

# OPTIMIZING YOUR PROCESS THROUGH LIGNIN MANAGEMENT

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## ABSTRACT

Removing lignin is a very necessary but costly part of the pulping process. Therefore having state-of-the-art measurements and control capabilities are extremely important in maximizing business performance and final pulp quality. It has been a constant challenge to accurately and reliably measure lignin from the blow line through the bleach plant. The industry has relied on laboratory measurements, and/or slower multi-point kappa analyzers to measure fiber kappa. An oftentimes equally important but neglected component is the dissolved (or filtrate) lignin moving through the process, which also can widely vary and consume a significant portion of bleaching chemicals. Until recently there have not been direct measurements of dissolved lignin and total bleach load. Operators have relied on surrogate values such as conductivity or chemical residual to account for black lignin carryover. Inaccurate measurement of dissolved lignin can lead to overuse of bleaching chemicals and increased costs to the operation. With these critical measurements being problematic, new innovative technologies have been developed to enhance the ability to optimize the pulping process and deliver significant sustainable gains in business performance for the industry. This paper will discuss the recent improvements in measuring fiber and filtrate lignin on-line and its impact on fiber line and bleach plant controls.

## MEASURING FIBER KAPPA

Lignin is the “glue” that holds the wood chip together. Chemical pulping relies on efficiently separating wood fibers from lignin. The most common measurement of lignin is the Kappa Number, which we can measure in the laboratory.

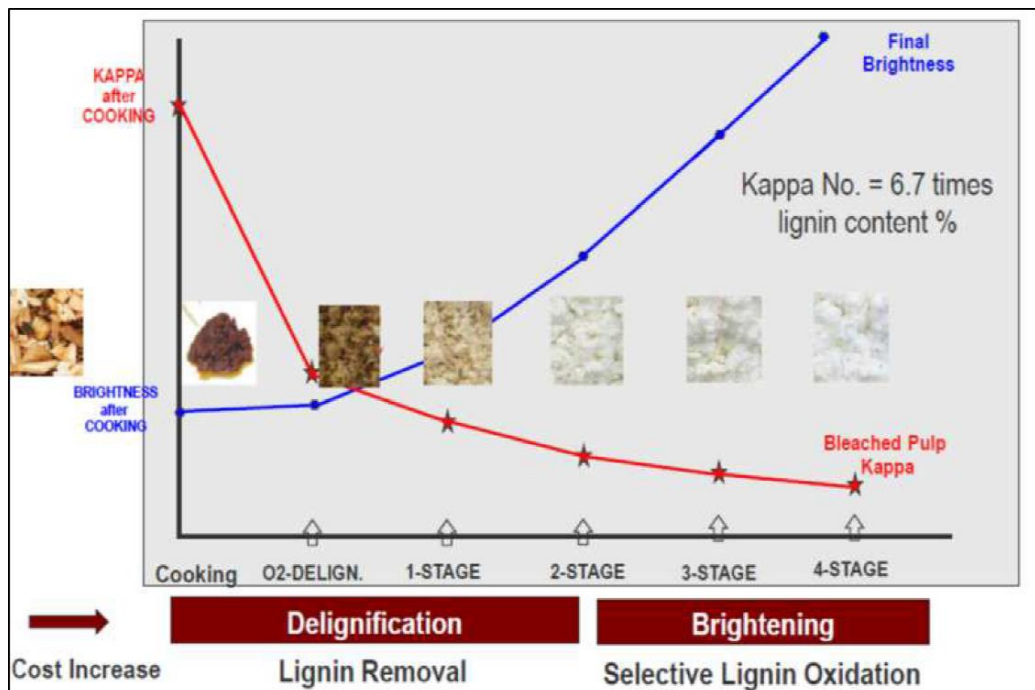


Figure 1: Measuring lignin in the Fiber Line

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Figure 2: Example of MultiPoint Kappa Analyzer

we focus on kappa measurement. Before online Kappa measurements were available, mills relied on a tester to run a test every 2 hours.

However, there can be numerous problems associated with the manual kappa test due to sample preparation, chemical strength, and method of titration. Testing and sampling error is a part of the Kappa variability from the digester, as shown in Figure 3. The Kappa result is used to make changes in digester cooking, control the Oxygen Delignification stage, and to cut or

When Kappa analyzers became commercially available in the early 1990's, Kappa Number (Kappa) has been measured on-line. Kappa is defined as the milliliters of 01N potassium permanganate solution consumed by 1 g of dry pulp using a standard laboratory procedure. For example, consider the amount of lignin in a 30 kappa softwood pulp. After the digester, the pulp has been delignified by 90 percent. The remaining 10 percent of lignin in the pulp is the focus of measurement and control after the blow line. Figure 1 is a conceptual diagram of lignin and brightness from the blow line through the bleach plant. The amount of lignin quickly reduces as we move through the bleach plant, while the brightness increases. The addition of extended cooking and oxygen delignification ahead of the bleach plant lowers the Kappa entering the bleach plant. Thus, Brightness may be a better measurement at the post extraction stage due to the very low Kappa numbers. Prior to the extraction stage,

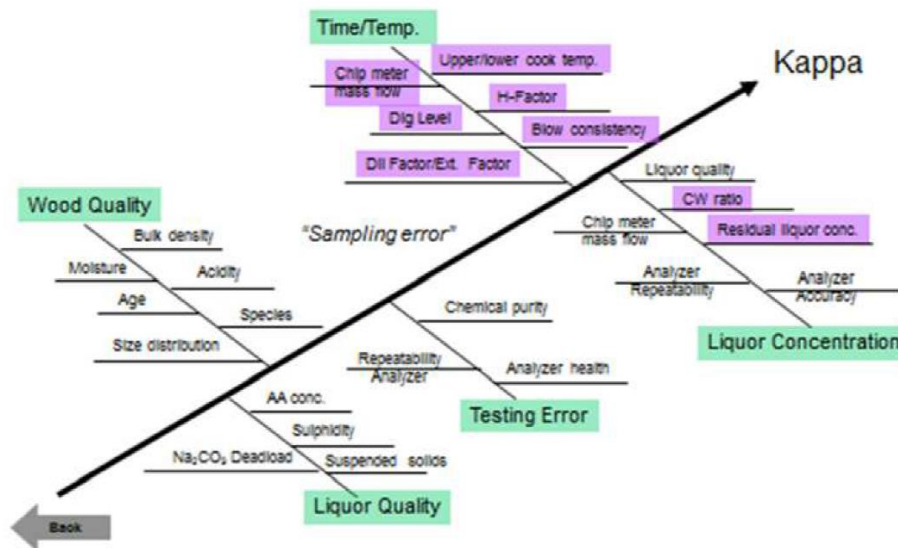


Figure 3: Variability Associated with the Kappa Number (Honeywell)

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Figure 4: Single Point Kappa Analyzer

increase chemical to the bleach plant.

Existing on-line technology has focused on the multi-point analyzer since the early 1990's. The device automatically samples and transports the pulp to a central analyzer. The central analyzer screens, washes, and measures Kappa. The measurement is based on the use of UV light passed through a diluted pulp sample of well washed pulp<sup>1</sup>. The analyzer uses an optical measuring method to determine the amount of lignin in the pulp. This method has been shown to correlate well with the results of laboratory chemical Kappa tests over a wide Kappa Number range, for a range of different wood species. The

analyzer consistently and repeatedly takes the measurement, eliminating differences between testers. Negatives associated with the analyzer are the high cost of installation, which can be up to \$250,000. Under optimal conditions, the traditional Kappa analyzer can process 4 to 6 samples per hour across up to 8 sample points, such as the digester blow line, pre and post oxygen delignification, feed to the bleach plant, and post extraction stage in the bleach plant. However, large gaps in Kappa updating to the DCS may occur because the analyzer must divide its processing time between samples. Mill control strategies may be affected by sporadic updating, and process changes may be missed due to long intervals between samples. Utilizing an eight point Kappa analyzer in unbleached Kraft applications requiring measurement of only one or two sample points is cost prohibitive. As such, some mills elect to measure Kappa manually, resulting in variable results and erratic update times. Operators are often reluctant to make digester changes based on infrequent samples. Automated control is not possible with the infrequent laboratory Kappa updates. Typically, operators will cook to a lower kappa to avoid discharging uncooked pulp. Overcooking leads to yield losses from the digester and higher production costs. More frequent kappa updates to the cooking control can lead to reduced variability and allow the mill to confidently raise their Kappa targets.

A new single point analyzer is now available that is mounted directly to the pulp processing pipeline. This analyzer contains all unit operations of the traditional multi-point Kappa analyzer, such as pulp sampling, screening, washing, and optical measurement of Kappa. Because of the single sample point,

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Kappa measurements for all fiber line applications are updated more promptly. The single point concept allows for incremental investment in key positions for mills with limited capital. It is now possible to update 12 samples per hour for a continuous digester, and 2 to 3 samples per blow for batch digesters. Yield increases of 0.5% to 2.0% are possible when the digester Kappa setpoint increases. The single point Kappa analyzer can also be placed pre- and post- Oxygen Delignification or in the bleach plant to control the dosage of chlorine dioxide.

An example from a southern unbleached Kraft mill running approximately 800 tons per day demonstrates how an affordable single point solution reduced Kappa variability and raised operator confidence in Kappa measurement. Green Bay Packaging in Morrilton, Arkansas has relied on laboratory kappa samples taken every 2 hours. Variability in tester methods has always been high, and operators were reluctant to raise the Kappa number due to sporadic feedback. A single point automated Kappa analyzer was installed in March 2014 after the hot stock refiners (Figure 4). Average Kappa number ranged from 91 to 101.

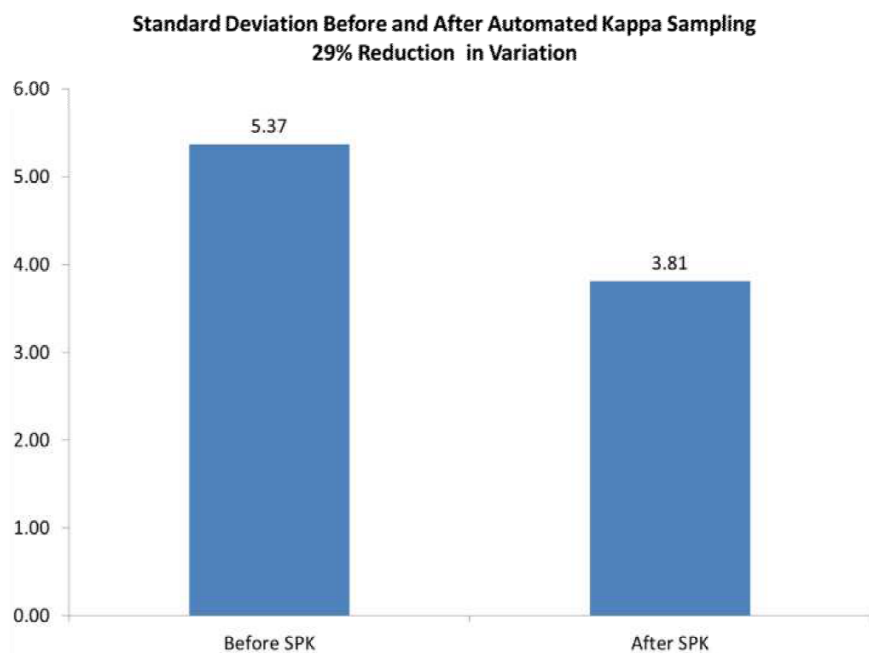


Figure 5: Reduction in Variability due to automated Kappa testing



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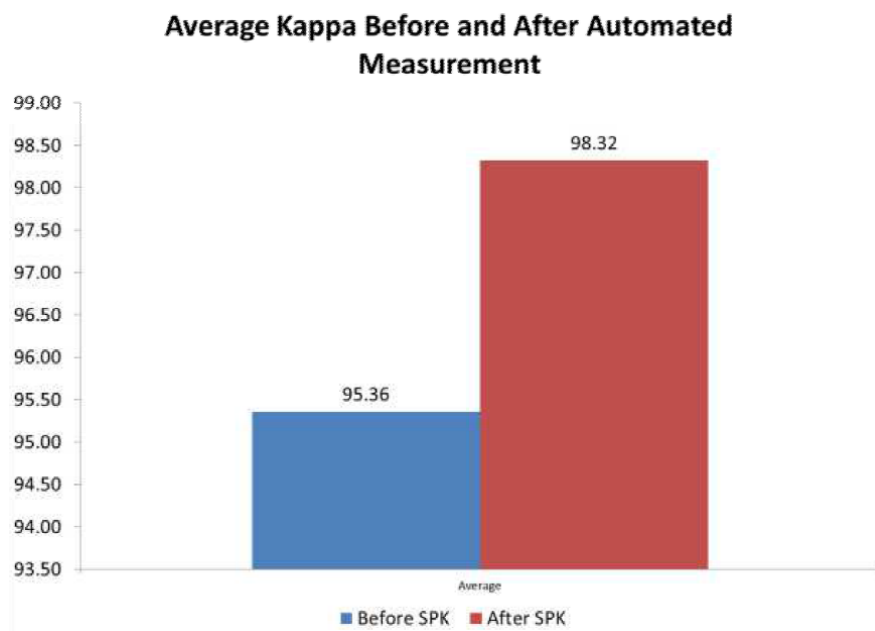


Figure 6: Increase in Blow Line Kappa After Installation of automated kappa

Figure 6 shows the rise in Kappa values after automating sampling and testing was installed. The 2.96 increase in Kappa roughly translates to a potential increase in yield that is now being verified by the mill. Green Bay Packaging has also seen a 29% reduction in Kappa standard deviation as shown in Figure 5. Operators are also making fewer changes to the alkali-to-wood ratio due to more frequent Kappa updates. Figure 7 shows that operators made 47% fewer corrections which in turn have resulted in lower Kappa variation out of the digester.

The Pulp Mill and Paper Mill Operations at Morrilton have accepted the new automated Kappa measurement and have cited many benefits of a single point automated kappa analyzer:

1. The sample point is now closer to the digesters so they can better adjust their cooks.
2. The control chart used to make changes in Alkali to Wood Ratio was only updated every 2 hours and is now updated every 30 minutes.
3. There are now less alkali-to-wood ratio changes. Previously, some changes were unnecessary and led to harmful kappa variability.
4. The operators are seeing a decrease in the daily standard deviation.

Alkali/Wood Ratio	Before SPK	After SPK
Ratio Changes	253	133
Average ratio	13.60	13.16
Standard deviation	0.59	0.31

Figure 7: Fewer changes to liquor charge with automated testing

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5. The operators raised the Kappa set point because they are more confident in the pulp mill's ability to control Kappa
6. Paper makers observe that they receive better and more timely data on the pulp that is coming to their machines. Manual Kappa samples were taken after the brown stock washers, which took 40 minutes per test. During this lengthy testing interval, the stock may have already reached the wet end of the paper machines. Usually, there was a one to two hour lag time in receiving test information
7. Paper makers can make preemptive moves to eliminate breaks from stock changes. One break causes 36 minutes of downtime. Previously, changes were made for anticipated off quality conditions that never materialized and the sheet broke due to the unnecessary process shift. This is currently being quantified.



Figure 8: Compounds in Oxygen Delignified Pulp

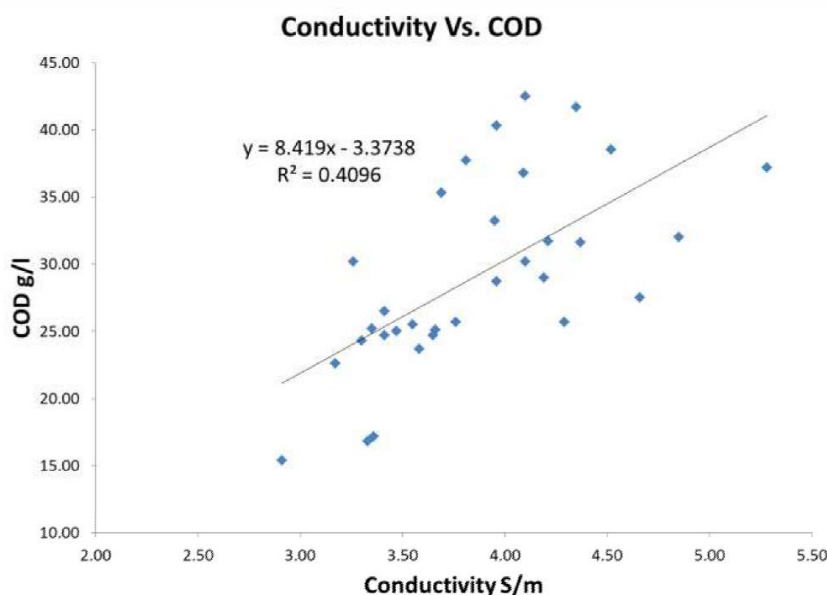


Figure 9: Relationship between COD and Conductivity

## MEASURING LIGNIN IN THE FILTRATE

Fiber lignin is not the only lignin in the process pipeline. Oxygen delignified pulp is comprised of fiber bound lignin and inorganic compounds and organic compounds, commonly referred to as black liquor or dissolved lignin.

The Kappa analyzer washes away the black liquor in its processing of the online sample, leaving the bleach plant operator to estimate what other compounds are entering the plant.

Mills struggle with proper washing in the brown stock area; they must balance the cleanliness of the pulp they are sending to the bleach plant along with the amount of solids they are sending to the evaporators.

When the washing is poor, the challenge in the bleach plant operation is to accurately

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quantify the amount of black liquor carryover, because it affects the first stage of processing.

At present, most mills rely on conductivity or chemical residual after chlorine dioxide addition to indicate carryover. These are at best surrogate measurements in place of quantifying dissolved lignin.

In the brown stock area, an accurate chemical oxygen demand test (COD) may be used to evaluate the washing performance, but the test requires more than 2 hours to process results. Thus, the operator receives the information long after the pulp has moved to the next processing stage in the plant. A real time indication of pulp cleanliness is needed to add to a dilution factor control strategy.

Dirty pulp entering the bleach plant with an excess of dissolved lignin will consume chlorine dioxide that is used to remove the lignin in the fiber in the first stage. Andersson et al<sup>2</sup> have shown through mill testing that the correlation of COD and conductivity is poor, as shown in the Figure 9 trend. This may lead to incorrect decisions in raising or lowering shower flows in the brown stock washing area, dosing chemical in the bleach plant, or both.



Figure 10: In-Line Dissolved Lignin Transmitter

Conductivity is based on the measurement of the ionic sodium species in the liquor, inorganic phase. Conductivity does not directly measure the organic phase, the dissolved lignin. We assume that the inorganic/organic phase remains constant, so a relationship can be built between conductivity and dissolved lignin. This ratio assumption holds for short time frames, but is inappropriate over longer time periods. This is due to changing white liquor concentration (TTA/EA), liquor to wood ratios in digesters, the extent of delignification in the digester, wood species changes, and other seasonal

effects.<sup>3</sup> The relationship between filtrate kappa and conductivity is also poor, which may lead to

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overdosing or under dosing chlorine dioxide in the bleach plant. The final result is missing the extraction Kappa and brightness target.

Andersson et al<sup>4</sup> introduced an in-line sensor in 2012 that can accurately quantify dissolved lignin. The Dissolved Lignin Transmitter, shown in Figure 10, is an in-line probe with UV and IR LED's. This sensor is a probe orientated in the pulp stream so only the filtrate portion is measured. The sensor exploits the relationship between lignin and UV absorption. We typically measure lignin in the Kappa analyzer at 280 nm. Figure 11 shows how UV absorbance of lignin is possible at a wider range of wavelengths with excellent correlations. The NIR signals compensates for any scattering effect caused by fines in the filtrate. This sensor ignores the inorganic components and focuses on the dissolved lignin only. The sensor is capable of adjusting for high and low dissolved lignin concentrations by adjusting the LED set point of the UV.

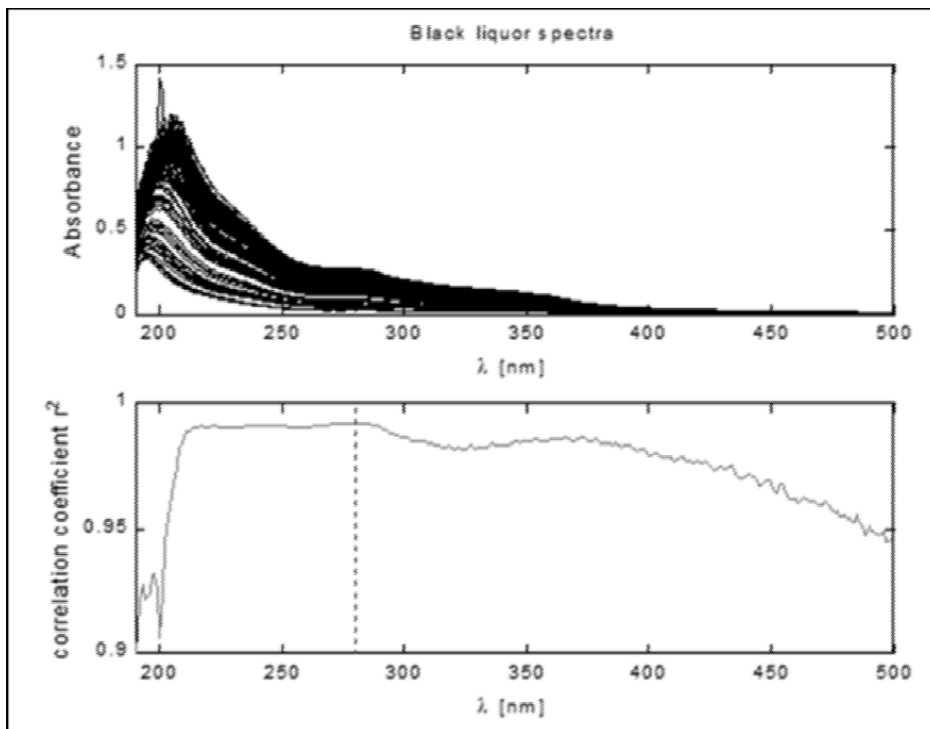


Figure 11: UV Absorbance is measured at a wide range of wavelengths

This transmitter may be installed in several locations of the fiber line to enhance process control:

- In brown stock washing prior to the bleach plant. Including an on-line dissolved lignin measurement in washer control will insure the cleanest pulp will be sent to the bleach plant.

- Prior to Oxygen Delignification stage: Incoming dissolved lignin can now be measured to dose the proper amount of chemicals to reach a percent delignification target.

- Bleach Plant to

measure carryover: This measurement may be added to Kappa factor control at the first stage of the plant and after the post extraction washer to measure the amount of extraction filtrate entering the D1 stage.

- Waste treatment: To measure sewer COD and not exceed environmental permit levels.
- Stock approach area to container board machines: Excessive lignin at the wet end creates more charge, pH, and foam. Measurement will help control the addition of additives, help reduce scale formation due to pH upsets, and reduce the need for defoamer.



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## MEASURING TOTAL LIGNIN



Figure 12: Bleach Load Transmitter In-Line

bleach load includes the carryover entering the bleach plant so the chlorine dioxide dosage is adjusted before the chemical is added. This removes the guesswork where the operator would adjust a "bias" or add more chlorine dioxide to account for the amount of estimated carryover coming into the bleach plant. In a North American bleach plant, the bleach load measurement was used to help control the first stage of the bleaching operation.

	Tons Per Day	Bleach Load	Kappa Factor	% ClO <sub>2</sub> Applied	Operator Bias
Before	522	18.52	0.232	1.64	0.651
After	461	18.27	0.231	1.60	0.000

Figure 13: Operator Bias Eliminated Using Bleach Plant

There are times when it is not necessary to separate the fiber and filtrate lignin and only report a total sum of the two components. VanFleet<sup>5</sup> has developed and defined the measurement of "Bleach Load" where the fiber and filtrate kappa is measured in an on-line sensor and used in Kappa factor control. The laboratory procedure to measure bleach load involves running a fiber Kappa test, consistency, and filtrate Kappa from the pulp squeezings of the sample. The filtrate Kappa is converted to a gram equivalent of the filtrate in the pulp suspension. The fiber and filtrate Kappa tests reported in grams are added together. Total

The chart in Figure 13 shows the overall lowering of chlorine dioxide usage. This is due to the elimination of operator bias from the existing compensated brightness control and resulted in an annual saving of \$60K US. The operator was able to achieve his target because the dissolved lignin was measured along with the fiber Kappa before chlorine dioxide was dosed.

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The Bleach Load Transmitter operates with five LEDs; blue, red, green, NIR, and UV, and works under the principle of optical reflection. Laboratory samples are collected along with raw LED values. The raw signals are used in combination to predict Bleach load. The number of signals used in the calibration will vary depending on the process and location of the bleach load sensor.

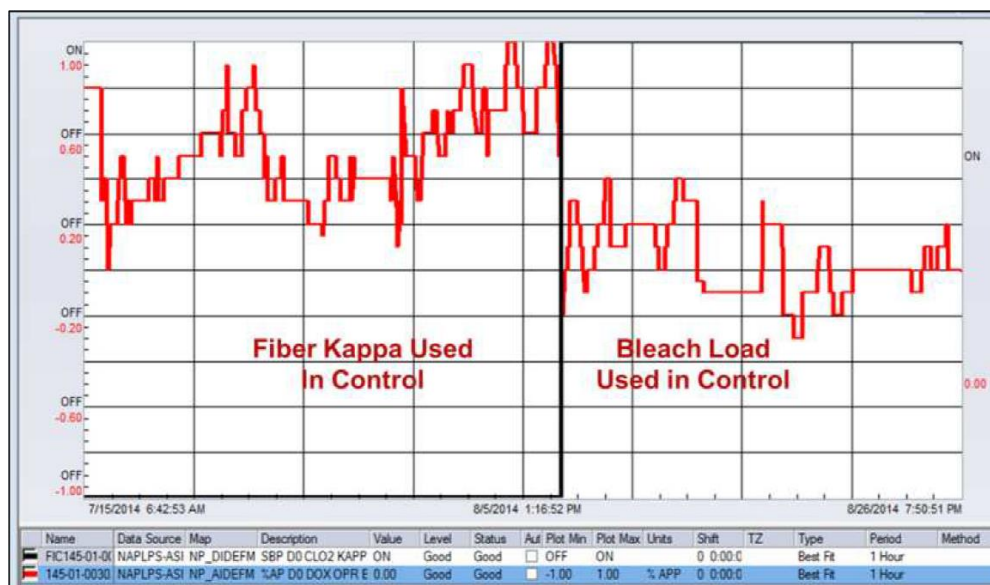


Figure 14: Fiber Kappa vs. Bleach Load used in Kappa Factor Control

Figure 15 is an example of a calibration and is using the red and UV LED in the model.

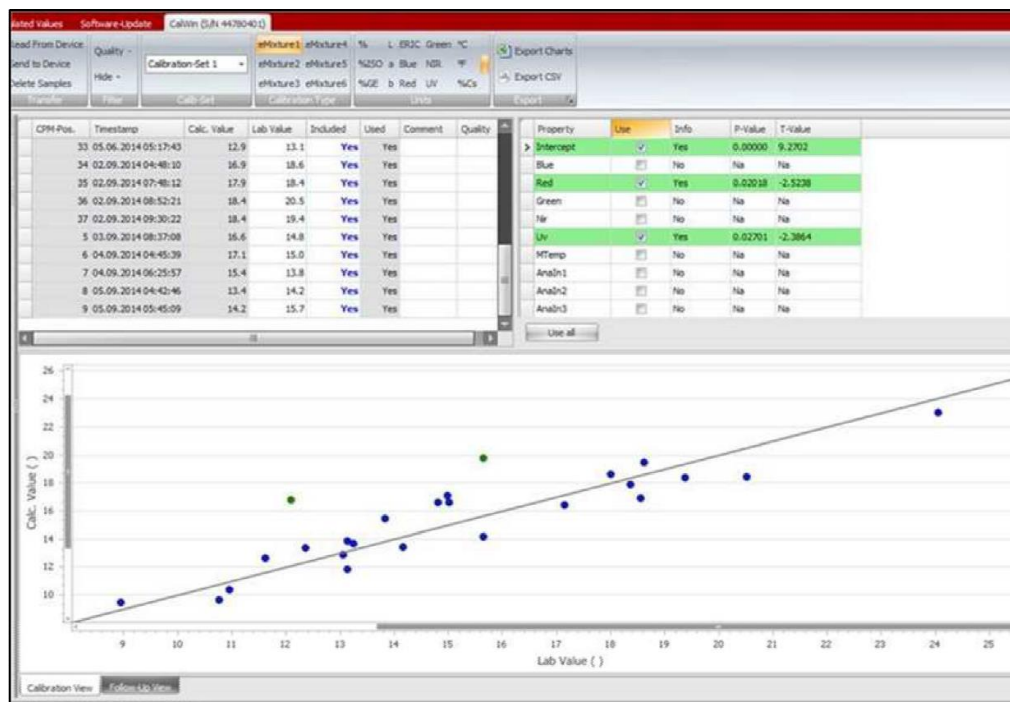


Figure 15: Example of Bleach Load Calibration

The Bleach Load Transmitter is usually installed in the first stage of the bleach plant and post extraction. Because the transmitter is measuring both fiber and filtrate, it can uncover problems in bleach plant washing. Poor washing has been shown to lead to higher percent of filtrate Kappa due to carryover of filtrate from the washer. In the example shown in

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Figure 15 and Figure 16, the mill acknowledged they were having problems washing the extraction filtrate from the pulp in a three stage bleach plant. This caused excessive use of chlorine dioxide in the

brightening stage. Laboratory testing revealed that the filtrate kappa was over 35% of the total bleach load. Adjustments were made to the washer showers, resulting in an almost 15 percent reduction in filtrate Kappa. A traditional multi-point analyzer would have missed this and extended troubleshooting time to determine why more chlorine dioxide was needed for the first

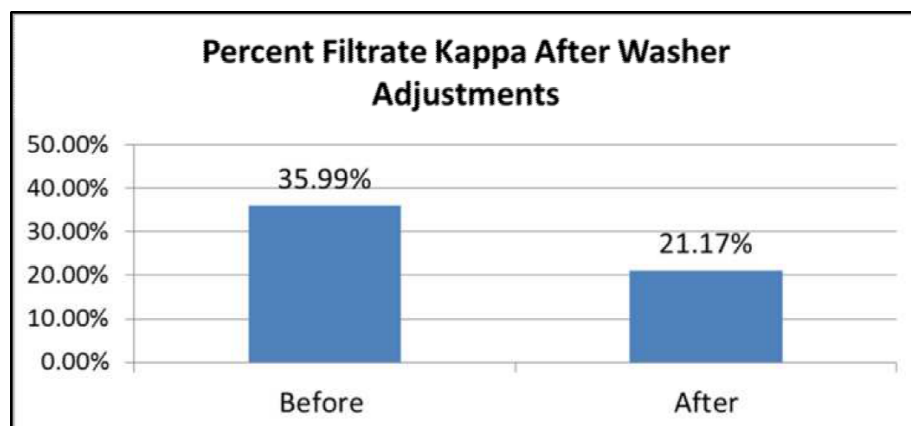


Figure 16: Impact of Washer Adjustments on Bleach Load

brightening stage.

## CONCLUSIONS:

There is available technology to measure both fiber and filtrate lignin using single point analyzers and in-line sensors. This is a significant change from the multi-point analyzers that can be maintenance intensive, require high installation costs, and have inadequate uptime times for sound process control. The dissolved lignin and total bleach load sensors are in-line probes that can be retracted while the process is running, providing continuous updates. Dissolved lignin in the filtrate can now be measured using a UV light source, which is the same measuring source as in the traditional fiber kappa analyzer. The third sensor discussed, the Bleach Load Transmitter, measured fiber and filtrate in a combined signal using optical reflectance and LED technology to determine total Bleach Load. Bleach Load represents the total chlorine dioxide demand to the bleach plant and allows the elimination of operator bias which often results in bleaching chemical overuse.

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