Medium consistency oxygen delignification performed with a controlled cavitation reactor

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ABSTRACT: A U.S. mill conducted a pilot-scale medium consistency oxygen delignification (MCOD) trial incorporating a proprietary reactor as part of an integrated delignification system. Delignification responses were obtained for various operating conditions and multiple chemical charges. Delignification levels of 35% or more were obtained for hardwood pulp. The trials were designed to determine potential operating costs, energy consumption, operational conditions, and benefits associated with a low capital cost installation of this system into an existing washer line. Material and energy balances in conjunction with pilot plant and lab data have been used to predict potential net effects of the MCOD system upon operating costs. The effects of the oxygen delignification operation upon pulp physical properties were also determined.

Application: This work demonstrates the importance of mass transfer in oxygen delignification and how mills can benefit from working to improve it. This work also showcases an easy oxygen delignification retrofit for mills.

In a previous pilot scale trial [1], an 8 tons/day low consistency (1%-5%) oxygen delignification (LCOD) system using a reactor in the place of a high-pressure retention tower and high shear mixer was installed at a U.S. kraft mill. The system was run with both hardwood (HWD) and softwood (SWD) pulps. Several potential process variables were explored along with the affect of the reactor on fiber integrity. The LCOD technology, when developed from efficient three-phase mixing technology, was shown to perform as well as conventional medium consistency (MC) systems (8%-14%). LCOD performance was found to be similar to MC operation when the LCOD system was located after brownstock washing. Locating an LCOD system before brownstock washers on a fiber line may require substantially more oxygen, depending on the composition of the residual black liquor, because the high residual black liquor content consumes a high percentage of the applied oxygen.

The LCOD trials showed comparable delignification to conventional medium consistency oxygen delignification (MCOD) systems. Unfortunately, the cost of operating the LCOD system was prohibitive, based upon chemical savings alone. Due to the more favorable economics found in MCOD, a new series of lab and mill trials were commissioned. These trials were conducted on relatively well-washed pulp pulled from the discharge of a drum displacement (DD) washer. Batch HWD kraft was subjected to washing in a compaction baffle (CB) filter followed by a DD washer. Before the addition of the MCOD system, the brown stock went to a brown high density storage chest and then over a rewasher prior to the bleach plant. Stock was taken from the DD washer discharge at consistencies averaging between 8% and 9%. A stock slip stream was directed through the reactor and back into the DD washer. Various operational conditions were used to determine if the reactor was capable of performing oxygen delignification under MC conditions. The work from these highly successful trials is discussed in this paper.

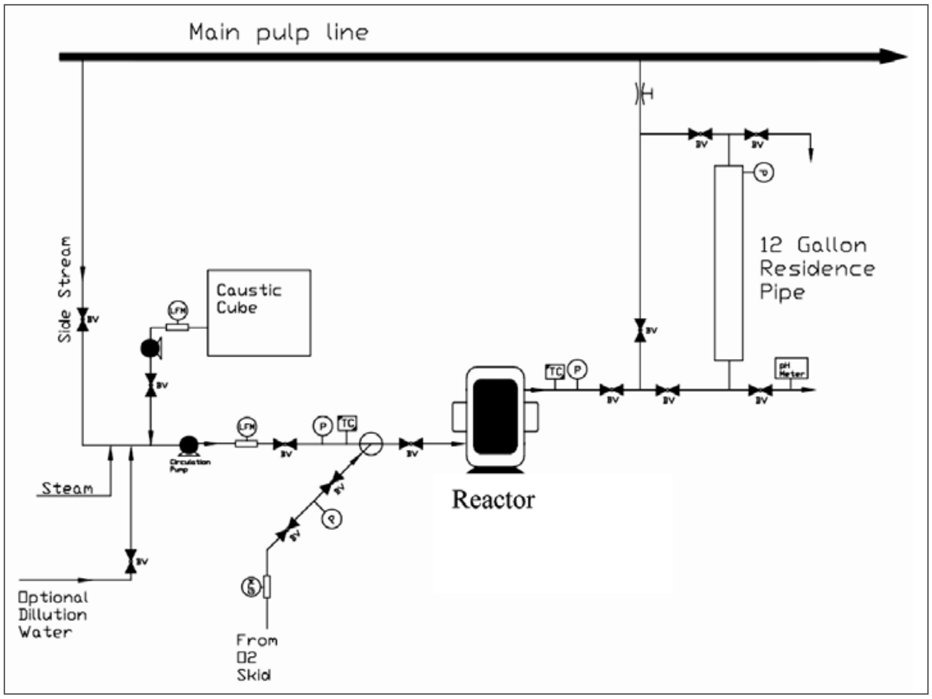
BACKGROUND

Oxygen delignification has many economic, product, and environmental advantages over traditional D_{100} bleaching sequences. Roughly 40% of North American bleach plants and almost 100% of the bleach plants in Scandinavia use oxygen delignification systems [2,3]. There has been a steady increase in the number of oxygen delignification systems in use worldwide has steadily increased since the first commercial installation in 1970 [4].

Much of the North American investment in oxygen delignification has been driven by mill-specific environmental concerns rather than economics. Oxygen delignification systems do not typically supply the fairly high rates of return on capital employed demanded by today's North American industry [5]. The environmental benefits and lower operating costs of oxygen delignification, as compared to alternate bleaching sequences, are well acknowledged throughout the industry. Recent research in the field has focused on improving the performance of the already proven technology in attempts to make it more economically viable and not just an environmental solution for mill specific problems.

Oxygen delignification systems can recycle effluent through the recovery system, thus lowering the amount of organic materials being sent to the bleach plant and ultimately to waste treatment. As a result, oxygen delignification systems exhibit many environmental advantages, including reductions in adsorbable organic halide (AOX), biochemical oxygen demand (BOD), chemical oxygen demand (COD), and effluent color. Another important benefit is lower bleaching chemical costs resulting from the displacement of more costly chlorine dioxide by oxygen, decreased chlorine dioxide demand in final bleaching stages, and decreased caustic required in the first extraction stage [6]. Additional benefits can include increased pulp yield.

Oxygen delignification is used in pulping operations to reduce kappa number, maintain pulp strength, reduce shives, and minimize environmental emissions. Oxygen is also used to lower overall bleaching cost, mainly by reducing the quantity of chlorine dioxide used. Almost 40% of the bleached pulp market uses oxygen delignification, which delignifies pulp to about 35%-50% of its original lignin content. Conventional oxygen delignification is run under MC (8%-14%)



1. Schematic of medium consistency oxygen delignification trial setup.

Property	Range
Mode of operation	Continuous or batch
Slurry flow, gal/min	25-75
Consistency, %	5-10
Slurry inlet temp., °C	60-95
Pressure, psig	85-110
Slurry pH	9-12
Reaction temp., °C	60-93
Reaction pressure, psig	90
Tank pressure, psig	0-90
End pH	11.5
Residence time, min	0-20

I. Test conditions.

conditions. Typical systems consist of a steam mixer, an MC pump, a high shear mixer, a pressurized residence tower with 30-90 minutes residence time, a blow tank, and washers. Higher consistency units, which run at pulp consistencies of 20%-30%, are also in use in pulp mills.

Mass transfer

Mass transfer is an important consideration in oxygen delignification due to the three-phase nature of the system. To react with lignin inside the fiber, oxygen must cross the gas-liquid interface, diffuse through the liquid film surrounding the fiber, and pass through the fiber wall itself. The ability to deliver oxygen to the inside of the fiber can be a limiting factor to the overall rate of the process [7]. Oxygen delignification is an environmentally friendly process, but the delignification rate has generally been thought to be relatively slow [8]. Rewatkar and Bennington [9] suggested that oxygen mass transfer limitations in retention towers cause the towers to operate 20% below their delignification potential, on average. Van Heiningen et al. [10] modeled oxygen delignification by taking into account oxygen mass transfer, pulp delignification kinetics, and mixer performance. Their studies found that industrial oxygen mixers are not effective in dissolving oxygen, even at the highest power input. Their work also found that most of the oxygenation and essentially all of the delignification occurred in the tower. Berry et al. [11] also demonstrated the importance of mass transfer in their recent laboratory study. Their work showed a wide range of delignification, 30%-55%, dependent upon the mixing conditions for a given mixer. Bennington and Pineault [12] found no single industrial oxygen mixer to be better than the next.

Pretrial work

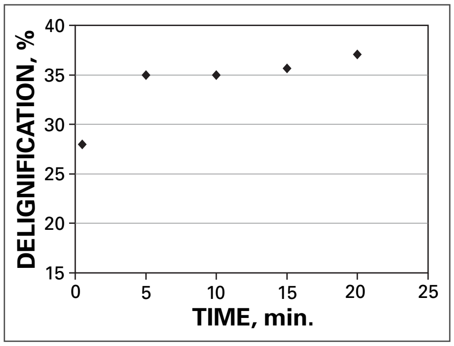
The Hydro Dynamics, Inc. reactor is designed to provide extremely high levels of mass transfer that can decrease the amount of residence time needed to reach industrially required levels of delignification. The reactor demonstrated high mass transfer capability in a series of laboratory studies conducted on a low and medium consistency oxygen delignification system incorporating the reactor as the gas-liquid mixer. The results from these studies showed the performance of this technology to be comparable to that of traditional MCOD systems.

The next logical step was a demonstration trial of this novel technology under a typical mill environment. The mill trial using the reactor-based low consistency oxygen delignification system was described in a previous paper [1]. Due to the minimization of mass transfer considerations in the reactor, oxygen delignification could be effectively conducted at low consistency, delivering results comparable to conventional MCOD. The improved mass transfer also resulted in substantially reduced retention times compared to conventional MCOD and similar times to those required in Mini-O systems. Medium consistency provided similar results in the lab and mill, with even better economic results due to the increased consistency. Lab results mirrored mill results for both low and medium consistency.

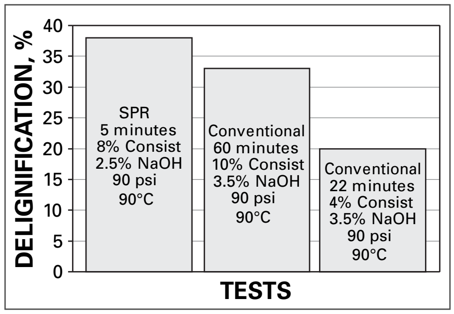
Medium consistency oxygen delignification trial

A 24 tons/day MCOD pilot plant trial was conducted at an integrated kraft mill. Hardwood brownstock pulp with kappa numbers between 12-18 was washed over a CD filter followed by a three-stage DD washer. The pulp was fully bleached to 87% ISO brightness using a D(EOP)D bleaching sequence. The objective of the trial was to demonstrate the MCOD technology under mill operating conditions and improve the economics over what was found in the low consistency trials presented earlier. **Figure 1** shows a schematic of the MCOD pilot skid. The test unit consisted of a 50 gal/min reactor unit, a Gould's circulation pump, and a 12 gal batch residence tank with accompanying instrumentation built on two skids.

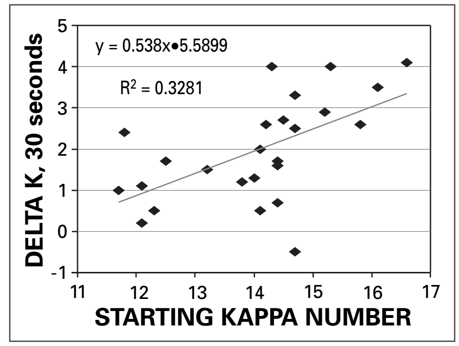
In a typical trial run, washed pulp discharged from the DD washer is drawn into the circulation loop through a side pipe on the discharge side of an MC pump at the prevailing consistency used in the MCOD system. The pump drive is



2. Percent delignification as a function of residence time.



3. Comparison of the reactor results versus the same pulp delignified in a Parr bomb reactor.



4. Effects of initial kappa number on the change in kappa number in the first 30 seconds of reaction.

equipped with a variable speed controller to accurately control the pulp flow rate and maintain pressure at the desired level. A piston-style transfer pump was used to add concentrated caustic for pH adjustment when necessary. Provision was also made for slurry temperature adjustment, when required, through steam addition.A side pipe delivers oxygen just before the reactor where the oxygen is mixed with the pulp slurry for the delignification reaction to begin. The reaction mixture flows through a holding tank that, along with the reactor unit, is held at pressure and is finally returned to the main brownstock line. There is also a bypass around the tank. The system can be run continuously and also allow pulp to be trapped in the tank and held for any desired residence time.

A test run begins by first adjusting the slurry and oxygen flows to the desired rates. After taking an initial sample, the pH of the slurry is checked and, if necessary, the caustic pump is used to adjust the pH to at least 11.5 – 12. Steam is added as necessary to increase the temperature to the desired level. Upon reaching steady state, pulp samples are collected for analysis (kappa number, brightness viscosity, and freeness).The reaction temperature, reaction pressure, slurry flow rates, oxygen flow rates, and reactor operational parameters are continuously recorded with a data acquisition system during each run. Samples were taken under both batch and continuous modes of operation. In the batch mode, the process was adjusted to a specified set of conditions and allowed first to run in the continuous mode while bypassing the residence tank until it reached steady state. At a time T_0, the stock was diverted into an insulated standpipe. The flow is turned off when the standpipe is full. The reaction mixture in the standpipe was maintained under prevailing conditions of temperature and pressure. Pulp samples were collected at 5- or 10-minute intervals for analysis. **Table I** shows the range of conditions tested.

RESULTS

The MCOD pilot scale trails were conducted over a 5-day period on a hardwood bleached pulp line at a northern U.S. integrated kraft mill. Experimental conditions were varied to determine the optimal operating conditions and to determine if the reactor would be able to perform oxygen delignification under MC conditions. Excellent levels of delignification were obtained during the MCOD trials. Optimized operating conditions were determined to be 1.8% applied caustic, 2% applied oxygen, 90ºC, at 90 psi, for 5 min. Fiber strength was not compromised under these operating conditions. Specific energy demand and brightness development were also investigated.

Residence time

In traditional MC delignification systems, delignification increases with residence time. In engineering a commercial system for minimum capital expenditure, residence time should be minimized. If a system can be operated with small enough retention times, all of the retention time can be obtained in piping systems instead of requiring an expensive pressurized tower. Various series of residence time versus delignification curves were obtained for multiple operating conditions. **Figure 2** shows a typical residence time curve obtained from these pilot plant trials for various operating conditions. Little value is obtained for residence time over 5 min (Fig. 2). Most of the delignification occurs in the first 5 min, with lesser but significant levels of delignification occurring over the next 25-55 min.

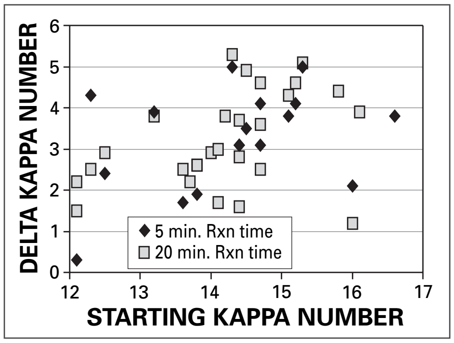
Mass transfer

After extensive pilot trials, the optimal residence time for the MCOD system was found to be about 5 min.Typically, MCOD systems require 30-60 min residence time. The reduced residence time is believed to occur because the reactor provides exceptional three-phase mass transfer, resulting in a reduced residence time requirement to obtain a given level of delignification.

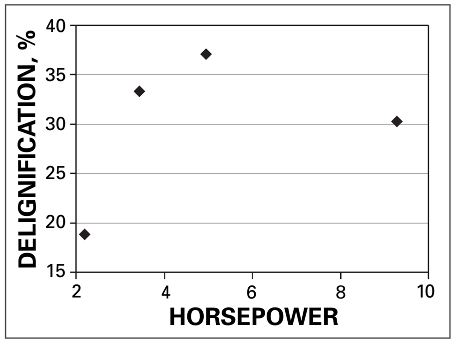
Laboratory studies were conducted using a Parr bomb to mimic conventional oxygen delignification technology. The resulting pulp was compared to pulp treated in the reactor followed with a 5 min residence time.The same starting pulp was used in all three tests. **Figure 3** shows a comparison of the Parr bomb low and medium consistency delignification studies as compared with pulp delignified in the reactor. Even with reduced caustic and reduced residence time, the reactor treated pulp exhibited higher levels of delignification than the Parr bomb treated pulps.

Starting kappa number and optimal residence time

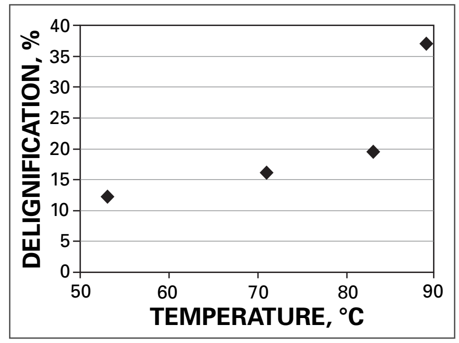
Several different conditions were run over extended reaction times in an attempt to determine what the optimal residence time should be. It appears that the majority of the delignification occurs in the first 30 s to 5 min. The starting kappa appears to influence how quickly a pulp responds, in both percentage and absolute kappa terms. **Figure 4** shows the change in kappa number during the first



5. Effects of reaction retention time on the change in kappa number as a function of starting kappa number.



6. Effects of horsepower applied to 50 gal/min flow on percent delignification obtained.



7. Effects of reaction temperature on percent delignification.

30 s of reaction. As the starting kappa number increases, a significant increase in the delta kappa number during the first 30 s of reaction occurs. Delignification continues to occur over the first 5 min then drastically slows down or stops altogether after that time. **Figure 5** shows the levels of delignification obtained for multiple pilot runs over both 5 and 20 min of retention time. No significant difference in kappa drop occurred due to extended reaction time for the pilot scale system.

The effects of horsepower

The reactor is a process intensification device. In process intensification, power usage is often a major concern. Applied horsepower per ton of pulp (specific energy) is a function of the volumetric flow rate governed by the consistency of the stock. Lower consistency stocks require more specific energy than higher consistency stocks. That is because fewer tons of stock are processed through the reactor in a given time for low consistency stock as opposed to higher consistency stock. For the MC test conditions, the reactor was found to have a relatively small power draw for the level of intensification needed to perform industrially acceptable levels of delignification. The reactor requires about 0.1 horsepower days/ton of stock treated to obtain good delignification levels. **Figure 6** shows the horsepower applied to a 50 gal/min flow of MC hardwood stock and the percent delignification achieved for each test condition.

Temperature

Temperature is a well-known requirement for oxygen delignification systems. Typical commercial systems operate around 90ºC. Many laboratory-based studies conducted in sealed Parr bombs or in lab-scale high shear mixing devices are conducted at around 100ºC. Reasonably high operating temperatures were obtained from the stock leaving the DD washer. Typically, the stock leaving the DD washer was in the 75ºC-85ºC range. To obtain higher reaction temperatures, a steam line was connected to the stock line going to the MCOD pilot plant. A cold water line (about 2ºC) also was connected to the stock line going to the MCOD pilot unit. Operating temperature was varied between 50ºC-90ºC by mixing either cold water or steam into the stock. No significant levels of delignification occurred until the temperature was elevated to 85ºC or higher (**Fig. 7**).

Caustic

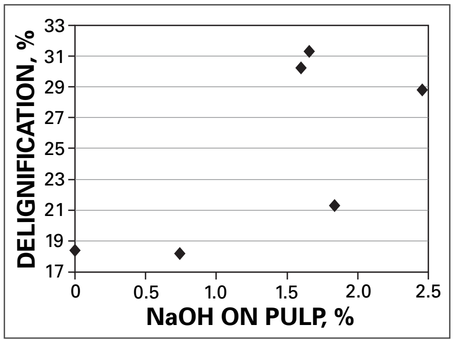
Caustic is used in commercial systems to drive delignification reactions. It is consumed in the delignification reaction and drives the level of delignification. At higher caustic charge better delignification is achieved. A downside to using higher levels of caustic to drive the delignification reactions is that pulp strength is damaged. Operating costs also increase as the use of oxidized white liquor, the typical caustic source for oxygen delignification systems, increases. Optimal caustic addition is determined by a careful balance of high levels of delignification with the need to maintain pulp strength and limit operating costs. For the MCOD pilot system, acceptable levels of delignification were obtained with about 1.8% caustic addition. Viscosity tests indicated that good pulp strength was maintained with caustic charges of up to 2.5%. Thus, the optimal charge appears to be between about 1.8% to 2.25% w/w on ovendry (o.d.) pulp resulting in a starting pH between 11.5 and 12. **Figure 8** shows the effects of caustic charge on percent delignification.

Oxygen

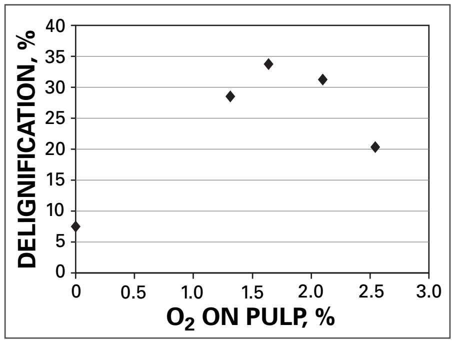
Oxygen is a relatively cheap chemical. As such, it is typically desirable to add excess chemical to ensure that enough is always present. For several years, commercial oxygen delignification systems simply added excess oxygen to ensure there was never a deficit. More recently, studies have shown that excessive oxygen addition can be detrimental to delignification [12]. This is most likely due to bubble coalescence that results in increased bubble size, which may slow the rate of diffusion. **Figure 9** shows the effect of oxygen addition levels on delignification. An optimal oxygen charge was determined to be about 1.8% w/w on o.d. pulp. The downward trend after 2% oxygen charges seen in Fig. 9 may be due to an increase in bubble size and a higher likelihood of bubble coalescence.

Brightness

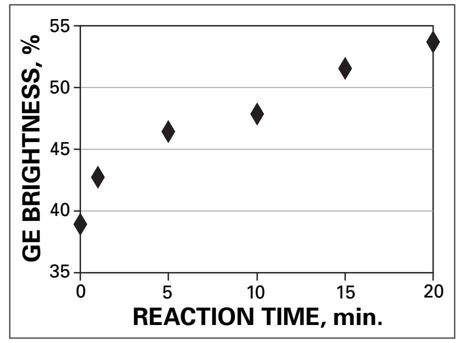
Brightness is not often the goal of oxygen delignification. Typically, oxygen delignification is used to lower the kappa number entering the bleach plant to reduce the chlorine dioxide required in the first bleaching stage. Therefore, delignification is more important than brightness development. The only time that brightness development would be a significant requirement is if the brightness ceiling of the bleach plant is being reached. Under this situation, extended residence time could be used to improve the final pulp brightness. **Figure 10** shows that significant brightening occurred during the reaction. Brightness was much slower to develop than delignification.



8. Effects of caustic charge on delignification performance.



9. Effects of oxygen addition levels on delignification performance.



10. Brightness development as a function of residence time.

Physical properties

Physical properties are measured on stock before and after delignification. Tear, tensile strength, tensile energy absorption (TEA), viscosity, and brightness were not impaired by the reactor. **Table II** shows the effects of the reactor delignification on these physical properties. Viscosity drop was in line for what would be expected from oxygen delignification.

The reactor unit is a high intensity device operating on MC stock. The reactor unit could possibly refine fiber at the same time that the unit was performing oxygen delignification. To determine the affect of the reactor on fiber refining, samples were collected before and after the reactor. CSF was measured on these samples to determine the influence of the reactor operation on fiber refining. **Table III** shows the freeness before and after the reactor. Within experimental error, no effects of reactor operating on fiber refining were found to exist.

Sample	Tear Index	Tensile Index	TEA Index	Viscosity	Brightness
#1, before	0.5	32.67	24.38	18.28	37.65
#1, after	0.63	38.38	33.62	18.6	50.1
#2, before	0.55	34.06	23.12	27.86	38.86
#2, after	0.69	39.29	46.23	19.47	51.48

II. Effects of oxygen delignification on physical properties of pulp.

	Before Reactor, CSF	After Reactor, CSF
Sample #1	543	550
Sample #2	553	547

III. Freeness before and after the reactor.

Effects of mill layout on reactor performance

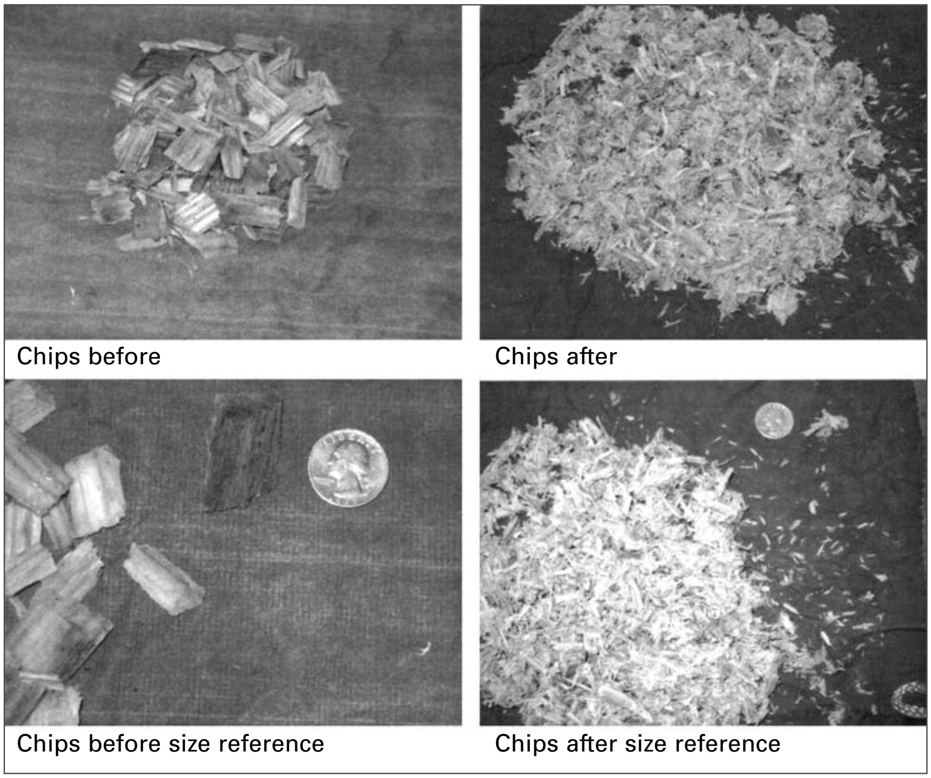
Conventional oxygen delignification systems require significant levels of post-oxygen washing. Frequently, two washers are installed after the oxygen system blow tank. Sufficient post-oxygen washing is required to ensure that the dissolved lignin and applied caustic can be separated from the fiber. If the washing is insufficient, dissolved lignin will carry forward into the bleach plant and negate the positive benefits of the delignification system. One of the problems with oxygen delignification systems is that the cost of these post-oxygen washers can make the project cost prohibitive.

One advantage of a reactor MCOD system is the potential to place it between existing washers, thus eliminating the need to buy new washers. To determine if the existing washing systems are able to handle an MCOD system, a WinGEMS model was developed for the mill. Two converged, verified WinGEMS models of two different washer lines in two different mills were developed. Each of these converged models was modified to include an MCOD system after the brownstock washing and before either a rewasher or decker system, depending upon the specific mill in question. In both models, a screen room was located after the MCOD system. Each of these WinGEMS models were then reconverged without altering the washing parameters set in the pre-MCOD model.

The results of these models were used to help determine the economic effects of retrofitting an MCOD system into an existing washing line. If the model predicts that 75% of the dissolved lignin and applied caustic could be separated from the fiber, the delignification levels used in the economic analysis were reduced to 75% of that obtained during the pilot scale trials.

Results obtained from the two different mill models suggested that the existing mill layout exhibited significantly different capabilities towards post-oxygen washing. The WinGEMS model of one mill suggested the MCOD system could be successfully incorporated into the existing mill layout and obtain upwards of 90% of the benefit available from the MCOD system. The other mill model suggested the current layout and operation of this mill would only be able to obtain 65% of the benefit available from the MCOD system. Performance can vary due to the differences in mill configuration and operation, which means the potential net economic benefits vary from mill to mill. For the test mill, savings could be as high as US$ 2.00/ton of pulp, considering chemical savings alone, while the potential net benefit for other mills might actually be higher or slightly negative. Additional savings are possible through increased yield, increased production, environmental benefits, and reduced use of make-up caustic.

In addition to determining the post oxygen delignification washing efficiency required to determine process economics, material and energy balance modeling is required to determine the temperature of the various stocks and filtrates. When the stock entering the reactor is heated to 90ºC and run over existing vacuum washer systems, it is possible to heat the filtrate to the point where



11. Wood chips before and after processing through a reactor.

one of the washers might start flashing in the drop leg. Carefully prepared and interpreted energy balances may be used to predict and eliminate potential operational problems resulting from the incorporation of an MCOD system into an existing washer line.

System robustness

One final area of concern is system robustness. Any commercial system has to be able to handle nuts, bolts, knots, and hard cooks without plugging or breaking. As the reactor is an energy intensifying device, its ability to handle these types of process upsets without plugging or breaking was a concern. A trial was conducted in which whole uncooked wood chips suspended in water were run through the reactor. The reactor disintegrated the chips. Upon dismantling the reactor no damage or excessive wear was found. **Figure 11** shows photographs from this trial.

CONCLUSIONS

Excellent delignification results were achieved in this study with low chemical and power additions and minimal residence time. The set of operating conditions sufficient to obtain about 35% delignification for hardwood pulp was found to be 1.8% caustic, 2.0% oxygen, 90°C, at 90 psi, for 5 min. CSF was unchanged during processing through the reactor and physical properties were not negatively affected by the delignification reactions.

The MCOD system can be incorporated into an existing brownstock washing line without adding additional post-oxygen washing. WinGEMS modeling results suggest that existing mill layout plays an important part in the economics of retrofitting an MCOD system into a brownstock washing line. Models of two different mills supplied vastly different results with respect to post-oxygen washing efficiency. One mill's configuration enabled the mill to obtain post-oxygen washing efficiency of about 90%. The second mill was only able to obtain a 65% washing efficiency. The difference in washing efficiencies alters the potential economics from a net cost savings of about US$ 2.00 per ton of pulp to a slightly negative net operating cost when considering chemical savings alone. **TJ**

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INSIGHTS FROM THE AUTHORS

We chose to research this topic as an application of an already proven process intensification and acceleration reactor to show how oxygen delignification retention time could be reduced through increased mass transfer.

This research complements work published in *TAPPI JOURNAL* in January 2005 (page 26) on low consistency oxygen delignification using the same reactor. It shows the effectiveness of the reactor on low and medium consistency stock, as well as the operating cost savings realized at medium consistency.

The most difficult aspect of this work was to conduct the experiments in a mill environment, with all the natural variability found there (and not in the lab).

The most surprising aspect of this work was the minimal increase in delignification achieved through extended residence time beyond 5 minutes. It leads one to wonder how much of the reaction is mass transfer limited and how much is kinetic.

A mill can benefit from this work by knowing the importance of mass transfer in oxygen delignification and working to improve it. This work also showcases an easy oxygen delignification retrofit for mills.

Our next steps include a full-scale installation at a mill.

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