

Applications of Reflectance Spectroscopy in Forest and Forest Products

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Abstract

Forests are a natural resource of major economic significance to Canada and many other countries. For example, Atlantic Canada forest industries exported over \$3.9 billion in wood products and had a direct employment of 37,900 persons in 2003. Secondary and value added products, particularly that of Canadian northern bleached softwood kraft market pulps represent revenues reaching \$7.5 billion in 2007. Canada leads the world in wood pulp exports. In addition to direct employment, the industry has an indirect effect through demand for goods and services and diversifies income sources for many communities.

More recently, wood is recognized as a renewable resource and can sequester CO₂. There is increasing emphasis on using wood for energy, bio-fuels and bio-chemicals production. Timely knowledge of wood characteristics is essential to ensure that the right fiber is directed to the appropriate wood manufacturing company at the right time and right cost to produce the right product.

Agenda 2020 has identified that a number of key variables that need to be measured accurately along the wood processing, from trees to logs to lumber and to secondary processing such as pulp and paper products. Measurements of wood and processing parameters will allow process monitoring and control,

leading to consistent high product quality at minimal costs and energy consumption.

Reflectance spectroscopy, particularly visible-NIR spectroscopy, has shown much promise as an adaptable technology that would meet the requirements for fast, non-contact, and process capable as online and at-line analysers for monitoring wood and wood products properties. This paper will discuss the application of reflectance spectroscopy as an at-line analyzer for measurements of kraft pulp properties such as kappa number and brightness, and an online analyser for pulp mill lime mud properties measurements. The paper will focus on adapting reflectance spectroscopy for process monitoring and control applications in processing requiring 24/7 operations.

Background:

In Canada, wood logs are selectively harvested from the plantations or crown and private land. The trees are fell, removal of thrash, and cut to dimensions. The logs are then stacked and transported to sawmills. The logs are stored and conditioned before they are sorted and cut to dimensional lumbers. Residual sawmill residues are then transported to pulp and paper mills where the wood chips are processed by chemical pulping (Kraft Process) or by mechanical action (Thermal Mechanical Pulping) to produce wood fibres. The pulp fibres are then bleached with

various chemicals, including chlorine dioxide, peroxide, and other bleaching agents, to produce white pulp. The bleached pulp is then fed into a head box which injects pulp slurry of known consistency (grams of fibres per liter) on a porous fabric moving conveyor. The fibre slurry dewater and results in a non-woven fibre matrix, which when dried through pressing and indirect heating, forms a fibre matrix to produce paper. Kraft pulping produces pulp fibres of longer length but at a lower yield (~40-42%) while thermal mechanical processes (TMP) produces higher yield (>90%) but lower fibre lengths due to shearing action of the mechanical grinding. Longer fibre lengths, in combination with a more collapsible fibre (along with other parameters), tend to form stronger paper strengths. Paper strength is especially needed for high quality paper which does not rip or break easily, such as origami paper and other specialty paper, including facial and bathroom tissues.

Figure 1 shows a cycle of Kraft pulping. Kraft pulping is performed by cooking wood chips in highly alkaline liquor which selectively dissolves lignin and releases the cellulosic fibres from their wooden matrix. The two major active chemicals in the liquor are sodium hydroxide and sodium sulfide. At the beginning of the kraft process, “white liquor” (high caustic solution of ~90g/L of NaOH (as Na₂O)) is fed into a digester or pressure vessel and allow to heat up to dissolve the lignin to form black liquor or spent liquor. The black liquor, which is low in caustic (< 20g/L NaOH as Na₂O) is extracted from the digester and sent to liquor evaporation units to remove water and concentrated to ~75% solids. The concentrated black liquor or “strong black liquor” is rich in dissolved organics from the wood matrix

(carbohydrates) and inorganics (sodium and sulfur) is then burnt in a recovery boiler to generate power and steam and a molten smelt rich in inorganic compositions. The steam in turn provides heat for drying the pulp, paper processing, and electrical power generation. The smelt is re-dissolved with contaminated water to generate “green liquor” which is rich in sodium carbonate and sulfur (as sulfide). The green liquor, or more accurate the carbonate, is reacted in a heterogeneous reaction with calcium oxide (CaO) to regenerate the white liquor that was originally feed into the digester for wood chip digestion.

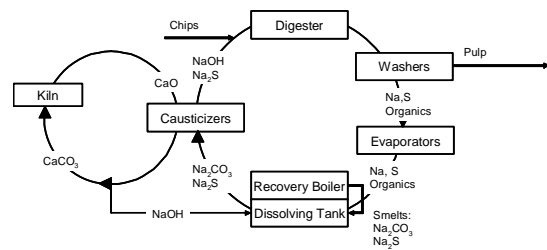


Figure 1. Kraft process operations and chemical recovery processes.

Calcium oxide, after reacting with sodium carbonate, generates CaCO₃ or “lime mud”. The lime mud is separated from the white liquor through special pressure filters or through settling tanks. The lime mud is then washed and concentrated using vacuum drum washers to produce mud solids of >70%, which in turn is fed into the rotary kilns where it is heated to 1200 degrees Celsius to first drive off the water before forming CaO. The knowledge of lime mud properties such as the solids content as well as the residual chemicals that was not washed from the mud and other inorganics will provide necessary data for controlling the operation of the mud washer and dewatering operation, which in turn has tremendous impact on the energy consumption of the kiln.

Overall, the Kraft pulping process relies heavily on the chemical recovery processes. When these cycles, the sodium-cycle and calcium-cycle, operates properly, chemical recovery/recycle is in the upwards of 95%. As such, the economic viability of the mill is highly dependent on the operations of the efficiency of the chemical recovery processes.

Similarly, other unit operations of the pulping operations are also critical to the overall success of a pulp mill, including pulp bleaching. In particular, the pulp fibres after digestion, is a brown fibrous material and brown in colour due to residual lignin content in the fibre walls, referred to as the kappa number of the pulp for the amount of permanganate require to fully oxidize the lignin. The brown fibre or “brown stock” is then washed to remove chemicals and the residual lignin is bleached through a five-stage tower with washing between each bleaching sequence, to produce a high brightness and whiteness pulp for paper production.

Timely knowledge of the residual lignin in the fibre and the brightness of the pulp from the digester product (brownstock) to the final white pulp are required. Kappa number of the pulp after digestion can be fed back to control digester operation so as to provide pulp at the targeted kappa as well as feedforward for controlling the bleaching chemical additions to achieve final brightness. In addition, the ability to measure kappa number and brightness of pulp in between bleaching towers further provide opportunities to reduce bleaching chemicals and reduce cost.

Near-Infrared reflectance spectroscopy has shown great potential as an adaptable technology that would meet

the requirements for fast, non-contact, and process capable for online and at-line measurements of such wood, chips, pulp, and processing chemicals properties. While there are many other potential applications of NIRS in forest and forest products [1-10], there have not been many commercial applications of instruments or analysers for heavy processing industry as routine measurement tools. Unlike the grain industry, wood and wood related products are bulky and difficult to handle with aqueous chemical reactions. This paper will focus on and discuss the application of reflectance spectroscopy for measurements of pulp kappa number (an at-line Vis-NIR analyzer) and lime mud properties measurements (an on-line Vis-NIR analyzer). Particularly, we the paper will highlight the success, challenges and shortcoming of adapting reflectance spectroscopy for on-line and at-line measurements in a heavy industry which operates 24/7 with high risk of out-of-spec operation due to instrument/analyzer errors.

Pulp Kappa and Brightness Measurements: KBTM

The accurate measurement of the Kappa number of chemical pulps obtained from rapidly varying furnishes had remained an unresolved issue for chemical pulp manufacturers. Mills that use residual sawmill-chips from various locations and those producing specialty grades are especially affected because of the variability of chip quality from various suppliers. This issue is even more prevalent now due to a shortage of available market wood chips and the fact that pulp and paper mills are being forced to purchase chips geographically distant from their manufacturing facilities. All too often, pulp produced during grade changes, or from poorly

characterized chip species mixtures, has to be downgraded because of a high proportion of improperly cooked pulp since each wood chip species cooks differently. Analysers capable of rapid determination of kappa number could help mills greatly reduce Kappa number variation, bleaching costs and the amount of off-grade pulp.

Current digester control algorithms use the blow-line Kappa number in conjunction with the H-factor [11] for controlling the pulping operation. Generally, Kappa number measurements are required as a feedback parameter to allow for adjustments in the liquor charged to the digester at the various zones of the cook and, much more critically, as a feed-forward parameter for control of the bleach plant. Higher kappa number pulp requires higher charges of bleaching chemical to reach target final pulp brightness levels, particularly during the oxygen-delignification stage.

The measurement of residual lignin content in pulp has been traditionally done on an hourly basis as a laboratory analysis according to TAPPI and PAPTAC standard method T236 [12-13] which use a back titration of residual permanganate with potassium iodide. However, the method requires extensive workup and can take 30 to 60 minutes per sample. Jiang *et al.* have improved on this standard technique by semi-automating the titration process with an automatic, multi-sample titrator [14]. The time delay limits the utility of this method for feedback control of the digester and for feed-forward control of the bleach plant.

Various methods for the at-line and online methods and analysers have been developed and they are discussed

elsewhere [15]. This paper illustrates the application of visible-near infrared spectrometer to measure kappa number and ISO brightness of wet pulp samples with varying consistencies and pulp furnishes. The Vis-NIR system was further packaged for mill environment and validate at three mill technical laboratories. Results showed that the Vis-NIR was capable of measuring wet pulp kappa number and ISO brightness with good accuracy and repeatability as well as having a lower detection limit as compared to manual and autotitrators. When compared to reference techniques, the Vis-NIR provided correlation with R^2 ranging from 0.95 to 0.99. Similarly, for ISO brightness measurement, results obtained with the Vis-NIR pulp analyser correlated well with reference technique such as the Technidyne and the DataColor systems with R^2 ranging from 0.97 to 0.99.

Experimental

Sample preparation

Pulp samples from process lines were collected and dispersed in water. Pads were made by filtering the dispersed pulp through a Buchner funnel lined with a wire mesh or paper-wire screen. Roughly 500 ml 2% consistency pulp, taken from the vat side of a washer or decker, is filtered through a 9.5cm (3.75") Buchner funnel. The sample is washed twice with 1 L of clean water until the pulp filtrate is reduced to a drip and the filtrate is seen to be clear with no contaminants. This would produce a pulp pad of approximately 11% consistency for the analysis. Calibration pulps were prepared to provide varying pulp pad consistencies. Sample analysis was done per PAPTAC and TAPPI standard for kappa number determination. ISO brightness

measurement was analysed with the Technidyne Technibrite optical brightness meter.

Equipment

Experiments were carried out with an Analytical Spectral Device Inc. (ASDI, Boulder, Co.) LabSpec Pro™ spectrometer capable of providing a complete scan from 350 nm to 2500 nm in 100 milliseconds. Spectral data was an average of 64 scans. A sample turn table, which has a 53.2mm illumination sampling spot size and rotates at approximately 22rpm, was used for sample presentation during spectral data acquisition. A standard halogen light source was used for illumination and the diffuse reflected light was collected with a fibre optic bundle recessed back in the holder and delivered the signal back to the three detectors.

Procedure

A white reference was first acquired using a Spectralon™ plate. The wet pulp pads are then placed under the illumination spot on the Turntable and rotated during spectral data collection. The height of the Spectralon plate was approximately equal with that of the samples. New reference was taken once an hour and more often during model development and calibration. Kappa number and ISO brightness values were automatically outputted to the screen and stored as data logs within the analyser. Repeatability results were acquired with the same sample, rotating in the tray while multiple spectral data are collected sequentially.

Calibration

Spectral data from various calibration dataset were correlated with standard methods for kappa number and ISO brightness values. In addition, sample

consistency was also incorporated as a component such that the calibration model would compensate for pad consistency variation. Grams32/AI and the PLS/IQ software package from Thermo Electron Corporation were utilized for all multivariate data analysis.

Results and Discussions

In one calibration (Mill-A), 111 samples were collected from process lines ranging from digester blowline (kappa 25) through to fully bleached pulp (kappa 0.9). The calibration pulps were taken every other day, once per shift, over a period of six weeks. The species mix within the mill is predominantly Lodgepole pine, up to 90%, with the remaining made up of other pines. By sampling over an extended operation period, we captured some pulp furnish variability in the calibration pulp. Figure 2 shows Vis-NIR spectra of pulp samples from W15 (washer) through to W65. The two dominant peak found at 1454 and 1930 nm are mainly due to water absorption as these samples are wet pulps. The difference spectrum, that of a W15 sample (ie. high kappa number) from that of W65 (ie. low kappa number), show that bands relating to lignin can be seen at various locations throughout the spectral region, as shown in Figure 3. As lignin is removed with bleaching, brightness increases and this band drop in intensity with bleaching. As such, the strong and large band between 400nm to 500nm indirectly relates brightness to the removal of lignin, consistent with UV-Vis measurement techniques. The correlation diagram shows that the visible spectral region is dominated by changes in brightness. Kappa number shows strong correlation above 1100nm in the NIR region. Lignin absorption can be seen at 1725nm, 1780nm, 1940nm, and

2130nm. Strong water absorption is noted in the correlation diagram with major bands at around 1460nm and 1930nm. These major water bands will shift with changes in consistency of the pulp. As such, a spectral region from 400nm to 2295nm was used for subsequent model development.

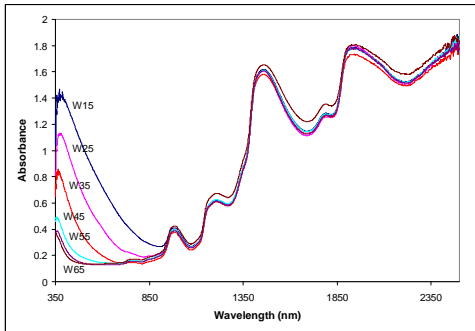


Figure 2. Vis-NIR spectra of wet pulp samples from brown stock(W15) washer through to fully bleached pulp (W65).

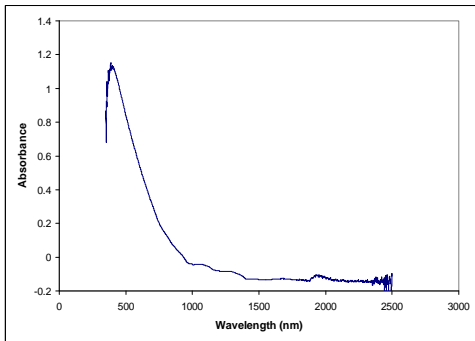


Figure 3. Difference spectrum between W15 and W65, showing strong absorbance associated with pulp brightness, indirectly related to lignin.

As reflectance spectra is dependent on pulp density, thickness, and water content, it was necessary to incorporate consistency variation in the training dataset. The calibration Pulp pads were made to varying consistencies by applying varying vacuum time and with and without the use of vacuuming aid or a vacuum pump with the Buchner funnel. The consistency of the pulp obtained in this manner varied from 8 percent to as high as 25 percent. At least

three levels of consistencies were made for each calibration pulp. Figure 4 shows a correlation of the Vis-NIR predicted kappa number versus titrated kappa number. For the same dataset, Figure 5 shows the correlation between Vis-NIR ISO brightness vs. Technidyne ISO brightness. Additionally, we can incorporate consistency as a component within the calibration. Figure 6 shows the correlation between Vis-NIR and oven-dry (OD) consistency determination.

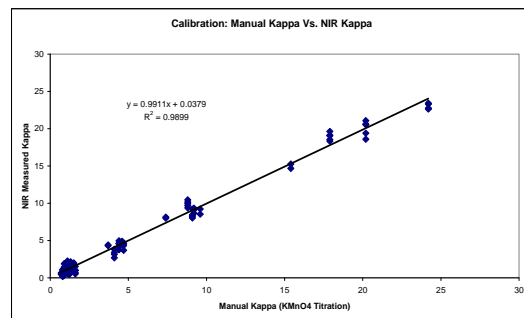


Figure 4. Mill-A Vis-NIR kappa predictions versus titrated kappa.

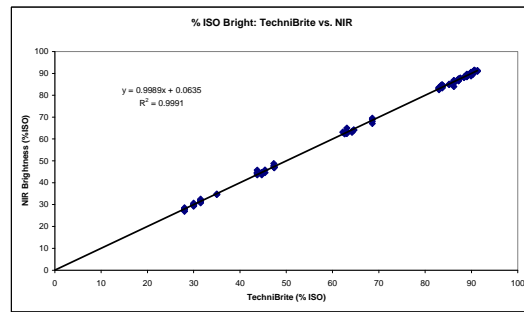


Figure 5. Mill-A Vis-NIR predictions versus Technidyne ISO brightness.

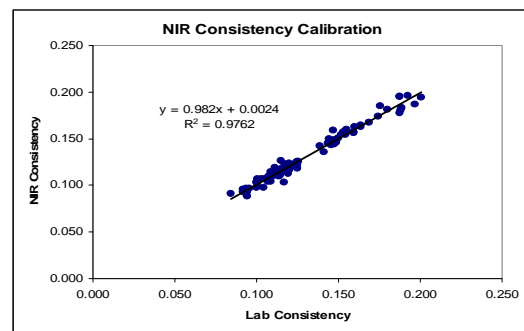


Figure 6. Mill-A Vis-NIR predictions versus oven-dry (OD) consistency.

Figure 7 shows the validation results obtained from Mill-A for kappa number, comparing Vis-NIR to mill titration. Good correlation was observed when validated against standard lab titration over a period of two weeks at the mill site. The standard deviation of the mill titrations were 3.9 kappa vs. 2.8 kappa number for the Vis-NIR technique, including process variability. A RMSEP (root mean-standard error of prediction) for the Vis-NIR dataset was 1.4 kappa number. The results indicate that the Vis-NIR technique is comparable to standard autotitrations when performed under strict laboratory conditions ($\sim \pm 1.3$ kappa), thus indicating that the technique is capable of providing accurate kappa number determination, directly from wet pulp.

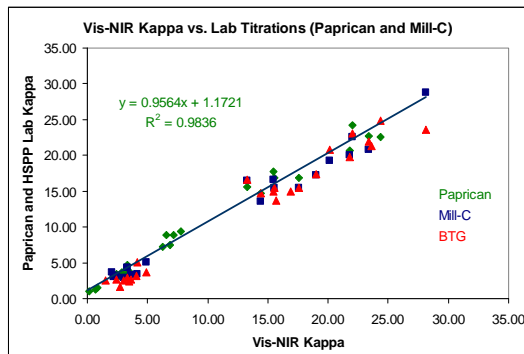


Figure 7. Kappa number validation comparing technical lab titrations, FPInnovations-Paprican titrations of same sample, and mill's online BTG kappa analyser.

The spectral data of the wet pulp was also used for ISO brightness measurements. Figure 8 shows the correlation obtained for ISO brightness at Mill-C vs. the Technidyne optical brightness system as well as the mill's Data Colour system. Figure 9 shows the correlations between the Vis-NIR vs. the QuickBrite system for ISO brightness. Even when the scale for brightness is expanded to provide a true look at useful

brightness range, the correlation between Vis-NIRS versus QuikBrite was very good. It is noted that we were not able to obtain good correlation initially with the QuikBrite until it was recalibrated against the Technidyne. Table I summarizes the results obtained for Mill-A. SEP is defined as the standard error of prediction and is used as an indication of the ability of the calibration to determine unknown pulp samples for kappa and brightness parameters. The ratio of performance to deviation (RPD) (Williams and Sobering 1993 [16]), calculated as the ratio of the standard deviation of the reference data to the Standard Error of Cross Validation (SECV), was also used to assess calibration performance. Determination of the RPD allows comparison of calibrations developed for different pulp properties that have differing data ranges, the higher the RPD the more accurate the data fitted by the calibration.

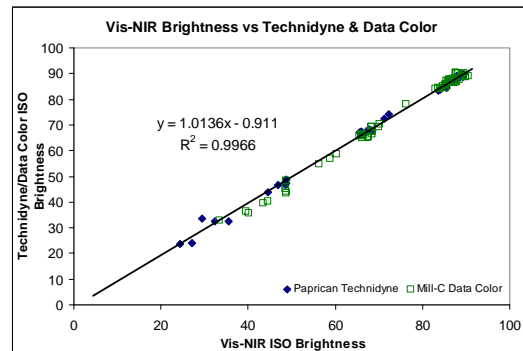


Figure 8: Vis-NIR pulp brightness validation results against the Technidyne reference technique as performed on dry handsheets.

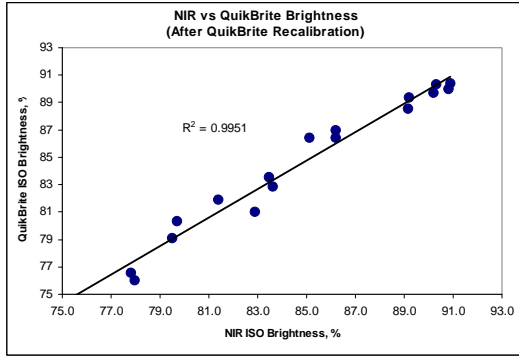


Figure 9: Vis-NIR Pulp brightness validation against QuikBrite technique, after the QuikBrite was recalibrated.

Table I: Summary of Mill-A results as performed by mill technical staff

Pulp Property	R ²	SEP	RPD
Kappa No. (Lab)	0.90	1.4	2.5
Kappa No. (Online BTG)	0.75	2.1	4.4
Kappa No. (Operator)	0.37	6.1	1.1
ISO Brightness (Technidyne)	0.95	1.4	4.8
ISO Brightness (QuikBrite)	0.99	0.6	3.3
ISO Brightness (Operator)	0.75	22.2	2.5

Table I shows that Vis-NIR technique correlated well with all reference techniques performed by mill technical staff, with the exception of the operator titrated kappa numbers. It is expected that the best correlation is seen with the reference methods for the two parameters (lab kappa titration and Technidyne ISO brightness). Good correlation was also obtained with the QuikBrite, but was only observed after re-calibration of the QuikBrite. Vis-NIR reflectance spectrometry technique shows good correlation with the online kappa number analyzer and could be used as a secondary technique to check these systems. The mill installation shows that Vis-NIR technique can accurately measure wet pulp kappa

number and ISO brightness, as performed by mill operation. Measurement time is approximately 3 minutes, including preparation time.

Factors Influencing Measurements

Consistency and water content affected the reflectance spectra and had large influence on the measurements. As such, variations in pulp pad consistency were induced and calibrated for in the calibration. Initially, the consistency was predicted from the Vis-NIR spectrum to ensure that sample preparation provided a pad of similarly water content that is within the calibrated range. By incorporating the consistency variation within the model, one has effectively removed the onus on the tester to prepare a pad of specific consistency. This further reduced the sample handling and increased ease of use with the Vis-NIR system.

Pulp pad thickness was also shown to have an effect on the spectral signature, particularly of those samples that are fully bleached. Specifically, the pad thickness or pad density affected surface characteristics and formation. For unbleached pulp, the fibres are stiff and does not collapse with pressure/vacuum and formed a thicker pad, though less dense. Bleached pulp is more collapsible and forms a thinner pad though higher density. This is further compounded with the use of a standard halogen lamp which is non-collimated. As such, height variations affected not only reflectance intensity, but also illumination spot size. In addition, light penetration through pulp pad was also observed, especially for pad samples from fully bleached pulp with thickness below 1cm.

Frequency of referencing and Spectralon handling was also found to be extremely important for accurate measurements of pulp properties. It was found that new reference update was required once per hour to maintain good accuracy from the predictions. However, this is complicated by the fact that the reference is a white Spectralon panel, which, through normal handling by operators and testers, soil the material surface, and ultimately shift the prediction results. This high frequency of referencing and reference material handling has proven to be a major issue in causing shifts in measurement results.

New KB™ (Kappa Brightness) Pulp Tester

To commercialize the KB System, we have found that it was necessary to package the spectrometer, computer and sampling chamber in an integrated cabinet (Figure 10a). Due to the corrosive environment, the material of choice is stainless steel (SS316). Working with our industrial partner, we developed an automated sample preparation device (Figure 10b) which automates the washing and pad forming procedures, minimizing the requirement of pulp mill testers and operators. The automated sample preparation station addresses two of the factors influencing the measurements: pad consistency and pad height variation.



Figures 10a (opp.) and 10b (above): illustration of ASD's KB™ pulp tester and the automated sample preparation device.

To minimize variations in pad consistency, the automated pulp pad sample station washes and presses the pulp to a constant pressure. Thus, a more consistent force is used for dewatering during pad formation. To address the height variability, the sample cup used for washing and pad formation can be separated at the screens level and at mid-cup level. As such, the sample cup itself acts as the sample holder and stage. Measurements are carried out on the pulp with the screen-side up, which is a constant height. A video showing the operation of the automated sample preparation device can be seen at the ASD website (www.asdi.com). Automated internal and external referencing will need to be address in the future. Without automated referencing, full adoption of any reflectance spectroscopy technique for processing operations requiring 24/7 measurements will not occur. The frequency and handling of the reference material will induce errors. Ideally, compensation for light bulb fluctuations, detector response and temperature should be compensated for during each measurement.

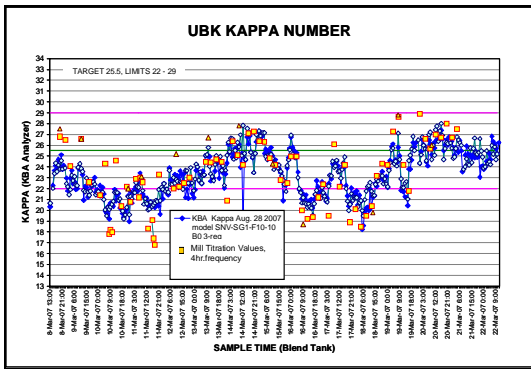


Figure 11: QA/QC data trend showing kappa number variability with mill processing. The upper and lower bands indicate acceptable range for specific client.

The KB pulp testers have now been installed and operational at over 10 pulp and paper mills. Figure 11 shows the use of the KB pulp tester to monitor final unbleached pulp kappa number destined for cement board applications. The tolerance lines brackets the upper and lower limit of acceptable pulp kappa number by the client. The rapid measurements of pulp kappa number allow each pulp bale to be stamped with a quality parameter to ensure compliance.

Online Lime Mud Properties Measurements: QS7000

The operation of the liquor recovery cycle of a kraft pulp mill is very important to its economic viability. Much of the costs associated with the pulp mill are attributed to the operation of the recovery cycle. Without efficient chemical recovery, cost of pulp production is not economical and sustainable. In a kraft mill operation, lime mud is separated from the active cooking liquor and is then thickened and washed with a rotary drum washer to recovery chemicals. Typical mud solids content after the drummer washer is in the range of 65% to 80% (wet weight basis). The thickened mud is then doctored off and fed into the kiln, via a

screw feeder, where is its heated to 1200 degrees Celsius to, first drive off water, and then convert the CaCO_3 to CaO (lime), for use in the generation of white liquor. At current, measurement of mud solids is done manually, once every 2 – 4 hours.

With kiln operation considered as one of the highest consumer of fossil fuel, with the rising cost of fossil fuel, small incremental savings could results in significant energy. It is suggested that if accurate lime mud moisture could be measured prior to entering the kiln, this information could be used to control and optimize kiln operation. A one percent increase in lime mud solids could result in 1% reduction in fuel consumption. For a 1000 t/d mill, a four percent increase in mud solids (68% to 72%) could result in \$300K to \$500K in fuel cost.

In addition, variations in mud moisture have been shown to have impact on operations of the rotary kiln. For example, a study reviewing 14 lime kilns have shown that variation in mud moisture was one of the main operating parameters influencing the kiln temperature profile, the location of the recarbonation zone and the propensity for ring formation. Kilns that had greater variations in mud moisture content had more ring formation problems, regardless of the average solids content of the mud feed. Measurements of silica (SiO_2), colour and other non-process elements, which can not be currently measured online, could provide additional information for further understanding of kiln operations and further reduce fossil fuel consumption.

Equipment

Experiments were carried out with an Analytical Spectral Device Inc. (ASDI, Boulder, Co.) FieldSpec 3™ spectrometer coupled to an over-the-conveyor probe head (QS7000). The probe head featured an automated internal calibration standard which can be run at any time between sample analyses. Spectral data was an average of 128 co-added scans. A sample turn table, consisting of a potter's wheel and a tray completes the sample handling setup. During calibration development, mud samples were collected from the mill, sealed in plastic bags, and shipped to FPInnovations for spectral data acquisition. Samples were spread over the tray and spun at a constant rate equivalent to 5 ft/s linear motion. The optimal distance from the sample to the probe was approximately 29-30"

Figure 12 illustrates the mill installation of the QS7000 probe-head over the conveyor. A SS316 cabinet housing the spectrometer is mounted nearby with fibre-optic bundle connecting the probe-head to the spectrometer. For online acquisition, 600 co-added scans were used for spectral data acquisition. The internal reference updating was carried out once every 10 minutes. This configuration allowed the analyser to monitor a representative sample from the process and maintain calibration. External reference is done during light bulb changes.



Figure 12. Lime mud solids analyser installation on a mud-feed line feeding a kiln. The probe-head is mounted 29" above the sample surface with an illumination spot of 6".

Calibration

Spectral data from various calibration dataset were correlated with standard methods for mud solids (oven-drying). Whiteness and brightness and other colour properties of the mud were correlated to standard CIE-LAB values, such as provided by the Technidyne. Non-process elements including Al, Fe, Si, Ca, Na, Mg, and Na were done by Atomic Absorption Spectroscopy. Grams32/AI and the PLS/IQ software package from Thermo Electron Corporation were utilized for all multivariate data analysis.

Results and Discussions

As mentioned above, the adoption of reflectance spectrometry as process analysers, there must be means to automate referencing and to maintain calibration over time. The QS7000™ provides a mean to update an internal reference (Spectralon), internal to the probe-head. During initial installation, or replacement of the illumination source, an external reference is carried out at a set distance from the probe-head to capture reflectance characteristics. Through a proprietary software

calibration, the external and internal references are mapped. Subsequent referencing can be simply done through the internal reference. This is truly critical for online process analysis. Without proper updating of references, any analyser would drift and fail. In addition, the QS7000 also features a collimated beam to minimize the effects of height variations associated with the mud filter operation. The probe-head was mounted nominally at a distance of 29" from the sample surface, thereby ensuring minimum risk of samples coming into contact.

The calibration model for wet mud solids content was constructed with 150 mill process samples collected over a period of three months. Mill samples were used as-is and samples were taken to perform standard OD for solids content immediately after spectral acquisition. The range in mud solids were from approximately 75% to 91% solids. Figure 13 shows the calibration model for mud solids, illustrating a correlation (R^2) of 0.95 and an SECV 0.30%.

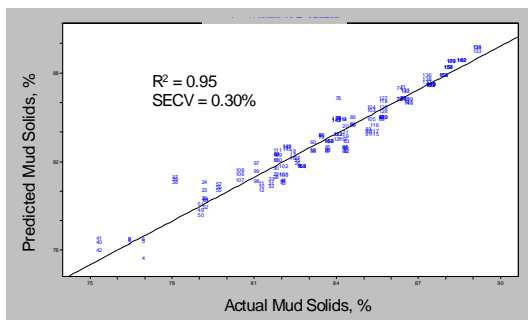


Figure 13. Lime mud solids calibration using an online probe-head (QS7000) and a FieldSpec 3 (ASDI).

The calibration model was transferred to the online analyser and used for continuous mud solids monitoring. Validation results showed that the calibration model can accurately predict

solids content of lime mud moving on a conveyor. Grab samples taken immediately after the probe-head on the conveyor surface provided an accurate (SEP) within 0.8% and a correlation (R^2) of 0.98, as shown in Figure 14.

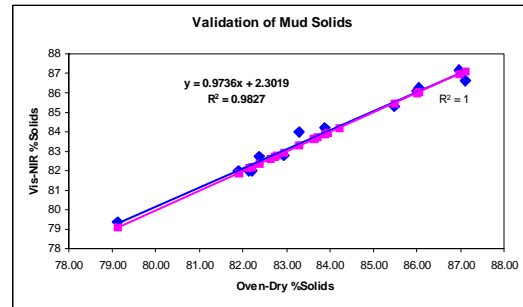


Figure 14. Validation of mud solids measurements using an online probe-head (QS7000) coupled to a FieldSpec 3.

Process data analyses were carried out to see what process parameters have the highest impact on mud solids content. The data show that, over a five day period, the mud solids content had an average value of 83.4% with a span of 77% to 92% and a standard deviation of 1.75%. The highest variation in mud solids is a direct result of a device used to clean the surface of the drum washer, a Continuous Precoat Renewal (CPR). The CPR utilizes a water jet to cut through the layer of the mud on the drum washer to clean the wire screen. Due to the operation of the CPR, excessive water is sprayed on the surface and did not allowed the mud cake/layer to form. As a result, the solids content dropped in relation to the CPR position, specific near position zero. Figure 15 shows an illustration of the process swing associated with the CPR operation. A swing of 4% in solids content is observed, with a quick drop in the solids and slowly rises with time and position of the CPR.

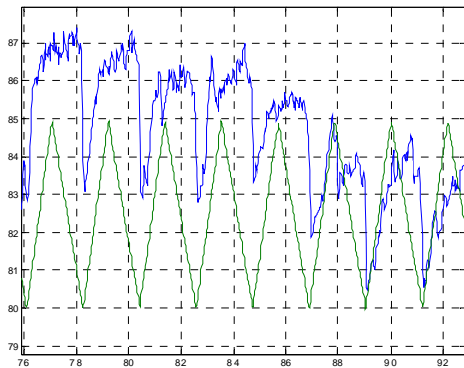


Figure 15. Swings in mud solids content with position of CPR.

In addition, results show that there is a slow drift of the solids content over time after each precoat drop. The entire sheet (precoat) is dropped once in a while to clean the wire mesh surface of the mud filter to ensure good vacuum and high solids content of the mud. This is observed in Figure 16 where the spike in the solids content is due to when the sheet is dropped and the mud solids analyser sees only belt. After each precoat is dropped, a higher solids content is observed, which is intuitive as the mesh surfaced has just been cleaned for higher vacuum efficiency. Overtime, as the solids drop, the coat is again dropped and renewed. This data shows that the mud solids analyser can be integrated for when indication as to when the precoat needs renewal.

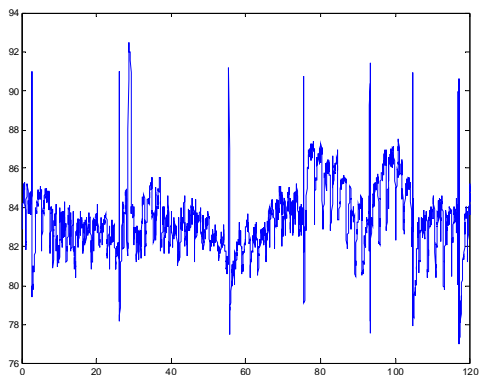


Figure 16. Vis-NIRS mud solids analyser trend showing slow drift in solids content over time.

Other operational parameters affecting the efficiencies of mud washing and filtering include vat slurry density, drum vacuum, shower water temperature, and rotational speed of the drum. Process bumtests and other studies are currently being conducted to develop simulation models relating various parameters to variations in mud solids content. The goal is to develop an automated control strategy of the mud filter operation utilizing mud solids content from the Vis-NIRS as feedback parameter. In addition, the mud solids content could also be incorporated into kiln control strategy to manipulate kiln firing and temperature according to the solids content.

Conclusions

The use of reflectance spectrometry, particularly Vis-NIRS, has many applications in forestry and forest products processing and manufacturing. Successful applications have been widespread, from in-tree wood properties measurements to logs to lumber and to pulp and paper processing. In this paper, we have demonstrated the two specific applications associated with the use of Vis-NIRS; at-line measurements of kraft pulp kappa number and brightness and online lime mud solids content.

Pulp kappa number at-line measurements incorporated a specially designed enclosure for the spectrometer and an automated sample preparation device to minimize measurement errors. Kappa and brightness measurements can be carried out in less than 3min. with no chemical requirements. Multiple commercial mill installations show that

the SEPs are within 1.4 kappa units and 0.6 ISO units for brightness.

Online lime mud solids showed that the calibration transfer from one spectrometer, with calibration model performed in the technical lab, to that of online process analyser can be done with good accuracy. Validations show that the SEP for mud solids is within 0.8% of oven-drying. The development of an automated internal referencing as well as the collimated beam has provided a real means of fully applying NIR reflectance spectroscopy as real-time process analysers for process control.

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