours produces pulp with the highest viscosity; beyond this cooking time viscosity decreases due to cellulose decomposition by formic acid.

The percentage of formic acid and cooking time are main factors that should be controlled when cooking bagasse in a formic acid/acetic acid/water environment, and pulp yield and viscosity depend predominantly on these factors. The findings of this study show that reasonable values for these factors for the delignification of sugar cane bagasse in a formic acid/acetic acid/water environment are cooking time – 3 h and percentage of formic acid – 30% (fa/aa /water ratio 30/50/20).

These values were used in subsequent trials to study the influence of other factors such as the water content and liquor/dry matter ratio on pulp properties.

Effect of water content of the cooking liquor on pulp quality

The main aim of these trials was to study the role of water on delignification when cooking in a fa/aa/water environment, and on the chemical characteristics of the pulp. The experiment results are shown in Table 2.

It is seen from Table 2 that the delignification of bagasse with a mix of fa/aa/water is at its maximum with 15% water in cooking liquor. Beyond this value, a decrease in delignification was noted as shown by an increase in Kappa number. When cooking with water contents of 2 and 30% rejects are very high, 28.4 and 12.3% respectively. Cooking with a fa/aa/water ratio of 30/55/15 produces a good yield pulp with the highest viscosity. A certain precise amount of water is therefore very important for optimal pulping of bagasse. This is because the breakdown of plant matter in an organic acid environment is favoured by the presence of solvate protons formed when water is added as follows:

$$RCOOH + H_2O \rightleftharpoons RCOO^- + H_3O^+ \quad [1]$$

In a concentrated organic acid solution, molecules are closely linked to each other by hydrogen bonds and protons are therefore not readily available. The addition of water is firstly used to break the links between the molecules of organic acid and then to favour the ionisation and dissociation of these acids, which then supply the protons. When the quantity of water molecules in the environment is very low (fa/aa/water of 30/68/2), the acid molecules, formic and acetic, are closely linked and liberate very few protons resulting in a low ability to destroy organic matter.

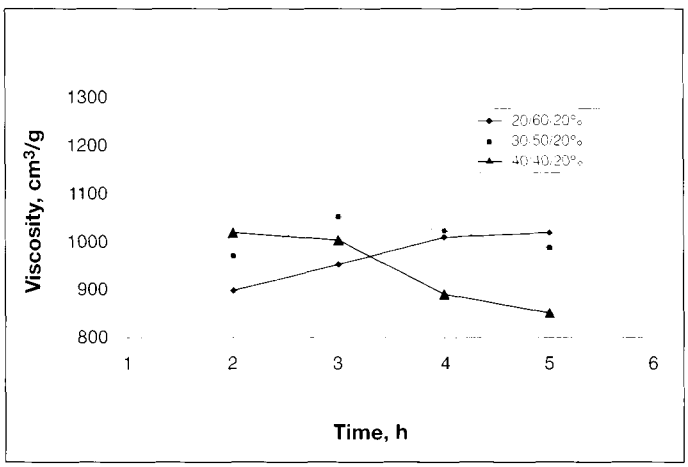

Fig. 1 The effect of cooking time and formic acid percentage on pulp viscosity.

Table 2
Effect of cooking liquor water content on pulp properties.

af/aa/ water (% v/v)	Yield (%)	Rejects (%)	Kappa number	Viscosity (cm^3/g)	DPv
30/68/2	31.8	28.4	71.1	966	1447
30/60/10	51.0	1.2	36.5	1067	1615
30/55/15	51.2	–	33.8	1072	1623
30/50/20	50.0	–	37.6	1055	1595
30/40/30	41.1	12.3	57.2	1021	1538

Cooking conditions: temperature, 107°C; L/M ratio, 10/1.

Table 3
Effect of the L/M ratio on pulp properties.

L/M ratio	Yield (%)	Rejects (%)	Kappa number	Viscosity (cm^3/g)	DPv
5/1	40.4	14.5	52.0	1024	1543
7/1	46.3	3.1	40.1	1067	1615
10/1	51.2	–	33.8	1072	1623
15/1	49.4	–	28.2	1075	1628
20/1	48.2	–	27.8	992	1490

Cooking conditions: fa/aa/water ratio, 30/65/15% v/v; temperature, 107°C; time, 3 h.

The poor results observed when sugar cane bagasse was cooked in a fa/aa/water environment of 30/40/30 can be attributed to the fact that water is not a very good lignin solvent. During their research into the solubilisation of lignin in different solvents Schuerch (*9*), and Young and Davis (*10*), specified that water, with a Hildebrand factor of 23, is a poor solvent. The increase in the quantity of water in cooking liquors therefore results in a decrease in the solubilisation of lignin fragments. Plant matter delignification is the result of various actions in the pulping environment: breakdown of the intramolecular lignin links, breakdown of the links between lignin and hemicelluloses, and solubilisation of the fragments obtained. The presence of a large quantity of water in cooking solutions results in a considerable drop in the solubilisation of lignin fragments and it is therefore not recommended.

These results show that 30/55/15% is the 'best' fa/aa/water ratio in the delignification of bagasse when the percentage of formic acid in the cooking solution is 30%. This value was consequently retained to study the effect of the liquor/dry matter ratio on pulp properties.

Effect of liquor/dry matter ratio (L/M) on pulp quality

Various liquor/dry matter ratios (from 5/1 to 20/1) were chosen to determine the impact of this factor on delignification and pulp quality (Table 3).

It is seen from Table 2 that the effect of L/M ratio on pulp yield, rejects level, and Kappa number, is highly significant. Increasing the L/M ratio from 5/1 to 20/1 results in a considerable reduction in Kappa (24.2 units); however, the main changes occur when the L/M ratio increases from 5/1 to 15/1. An L/M ratio above 10/1 is required to eliminate rejects. A relatively high L/M ratio value (15/1) can be used on a laboratory scale to produce pulp from bagasse with a high yield and viscosity value as well as a fairly low Kappa number.

The favourable effect of L/M ratio on the delignification of bagasse in a formic acid/acetic acid/water environment is because a high L/M ratio is necessary to sufficiently impregnate the bagasse, to transfer hydrolysed bagasse products in a fa/aa/water environment into solution and for solubilisation of lignin and hemicelluloses fragments.

The experiment results show that the 'best' operating conditions for batch pulping of bagasse in a formic acid/acetic acid/water environment are as follows:

Cooking times: 3 h.

L/M ratio: 15/1.

Fa/aa/water ratio: 30/55/15.

Cooking temperature: 107°C

The properties of pulp obtained by cooking bagasse under these optimal conditions are shown in Table 4.

The results in Table 4 show that the mechanical properties of pulp obtained from bagasse under optimal pulping conditions are suitable for the manufacture of various kinds of paper and board. Moreover, when cooking in a formic acid/acetic acid/water environment a large proportion of the silicon by-products initially present in the raw organic matter is retained in the pulp. The remaining silicon by-products in the pulp constitute excellent quality filler. This retention of silicon by-products also makes the recovery of chemicals used during the cooking process easier.

Cooking in a formic acid/acetic acid/water environment can thus be seen to be suitably adapted to the production of paper grade pulp from bagasse. Furthermore, the lignin and soluble sugar in the cooking liquor can be easily isolated (*3,6,8*), and these different components of plant matter can be used for the synthesis of various chemical compounds such as lignin/formaldehyde resin, xylitol, furfural etc. Pilot scale evaluation of this pulping process is planned.

Bleaching of optimal unbleached bagasse pulp

A preliminary delignification sequence was applied to the optimal bagasse pulp using organic peracids (Pa) and ozone (Z) followed by a bleaching stage using hydrogen peroxide (P) in a basic TCF (PaZEP) sequence. The performance of this

Table 4
Properties of pulp obtained by cooking bagasse under optimal conditions.

Property	Value
Screened yield, (%)	49.4
Kappa number	28.2
Viscosity, (cm^3/g)	1075
Silica content, (g)	1.23
Silica retention, (%)	95.2
Beating degree,(°SR)	45
Grammage, (g/m^2)	71.0
Breaking length, (m)	5350
Tear index, ($mN.m^2/g$)	4.23
Burst index, ($kPa.m^2/g$)	3.21

Table 5
Chemical and optical characteristics of bleached bagasse pulp.

Characteristics	Pulp bleached using the PaZEP sequence*	Pulp bleached using the OD1EPD2EP sequence**
Total yield, (%)	42.1	41.6
Silicon by-products content, (%)	2.4	0.4
Silica derivates retention, (%)	77.7	12.8
Viscosity, (cm^3/g)	755	728
Whiteness index, (ISO)	85.0	87.0
Beating degree, (°SR)	45	45
Breaking length, (m)	4637	4250
Tear index, ($mN.m^2/g$)	4.9	5.0
Burst index, ($kPa.m^2/g$)	2.4	3.0

*** PaZEP sequence**
Pa: hydrogen peroxide, 5% on o.d. pulp; stock concentration, 10%; formic acid/acetic acid ratio, 30:70 % v/v; temperature, 70°C; sodium pyrophosphate, 0.5% on o.d. pulp. Peracids were prepared at ambient temperature for 24 hours.
Z: ozone, 1% on o.d. pulp; ambient temperature; stock concentration, 35%.
EP: soda charge, 4% on o.d. pulp; hydrogen peroxide, 2% on o.d. pulp; stock concentration, 10%; temperature, 70°C; retention time, 120 min.

**** OD_1EPD_2EP sequence**
O: Oxygen pressure, 5.10^5 Pa; temperature, 100°C; retention time, 60min; stock concentration, 10%; magnesium sulfate, 0.5% on o.d. pulp; soda charge, 5% on o.d. pulp.
D_1: chlorine dioxide, 2.8% avail. chlorine on o.d. pulp; final pH, 3.0; stock concentration, 10%; temperature, 70°C; retention time, 80 min.
EP: hydrogen dioxide, 0.5%; soda charge, 2% on o.d. pulp; stock concentration, 10%; temperature, 70°C; retention time, 120min.
D_2: chlorine dioxide, 1.8% avail. chlorine on o.d. pulp; final pH, 5.0; stock concentration, 10%; temperature, 70°C; retention time, 180min.

bleaching sequence was compared to results obtained using a so-called 'traditional' ECF sequence (OD_1EPD_2EP) of oxygen delignification (O) followed by various stages of bleaching with chlorine dioxide (D) and peroxide. The conditions applied and the results obtained are shown in Table 5.

The results in Table 5 show that bleaching bagasse pulp using the TCF sequence, PaZEP, is almost as efficient in terms of whiteness as bleaching with the regular ECF sequence OD_1EPD_2EP. The TCF bleached pulp mechanical properties are comparable to those obtained with the ECF sequence. The TCF sequence simplifies the bleaching process and facilitates reduced water usage in comparison to the ECF sequence.

CONCLUSIONS

The unbleached pulp obtained by cooking bagasse in a formic acid/acetic acid/water environment has many properties suited to its use in paper production. By studying the influence of the percentage of formic acid, cooking time, the water content of the cooking liquor, and the liquor to solid matter ratio, it was possible to define an effective delignification regime.

Unbleached bagasse pulp obtained by cooking in this optimal organic acid environment could be TCF bleached to a suitably high brightness (85% ISO) while retaining mechanical strength properties that could be acceptable in the manufacture of printing and writing grades of paper.

The development of this technology on a pilot scale is pending.'

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