Recent advances in the commercialization of NIR (near-infrared) based liquor analyzers in the pulping and recovery area

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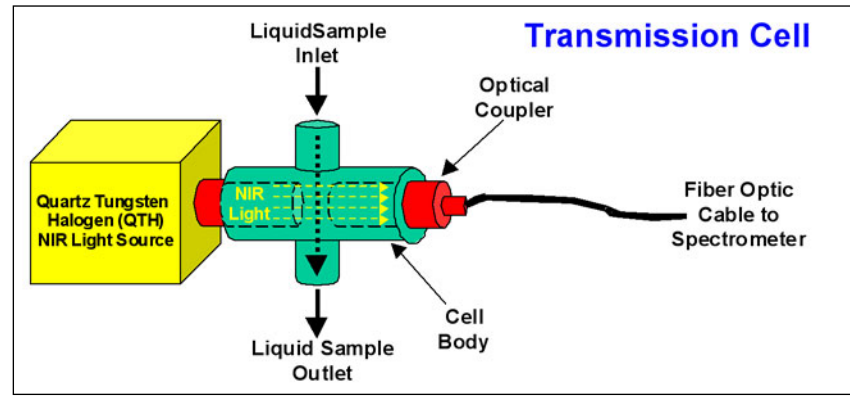
ABSTRACT: Near-infrared (NIR) spectroscopy techniques have held the promise of a new generation of online and lab based process analyzers for the pulping and recovery areas for some years now. NIR technology has been shown to be a quite effective method for the analysis of white, green and black liquors by multiple investigators. Recently, commercial analyzers have been developed for green liquor analysis on the dissolving tank, white and green liquor analysis in the recaustcizing area and black liquor analysis on batch and continuous digesters. These analyzers are currently running at different mill sites and are proving to be superior to current technology choices for these applications. Results will be presented on the performance of these analyzers relating to accuracy, maintenance and total installed cost.

Application: If a mill is looking for a turnkey liquor analysis solution in one of the key areas of the pulping and recovery processes then the NIR solution is now a viable candidate.

The kraft process is the most widely used pulping technique in the world accounting for over 80% of all chemical pulps. Advanced control of the pulping and recovery processes requires precise monitoring with online instruments. However, low maintenance cost effective instrument technology to continuously monitor liquor composition at the key locations in the kraft process has not been readily available. Accordingly, online liquor measurements have been a long-standing need for improved quality control in the kraft pulping and recovery processes. Near infrared (NIR) spectroscopy has shown much promise for providing reliable, cost effective solutions for all of the online and lab based liquor analysis requirements in the kraft process.

Several industrial installations by multiple companies have demonstrated the viability of NIR spectroscopy as a process sensor for liquor analysis applications. However, no commercially viable analyzer has resulted from these efforts for a variety of reasons. Some of the main issues that have hindered the commercialization of NIR based liquor analyzers have included sampling related problems, spectrometer robustness issues and the lack of a standardized design. In light of these issues a comprehensive effort was initiated to develop a common technology platform, with NIR spectroscopy as the sensor technology, to address all of the liquor analysis requirements in the kraft pulping and recovery process. The results of this effort have yielded a set of commercially available analyzers for white, green and black liquor analysis at key measurement locations in the kraft process.

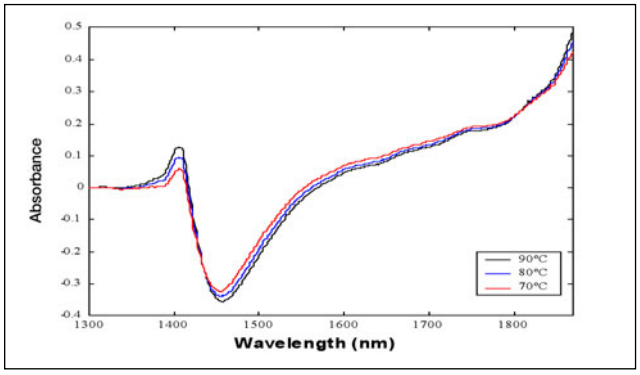


1. Typical transmission mode arrangement for liquid sample analysis.

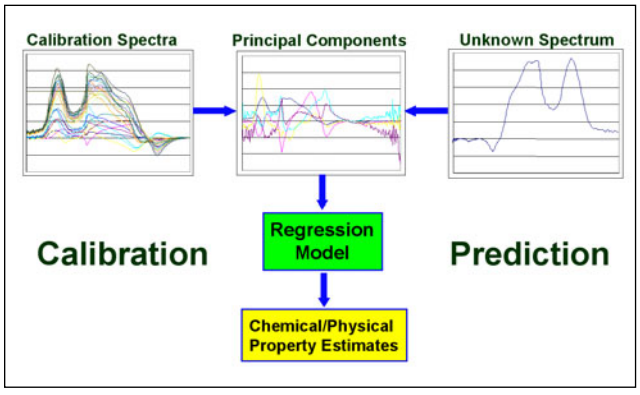
DISCUSSION

NIR spectroscopy

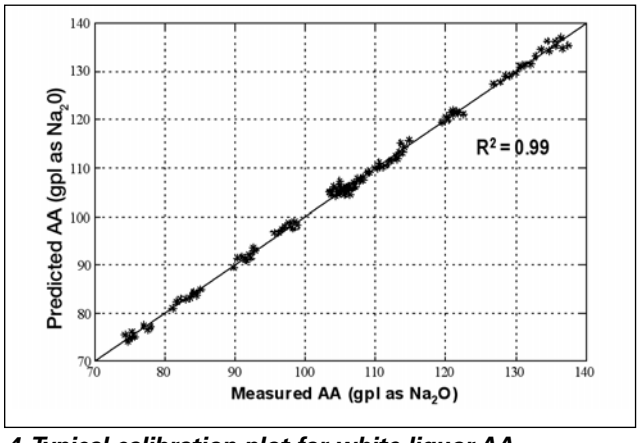
The near-infrared (NIR) spectral region is loosely defined as the band of wavelengths from 0.7 to 2.5 μm. Many molecular vibrations have overtone frequencies that give rise to absorption bands in this spectral region. The wavelengths at which these vibrations occur for a particular chemical are a function of its structure and composition. NIR spectra can thus be used to identify molecular species and evaluate their concentrations or mole fractions in com-



2. Absorbance spectra of white liquor at three different temperatures.



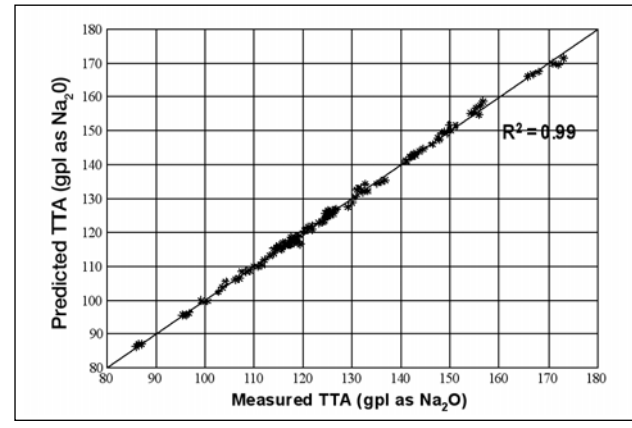
3. Partial least squares regression (PLSR) in a nut shell.



4. Typical calibration plot for white liquor AA.

plex chemical mixtures.

There are two practical advantages of NIR over conventional infrared spectroscopy. First, direct observation of solution samples is difficult because infrared wavelengths are strongly attenuated by water, solvents and the analytes themselves. Second, remote monitoring is difficult because these wavelengths cannot be efficiently transmitted through optical fibers. For these reasons, it is difficult to use IR spectroscopy for real-time monitoring of chemical and biochemical mixtures. NIR spectroscopy has no such limitations; it can be used directly with aqueous and organic so-



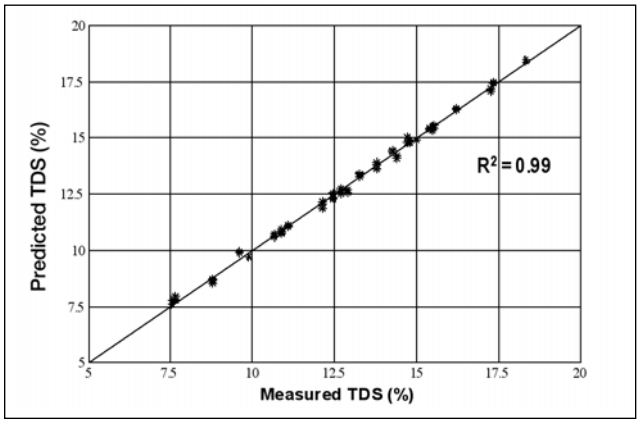
5. Typical calibration plot for green liquor TTA.

lutions, in both transmission and reflection modes. Also, it is well suited to fiber-based remote sensing using conventional, low-cost fiber optics. Figure 1 shows a typical transmission mode arrangement for analyzing liquid samples such as white, green, and black liquors.

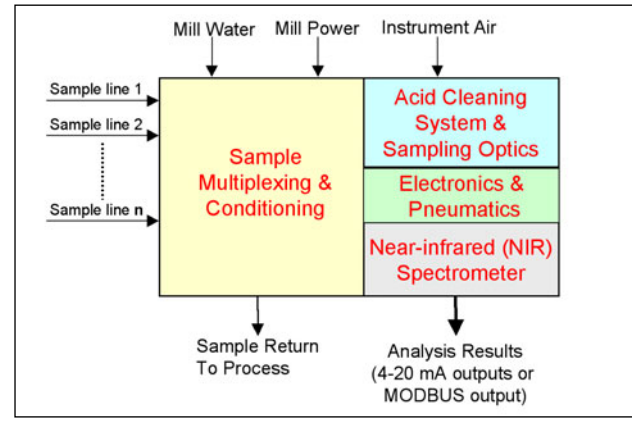
Quantitative analysis of materials using spectroscopic methods begins with acquiring a spectrum of the sample over the wavelength range of interest. For NIR analysis this wavelength range is between 750 and 2500 nm as previously stated. For liquid analysis, light absorption or transmission is measured at a number of wavelengths to generate a spectrum using a transmission cell arrangement as shown in Fig. 1. The spectrum is then linearized using the Beer-Lambert relation. Figure 2 shows linearized absorption spectra for a white liquor sample at three different temperatures. Physical and chemical information is contained in the spectral signatures of the samples and can be extracted using various regression techniques.

Spectral and chemical information

Extraction of physical and chemical information from spectral data can be achieved through a number of regression techniques. The field of chemometrics has provided a number of mathematical techniques to deal with this issue. One of the most popular techniques in use for relating spectral data to physical and chemical data is partial least squares regression (PLSR). This technique takes a set of spectral data derived from samples with known properties (e.g., effective alkali [EA], active alkali [AA], total titratable alkali [TTA], etc.) and builds a regression model. This regression model can then be used with spectra from unknown samples to predict the chemical/physical properties of the unknown sample. Figure 3 shows a graphic summarizing the calibration and prediction operations. In general a spectrum will have many more independent variables than available known samples which precludes the use of a standard regression approach. PLSR reduces the size of the calibration spectral data set by computing a set of principal components. Any spectrum in the original data set can then be reproduced as some linear



6. Typical calibration plot for black liquor TDS.



7. Block diagram of NIR based online liquor analyzer.

combination of these principal components. The principal components (PCs) are computed in such a way that a small number of PCs can be used to reproduce any spectra to any degree of precision desired. The original spectral data set is thus reduced to a small number of PCs and the corresponding coefficients required to reproduce each spectrum. Since the PCs are fixed for a given calibration set the coefficients now become the independent variables and represent the information content of the corresponding spectrum. At this point a standard regression model can be computed relating the spectral data to the properties of interest for the sample and the calibration is done. To predict an unknown sample a new spectrum is decomposed using the PCs from the calibration set. The information content of the new spectrum is reduced to a small number of coefficients based on the PCs. These coefficients are passed through the regression model to generate estimates of the desired physical/chemical properties of the sample.

NIR spectroscopy and liquor analysis

Previous and current work has shown that NIR spectroscopy can be used to measure the following chemical properties in white, green and black liquors:

- White Liquor – (EA, AA, TTA, TDS, TDD)
- Green Liquor – (EA, AA, TTA, TDS, TDD, RE)
- Black Liquor – (REA, RAA, Lignin, TDS)

Where these properties are defined as follows:

EA – Effective alkali (NaOH + ½Na_2S)

AA – Active alkali (NaOH + Na_2S)

TTA – Total titrateable alkali (NaOH + Na_2S + $Na2CO_3$)

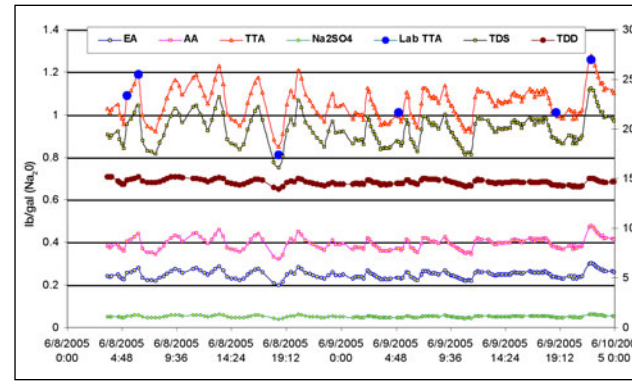
TDS – Total dissolved solids 100% * (Mass of dry solids/Mass of solution)

TDD – Total dissolved deadload 100% * (Mass of (Na_2SO_4 + Na_2SO_3 + $Na_2S_2O_3$ + Cl-)/TDS)

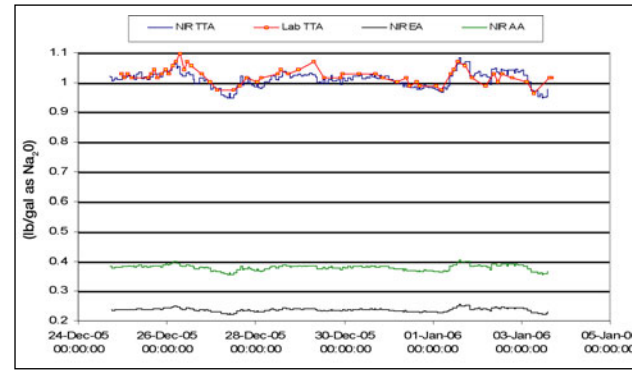
RE – Reduction efficiency 100%* (Na_2S/(Na_2S + Na_2SO_4))

REA – Residual effective alkali (residual NaOH + _ residual Na_2S)

RAA – Residual active alkali (residual NaOH + residual Na_2S)

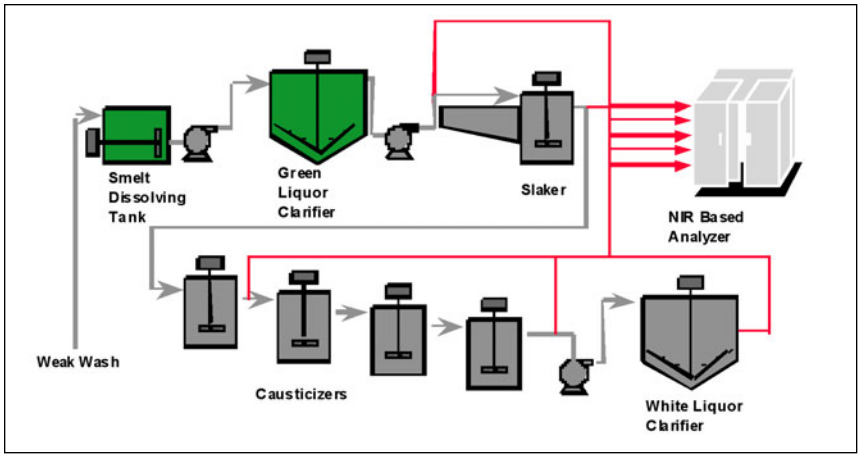


8. Trend data collected from a dissolving tank installation over a two day period using an online NIR liquor analyzer.



10. Trend data collected from a four sample line causticizing installation over a 10-day period using an online NIR liquor analyzer (green liquor to slaker).

Calibration of a NIR analyzer to accurately measure these properties involves collecting spectral data from a combination of synthetic and mill samples with known values and applying the PLSR method as previously described. Once the initial calibration has been performed, a single point calibration can then be carried out at the installation site based on lab testing at the site. The initial calibration is a one time operation that is carried out before the analyzer is installed. This calibra-



9. Diagram of typical causticizing application.

tion guarantees that the analyzer accurately measures changes in the chemical properties of interest. The single point calibration at the installation site sets the base line for the analyzer. Figures 4-6 show typical calibration curves for various components in the different liquor streams. The accuracy of NIR liquor analysis compared to conventional manual testing is summarized in Table I.

Lab-based NIR Liquor Analyzers

Some of the key advantages of this system include the items discussed below.

1. Elimination of operator bias due to sample volume variations. Repeatability of titration methods requires accurate volumes of liquor sample to be used and that each operator uses the same sample volume. An NIR analyzer does not need an accurate volume of sample for analysis, just a minimum volume.

2. Elimination of operator bias due to endpoint variations. If pH indicators are used to signal titration endpoints, repeatability can suffer if different operators interpret the endpoints differently or if an operator adds titration acid to rapidly when using pH probes. An NIR analyzer does not need pH indicators or pH probes or titration chemicals or any of the other peripheral support materials required for titration based testing. Therefore all bias and errors associated with titration methods can be eliminated with a spectroscopic based measurement.

3. Elimination of excess lab equipment at the testing station. Since properly calibrated NIR analyzers can measure other liquor properties such as total dissolved solids, a moisture balance can be eliminated from the testing station.

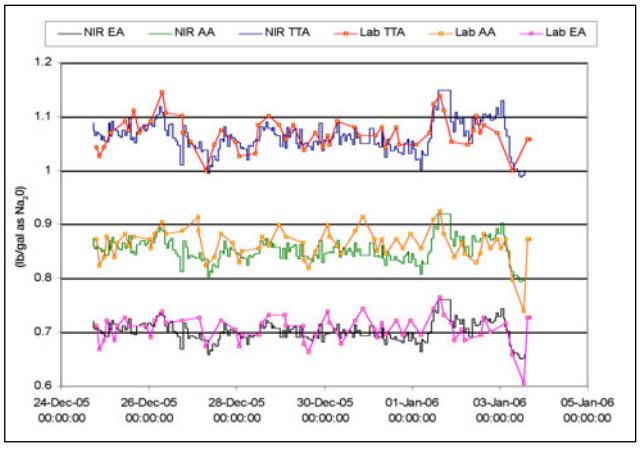
4. Rapid analysis leading to enhanced repeatability. Once the NIR analyzer is presented with a sample for analysis the results are available within seconds. This gives the opportunity to test the same sample several times to guarantee repeatability.

Online liquor analysis using NIR spectroscopy

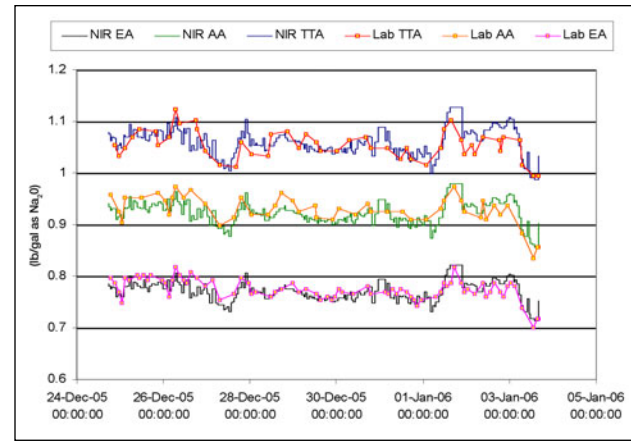
Online liquor analyzers, unlike their lab based counterparts, are significantly more involved. The problem is simple enough to define but quite difficult to solve. Automating the liquor analysis process simply comes down to reliably delivering a sample for the spectrometer to analyze. The implementation of this task is quite a challenge in practice. The use of NIR spectroscopy as the analyzer technology provides two approaches to solving this problem. One approach is to simply install transmission cells into any sample stream of interest. A sample cell, spectrometer pair can be installed at each analysis point to measure the desired liquor properties. This can become cost prohibitive if a number of sample points need to be analyzed. For many sample points an optical multiplexer can be used which takes the output from each transmission cell and sequentially passes the output to a single spectrometer. This approach seems simple enough to implement but ignores many issues required to create a truly reliable and stable analyzer. Some of the key issues include the buildup of scale on sampling optics, no convenient way to autoreference the spectrometer to guarantee long term stability and the need for multiple long runs of fiber optic cable to connect transmission cells to the spectrometer. A more reliable and cost effective approach is to multiplex the sample streams and not to multiplex the optical signals. This approach allows all of the aforementioned issues to be eliminated. The down side to this approach is the necessity to construct a sampling system that is comparable in reliability to the NIR spectrometer. Fortunately, the characteristics that make NIR spectroscopy attractive for liquor analysis applications also contribute to greatly simplifying the design of the supporting sampling system. The fact that NIR analysis requires little sample preparation allows for a significantly simpler sampling system design compared to online titration systems. Using a fiber optic based transmission cell completely isolates the spectrometer from the process samples, greatly improving reliability as compared to inline conductivity meters and refractometers. Finally, since NIR spectroscopy is not sensitive to trace components, standard mill water can be used as a reference material for autoreferencing the analyzer and thus guaranteeing long term stability.

Property	Agrees With Manual Testing Within
EA (gpl as Na_2O)	± 0.5 (gpl as Na_2O)
AA (gpl as Na_2O)	± 1.00 (gpl as Na_2O)
TTA (gpl as Na_2O)	± 1.00 (gpl as Na_2O)
TDS (%)	± 0.5 %
Repeatability	± 0.1 % of Full Scale

I. Summary of NIR measurement performance compared to manual testing.



11. Trend data collected from a four sample line causticizing installation over a 10-day period using an online NIR liquor analyzer (# 1 causticizer inlet).



12. Trend data collected from a four sample line causticizing installation over a 10-day period using an online NIR liquor analyzer (# 3 causticizer outlet).

Figure 7 shows a generic block diagram detailing the major components and connections for an NIR based liquor analyzer. This arrangement isolates all of the major components from each other to enhance system reliability. This arrangement yields a completely integrated system with a small installation footprint. All of the required hardware is packaged into a portable unit which significantly reduces system cost and installation cost. The main components of the NIR based analyzer are described below with their corresponding function.

- Sample multiplexing and conditioning: This portion of the analyzer is composed of valves and peripheral hardware required to reliably deliver process samples to the analyzer for analysis.
- Acid cleaning system and sampling optics: This portion of the analyzer is composed of the optical hardware required to interface the spectrometer to the sample under analysis. Additionally, an automated acid based cleaning system keeps the sampling optics free from scale buildup.
- Near-infrared (NIR) spectrometer: The NIR spectrometer is the brains of the system. This device controls all aspects of sample extraction and preparation as well as the acid cleaning system. The spectrometer also analyzes the sample for chemical composition and then makes the results readily available through industry standard IO.

NIR liquor analyzer applications

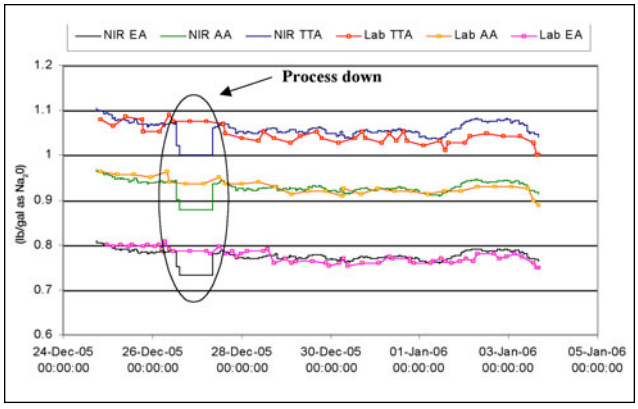
Initially, four liquor analysis applications were identified that have the greatest impact on process control in the pulping and recovery processes and potentially the highest payback. They include, green liquor analysis at the dissolving tank or stabilization tank, green and white liquor analysis in the recausticizing area, white liquor analysis on batch and continuous digesters, black liquor analysis on batch and continuous digesters.

Some of the current sensors and measurement techniques include hybrid systems composed of some arrangement of refractometer, density meter, conductivity meter and UV absorption meters. These sensors have calibration, scaling, base line drifts and maintenance issues. More over multi-component analysis is not viable with these sensors when used individually. On the other hand, on line titraters have the ability to measure multiple components in the liquor streams but suffer from high maintenance, cost and special housing requirements.

Reliable and accurate liquor analysis at the dissolving tank and stabilization tank locations is key to controlling the quality of the green liquor that is sent to the causticizing process. Stabilization of the green liquor at the dissolving tank is also important for controlling the settling characteristics of the liquor and minimizing scale buildup in piping and process equipment. In general the NIR analyzer is a two sample line system. One sample line monitors the exiting green liquor and the other sample line monitors the incoming weak wash, which provides the necessary measurements to implement a feed forward-feedback control arrangement. Reliable on-line liquor analysis at the dissolving tank is difficult in many cases due scale buildup on the sensor. To solve this problem, some sort of automated cleaning system has to be installed with the sensor. Green liquor concentrations can also change rapidly due to smelt rush conditions from the recovery boiler therefore rapid sample analysis is required to observe these changes.

The NIR analyzer approach, as implemented provides rapid multi-component analysis. The automated acid cleaning system guarantees that scale buildup on the optical components in contact with the green liquor is not an issue. Additionally, the NIR analyzer can measure components and properties that an automated titration approach cannot (e.g. Na_2SO_4, TDS, and TDD).

Figure 8 shows typical trend data from an NIR analyzer installed on a dis-



13. Trend data from a single sample line NIR white liquor analyzer collected over a 10-day period.

solving tank collected over a 2-day period. This dissolving tank is controlled based on a combination of bubble tube and density meter measurements. It can be seen that the density meter and bubble tube combination lack the precision to accurately track the green liquor concentration changes. Sample analysis was varied from 10 to 20 min intervals to see how rapidly the tank changed. Based on the trend data a sample update time of about 5 min is required to catch all of the process variation quickly enough to compensate. The NIR analyzer can sample and analyze the green liquor stream down to 3 min intervals.

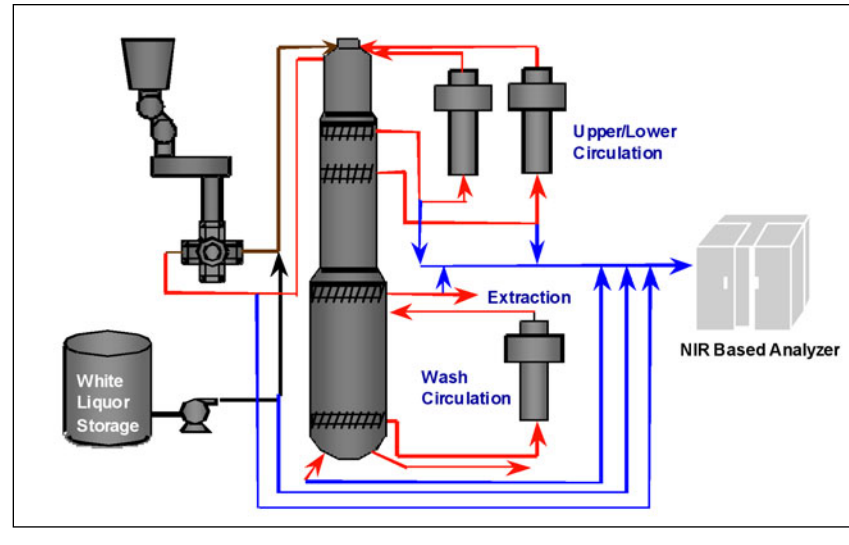
Maintenance requirements for the NIR analyzer are far less than any currently available green liquor sensor. The primary maintenance requirements for this system are light source replacement once per year and refreshing the optics cleaning acid every 3-4 months. Total installed cost for this system is comparable to that of a refractometer based solution.

Reliable and accurate white and green liquor analysis at key locations in the causticizing process is required to produce a stable high quality supply of cooking white liquor. Variations in green liquor composition and variations in lime quality require an online measurement solution to optimize control of the causticizing process. Operating the process at the highest causticizing efficiency possible without using too much lime is very difficult based on manual tests and delta-T control. Measurements for green liquor carbonate levels going into the slaker and white liquor composition downstream provide the necessary information to compensate for the majority of the process variations that will be experienced in practice. The NIR analyzer solution easily provides these measurements in a timely and reliable manner. Figure 9 shows a graphic of a typical causticizing process with the NIR analyzer as the monitoring sensor. In general the NIR analyzer is a four or five sample line system for this application. One sample line monitors the green liquor coming into the slaker, one line monitors the white liquor exiting the slaker, one line monitors the white liquor exiting the first causticizer one line monitors the white liquor exiting the last causticizer and optionally one line can monitor the clarified white liquor.

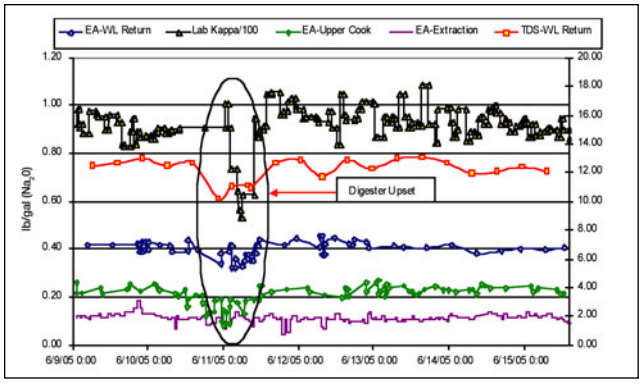
Again, the NIR analyzer approach, as implemented in Fig. 9 provides rapid multi-component analysis with near zero maintenance. The automated acid cleaning system guarantees that scale buildup on the optical components in contact with the process liquors is not an issue. The greatly simplified sampling system and small footprint of the NIR analyzer reduces system installation costs and maintenance costs to a fraction of those of a titration system. Again, the NIR analyzer can measure components and properties that an automated titration approach cannot (e.g. TDS and TDD).

Figures 10-12 show typical trend data from an NIR analyzer installed in a causticizing process collected over a 10-day period. Currently this process is controlled by a combination of delta-T and manual liquor tests. Variations in green liquor carbonate levels can easily be seen. Too high lime conditions and lime screw problems can also be observed from the trend data. The green liquor carbonate measurement is required to implement feed forward control of the lime screw for dosing the appropriate amount of lime to avoid an excessive lime condition. The effective alkali levels of the downstream white liquor can then be used to fine tune lime addition to compensate for variations in lime quality. Sulfide levels in the downstream white liquors are used to set the target causticizing efficiency. The combination of feed forward and feedback control based one these measurements will ensure that the causticizing process is run at the maximum causticizing efficiency without using too much lime.

Maintenance requirements for the NIR analyzer are far less than



14. Diagram of typical continuous digester application.



15. Trend data collected from a six sample line digester liquor analyzer over a 7-day period using an online NIR liquor analyzer.

any hybrid sensor package or titration based solution. The primary maintenance requirements for this system are light source replacement once per year and refreshing the optics cleaning acid every 3-4 months. Total installed cost for an NIR based analyzer is typically less than half that of a titration based system.

Reliable and accurate white liquor analysis for batch and continuous is important for minimizing pulp quality variations. For both continuous and batch digesters white liquor composition needs to be accurately known to ensure that the correct effective alkali is charged for the given chip mass entering the digester. The two sources of variation when charging a batch or continuous digester are chip moisture variations and white liquor variations. If white liquor composition is accurately known then one source of variation can be eliminated. With an online white liquor analyzer the white liquor can be trimmed in real time to meet target effective alkali and sulfidity levels allowing one to maintain a constant liquor to wood ratio. Alternatively, variations in white liquor composition can be compensated for by only adjusting the liquor to wood ratio. Either approach requires an accurate online analysis of the cooking white liquor. An NIR based analyzer provides the required effective alkali and sulfidity measurements in a timely, accurate and reliable manner. This application is typically a single sample line instrument monitoring the white liquor right before it is charged into the digester or monitoring the white liquor downstream of a trimming arrangement.

Figure 13 shows typical trend data from an NIR analyzer monitoring cooking white liquor collected over a 5-day period. Variations in effective alkali, sulfidity and carbonate levels can be seen on this graph. Variations in sulfide and carbonate levels are not extreme but they are enough to be noticed with a conductivity meter and thus introduce error in the effective alkali measurement obtained from such an instrument.

Maintenance requirements for the NIR white liquor analyzer are the same as the previous analyzers described. The primary maintenance requirements for this system are light source replacement once per year and refreshing the optics cleaning acid every 3-4 months. Total installed cost for this system is about twice that of a high quality conductivity probe.

Monitoring black liquor composition in a continuous digester provides a means for implementing advanced control schemes in the pulping process. By monitoring the residual effective alkali profile and stabilizing this profile in a continuous digester, pulp quality variations can be greatly reduced. Digesters running standard cooks can compensate for residual EA variations by adjusting the temperatures in the cooking zones. Variations in residual EA can also be compensated for by trimming the incoming white liquor or adjusting the liquor to wood ratio. Continuous digesters fitted with liquor feed points in the cooking zones have the added flexibility of accurately and quickly controlling the residual EA profile throughout the digester by directly injecting white liquor into these zones. Controlling the residual EA profile and cooking conditions in a continuous digester by either method requires a reliable and accurate measurement. NIR analysis is well suited for measuring residual EA in black liquor. Besides providing the residual EA measurement, residual AA, lignin and TDS measurements are also available from the same instrument. The residual EA profile is an indicator of exiting kappa number, the residual AA profile, lignin profile and TDS profile are indicators of exiting pulp yield. Black liquor composition contains a wealth of information relating to various pulp properties. The NIR analyzer captures this information in the spectral signature of the black liquor sample. So in addition to providing the previously mentioned measurements, a variety of pulp properties (e.g. kappa number and relative yield) can be directly correlated to the liquor spectral signature. This can be done for both batch and continuous digester. Figure 14 shows a graphic of a typical continuous digester installation. This implementation has six sample lines, monitoring the incoming white liquor, liquor return, upper and lower cooking zones, extraction zone and the incoming filtrate liquor. This arrangement gives a complete picture of the digestion process from the perspective of liquor variations.

The NIR analyzer solution provides the collection of measurements in one low maintenance instrument. Calibration for the NIR analyzer is reduced to a single point calibration that is performed once at the installation site.

Figures 15 shows typical trend data from an NIR analyzer installed on a continuous digester, running a conventional cook, collected over a one week period. Currently kappa control is implemented by using a combination of cooking zone heater adjustments and liquor to wood ratio adjustments. By monitoring the white liquor input and the white liquor return it is possible to compensate for variations in chip moisture early in the cook. Monitoring the cooking zones and extraction zone provide a further means of stabilizing the liquor profile throughout the digester, resulting in a final pulp with less variations in physical and chemical properties. Although the lab kappa measurements are noisy it is easily seen that there is a correlation between the liquor composition profile of the digester and exiting kappa number of the pulp.

Black liquor analysis on batch and continuous digesters is the most demanding of all of the liquor analysis applications. The high temperatures and pressures of the incoming samples place a

great strain on the sampling system. The NIR based analyzer allows for a very simple sampling system design, which results in a system with the required durability and reliability to handle this application. Maintenance requirements for the NIR digester analyzer are the same as the previous analyzers described. The primary maintenance requirements for this system are light source replacement once per year and refreshing the optics cleaning acid every 3-4 months.

CONCLUSIONS

Spectroscopic techniques hold the solution to most if not all of the difficult measurement applications in the kraft pulping and recovery process. In particular, NIR based process analyzers provide a complete liquor analysis solution for the pulping and recovery area based on a common analyzer technology. These systems have been designed to take every advantage offered by NIR spectroscopy. Combining the advantages of NIR analysis with the simplified sampling arrangement and an appropriate optics cleaning system produces an online analyzer with the following characteristics:

- Very low maintenance
- Multi-component analysis
- Simple architecture
- Integrated turnkey design
- Simple, low cost installation
- Minimal training of mill personnel
- Standardized technology across all process analyzers

NIR based process analyzers are showing much promise for other applications well beyond liquor analysis. **TJ**

Submitted: May 8, 2006
Accepted: August 19, 2006

ACKNOWLEDGEMENTS

The authors wish to thank the Alabama Center for Paper and Bio-resource Engineering for past, current and future contributions leading to the successful commercial development of this technology.

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14. TAPPI Test Methods T 236 cm-85 "Kappa number of pulp."

INSIGHTS FROM THE AUTHORS

This work was undertaken to develop viable commercial liquor analyzer systems based on NIR technology. Several prior mill trials had shown the potential of this technology to provide a comprehensive online analyzer solution to all of the key liquor measurement needs in the pulping and recovery areas. None of these prior systems evolved into a true commercial solution for a variety of reasons including sampling system reliability issues, spectrometer reliability issues and the lack of a standard design. To overcome these issues a standardized design was developed from which all of the analyzers are based. For each application only the sampling system varies, all other system components remain constant. A simple cost effective spectrometer was designed that would survive in the harsh mill environment and would also provide all of the functionality necessary to run the sampling system, analyze the samples and report the results. Mill trials for each application were initiated to test and refine sampling system designs as well as proving the overall design approach. The results of these trials have produced a set of near-infrared based liquor analyzers that are simple in design and installation, low in total installed cost and low in maintenance compared to currently available technology.


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