

# Kinetic Model-Based State Estimation and Inferential Control of the Causticizing Process

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## ABSTRACT

In this paper we present a new causticizing control system that combines liquor composition measurements and model-based inferencing of lime quality to achieve lime dosage control. Lime addition to the slaker is determined by a stoichiometric calculation where the clarified green liquor flow and chemical composition, the inferred lime quality and the white liquor causticity target value are inputs. A lime quality estimator, based on a high fidelity kinetic-dynamic model of the causticizing process, is used to predict the downstream liquor temperature and composition measurements from the process inputs and the current state estimates, and then the difference between the model predictions and the actual measurements is used to correct the states. The system is extremely flexible as it is able to integrate conventional measurements such as temperature and conductivity, as well as liquor composition measurements from an online analyzer, manual ABC titrations, or a combination of these. Two mill case studies, one involving conventional measurements and one based on an online analyzer, are presented and discussed.

## INTRODUCTION

The main purpose of the causticizing process is to maximize the conversion of sodium carbonate to sodium hydroxide while simultaneously producing a strong, uniform white liquor and lime mud particles that are easily separated. Unconverted sodium carbonate in white liquor is an undesirable deadload that increases energy consumption and chemical losses, reduces equipment capacity, and creates operating problems [1,2]. Causticity (CE) is inversely related to the sodium carbonate deadload and, therefore, the aim of most causticizing control systems is to regulate the CE of the white liquor to a uniformly high value close to the equilibrium CE, but without over-liming. Over-liming causes a completely different but no less severe set of operating problems characterized by low mud settling rates, free-lime in the white liquor, increased digester and evaporator scaling, and low lime kiln thermal efficiency [3]. The correct lime dosage is difficult to determine since it depends on the CE target value, the flow rate and chemical composition of the green liquor, and the availability, reactivity and bulk density of the lime.

Common practice in causticizing process control is to regulate the incoming green liquor total titratable alkali (TTA) upstream, starting at the smelt dissolving tank and with an optional trim just downstream of the green liquor clarifier, and then to control the conversion of sodium carbonate to sodium hydroxide in the slaker/causticizers through the regulation of CE. Careful choice of the TTA and CE targets, followed by good regulation around those targets, will produce a uniform white liquor at the target effective alkali (EA). Despite the fact that it is not overly complex, there are still very few mills that have closed-loop control of the causticizing process. This is likely due to several factors, including; a continued dependence on conventional measurements such as temperature, density and conductivity that are subject to scaling, extremely long process dynamics, and a lack of high quality liquor composition measurements. Spring et al. [4] implemented a causticizing control system consisting of lime to green liquor ratio control and 1<sup>st</sup> causticizer conductivity control. They also used the green liquor temperature to regulate the slaker temperature independent of the lime dosage. While this strategy reduced conductivity variability in the 1<sup>st</sup> causticizer, it still required the operator to manually adjust the conductivity target based on the final white liquor CE. Swanda et al. [5] compared slaker  $\Delta T$  control (slaker temperature minus green liquor temperature) to inferred 1<sup>st</sup> causticizer active alkali (AA) control in simulation. The inferred-AA control outperformed the  $\Delta T$  control by maintaining 1<sup>st</sup> causticizer and white liquor AA values closer to their targets. However, the 1<sup>st</sup> causticizer AA control system could not fully return the white liquor AA to its target value.

In this paper we present a new causticizing control system that combines liquor composition measurements and model-based inferencing of lime quality to achieve stoichiometric lime dosage control. The proposed strategy combines most of the benefits of the above mentioned approaches while simultaneously addressing the drawbacks.

Specifically, the system is able to integrate all of the available measurements and, thereby, minimize the need for manual intervention. This includes conventional measurements such as temperature and conductivity as well as liquor composition measurements from an online analyzer, manual ABC titrations, or a combination of these. This paper deals only with CE control as related work on smelt dissolving tank TTA control was reported previously in [6]. Two mill case studies, one using conventional measurements and one based on an online analyzer, are presented and discussed. These build on two prior applications recently reported in [7,8]. In all cases, the controls have been implemented directly in the mill's distributed control system (DCS).

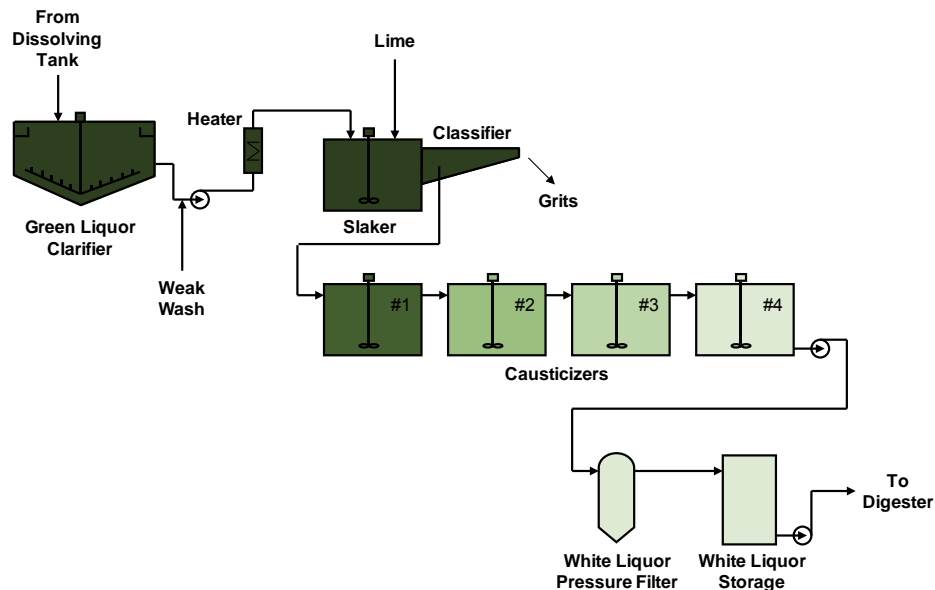
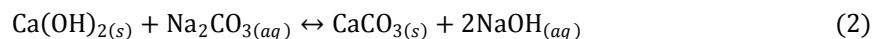
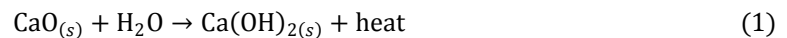


Figure 1: Simplified schematic diagram of the causticizing process showing green liquor clarifier, slaker/classifier, four causticizers, white liquor pressure filter and white liquor storage tank.

## PROCESS DESCRIPTION

The theory and practice of causticizing is well known and all of the information that is presented here is available in standard texts [9]. The slaking and causticizing reactions (Equations 1 and 2, respectively) are carried out in a series of continuous flow stirred tank reactors (CSTR) typically consisting of a green liquor clarifier, slaker/classifier, three or four causticizers, a white liquor pressure filter (or clarifier) and white liquor storage tank, see Figure 1.



The heart of the process is the slaker/classifier where the correct amount of lime ( $\text{CaO}$ ) must be applied to convert sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) in green liquor into sodium hydroxide ( $\text{NaOH}$ ) in white liquor for subsequent use in the digester. As sodium carbonate is inert in the cooking process, unconverted  $\text{Na}_2\text{CO}_3$  is an undesirable deadload that accounts for about 70% of the total deadload in white liquor [1]. It is not possible to completely eliminate this deadload as the causticizing reaction is reversible and therefore stops when the equilibrium conversion is reached. If more  $\text{CaO}$  is added than can be consumed in causticizing, unconsumed calcium hydroxide ( $\text{Ca(OH)}_2$ ) particles (free lime) end up in the unclarified white liquor. Although large clarifiers can handle 1-3% free lime without hampering the mud handling systems, pressure filters are very sensitive to free lime as this causes clogging of the socks. Since most installations have now switched to pressure filters, accurate dosing of lime and prevention of over-liming is more important than ever [3].

## STEADY-STATE ANALYSIS

In the following, it will be assumed that the process provides sufficient residence time for the above reactions to go to steady state. Equations (1) and (2) indicate that 1 mol of CaO reacts with 1 mol of Na<sub>2</sub>CO<sub>3</sub>. Define dosage to be the molar ratio of CaO to Na<sub>2</sub>CO<sub>3</sub> charged to the slaker, i.e. Equation (3)

$$\text{Dosage } (D) = \frac{\text{mol CaO}}{\text{mol Na}_2\text{CO}_3} = \frac{\text{g CaO (as Na}_2\text{O)}}{\text{g Na}_2\text{CO}_3 \text{ (as Na}_2\text{O)}} = \frac{[\text{CaO}]_0}{[\text{Na}_2\text{CO}_3]_{GL}} \quad (3)$$

Here, [CaO]<sub>0</sub> is the initial “effective concentration” of CaO charged to the slaker, i.e. that initial concentration, expressed as Na<sub>2</sub>O equivalents, which results from metering a given mass of CaO per litre of green liquor, assuming no change in volume. Sodium carbonate conversion is given by Equation (4)

$$\text{Conversion } (Y) = \frac{[\text{Na}_2\text{CO}_3]_{GL} - [\text{Na}_2\text{CO}_3]_{WL}}{[\text{Na}_2\text{CO}_3]_{GL}} = \frac{[\text{NaOH}]_{WL} - [\text{NaOH}]_{GL}}{[\text{Na}_2\text{CO}_3]_{GL}} \quad (4)$$

Note that the above results from the fact that the reduction in [Na<sub>2</sub>CO<sub>3</sub>] between green and white liquor is equivalent to the increase in [NaOH] when all concentrations are in Na<sub>2</sub>O equivalents and one ignores the small amount (< 2%) of H<sub>2</sub>O consumed during slaking. Assuming the causticizing reaction goes to steady state and that all the CaO is consumed (no over-liming) then, due to stoichiometry, the sodium carbonate conversion is numerically equivalent to the dosage. This is stated mathematically in Equation (5)

$$Y = D, \text{ provided } Y < Y_{eq} \quad (5)$$

where  $Y_{eq}$  is the sodium carbonate conversion at equilibrium. Substituting (3) and (4) into (5) we get Equation (6)

$$[\text{CaO}]_0 = [\text{NaOH}]_{WL} - [\text{NaOH}]_{GL} \quad (6)$$

[CaO]<sub>0</sub> is related to slaker inputs according to Equation (7)

$$[\text{CaO}]_0 = \frac{1}{K_L} \frac{M_L A_L}{F_{GL}} \quad (7)$$

where  $M_L$  is lime mass flow rate,  $A_L$  is lime availability,  $K_L$  is a stoichiometric constant and  $F_{GL}$  is green liquor flow rate. Furthermore, using standard definitions, the sodium hydroxide concentration may be expressed as Equation (8)

$$[\text{NaOH}] = \text{CE}(1 - S)\text{TTA} \quad (8)$$

where S is sulfidity. Substituting (7) and (8) into (6) and rearranging gives Equation (9)

$$\frac{M_L}{F_{GL}} = \frac{K_L}{A_L} (\text{CE}_{WL} - \text{CE}_{GL})(1 - S_{GL})\text{TTA}_{GL} \quad (9)$$

Here, since S and TTA are determined upstream and do not change appreciably across the causticizing process, we take the values at the feed (green liquor) condition.

Equation (9) illustrates the relationship between the main causticizing variables; the incoming clarified green liquor properties (flow rate and composition), lime quality and addition rate, and the final white liquor CE. It, therefore, provides useful insights into the causticizing process. The following comments may be made;

1. The left hand side of Equation (9) is the lime ratio, i.e.  $R_L = M_L/F_{GL}$ . Operating according to  $R_L$  serves two purposes; (i) it provided feedforward compensation for production rate changes (i.e. changes in  $F_{GL}$ ) and (ii) it modifies (linearizes) the lime mass flow rate changes required to compensate for changes in green liquor composition and/or lime quality, i.e. the compensating control actions are flow rate dependent.

2. The correct lime ratio depends, in part, on the composition of the incoming green liquor composition, i.e. green liquor CE, S and TTA. Sulfidity is closely controlled in mills and any changes are expected to be small in magnitude under normal operating conditions. Green liquor CE is primarily a function of weak wash strength, which also is not normally expected to vary significantly. TTA is the largest potential source of green liquor variability. Thus, it is important to control the TTA upstream of the slaker. As mentioned earlier, TTA is largely determined at the smelt dissolving tank, and in many cases is trimmed downstream of the green liquor clarifier.
3. Since S and TTA are reaction invariant, that leaves one additional variable that may be controlled in the white liquor through manipulation of lime ratio. In Equation (9) that variable is the final white liquor causticity,  $CE_{WL}$ . Note that expressions similar to Equation (9) may be developed for the final white liquor EA or AA.
4. The largest source of uncertainty in causticizing is the CaO mass flow to the slaker. CaO mass flow rate is the product  $M_L A_L$  in Equation (9). Firstly, since lime is fed to the slaker volumetrically via a screw feeder, changes in lime bulk density in the flights, channelling in the lime silo and wear of the screw introduce unmeasured variability in lime mass flow rate,  $M_L$ . Secondly, depending on the lime kiln operation, and requirements for and methods of adding purchased lime, the lime availability,  $A_L$ , may be also be highly variable. Thus, knowing one quantity without the other is of no practical use. So, although there is a manual laboratory test for  $A_L$ , its use would depend on precise control of  $M_L$  which, for reasons mentioned above, is impractical. Therefore, the need to compensate for CaO mass flow rate variability, regardless of its source, points towards the requirement for some type of feedback control.

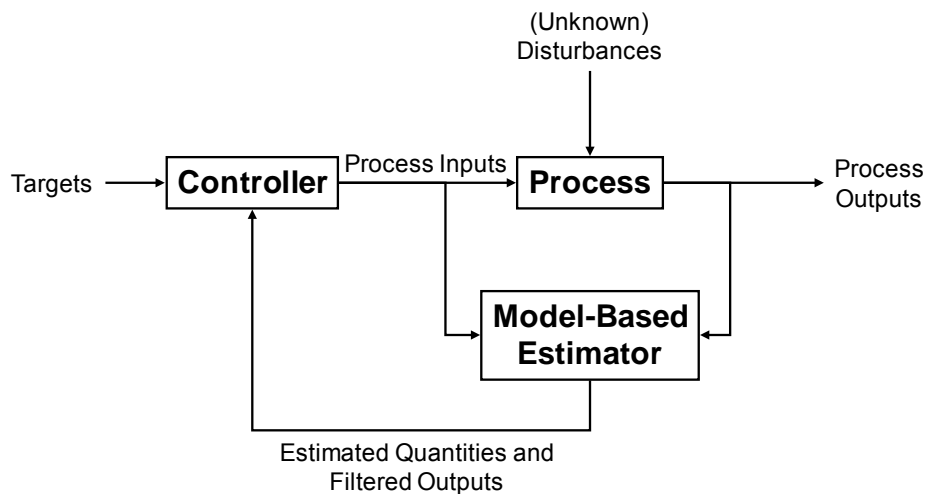


Figure 2: Block diagram of generic inferential control scheme.

## INFERENCEAL CONTROL STRATEGY

The general idea behind inferential control (Figure 2) is to run a model of the process in a computer, and use the difference between predicted model outputs and actual process outputs to infer the modeled, but unknown, quantities, which are then used either directly or indirectly for control. As indicated in point 4 above, the CaO mass flow rate,  $M_L A_L$ , is the main source of uncertainty in the causticizing process. As will become evident in the sequel, it is necessary to maintain separation between estimation and control functionality and, therefore,  $M_L$  and  $A_L$  are kept as separate variables.

Normally, the lime mass flow is calculated from the screw speed (DCS output) by a lime screw characterizer, typically a linear function, e.g. Figure 3. Denote the true lime mass flow rate by  $M_L$  and the calculated value by  $\hat{M}_L$ . Since there is no way to know the true lime mass flow,  $\hat{M}_L$  is at best a rough approximation of  $M_L$ , and cannot quantify unmeasured disturbances in the lime mass flow rate of the type discussed above. Now assume that lime availability is one of the estimated variables according to the general inferential control scheme of Figure 2, and

denote the true availability by  $A_L$  and the estimated value by  $\hat{A}_L$ . Provided certain conditions are met, then it may be established that  $\hat{M}_L \hat{A}_L \rightarrow M_L A_L$ . Therefore, substituting  $\hat{M}_L$  and  $\hat{A}_L$  in place of the true values in Equation (9) provides a means of achieving process control.

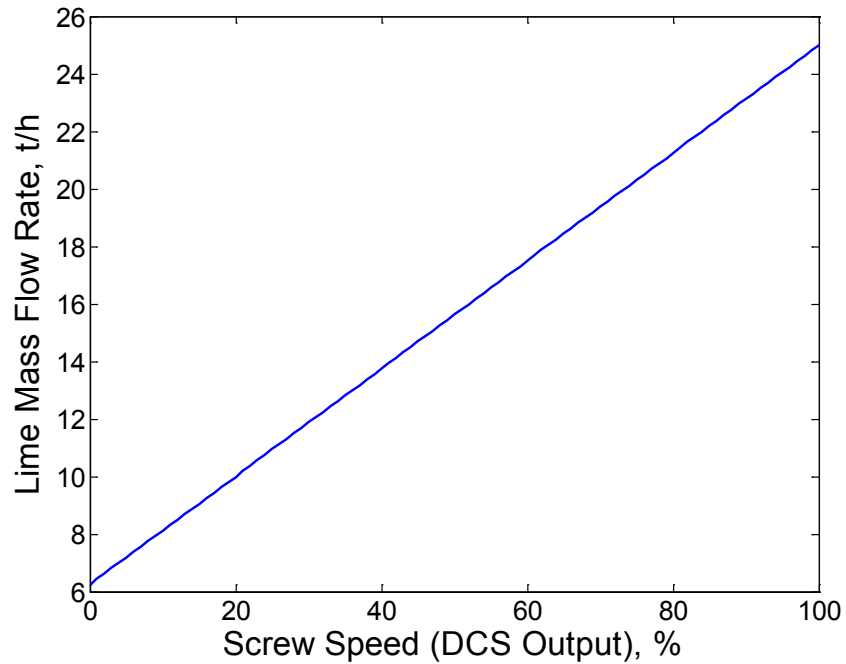


Figure 3: Typical lime screw characterizer function relating DCS output to lime mass flow rate.

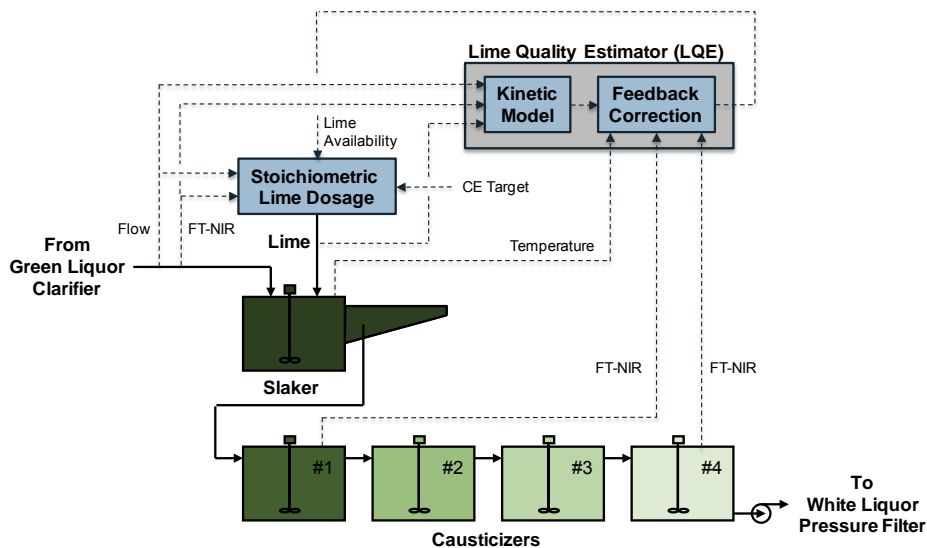


Figure 4: Causticizing inferential control scheme superimposed on the process schematic.

Figure 4 shows the causticizing inferential control scheme superimposed on the process schematic of Figure 1. Lime addition to the slaker is determined by a stoichiometric lime dosage calculation (Equation 9) where the clarified green liquor flow and chemical composition, the inferred lime availability and the white liquor CE target value are inputs. The lime availability estimate is determined by a lime quality (state) estimator (LQE). The LQE is based on a high fidelity kinetic-dynamic model of the causticizing process. The unknown lime availability is one of several

estimated variables in the model. The model is used to predict the downstream liquor temperature and composition measurements from the process inputs and the current state estimates. The difference between the model predictions and the actual measurements are then used to correct the states. This correction step takes place each time a new set of measurements becomes available.

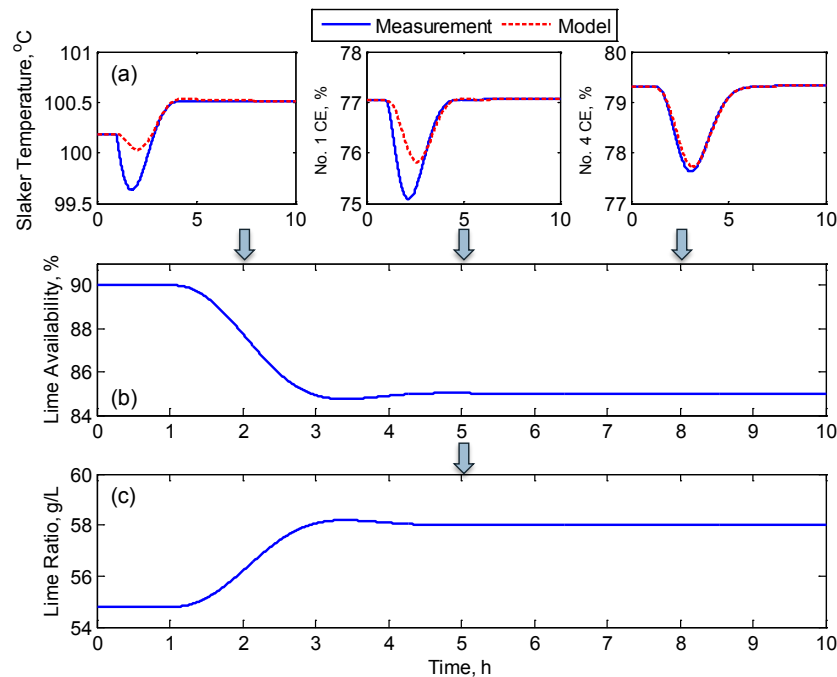


Figure 5: Simulation of inferential control scheme response to a lime availability disturbance.

Figure 5 shows a simulation of how the system of Figure 4 responds to a change (decrease) in lime availability. The measurements are plotted together with the model predictions in Figure 5a. Note that, after an initial period of steady state operation, a step change in the true lime availability (90% to 85%) causes all three measurements to decrease relative to the model predictions. This causes the lime availability estimate to decrease (Figure 5b) in order to correct the tracking error which, in turn, causes an increase in the lime ratio (Figure 5c) and an eventual correction to the CE values.

Some requirements on the model are that it needs to be causal, have relatively few parameters and be amenable to numerical integration in a digital computer. As already mentioned, one of the unknowns is the lime availability state,  $\hat{A}_L$ . The model also includes a lime reactivity state estimate, the lime specific surface  $\hat{S}_L$ , which appears as a proportionality constant in the rate expressions. The measurements consist of the slaker temperature, first causticizer composition and temperature, and last causticizer composition. One of the advantages of this inferential control approach is that the contribution of individual measurements to the state estimates may be tailored to the specific application, and additional measurements are readily integrated.

Before proceeding to the next section, some comments on these “lime quality” state estimates ( $\hat{A}_L$  and  $\hat{S}_L$ ) are warranted. As pointed out in [10], the inclusion of non-stationary stochastic disturbance states give the Kalman Filter the ability to estimate or track the observable states of the process using information from measurements taken on only a few of the states. Just as in process control, non-stationary disturbances cause the process to drift away from the deterministic model predictions, and it is only through the introduction of these additional state variables that information from the outputs gets fed back into the state estimates. This is entirely analogous to how integral action functions within feedback controllers. Hence, one of the most critical steps in designing Kalman Filters is to formulate a model for the unmeasured stochastic disturbances using as much theoretical insight as possible. The lime quality states result from the simple fact that the majority of the unmeasured disturbances in the causticizing process enter with the lime. However, it needs to be emphasized that, although some effort has been made to assign physical significance to these states, the estimates will not, in general, be numerically equivalent to the values that

one would measure by manually testing lime samples in the laboratory. For example, based on the above discussion of the limitations of the lime mass flow characterizer, it follows that any error between  $\hat{M}_L$  and  $M_L$  will result in bias of the lime availability estimate, i.e.  $\hat{A}_L \rightarrow (M_L/\hat{M}_L)A_L$ . In other words, under the proposed scheme,  $\hat{A}_L$  becomes a sink for all CaO mass flow uncertainty, including modeling errors, regardless of the source. Similar comments apply to  $\hat{S}_L$ . Thus, at best, the lime quality state estimates are proportional, but not necessarily equal, to the true values. Trending the estimates may still provide useful insights into the operation of the chemical recovery system.

## MEASUREMENTS

Advanced process control (APC) applications often rely on high quality measurements, and causticizing control is no exception. In order to implement the above strategy, information equivalent to an ABC test is needed on a minimum of three streams; the clarified green liquor inlet to the slaker, and the outlet of the first and last causticizers. Spectroscopic techniques are ideally suited to on-line measurement of key process variables and product quality attributes in the process industries because of its solid instrumentation, insensitivity to environmental factors and wide range of potential applications. The use of spectral data over a range of frequencies combined with multivariate calibration methods enables a multitude of product attributes to be measured simultaneously. When applied to process liquors, these techniques have the added advantage that multiplexing of process streams allows a single spectrometer to be used for an entire process area, or even across several areas. Furthermore, unlike automatic titrators, no reagents are required. In the mid 1990's, the Pulp and Paper Research Institute of Canada (Paprican) developed, patented and commercialized Fourier transform near infrared (FT-NIR) spectroscopy for kraft liquor measurements. The reader is referred to [11] for a thorough review of this and other developments in the area.

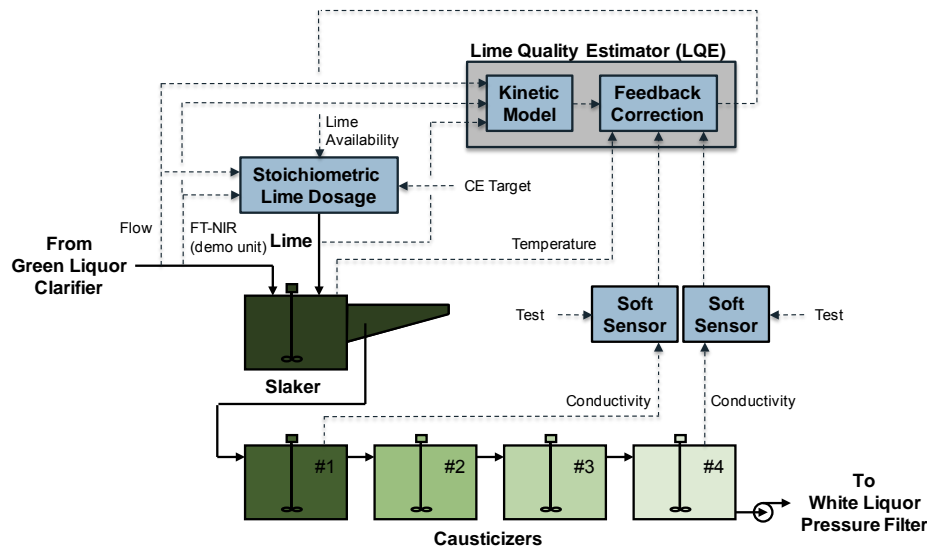


Figure 6: Causticizing inferential control scheme implemented based on soft sensors.

The FT-NIR liquor analyzer measures sodium carbonate, sodium sulfide and effective alkali (EA) concentrations, which are required for the causticizing control system described here, as well as sodium sulfate and thiosulfate, which may be used to monitor recovery boiler operations. The analyzer periodically takes a liquor sample from the process and determines the composition by spectroscopy. It provides new information to the estimator/controller at the completion of each batch analysis, which can be automated at a period depending on the amount of solids in the sample and the number of streams being analysed. From the viewpoint of control performance, [12] suggests the sampling rate should be approximately ten times the desired closed-loop bandwidth. For the causticizing process, in order to avoid significant overshoot in the lime dosage, a reasonable control objective is for the closed-loop dynamics to be roughly equivalent to the open-loop. This implies that the ideal sampling period for a typical process consisting of a slaker with a 20 minute hydraulic time constant and four causticizers, each with a 45 minute time constant, is approximately 25 minutes.

When a liquor analyzer is unavailable, the control strategy of Figure 4 may be implemented based on soft sensors, as shown in Figure 6. Here, pseudo-continuous CE measurements are determined by CE soft sensors, where conductivity is used to predict the CE values between manual ABC tests. As shown in Figure 7a, the soft sensors use a static linear (gain and offset) model to relate the CE values to conductivity. The gain is a fixed value, but the offset is estimated, i.e. when a new test is entered into the DCS, the offset is adjusted based on the error between the CE measurement and the soft sensed value, as shown in Figure 7b. In this way, the soft sensor can adjust to changes in the conductivity signal as, for example, scale accumulates on the probes. When the soft sensor is working well, it should be a good predictor of incoming CE tests.

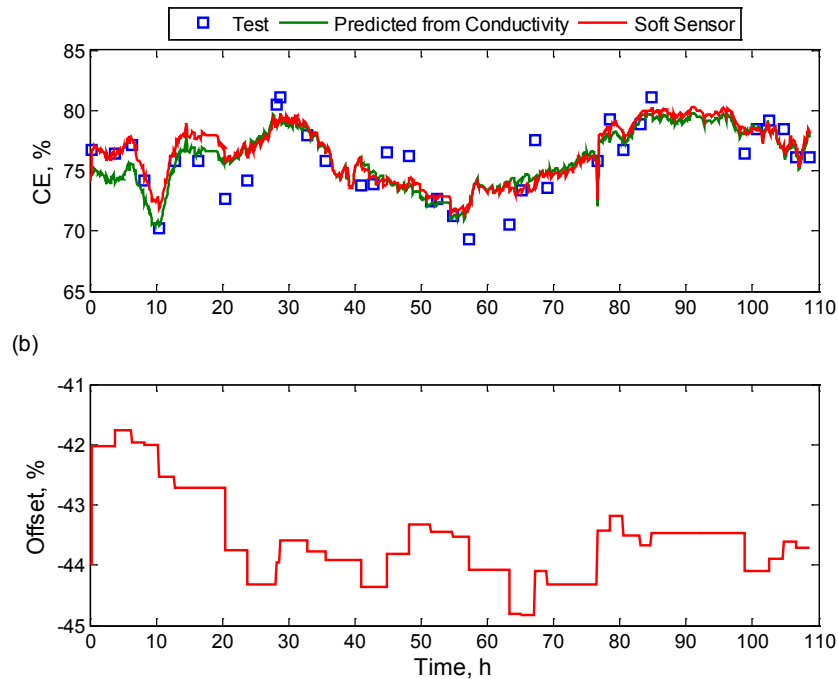
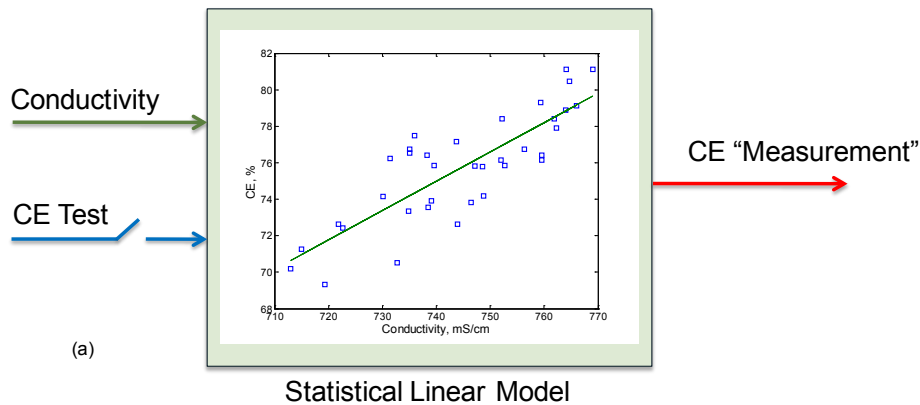


Figure 7: Causticity (CE) soft sensor; (a) static linear model, (b) offset adjustment new test is entered.

## RESULTS

In this section, two mill case studies, one using conventional measurements and one based on an online analyzer, are presented and discussed.



## Mill A

Mill A produces approximately 1850 ADMt/d of bleached kraft pulp, primarily from hardwood with occasional campaigns of softwood. The causticizing process consists of a slaker, 4 causticizers and a white liquor pressure filter. The DCS is a Honeywell TDC-3000 system. In 2010 the process was equipped with an online FT-NIR liquor analyzer. The design, installation and startup of this system is fully documented in [13]. Measurement points include the raw green liquor, clarified green liquor (CGL), classifier overflow (CO), 4<sup>th</sup> causticizer (C4), final white liquor and weak wash. The analyzer continuously cycles through a schedule consisting of CGL, CO and C4 samples, since these measurements are used for control. This provided a sampling period of 25-30 minutes for each stream, which is close to the ideal sampling period mentioned earlier. The remaining streams are inserted into the schedule on a time basis (i.e. every two hours, twice per shift, etc). Note that Mill A has CGL TTA trim control at the outlet of the green liquor clarifier and slaker temperature controller via control of the incoming green liquor temperature.

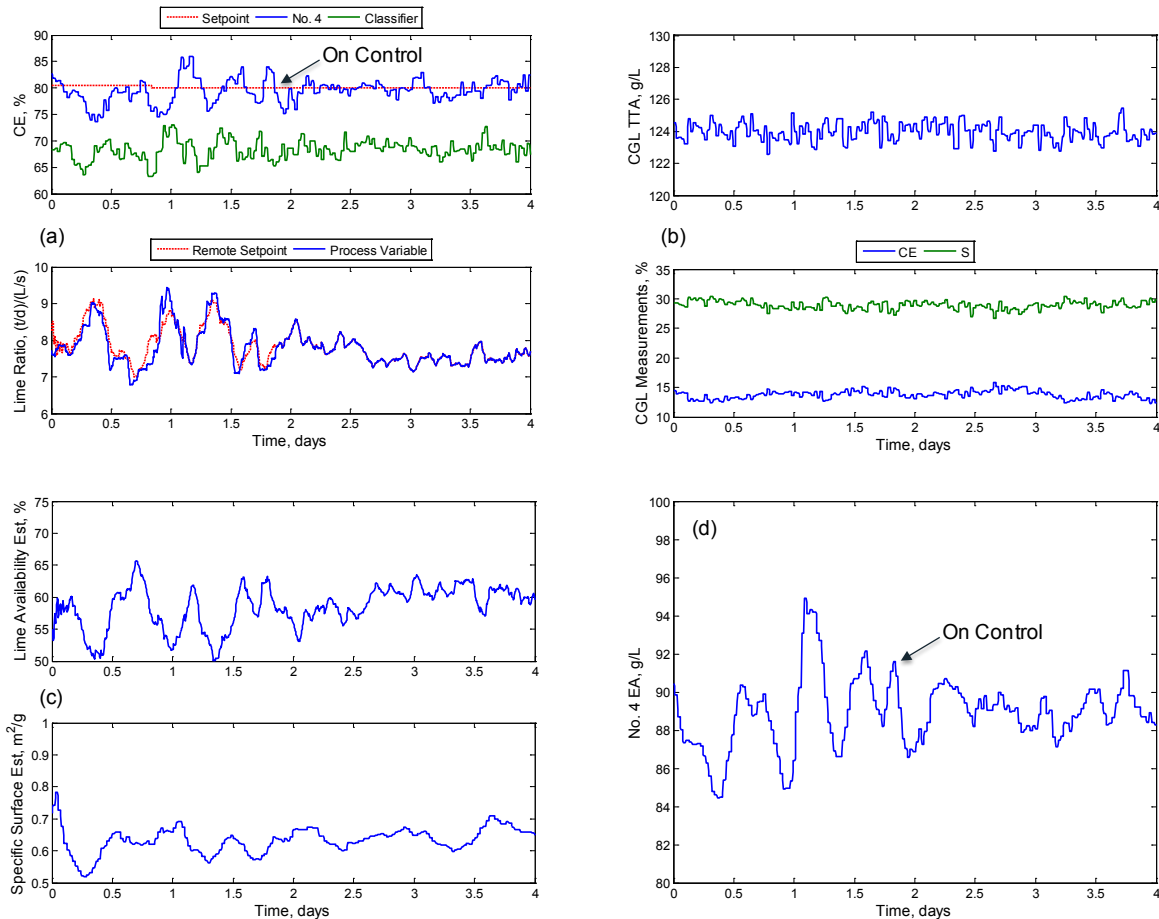


Figure 8: Four days of operation from Mill A comparing manual and automatic control performance.

Figure 8 shows four days of operation from Mill A where the CE controls were in manual for the first two days before being switched to automatic. Figure 8a shows the CE values in the upper plot and the lime ratio in the lower plot. In this case, automatic control reduced the C4 CE standard deviation from 2.9% to 1.3%, which represents a 55% reduction in variability. As shown in Figure 8b, this improvement is unrelated to any changes in incoming CGL composition variability. The CGL flow rate was also fairly steady over this period. Figure 8c shows the estimated lime quality parameters, which indicates that most of the variability is in the lime availability. Note that the control system calculates a lime ratio remote setpoint independent of the control mode. This value is used by the operators to help guide manual operation and to decide when to switch modes.

Earlier it was stated that careful choice of the TTA and CE targets, followed by good regulation around those targets, will produce a uniform white liquor at the target EA. This is illustrated in Figure 8d. As a direct result of the reduction in C4 CE variability, the C4 EA standard deviation dropped from 2.4 to 0.9 g/L, which is a 60% reduction in variability.

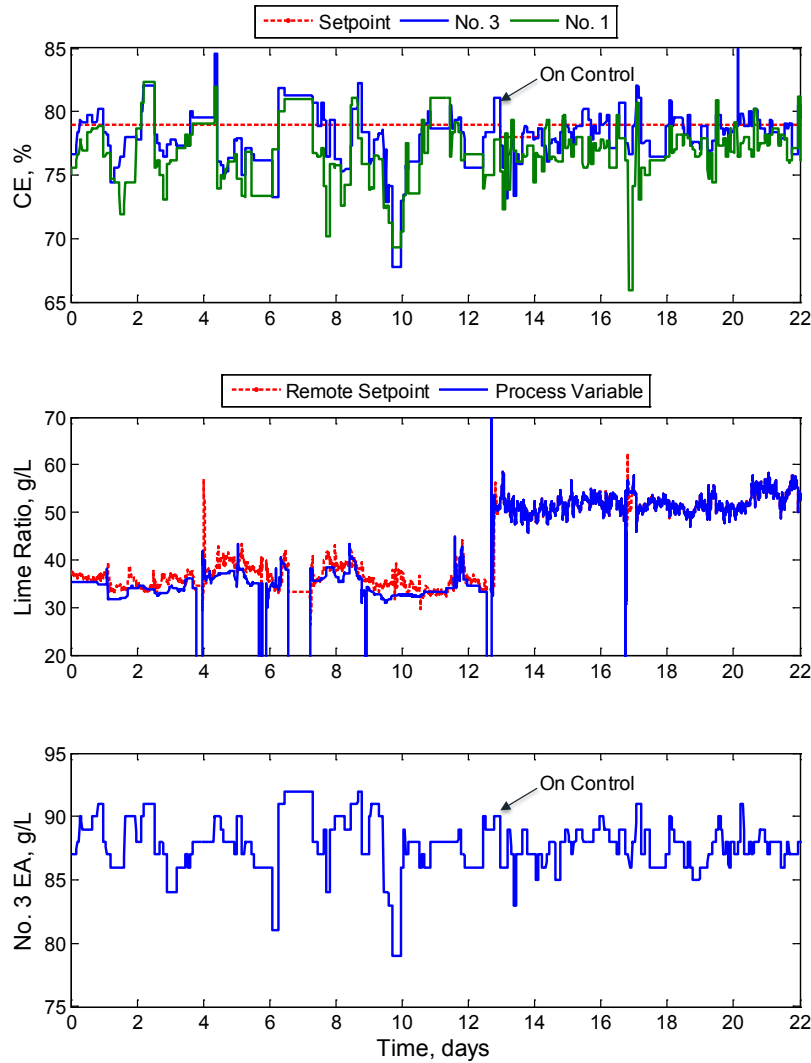


Figure 9: Three weeks of operation from Mill B where the CE controls were commissioned on day twelve.

### Mill B

Mill B produces roughly 1500 ADMt/d of northern bleached softwood kraft (NBSK) pulp. It also has a causticizing process consisting of a slaker, 4 causticizers and a white liquor pressure filter, and a Bailey INFI 90 DCS. Since Mill B does not have an online liquor composition measurement system, an FT-NIR demonstration unit was installed for a six month trial. The trial unit consists of a flow cell, industrial spectrometer and computer in a single cabinet located on the plant floor in close proximity to the sample location. Since the demonstration unit is only capable of sampling a single stream, the CGL stream was chosen because it provides the opportunity to do TTA trim control upstream of the slaker as well as the composition measurements required in the stoichiometric lime dosage

calculation. Note that since the trial unit is dedicated to a single stream, the sampling frequency can be extremely fast – in this application it was 10 minutes. Furthermore, the downstream liquor composition measurements used in the LQE were provided by CE soft sensors of the type discussed earlier (see Figure 7), which in this case were driven by conductivity probes located in the 1<sup>st</sup> and 3<sup>rd</sup> causticizers.

Figure 9 shows roughly three weeks of operation from Mill B where the CE controls were commissioned on day twelve. Note that the hot lime screw was recalibrated just prior to the switch to automatic. This was required to achieve smoother operation during manual adjustments of the pebble lime screw. Here, automatic control reduced the C3 CE standard deviation from 2.7% to 1.5%, or a 45% reduction in variability. As a direct result of this, the C3 EA standard deviation dropped from 2.6 to 1.4 g/L.

Mill B is now reliably running to a CE target of 81%. This is within a few percentage points of the equilibrium CE since this mill typically runs a white liquor TTA approaching 130 g/L. Although there has not been sufficient run time to do a full economic benefit analysis as yet, the early evidence points towards a reduction in soda make-up chemicals (caustic and soda ash), purchased lime consumption, and natural gas consumption in the kiln. Savings of a similar nature were reported in an earlier mill application [7]. The link between high deadload and soda losses is well known [1,2]. High deadload operation requires an increase in wash water to reach the target weak black liquor solids, thus increasing washing losses. Also, high deadload increases chemical losses through increased generation of ash in the recovery boiler, which is subsequently purged as precipitator catch. Reductions in purchased lime consumption and improvements in lime kiln thermal efficiency are a direct result of decreased over-liming, which results in better white liquor filterability, lime mud processing and lime kiln operation [3].

## CONCLUSIONS

In this paper, we presented a new causticizing control system that combines liquor composition measurements and model-based inferencing of lime quality to achieve stoichiometric lime dosage control. The system is extremely flexible as it is able to integrate conventional measurements such as temperature and conductivity, as well as liquor composition measurements from an online analyzer, manual ABC titrations, or a combination of these. Results from two mill case studies, one involving conventional measurements and one based on an online analyzer, demonstrated variability reduction in the range of 45 – 55% in the last causticizer causticity (CE) values. Improved CE control, combined with control of the incoming clarified green liquor total titratable alkali (TTA), achieved corresponding reductions in white liquor effective alkali (EA) variability. Economic benefits include reductions in soda make-up chemicals (caustic and soda ash), purchased lime consumption, and natural gas consumption in the kiln.

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