Chemical Recovery and Pulp Mill Optimization and Control

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ABSTRACT

With increasing competition worldwide, the pulp and paper manufacturers, require greater emphasis on cost reduction and increased product efficiency and quality. Chemical recovery process continues to be a critical component of a pulp mill, providing chemical, steam, and power. The mill's economic viability rests in large part from efficient and optimized chemical recovery operations.

The application of new measurement tools has led to new advancements in control strategies which take advantage of the multitude of liquor chemical compositions. Implementations of these analyzers and control strategies over the past four years, mills have reported significant process variability reduction, typically from 40% to 60%, allowing for target shifts to higher TTAs and higher CEs. In addition, these mills have reported significant reduction in purchase lime usage and all mills reported reduction in pressure filter maintenance, reduced plugging in evaporators and plugging in recovery boiler. In one case, increase WL strength and debottlenecking recausticizing area resulted in significant increase in digester production. ROI ranged from $500K to $3M.

This paper will briefly review the analyzer technology and will focus on economic impacts achieved through efficient and optimized chemical recovery operations with the use of process analyzers and control.

INTRODUCTION

Historically, the lack of on-line sensors for key process variables and product quality attributes has hampered the development of advanced process control [1-5]. Comparing sensor needs from twenty-five years ago [6-7] to those of today, it is interesting to note that while some needs such as kappa number and dissolved solids content have been met, others such as chip moisture, bulk density and species, pulp yield, recovery boiler bed conditions, chip level in hydraulic continuous digesters, etc. still remain a challenge. Furthermore, according to a survey completed just ten years ago [8], the vast majority of new sensors are being used primarily for process monitoring, i.e. very few have been successfully incorporated into process control strategies. In addition, many rely on indirect measurements, such as conductivity, density, and refractometry, to infer at the properties of interests.

Real-time monitoring of chemical composition and physical properties, when coupled to supervisory process control, results in higher yields, higher throughput and improved quality by providing the information necessary to achieve tight control.

Routine on-line measurement of the effective alkali (EA), sodium sulphide (Na_2S) and sodium carbonate (Na_2CO_3) content of kraft liquors can lead to significant improvement in the practice of controlling key mill processes, especially the pulping and causticizing operations. Conductivity sensors have been used extensively for this purpose [9], but need to be corrected for variations in temperature and total titratable alkali (TTA) [10]. Furthermore, these devices lack chemical specificity, particularly when applied to black liquor [11]. To obtain the required specificity, methods such as ion chromatography (IC) [12], capillary electrophoresis (CE) [13-14], and titration [15] have been explored. Except for titration, none of these techniques have been adapted for on-line analysis. Even though automatic titrators can be used for determining sulphide and carbonate concentrations in white and green liquors [16], these systems require extensive sample pretreatment and precise volume delivery of sample and titrant, thereby limiting the sample throughput to a few samples per hour.

FT-NIR LIQUOR ANALYSER

Principle of Measurements

Near infrared spectroscopic techniques are well suited for on-line applications because of their rugged design, high throughput and ability to accurately determine the concentration of several liquor components simultaneously. They require less sample preparation, no reagents, and have fewer moving parts and, therefore, require less maintenance. In addition, the ability to use fibre optic cable for remote sensing allows for integration of one spectrometer for a multitude of applications.

In the past 15 years, we have seen much activity in the development of vibrational spectroscopy [17-20], particularly the maturation of the FT-NIR (Fourier-transform Near Infrared) liquor analyzer. Principally, FT-NIR utilizes the information associated with the absorption of Near Infrared (NIR) electromagnetic radiation by molecules or ion interactions with molecules to probe at the presence and extent of concentration. This methodology excels over other techniques as it provides true liquor compositions and can provide a much higher sample analysis frequency over traditional manual titrations, allowing for coupling with supervisory control.

Acousto-Optical Tunable Filter (AOTF)

The success of FT-NIR technology as online process analyzers for kraft pulp mill operations have led to other NIR technology, particularly dispersive based instruments. A recent trend in dispersive NIR spectroscopy is to use an acousto-optical tunable filter as a monochromator [17]. The advantage of this monochromator is that it provides faster scan rate. Despite a fast scanning speed and a relatively large aperture, the spectral resolution of an AOTF is modest at best (approximately 50 cm^{-1}). Poor spectral resolution can have adverse effects on calibration model transferability. Also, AOTF instrumentation needs occasional recalibration during use (offset compensation) and after servicing, thereby generating greater calibration maintenance costs. An account of early pulp and paper experience with AOTF-NIR technology has been given in [19]. The author reported persistent fouling problems characterized by severe film formation on the optical window, which caused inconsistent results. Due to the use of a small pathlength cell (<1mm) which could pose a severe plugging potential, any scale or film formation would account for a relatively large change to the optical pathlength and results in large influence on measurement results.

Fourier-Transform Near Infrared (FT-NIR)

On the other hand, FT-NIR spectrometers provide superior throughput (25x more energy) and wavelength accuracy which are essential for more demanding applications, such as those for pulp and paper applications, such as weak black liquor and dissolving tank green liquor. The use of a large optical pathlength (8mm – 20mm) enables greater sensitivity (A=molar absorptivity*pathlength*concentration) while minimizing possibility of cell plugging. This last point is particularly true when one considers the solids content present in 1st and 4th causticizing plant samples or the high solids content of a chlorine dioxide generator liquor. In addition, inherent with the higher spectral resolution, calibration model transferability is enhanced; allowing models developed to be used from mill to mill.

Since the mid 1990's, more than 35 installations of FT-NIR technology have been implemented in North American mills. Most of these systems are still operational, and many are still running on the original calibration models. A more complete review of the historical development and advantages of this technology are reviewed in [20]. US patent 6,339,222 B1 [21] further describes the technology and applications for pulp and paper operations. Numerous literatures are available including international and U.S. patents, peered reviews journal articles, and conference papers which document the use of the analyzer technology [22-34]. Table I shows a list of liquor streams and corresponding compositions that are available. Measurement accuracy as determined against manual titrations is summarized in Table II. Some of the more practical aspects of implementation are discussed below. This is followed by example applications.

TABLE I: FT-NIR LIQUOR MEASUREMENTS

Liquor Type	Component Measured
White & Green Liquors	EA, AA, TTA, Na_2CO_3, Na_2S, Na_2SO_4, $Na_2S_2O_3$, %RE, %CE
Black Liquor	EA, Organic, Inorganic & Total Solids
Weak Wash	TTA, NaOH, Na_2SO_4, $Na_2S_2O_3$
ClO_2 Generator	Acid (H_2SO_4), Chlorate ($NaClO_3$), Solids content
EA = Effective Alkali (NaOH + 1/2Na_2S) AA = Active Alkali (NaOH + Na_2S) TTA = Total Titratable Alkali (NaOH + Na_2S + Na_2CO_3) %RE = % Reduction Efficiency %CE = % Causticizing Efficiency	

TABLE II: ACCURACY OF FT-NIR MEASUREMENTS

Component	Accuracy
EA	± 1.0 g/L as Na_2O
AA & Na_2CO_3	± 1.2 g/L as Na_2O
TTA	± 1.5 g/L as Na_2O
Na_2SO_4 & $Na_2S_2O_3$	± 0.50 g/L as Na_2O
Organic & Inorganic Solids	± 0.5 %
Total Solids & Lignin	± 1.0 %
H_2SO_4	± 2.0 g/L
$NaClO_3$	± 3.0 g/L

FT-NIR Analyzer Hardware and Operation

The FT-NIR-based analyzer consists of two separate components; an instrument rack consisting of a spectrometer and computer and a sample skid consisting of a sample cell and various valves that deliver liquor from the process points to the cell for measurements. Near Infrared light from the spectrometer is carried through fibre-optic cable to the cell, passes through the large optical pathlength, and is delivered back to the detector at the spectrometer where it is processed to determine liquor compositions (Figure 1). All spectral data were collected with a spectral range of 14000 cm^{-1} to 6500 cm^{-1} (714 nm to 1538 nm).

At the cell, one or more samples (up to 6 samples) from different sample sources (ie. clarified GL, #1causticizer, etc.) can be tied into the cell through a sampling station, see Figure 2. Each spectrometer supports up to 8 cells and each cell may be located up to 300 m (cable length) from the spectrometer. This fibre optic connection allows for one unit to be used for multiple applications, such as those listed earlier.

As with all online analyzers, potential scaling of sample lines and the cell is of major concern. Demineralized water is used to flush the flow cell as well as the sample lines to minimize plugging. Automatic referencing to zero the system (equivalent to a blank) is performed once an hour, thereby allowing the system to compensate for any possible fluctuations in light source characteristics and/or cell condition.

In addition, a multitude of permissives, such as liquor flow, temperature, pressure, and conductivity, are monitored to indicate the quality of the liquor being measured. These diagnostic features, as well as spectral data diagnostics, can be monitored directly and can be configured to trigger alarms and other smart logics for control.

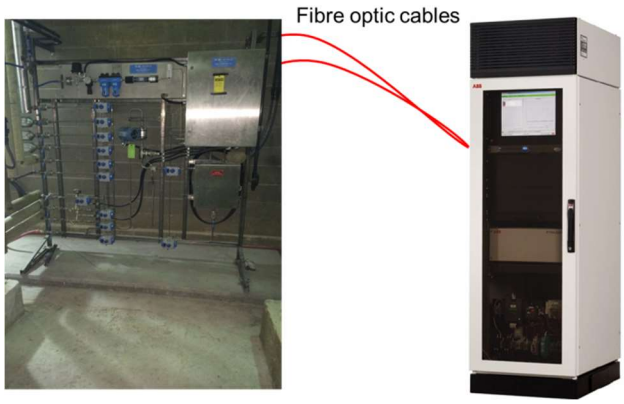


Figure 1: FT-NIR sample skid and spectrometer rack. The spectrometer rack is typically located in the MCC room and connects to the sample skid via fibre optic cable.



Figure 2: FT-NIR liquor analyzer sampling station showing sampling tree and transmission cell.

Maintenance

Spectrometer maintenance involves replacing the light source once per year and an internal laser once every three years. The sample cell windows are made of sapphire and have excellent hardness and chemical resistance. If the windows require cleaning, the inner flow cellblock can be removed without detaching the fire-optic cables. Sampling valve repairs are typically required once a year, depending on application and solids content, with most of the sampling station parts being typical mill store stock items.

Integrated Platform Technology

The ability to have up to 8 sample cells connected to a single spectrometer has inherent advantages for process analyzers. Figure 3 illustrates a single spectrometer connected to four different sample stations with their own flow cells, with each sample station being able to accommodate multiple sample streams; one cell at the digester with 5 sample streams, one cell at the ClO2 generator with a single sample stream, and two cells at the recaust plant with multiple sample streams, all working concurrently. This integrated platform system allows for flexibility with the analyzer. Mills can effectively expand the measurement capability as the needs arise, for example, starting with recaust application and then expanding to digester or chlorine dioxide generator. Future applications can easily be integrated by adding additional sample stations and connecting to spare detectors at the spectrometer.

Since the platform spectrometer is the same, lower cost is realized. More importantly, technology familiarity and servicing for the E/I department is maintained, even if E/I personnel are shuffled from one area to another.

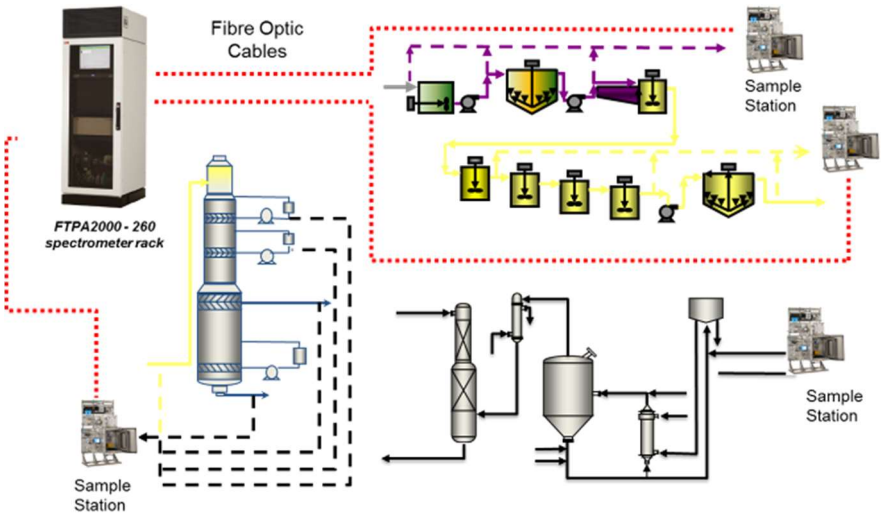


Figure 3: Integrated platform FT-NIR system showing the use of one spectrometer with four sample station at different unit operations of a mill.

FT-NIR ANALYER APPLICAIONS in PROCESS MONITORING and CONTROL

Process analyzers provide information on the process, but alone can only utilized for monitoring. Process control strategies need to be integrated with the measurements to obtain a pay-back and to provide incentives for further optimization. Below, we provide some examples of recent projects and case studies relating to the use of FT-NIR analyzer technology for process measurements coupled with advanced control.

Causticizing Control

The main purpose of the causticizing process is to maximize the conversion of sodium carbonate to sodium hydroxide while simultaneously producing a strong, uniform white liquor and lime mud particles that are easily separated. Unconverted sodium carbonate in white liquor is an undesirable deadload that increases energy consumption and chemical losses, reduces equipment capacity, and creates operating problems [35]. Causticity (CE) is inversely related to the sodium carbonate deadload and, therefore, the aim of most causticizing control systems is to regulate the CE of the white liquor to a uniformly high value close to the equilibrium CE, but without over-liming. Over-liming causes a completely different but no less severe set of operating problems characterized by low mud settling rates, free-lime in the white liquor, increased digester and evaporator scaling, and low lime kiln thermal efficiency. The correct lime dosage is difficult to determine since it depends on the CE target value, the flow rate and chemical composition of the green liquor, and the availability, reactivity and bulk density of the lime.

Common practice in causticizing process control is to regulate the incoming green liquor total titratable alkali (TTA) upstream, starting at the smelt dissolving tank and with an optional trim just downstream of the green liquor clarifier, and then to control the conversion of sodium carbonate to sodium hydroxide in the slaker/causticizers through the regulation of CE. Careful choice of the TTA and CE targets, followed by good regulation around those targets, will produce uniform white liquor at the target effective alkali (EA). Despite the fact that it is not overly complex, there are still very few mills that have closed-loop control of the causticizing process. This is likely due to several factors, including; a continued dependence on conventional measurements such as temperature, density and conductivity that are subject to scaling, extremely long process dynamics, and a lack of high quality liquor composition measurements.

Here we summarize the results obtained from applying the FT-NIR technology and a new causticizing control system [35-36] that combines liquor composition measurements and model-based inferencing of lime quality to achieve stoichiometric lime dosage control.

The system is able to integrate all of the available measurements and, thereby, minimize the need for manual intervention. This includes conventional measurements such as temperature and conductivity as well as liquor composition measurements from the above discussed analyzer. Figure 4 is a schematic of the CE control strategy. The reader is referred to [36] for further information.

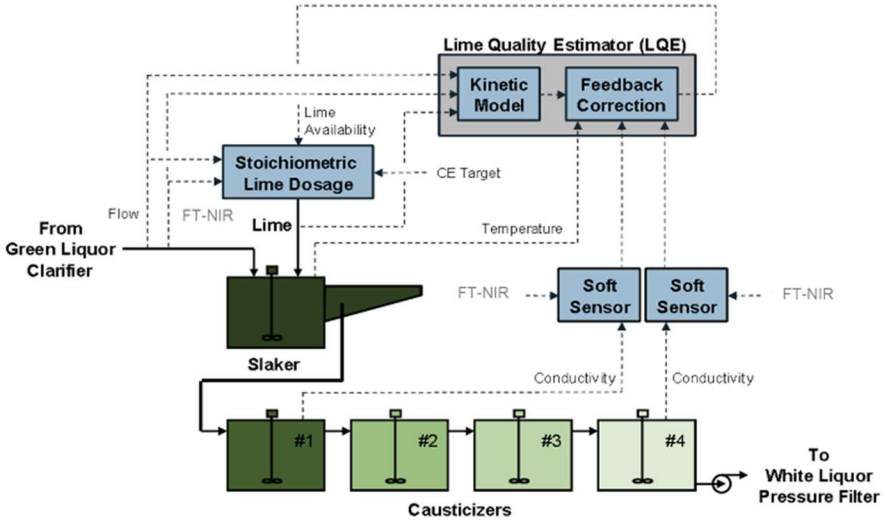


Figure 4: FT-NIR based Slaker and CE control which incorporates all information including online FT-NIR values for liquor

CASE STUDIES

Mill A

Mill A produces approximately 1850 ADMt/d of bleached kraft pulp, primarily from hardwood with occasional campaigns of softwood. The causticizing process consists of a slaker, 4 causticizers and a white liquor pressure filter. In 2010 the process was equipped with an online FT-NIR liquor analyzer. The design, installation and startup of this system are fully documented in [34]. Measurement points include raw green liquor, clarified green liquor (CGL), classifier overflow (CO), 4th causticizer (C4), final white liquor and weak wash. The analyzer continuously cycles through a schedule consisting of CGL, CO and C4 samples, since these measurements are used for control. This provides a sampling period of 25-30 minutes for each stream, which is close to the ideal sampling period for causticizing process. The remaining streams are inserted into the schedule on a time basis (i.e. every two hours, twice per shift, etc).

Results from the implementation of the supervisory control using the analyzer's results are shown in Figure 5 – 6. The lower portion of each graph shows the manipulated variable to achieve the desired target results, as shown in the upper portion. TTA trim control reduces the TTA variability and achieved target values, both variable being critical to feedforward control of slaker. Automated manipulation of lime screws provide improved CE control as anticipated by lime quality variability.

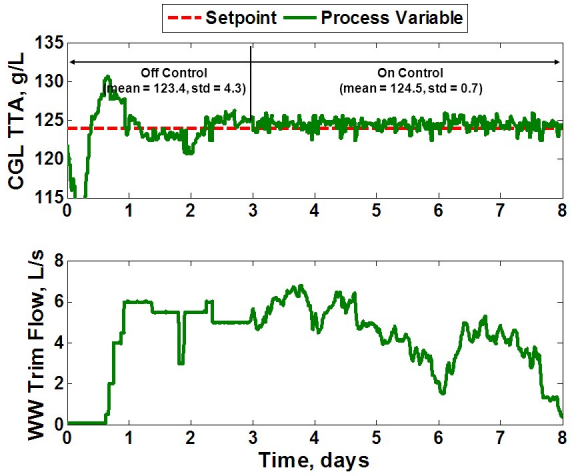


Figure 5: CGL TTA ON versus OFF control. Top graph illustrates reduction in variability with control while bottom graph shows the needed weakwash trim flow adjustments to maintain steady TTA.

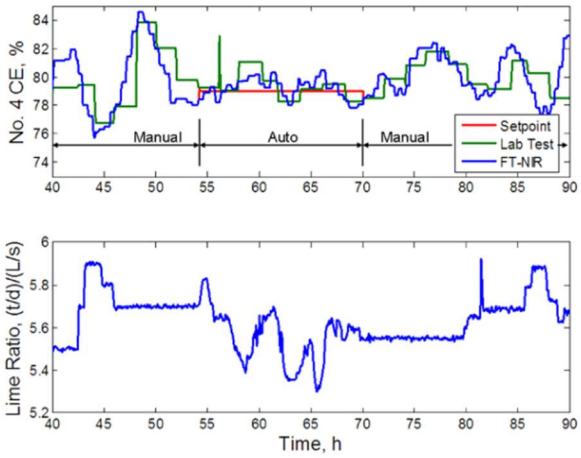


Figure 6: #4causticizer CE ON versus OFF control. Top graph illustrates reduction in CE variability with control while bottom graph shows the needed lime ratio adjustment required to maintain steady #4C %CE.

Mill B

Mill B is a softwood kraft mill in the interior of British Columbia, Canada. The mill produces roughly 800t/d of fully bleached NBSK. Similarly, the causticizing process includes a slaker and four causticizers and utilizes a pressure filter for white liquor clarification. Historically, the mill's recaust operation achieves an average CE of 74.4% utilizing operator tests and manual adjustments. The mill actually did have conductivity control but it has since been. For this mill, an ideal CE of 79 – 80% for the final WL but operators cannot run near this target due to infrequent testing and inaccuracies of manual testing.

An FT-NIR system was installed at the mill to measure a single stream, the CGL liquor for TTA control as well as feedforward slaker and CE control, while the feedback control relied on the slower manual testing. Over a six months period, the average CE was raised from 75% to 81% while using the FT-NIR based control strategy. Table III summarizes the economic benefits when one compares six months before and after control implementation.

Table III: Economic Benefits, Mill B

Sl. No.	**Performance Variables**	**FT-NIR-80% final CE**	**Pre FT-NIR**
1	CE estimate at # 3 Causticizer (%)	78.90	74.40
2	Av. WLPF Pressure (psi)	8.1	8.00
3	Av. Hot lime Screw Speed (%)	47.70	46.60
4	Av. Purchased lime Screw speed (%)	0.30	3.10
5	Av. GL flow to Slaker (USGPM)	679.50	698.00
6	Av. Lime Addition Rate (g/USG of GL)	180.70	141.59
7	Total Hot lime added (Tons/day)	145.68	142.32
8	Tota Purchased lime added (Tons/day)	0.51	5.27
9	Total lime added (Tons/day)	146.19	147.58
10	**% of purchased lime in total lime added (%)**	**0.35**	**3.57**
11	Total lime added for same GL flow rate (Tons/day)	146.19	138.54
12	**Adjusted quantity of purchased lime for same GL flow (Tons/day)**	**0.51**	**4.94**
13	Av. Lime quality number	1.20	Not measured
14	**Av. Digester production rate (ADMT/Day)**	**783.00**	**781.00**
15	**Av. steam flow to Evaps for same Digester production (klb/hr)**	**169.00**	**176.00**

Mill C

Mill C produces approximately 1000 ADMT/d and has two fibrelines, SW and HW and is equipped with one common chemical recovery and recausticizing plant. The recausticizing plant is fed from two green liquor clarifiers into one slaker. Two lime silos (one for purchase lime and one for reburnt lime) allow for metered lime addition into the slaker. From the slaker, a train of three conventional causticizers is then followed by one turbo causticizer. White liquor is then pumped to two clarifiers before pumping pumped to the WL storage tank. The bottleneck for the mill is the recausticizing plant's ability to produce sufficient white liquor to keep up with digester demand. Since the implementation of the supervisory control using true liquor compositions from FT-NIR, the mill has documented the following benefits

Decreased Liquor Deadload

The improvements in recaust process stability have resulted in a significant and measureable decrease in carbonate deadload in the liquor cycle. Since the implementation of the fully optimized strategy, the mill has observed a decrease of 1.75g/L (as Na_2O) in carbonate in the white liquor feeding the digester for the same CE target. This indicates that prior to commissioning the analyzer and control, the mill was overliming. This is likely due to the fact that operators will tend to overlime to ensure that they produce sufficient WL strength and not fall behind with digester demand.

According to Grace et. al. [37], and Empie et. al. [38], this reduction in deadload content also provides an energy savings component across the digesters (in heating up water that is required to transport the deadload), evaporator train, and recovery. The calculated energy savings from the decrease in carbonate deadload is equivalent to increasing CE by 1%, and on the order of 38GJ/day. Further energy savings can be realized by actually increasing the CE target by 1 – 2%.

Reduced Purchase Lime Usage

As a direct consequence of reduction in deadload the mill has observed a 50% decrease in purchased lime usage during normal operations. This result is consistent with other mill implementations [35]. More data will further validate this observation and may show an even more significant reduction in purchased lime.

Increased Maximum Sustainable Recaust Rate

The recaust maximum sustainable rate (MSR), with the implementation of the FT-NIR analyzer and advanced control strategy, has increased from 1050gpm to as high as 1080gpm. This extra 30gpm liquor translates to two extra blows per day, or 40 ADMT/d, of dense hardwood. A more conservative MSR for the process over very long periods utilizing fully tuned control is around 1065gpm, which could translate into 1 extra blow per day or 20 ADMT/day of dense hardwood. Assuming a production schedule of 15 days per month on dense hardwood, or about 170 days per year, and good mill runnability, up to 3100 ADMT/year of additional dense hardwood could be produced. The higher recaust MSR achieved with the FT-NIR and advanced controls strategy has shifted the mill bottleneck from the recaust to the recovery boiler, which is where a mill's bottleneck should be.

Reduced Scaling in Recaust Train

It has been observed that with improved green liquor TTA control, conditions leading to recaust train scaling occurred far less often than prior to the FT-NIR analyzer and advanced control implementation. The mill will continue to document these observations, but anticipate that cleaning cycle for the green liquor heater/cooler will be reduced, along with the causticizer train high pressure washes. The mill plans to inspect and document the scaling during their annual shutdown and possibly quantify scaling propensity.

The documented economic benefits arising from these case studies show that the FT-NIR based control strategy significantly improved operation while reducing deadload content. As the mill improves one area of operation, the benefits can be seen propagated to other unit operations. ROIs are typically six months to a year plus additional benefits associated with entire pulping and chemical recovery processes. As the chemical recovery unit is vital to the pulping processes, its improvements will play a major impact on the competitiveness of the mill.

DIGESTER AND RECOVERY BOILER OPTIMIZATION

Development of measurement devices aimed at digester control has not seen much activity. Traditional information regarding blow line Kappa number as well as H-factor is well documented. Modo and Modo-Chemetics added the measurement of black liquor REA (residual effective alkali) using a modified titration technique to further refine the control. However, parameters including chip moisture content, WL EA and sulfidity, as well as reliable black liquor REA have been mostly carried out as manual testing. FT-NIR technology was first applied as process analyzers for weak black liquor (WBL) REA and white liquor feed to the digester has seen good acceptance and the reader is referred to references [26-28] for further information.

In addition to WL EA and WBL REA for the digester, the FT-NIR system has been calibrated to measure lignin, organic, and inorganic solids content. Figure 7 shows a plot of the FT-NIR results for digester extraction black liquor properties as the digester migrates from softwood to hardwood. The trend shows that WBL extraction REA, lignin, and organic content dropped while the inorganic solids remain relatively constant. This is consistent since the inorganic charge (WL) to the digester remained the same during the transition.

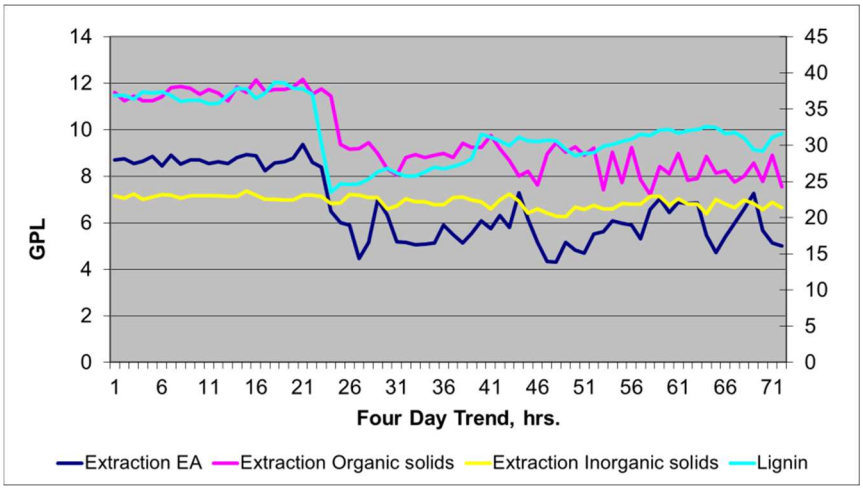


Figure 7: Extraction WBL properties trend showing changes in lignin and organics as a result of softwood to hardwood transition.

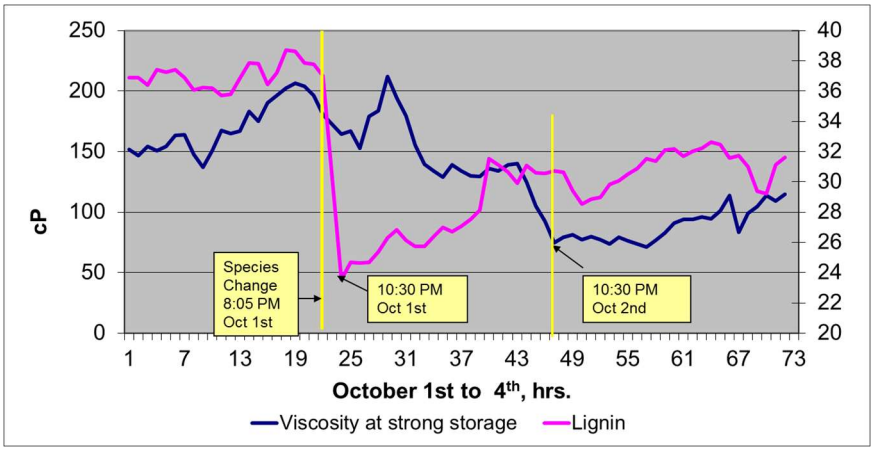


Figure 8: Viscosity and lignin content showing onset of liquor change with drop in lignin and associated viscosity drop 1 day later.

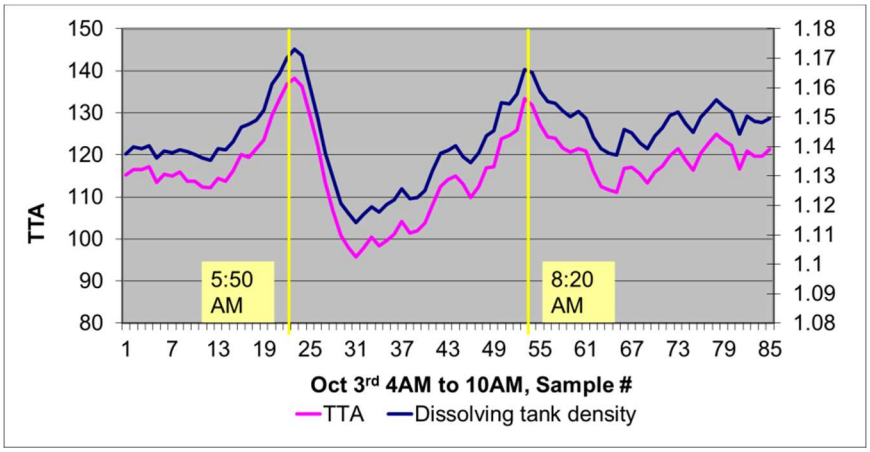


Figure 9: Smelt dissolving tank FT-NIR TTA and density capturing smelt run-off.

If we monitor the viscosity changes with the associated change in lignin content (figure 8), we see that the drop in lignin content hit the strong BL storage one day later (Oct. 2), with viscosity dropping to a low of ~75cP. On the third day, the mill experienced a massive run-off. The level in the dissolving tank spiked due to a surge in smelt flow which in turn triggered large flows of weakwash to compensate for high TTA. FT-NIR analyzer installed at dissolving tank for monitoring raw GL TTA captured the spikes (figure 9) and was confirmed and validated by density measurements. The run-off, when diagnosed, was attributed to changes in liquor properties (due to lignin content changing as captured by FT-NIR) resulted in lower viscosity. Since the firing temperature was left high, small droplets and inflight burning occurred, resulting in poor bed formation. To address this, the firing temperature was dropped.

It is clear that the information provided by the FT-NIR at the WBL extraction provided valuable information that could be used to avoid such a run-off, particularly if tied to some high-level monitoring system. These additional properties relating to the WBL, along with information such as carbohydrates, could and will play a vital role as we develop better understanding of digester operations and how these properties influence evaporator and recovery boiler operations.

CONCLUSIONS

Pulp mill applications using FT-NIR spectroscopic techniques have matured significantly. On-line measurement of key process variables in the kraft chemical recovery process has been well documented with applications ranging from dissolving tank TTA control, slaker control, causticizing CE control, digester monitoring and control, as well as chlorine dioxide generator control, all with good ROI for the industry. Case studies have shown that CGL TTA variability can be reduced by more than 50% while WL CE variability can dropped by more than 40%. The control strategy at the slaker has optimally controlled lime dosage, leading to carbonate deadload reduction, even without increase in CE target as well as a decrease in purchased lime consumption by 50% (under normal kiln operation), and reduced scaling in the recaust train. More importantly, smoother operations have led to an increase in recaust maximum sustainable liquor flow rate by as much as 30gpm, leading to a potential increase in pulp production up to 3100 ADMT/year for dense hardwood.

Future trends indicate that FT-NIR technology will continue to play an important role as a sensing platform arising from its inherent flexibility and ability. As the industry moves into value added products from cellulosic feedstock, FT-NIR technology is poised to take advantage of its capability to sense organic compositions. It is anticipated that spectrometric techniques will continue to grow and see new applications that will include solid woods and wood composite materials as new hardware becomes available.

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