The effect of varying air injection rates on tall oil soap skimming efficiency for low fatty acid/resin acid ratio soaps produced by pulping mountain pine beetle-infected wood

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Abstract

Trials on the evaporator soap skimmer at a British Columbia Interior mill were conducted to assess the impact of higher air injection rates on skimmer performance. Those tests indicated that the mill now has very poor skimming efficiencies: only 13%–38% of the total soap in the feed liquor was skimmed for low acid number (114–118) and low fatty acid/resin acid ratio (0.73–0.83) soap. In addition, there were extended periods over the two-day trial when soap skimming stopped completely and the soap levels in the skimmed liquor equalled or exceeded those in the feed liquor. Higher air injection rates were observed to have little beneficial impact on soap-skimming efficiency. As expected, the higher air injection rates decreased the soap density by about 8% and increased black liquor entrainment in the skimmed soap by nearly 35%. As high percentages of black liquor can cause high H_2S emissions during soap acidulation, and the precipitated lignin can hinder tall oil separation, the use of higher air-injection rates cannot be recommended.

Keywords: mountain pine beetle, lodgepole pine, Kraft mills, wood extractives, resin acids, fatty acids, soap solubility, soap density, skimming efficiency, acid number

Résumé

Des essais sur l’évaporateur du récupérateur à une scierie de l’intérieur de la Colombie-Britannique ont été réalisés, afin d’évaluer l’incidence des taux plus élevés d’injection d’air sur le rendement du récupérateur. Ces essais ont indiqué que les taux d’efficience du récupérateur de la scierie sont actuellement très faibles : de 13 % à 38 % seulement de la quantité totale de savon dans la liqueur d’alimentation était récupérée pour un indice d’acidité faible (114 à 118), un savon dont le rapport acide gras/acide résinique est faible. Il y a eu, en outre, des périodes prolongées au cours de l’essai de deux jours quand la récupération de savon s’est arrêtée complètement et que les niveaux de savon dans la liqueur « récupérée » étaient égaux ou plus élevés que ceux de la liqueur d’alimentation. On a observé que des taux d’injection d’air plus élevés n’avaient que peu d’incidence bénéfique sur l’efficacité de la récupération de savon. Comme prévu, les taux plus élevés d’injection d’air ont réduit la densité du savon d’environ 8 % et augmenté l’entraînement de la liqueur résiduaire dans le savon récupéré de près de 35 %. Comme les pourcentages élevés de liqueur résiduaire peuvent causer des émissions élevées de H_2S au cours de l’acidulation du savon, et que la lignine précipitée peut empêcher la séparation du tallöl, l’utilisation de taux d’injection d’air plus élevés n’est pas recommandée.

Mots clés : dendroctone du pin ponderosa, pin tordu latifolié, fabriques de pâte kraft, produits d’extraction du bois, acides résiniques, acides gras, solubilité du savon, densité du savon, efficacité de la récupération, indice d’acidité

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1. INTRODUCTION

Kraft pulping of softwoods converts the free resin acids, fatty acids, and much of the triglycerides in wood into their sodium salts or *soaps*. Associated with the salts are neutral or *unsaponifiable* compounds, such as sterols. The salts and unsaponifiable compounds separate from the spent cooking liquor as *black liquor soap*. Wood species, dissolved solids content, and the residual effective alkalinity (NaOH + ½ Na_2S) in the black liquor can influence this separation (Foran 1992; Uloth et al. 1987).

The recovered soap is either burnt in the recovery boiler or converted into a blend of fatty and resin acids and neutrals, known as *crude tall oil*, through acidulation in a tall oil plant. About 12 kraft mills in Canada process recovered soap to produce 75,000 tonnes of crude tall oil (CTO) per year (Norman 1982). Although Canadian tall oil is generally of much lower quality than that from southern US kraft mills (Norman 1982) and is often difficult to market, tall oil soap should be recovered to minimise its adverse affects on the unit operations in the kraft recovery system (Uloth and Wong 1986). Tall oil also has a high heating value, as high as 37.9 MJ/kg of dry solids (16,300 BTU/lb), and many mills now burn tall oil in their lime kilns to offset much of their purchased fossil fuel (Young 1989). With natural gas selling for just over $8/GJ, the fuel value of tall oil is close to $300/tonne, even before considering potential emission reduction credits for burning a renewable fuel.

Recent soap solubility tests of black liquor and soap from four BC mills, and one Alberta mill pulping very little beetle-killed wood, indicate that mills pulping beetle-infested wood may drop their tall oil production substantially for three reasons: higher soap solubility in black liquor, a reduced tendency for the soap to "float" off in storage tanks and skimmers, and lower soap acid numbers (Uloth et al. 2007). The minimum soap solubility in the BC mills has increased from 6.0–8.5 kg per tonne of dry black liquor solids (kg/tDBLS) in the mid-1980s, to 10.2–12.7 kg/tDBLS. The changes in soap solubility and buoyancy are largely due to changes in the fatty acid (FA) and resin acid (RA) content of the wood in response to beetle attack and subsequent fungus infestation. The FA/RA ratio in tall oil produced from soap skimmed in the mills pulping beetle-infested wood has dropped from 1.49–1.86 in the mid 1980s, to 0.72–1.28. This has shifted the soap solubility versus black liquor solids curves up and to the right. The solids concentrations at which soap solubility is minimised are now consistently in the 35%–49% solids range. In the 17%–32% solids range, where most BC mills try to skim soap, soap solubility increased significantly and now generally ranges from 10.5–23 kg per tonne of dry liquor solids. The tall oil soap concentrations in weak black liquor samples from these mills ranged from 11.6–32.6 kg/tDBLS, and did not greatly exceed the solubility limit in the 17%–32% solids concentration range. Soap could, however, be expected to precipitate at higher solids concentrations, aggravating evaporator and concentrator scaling problems, and accumulate in strong liquor storage tanks where it could cause further operating problems and lead to unstable recovery boiler operation. Instead of forming a distinctive soap layer on top of the black liquor, soap can also form a sludge that sinks when the fatty acid/resin acid ratio drops below about 0.9.

Uloth and Wearing (1988) showed that adding air to the black liquor going to the evaporator soap skimmer can increase tall oil soap skimming efficiency. When the feed liquor soap content exceeded 25–35 kg/tDBLS, air injection increased soap skimming efficiency by an average of 22%, based on the total soap available (i.e., from 50%–65% to 70%–90%). In agreement with earlier studies by Foran (1984), adding air at a rate of about 7 L/m^3 of liquor provides near-optimum results, although lower injection rates (3.3–4.5 L/m^3) were found to be equally effective in one mill. The soap's tendency to sink when the fatty acid/resin acid ratio drops below 0.9 was hypothesised to be due to higher soap density (Pearl et al. 1976), and so testing the effects of higher air injection rates on tall oil skimming efficiency was recommended at mills pulping mountain pine beetle-infested wood (Uloth et al. 2007).

2. MATERIALS AND METHODS

Skimmer studies were conducted around the evaporator soap skimmer in the A line at Mill D in late July 2008. Both the soap skimmer feed lines in the A and B lines at the mill were equipped with sintered metal air diffusers and rotameters just downstream of the skimmer feed pump off the #4 effect evaporator in the mid-1990s. Black liquor from the #4 effect is pumped about 50 m to the evaporator soap skimmer, adjacent to the soap loading bay and the rail line. At about 3:00 p.m. on July 29, the air flow rate to the A side skimmer was adjusted to approximately 8.9 L/m^3 (1 ft^3/min or cfm on the rotameter) for our first day of testing. The set air flow rate was about 27% greater than the optimum established in tests at the same mill in the late 1980s (Uloth and Wearing 1988), but about 20% lower than the flow rate currently used by the mill's operators. The air flow was left at this rate overnight and liquor sampling was started at 9:00 a.m. the next morning.

As composite samples greatly reduced the scatter in the experimental data (Uloth and Wearing 1988), composite samples of the approximately 25% solids black liquor going to and leaving the soap skimmer were taken over a period approximating the liquor retention time (3 h) in the skimmer. Three hourly grab samples of each were composited for analyses. The solids content of each sample was determined using the TAPPI standard test (T 650 om-89 1995). The tall oil content of each sample was determined using a modification of the Saltsman and Kuiken procedure (1959) and PCA-24 (1977). The crude tall oil (CTO) content of the skimmed liquor was converted to soap content by assuming a 60% yield of CTO from soap (Uloth et al. 1987).

At 9:00 a.m. on July 31, a sample of the skimmed soap was taken from the A side soap skimmer. Its volume was marked so it could be weighed and compared to an equal volume of water to estimate the soap density. After the skimmer tests, the black liquor content of the skimmed soap was measured using procedures outlined by Ouchi et al. (1994). The tall oil content of the soap sample and acid number of the extracted tall oil were determined using the standard Pulp Chemicals Association test procedures (PCTM 7 1996, followed by PCTM 1 1996).

The air injection rate was then increased by about 17% to about 10.4 L/m^3 of liquor, the maximum allowed by the air tubing, diffuser, and rotameter on the A line and the rate currently applied at the mill. This is about 50% higher than the optimum seen in the mid-1980 tests (Uloth and Wearing 1988). We started taking composite samples around 10:00 a.m. and continued sampling until 6:00 a.m. the next morning. At 9:00 a.m., the skimmed soap was sampled again so we could look at the effects of the higher air flow rates on the skimmed soap density and black liquor content, and determine the acid number for the soap skimmed over the second day of testing.

After double-checking the analyses for several black liquor samples from the skimmer efficiency testing, three of the feed liquor samples from each day were mixed, covered, and sat overnight in an oven at 90°C. The insoluble soap was skimmed from each sample, and the still-hot skimmed liquor was sampled. The solids content and crude tall oil (CTO) content of the skimmed liquors were then determined to estimate tall oil soap solubility in the skimmer so skimming efficiency could be calculated based on both the total soap available and the insoluble soap. Since the soap solubility limit for the first set of samples (8.1 kg/TDBLS on July 30, and 8.5 kg/TDBLS for July 31) were much lower than expected, the solubility test was repeated using three more of the feed liquor samples from each day of testing. Very similar results (9.8 kg/TDBLS on July 30, and 8.4 kg/TDBLS for July 31) were obtained. The averages from the two sets of tests for each day were used to account for limits on skimming caused by soap solubility. Since soluble soap cannot be skimmed, calculations based on insoluble or recoverable soap are a better and more consistent measure of the performance of a given soap skimmer than is soap recovery on the basis of total soap available (Uloth and Wearing 1988).

3. RESULTS AND DISCUSSION

Soap skimmed on July 30 had an acid number of 114.1; soap skimmed on July 31 had an acid number of 110.1. The acid numbers determined for these samples are slightly lower than the averages for soap shipped from Mill D to Chemtrade Logistics in both June (122.4) and July (122.3) 2008. Soap with an acid number in the 110–115 range would be expected to have a fatty acid/resin acid (FA/RA) ratio between 0.73 and 0.83 (see Figure 9 in Uloth et al. 2007). Even soap with an acid number of 122 would have an FA/RA ratio of only about 1.0. We previously observed (Uloth et al. 2007) that, instead of forming a distinctive layer above the black liquor, soap can form a sludge that sinks to the bottom of storage vessels when the FA/RA ratio drops below about 0.9. On the basis of the skimmed soap analyses, problems with soap skimming could be anticipated.

Soap solubility in the feed liquors averaged 9 kg/tDBLS for the 25.3% solids liquor on July 30, and 8.5 kg/tBLS for the 26.6% solids liquor on July 31, both significantly lower than the 14.8 kg/tDBLS (0.89% CTO on dry black liquor solids) seen for 25% solids liquor at this mill (Mill D) in the 2007 tests. Three (out of 15) of the skimmed liquor samples from the two days of testing were observed to contain 10–12 kg of soap/tDBLS, indicating that the laboratory-measured solubility limit was not unrealistically low. The change in soap solubility between the 2007 laboratory samples and the 2008 full-scale tests is likely due to changes in the soap or black liquor composition, but considering the low soap acid numbers, the observed low soap solubility in 25% solids liquor was unexpected.

Averages of the skimmer tests and impacts of varying the air injection rate on tall oil soap quality are summarised in Table 1. Only 37.7% and 13.1 % of the total available soap was skimmed on July 30 and July 31, respectively. When skimming limits due to soap solubility are considered, only 53.2% of the insoluble and recoverable soap was skimmed on July 30. On July 31, the skimming efficiency for the lower quality (110 acid number) soap averaged only 19.2%, even when the limits caused by soap solubility were taken into account.

Table 1. Effects of air injection rates on tall oil soap skimming efficiency and soap quality at Mill D

	Day 1 (July 30)	Day 2 (July 31)
Average air injection rate, L/m^3 of liquor	8.9	10.4
Average quantity of soap in the feed liquor, kg/tDBLS	31.0	26.7
Average quantity of soap in the skimmed liquor, kg/tDBLS	19.3	23.2
Number of hours where skimming stopped	3 - 5	9
Average skimming efficiency based on total soap, % (disregarding periods where skimming stopped)	37.7 (53.2)	13.1 (19.2)
Average soap solubility in the skimmer feed liquor, kg/tDBLS	9.0	8.4
Average soap skimming efficiency based on insoluble soap only, % (disregarding periods where skimming stopped)	45.7 (63.0)	44.9 (65.9)
Density of skimmed soap, kg/L	0.89	0.82
Weight % black liquor in the skimmed soap	12.8	17.3

More disturbing than the observed low skimming efficiencies were the extensive periods (3–5 h on the first day and 9 h on the second day) where skimming stopped and the concentration of soap in the skimmed liquor equalled or exceeded the concentration in the feed liquor. Potential reasons for the lack of skimming over these periods were investigated. If the periods of no skimming are dropped from the data set, on average, 45.7% of the total soap was skimmed on July 30, and 44.9% of the total soap was skimmed using a higher air injection rate on July 31; further, 63.0% of the insoluble soap was skimmed on July 30, and 65.9% of the recoverable soap was skimmed on July 31. Higher air injection rates were thus observed to have little benefit to soap skimming efficiency. Observed soap skimming efficiencies (about 45% based on total soap, with poor skimming periods dropped) were much lower than those seen with air injection (75%–89%, based on total soap available) at even lower air injection rates in the initial 1985 trials (Uloth and Wearing 1988). The reduced skimming efficiency reflects the decline in soap quality, the increase in soap solubility, and the decline in soap buoyancy when coming from beetle-infested wood (Uloth et al. 2007).

As expected, the higher air injection rates decreased the soap density by about 8%, and increased black liquor entrainment in the skimmed soap by nearly 35% (Table 1). As high percentages of black liquor can cause high H_2S emissions during soap acidulation, and the precipitated lignin can hinder tall oil separation (Uloth et al. 1994), the use of higher air injection rates cannot be recommended. Interestingly, air injection at these higher flow rates produced soap with a density very close to the 0.85 kg/L optimum (Uloth and Wearing 1988; Foran 1984), and the black liquor content of skimmed soap was no higher than that observed by Foran (1984) (14%–17% by weight) at the optimum air injection rates. These two observations suggest that the low FA/RA soaps have higher density than those skimmed in the earlier tests (Uloth and Wearing 1988; Foran 1984).

The soap content of each feed and skimmed liquor sample taken over the two days of testing (based on the weight of extracted tall oil measured in the PCA-24 [1977] test) are summarised in Figure 1.

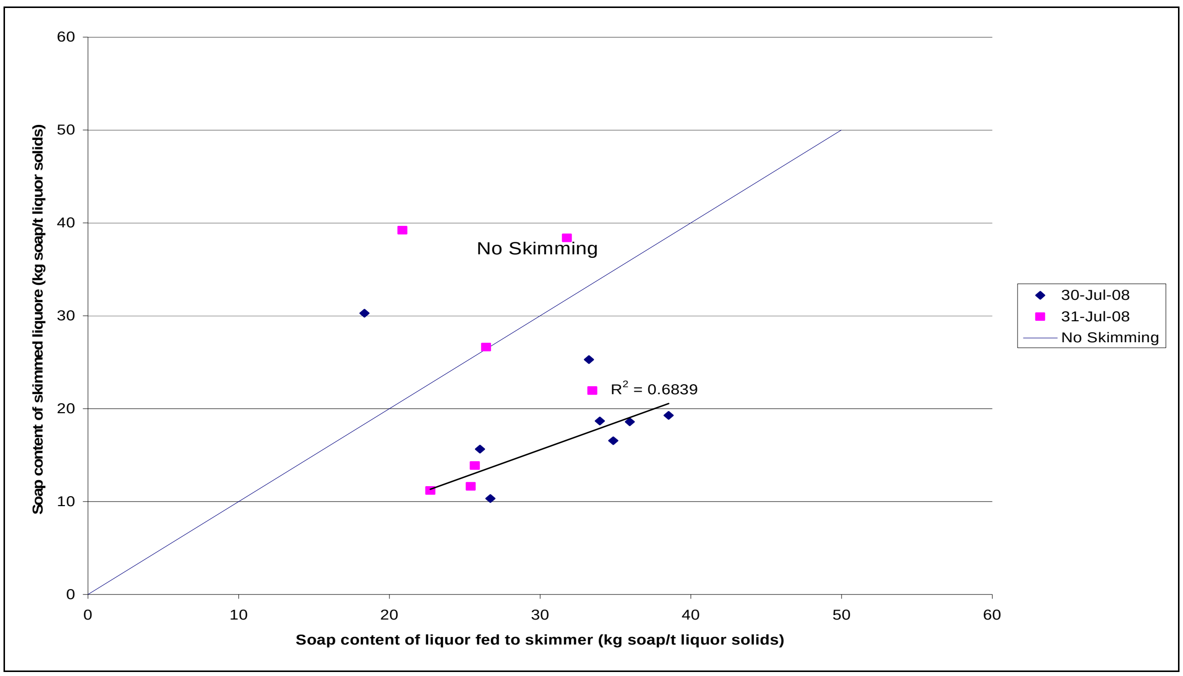

Figure 1. Effect of air addition and the feed liquor soap content, based on weight, on skimming efficiency over two days of testing on the A line evaporator soap skimmer at Mill D.

Note: Air addition rates were 8.9 L/m^3 of liquor on July 30, and 10.4 L/m^3 of black liquor on July 31.

Most data is grouped around the fitted line (R^2 = 0.68), which shows skimming efficiency, based on insoluble soap only, increasing from 62% with the feed liquor soap concentration at 40 kg/tDBLS, to 87% when the feed liquor soap concentration is 22 kg/tDBLS. However, three data points on July 31 and one data point on July 30 sit well above the line indicating no soap skimming. Two more data points, one on each day, sit well above the fitted line and show an adverse effect on the average measured skimming efficiencies (Figure 2). Soap skimming declined dramatically for 3–5 h on July 30 (sample D1 to D8), and stopped completely for at least 9 h on July 31 (samples D9–D15) (Figure 2). The negative skimming efficiencies indicate that the concentration of soap in the outlet liquor was higher than that in the feed liquor and suggest that soap already removed from the liquor periodically dissolved again or entrained in the exiting liquor. Negative soap skimming efficiencies could be due to sinking soap, or increased tall oil soap solubility.

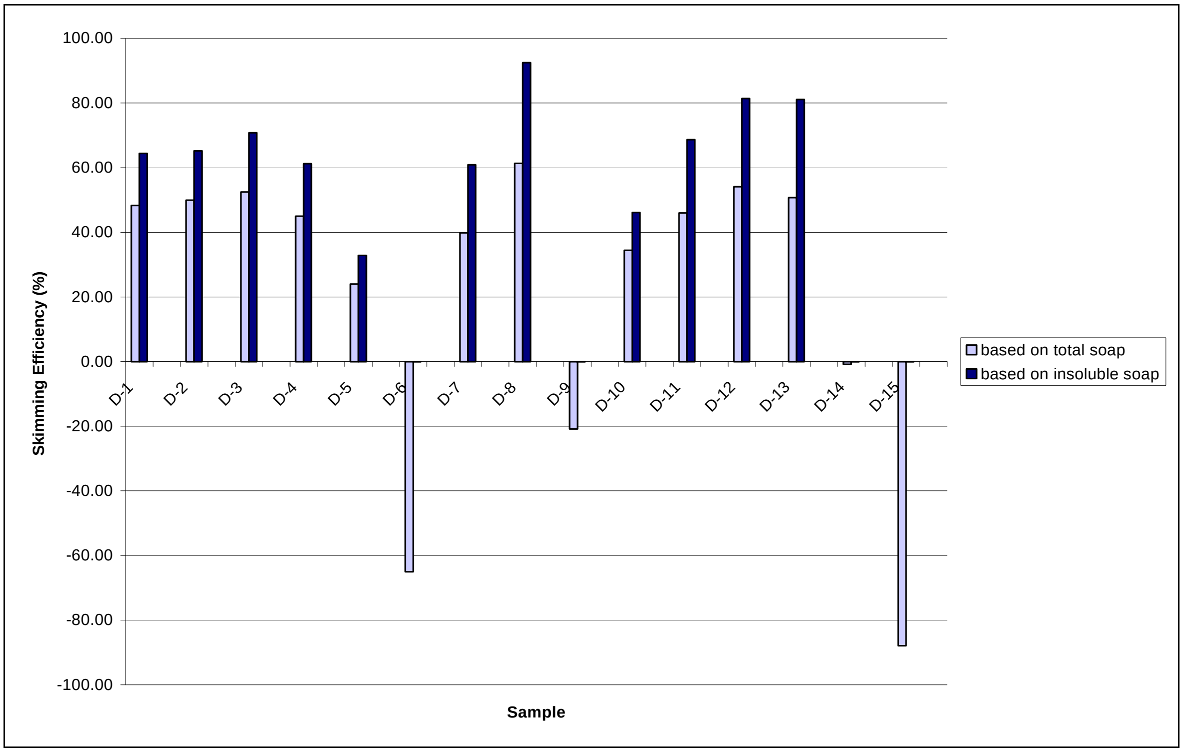

Figure 2. Tall oil soap skimming efficiencies, based on weight, over the two days of testing on Mill D's A line evaporator soap skimmer.

Note: For July 30, 2008 (Samples D 1–8), the air injection rate was about 8.9 L/m^3 of liquor. For July 31, 2008, the air injection rate was about 10.4 L/m^3.

To extract maximum data from the skimming trials, we weighed the extracted tall oil, redissolved it in isopropanol, and then titrated the extracted tall oil for each liquor sample using methanolic potassium hydroxide.

To use the titration results, one must plug an estimated acid number into the denominator of an equation to calculate the weight of tall oil and estimate the weight % tall oil on dry black liquor solids. Figure 3 compares the weight % crude tall oil (CTO) estimated for each July 30 sample from weighing the extract to that from titrating it. For the titration, tall oil weight was estimated assuming a tall oil acid number of 115, slightly higher than the 114.1 determined for the soap sample taken at the end of the test run.

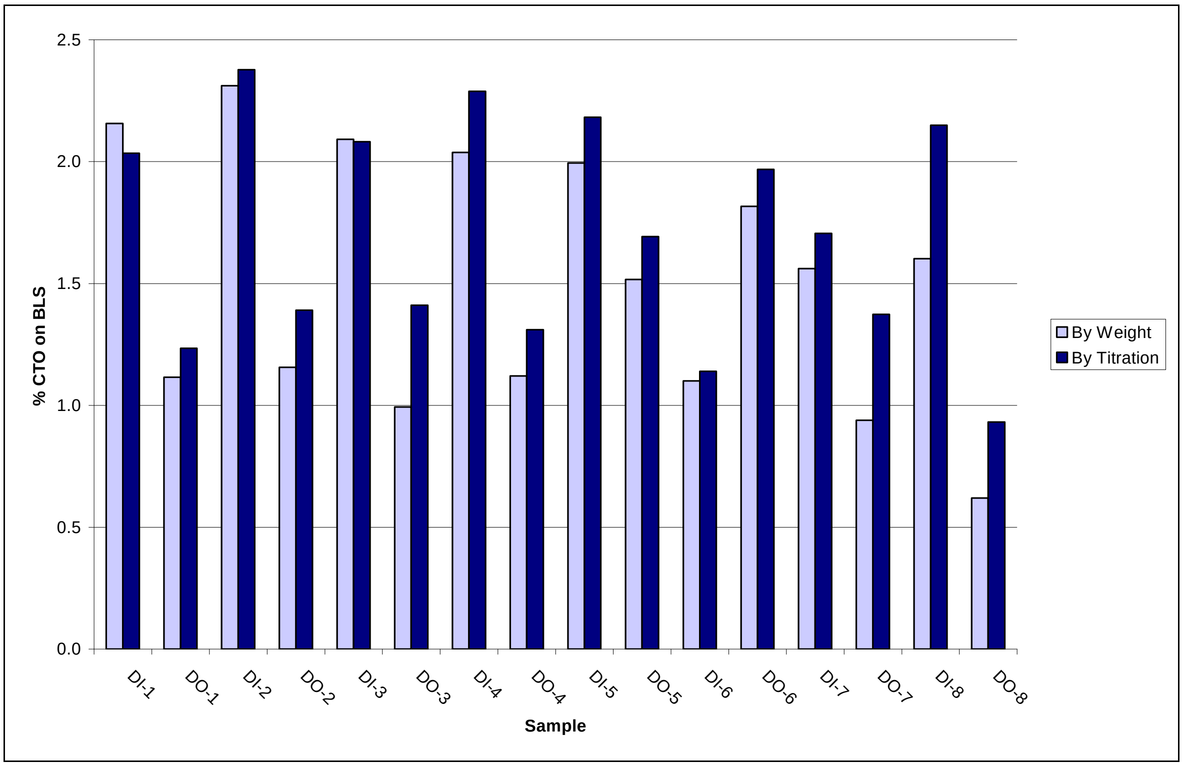

Figure 3. Weight % crude tall oil (CTO) on dry black liquor solids in the feed and skimmed black liquor samples on July 30.

Note: The weight of CTO was determined by both weighing and titrating the extracted tall oil. The titration was converted to a weight estimate assuming a tall oil acid number of 115, based skimmed soap collected at the end of the test.

While the results from the two estimates are similar for most of the samples, particularly at the start of the test, the titration gave higher estimates of the weight % tall oil on dry black liquor solids for 14 of the 16 samples. Since the acid number estimate goes in the denominator of the equation used to convert the titration volume to a weight, this suggests that the actual soap acid number was probably higher than 115.

Figure 4 compares the % crude tall oil estimated for each July 30 sample from weighing the extract to that from titrating it assuming an acid number of 124; titrations now give higher estimates of the weight % tall oil on dry black liquor solids for only 9 of the 16 samples, and most results from the two tests are very close.

The skimming efficiency dropped for the D5 samples, and was negative for the D6 sample set. Lower soap acid numbers were correlated to lower soap fatty acid content (see Figure 9 in Uloth et al. 2007) and lower soap FA/RA ratios, which were observed to reduce the soap's buoyancy. If the acid number of the soap in the feed liquor had dropped, we would expect the measured weight of tall oil to exceed that determined from the titration. Such is not the case for either the D5 or D6 samples. The fact that the weight of tall oil determined from the titration is significantly higher than that measured for 3 out of the 4 D7 and D8 samples suggests that the soap acid number may have increased in the last part of the test, resulting in the improved skimming efficiency seen in Figure 2.

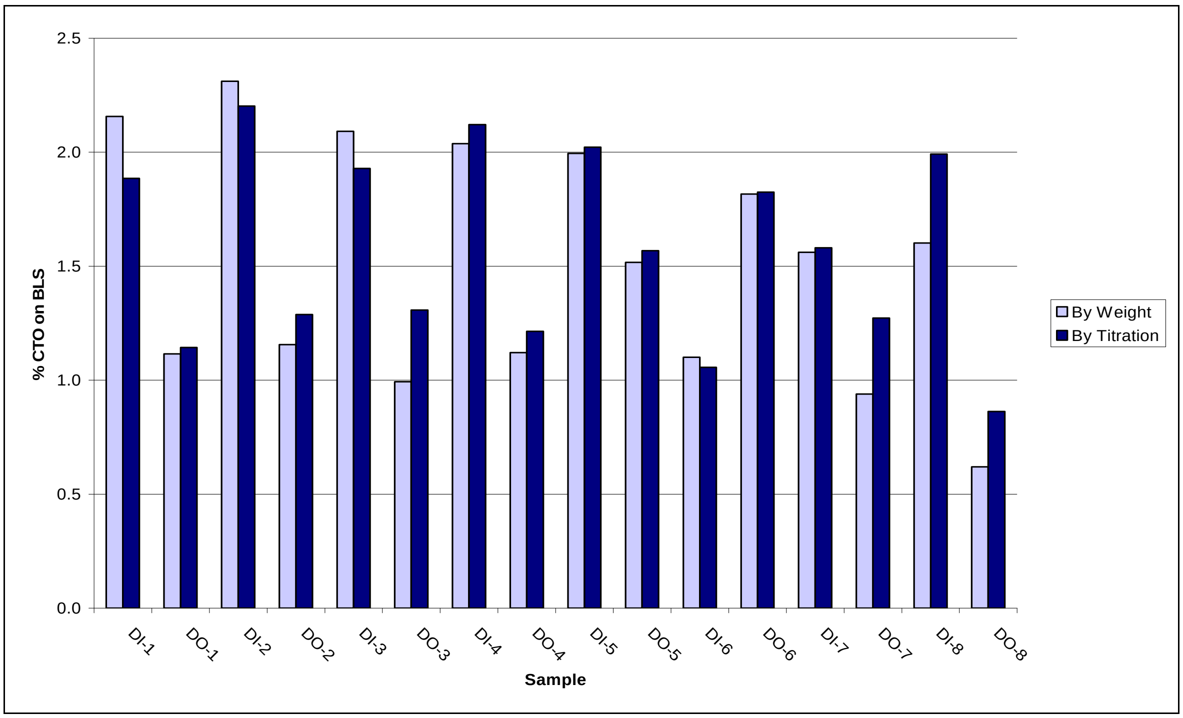

Figure 4. Weight % crude tall oil (CTO) on dry black liquor solids in the feed and skimmed black liquor samples on July 30.

Note: The weight of CTO was determined by both weighing and titrating the extracted tall oil. The titration was converted into a weight estimate assuming a tall oil acid number of 124, which was required to get the two estimates to closely agree.

Figure 5 shows a similar comparison between the % crude tall oil estimated for each July 31 sample from weighing the extract and from titrating it when an acid number of 138 is assumed. There was much more scatter between the weight % results determined by weighing and by titration on July 31, and this (perhaps unrealistically) high acid number was required to get fairly good agreement between the two test methods. The measured skimming efficiency was negative for samples D9, D14, and

D15, and quite low for the D10 sample set. Again, if the acid number of the soap in the feed liquor had dropped, we would expect the measured weight of tall oil to be greater than that determined from the titration. Such is not the case for either the D14 or D15 samples. Although the measured weight is greater than that estimated from the titration for the D9 samples, the difference is quite small.

Little evidence was thus found to support the hypothesis that the skimming efficiency dropped due to a decrease in the soap acid number or fatty acid/resin acid ratio, although the drop was most likely due to changing soap quality. While the drop in skimming efficiency cannot be explained, it is consistent with the experiences at Mill E, the mill that pulped the highest percentage of grey-stage beetle-killed wood described in our 2007 study. Mill E reported that their skimming efficiencies gradually decreased as they pulped more grey-stage beetle-killed wood, even though they, too, were using air injection on their soap skimmer. Eventually, soap recovery ceased and soap, which had been present in the skimmer, disappeared overnight, presumably into the liquor leaving the skimmer. Mill E took their skimmer out of service about 3 years ago and have since been plagued by high concentrator-scaling rates and liquor-firing problems in the recovery boiler (high-calorie, high-viscosity liquor that burns poorly). These problems are consistent with our findings on the effects of high concentrations of soap in fired black liquor (Uloth et al. 2007).

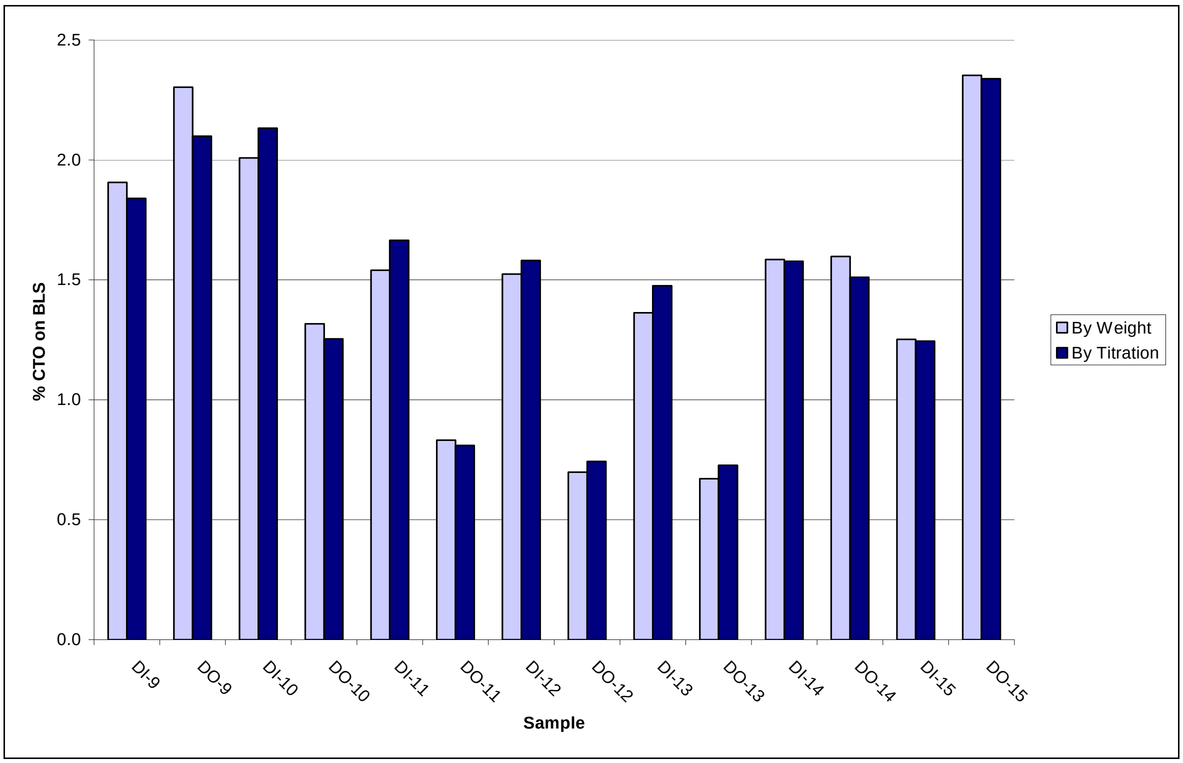

Figure 5. Weight % crude tall oil (CTO) on dry black liquor solids in the feed and skimmed black liquor samples on July 31.

Note: The weight of CTO was determined by both weighing and titrating the extracted tall oil. The titration was converted to a weight estimate assuming a tall oil acid number of 138, which was required to get the two estimates to closely agree, but is much higher than the acid number 110, determined from skimmed soap collected at the end of the trial.

Baseline skimming tests will be done on the soap skimmer at Mill E later in 2008 as part of a fatty acid addition trial. In light of the results observed at Mill D, we will run baseline tests for at least 12–18 h to see if we observe similar periods where skimming efficiency drops to zero. Waste fatty acids from canola oil production will be added to the weak black liquor at the mill to raise the FA/RA in the product soap to 1.5–2.0 over a 2-day period. Laboratory tests have shown that this should reduce the tall oil soap solubility to 6.0–8.5 kg/tDBLS in the 28%–30% solids liquor going to their soap skimmer. With two days of testing at the higher FA/RA ratios, we will also be able to see if fatty acid addition raises the soap skimming efficiency and eliminates periods of very low skimming efficiency.

4. CONCLUSIONS

Soap skimming efficiency at Mill D is currently poor. On average, only 37.7% of the total available soap was skimmed on July 30 and 13.1% July 31. When skimming limits due to soap solubility are considered, on average, only 53.2% of the insoluble and recoverable 115 acid number soap was skimmed on July 30. On July 31, the skimming efficiency for the lower quality (110 acid number) soap averaged only 19.2%, even when the limits caused by soap solubility were taken into account.

More disturbing were the extensive periods (3–5 h on the first day and 9 h on the second day) where skimming stopped and the concentration of soap in the skimmed liquor was equal to or greater than the concentration in the feed liquor.

If the periods of no skimming are dropped from the data set, on average, 63.0% of the insoluble soap was skimmed on July 30, and 65.9% of the recoverable soap was skimmed using a higher air injection rate on July 31. Higher air injection rates were thus observed to have little beneficial effect on the soap skimming efficiency.

Soap skimming efficiencies (about 45% based on the total soap available, even when the poor skimming periods are dropped) were much lower than those seen (75%–89% based on total soap available) with air injection at even lower rates in the initial 1985 air injection trials (Uloth et. al. 1988). The reduced skimming efficiency reflects the decline in soap quality, the increase in soap solubility, and the decreasing tendency for the soap to float, that was observed in earlier work to result from the pulping of mountain pine beetle-infested wood.

As expected, the higher air injection rates decreased the soap density by about 8%, and increased black liquor entrainment in the skimmed soap by nearly 35%. As high percentages of black liquor can cause high H_2S emissions during soap acidulation, and the precipitated lignin can hinder tall oil separation, the use of higher air injection rates cannot be recommended.

Little evidence was thus found to support the hypothesis that the skimming stopped due to a decrease in the soap acid number or the fatty acid/resin acid ratio, although the drop was most likely due to changing soap quality. While the drop in skimming efficiency cannot yet be explained, it is consistent with the experiences at Mill E, which pulped the highest percentage of grey-stage beetle-killed wood in our 2007 study.

5. ACKNOWLEDGEMENTS

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