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A PRELIMINARY ASSESSMENT OF AQUEOUS ETHANOL PULPING OF BAGASSE: THE ECOPULP PROCESS

By

B.P. LAVARACK[1], T.J. RAINEY[1], K.L. FALZON[1] AND G.E. BULLOCK[2]

[1]*Sugar Research Institute, Mackay, Queensland,* [2]*Retired from Sugar Research Institute, Mackay, Queensland.*

b.lavarack@sri.org.au

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Abstract

SRI has examined the organosolv (organic solvation) pulping of Australian bagasse using technology supplied by Ecopulp. In the process, bagasse is reacted with aqueous ethanol in a digester at elevated temperatures (between 150ºC and 200ºC). The products from the digester are separated using proprietary technology before further processing into a range of saleable products.

Test trials were undertaken using two batch digesters; the first capable of pulping about 25 g of wet depithed bagasse and the second, larger samples of about 1.5 kg of wet depithed bagasse. From this study, the unbleached pulp produced from fresh bagasse did not have very good strength properties for the production of corrugated medium for cartons and bleached pulp. In particular, the lignin contents as indicated by the Kappa number for the unbleached pulps are high for making bleached pulp. However, in spite of the high lignin content, it is possible to bleach the pulp to acceptable levels of brightness up to 86.6% ISO.

The economics were assessed for three tier pricing (namely low, medium and high price). The economic return for a plant that produces 100 air dry t/d of brownstock pulp is satisfactory for both high and medium pricing levels of pricing.

The outcomes from the project justify that work should continue through to either pilot plant or upgraded laboratory facility.

Introduction

Many of the major competitors of the Australian sugar industry have pursued value-adding opportunities in order to lessen the impact of low prices for sugar. These policies have led to more sustainable cane sugar industries in these countries. The independent assessment of the sugar industry undertaken by Hildebrand (2002) recommended that the industry in Australia should diversify into value adding activities. Although Hildebrand specifically mentioned cogeneration and ethanol production, other value adding activities such as the production of pulp and paper from bagasse should be included.

The conventional process for the production of bleachable chemical pulp is the kraft process which is highly capital intensive and is unlikely to be economic unless a very large production facility is established (Rainey and Clark, 2004). Other technologies for the production of pulp such as organic solvation (or organosolv) may provide an alternative process route since these processes can both reduce the capital requirements and increase the revenue stream from a mix of value added products (e.g., pulp, lignin, acetic acid and furfural) thus substantially reducing the size of the plant required for economic viability.

This paper provides an initial assessment of a proprietary organosolv process for the production of pulp and other value-added products from bagasse. The organosolv technology assessed is known as the Ecopulp process and is partially based on the Alcell process (Pye and Lora, 1991) tested on wood chips in a 30 t/d demonstration plant at Miramichi in Canada. The Ecopulp process has been modified to make it more appropriate for pulping agricultural residues rather than wood chips.

Overview of the process

A simplified schematic of the Ecopulp process is shown in Figure 1. In the process, bagasse is taken from storage and depithed to separate the pith from the whole fibre. The depithed bagasse (true fibre) is fed into a digester[1], impregnated with steam and aqueous ethanol and reacted in the digester at the required conditions (e.g. between 150ºC and 200ºC). The pulp is discharged from the digester through a let-down valve into a blow tank. The pulp is screened and washed to separate the unbleached pulp (i.e. brownstock) from the spent organosolv liquor (i.e. black liquor).

The brownstock can feed a bleaching plant or may be dried and baled, and sold as unbleached pulp, primarily for packaging applications. The chemicals in the black liquor are reclaimed in a recovery plant. The organosolvent (i.e. aqueous ethanol) is recovered by (i) flashing the hot black liquor and (ii) stripping ethanol in a distillation column. Lignin, furfural, acetic acid and sugars (i.e. pentose and hexose) are also recovered.

The conditions for the trials and the preliminary test work were prescribed by the Ecopulp consultants and followed in practical test work.

[1] Ideally the bagasse digester should be continuous as both the solvent requirements and the volumes of product will reduce (viz. the chemicals will be more concentrated and recovery costs will reduce), but it is possible to use a series of smaller batch reactors and recycle solvent (liquor) to the reactors in an appropriate manner.

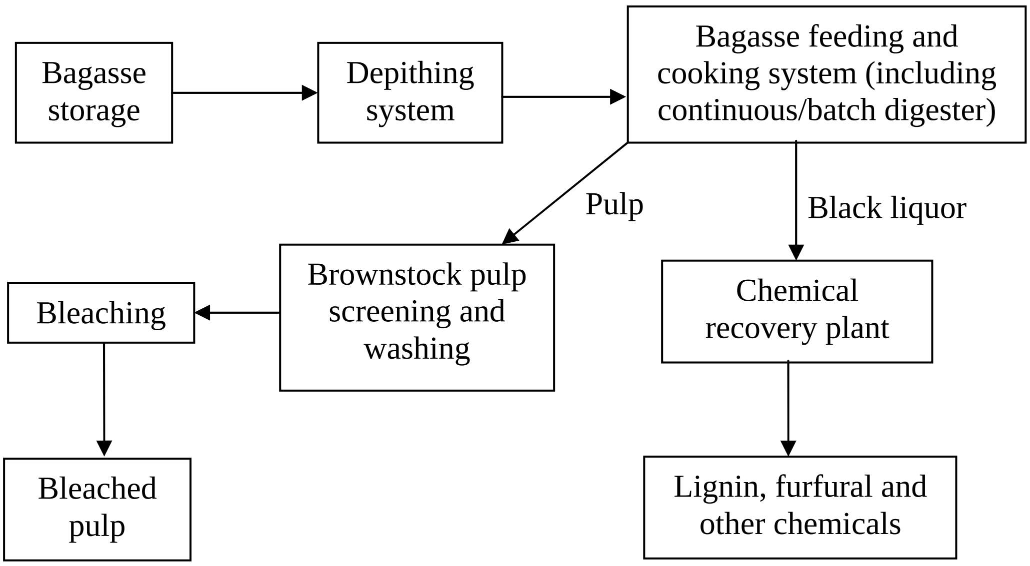

Fig. 1 Schematic of the Ecopulp process

Advantages of the process

The main features claimed for the Ecopulp process are:

1. recycle of organosolvent (aqueous ethanol) with reduced potential for contamination of the environment (no sulfur odours as in the kraft process and reduced water consumption);

2. reduced capital cost as no large chemical recovery boilers are required to recover chemicals (e.g. as is the requirement for the kraft, soda and the sulfite processes); and

3. recovery of co-products (e.g. lignin, xylose and furfural) for increased revenue.

4. integration of pulping with the fermentation of sugars from the process and from the sugar mill to generate high protein animal feed, fertilizer and ethanol for use in the process with excess for sale.

Both the reduced capital costs and the increased income from co-products are claimed to increase the economic viability of relatively small pulp mills, making it possible to locate relatively small pulp mills of about 100 t/d pulp close to the source of the fibre feedstock. The potential for the co-location of pulp mills at sugar factories based on this technology is claimed to be enhanced for these reasons.

Laboratory scale solvation trials (ranging tests)

Trials were undertaken on depithed bagasse with a small batch digester (500 mL Parr reactor) to determine the optimum conditions for the production of pulp of suitable quality for papermaking. The main operating parameters investigated were: (i) the cooking temperature; (ii) the cooking time; (iii) ethanol concentration; and (iv) the liquor to fibre ratio[2].

It was necessary to depith the bagasse prior to processing in order to obtain a pulp with suitable drainage characteristics. The pith was removed from the bagasse by dry screening through a large aperture sieve. Increased liquor to fibre ratios were found necessary for uniform pulping conditions in the Parr batch reactor. In all the tests, a liquor to fibre ratio (10:1) was applied to achieve adequate wetting of the bagasse in the batch digester. The liquor to fibre ratio is higher than used industrially for pulping woodchips by other processes. In a continuous digester the liquor to fibre ratio is a function of the relative feed rates of fibre and liquor and is not a function of the internal consistency (mass of fibre to mass of liquor) in the digester.

The quality of the pulp was assessed in terms of (i) the extent of delignification as measured by Kappa number and (ii) the degree of cellulose polymerisation as measured by pulp viscosity. The Kappa number is an indicator of the residual lignin content of the pulp and is an important quality parameter if the pulp is to be bleached. In general, the higher the Kappa number, the higher the lignin content and the higher the requirements for both bleaching chemicals and effluent treatment. Consequently, low Kappa numbers should be targeted if the pulp is to be bleached. Also, for the Ecopulp process, a higher Kappa number means less lignin will be recovered for sale. The Kappa number is determined in a standard TAPPI method (TAPPI, 1985) and is commonly used in the pulp industry.

There are several well-known tests to determine the specific physical properties of pulp (e.g. burst, tear and breaking length). Pulp viscosity was used to estimate the degree of polymerisation of the pulp from the laboratory trials. Pulp viscosity provides an indicator for the tensile strength of the pulp. However, the measurement of the pulp viscosity for industrial pulps is not widely used in Australia and the measurement of tensile strength (i.e. breaking length) is required for validation. The method for determining pulp viscosity is given in a standard TAPPI method (TAPPI, 1989). A low pulp viscosity indicates that the pulp may have poor tensile strength. Good tensile strength is generally important for printing and writing as well as tissue grades. Pulp viscosity and tensile strength are not important physical properties for packaging applications.

Results of the ranging tests for Kappa numbers indicated that the amount of residual lignin in the pulp generally decreased with increase in pulping time. However, in some cases, lignin redeposited onto the pulp fibres after long cooking times. The results also indicated that the pulp viscosity decreased up to a particular cooking time and, thereafter, it increased with cooking time. The reason for this phenomenon is likely that, with longer cooking times, the amount of hemicellulose remaining in the pulp decreases and the pulp yield declines. The presence of hemicellulose causes a decrease in the pulp viscosity because it has a shorter polymer

[2] The liquor to fibre ratio is the ratio of the mass of liquor (in both liquid and bagasse phases) to the mass of dry solid fibre.

chain length than cellulose. In general, the higher the operating temperature, the lower the residual lignin content of the pulp, as indicated by lower Kappa numbers. There was some scatter in the results for the effect of cooking temperature on pulp viscosity at long cooking times. Nevertheless, at shorter cooking times, the pulp viscosity increased with increasing cooking temperature. For the range of ethanol concentrations used, the Kappa number of the pulp decreased with increasing ethanol concentration in the cooking liquor. This phenomenon is expected since lignin is more soluble in ethanol than in water. The cooking liquor composition had less impact on pulp viscosity than the cooking temperature.

Pilot scale solvation pulping trials

An 18.5 L Parr reactor was acquired for increased scale testing of the Ecopulp process. During this phase of the project, a test laboratory was established with assistance from CSIRO Forestry and Forest Products (now called ENSIS). About 3 m^3 of bagasse was collected from the milling train at Racecourse Mill, Mackay. The bagasse was derived from the cane variety Q135[3]. Nearly all pilot test work on the large reactor was undertaken using this sample. The bagasse was depithed in a standard sieving procedure. In order to investigate the effect of aging bagasse on final pulp properties, smaller samples of bagasse were regularly collected from the storage pile at Proserpine Mill[4].

Increased quantities of pulp were produced from the 18.5 L Parr reactor in the pilot tests to enable the production of handsheets for pulp testing. The mass of the pulp sample prepared in the large reactor was about one hundred times more than that produced in the 500 mL Parr reactor.

Pilot trial results for pulp

The Kappa numbers of the pulp samples from these trials were typically in the range from 69.5 to 81.8 and considerably higher than the expected Kappa number of 40 (or less). Upon washing with water, the Kappa number of the pulp reduced to about the low 60s. The viscosity[5] of the pulp using fresh bagasse for the recommended conditions ranged from about 856 cm^3/g to about 1255 cm^3/g implying the pulp had fair to satisfactory degree of polymerisation. Viscosities for the pulp of 1000 cm^3/g or greater are preferred.

The Kappa number reduced to 57.1 when a multistage cook was undertaken on the large reactor. Low Kappa numbers (38.5 and 30.6) were obtained for the pulp produced from degraded, depithed bagasse obtained from Proserpine Mill in the storage trial evaluation. However, the viscosities for these pulps were low (657 cm^3/g and 549 cm^3/g respectively, indicating poor tensile strength).

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[3] The analysis for the source cane Q135 from Racecourse Mill was 16.4% fibre and 15 CCS. Q135 is a common variety for the Mackay district.

[4] The cane supply source for these bagasse samples could not be traced.

[5] The units for the viscosity are not dimensionally consistent with either dynamic viscosity (Pa.s or Poise) or kinematic viscosity (m^2/s) used in SI. The units are traditional units used by the Australian pulp industry. To convert to mPa.s or cP, the viscosity in traditional units should be multiplied by 1.052/100.

In Australia, unbleached pulp targeted for corrugating medium must compete with recycled fibre. The critical properties for corrugating medium are tested using the Concora Medium Test and the Ring Crush Test. CSIRO undertook these tests (Catela and Farrington, 2004) and the pulp as submitted for testing is currently not suitable based on these preliminary results. Further development work is required to make the pulp suitable for this market.

Bleaching and other tests were undertaken on pulp samples submitted to CSIRO for analysis. The Kappa numbers of the brownstock samples for bleaching tests are high (63.1 for Racecourse Mill sample and 62.4 for Proserpine Mill sample). Both samples of the feed bagasse were not aged before pulping. It has been noted previously by Alcell

The nominated bleaching scheme[6] is (E)(O)(D_0)(E_P)(D_1), where the first stage, the E stage, refers to caustic extraction, the O stage is oxygen delignification under alkali conditions, the D_0 and D_1 stages are chlorine dioxide stages and the E_P stage is caustic extraction with peroxide. The pulp is washed between each stage as identified by the brackets. The bleaching response is reported in Table 2. It should be noted that the scheme is a preliminary bleaching investigation and demonstrates what is achievable. The bleach scheme was not optimised. Organosolv pulps at equal Kappa numbers to kraft pulps are reported by Alcell (Ni *et al.*, 1996) to bleach more easily and with lower chemical demand.

Table 2 Comparison of bleaching response for both Racecourse and Proserpine Mill samples.

Bleach stage	**Racecourse Mill ISO Brightness (%)**	**Proserpine Mill ISO Brightness (%)**
E	17.2	25.6
O	34.3	73.8
D_1	58.9	73.9
E_P	72.9	83.9
D_2	78.2	86.6

The bleached bagasse pulp produced by CSIRO (Catela and Farrington, 2004) showed that the pulp could achieve suitable colour and brightness for tissue and towelling and perhaps low quality writing papers. However, the poor breaking length (about 5.1 km) and opacity (about 100.2% ISO) of the sample may make the pulp less suitable for these grades. In mitigation, it should be stated that pulps produced from bagasse are known to be inferior to wood pulps in terms of properties such as freeness, breaking length and other physical properties. It is common practice to blend short fibre bagasse pulp with other pulps in the furnish to achieve acceptable properties of the final product from the paper machine. Each pulp in the furnish provides unique properties to the paper product. Short fibre pulps have properties other than strength, which is provided by the long fibre pulps. Many white office papers contain up to 20% calcium carbonate, which provides no strength to the sheet, but improves paper opacity. Weak short fibre pulps can achieve similar results.

[6] The bleaching scheme was nominated by Ecopulp.

The brightness levels achieved in the bleaching tests undertaken by CSIRO were significantly different for two samples (78.2 and 86.6). Differences in cane variety and other factors such as the extent of depithing and dirt and ash levels are likely causes of the variations.

Pilot trial results for other co-products

Yields of the products of the Ecopulp process (pulp, lignin, xylose, furfural, acetic acid and other chemicals) were determined in the pilot tests. However it was difficult to obtain complete closure for the mass balance and the results are not presented.

Typically about 0.75 kg of bone-dry[7] depithed bagasse was digested to produce about 0.33 kg of bone dry pulp (about 44% pulp yield) and about 0.08 kg of bone dry lignin (about 11% lignin yield). The yield of lignin increased for two tests on aged bagasse (from about 11% to about 12% and 15% respectively).

Ecopulp claim that the lignin may be used as a binder in brake linings, particle board, orientated strand board and other products. Other applications include use in circuit boards and phenol-formaldehyde resins. The use of lignins in polymers may provide a substitute for polymers derived from fossil fuels. A less economically desirable option would be to use the isolated lignin as sulfur-free fuel to provide process energy. The lignin from wood is reported (Pye and Lora, 1991) to have a slightly lower heating value than coal (25.6 MJ/kg on a dry basis). The samples of lignin recovered on the 18.5 L Parr reactor were relatively small (about 80 g per cook). No trials have yet been undertaken to test the lignin product in the applications mentioned.

Preliminary economic assessment

The economics of the Ecopulp process were assessed for three tier pricing (namely low, middle and high price levels for all co-products). The economic return for a plant that produces 100 adt/d[8] of brownstock pulp is satisfactory for both high and medium levels of pricing. The economics are determined assuming pricing for lignin based on the polymeric properties of the product and not on the heating value. Economic modelling for the process is complicated in that the process is novel and obtaining reliable capital costs and operating costs for the process are difficult. By refining the basic process, costed in the study, it may be possible to reduce capital costs and operating costs to achieve better outcomes for the process. Better cost information will be obtained in acquiring pilot plant equipment.

The costs for steam were determined to be the highest operating cost item, but these are likely to reduce through the application of energy integration techniques, stream recycling and reduced liquor to solids ratio in a continuous reactor.

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[7] Pulp products are normally sold on the basis of being air dry (containing 10% moisture). Bone dry emphasises that all water is removed. Pulp is never sold bone dry for technical reasons.
[8] adt is short for air dry tonnes, and includes 10% moisture.

Conclusion

The outcomes from the project justify that work should continue through either pilot plant or upgraded laboratory facility with the aims of:

- demonstrating that a continuous process will achieve better delignification than the batch process tested;
- developing an unbleached product suitable for corrugating medium and other markets;
- producing increased quantities of products (particularly lignin) for market assessment; and
- refining the technology (including reducing overall energy consumption through applying energy integration technology and evaluating other low cost lignin removal options).

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Ecopulp is thanked for reviewing the paper. The Ecopulp organsolv technology evaluated by SRI is the property of Ecopulp Pty Ltd and any further detail and enquiries of a commercial nature should be directed to Ecopulp, 7 Forswood Avenue, Turramurra NSW 2074.

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