Formation of pirssonite in green liquor handling systems

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ABSTRACT: The formation of pirssonite is commonly believed to be the cause of hard-scale buildup in green liquor handling systems in kraft pulp mills. The precipitation occurs when the concentration of sodium carbonate in the liquor exceeds the solubility of pirssonite. A laboratory study was conducted to systematically determine the causticizing conditions under which pirssonite precipitates. The results confirmed literature data showing that the solubility of pirssonite increases with temperature but decreases with increased concentrations of sodium carbonate and other sodium salts present in the green liquor. The solubility data obtained were used to create a database for pirssonite formation in OLI, a thermodynamic program for predicting phase stabilities of alkali salts in aqueous solutions. The OLI program, with the newly created database, was subsequently used to generate a series of pirssonite solubility curves in terms of saturated total titratable alkali that can be used as operational guidelines to prevent pirssonite precipitation and hard-scale formation in green liquor handling systems. A case study was performed using these solubility curves to explain the occurrence of the pirssonite deposition problem at a kraft pulp mill.

Application: Mill engineers can determine the maximum total titratable alkali that the mill can operate without having problems with pirssonite precipitation and hard-scale formation in green liquor handing systems.

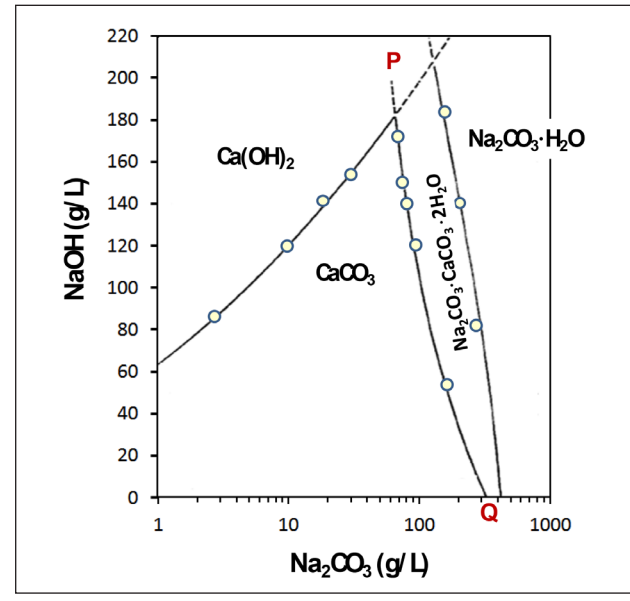


In the kraft chemical recovery plant, molten smelt from the recovery boiler is dissolved in the dissolving tank with weak wash from the causticizing plant to produce green liquor. Scaling in green liquor processing equipment and transportation lines, such as dissolving tanks, storage tanks, pipelines, clarifiers, and slakers has been a persistent problem in many mills. Scale deposits are usually rock hard and can drastically restrict green liquor flow. In severe cases, they can lead to lengthy process downtimes for scale removal.

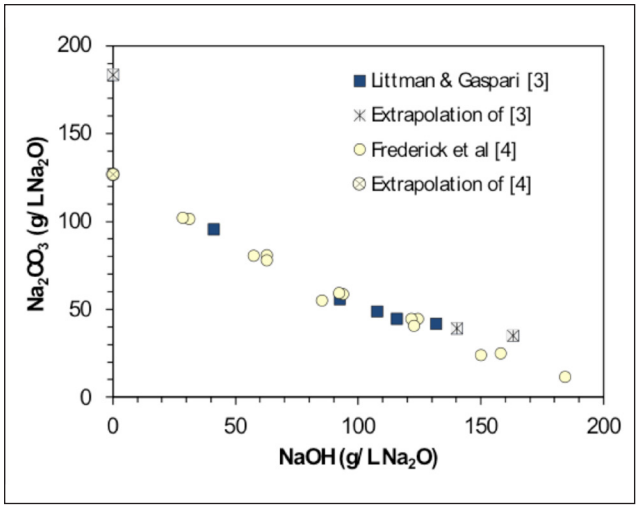
In a recent study [1], we characterized 12 scale samples from 10 kraft mills and found that only 4 of the samples were composed of pirssonite ($Na_2CO_3 \cdot CaCO_3 \cdot 2H_2O$); the remaining 8 samples were mainly composed of calcium carbonate ($CaCO_3$). We postulated that the presence of $CaCO_3$ in the majority of the scale samples was likely due to the precipitation of pirssonite scale under supersaturated conditions, followed by selective dissolution of sodium carbonate (Na_2CO_3) from the pirssonite scale, leaving the insoluble $CaCO_3$ behind during the weak wash switch in the pipelines. It is, therefore, important to understand the solubility of pirssonite in green liquor and how it is affected by the operating conditions of the causticizing plant, in order to minimize scale formation.

Only a few studies on pirssonite solubility have been performed to date. Bury and Redd [2] were probably among the first to examine the solubility of pirssonite. Their work in 1933 on the phase stability of the system Na_2CO_3-$CaCO_3$-H_2O in a temperature range of 15°C–95°C showed that pirssonite forms only in concentrated solutions of Na_2CO_3 (>20 wt%) at temperatures >40°C. More than two decades later, Littman and Gaspari [3] studied the causticizing reaction between Na_2CO_3 with slaked lime at 95°C. They proposed a phase stability diagram for the system Na_2CO_3-$Ca(OH)_2$-NaOH-H_2O (**Fig. 1**), showing that pirssonite can exist only within specific concentration ranges of Na_2CO_3 and sodium hydroxide (NaOH) in the solution.

Frederick et al. [4] investigated the effects of sodium compounds, mainly NaOH, sodium sul-



1. System of $Ca(OH)_2$-NaOH-Na_2CO_3 in water at 95°C [3].



2. Effect of NaOH and other sodium compounds on the solubility of pirssonite at 95°C.

fide (Na_2S), and sodium sulfate (Na_2SO_4), on the solubility of pirssonite in various slurries of $CaCO_3$ in Na_2CO_3 solutions at 55°C, 75°C, and 95°C. They reported that the solubility increases with temperature but decreases with an increase in concentrations of other sodium compounds. As shown in **Fig. 2**, their data at 95ºC were in good agreement with Littman and Gaspari's data [3] at the same temperature. However, there is a large discrepancy between the extrapolated values for these two sets of data at the zero-NaOH concentration. Frederick et al.'s extrapolated data [4] show that the Na_2CO_3 concentration required for pirssonite to precipitate at the zero-NaOH concentration was 127 g/L sodium oxide (Na_2O), significantly lower than that suggested by Littman and Gaspari's data (point Q in Fig. 1), which is 314 g/L Na_2CO_3 or 184 g/L Na_2O.

Ulmgren et al. [5] extended the work of Frederick et al. [4] by carrying out laboratory experiments at higher temperatures (95°C–125°C) to include the operating conditions of the green liquor produced from a black liquor gasifier. They proposed a linear model that calculates the pirssonite solubility in green liquor solutions. However, they did not provide experimental data in regions where the concentrations of other sodium salts are low.

Green liquor contains mostly Na_2CO_3 and Na_2S with small amounts of NaOH, Na_2SO_4, sodium chloride (NaCl), and potassium salts. The total concentration of Na_2S and Na_2SO_4 typically ranges from 30 to 50 g/L Na_2O, depending on the liquor sulfidity. The concentrations of NaOH and NaCl are, respectively, 5–10 g/L and <2 g/L Na_2O. Thus, the total concentration of sodium salts other than Na_2CO_3 in the green liquor may range from 35 to 60 g/L Na_2O. While this falls within the experimental range of 30–185 g/L Na_2O for other sodium salts as shown in Fig. 2, the lack of solubility data in the concentration range <30 g/L Na_2O for other sodium salts makes it difficult to evaluate whether the extrapolated value at 0 g/L Na_2O is accurate.

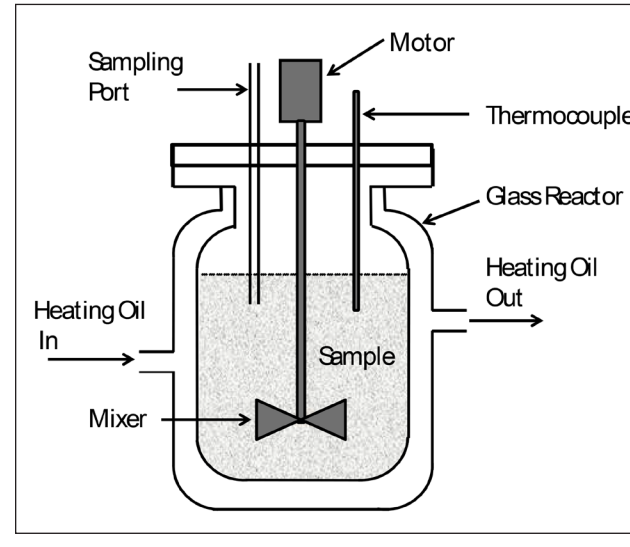
The accuracy of the solubility data at or near 0 g/L Na_2O for other sodium salts is critically important for the development of a reliable database for pirssonite formation in OLI, a commercially available thermodynamic program for predicting phase stabilities of alkali salts in aqueous solutions. The program has been used to successfully predict the solubility of recovery precipitator ash [6], as well as to evaluate the effects of chloride, potassium, and sulfate on the equilibrium causticizing efficiency of kraft liquor [7].

In this study, a large number of laboratory experiments were carried out to examine the effects of various causticizing conditions on pirssonite formation. The objectives were to obtain the solubility of pirssonite, particularly at low concentrations of other sodium salts (<30 g/L Na_2O), to incorporate the obtained solubility data into an OLI-based model and to use it to predict a wide range of conditions leading to pirssonite formation in the causticizing plant. It is important to note that the pirssonite scale samples in our previous work on green liquor scale characterization [1] and those collected later from various mills were all found to contain about 18 wt% sodium (as Na) and <0.05 wt% potassium (as K). The presence of such a small amount of potassium in the scale samples implies that potassium compounds do not coprecipitate with pirssonite and, thus, do not take part in pirssonite scale formation. As a result, the effect of potassium was not investigated in this study.

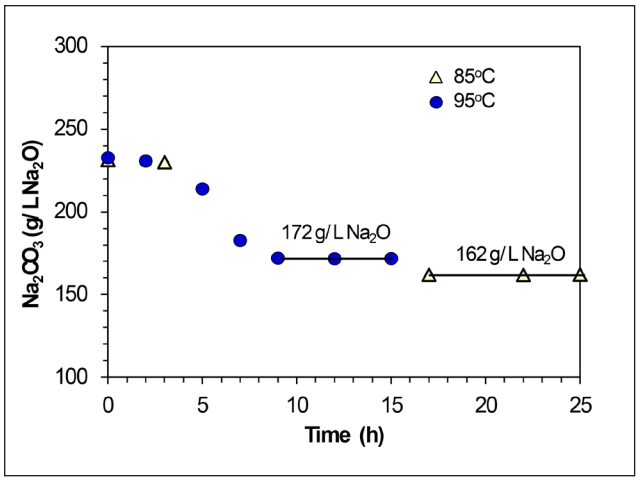
This paper discusses the results of the study and how the information can be used to minimize hard-scale formation in the green liquor handling system.

EXPERIMENTAL

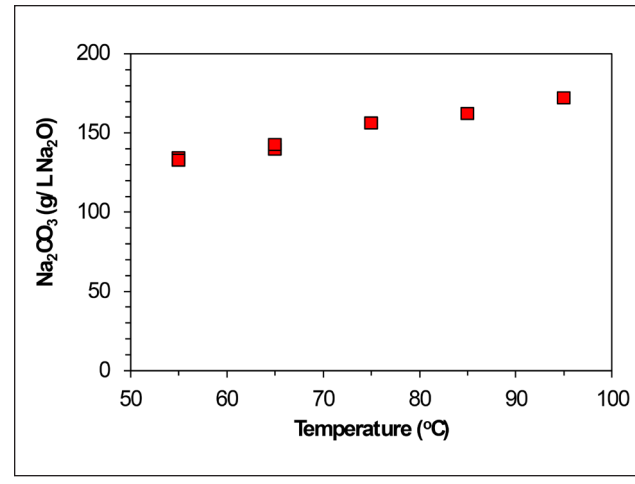
Figure 3 shows the experimental setup used in this study. It consisted of a 1-L jacketed reactor vessel, a thermocouple, a stirrer, and a sampling port. The temperature of the slurry (a mixture of $CaCO_3$ and alkaline solution) in the reactor was controlled by circulating hot oil from a temperature-con-



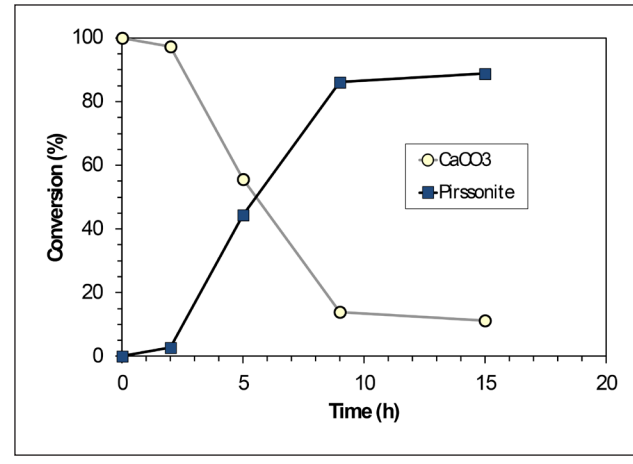
3. Experimental setup used for solubility experiments.



4. Reaction kinetics in the pure system of Na_2CO_3-$CaCO_3$-H_2O at 85°C and 95°C.



5. Pirssonite solubility in the Na_2CO_3-$CaCO_3$-H_2O system.



6. Conversion of $CaCO_3$ to pirssonite with time at 95°C.

trolled oil bath through the reactor jacket using a pump. The slurry was well mixed using a stirrer inserted from the top of the reactor.

All alkaline solutions were prepared by dissolving appropriate amounts of reagent-grade chemicals (Na_2CO_3, Na_2S, and NaOH) in deionized water. At a set time interval, a small amount of slurry (solid $CaCO_3$ particles in the alkaline solution in the reactor) was withdrawn using a syringe attached to a dip-tube inserted through the sampling port and filtered using a 0.45-µm nylon syringe filter. The filtrate was immediately diluted with deionized water to prevent further precipitation. The solids were further extracted using 0.15-µm filter paper. The syringe, dip-tube, and syringe filters were all preheated to the solution temperature to assure the withdrawn sample was maintained at the solution temperature.

Pirssonite solubility

The solubility of pirssonite was studied for various slurries of $CaCO_3$ mixed with aqueous solutions of Na_2CO_3, NaOH, and Na_2S in the Na_2CO_3-$CaCO_3$-H_2O; Na_2CO_3-NaOH-$CaCO_3$-H_2O; and Na_2CO_3-Na_2S- $CaCO_3$-H_2O systems.

Na_2CO_3-$CaCO_3$-H_2O system

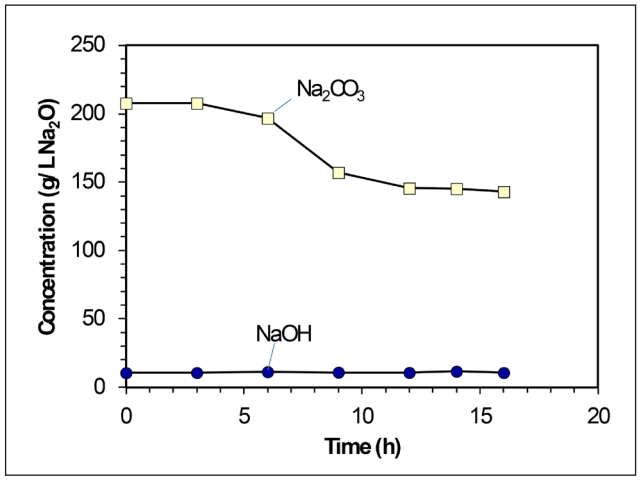
The solubility of pirssonite for this system was examined at 55°C, 65°C, 75°C, 85°C, and 95°C. An excess amount of $CaCO_3$ was added to a solution of Na_2CO_3. As the reaction proceeded at each temperature, a small amount of the solution was collected at different time intervals, and the Na^+ concentration in the solution was determined using atomic absorption spectroscopy (AAS). The Ca^{2+} concentration in the solution was also analyzed using AAS but was undetectable due to the low solubility of $CaCO_3$ in an alkaline solution.

Figure 4 shows the change in Na_2CO_3 concentration in the solution with time for the reactions performed at 85°C and 95°C. The Na_2CO_3 concentration decreased steadily with time until it reached a plateau, indicating that equilibrium had been reached. The equilibrium Na_2CO_3 concentration at 85°C was about 162 g/L Na_2O, slightly lower than 172 g/L Na_2O at 95°C, and it took 10–20 h to reach equilibrium. These equilibrium concentrations are essentially the solubility of pirssonite at 85°C and 95°C. Similar experiments were also performed at lower temperatures of 55°C, 65°C, and 75°C. The results are summarized in **Fig. 5**, which shows that the equilibrium Na_2CO_3 concentration or the solubility of pirssonite in the Na_2CO_3-$CaCO_3$-H_2O system increases with an increase in temperature.

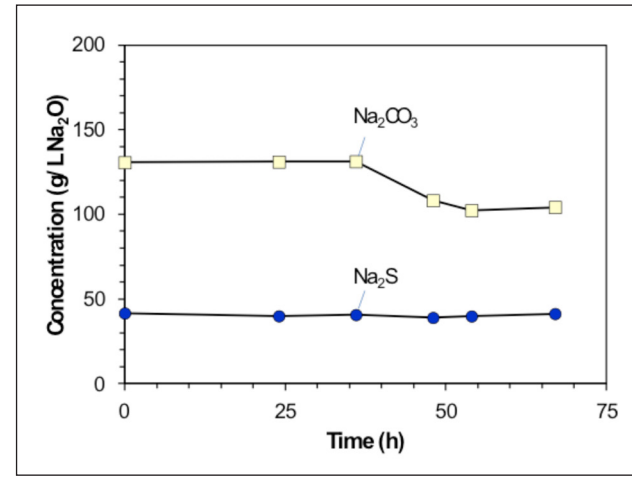
The solid samples were also collected from the reaction vessel at different time intervals and analyzed using a simultaneous thermogravimetric analyzer and differential scanning calorimetry (TGA/DSC) for the pirssonite that formed and the $CaCO_3$ that still remained in the samples. This analytical technique has been described in the previous paper [1]. As shown in **Fig. 6**, the solids consisted of mainly $CaCO_3$ at the beginning of the reaction and converted to pirssonite as time progressed.

Na_2CO_3-NaOH-$CaCO_3$-H_2O system

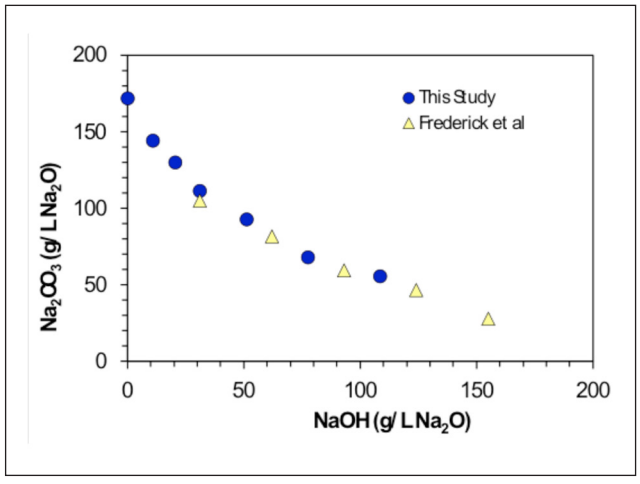
For this system, instead of only Na_2CO_3, alkaline solutions were prepared by dissolving appropriate amounts of NaOH



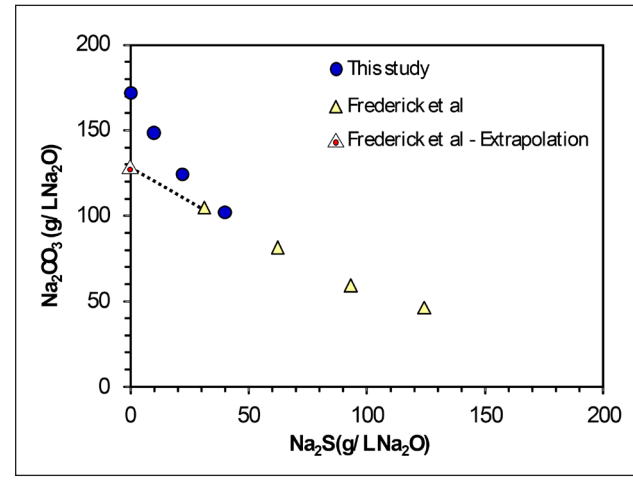
7. Reaction kinetics for NaOH = 10.5 g/L of Na_2O at 95°C.



9. Reaction kinetics for Na_2S = 40 g/L of Na_2O at 95°C.



8. Solubility of pirssonite in the Na_2CO_3-NaOH-$CaCO_3$-H_2O system at 95°C.



10. Solubility of pirssonite in the Na_2CO_3-Na_2S-$CaCO_3$-H_2O system at 95°C.

and Na_2CO_3 in deionized water at 95°C. As in the previous case, an excess amount of $CaCO_3$ powder was placed in the reactor. The slurry was continuously agitated and samples were taken at regular intervals. The concentrations of OH- and CO_3^{2-} ions in the liquid samples were determined by titrating with 1N hydrochloric acid. The solids were dried and analyzed for pirssonite and $CaCO_3$ using TGA/DSC.

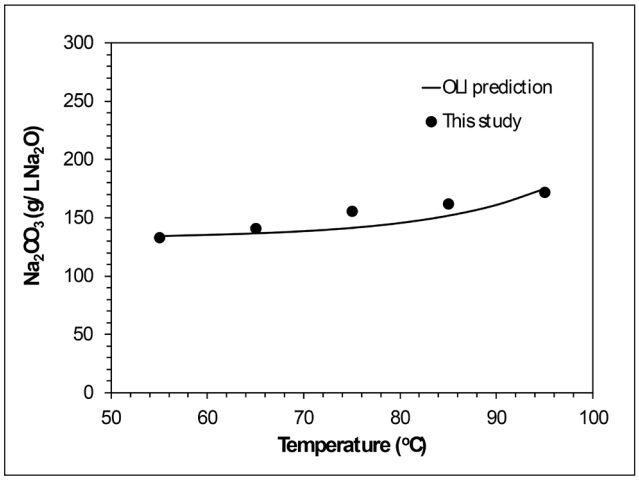
Figure 7 shows the change in NaOH and Na_2CO_3 concentrations observed in one of the experiments. The NaOH concentration in the solution remained constant at 10.5 g/L Na_2O, while the Na_2CO_3 concentration decreased from its initial concentration of 208 g/L Na_2O as more and more Na_2CO_3 from the solution reacted with $CaCO_3$ to form pirssonite. It took about 12 h for the Na_2CO_3 concentration to reach an equilibrium value of 144 g/L Na_2O, which is essentially the solubility of pirssonite under the experimental conditions.

Figure 8 shows the solubility of pirssonite as a function of the concentration of NaOH. The results are in good agreement with that of Frederick et al. [4]. However, the solubility data in the dilute NaOH concentration range (<30 g/L Na_2O) were higher than those predicted by extrapolation of Frederick et al.'s data in Fig. 2.

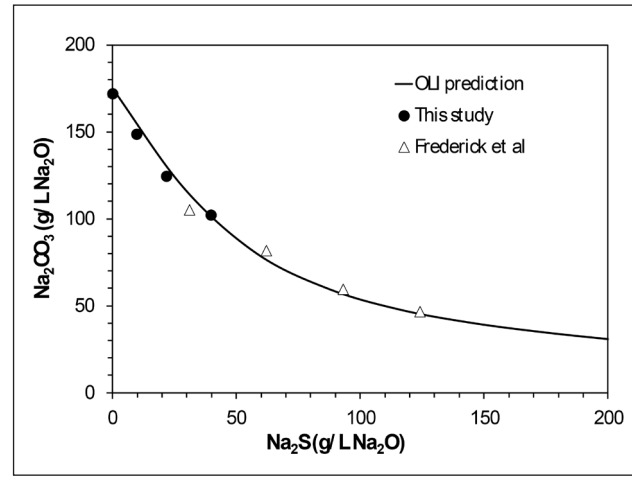
Na_2CO_3-Na_2S-$CaCO_3$-H_2O system

This system was studied to examine the effect of Na_2S on the solubility of pirssonite at 95°C. The concentrations of OH-, HS-, and CO_3^{2-} ions in the solution were determined using the ABC titration method in accordance with the TAPPI standard for green liquor analysis (T624os-68 "Analysis of soda and sulfate, white liquor and green liquors"). **Figure 9** shows the change in Na_2S and Na_2CO_3 concentrations observed in one of the experiments. The Na_2S concentration in the solution remained constant at 40 g/L Na_2O, while the Na_2CO_3 concentration decreased from its initial concentration of 130 g/L Na_2O to an equilibrium value of 110 g/L Na_2O.

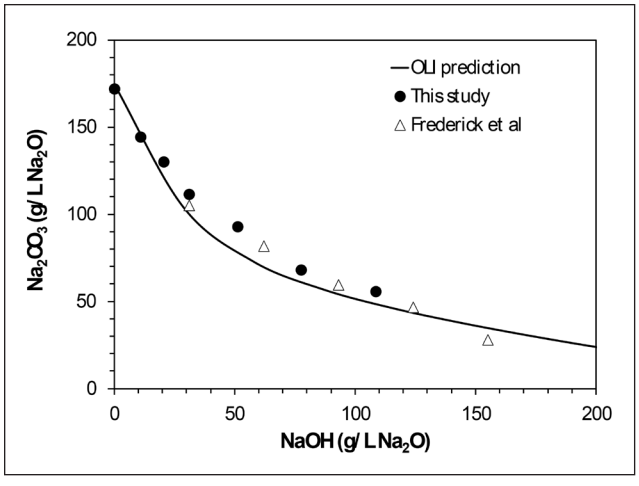
Note that in this case it took a much longer time (more than 50 h) for the Na_2CO_3 concentration to reach an equilibrium value compared to the two other systems with no Na_2S. No explanation for this observation can be given at this time. It was also observed that as soon as $CaCO_3$ powder was intro-



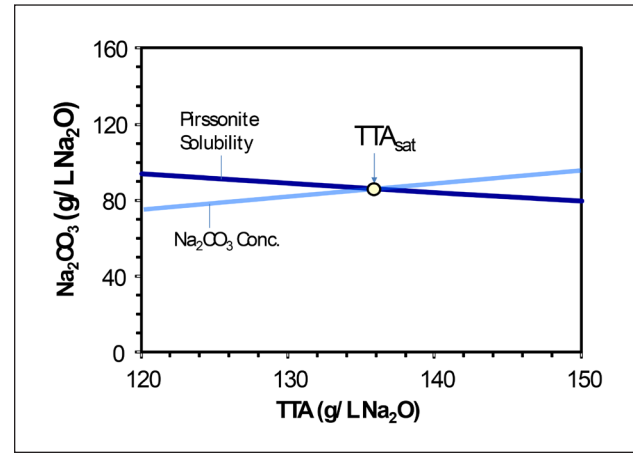
11. Comparison of experimental data and model predictions for pirssonite solubility in the Na_2CO_3-$CaCO_3$-H_2O system from 55°C to 95°C.



13. Comparison of experimental data and model predictions for pirssonite solubility in the Na_2CO_3-Na2S-$CaCO_3$-H_2O system at 95°C.



12. Comparison of experimental data and model predictions for pirssonite solubility in the Na_2CO_3-NaOH-$CaCO_3$-H_2O system at 95°C.



14. Calculation of TTA_{sat} value from green liquor solutions with 30% sulfidity and 10% causticity at 95°C using OLI.

duced in the clear solution of Na_2CO_3 and Na_2S, the slurry turned into a pastel green color whereas in the other two systems the slurry was white throughout the experiments.

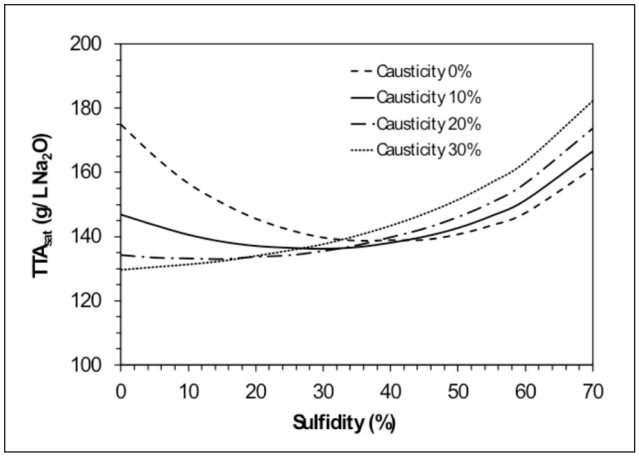
Figure 10 shows the solubility of pirssonite in the presence of Na_2S. The results are in good agreement with Frederick et al. [4] in the higher Na_2S concentration range (near 40 g/L Na_2O). At low Na_2S concentrations (particularly near 0 g/L Na_2O), however, the solubility data obtained from this study were higher than those obtained by extrapolation of Frederick et al.'s work (broken line in Fig. 10).

Prediction of pirssonite solubility using OLI

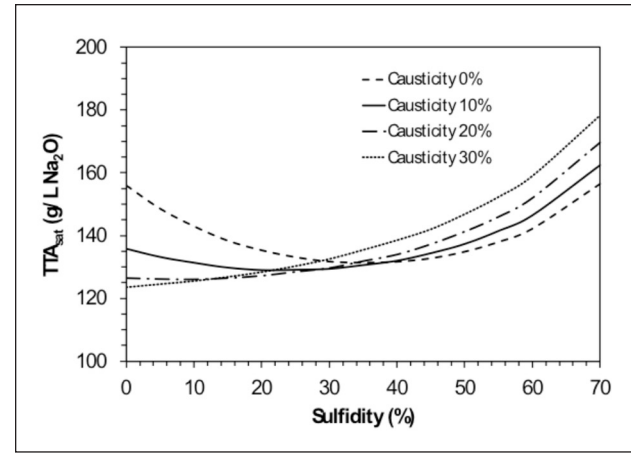
OLI is a thermodynamic-based computer program that has been used in various industries to simulate aqueous processes. It has a public database that contains thermodynamic data for most common compounds, such as Na_2CO_3 and Na_2S, although not for pirssonite. In this study, the experimental data presented above were first used to obtain the thermodynamic data for pirssonite, which were then incorporated into OLI's main database to create a computer model that can be used to calculate the solubility of pirssonite under various conditions. **Figures 11–13** show the comparison between the results predicted by the model (solid curves) and the experimental data under the same conditions. The agreement was good in all cases.

Effects of causticizing plant operating conditions

The model was used to calculate the solubility of pirssonite under various operating conditions prevailing in the causticizing plant of kraft pulp mills. At a given sulfidity and causticity, the concentrations of Na_2CO_3, Na_2S, and NaOH increase with an increase in TTA. **Figure 14** shows an example of this at 30% sulfidity and 10% causticity. The concentration of Na_2CO_3 in green liquor (light line) increases as TTA increases, while the solubility of pirssonite (dark line) decreases. The intersec-



15. Calculated pirssonite solubility curves for green liquor solutions with 0 mol% Cl/(Na+K) at 95°C.



16. Calculated pirssonite solubility curves for green liquor solutions with 10 mol% Cl/(Na+K) at 95°C.

tion of these two lines represents the point at which the concentration of Na_2CO_3 in g/L Na_2O is equal to the solubility of pirssonite in g/L Na_2O in the solution. The TTA value at this point is called saturated TTA, or TTA_{sat}. This green liquor TTA value is critical because above it, pirssonite will precipitate if $CaCO_3$ is present.

Effect of sulfidity and causticity

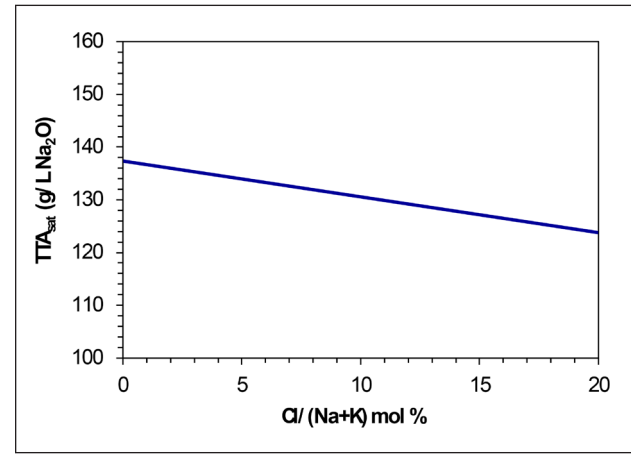
The TTA_{sat} of 136 g/L Na_2O shown in Fig. 14 is only for green liquors with 30% sulfidity and 10% causticity and at 95°C. It is expected to change with liquor composition and temperature. A series of graphs similar to those in Fig. 14 were created to determine the TTA_{sat} at 95°C for green liquor with sulfidity ranging from 0% to 70% and causticity varying from 0% to 30%.

The results are summarized in **Fig. 15**. The curves in this graph can be used by mill operators or process engineers to determine the maximum TTA their mill could operate before experiencing pirssonite formation problems. For example, at 28% sulfidity (on TTA), 10% causticity, and 95°C, TTA_{sat} is about 136 g/L Na_2O. This means that the operating TTA must be kept <136 g/L Na_2O to avoid pirssonite deposition.

Effect of chloride

The effect of chloride on pirssonite solubility is of interest to many kraft mills, particularly those located in coastal regions. Chloride (Cl^-) may exist as NaCl and/or KCl in green liquor. In this work, the effect of chloride on pirssonite solubility was investigated by adding NaCl and KCl to the inputs of the OLI model.

Figure 16 shows the TTA_{sat} curves for conditions similar to those in Fig. 15, but with 10 mol% Cl/(Na+K). Comparing these two figures shows that at a given sulfidity and causticity, the pirssonite solubility in terms of TTA_{sat} is lower with Cl^- than without Cl^-. The effect can be seen more clearly in **Fig. 17**, which shows the correlation between TTA_{sat} and Cl^- concentration in green liquor with 30% sulfidity and 5% causticity. The TTA_{sat} decreases linearly as chloride concentration

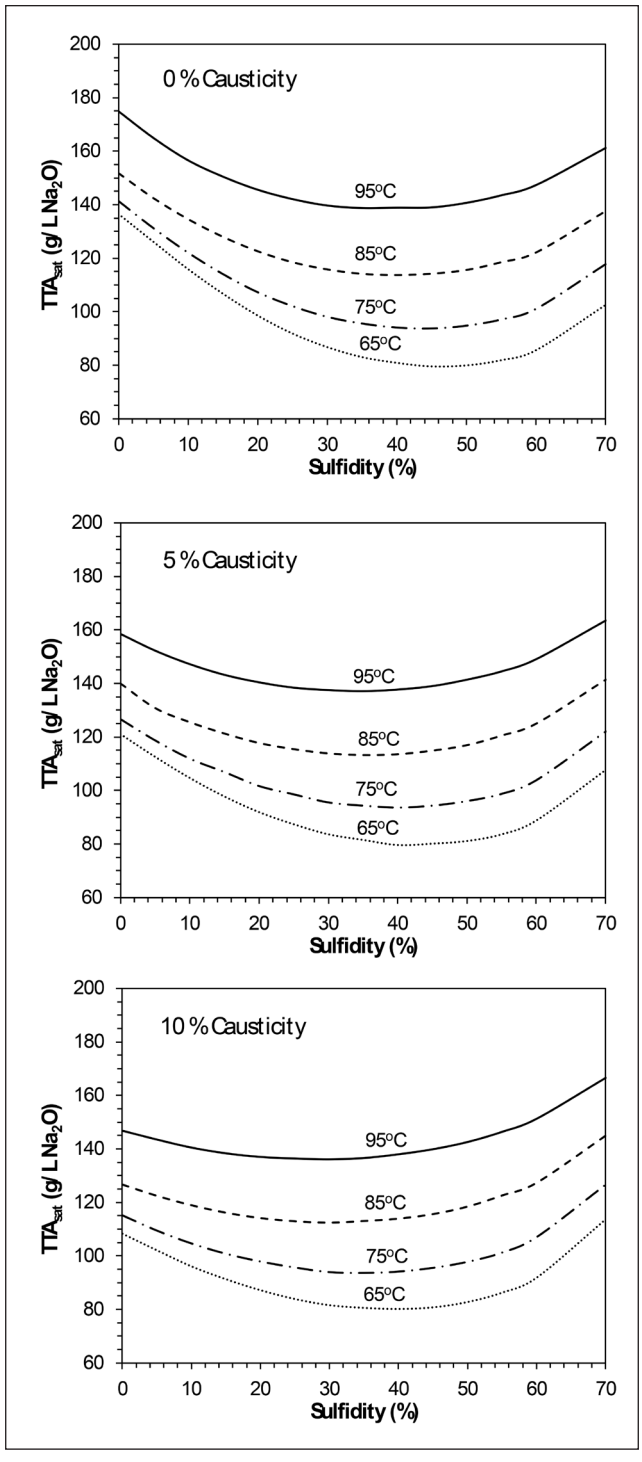


17. Effect of chloride on calculated pirssonite solubility at 95°C with 30% sulfidity and 5% causticity.

increases, from 138 g/L Na_2O at 0 mol% Cl/(Na+K) to 124 g/L Na_2O at 20 mol% Cl/(Na+K). For inland mills, however, the variation in Cl^- concentration in green liquor is typically small, 1–2 mol% Cl/(Na+K). This means that although Cl^- has an effect on TTA_{sat}, the change in pirssonite solubility due to Cl^- variation in an inland mill is insignificant. For coastal mills, the Cl^- concentration may reach as high as 12 mol% Cl/(Na+K), and thus can have substantial effects.

Effect of temperature

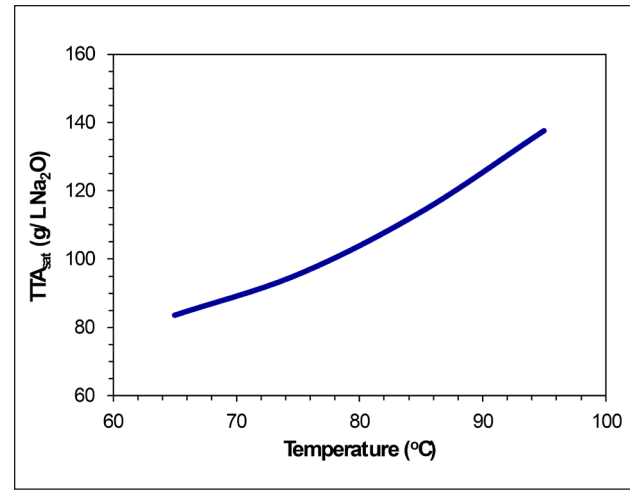
As shown in Fig. 5, the solubility of pirssonite in the Na_2CO_3 solution increases by about 10 g/L Na_2O for every 10°C increase in temperature. Green liquor contains not only Na_2CO_3, but also Na_2S, NaOH, and other sodium salts. Its temperature usually varies between 70°C and 100°C. Therefore, it is important to examine how the TTA_{sat} for green liquor might vary in this temperature range. Because the causticity of green liquor under normal conditions is between 5% and 10%, three sets of TTA_{sat} (or solubility) curves at four temperature levels (65°C, 75°C, 85°C, and 95°C) were created using the OLI



18. Effect of temperature on calculated pirssonite solubility at 0%, 5%, and 10% causticity.

model. The results are shown in **Fig. 18** at 0%, 5%, and 10% causticity levels. These graphs can be used by kraft mills to determine the TTA_{sat} at their operating conditions.

Figure 19 shows the effect of temperature on TTA_{sat} for green liquor under typical causticizing plant operating conditions of 30% sulfidity and 5% causticity.



19. Effect of temperature on calculated pirssonite solubility at 30% sulfidity, and 5% causticity.

CASE STUDY: APPLICATION OF TTA_{SAT} CURVES

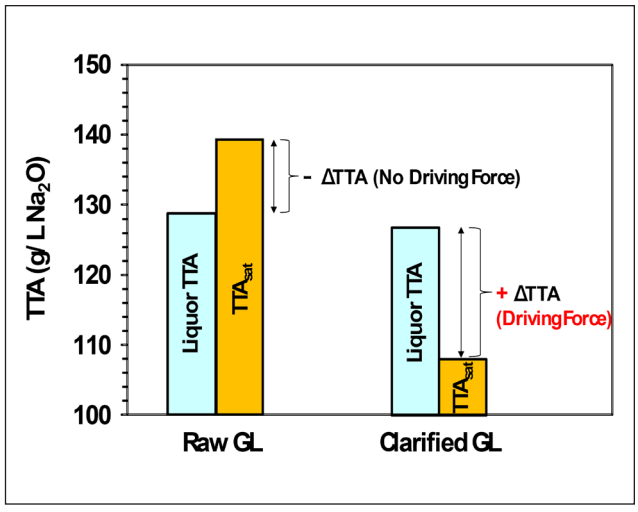
A kraft mill experienced severe scaling problems in its green liquor system in early 2011. Hard scale was found to build up at a location downstream of the pump in the pipeline that carried clarified green liquor. Chemical and thermal analyses of the scale sample showed that it consisted mainly of pirssonite [1].

The average temperature of the clarified green liquor at this mill was 81°C. The average operating values of TTA, sulfidity, and causticity were about 127 g/L as Na_2O, 28.7%, and 3.4%, respectively. Using Fig. 18, the TTA_{sat} under such conditions is estimated to be 108 g/L Na_2O, which is well below the average operating TTA (127 g/L Na_2O). The difference between $TTA_{operating}$ and TTA_{sat} in this case is 127 - 108 = 19 g/L Na_2O, implying that there was a large positive driving force for pirssonite to form in the clarified green liquor handling system.

During the same period, the mill did not have scaling problems in its raw green liquor handling systems, such as the dissolving tank, the storage tank, or the pipeline that carries raw green liquor. The average operating temperature, TTA, sulfidity, and causticity of the raw green liquor were 94°C, 129 g/L Na_2O, 26.6%, and 3.3% respectively. Under these conditions, TTA_{sat} as determined from Fig. 18 was about 140 g/L Na_2O, which is well above the average operating TTA of the raw green liquor (129 g/L Na_2O). The TTA difference in this case is 129 - 140 = -11 g/L Na_2O, meaning that there was no driving force for pirssonite to precipitate.

The above scaling experience can be explained more explicitly with the aid of **Fig. 20**, which shows the comparison between the positive driving force that led to pirssonite precipitation in the clarified green liquor system and the negative driving force that prohibited pirssonite precipitation in the raw green liquor handing system equipment.

Using the curves in Fig. 18 and the correlation in Fig. 19, it



20. Comparison of the driving forces for pirssonite precipitation in raw and clarified green liquor systems.

can be shown that under the average operating conditions of TTA 127 g/L Na_2O, 28.7% sulfidity, and 3.4% causticity, the liquor temperature at this mill must be controlled above 90°C to avoid pirssonite scale formation in the clarified green liquor handling system.

SUMMARY

The formation of pirssonite is commonly believed to be the cause of hard-scale buildup in green liquor handling systems in kraft pulp mills. The precipitation occurs when the concentration of Na_2CO_3 in the liquor exceeds the solubility of pirssonite. A laboratory study was conducted to systematically determine the causticizing conditions under which pirssonite precipitates. The results confirmed literature data demonstrating that the solubility of pirssonite increases with temperature but decreases with increased concentrations of sodium carbonate and other sodium salts present in the green liquor.

The study also extended the pirssonite solubility data to cover the low concentration range for NaOH and Na_2S. The information was used to generate reliable thermodynamic data for pirssonite formation and incorporate the data in OLI, a thermodynamic program that predicts phase stabilities of alkali salts in aqueous solutions. The OLI program was subsequently used to generate a series of pirssonite solubility curves in terms of TTA_{sat} that can be used as operational guidelines to prevent pirssonite precipitation and hard-scale formation in green liquor handing systems.

These TTA_{sat} curves were successfully used to explain why the pirssonite scaling problem was experienced in the clarified green liquor system but not in the raw green liquor handling system at a kraft pulp mill. **TJ**

ACKNOWLEDGMENTS

This work was conducted as part of the research program Increasing Energy and Chemical Recovery Efficiency in the Kraft Process, jointly supported by the Natural Sciences and Engineering Research Council of Canada (NSERC) and a consortium of the following companies: Andritz, Babcock & Wilcox, Boise, Carter Holt Harvey, Celulose Nipo-Brasileira, Clyde-Bergemann, DMI Peace River Pulp, Fibria, Kiln Flame Systems, International Paper, Irving Pulp & Paper, Metso Power, MeadWestvaco, StoraEnso Research, and Tembec.

ABOUT THE AUTHORS

We chose this topic to research because there was insufficient data on pirssonite solubility to allow us to predict whether pirssonite can form in the green liquor handling systems under a wide range of causticizing plant operating conditions. This research was complementary to the previous work by our and other research groups. The difference is that it focused on the solubility of pirssonite over a wide range of mill operating conditions.

The most difficult aspect of this work was to reconcile several small (but important) differences between our results and literature data. We addressed this by going through the data carefully, and by conducting experiments to obtain data under conditions where no data were previously available. The most interesting finding was that the solubility curves that we developed could be used to explain well the occurrence of the pirssonite deposition problem in a case study.

Mills may use this information as operational guidelines to prevent pirssonite precipitation and hard-scale formation in green liquor handling systems. The next step is to investigate in detail why hard scale in many cases contains only calcium carbonate, and whether its formation process is related to pirssonite formation.



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