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Sylvie Gagné
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New Forest Paper Mills LP

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The North American pulp and paper industry – a look forward

35 years ago I joined the pulp and paper industry working for an engineering company that offered a new technology for sulphite mills. Timing was not good. Sulphite mills were rapidly disappearing and for me it was the time to move on. After that, I was in a small R&D department of a tissue mill. That was a much better experience: all production was sold at good profit, the mill was expanding and there was no lack of R&D funds. Later, I found a similar spirit of prosperity and growth, at least initially, when I joined Paprican at the end of the 70’s. At that time, paper consumption correlated per capita income and parameters were rising monotonically.

Now let’s fast forward 20-30 years. At first sight it might appear that the pulp and paper industry is still going strong. Between 2000 and 2010 global production increased by 20%, to reach 400 Mt/y. But, in the same time period, the production of the two formerly largest producing regions declined abruptly, North American by some 15-20% and Japanese by 25%. Most of the production increase occurred in Asia (from 95 to 170 Mt/y). China alone accounted for 60 Mt of annual increase and became the world’s largest producer. Paper consumption is still rapidly growing in developing countries, but it is not expected to reach the present North American consumption.

What is then the future of the North American pulp and paper industry? Even if the economic situation will improve, paper consumption, which is no longer coupled with per capita income, will not come back to levels of the late 90’s. Yet, not all grades of pulp and paper are in decline. Producers of tissue and hygienic products prosper, as do many paperboard and pulp producers. The consumption decrease affects mainly papers used for recording and disseminating information. The producers of newsprint or fine paper might today feel as I did in the 70’s marketing the technology for sulphite mills. As an ever-larger proportion of information will be recorded and distributed by electronic media, the role of paper as the custodian of human knowledge will diminish. This year, for the first time, US spending for online advertising at $US 40 billion will exceed combined spending of $US 34 billion for advertising in newspapers and magazines. Printing and writing papers will still be used for the foreseeable future, but they will not be the main future grades. There are good prospects for those grades that are doing well today, such as pulp, packaging grades and hygienic products. Current R&D efforts will probably bring about novel products in these areas. Large countries with rapidly increasing paper consumption such as China and India, have only limited potential for fibres production and with the declining paper consumption the availability of recycled fibres will also decrease. This promises a good future for pulp suppliers who, in addition to pulp, might offer a series of new products based on lignin and hemicellulose.

More exciting results can be expected from the intensive, world-wide research seeking completely novel, forest-based, ecological alternatives to products made from materials such as plastics or metals. The North American pulp and paper industry will eventually prosper, but it will with continuously changing products.

Congratulations to J-FOR’s Revision Panel

We hope you will enjoy this new issue of J-FOR including very interesting papers related to pulping and papermaking, and we take this opportunity to thank Dr. Dave McDonald and Dr. Ramin Farnood for their hard work as Associate editors on this issue. We are also pleased to welcome our newest Associate Editor, Dr. Ivan Pikulik, who will provide further expertise and review support in the field of papermaking.
Industry CEOs to kickoff each day at PaperWeek Canada 2013

John D. Williams, President & CEO of Domtar, will give the official kick-off to PaperWeek with a keynote presentation on the state of the industry and its future and share Domtar’s vision on the industry’s sustainability.

James Lopez, President & CEO of Tembec, will present Tembec’s approach to face the challenges and opportunities tied to the transformation of the industry and the recent progress we have made. Mr. Lopez will be followed by Kurt Schafer, VP Fiber with RISI, who will give a world outlook on the pulp and paper market trends and forecasts.

Alain Lemaire, President & CEO of Cascades, will discuss Cascades’ vision and its unique approach to tackle the industry’s markets and develop a successful sustainable development strategy. Frédéric Bouchard, Managing Director, Forest, Paper & Packaging Practice at PricewaterhouseCoopers will follow with a financial analysis and how major projects around the world get financed.

PAPTAC Launches Business Awards to Recognize Leadership Excellence

In its objective to underline excellence for the pulp and paper industry, PAPTAC is proud to be launching three Business Awards in addition to its renowned technical and life achievement awards. The PAPTAC Business Awards recognize excellence in three specific management areas and provide an opportunity to promote the highest levels of business leadership in the Canadian pulp & paper and forest-based industry. This recognition is awarded on the largest National platform during PaperWeek Canada, organized by PAPTAC.

The 2013 Business Award categories are Mill Manager of the Year Award, Safety Leadership Award, and Environmental Strategy of the Year Award. We encourage all companies to participate and submit entries and take part in celebrating business excellence and leadership in the Canadian industry.

Visit www.paptac.ca for details.

New Leadership for the Maintenance Community

Andre D. Murphy is Manager, Maintenance & Engineering at Tolko Industries, Kraft Papers Division. With more than 25 years of experience in the kraft paper industry, Andre Murphy remains focused on driving achievement and success in maintenance reliability and increased paper production for improved performance and operation efficiency. Andre brings insight, knowledge and leadership in managing the challenges faced within maintenance of the pulp and paper industry today.

Engineering Data Sheet Update G-4 - PULP, PAPER AND BOARD MACHINES: Fan Pump Calculations

This data sheet, produced and updated by the Paper Machine Technology Community, provides under a spreadsheet format a simple way to calculate various wet-end flow rates based on a simplified material balance approach, as well as pump power requirements. Calculations are available under separate tabs in either US or metric units.

Bleaching Community Spring Meeting Recap

The Spring 2012 meeting of the PAPTAC Bleaching Community was graciously hosted by Domtar Inc. in Dryden, Ontario, June 11-13. Forty-four attendees were present, including members from 13 different pulp mills and pulp & paper companies. Two technical sessions were held at this meeting. The always-popular Problems & Opportunities round-table was filled with practical questions and discussions on mill issues and problem-solving. Sulphuric Acid was the safety topic for this meeting, and attendees also discussed recent safety incidents and heard short presentations on a variety of bleaching topics. The group toured the Domtar Dryden mill on the Wednesday.

Sylvie Gagné
Process Engineer and Wastewater Treatment Plant Supervisor
New Forest Paper Mills LP

Sylvie Gagné is a graduate from the University of Ottawa, earning a degree in Chemical Engineering-Biotechnology in 1993 and a Masters of Applied Science in Chemical Engineering in 1996. Her first pulp and paper experience was as a Coop Student at the James-River (Marathon, Ontario) pulp mill and was hooked by the mill environment: working with people to achieve technical objectives. Upon graduation she worked as a Technical Service Engineer with Nacan Products Limited, providing technical expertise to papermills in the use of wet-end starch, size press starch applications and size emulsification. Subsequently, she was the Process Engineer at Eka Chemicals in Toronto, a plant producing AKD size and de-inking additives for the paper industry. Currently Sylvie provides technical expertise, process analysis and project management for stock preparation, paper machine operation and the wastewater treatment plant at New Forest. Sylvie is not happy with the status quo and continuously looks for ways to improve safety, paper quality, process performance, and to reduce production costs. She is also committed to improving herself, taking part in numerous PAPTAC courses and seminars over the years and recently earned her Project Management Professional (PMP) designation (2011).

Sylvie’s has been a member of PAPTAC for many years and in 2009, she won the 3rd prize in PAPTAC’s Energy Conservation Opportunity Awards. She promotes the pulp and paper industry by speaking to schools, Girls Guides groups and as a guest speaker at the University of Toronto “Pulp and Paper Processes” course. “I also encourage all my Co-op students to join PAPTAC!”

Visit www.paperweekcanada.ca
It has recently been shown that the energy efficiency during first-stage TMP (thermo-mechanical pulp) refining can be improved using a modified chipping method (collimated chipping), where the angle between the fibre direction of the wood specimen and the cutting plane is increased. This paper reports the differences in properties of TMP refined from wood chips produced at two different spout angles, 30° and 50°, with and without the addition of sodium bisulphite (NaHSO₃) to the dilution water. It was found that the specific energy input for a certain CSF (Canadian Standard Freeness) value was lower for chips produced at spout 50°, but that the addition of chemicals to the dilution water had no influence on the specific energy value for a given CSF value. However, the tensile index and specific light-scattering coefficient were substantially higher for handsheets made from the pulp refined from chips produced at spout angle 50° and with NaHSO₃ added compared to handsheets from pulp made from 30° and 50° chips without chemicals added.

**INTRODUCTION**

The interest in mechanical chip pre-treatment processes for TMP (thermo-mechanical pulp) has increased in recent years. The main driving force for this interest is the wish to reduce the energy consumption during refining while maintaining or improving strength and optical properties. To induce damage in a wood chip, axial pre-compression has been shown to be the most efficient method [1]. It has also been shown to be possible to achieve a reduction in refining energy of axially pre-compressed western hemlock blocks by 9% for TMP and 40% for CTMP (chemi-thermo-mechanical pulp). Pre-compression of wood chips opens up the wood structure, which increases the specific surface area [1,2] and facilitates the penetration of chemicals and water.

A number of kinds of equipment are used in mechanical and chemi-mechanical pulping which can be claimed to provide compressive mechanical pre-treatment. The most well-known of these are: the Impressafiner [3, 4], the plug screw feeder [5], the PREX impregnator [6], and the Bi-Vis Extruder [7]. The compressive action of some of these devices also makes it possible to press out substantial amounts of water and thus to reduce the load of extractives entering the chip refiner [3,4]. Drawbacks of these devices are that the pre-treatment is not oriented solely in the axial direction of the wood fibre, that they are expensive, that they require space for installation, and that they often become bottlenecks for production.

The basic idea behind the concepts presented in this paper is the observation...
that when wood is loaded in compression parallel to the fibres, cracks running along the fibres are initiated. These cracks are known to be beneficial for energy efficiency during chip refining because they serve as growth sites for cracks during the subsequent refining process. Numerical simulations have shown that chips produced at a large spout angle experience extensive plastic deformation [8]. A theoretical model [9] has indicated that large compressive stresses might occur during chipping.

It has been observed that mechanical refining of axially compressed chips results in TMP pulps with significantly improved strength properties and lower shive content [3]. It has also been observed that chemical treatment of pre-treated (axially compressed) chips improves the pulp quality considerably more compared to the corresponding chemical treatment of non-pretreated chips [4]. In a previous investigation [10], it has been shown that chipping in such a way that the compressive damage induced to the chips is maximized will reduce the specific energy to achieve a certain CSF (Canadian Standard Freeness) value at the same mean fibre length. A patent application regarding the modification of the chipping process as presented in [10] has been filed and is pending.

The main goal of this study is to investigate whether it is possible to achieve an improvement of some of the main properties used to define the quality of printing grades, i.e., tensile index and specific light-scattering coefficient, by combining refining of chips produced using a spout angle of 50° (more prone to induce directed cracks) with addition of bisulphite solution (\(\text{NaHSO}_3\)) to the dilution water during chip refining. It was shown earlier that by using somewhat acidic conditions in CTMP production, it is possible to improve strength properties at constant energy consumption without reducing the light-scattering coefficient [11], which is important for printing paper.

**EXPERIMENTAL**

**Materials**

To produce the amounts of chips needed for the refining trials, a specially developed laboratory chipper was used to chip fresh Norway spruce (\(\text{Picea Abies}\)) in the form of planks with a cross section of 50 mm x 100 mm. The chipper is described in detail in [12]. The chips were produced using two different values of the spout angle, \(\epsilon\) (30° and 50°) (see Fig. 1), keeping all other parameters constant. To be more specific, the spout angle is the angle between the cutting direction (indicated by an arrow in Fig. 1) and the wood fibre direction. The cutting rate, \(v_c\), was 20 m/s, and the nominal chip length was 25 mm.

The other angles in Fig. 1 are the clearance angle \(\alpha\), the sharpness angle \(\beta\), and the complementary angle \(\delta\), defined simply by \(\delta = 90° - (\alpha + \beta + \epsilon)\). For each value of the spout angle (i.e., for each refining trial), approximately 80 kg of chips was produced. For chip dimensions, i.e., chip thicknesses for the two spout angles, and for details regarding the knife geometry, chip dimensions, etc., see [10].

Mechanical first-stage refining of chips produced as described above was performed using a pilot single-disc refiner (OVP-MEC) with technical data as given in Table 1. Before first stage refining, the chips were pre-steamed for 15 minutes in an open bin at atmospheric pressure, and to produce a pulp adapted for printing-paper quality, bisulphite was used, in this case, approximately 30 kg \(\text{NaHSO}_3\) for each ton of dry pulp. The chemical was added to the dilution water direct into the inlet of the refiner in the first stage. Refiner type, size, refiner plates, etc. for second-stage refining was the same as in the first stage. The second-stage refining was performed at atmospheric pressure (ROP-20). Canadian Standard Freeness (CSF) measurements were carried out in accordance with the ISO 5267-2001 TAPPI test method. The PQM (Pulp Quality Monitor) technology [13] was used to determine the average fibre length. It was concluded that the pulp from chips produced at spout angle 50° had roughly the same average fibre length as the fibres from the other type of chips.

**RESULTS AND DISCUSSION**

Figures 2–4 show the outcome of the refining trials. The circles denote chips produced at spout angle 30°, and triangles those produced at 50° (collimated chipping). The unfilled markers denote pulp production without sodium bisulphite (\(\text{NaHSO}_3\)), and the filled markers denote the situation where sodium bisulphite was added. The term “B” represents bisulphite in the legend. Figure 2 presents a plot of freeness vs. specific energy. The results show that there is a reduction in refining energy on the order of 15% for pulp produced by refining chips created by collimated chipping, i.e., with a spout angle of 50°. Moreover, it is obvious from Fig. 2 that the addition of \(\text{NaHSO}_3\) had no noticeable effect of the refining energy for a given CSF value.

For printing grades, the tensile index is an important parameter. Figure 3 shows a
curves correspond to cases without chemicals added, and the highest curve is obtained after addition of NaHSO₃. It can be concluded that for tensile index values below 34 Nm/g, the effect of a spout angle of 50° represents an energy saving, while the effect seems to disappear at larger tensile index values. The tensile index is substantially higher for a spout angle of 50° with addition of NaHSO₃. The largest standard deviation was 5.4% for a spout angle of 50° without chemicals, 5.5% for a spout angle of 30°, and 5.8% for a spout angle of 50° with chemicals added.

Another important quality parameter for printing grades is the specific light-scattering coefficient, which is shown in Fig. 4. The three curves represent the same three cases as in Fig. 3 and rank in exactly the same way. The specific energy consumption for a light-scattering coefficient of 44 m²/kg is 10%–13% less for a spout angle of 50° with addition of NaHSO₃ than for a spout angle of 30° with no chemical added. However, due to a mistake, the tensile index and light-scattering data for a spout angle of 30° and with chemicals added were lost, so nothing can be said about the influence of chemicals on the properties of pulp produced from conventional chips.

The results are in accordance with the hypothesis that an increased amount of compressive damage induced to the wood hips during the chipping process makes it possible to improve quite substantially the energy efficiency of the subsequent refining process. The energy efficiency improvement potential seems to be approximately 200 kWh/t, and the full effect is seen already at very low energy consumption. This can be interpreted as meaning that the initially created surfaces explain most of the energy efficiency improvements. The subsequent refining seems to develop tensile and light scattering in such a way that the same slope as for conventional TMP is followed.

This work also shows that the spout angles normally used in the chipping process are not as well adapted to mechanical pulping as they are to chemical pulping. It was noted that, quite opposite to what one might expect, the bulk density for chips produced at spout angle 50° was greater than for chips produced at spout angle 30°. It should also be pointed out that no plot of tensile index vs. specific energy for handsheets made from pulps obtained by refining of chips produced at 30° and 50° spout angles. The lowest and intermediate curves correspond to cases without chemicals added, and the highest curve is obtained after addition of NaHSO₃.

![Fig. 2 - Freeness vs. specific energy.](image)

![Fig. 3 - Tensile index vs. specific energy.](image)
attempts have yet been made to maximize the compressive damage by optimizing spout angle and chip length. This can in fact be expressed mathematically as an optimization problem. Finally, it should be pointed out that the chips used in this investigation were cut from planks and not from logs, and it might well be that the impact of an increased spout angle on chips cut from logs is different. Understanding better the differences between pilot-scale chipping on planks and full-scale chipping on whole logs could be a topic for future investigations.

CONCLUSIONS

It has been shown through pilot-scale experiments that chipping in a collimated way, i.e., in such a way that the amount of compressive damage induced to the wood chips is increased, will decrease the specific energy needed to achieve a certain tensile index, specific light-scattering coefficient, and CSF value. This reduction is on the order of 15%. Moreover, adding bisulphite to the dilution water during refining will increase the beneficial effects of collimated chipping on two important quality parameters for printing grades, namely the tensile index and the specific light-scattering coefficient.

ACKNOWLEDGEMENTS

The European Regional Development fund is acknowledged for financial support, while Anders J. Persson at Metso Paper is greatly acknowledged for carrying out the refining trials.

REFERENCES

OPTIMIZATION OF SELECTIVE REFINING OF SULPHONATED LONG FIBRES FROM JACK PINE: FIBRE CHARACTERISTICS

YOUFENG LIN, ROBERT LANOUETTE*

In the research reported in this paper, modelling was performed to study the effect of sulphonation conditions on fibre properties of the long-fibre fractions of Jack pine thermo-mechanical pulp during selective refining. Then three different chemi-mechanical pulping procedures were compared: sulphonation of long fibres after fractionation, inter-stage whole-pulp sulphonation, and chip sulphonation during primary-stage refining (CTMP process). The results showed that various sulphonation conditions had different impacts on the fibre properties of the long-fibre fraction. Sulphonation associated with selective refining required less energy and developed fibres more efficiently than sulphonation of chips or whole pulp.

INTRODUCTION

Representing about 20.4% of conifer wood species, Jack pine is an abundant commercial tree species in Canada [1]. However, compared to black spruce, Jack pine has seen limited use in mechanical pulp mills because of drawbacks such as pitch deposits, high energy consumption, and poor strength properties, which are closely related to its specific fibre characteristics. Jack pine has high extractives content, high fibre-wall thickness in summer wood, and a high summer/spring wood ratio.

Selective refining based on fractionation can separate primary pulp effectively into uniform fractions. This process has attracted attention in recent years because of its ability to save energy while maintaining certain advantages associated with untreated fines in comparison with whole-pulp refining [2,3,4]. From the authors’ recent pilot study on mechanical pulping of Jack pine [5] compared to conventional thermo-mechanical pulping, selective refining of long-fibre fractions with chemical treatments, especially sulphonation, facilitates the development of Jack pine fibres, providing better tensile strength while reducing refining energy consumption. The first part of the present experiments was conducted to evaluate the influence of sulphonation conditions on selective refining of Jack pine and to obtain optimum sulphonation conditions [6].

In this experiment, the objective was first to evaluate the influence of sulphonation conditions on fibre characteristics developed during selective refining of sulphonated long fibres, and second to compare the development of fibres from three chemi-mechanical pulping processes: sulphonation of long fibres after fractionation, inter-stage whole-pulp sulphonation, and chip sulphonation during primary-stage refining (the CTMP process).

EXPERIMENTAL

Material

Jack pine (Pinus banksiana Lamb.) used in this research was obtained from the northern Mauricie region in the province of Quebec. Jack pine chips were screened using Rader disc screens (Rader Company, Inc.) to remove the over-thick chips and fines, then washed and drained.

Methodology

A schematic representation of the experimental work performed is shown in Fig. 1. Pulps were produced using four types of chemi-mechanical pulping processes,
including selective refining with long-fibre sulphonation (CTMP\textsubscript{LF}), pulp recombined with its short-fibre fraction (M-CTMP\textsubscript{LF}), inter-stage whole-pulp sulphonation (CTMP\textsubscript{WP}), and CTMP based on chemically treated chips (CTMP\textsubscript{CHIPS}). The procedure consisted of first-stage refining, fractionation, and sulphonation, as illustrated in Fig. 1.

**Refining**

Refining consisted of two stages: a primary and a secondary refining stage, both conducted using the Metso CD300 pilot refiner at Lignocellulosic Material Research Centre (LMRC) in Trois-Rivières, Quebec. For primary refining, Jack pine chips steamed at atmospheric pressure were fed by a plug screw into the preheater at 262 kPa for five minutes before being refined at approximately 138°C at a production rate of approximately 0.59 kg/min and 26% discharge consistency. The specific energy consumption for primary refining was 4.41 MJ/kg. Under atmospheric conditions and with the measured discharge pulp consistency ranging from approximately 9% to 15%, second-stage refining was performed in a single pass to produce pulp samples with different freeness by adjusting the refiner plate gap each time. All pulp samples were kept in a cold chamber.

**Fractionation**

Primary pulp (at approximately 700 CSF) was fractionated using a Black-Clawson model 8P pressure screen equipped with a two-foil open rotor rotating at a tangential speed of 20 m/s to obtain two fractions: a short- and a long-fibre fraction. The fractionation process consisted of a two-stage cascade with a 0.25-mm smooth-hole basket. Ferluc et al. [7] have proven, after testing several fractionation strategies, that this method is an efficient way to separate primary Jack pine pulp very well [5].

**Sulphonation**

For wood chips, sulphonation was performed in the preheater before primary refining by adding 4% sodium sulphite at the outlet of the plug screw. For long-fibre fractions and whole fibres, sulphonation was carried out in the inter-stage reactor of the LMRC using 4% sodium sulphite at 130°C for 30 minutes. To study the impact of sulphonation conditions on selective refining of long-fibre fractions, a custom design of JMP (version 8.0, SAS Institute) was used to prepare the experimental plan (Table 1), using coarseness...
and acidic-group content as the responses. The data were initially evaluated by stepwise regression to eliminate the least significant variables, and then the remaining significant variables were refitted to produce a satisfactory model using the standard least-squares error-fitting model.

**Fibre and Handsheet Properties**

The fibre-length distribution of pulp was characterised using a Bauer-McNett apparatus. Fibre coarseness was measured using a Fibre Quality Analyzer (OpTest Equipment, Canada), and the coarseness of fibres at 100 CSF was obtained by interpolation. The fibre-wall thickness was determined by MWT (MorFi Wall Thickness, Techpap, France). The samples for coarseness and wall thickness were prepared from R28 Bauer-McNett fractions. Sulphonic and carboxylic groups were measured using conductometric titration [8].

The sedimented volume of fines was determined by the method described by Marton and Robie [9], which mainly consists of the collection and sedimentation of fines.

Latency was removed from all pulp samples using the Domtar disintegrator. 60-g/m² handsheets were prepared using a British sheet-mould former and then were kept in a constant temperature and humidity room. The freeness, physical, and optical properties of the handsheets were evaluated in accordance with PAPTAC standard testing methods. The data for each property at 100 CSF were obtained by interpolation.

### RESULTS AND DISCUSSION

**Influence of sulphonation conditions**

Figures 2, 3, and 4 show the response surfaces from the models used to interpret the content of acidic groups and coarseness, as indicated in Eqs. 1, 2, and 3. The coefficients of determination ($R^2$) of all three models are high, indicating that most of the experimental variance can be explained by the models.

Sulphite treatment results in the formation of acidic groups, including sulphonic and carboxylic groups, during pulp production. These acidic groups on fibres play an important role in influencing fibre swelling and fibre bonding [10,11]. As shown in Fig. 2, high pH could produce a pulp with more carboxylic groups because uronic materials in pectin and hemicellulose are more easily hydrolysed into new

---

**TABLE 1** Experimental factors and levels.

<table>
<thead>
<tr>
<th>Factor</th>
<th>Level</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>7–11</td>
</tr>
<tr>
<td>Time, min</td>
<td>2–31</td>
</tr>
<tr>
<td>Dosage of sodium sulphite, %</td>
<td>2–6</td>
</tr>
</tbody>
</table>

---

Carboxylic acid = $94.93 + 2.40(X - 9) + 0.0714(Y - 17) + 3.24(Z - 4) - 0.079(Y - 17) * (Z - 4) + 0.01(Y - 17)^2$

(1)

Sulphonic acid = $41 + 4.941(X - 9) + 0.132(Y - 17) + 12.44(Z - 4) - 0.353(X - 9) * (Z - 4) - 0.0625(Y - 17) * (Z - 4) + 0.021(Y - 17)^2 + 0.616(Z - 4)^2$

(2)

Coarseness = $0.233 - 0.00138(X - 9)^2 - 0.000224(Y - 17) - 0.00184(Z - 4) - 0.0000138(X - 9) * (Y - 17) + 0.00138(Z - 4)^2$

(3)

where X is pH, Y is sulphonation time, and Z is the dosage of sodium sulphite (uncoded values).

---

**Fig. 2** - Response surface of carboxylic group content as a function of time and pH, keeping dosage of sodium sulphite at a moderate value (4%).

**Fig. 3** - Response surface of sulphonic group content as a function of time and pH, keeping dosage of sodium sulphite at a moderate value (4%).

**Fig. 4** - Response surface of coarseness as a function of time and pH, keeping dosage of sodium sulphite at a moderate value (4%).
carboxylic groups under alkaline conditions [12,13]. Under alkaline conditions, more sulphonic groups can be generated (Fig. 3), an observation which agrees with the result that more sulphonic groups can be obtained in the presence of sodium hydroxide during sulphonation of wood chips [14]. The larger quantity of acidic groups generated in long fibres treated under high-pH conditions partly contributes to the high tensile strength of paper [6]. Extending sulphonation time does not significantly increase the number of acidic groups, whereas at maximum sulphonation time, the acidic groups increase slightly, which could be one reason for the increase in tensile strength [6].

As can be observed in Fig. 4, under conditions of high pH and long sulphonation time, selective refining can produce fibres with lower coarseness. Increasing the dosage of sodium sulphite can also reduce coarseness (effect not shown here). The reduction in coarseness can increase fibre flexibility, improving the bonding surface of fibres and thus having a positive impact on tensile strength. Meanwhile, the reduced strength of single fibres related to the reduction in coarseness might explain the decrease in tear strength of pulp with better sulphonation [6].

**Specific Energy Consumption**

One of the existing drawbacks in Jack pine mechanical pulping is its high refining-energy consumption. The selective refining of long-fibre fractions of Jack pine instead of whole-fibre treatment could effectively reduce refining energy during TMP production [5].

In Fig. 5, the energy consumption levels for different chemi-mechanical pulping procedures are compared. Inter-stage whole-pulp sulphonation (CTMP(wp)) consumes less energy than refining sulphonated chips, which supports the conclusion that inter-stage sulphonation is better than wood-chip sulphonation for saving refining energy [15]. However, compared to these two procedures, recombination of sulphonated long fibres with short-fibre fractions requires less energy to reach a given freeness, showing that fibres can absorb energy more efficiently during selective refining.

**Fibre properties**

It is well known that CTMP refining of sulphonated chips is characterised by a high proportion of long-fibre fractions compared to TMP. Fibre rupture occurs in the middle lamella/primary wall, keeping fibres more intact [16]. As illustrated in Fig. 6, the recombined pulp (M-CTMP(L)) from selective refining of sulphonated long fibres has a high R14+R28 content, similar to that of CTMP (CTMP(Chips)). The recombined pulp also has slightly more fines, but less of the R48 and R100+R200 fractions, than CTMP. This indicates that coarse fibres can be developed rather than broken down or shortened into small fragments as with inter-stage whole-pulp sulphonation, which produces a pulp with higher R48 and R100+R200 fractions, but less R14+R28 and fines.

Figure 7 shows the behaviour of individual fibres responding to the force inside the refiner for the three processes.

The cell-wall thickness of the fibres from selective refining is lower than that of the fibres from inter-stage whole-pulp sulphonation, indicating more peeling-off and delamination of fibres during selective refining. However, due to the presence of short-fibre fractions during whole-pulp refining, coarse fibres receive a less uniform force from the refiner, which hinders fibre development.
After addition of the short fraction to produce recombined pulp (M-CTMP LF), the sedimented volume of fines decreases due to the introduction of flake-like particles from the short-fibre fraction. The sedimented volume of fines from recombined pulp, which consists of selectively refined pulp with the addition of short-fibre fractions, is less than that of pulp from whole-pulp refining, a difference which is probably related to differences in fibre development during second-stage refining. More specifically, middle lamella materials that remain on the fibre surface after the first refining stage are broken down into fines by more intense impacts on fibres during selective refining. These materials enter the R100 and R200 fractions during inter-stage whole-pulp sulphonation.

Physical and Optical Properties of Handsheets

Physical and optical properties of hand-sheets from recombined chemi-mechanical pulp and whole-pulp refining chemi-mechanical pulp are compared in Table 2. Table 2 shows that, at a given freeness, the recombined pulp has higher mechanical resistance, opacity, and light-scattering coefficient, while maintaining almost the same optical properties as inter-stage whole-pulp sulphonated fibres. These materials enter the R100 and R200 fractions during second-stage refining.

<table>
<thead>
<tr>
<th>Property</th>
<th>M-CTMP LF</th>
<th>CTMP WP</th>
<th>CTMP CHIPS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Freeness, mL</td>
<td>100</td>
<td>100</td>
<td>100</td>
</tr>
<tr>
<td>Specific energy, MJ/kg</td>
<td>11.6</td>
<td>11.6</td>
<td>13.3</td>
</tr>
<tr>
<td>Apparent density, g/cm³</td>
<td>0.352</td>
<td>0.352</td>
<td>0.356</td>
</tr>
<tr>
<td>Tensile index, N/m²/g</td>
<td>43.9</td>
<td>41.5</td>
<td>46.1</td>
</tr>
<tr>
<td>Tear index, mN·m²/g</td>
<td>8.2</td>
<td>7.8</td>
<td>8.85</td>
</tr>
<tr>
<td>Brightness, % ISO</td>
<td>45.2</td>
<td>45.3</td>
<td>50.4</td>
</tr>
<tr>
<td>Light-scattering coefficient, m²/kg</td>
<td>52.1</td>
<td>52</td>
<td>50.8</td>
</tr>
<tr>
<td>Opacity, %</td>
<td>97.6</td>
<td>97.3</td>
<td>96.0</td>
</tr>
</tbody>
</table>

CONCLUSIONS

Based on the models presented here for selective refining of sulphonated long fibres from Jack pine, high pH and high dosages of sodium sulphite are favourable for producing pulps with more acidic groups. Treatment under high pH with a long treatment time can decrease coarseness.

For Jack pine chemimechanical pulp, selective refining can reduce refining-energy consumption compared to whole-pulp refining. Selective refining can facilitate delamination and peeling-off of fibres, which generates pulp with better mechanical resistance than pulp from inter-stage whole-pulp sulphonation. Selective refining can produce pulp with a relatively higher R14/R28 fraction and slightly more fines with lower specific volume.

ACKNOWLEDGEMENTS

The financial support of NSERC and the contribution of technicians Alain Marchand and Daniel Bégin to this research are deeply appreciated.

REFERENCES

2. Ferluc, A., Lanouette, R., Bousquet,
TRADITIONAL AREA CONTRIBUTIONS


FACTORS AFFECTING THE MEASUREMENT OF PROPERTIES OF HIGH-YIELD PULP

ZHIRUN YUAN*, CHANGBIN MAO, WAYNE BICHARD, MICHAEL HELLSTERN, DANIEL GILBERT, YAJUN ZHOU, DARREN GULIOV

High-yield pulps (HYP, including BCTMP and APMP) have been increasingly used in various paper grades because of their unique properties such as high bulk, high opacity, and good printability. Among the various properties of HYP, bulk is usually the most important quality for end users. However, discrepancies have often been observed among various laboratories in the measurement of HYP bulk and other properties, even when the same standard test method is used. In this paper, key factors affecting the measurement of HYP properties have been identified, ranging from pulp preparation, handsheet making, and caliper measurement to the grammage basis used in the bulk calculations. It was found that the measured freeness, bulk, and strength of HYP changes with the time delay between hot disintegration and freeness testing or handsheet making. HYP bulk and freeness increase during the first four hours after hot disintegration, while the tensile strength decreases. Pre-soaking of HYP can minimize, but not eliminate, the changes in bulk and freeness with time after hot disintegration. This contributed to the difference in measurements, even though the same standard test method was used. Speed drying with a fan decreases bulk, but increases tensile strength compared to overnight drying. The number of handsheet plies in the caliper measurement also affects the bulk measurement.

INTRODUCTION

High-yield pulps (HYP, including BCTMP and APMP) have been increasingly used in various paper grades because of their unique properties such as high bulk, high opacity, and good printability. Among the various properties of HYP, bulk and strength are usually the most important qualities for end users. Correctly evaluating these HYP properties is of critical importance in predicting end-product quality and paper machine runnability.

Several standard methods available for the testing of mechanical pulps. These methods typically involve hot disintegration, testing on hot-disintegrated pulp, standard handsheet making, conditioning, and measurement on handsheets for physical and optical properties. Because market HYPs are usually flash-dried to below 10%–15% moisture content and then pressed into a bale, it is recommended to pre-soak the dry pulp for at least four hours before hot disintegration. However, there is a lack of information on how this pre-soaking affects the measurement of HYP properties.

Discrepancies have often been observed among various laboratories in the measurement of HYP bulk and other properties like freeness and tensile strength. Although this could be partially due to the differences in various standard testing methods, it did not explain the difference in measurement data when tests were carried out following the same testing method. It has been observed by the authors that the time delay between

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hot disintegration and freeness testing or handsheet making has a significant effect on HYP properties. However, there is no information on this in the literature, nor is it specified in the existing standard testing methods.

Therefore, the objectives of this paper are: 1) to study systematically the effect of pre-soaking and the time delay between hot disintegration and freeness testing or handsheet making on the measurement of key HYP properties, including bulk, tensile strength, and freeness; and 2) to clarify the differences between the main standard testing methods and their potential effect on HYP property measurements.

**EXPERIMENTAL**

**Material**

Three commercial flash-dried HYPs (bleached chemi-thermo-mechanical pulp, or BCTMP) were obtained from Canadian HYP producers: maple (M) 350/70, aspen (A) 400/85, and spruce (SP) 400/80. Never-dried (ND) pulps from the same batches were also collected just before the flash dryer as references for aspen and spruce HYP.

**Hot Disintegration and Pulp Testing**

Hot disintegration of HYP was carried out in a “Domtar-type” disintegrator following the PAPTAC C.8P standard test method. In some cases, cold disintegration was also carried out in a “British Standard type” disintegrator at room temperature for two minutes. Canadian Standard Freeness was measured according to PAPTAC Standard C.1. Handsheet properties were measured by the corresponding PAPTAC Standard methods.

**RESULTS AND DISCUSSION**

**Factors Affecting Measurement of HYP Properties**

Table 1 lists the main sources of standard test methods and their abbreviations. The main differences among these methods are reflected in the following aspects and will be discussed in this paper:

- Pre-soaking and latency removal.

<table>
<thead>
<tr>
<th>TABLE 1</th>
<th>Main sources of standard test methods.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Abbreviation</td>
<td>Sources</td>
</tr>
<tr>
<td>PAPTAC</td>
<td>Pulp &amp; Paper Technical Association of Canada</td>
</tr>
<tr>
<td>TAPPI</td>
<td>Technical Association of the Pulp &amp; Paper Industry</td>
</tr>
<tr>
<td>ASTM</td>
<td>American Society for Testing and Materials</td>
</tr>
<tr>
<td>ISO</td>
<td>International Organization for Standardization</td>
</tr>
<tr>
<td>APPITA</td>
<td>Technical Association of the Australian and New Zealand Pulp &amp; Paper Industry</td>
</tr>
</tbody>
</table>

- Time delay between hot disintegration and freeness testing or handsheet making.
- Handsheet making, drying, and conditioning.
- Handsheet testing, including bulk, tensile strength, and brightness.

Table 3 compares the properties of the three HYPs with and without pre-soaking. It shows that pre-soaking does not have a significant effect on HYP property measurement. However, in this set of experiments, all the handsheets were made four hours after hot disintegration. As will be shown later, the HYP properties will change with the time delay between hot disintegration and freeness testing or handsheet making, and pre-soaking can help minimize the scale of this change.

<table>
<thead>
<tr>
<th>TABLE 2</th>
<th>Key testing variables and differences between standard laboratory methods of latency removal.</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test Method</td>
<td>Minimum Soak Time for Dry Pulp, h</td>
</tr>
<tr>
<td>PAPTAC C.8P</td>
<td>4</td>
</tr>
<tr>
<td>TAPPI T 262</td>
<td>24</td>
</tr>
<tr>
<td>ISO 5263-3</td>
<td>4</td>
</tr>
</tbody>
</table>

**Pre-Soaking and Latency Removal**

It is a well-known fact that mechanical pulp has latency built into it during the refining process. The decrease in pulp freeness and the increase in handsheet strength brought about by the hot disintegration of mechanical pulps have been well documented [1−4].

Table 2 lists the key testing variables and the differences among the three standard laboratory methods of latency removal. All three methods recommend at least four hours of pre-soaking time for dry pulp. The main differences are the requirement for hot disintegration temperature.

**Time Delay Between Hot Disintegration and Freeness Testing or Handsheet Making**

After hot disintegration of HYP, the usual next step is to measure pulp freeness and to make handsheets for testing of physical and optical properties. PAPTAC C.8P requires that this testing be carried out after the pulp cools down, while...
Figure 1a shows that pulp freeness increased with time after hot disintegration, especially in the first four hours. The freeness leveled out after approximately eight hours. Figure 1a also shows that the change in freeness with time was less if the flash-dried pulp was pre-soaked for four hours before hot disintegration. Figure 1b shows that corresponding to the freeness change with time, the pulp bulk also increased with time after hot disintegration, especially in the first four hours. Pre-soaking also minimized the change in pulp bulk when compared to the non-pre-soaked flash-dried pulp. As a result, pulp tensile strength decreased slightly with time after hot disintegration, as can be seen in Fig. 1c.

Figure 1 indicates that the flash-dried pulps had a slightly higher freeness and bulk, but slightly lower tensile strength than the never-dried pulp, which probably occurred because the flash-drying process introduced more latency into the pulp than was present in the never-dried pulp [5–7].

Figure 2 shows the change in pulp temperature with time after hot disintegration. Pulp temperature decreased quickly during the first four hours from 90°C to 43°C and then further dropped from 43°C to 23°C during the next 20 hours. The significant decrease in temperature during the first four hours corresponds to the observed fast change in pulp freeness and handsheet properties. Figure 3 shows microscopy images of the spruce HYP. When the pulp underwent only cold disintegration (two minutes in a British Standard-type disintegrator), there were still many kinked, curled, and twisted fibers (Fig. 3a), because the latency had not been removed under cold disintegration. The pulp freeness of the cold disintegrated pulp was 460 mL compared with 405 mL for the hot disintegrated pulp. The pulp tensile index was also lower for the cold disintegrated pulp (26 vs. 30 N.m/g). After hot disintegration, the fibers become straighter and less twisted (Fig. 3b). However, after 24 hours of storage at room temperature, there was a noticeable
increase in twisted fibers, which is a clear indication of the return of latency when the pulp cools down. This is in agreement with observations in the literature [8].

Similarly, for aspen HYP, pulp properties changed with time after hot disintegration, especially in the first four hours (Fig. 4). Compared to spruce HYP, the change in properties with time for aspen HYP was slightly less. HW HYP has less latency than SW HYP. Again, the extent of change in pulp properties was less if the flash-dried pulp was pre-soaked before hot disintegration.

In summary, it is important to control the time delay between hot disintegration and freeness testing or handsheet making when comparing HYP pulp properties. Pre-soaking is recommended because it can minimize the change in pulp properties with the time delay between hot disintegration and testing. However, pre-soaking does not eliminate the change in HYP properties with time after hot disintegration.

**Handsheet Making, Drying, and Conditioning**

Several standard test methods are available for making reproducible test sheets for the measurement of pulp physical and optical properties. Although there is no direct correlation with a machinemade product, standard handsheets may be used for relative comparison of pulp properties. The British standard sheet machine method (PAPTAC C.4, TAPPI T 205, ISO 5269-1; methods are technically equivalent) is the most commonly used and is available in manual and semi-automatic versions. Internal comparison at FPInnovations has shown that there is negligible difference in handsheet properties made with these two types of machines. Another method used in some countries in Europe and Asia is the Rapid-Koethen method (ISO 5269-2), which quickly dries test sheets at an elevated temperature.

All the standard methods require that the handsheets be pressed twice at 50 psi (5.5 and 2.5 minutes respectively), with a change of blotters between these two pressings. Figure 5 shows that the bulk of spruce HYP decreased with the number of blotter sheets changed. The new and dry blotter helps to remove more water from the handsheet during pressing and therefore helps sheet consolidation.

After pressing, the handsheets should be placed into a drying ring with a drying plate and be conditioned in a standard conditioning room (23°C and 50% relative humidity) for a minimum of 12 hours. In certain mill situations, speed drying is also used for operational control purposes. A rapid method of drying and conditioning the sheets using a fan, which shortens the time required to two to four hours while still conforming to the standard procedure, has been developed by the Pulp and Paper Research Institute of Canada (see the annex in TAPPI T 205). However, Fig. 6 shows that speed drying of maple HYP handsheets results in higher tensile but lower bulk when compared with overnight drying. Apparently, the quick and forceful removal of moisture from handsheets helps sheet consolidation and results in a lower sheet bulk.

**Handsheet Testing**

**Bulk measurement** - Paper bulk refers to the compactness of a sheet in relation to its weight (volume per unit weight). Therefore, it is calculated as sheet thickness (caliper) divided by grammage (mass per unit area). The key test method variables and the differences among some standard testing methods for bulk are listed in Table 4.

Figure 7 shows that for the same maple HYP, bulk decreased when more plies of handsheets were stacked together for measurement. This may be explained by the conformation of surface peaks...
Fig. 4a - Freeness vs. time after hot disintegration for aspen HYP.

Fig. 4b - Bulk vs. time after hot disintegration for aspen HYP.

Fig. 4c - Tensile strength vs. time after hot disintegration for aspen HYP.

Fig. 5 - Effect of blotter change on bulk of spruce HYP.

Fig. 6 - Comparison of the effect of drying methods on the properties of maple HYP.

Fig. 7 - Decrease in handsheet bulk with increasing number of sheet plies measured.
and valleys of the handsheets under pressure during the caliper measurement. Morgan [9] studied the effect of anvil pressure and head surface of the measuring head on handsheet caliper measurement, as reproduced here in Fig. 8. The sheet caliper decreased linearly with the increase in anvil pressure when the same measuring head was used. At the same anvil pressure, the sheet caliper decreased with a smaller measuring head face area.

Another key difference among these test methods that could significantly affect the determination of bulk is the basis of the sheet grammage used in the calculation: oven-dry or air-dry. The bulk could be 11% higher if the PAPTAC method were used as opposed to the ISO and TAPPI methods. Considering the difference in the number of plies measured and the grammage basis used in the calculation, bulk determined using the PAPTAC test method is expected to be higher than that from the TAPPI method because the latter uses a higher number of sheet plies and calculates the bulk on an air-dry basis.

**Tensile strength measurement** - Table 5 lists the key differences among some standard test methods for tensile measurement. The main difference is in the rate of elongation, which varies from 10 to 25 mm/min. TAPPI T 205 states the following in an annex: “Doubling the test speed (for the same length of specimen) will increase the apparent tensile strength and may increase TEA for some papers by approximately 3%. In other cases, stretch will be reduced, thus acting to keep TEA nearly constant.”

**Measurement of optical properties** - The optical properties of HYPs are routinely determined for specification purposes, but can also provide useful information about pulping processes. Optical properties are commonly determined by measuring reflectances with a colorimeter. Two different instrument geometries are in current use in North America: diffuse illumination (commonly called PAPTAC, ISO, SCAN, Elrepho, or Technibrite reflectance) and directional illumination (also known as TAPPI, GE, or IPC reflectance). This can be a source of confusion when individuals attempt to compare test results obtained using different geometries. Differences of up to several reflectance percentage points may be observed between these two types of instrument geometry. No simple relationship exists between the two scales.
Brightness. The most often measured optical property is brightness, which is a useful index for comparing similar white materials. Brightness is measured as the reflectance value in the blue region of the visible spectrum (specifically, at a wavelength of 457 nm). Bleaching agents strongly influence the reflectance of pulp in this portion of the visible spectrum, and therefore brightness is widely used to measure bleaching efficiency.

Brightness can be measured using either the C or D65 illuminant, which simulates indoor and outdoor lighting respectively. The difference in measured brightness between these two illuminants is very small when there is no optical brightening agent (OBA) present in the paper. For OBA-containing paper, D65 brightness is higher than C brightness because the D65 illuminant contains more UV component than the C illuminant. The difference in brightness between these two illuminants is illustrated in Table 6 for an aspen HYP. As is well known, OBA is a significantly increased the whiteness of HYP properties in the laboratory. The whiteness derived from measurements of CIE whiteness differs fundamentally from paper brightness in that whiteness includes the entire visible spectrum in its assessment, whereas brightness includes only the blue portion of the spectrum. It has been shown that CIE whiteness correlates well with visual whiteness assessment when the UV level of the source approximates that of average indoor lighting [10].

CIE whiteness. In certain applications where measured brightness is found to correlate poorly with visual assessment of whiteness because of variations in areas outside the blue region of the visible spectrum, other indices of whiteness have been found to be useful. In general, colorimetric measurements made in three regions of the visible spectrum (known as tristimulus values) are necessary to describe any white shade completely: As a compromise, a single-number identification of a white shade has been defined for ease of understanding and communication. “CIE whiteness” is a measure of whiteness derived from measurements of the CIE tristimulus values. Table 6 shows the differences in whiteness for an aspen HYP. Without OBA, the whiteness reading is much lower than the brightness due to the yellowish colour of HYP. OBA significantly increased the whiteness reading, especially with D65 illuminant, because it has more UV component than the C illuminant.

CIE whiteness differs fundamentally from paper brightness in that whiteness includes the entire visible spectrum in its assessment, whereas brightness includes only the blue portion of the spectrum. It has been shown that CIE whiteness correlates well with visual whiteness assessment when the UV level of the source approximates that of average indoor lighting [10].

CONCLUSIONS

The key factors affecting the measurement of HYP properties in the laboratory have been identified and studied. The main findings are:

1. Measured HYP properties like freeness, bulk, and tensile strength change with the time delay between hot disintegration and freeness testing or handsheet making. HYP bulk and freeness increase during the first four hours after hot disintegration, while the tensile strength decreases. Pre-soaking of HYP can minimize, but not eliminate, these changes.
2. Speed drying with a fan decreases bulk and increases tensile strength compared to overnight drying.
3. HYP bulk measurement is affected by the number of sheet plies measured, together with the test specimen, the anvil pressure, and the measuring head surface, and, more importantly, the grammage basis of the calculation – whether it is based on oven-dry or air-dry mass.
4. It is necessary to understand the difference in brightness and whiteness as well as the difference in illuminant source and instrument geometries, especially when measuring optical properties of OBA-containing papers.
5. It is important to realize the difference between standard testing methods and their effect on HYP property measurement.

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REFERENCES

In a mill pilot trial using the FPInnovations pilot flotation column, we examined the potential of using dispersed-air flotation to purge dark pitch-laden fines selectively from four different water streams in a mill making newsprint from 100% thermomechanical pulp. Flotation removed between 21% and 52% of the total organic extractives from the paper machine excess white water, save-all cloudy white water, Uhle box filtrate, and TMP cloudy white water. Without coagulation, the flotation process removed substantial quantities of less stable pitch in the colloidal state as well as fines. Further removal of pitch from TMP cloudy white water was obtained through the addition of a coagulant before flotation. Substantial gains in the brightness of fines handsheets made from paper machine excess white water and save-all cloudy white water were observed. Decontamination of white water by flotation may lead to improved sheet properties, reduction in the use of papermaking and bleaching chemicals, improved paper machine runnability, and lower fresh water and energy requirements. Alternatively, the technology may enable the mill to increase its usage of wood with high extractives content such as pine and mountain pine beetle-attacked wood.

INTRODUCTION
Western Canadian mills are currently faced with the challenge of Mountain Pine Beetle (MPB) attack. To counter this infestation, a healthy pine strategy calls upon industry to harvest attacked and susceptible pine trees to prevent the spread of the MPB. The strategy fosters a healthy young vibrant forest to replace the older pine trees that are susceptible to attack and minimizes the fire hazards from dead standing wood. Trees under MPB attack have a limited useful life for industry due to a combination of severe checking (drying), darkening from blue stain fungus, and pitch build-up from resisting attack. MPB wood use could severely limit the ability of mechanical pulp and paper producers to manufacture a premium product. Concurrently, mills are also trying to reduce their fresh water consumption to meet environmental targets. The increased usage of green and beetle-attacked pine increases the water requirement to purge pitch and extractives adequately from the mill water system. Such purges are necessary to prevent deposition on the paper machine and maintain paper quality. Industry needs a technology to remove pitch and contaminated fines selectively in a concentrated form without the discharge of large volumes of water or quality fibre.

The Recycling Group of FPInnovations has successfully implemented three industrial flotation columns to recover fibres from deinking-plant waste streams. This technology was adapted to white-water streams, where it was shown that partial treatment of the mill white water may be sufficient to purge contaminated dark fines rich in extractives [2–5]. Indeed, the flotation process is selective for hydrophobic materials such as extractives-laden fines. Depending on the pulp furnish, these rejected fines and froths may also be
heavily contaminated with ink, synthetic polymers such as stickies, iron and aluminum, nitrogen from papermaking additives, lignin, and dyes [2–6]. Several laboratory and mill pilot trials have been performed using dispersed-air flotation to remove dark, extractives-rich fines selectively from mill white waters [2,3,5–9], which for the most part contain deinked pulp in their furnish. In 100% TMP, flotation was shown to be effective for removing extractives and colloidal pitch from pulp [10–12], but fines loss was not mentioned. We are aware of only one study using a small laboratory bench flotation cell that looked at flotation of white water from a TMP and peroxide-bleached TMP furnish. Flotation of this white water showed that the fines in the bench-top flotation cell were dark and very rich in extractives and metals such as aluminum and iron. To our knowledge, no extensive study on the use of flotation for de-resination of paper machine and TMP waters from a 100% TMP mill has yet been performed. Although the principles of white-water flotation have been proven using 15 mill water samples from different mills, to our knowledge, no mill-scale implementation of this process has yet been tried to treat large volumes of white water.

At 20 m³ of fresh water used per ton of paper, the Alberta Newsprint mill has the second highest level of water closure in Canada for a TMP newprint mill. The mill has adopted several practices to control troublesome extractives, including purging of water streams with the highest pitch content, adding fixatives at the paper machine to remove extractives with the paper, limiting the content of extractives-rich wood in the pulp furnish, and using dissolved-air flotation to remove suspended solids and dissolved and colloidal substances from white water. Despite these control measures, further reduction in fresh-water usage or further increase in the amounts of green and MPB-attacked wood used are difficult due to sticky deposition problems on the paper machine. Cleaning mill waters by dissolved-air flotation would give the mill another tool to control extractives. Unlike dissolved-air flotation, which removes all solid substances and fibre, dispersed-air flotation would limit yield loss by removing selectively only the most contaminated fines. As a first step to transferring white-water flotation from the laboratory scale to a mill-scale application, we examined one of two strategies: flotation of a severed white-water stream, or treatment of a small portion of the mill recirculated white water. In the first strategy, flotation would enable recuperation of valuable fibre, fresh water, and energy from severed streams such as TMP purge water or the water removed from the paper machine wire under vacuum. By eliminating a proportion of dark, extractives-laden fines, the second strategy would lead to chemical savings in bleaching, extractives treatment, and retention aids for papermaking. Alternatively, efficient pitch removal could enable the mill to increase its use of inferior fibre (green and beetle-attacked pine) and still make premium product with less usage of chemicals, water, and energy.

As a first step in transferring white-water flotation as a cleaning process for mill waters, we installed a 40-L pilot flotation column at Alberta Newsprint Company to measure the response of different mill streams to dispersed-air flotation. Of the four mill waters evaluated, two streams are currently sewered, and two streams are continuously recirculated. One of the severed streams acts as a purge to remove high amounts of extractives, thereby lowering extractives levels at the paper machine. Because this stream was not exposed to fixatives, the effect of adding fixatives to this TMP purge on flotation was measured. These evaluations will enable the mill to evaluate the benefits of flotation treatment and to select a stream for a larger pilot-scale demonstration flotation column.

### TABLE 1 Pilot column flotation parameters for each mill water.

<table>
<thead>
<tr>
<th>Flotation Parameters</th>
<th>Paper Machine Whitewater</th>
<th>TMP Whitewater</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flotation Time, min</td>
<td>Excess 0 - 60</td>
<td>0 - 60</td>
</tr>
<tr>
<td>Air Content, %</td>
<td>11 - 14</td>
<td>20 - 24</td>
</tr>
<tr>
<td>Froth Height, cm</td>
<td>15</td>
<td>15</td>
</tr>
<tr>
<td>Bubble Diameter, mm</td>
<td>0.3 - 1.5</td>
<td>0.3 - 1.5</td>
</tr>
<tr>
<td>Gas Velocity, cm/s</td>
<td>1.2 - 1.8</td>
<td>1.2</td>
</tr>
<tr>
<td>Accepts Velocity, cm/s</td>
<td>0.4 - 2</td>
<td>0.3 - 2.1</td>
</tr>
<tr>
<td>Temperature, °C</td>
<td>35.0 - 52.1</td>
<td>35.5 - 51.7</td>
</tr>
</tbody>
</table>

### TABLE 2 Characteristics of mill waters.

<table>
<thead>
<tr>
<th>Consistency, %</th>
<th>Paper Machine Whitewater</th>
<th>TMP Whitewater</th>
</tr>
</thead>
<tbody>
<tr>
<td>pH</td>
<td>4.7</td>
<td>6.4 - 6.6</td>
</tr>
<tr>
<td>Flow Rate, LPM</td>
<td>14 000 - 15 000</td>
<td>2 500</td>
</tr>
<tr>
<td>Temperature, °C</td>
<td>56</td>
<td>33-35</td>
</tr>
<tr>
<td>Turbidity, NTU</td>
<td>13 ± 3</td>
<td>20 ± 7</td>
</tr>
<tr>
<td>COD, mg/L</td>
<td>1 092 ± 236</td>
<td>892 ± 50</td>
</tr>
<tr>
<td>Cationic Demand, µeq/L</td>
<td>151 ± 18</td>
<td>102 ± 4</td>
</tr>
</tbody>
</table>

| Consistency, %        | Excess 0.73 - 0.84       | 0.24 - 0.30   |
| pH                    | 4.7                      | 6.4 - 6.6     |
| Flow Rate, LPM        | 14 000 - 15 000          | 2 500         |
| Temperature, °C       | 56                       | 33-35         |
| Turbidity, NTU        | 13 ± 3                   | 20 ± 7        |
| COD, mg/L             | 1 092 ± 236              | 892 ± 50      |
| Cationic Demand, µeq/L| 151 ± 18                 | 102 ± 4       |
MATERIALS AND METHODS

Samples
The FPInnovations pilot flotation column [2,3,13] was temporarily installed in a 100% TMP mill to treat three paper machine (PM) waters and one TMP white water (WW): 1. PM Excess WW; 2. PM Save-all Cloudy WW; 3. PM Uhle Box Pressate; and 4. TMP Cloudy WW. The flotation trials were performed on fresh mill samples from May 13 to 25, 2010.

Handsheets to determine percentage fines, turbidity, cationic demand, pH, and chemical oxygen demand (COD) were prepared and tested within 24 h of flotation treatment and sampling. Before testing COD, cationic demand and turbidity, flotation samples were filtered on Whatman No. 1 filter paper. Colloidal pitch measurements were made on centrifugal supernatants before and after filtration. Analysis of extractives and metals content were performed on dried handsheets or freeze-dried whole pulp.

Column Flotation Conditions and Sampling
Studies of the various mill waters were performed on a pilot flotation column built at FPInnovations that is 10.2 cm in diameter and 4.65 m in height [2,3,13]. Table 1 gives the range for each of the flotation parameters used in treating each mill water. Air flow rates were kept constant at the desired air content (i.e., gas hold-up), which was controlled by varying the air velocity between 1.2 cm/s and 1.8 cm/s. The collection time in the column was varied in the range of 0 to 60 min by changing the liquid velocity from 0.3 cm/s to 2.1 cm/s while maintaining a froth height of 15 cm. Flotation times were controlled by proportionally increasing the feed and accept flow rates. Measurement of online air content and estimation of average bubble diameter have been described in previous publications [4,13]. For TMP Cloudy WW, a fixation agent or fixative, Bufloc 5376 from Buckman Laboratories, was added to the column feed tank to fix colloidal pitch to suspended solids or fibre before flotation.

Mill white waters were sampled before (feed) and after flotation (accepts, rejects, or froth). Yields and flotation losses were calculated using the flow rates and pulp consistencies of the flotation feed, accepts, and rejects. Because air was entrained in the flotation accepts and rejects, flow rates were corrected by weighing a portion of each stream over a given time.

Handsheet Preparation and Analysis
White-water consistencies were measured according to PAPTAC Standard Method D.16. Fines handsheets were prepared as described previously [14]. Samples were poured directly into the deckle of the standard British Handsheet Maker into which a Whatman No. 1 filter was placed over the standard 150-mesh screen without addition of retention aids. To prevent washing of the fines during handsheet formation, no fresh water was added to the samples in the deckle. Optical properties were measured on six layers of 1.2-g handsheets according to PAPTAC Standard Method E.1. Optical properties are expressed as the average of measurements on both the felt and screen side of the handsheets.

Chemical Analysis
Cationic Demand - Cationic demand was determined using the Mütek Particle Charge Detector (PCD-3) and an automated titrator (PCD-T03). Cationic demand was determined on 10 mL of sample filtrate using 0.001 N PolyDADMAC as the titrant.

Chemical Oxygen Demand (COD) - COD was measured on 25 mL filtrate of PM Excess WW, PM Save-all Cloudy WW, or PM Uhle Box Pressate diluted with 25 mL of distilled water or on 10 mL of TMP Cloudy WW filtrate diluted with 40 mL of distilled water. Diluted filtrates (2 mL) were transferred to HACH COD digestion vials and reacted in the HACH COD reactor at 150°C for 2 h, then cooled as indicated by HACH for the treatment of high-range COD samples. COD values were then read in the HACH DR2800 spectrophotometer and expressed as mg/L.

Colloidal Pitch - Colloidal pitch concentration was measured by spectrophotometer on TMP Cloudy WW flotation samples as previously described [15–18]. Centrifugation was performed at 3000 rpm for 15 min. Centrifugal supernatants were then filtered through a 0.2-µm filter. Concentration of colloidal pitch was measured as the difference in light attenuation of unfiltered centrifugal supernatant and of filtered centrifugal supernatant and is expressed as the light attenuation of colloidal particles measured at 525 nm.

Extractives Content of Fines Handsheets - For extractives content determinations, handsheets were freeze-dried and extracted using the Soxtec method [19] with dichloromethane as the solvent in a single extraction step. The extracts were then weighed and reported as % extracts on oven-dried pulp weight.

Metals - Aluminum, calcium, copper, iron, magnesium, manganese, and sodium metal contents were determined on handsheets of PM Excess WW and PM Save-all Cloudy WW flotation samples. For metal content, lyophilized pulps were digested with nitric and perchloric acids (wet ashing) according to PAPTAC Standard Method G.30P before metal content determination by inductively-coupled plasma spectroscopy.

Particle Size Distribution - The particle size distributions of PM Excess WW and PM Save-all Cloudy WW flotation samples were determined using the Mastersizer S Particle Size Analyzer (Malvern Instruments).

Turbidity - Turbidities, expressed as NTU, were determined on pulp filtrates using the HACH 2100N turbidimeter.

RESULTS AND DISCUSSION
To identify which of the waters from an integrated TMP newsprint mill would benefit most from dispersed-air flotation; four TMP mill waters were treated in the FPInnovations pilot flotation column: 1. PM
Excess WW; 2. PM Save-all Cloudy WW; 3. PM Uhle Box Pressate; and 4. TMP Cloudy WW. In the mill, the PM Excess WW and PM Save-all Cloudy WW are continuously recirculated, whereas the PM Uhle Box Pressate and TMP Cloudy WW are purged. After reviewing the characteristics of each mill water stream, the benefits of white-water cleaning will be described for each stream.

**Characteristics of Mill Streams**

Table 2 summarizes the characteristics of each white water before flotation without the addition of fixation chemicals. Values are expressed as ranges or mean values of 2–13 replicates with standard deviation. The flow rates are included to give the reader an idea of the quantities of white water that would need to be treated. Of particular note, the PM Save-all Cloudy WW and the PM Excess WW have high flow rates, between 13 and 15 m³/min. Of the four waters, the TMP cloudy white water has eight times higher turbidity and two times more cationic demand and COD than the paper machine white waters. The fibre content of this water is, however, the lowest.

Figure 1 compares the extractives content and brightness of a whole TMP sample to that determined from fines handsheets made from each of the PM and TMP water streams. Fines from Uhle Box Pressate contained 23 times more extractives and had brightness 38 points lower than the whole TMP fibre. The PM Excess WW and PM Save-all Cloudy WW had respectively 5.6 and 6.7 times more extractives than the whole TMP. Clearly, white-water recirculation promotes accumulation of extractives on the fines, as described in earlier studies [19]. Brightnesses of handsheets from PM Excess WW and PM Save-all WW were 3.2 brightness points lower than handsheets prepared from whole TMP. Note the 8.6 points-lower brightness of the TMP Cloudy WW...
handsheets compared to the brightness of handsheets made from whole TMP fibres, which was in the range previously reported [14].

**PM Excess WW** - An air content of 11%–15% with bubble diameters of 0.9–1.5 mm was sufficient to establish a stable froth during flotation of the PM Excess WW. Figure 2 shows that at 9 min flotation time, 21% of the extractives were removed at a flotation rate constant of 0.035 min⁻¹. The rejected froth was very sticky to the touch. The rejected fines from 3–10 min flotation time were highly contaminated, with a brightness of 20%–44% and 22%–30% extractives. At 9 min flotation time, the brightness of fines handsheets increased by 4.5 points, but with a corresponding 11.5% fibre loss, as seen in Fig. 3. These solids losses are similar to those reported for white-water flotation of a TMP/DIP mixture by Haapala et al. [6,9] for first-loop flotation. Decreasing the bubble size to 0.3–0.5 mm increased extractives removal, but also promoted a higher fibre loss, 34% for 9 min flotation time. A similar decrease in bubble diameter was observed during hyperflotation trials at an extended flotation time of 60 min.

No significant changes in cationic demand, COD, or turbidity were observed following flotation treatment. Although particle size numbers obtained by laser diffraction are not perfect because the analyzer measures the volume that a particle occupies in a suspension and assumes a spherical shape for filamentous fines, the tool can show relative changes in the particle size range of flotation samples. For the PM Excess WW, the average size of the rejected fines was 10–17 μm, which was 2–2.5 μm smaller than the average size range of the fines from flotation feed and accepts. Because smaller fines have a higher surface area, they often contain more extractives and as such are more hydrophobic. Both hydrophobicity and particle size influence flotation efficiency. Similarly, these rejected or smaller fines also contain more nitrogen from retention aids, lignin, and metals [5,19]. Rejected fines from PM Excess WW flotation contained 2242 mg/kg of calcium, 10.2 mg/kg of copper, 45.8 mg/kg of iron, 55.2 mg/kg of manganese, and 56.5 mg/kg of aluminum, amounts that were 1.6, 1.8, 2.4, 1.1, and 2.7 greater respectively than in flotation accepts. There were no significant changes in mean levels of sodium (210.3 mg/kg), magnesium (186.7 mg/kg), and manganese (53.5 mg/kg) for fines handsheets made from flotation feed, accepts, and rejects.

Figure 4 shows a linear increase in brightness and linear removal of blue coloration (b*), where the quantity of extractives removed is dependent on the amount of fines removed (Fig. 3). The b* values decreased in all cases but remained in the positive range, from 2.9 to 5.9 for the flotation accepts and 4.9 to 6.5 for the flotation feed. Brightness loss is related to the darker color of native fines when compared to longer fibres and their level of contamination through white-water recirculation [14,19,20]. Because of their high surface area and their greater level of recirculation in mill waters, the fines have higher pitch, dye, lignin, and metal contents [5,14,19–21]. Heavy metal ions, like copper and iron, could accelerate thermal darkening of mechanical pulp to form thermal complexes with lignin and phenol compounds [20]. Monitoring the extractives content of fines is a tedious and lengthy process that involves the preparation of fines handsheets followed by solvent extraction. This process, although necessary to understand white-water flotation, would be difficult to perform in a mill setting. With the ready availability of brightness sensors and the establishment of a curve such as that shown in Fig. 4, the extractives loss could be estimated. This relationship will be mill-dependent because it will be influenced by the pulp furnish, the level of white-water recirculation, and the type of dye used in the furnish.

**PM Save-all Cloudy WW** - With air contents of 13%–18% and bubble diameters of 0.7–1.5 mm, a stable froth was
established during PM Save-all Cloudy WW flotation, similarly to that seen for the PM Excess WW. Figure 5 shows that at 9 min flotation time, 52% of the extractives were removed, and the brightness of fines handsheets was increased by 4.8 points. At 3–10 min flotation times, rejected solids had a brightness of 21%–34% and contained 23%–29% extractives. Fibre loss at 9 min flotation time was 18%. At 0.15 min⁻¹, the flotation rate constant at 9 min was 4.3 times higher for the PM Save-all Cloudy WW than for that of the PM Excess WW. Although the effect of flotation feed consistency was not measured in this trial, the 2.9 times lower consistency of the PM Save-all Cloudy WW may be one of the reasons that the flotation rate constant was higher than that of the PM Excess WW.

No significant changes in cationic demand, COD, or turbidity were observed following flotation treatment. Rejected fines contained 2012 mg/kg of calcium, 27.2 mg/kg of copper, 96.0 mg/kg of iron, and 50.3 mg/kg of aluminum, amounts that were 1.6, 3.1, 3.4 and 2.5 times greater respectively than in the flotation accepts. There were no significant changes in mean levels of sodium (189.2 mg/kg), magnesium (210.0 mg/kg), and manganese (40.9 mg/kg) for fines handsheets made from flotation feed, accepts, and rejects.

Using the scale-up procedures proposed by Hernández-Alverez, Finch, and Dobby [22,23] for a one-loop flotation system treating 21% or 2800 LPM of the PM Save-all Cloudy WW, we estimated the flotation losses at flotation times of 6.5–7 min for a column with a diameter of 2.4 m and a width of 7.78 m. With a 15% flotation loss, the mill would remove 0.30–0.35 t/d in extractives from its white-water circuits with an associated loss of 1.1–1.2 t/d loss of contaminated fines. Solids and water removal rates can be controlled by adjusting the residence time of feed and accepts flow rates, air content, and bubble diameter.

**PM Uhle Box Pressate** - The fines of the PM Uhle Box Pressate were dark and contained the highest proportion of extractives of all the mill waters tested. Although this stream has 5–6 times lower flow rates than the other two PM streams, it was thought that if the flotation treatment could remove the extractives, the mill could save on fresh water and energy. Figure 6 shows that a flotation treatment of 9 min removed 48% of the extractives and improved the brightness of fines handsheets by 20 points. At a rate of 0.375 min⁻¹, the flotation rate constant was more than double that observed for the PM Save-all Cloudy WW. Air content levels of 20%–24% with bubble diameters of 0.5–0.7 mm were obtained at air velocities of 1.2–1.6 cm/s. No significant changes in cationic demand or COD were noted; however, the turbidity decreased by 21%–48% during periods of 3–15 min flotation times. Although flotation reduced the extractives content of the fines from 25% w/w in the flotation feed to 13% w/w extractives remaining on the accepted fines, the end result would be further contamination of the PM white water because these levels of extractives in the flotation accepts are still higher than those in the PM Save-all Cloudy WW and the PM Excess WW. A dissolved-air flotation treatment in which all the solids are removed would be a better strategy if the mill wished to recuperate this water.

**TMP Cloudy WW** - As with the PM Uhle Box Pressate, the mill could also recuperate non-contaminated fines from TMP Cloudy WW, which is typically sewered. The TMP Cloudy WW has high levels of COD, cationic demand, turbidity (Table 2), and colloidal pitch. By fixing the colloidal pitch onto the fines before flotation, the flotation treatment may be made more effective. To assess whether dispersed-air flotation could be used to clean this water loop of colloidal pitch effectively, the colloidal pitch in TMP Cloudy WW was measured before and after flotation with the addition of a polymeric cationic fixative.

A first series of experiments with this water kept the flotation time constant at 10 min with increasing fixative dosage.
from 0–110 ppm. No adjustments were made to the air and liquid flow rates or to froth height. Figure 7 shows that as the fixative dosage increased, the air content in the column rose from 16% to 24%. Bubble diameter simultaneously shrunk from 1.5 mm to 0.7 mm over the same fixative dosage change. Although the increase in gas hold-up was linear, the extracts removed increased from 21% to 68% with increasing fixative concentration, but attained a plateau at 40–75 ppm of fixative. Brightness of fines handsheets increased from 48.3 to 51.4 points with no fixative and to 52.4 points with 15 ppm of fixative. Fibre and water losses also rose with increasing fixative concentration to 54% and 57% respectively. Because the air and liquid flows were kept constant, the flotation losses at high fixative doses were large. In a controlled flotation process, the air flow would have been reduced and the froth height controlled to limit fibre and water loss.

Figure 8 shows colloidal pitch removal before and after flotation. The selectivity of the flotation process in terms of removal of colloidal pitch was greatest with the lowest amount of fixative. With no addition of fixative, the water and fibre loss were approximately 10%, the colloidal pitch concentration in the accepts decreased by 23%, and the amount in the rejects increased by 65%. When fixative is added, the amount of colloidal pitch decreases before flotation because the pitch becomes fixed to the suspended solids. The enrichment of the rejects with colloidal pitch is marginal and potentially not worth the substantial water and fibre losses, as shown in Fig. 7.

The relative differences in the concentration of colloidal pitch in samples taken before and after flotation is shown in Fig. 8. In these experiments the decrease in colloidal pitch may involve coagulation, agglomeration, and fixation of colloidal pitch on fines and fibre or direct removal by flotation of colloids. A comparison of the removal processes from suspension is shown in Fig. 9. Note that fixation removes 66% of the colloidal pitch from the water fraction through precipitation, deposition on the fines, or both. Up to 33% of the colloidal pitch is removed by the flotation process alone without use of fixative. The solid black bar represents the additive effect of both flotation and fixation on the removal of these troublesome colloids from TMP Cloudy WW. When extractives removal is compared to the fibre and water losses for this experiment, it becomes clear that the flotation becomes less selective with addition of cationic polymeric fixative. The results indicate that a small amount of fixative or coagulant (7.5 to 15 ppm) may enhance colloidal pitch removal by flotation. Some agglomeration, coagulation, and fixation on high surface-area fines is probably beneficial, but substantial fixation appears to lead to other problems that decrease selectivity, as shown in Fig. 7.

Unlike paper machine waters that have been exposed to cationic fixatives and retention aids, the cationic demand, turbidity, and COD values are high in TMP Cloudy WW. Figure 10 shows that values of cationic demand, turbidity, and COD decrease linearly with increasing fixative addition. With the addition of 75 ppm of fixative, the extractives content of the fines increased from 4.7% to 7.5% w/w, with a simultaneous drop of 55% in the turbidity from 117 NTU to 53 NTU. With 15 ppm of fixative, the COD dropped by 10%, the cationic demand by 8%, and the turbidity by 31%.

Overall, the results indicate that if TMP purging were carried out in combination with dispersed-air flotation, the concentration of colloidal pitch could be decreased by 25%–45% in TMP white water. In addition, the brightness of the fines retrieved would be substantially improved, by 4.1 points, and the removal of DCM extractives with the fines could reach 52%. Raising the fixative concentration to 15 ppm resulted in an additional 31% w/w of DCM extractives being deposited on the fines. Flotation of the TMP Cloudy WW could lead to a substantial reduction in the amount of water purged. If this water were not sewered, the impact of added COD, cationic demand, and turbidity on paper machine operations could be considerable.
CONCLUSIONS

To our knowledge, this is the first pilot flotation trial to treat mill white-water streams from a 100% TMP mill. In all four streams tested, dispersed-air flotation effectively cleaned mill TMP and paper machine white-water streams of dark and extractives-rich fines. Flotation removed between 21% and 52% of total organic extractives from the paper machine excess white water, save-all cloudy white water, Uhle box filtrate, and TMP cloudy white water. Substantial gains in the brightness of fines handsheets made from flotation accepts of all waters were observed.

Although flotation improved the quality of all waters, the two sewered waters still contained contaminants. For example, even though flotation removed 48% of the extractives from the PM Uhle Pressate, because the pressate feed contained 2.7 times more extractives than the PM Excess WW and the PM Save-all Cloudy WW, the resulting flotation accepts would contaminate recirculated paper machine waters instead of cleaning them. In the TMP Cloudy WW, the flotation process removed substantial quantities of less stable pitch as well as fines without the addition of a fixative. Further removals of pitch from TMP cloudy white water were obtained through the addition of a low dosage of a fixative before flotation. Higher dosages of fixative reduced the selectivity of dispersed-air flotation for hydrophobic material, resulting in high fibre and water losses. Although some of these losses could be reduced through control of the flotation process, the cationic demand and COD of the TMP Cloudy WW accepts still remains high. For these reasons, the mill has decided to treat the PM Save-all Cloudy WW as the first candidate for flotation in the pilot-scale industrial demonstration of the technology.

Decontamination of white water by flotation may lead to improved sheet properties, reduction in the use of papermaking and bleaching chemicals, improved paper machine runnability, and lower fresh water and energy requirements. Alternatively, the technology may enable the mill to increase its usage of wood with high extractives content such as pine and mountain pine beetle-attacked wood.

ACKNOWLEDGEMENTS

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REFERENCES

Due to the high costs of natural gas and fuel oil in recent years, many Kraft pulp mills now use lower-cost fuels with high sulphur content in their lime kilns. Depending on the total sulphur input to the kiln, burning high-sulphur fuels in lime kilns may increase SO$_2$ emissions, decrease lime availability and reactivity, and aggravate ring formation. The sulphur absorbed in the product lime ends up as Na$_2$SO$_4$ deadload in the liquor cycle, which is reduced to Na$_2$S in the recovery boiler, leading to an increase in liquor sulphidity. These effects, however, are manageable.

**ABSTRACT**

Due to the high costs of natural gas and fuel oil in recent years, many Kraft pulp mills now use lower-cost fuels with high sulphur content in their lime kilns. Depending on the total sulphur input to the kiln, burning high-sulphur fuels in lime kilns may increase SO$_2$ emissions, decrease lime availability and reactivity, and aggravate ring formation. The sulphur absorbed in the product lime ends up as Na$_2$SO$_4$ deadload in the liquor cycle, which is reduced to Na$_2$S in the recovery boiler, leading to an increase in liquor sulphidity. These effects, however, are manageable.

**INTRODUCTION**

In kraft pulp mills, lime kilns are used to convert lime mud (CaCO$_3$) to lime (CaO) for reuse in the causticizing plant. The process requires a large amount of heat (6–10 GJ/t CaO production), which is supplied to the kiln mainly by burning fossil fuels such as fuel oil and natural gas. Due to high energy costs in recent years, there has been a growing interest in burning alternative fuels in lime kilns, such as petroleum coke (petcoke), wood residue-based biofuels (directly fired, gasified, or pyrolysed), and precipitated lignin [1].

The composition, heating value, and adiabatic flame temperature of fuel alternatives can significantly affect lime kiln operations. The fuel sulphur content is particularly important because it influences lime availability, ring formation, and SO$_2$ emissions. Table 1 shows the sulphur content and heating value of traditional and alternative kiln fuels. In addition to these fuels, waste streams that are commonly burned in lime kilns also contain sulphur, such as non-condensable gases (NCGs), stripper off-gases (SOGs), and tall oil. Although burning sulphur-containing fuels and waste streams in lime kilns is a common practice, the fate of fuel sulphur and its impact on lime availability, ring formation, and emissions is not well understood.

In this paper, literature information and a sulphur balance model were used to gain a better understanding of the fate of fuel sulphur and the impacts of burning high-sulphur fuels on lime kiln and chemical recovery operations.

**FATE OF FUEL SULPHUR IN LIME KILNS**

When a sulphur-containing fuel is combusted in a lime kiln, the sulphur is oxidized to sulphur dioxide (SO$_2$):

\[ \text{S} + \text{O}_2 \rightarrow \text{SO}_2 \]  

[L reaction 1]

Lime-based sorbents, such as limestone and dolomite, are widely used in fluidized-bed combustion of sulphur-containing fuels to control SO$_2$ emissions [3] because they absorb SO$_2$ through sulphation reactions. In lime kilns, product lime is known to react with the majority of SO$_2$ and O$_2$ to form CaSO$_4$ and Na$_2$SO$_4$ [4], and the remaining SO$_2$ that is not absorbed flows out of the kiln with the flue gases.

**CaSO$_4$ Formation**

Sulphur dioxide in the kiln flue gas can be absorbed by calcium oxide (CaO) through sulphation reactions between CaO in lime and SO$_2$ in kiln flue gas to form calcium sulphate (CaSO$_4$):

\[ \text{CaO} + \text{SO}_2 + \frac{1}{2} \text{O}_2 \rightarrow \text{CaSO}_4 \]  

[L reaction 2]

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Although the calcination reaction of lime mud ($\text{CaCO}_3 \rightarrow \text{CaO} + \text{CO}_2$) occurs at approximately 800°C, the solids in the kiln are typically heated to a higher temperature to achieve agglomeration and an acceptably large size of lime nodules [5]. The sulphation reaction (Reaction 2) occurs in a narrow temperature range between 900–1200°C. The reaction rate is low below 900°C, but increases markedly with temperature, reaching a maximum at approximately 1100°C. Above 1200°C, the reaction reverses due to the thermal instability of $\text{CaSO}_4$ [6–8]. Sulphation is a reaction that commonly occurs on the surface of ring deposits, and the above kinetic observations are consistent with mill experience that shows that ring deposits usually have the highest sulphur content in a narrow region close to the front end of the kiln [4].

The sulphation of CaO is a gas-solid reaction and is often described using the unreacted core model, which is a sulphation pattern leading to the formation of particles with sulphated rims and unsulphated centres. Sulphur absorption by a lime particle may occur in two stages. In the first stage, $\text{SO}_2$ and $\text{O}_2$ diffuse through the pores of the particle and react with CaO at the solid-gas interface. The second stage begins once a $\text{CaSO}_4$ layer has formed on the particle surface, limiting the diffusion of $\text{SO}_2$ and $\text{O}_2$ through the layer. Eventually, diffusion becomes so slow that the sulphation process is considered to have stopped [3].

In lime kilns, when sulphur-containing fuels are burned, the resulting $\text{SO}_2$ reacts with $\text{O}_2$ and CaO to form a $\text{CaSO}_4$ layer on the surface of lime particles, as shown schematically in Fig. 1. This $\text{CaSO}_4$ layer acts as a barrier, preventing $\text{SO}_2$ and $\text{O}_2$ from further reacting with CaO. Eventually the sulphation process stops, leaving an unreacted core in the centre of the lime nodule. Therefore, $\text{SO}_2$ emissions from lime kilns are possible even though there is more than enough CaO to bind with all the $\text{SO}_2$ present. In addition, the lack of prolonged, intimate contact between the flue gas and the lime nodules and the relatively large lime nodule size further prevent the lime from reacting with $\text{SO}_2$.

**$\text{Na}_2\text{SO}_4$ Formation**

Sodium enters the kiln with residual white liquor in lime mud in small amounts, typically < 1.2 wt% $\text{Na}_2\text{O}$ or 0.9 wt% Na. It occurs mainly in the form of $\text{NaOH}$, $\text{Na}_2\text{S}$, $\text{Na}_2\text{CO}_3$, $\text{Na}_2\text{SO}_4$, and sodium silicates. Of these compounds, only $\text{NaOH}$ and $\text{Na}_2\text{CO}_3$ can form $\text{Na}_2\text{SO}_4$ directly upon reacting with $\text{SO}_2$ and $\text{O}_2$ (Reactions 3 and 4):

\[
2 \text{NaOH} + \text{SO}_2 + \frac{1}{2} \text{O}_2 \rightarrow \text{Na}_2\text{SO}_4 + \text{H}_2\text{O} \quad \text{[Reaction 3]}
\]

\[
\text{Na}_2\text{CO}_3 + \text{SO}_2 + \frac{1}{2} \text{O}_2 \rightarrow \text{Na}_2\text{SO}_4 + \text{CO}_2 \quad \text{[Reaction 4]}
\]

A small portion of $\text{Na}_2\text{S}$ may also indirectly capture $\text{SO}_2$ by first releasing its white liquor $\text{S}$ to form $\text{H}_2\text{S}$ and $\text{Na}_2\text{CO}_3$ in the cold end of the kiln (Reaction 5); the resulting $\text{Na}_2\text{CO}_3$ then reacts with $\text{SO}_2$ (from fuel $\text{S}$) and $\text{O}_2$ in the hot end of the kiln (Reaction 4):

\[
2 \text{Na}_2\text{S} + \text{CO}_2 + \text{H}_2\text{O} \rightarrow \text{Na}_2\text{CO}_3 + \text{H}_2\text{S} \quad \text{[Reaction 5]}
\]

Although the total amount of Na compounds is small, they are capable of reacting with up to ~5 kg S per ton of CaO. This is the equivalent of absorbing all the $\text{SO}_2$ produced from co-firing pet coke with fuel oil in a lime kiln at ~20% substitution.

**LIME KILN SULPHUR BALANCE**

A sulphur balance model was developed using a commercial heat and mass balance computer program to gain a better understanding of the fate of fuel sulphur in lime kilns. The model incorporated empirical correlations between the sulphur (S) absorption efficiency, $\text{SO}_2$ emissions, and the total S input derived from a field study on the effect of burning concentrated NCGs on the performance of an oil-fired kiln at a Brazilian kraft pulp mill [9]. In that study, the sulphur absorption efficiency was defined as:

\[
\text{S Absorption Efficiency} = \frac{\text{S Leaving Kiln with Lime}}{\text{Total S Input to Kiln}} \times 100\%
\]

**Fig. 1 - Formation of $\text{CaSO}_4$ layer on the surface of a lime particle [9].**

**Fig. 2 - Effect of sulphur input on S absorption efficiency and SO2 emissions at a Brazilian mill [9].**
As shown in Fig. 2, the efficiency was 98.2% when the S input was 6.3 kg/t CaO, but decreased to 88.7% when the total S in the fuel was 17.9 kg/t CaO. The high S absorption efficiency at low S inputs implies that most of the S entering the kiln with the fuels was absorbed by the lime and the lime dust. These correlations enable the amount of fuel sulphur in the product lime and SO₂ emissions to be calculated.

The model was run for six different cases of varying levels of petcoke substitution: 0, 20, 40, 60, 80, and 100% TL (kiln thermal load) provided by petcoke. These cases cover a fuel sulphur input range of 3.77–12.75 kg S/t CaO.

**IMPACTS ON LIME KILN OPERATIONS**

In general, the major impacts of high-sulphur fuel use on lime kiln operations are felt in three main areas: lime availability and make-up requirements, air emissions and compliance, and ring formation. It is important to note that “high” sulphur content as referred to in this paper is only relative to the original fuel being burned in the kiln. A noticeable increase in SO₂ concentration in the kiln will occur only if the new fuel contains significantly more sulphur than the fuel that was previously burned. As shown in Table 1, petcoke (6.5 wt% S) has a high sulphur content relative to both natural gas and fuel oil, while precipitated lignin has a sulphur content ranging between 1–3 wt% [2], which is about the same as fuel oil but higher than natural gas.

**Lime Availability and Make-Up Requirements**

When high-sulphur fuels are burned in the kiln, the absorption of sulphur through the sulphation of CaO inevitably leads to a decrease in lime availability. Modelling results (Fig. 4) show that the use of 100% petcoke in the lime kiln can reduce lime availability by 3%. To demonstrate the extent of fuel sulphur absorption by lime, lime availability based on modelling results is compared to two hypothetical cases in Fig. 4. In this graph, three curves are plotted: one derived from the modelling results and two hypothetical curves representing respectively no fuel sulphur absorption by lime and complete fuel sulphur absorption by lime. These two hypothetical curves represent the estimated upper and lower limits of the possible changes in lime availability. The proximity of the modelling curve to the complete sulphur absorption curve shows that most of the fuel sulphur is absorbed by lime, particularly at low petcoke substitutions.

The decrease in lime availability due to sulphur absorption will lead to a higher make-up lime requirement. However, it should be noted that in the slaker and causticizers, the calcium in CaSO₄ ends up as CaCO₃, which is lime mud (Reaction 5). To understand how this influences the make-up lime requirement, it is useful to consider a simplified analysis of the kiln on a 1 mol CaO basis. Before the use of petcoke, 1 mol of CaCO₃ is required to produce 1 mol of CaO, as shown in Fig. 5.

At 100% petcoke substitution, 1 mol of CaO is produced along with approximately 0.024 mol of CaSO₄, as shown in Fig. 6. This means that 1.024 mol of CaCO₃ is required to produce 1 mol of CaO and 0.024 mol of CaSO₄. This leads to a higher mud load to the kiln (up to 2.4%). Initially, there will be a transient period where the make-up lime will need to be increased to account for the calcium being consumed and converted to CaSO₄. Eventually, however, the make-up requirement will level off because the Ca in the CaSO₄ returns to the kiln as CaCO₃ after going through the causticizing plant. There will be no build-up of sulphur in the lime mud because the sulphate in CaSO₄ exits the causticizing plant as soluble Na₂SO₄ and does not return to the kiln with the lime mud. This is in agreement with S/Ca ratio measurements in lime mud and lime from a kiln burning 70% petcoke (Fig. 7), where the S/Ca molar ratio of mud is significantly lower than that of lime.

**Air Emissions and Compliance**

SO₂ emissions may increase with the use of high-sulphur fuels depending on the

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Fig. 3 - Model results: lime availability versus petcoke (6.5 wt%S) substitution.

Fig. 4 - Comparison of lime availability based on modelling results and two hypothetical situations: no fuel sulphur absorption and complete fuel sulphur absorption.
total input of sulphur to the kiln and whether the kiln is equipped with a flue gas scrubber or an electrostatic precipitator. Based on the modelling results (Fig. 8), there should be a small increase in SO$_2$ emissions for kilns that burn petcoke at low substitution rates, below 40% (i.e., fuel sulphur input less than 6 kg S/t CaO). For kilns that are equipped with flue gas scrubbers, SO$_2$ emissions are usually a non-issue because most of the remaining SO$_2$ in the flue gas is removed by the scrubber. On the other hand, for kilns that burn petcoke at higher substitution rates, or petcoke together with concentrated NCGs (CNCGs) and SOGs, high SO$_2$ emissions are inevitable, particularly for kilns without scrubbers.

Figure 9 shows SO$_2$ concentration in the kiln gas as a function of fuel oil substitution and total sulphur in the alternative fuel. The calculations were based on the same lower heating value of 34 MJ/kg for the alternative fuel. As expected, for a given fuel substitution level, burning alternative fuels with higher sulphur contents results in higher SO$_2$ emissions. The SO$_2$ concentration rises more steeply with increased substitution for high sulphur-containing fuels. Similar results were obtained for the replacement of natural gas by an alternative fuel (Fig. 10), although the SO$_2$ emissions were much lower because natural gas contains no sulphur.

TRS emissions are usually not an issue when burning high-sulphur fuels as long as adequate excess O$_2$ is provided and good burner performance is maintained.

**Ring Formation**

Ring formation is normally due to the presence of “sticky” molten sodium compounds in lime that encourage the adhesion of material on kiln walls, while ring growth is mostly driven by recarbonation reactions [4,7]. The burning of high-sulphur fuels may aggravate ringing because the sulphation of CaO can make ring deposits hard and more resistant to the abrasion caused by solids movement in the kiln. However, due to the narrow temperature range of the sulphation reaction (900–1200°C), ring deposits usually have the highest S content in a narrow region close to the front end of the kiln [4].

In the example of the mill burning 70% petcoke (Fig. 7), the ring deposits have a much higher S/Ca molar ratio than reburned lime and lime mud. The standard deviation is relatively large for the ring deposit data because the S content depends greatly on where the ring is located and from where within the ring the sample is taken. Nonetheless, the
average S/Ca molar ratio of 0.03 in this case means that the ring deposits contained only 1.6 wt% S (or 6.8 wt% CaSO\(_4\)), an amount too small to be the main cause of ringing. In kilns that burn pet coke, if ringing is an issue, it is more likely caused by flame instability that leads to wide temperature variation and ring growth through recarbonation [7] than by the high sulphur content.

### IMPACTS ON CHEMICAL RECOVERY OPERATIONS

Product lime, which contains absorbed sulphur in the form of CaSO\(_4\) and Na\(_2\)SO\(_4\), exits the lime kiln and enters the slakers, where it reacts with green liquor. For large lime nodules, particularly “dead-burned” lime, the retention time in the slaker is insufficient to ensure full break-up and causticization. As a result, large nodules tend to settle at the bottom of the slaker and are then removed as grits. If these nodules are coated in a hard layer of CaSO\(_4\), then there will be some sulphur removal from the cycle in the form of grits. Another place in the causticizing plant where sulphur is removed is with the dregs from the green liquor clarifier. Dregs may contain residual sodium from the green liquor.

The slaking and causticizing reactions cause lime nodules to break up into smaller particles and to release CaSO\(_4\) into the liquor. The “released” CaSO\(_4\) reacts rapidly with sodium carbonate (Na\(_2\)CO\(_3\)) in the liquor to form calcium carbonate (CaCO\(_3\)) and sodium sulphate (Na\(_2\)SO\(_4\)):

\[
\text{CaSO}_4 + \text{Na}_2\text{CO}_3 \rightarrow \text{CaCO}_3 + \text{Na}_2\text{SO}_4
\]

[Reaction 6]

The precipitated CaCO\(_3\) is essentially lime mud, and therefore there is an increase in the mud load to the causticizing plant and lime kiln. Na\(_2\)SO\(_4\), on the other hand, is water-soluble and ends up leaving the causticizing plant with the white liquor, becoming part of the sulphate deadload in the liquor cycle. Na\(_2\)SO\(_4\) eventually enters the recovery boiler, where most of it is reduced to Na\(_2\)S. This process inevitably results in an increase in liquor sulphidity if the NaOH concentration in the liquor is not adjusted. The fate of fuel sulphur and the resulting sulphate deadload is illustrated in Fig. 11.

It is difficult to estimate how high the liquor sulphidity may become and how much sodium make-up may be required as a result of burning high sulphur-containing fuel in the lime kiln. These effects depend on many factors, including the
sulphur absorption efficiency of the kiln, the kinetics of Reaction 6, the amounts of grits, dregs, and lime dust being disposed of, and the amount of sulphur loss through $\text{SO}_2$ and particulate emissions from the recovery boiler and lime kiln stacks. Mills may choose either to operate at a higher sulphidity or to try to maintain the same sulphidity through lime dust and recovery boiler precipitator dust purges. A combination of the two options is probably the best solution. Figure 11 shows the possible sulphur purge points from the chemical recovery cycle.

**SUMMARY**

Burning high-sulphur fuels affects both lime kiln and chemical recovery operations. Fuel sulphur can be absorbed in the kiln by $\text{CaO}$ and by sodium compounds in the lime. Depending on fuel sulphur content, kiln operating conditions, and lime mud characteristics, a portion of the $\text{SO}_2$ may pass through the kiln without reacting and contribute to increased $\text{SO}_2$ emissions. The absorbed sulphur compounds in the product lime end up as $\text{Na}_2\text{SO}_4$ deadload in the liquor cycle. Most of the $\text{Na}_2\text{SO}_4$ is eventually reduced to $\text{Na}_2\text{S}$ in the recovery boiler, leading to an increase in liquor sulphidity if Na input is not adjusted.

Increasing the fuel sulphur input to the kiln would also result in lower lime availability (3% maximum) and hence a higher make-up lime requirement and the formation of hard rings in the kiln. These effects, however, are all manageable.

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**REFERENCES**

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