

New challenges regarding nonprocess elements in the liquor and lime cycle

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ABSTRACT: Optimal performance of the green liquor plant, recausticizing plant, and lime cycle is vital for adequate white liquor availability and quality as well as for a mill's energy efficiency. Recently, various problems in the liquor and lime cycle have been frequently reported by our industrial partners, including poor performance of green liquor filters or sludge filters, decreased filterability of lime mud, increased lime kiln dead load, and poor-quality white liquor. Those problems are most likely caused by an accumulation of nonprocess elements in the liquor and lime cycle due to increased mill closure, increased use of biofuels, or both.

Data from the literature and earlier studies have been analyzed with regard to the occurrence and concentration of nonprocess elements in various process streams, including filtered green liquor, green liquor sludge, lime mud, and white liquor. The mineral forms in which nonprocess elements often precipitate were also studied, together with the common knowledge and rules of thumb used by mills for dealing with the problems. The literature data are compared with the newest analytical results from a sampling campaign involving several mills with varying process solutions with respect to nonprocess elements. The consequences and possible recommendations for the mills are presented.

Application: Kraft pulp mills can use this information to diagnose and possibly solve problems related to non-process elements in day-to-day mill operation.

Optimal performance of the green liquor plant and recausticizing plant is vital for the operation of a kraft mill. Any disturbance in the liquor and lime cycle, such as interrupted green liquor production, will immediately manifest itself in decreased white liquor availability and, consequently, a potential loss of production. If the lime cycle starts to perform poorly, the quality of white liquor may be seriously compromised; the mill's energy efficiency may also deteriorate due to the accumulation of dead load in the lime reburning stage.

During the past few years, a trend of more frequent problems in the liquor and lime cycle has been observed, both in the literature [1-3] and among our industrial partners. An increasing number of mills have reported deteriorated performance of green liquor filters or sludge filters-thickeners. Even when these problems are apparently solved locally, they tend to propagate to other process stages, such as the lime cycle. The results of nonprocess elements (NPE) accumulation in the lime cycle include decreased filterability of lime, increased tendency for ring formation, and increased dead load in the kiln product. In certain cases the problems could spread even further, affecting white liquor composition, causing scale formation in the digester or evaporation plant, or causing problems in oxygen delignification if oxidized white liquor is used as an alkali source. The most probable cause for these problems is an accumulation of NPE in the liquor and lime cycle. This accumulation is, in turn, believed to depend on increased mill closure (e.g., recirculation of

internal water streams and combustion of biosludge from external treatment in the recovery boiler), increased use of biofuels, or both.

Most of the industrial rules of thumb and practical approaches to solving NPE-related problems in the liquor and lime cycle have relied on theoretical and experimental studies from the early 1980s to the beginning of the 21st century [4-7]. A classic example is the work of Ulmgren [4] describing the removal of aluminum from green liquor as a function of the magnesium (Mg)/aluminum (Al) molar ratio, due to a formation of solid magnesium aluminum hydrotalcite, commonly written as $\text{Mg}_6\text{Al}_2(\text{CO}_3)(\text{OH})_{16} \cdot 4(\text{H}_2\text{O})$. These approaches were broadly validated and have performed very well in the conventional kraft process.

It is, however, frequently observed that these traditional approaches often fail in modern mills with increased NPE input (e.g., via various biofuels, increased recirculation of waste process streams, and decreased purge). The plausible explanation seems to be that the process conditions have moved outside the boundaries of the traditional chemical model. Several studies have been published [1,2] showing that the proven methods for dealing with NPE-related problems (e.g., adjusting Mg/Al ratio in the smelt dissolver) did not help; moreover, chemical compounds previously unreported in the studied process stages have been found. Consequently, there is a need for a new approach to predicting the effects of NPE accumulation in the liquor and lime cycle.

It is also important to focus on the interactions between

NONPROCESS ELEMENTS

the unit operations of the entire recovery cycle, so the encountered problems would be solved instead of simply moved around. Also, many earlier studies focused on the effects of one or two NPE (e.g., Al and Mg) at a time. It is now suspected that minerals that may form in the liquor and lime cycle are so complex that interactions of four to five NPE should be considered. An example of an attempt to broaden the understanding of the interactions of various NPE in the liquor and lime cycle is a work of Wannenmacher et al. [8], which described the solubility of complex aluminosilicates in green and white liquor.

The overall objective of our work is to improve the performance of the green liquor plant and recausticizing plant, as well as white liquor and lime mud quality, through increased understanding of the behavior of NPE in the liquor and lime cycle, taking into account the new conditions regarding increased NPE input. This paper covers the first part of the project, namely literature and field studies regarding NPE-related problems in the liquor and lime cycle of a modern kraft mill. The data from earlier studies have been analyzed with regard to the occurrence and concentration of NPE in various process streams: green liquor (filtered and unfiltered), sludge, white liquor, lime, lime mud, and lime kiln dust. The mineral forms in which NPE often precipitate were also studied, together with the common knowledge and rules of thumb used by mills for dealing with NPE-related problems. The literature data are compared with analytical results from a recent sampling campaign involving several mills with varying process solutions with respect to NPE. The consequences and recommendations for the mills are presented.

BACKGROUND

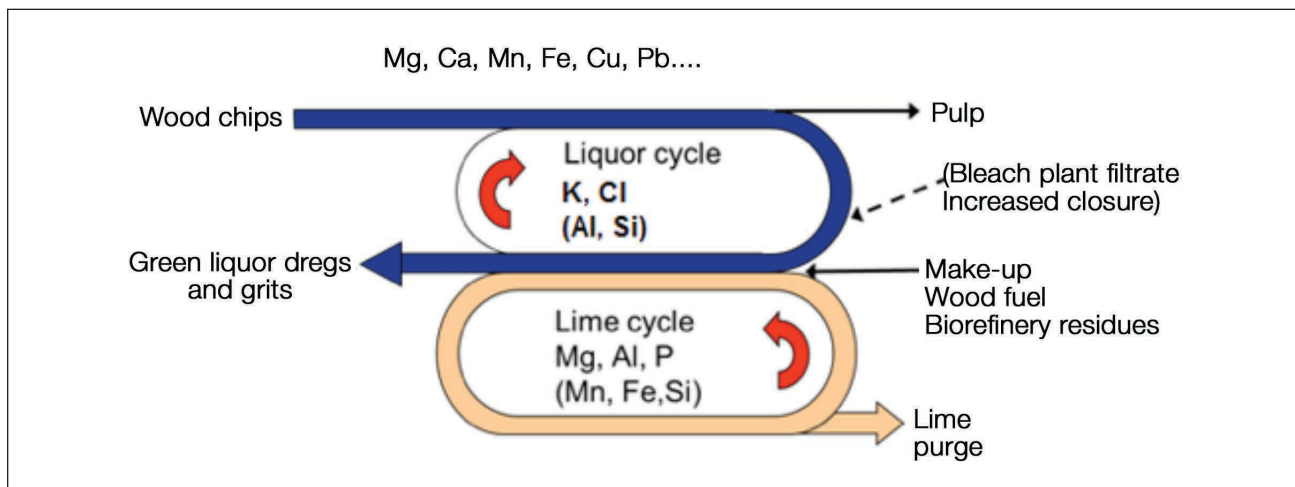
NPE in a pulp mill

The main source of most NPE in a kraft pulp mill is wood raw material. For potassium (K), phosphorus (P), or manganese (Mn), wood is often the only significant source. Other inputs may include process chemicals such as magnesium from magnesium sulfate (MgSO_4) added to oxygen bleaching and pos-

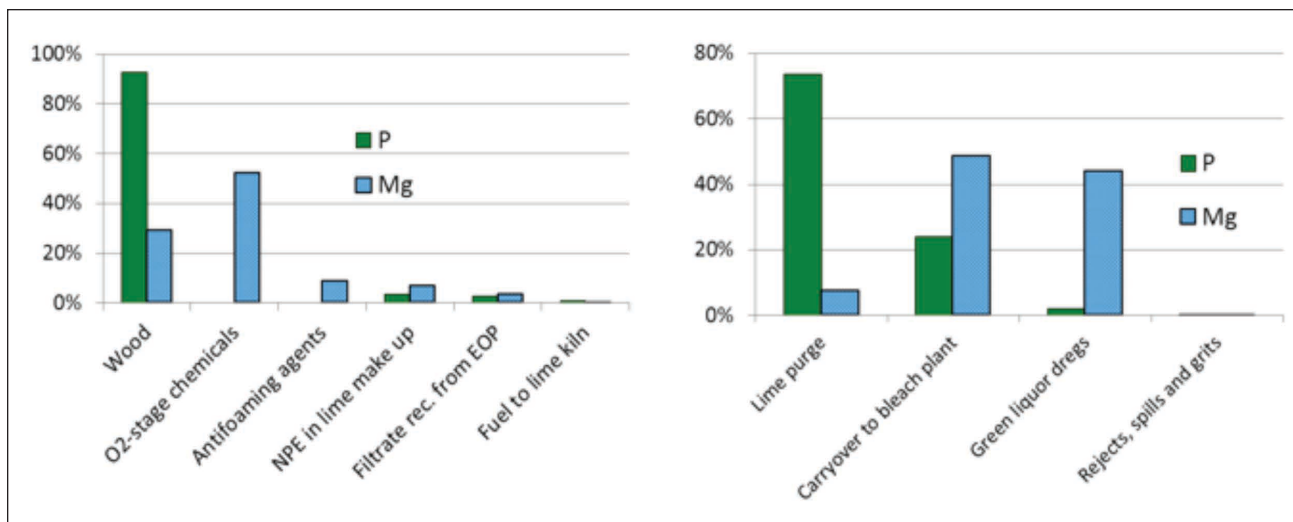
sibly other alkaline stages; silicon (Si) from talc used as a fiber line washing aid; biosludge from external treatment combusted in the recovery boiler (often containing Al); biofuels in the lime kiln such as Mn and Si from bark powder; fresh water; makeup chemicals such as Al and Si in low-quality lime makeup; and corrosion such as zinc (Zn) from anodic corrosion protection. The main output streams may vary depending on a particular NPE, but green liquor sludge is normally regarded as the most important purge for Mg, Mn, iron (Fe), Zn, Al, and often Si. Two other significant streams for NPE purging include mill effluents and the pulp product. Certain NPE are also removed with the lime mud purge (P), ESP dust from the recovery boiler (K and chloride [Cl]), or possibly with flue gases. **Figure 1** presents the general concept regarding the behavior of NPE in the liquor and lime cycle as it has been used as a rule of thumb during the last years.

Accumulation of NPE in the process may lead to a series of problems, the most well-known of which include scale formation on process equipment (e.g., calcium carbonate in cooking and manganese oxohydroxide in the fiber line); filtration problems (e.g., poor dewatering of green liquor sludge or lime mud caused by Mg); decomposition of oxygen-based bleaching chemicals by transition metals; increased energy consumption (e.g., due to P-based dead load in the lime kiln); and sticky dust problems in the recovery boiler caused by K and Cl. Proper management of NPE, by minimizing their input and optimizing their purge, is therefore vital for the overall process economy of the pulp mill.

Figure 2 presents mass balances for two NPE, Mg and P, in a reference pulp mill excluding the bleach plant. The mill was constructed as a model simulation based on representative sampling campaigns in a number of typical Nordic mills as well as literature data for best-available technology [9]. The mill returns its alkaline filtrate to brownstock washing and uses a wood biofuel in the lime kiln. As mentioned before, wood is one of the most important sources of NPE in a modern mill; while most Mg purges as green liquor dregs, P accumulates in lime mud and must be removed from the lime cycle.



1. General concept regarding accumulation and purge of nonprocess elements in liquor and lime cycle [6].



2. Mass balance for magnesium and phosphorus in a modern pulp mill. Left: input; right: output.

NPE in the liquor and lime cycle

In the following sections, we present the prevailing knowledge regarding the characteristic behavior of the most common NPE. The summary includes results from traditional literature sources, new research, and our project experience.

Magnesium

Magnesium enters the mill via MgSO_4 , talc, wood, biosludge, and lime makeup. Magnesium and Al should be purged from the liquor cycle via green liquor dregs as hydrotalcite. A common practice for kraft mills has been to keep the correct molar proportion between these two elements in green liquor. If the concentration of Al is too high, it will not be completely precipitated, but rather carried away to white liquor and lime. If the Mg content is too high, part of it will precipitate as magnesium hydroxide ($\text{Mg}(\text{OH})_2$), giving the dregs very poor dewatering properties. Magnesium does not seem to dissolve well in white liquor but tends rather to accumulate in the lime cycle and is present as $\text{Mg}(\text{OH})_2$ in the lime mud, decreasing its filterability. The dead-load component formed after calcination, magnesium oxide, does not causticize sodium carbonate.

Aluminum

The main sources of Al include wood, biosludge, dust (sand), and makeup chemicals. Aluminum should be purged from the liquor cycle together with Mg as hydrotalcite. Excess Al concentration in the filtered green liquor has traditionally been controlled by the addition of MgSO_4 .

It seems that the capacity of white liquor to dissolve Al is very high. If Al is added directly to the lime kiln, such as via biofuel, it will dissolve in white liquor. Theoretically, the maximum concentration in white liquor should be controlled by the amount of Mg available; however, some of our recent observations suggest that if the Al input is large, its concentration in white liquor may rise uncontrollably.

Silicon

The main sources of Si include wood, talc, biosludge, and dust (sand). Silicon has long been believed to have relatively high solubility in green liquor and therefore be poorly removed with green liquor sludge. However, newer data [1,2,8] suggest that coprecipitates of Si and Al with Mg or P are possible. We have also found Si precipitates in green liquor sludge, on green liquor filters, or both; these are probably unknown Mg-Al-Si compounds with exceptionally poor filtration properties. The removal of Si from the green liquor is, however, not as effective as removal of Mg, for example, and a large fraction can be carried over to the lime cycle.

The solubility of Si in white liquor is believed to be rather high compared with other NPE, approximately 12–15 mmol/L as dihydrogensilicate (approximately 340–420 mg Si/L) or more in certain cases. High concentration of Si in the white liquor is undesirable as it may lead to troublesome aluminosilicate scales in black liquor evaporation. The excess Si in white liquor has traditionally been assumed to precipitate as calcium hydroxysilicate, which is recalcinated to calcium silicate–calcium oxide solid, whose contribution to the lime cycle dead load is rather large. However, recent results [1,2] suggest that Si may form new dead-load compounds: kaolinite and gehlenite, as well as silicobermanite. The authors of this work have also identified particles containing Ca, Si, and P in lime kiln dust with the help of environmental scanning electron microscopy element mapping. Upon reacting with S^{2-} ion in green liquor, these compounds may give the lime mud an intense green color; they are also believed to be responsible for significant water retention in lime mud and filtration problems.

Phosphorus

The main input points for this NPE include wood, biosludge, possibly lime kiln fuel, or low-quality makeup. The solubility of P is believed to be higher in green liquor than in white liquor; the maximum concentration in white liquor is believed

NONPROCESS ELEMENTS

to be lower than approximately 0.5 mmol/L (15 mg/L) [10]. Some mills reported higher values, however, which suggest that the general guidelines should be verified. Only insignificant amounts of P are removed with green liquor sludge; instead, P tends to accumulate in the lime cycle. Traditionally, it has been believed that the compounds involved include calcium phosphate or calcium hydroxylapatite, possibly with some degree of ionic substitution. Recent research suggests a possible formation of other dead-load components in the lime cycle, possibly silicobernanite. In such case, the relative decrease of free calcium oxide due to the accumulation of P and Si would be somewhat lower than if these two NPE deactivated Ca alone. In some mills, P seems to be enriched in lime kiln dust; the degree of enrichment may possibly be correlated with the total concentration.

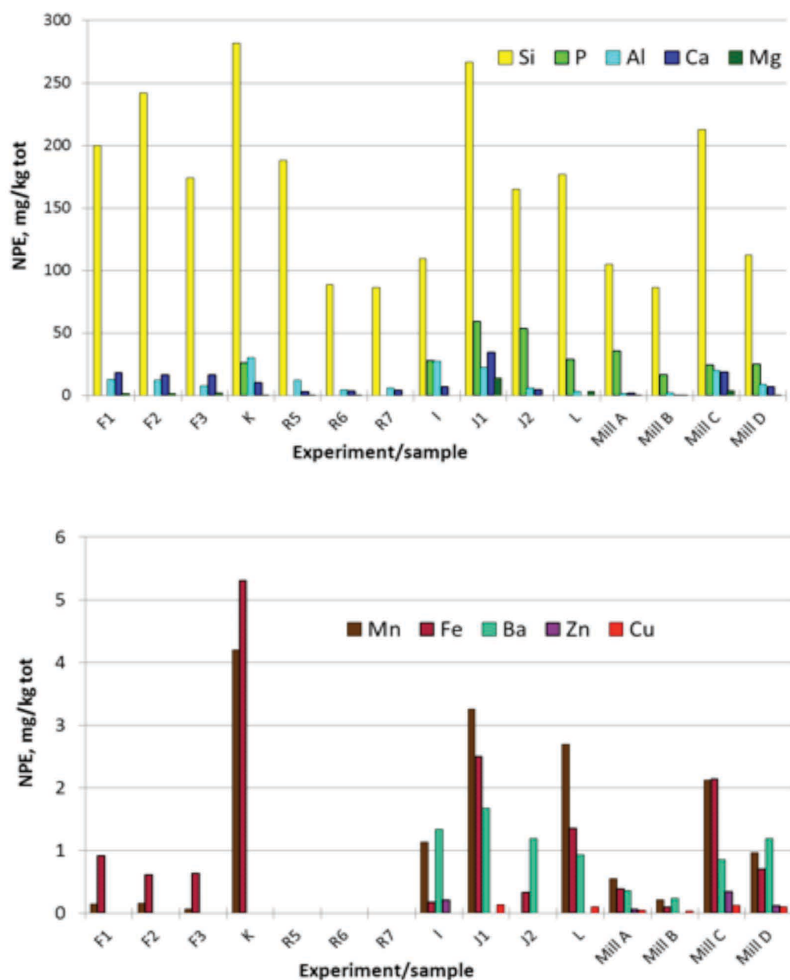
Manganese

The main source of Mn in a pulp mill is wood. Manganese is effectively removed from the liquor cycle via green liquor dregs

as solid manganese sulfide (MnS), manganese hydroxide, or both. If the dregs removal is poor or if Mn is introduced directly with the fuel to the lime cycle, it may accumulate there as MnS, which will be transformed to various, relatively inert Mn oxides during recalcination. A formation of complex minerals containing Mn, Mg, and Ca may also take place, but this hypothesis has not been unequivocally confirmed. The solubility of Mn in white liquor is believed to be low, approximately 0.4 mmol/L (approximately 22 mg/L) [10], but the mill experiences vary.

Iron

Iron is introduced to the pulp mill with wood and makeup chemicals and as a result of corrosion. Iron is effectively removed from the liquor cycle via green liquor dregs as ferrous sulfide (FeS). If Fe is introduced to the lime cycle (e.g., due to poor dregs removal, corrosion of the equipment in the liquor pathway, or directly with the fuel), it will accumulate there as FeS and be transformed to dicalcium ferrite during recalcination. The lime mud color changes to green, but the



3. Nonprocess elements (NPE) levels in clarified/filtered green liquor (F1-F3 [11]; K [2]; R5-R7 [7]; I-L, previous work; Mill A-Mill D, this work).



4. NPE levels in green liquor sludge (F1-F3, S not analyzed; S1-S5 and U1-U5 [11]; R1-R4 [7]; Mill D0, previous work; Mill A-Mill D, this work).

causticizing or filtration properties do not seem to be significantly affected. The accumulation of Fe in the lime is also believed to significantly increase the dust formation in the lime kiln. The solubility of Fe in white liquor seems to be low, approximately 0.1 mmol/L (approximately 6 mg/L) [10].

NPE LEVELS IN LIQUOR AND LIME CYCLE

Data choice

The sources of concentration data for this comparison include the work of Taylor and McGuffie [2] (K), Richardson et al. [7] (R1-R7), Empie et al. [11] (F1-F3, S1-S5, U1-U5), Innventia's data from earlier mill samplings for mill modeling (I-L), and data from this work (Mill A-Mill D).

Four mills have been chosen for the sampling campaigns during this project, taking into account varying prerequisites for NPE accumulation.

Mill A

Mill A is a softwood mill with relatively good capacity margins in the recovery boiler and the lime kiln. The mill practices partial recovery of bleach plant filtrate. Biosludge from external water treatment is combusted in the recovery boiler; green liquor sludge is separated by filtration, and a relatively clean biofuel (wood) is used in the lime kiln. No significant problems in the liquor and lime cycle were reported, besides Mn precipitates in evaporation.

Mill B

Mill B is a eucalyptus mill with rather low NPE input and degree of closure. Green liquor sludge is separated by filtration, and the lime kiln uses natural gas. The mill has not reported significant NPE-related problems during normal operation; however, when the lime kiln capacity was temporarily decreased and large amounts of reburned lime makeup were used, the mill experienced some problems with lime mud filtration.

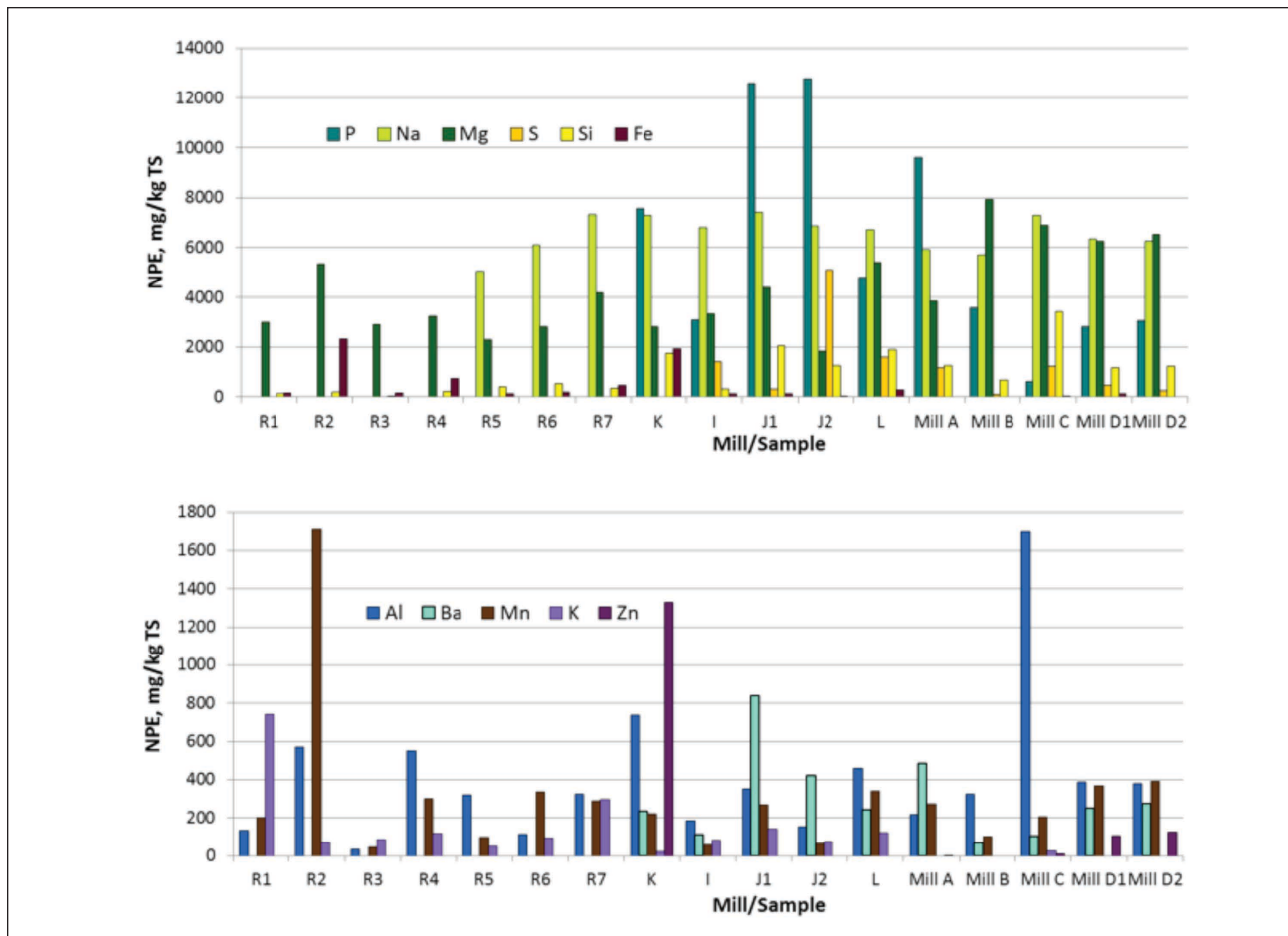
Mill C

Mill C has a softwood and hardwood line with a high degree of closure implying 90% recirculation of bleach plant filtrates; green liquor sludge is separated by clarification. The capacity of the lime kiln system is highly overrun, and large amounts of fresh lime makeup are used in causticizing. The makeup is of rather poor quality, and the mill has experienced some problems with aluminosilicate scaling in evaporation.

Mill D

Mill D runs softwood and hardwood campaigns with partial recirculation of bleach plant filtrate. Biosludge from the external treatment is combusted in the recovery boiler, and green liquor sludge is separated by filters. The lime kiln system operates slightly over its design capacity, and the fuel oil is partially substituted by bark biofuel. The mill has experi-

NONPROCESS ELEMENTS



5. NPE levels in lime mud (R1-R7 [7], phosphorus and barium not measured; K [2]; I-L, previous work; Mill A-Mill D, this work).

enced problems with poor green liquor sludge dewatering, scaling on sludge filters, some problems with lime mud dewatering, and ring formation.

From the characteristics above, we expected to find the highest levels of NPE in the liquor and lime cycle of mill C, followed by mills D, A, and finally B.

NPE in the liquor and lime cycle

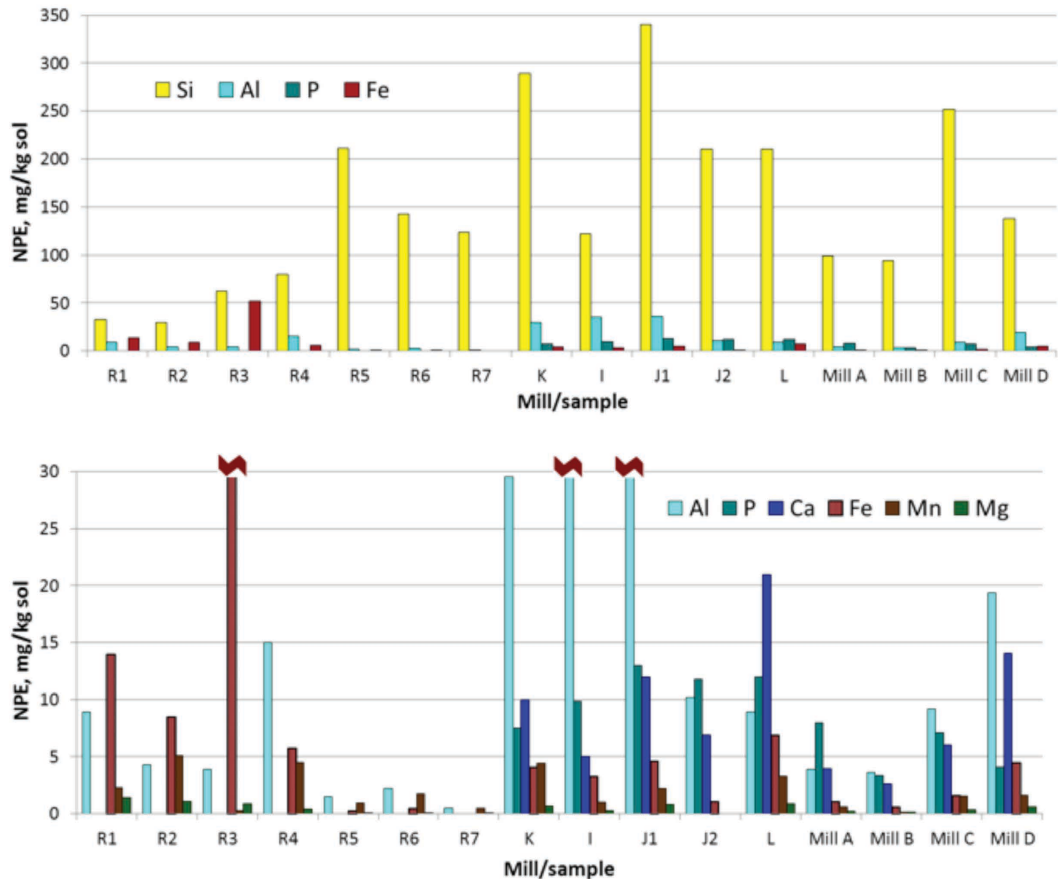
Figure 3 presents NPE levels in clarified-filtered green liquor. Silicon is the most abundant NPE besides K and Cl (not shown), and its levels can vary significantly. Literature mill K [2] and mill C separate their sludge by clarification rather than filtration, and the concentrations of Fe and Mn are rather high, as expected.

Figure 4 shows NPE levels in green liquor sludge; several data points can be correlated with those from Fig. 3. The concentrations of Mg and Al can possibly be correlated, while the levels of Si vary significantly. Measurements of green liquor sludge composition often carry large error, as it is difficult to take a representative sample. Moreover, the accuracy of the analytical methods for some NPE (especially Si) may differ from early data. Taking the previous into consideration, it is generally believed that the levels of NPE in green liquor sludge in relation to those of other streams, especially green

liquor, may often give valuable information about the overall NPE situation in the mill. However, if observed alone, the measurements should be treated with limited confidence.

The concentrations of NPE in lime mud presented in **Fig. 5** seem to vary between mills and over time. Traditionally, P has been regarded as the most important NPE of the lime cycle, but it seems that some modern mills may have larger problems with Mg accumulation. High levels of P in mill A may possibly be attributed to biofuel in the lime kiln, while high content of Al and Si in mill C clearly reflect the quality of lime makeup. Accumulation of Zn and Fe in literature mill K [2] may be attributed to poor green liquor sludge separation, while high levels of Mn in mill D come from bark fuel.

Finally, **Fig. 6** presents NPE levels in white liquor. As in green liquor, Si is the most abundant NPE besides K and Cl. Relatively high levels of Si in mill C are not surprising taking into account the lime mud results. Analogically, a high concentration of Al in mill C was also expected; it was instead found in mill D. It can be speculated that relatively high levels of Al, Mn, and Fe in mill D compared to other mills from this work may be the result of biofuel use in the lime kiln, but generally the solubility behavior of Al is not well established. Ulmgren [4] suggested that Mg-Al-hydrotalcite could be precip-



6. NPE levels in white liquor (R1-R7 [7], phosphorus and barium not measured; K [2]; I-L, previous work; Mill A-Mill D, this work).

itated in lime mud in an analogic fashion to green liquor sludge; consequently, high levels of Mg in lime mud would prevent excess dissolution of Al in white liquor. Therefore, it is not understood why mill D has much higher white liquor Al concentration than mill C.

CONCLUSIONS AND RECOMMENDATIONS

NPE separation in green liquor sludge

Green liquor dregs separation should be treated as the most important purge point for many troublesome NPE; it is therefore important that this process stage works properly. The green liquor filter is recommended as a first choice; if, however, a clarifier is the only alternative, the mill should make sure that it does not operate much above its design capacity so that minimal dregs carryover is experienced. Manganese, Fe, and Zn are generally removed very well as long as the dregs separation itself is efficient. Calcium and Mg are also removed quite well, while experiences regarding Al and Si vary among mills. The removal of P is very poor (possibly some small amounts are precipitated with Ca), and virtually none of K and Cl is removed.

Problems with poor filtration and dewatering properties of dregs have traditionally been ascribed to high Mg content. Magnesium should preferably be precipitated together with

Al as hydrotalcite; if not enough Al is present, Mg will form hydroxide (possibly colloidal) with high water retention. However, even as hydroxide, Mg will still be removed from green liquor rather efficiently. Therefore, the mills experiencing a dregs dewatering problem have adjusted the Mg/Al molar proportion, and this practice is still recommended as a first measure to try. However, poor dregs filtration and dewatering may also depend on the formation of complex aluminosilicates, which may form scales on the dregs filter; the dregs separation process will then be disturbed despite apparently favorable Mg/Al ratio. In this case, lowering the Si input as well as other means of improving dregs separation (e.g., increasing the filter volume and improving flow properties or increasing the residence time in the raw green liquor tank) are recommended.

NPE in lime cycle

Problems with lime mud filtration

As in the case of green liquor sludge, poor lime mud filtration properties can depend on accumulation of Mg and the formation of $Mg(OH)_2$ in causticizing. However, this situation is estimated to happen rather seldom: first, separation of Mg with green liquor sludge is rather good and significant

NONPROCESS ELEMENTS

amounts of this NPE should not be present in the lime cycle. Second, even in the lime cycle, Mg can be precipitated as hydrotalcite with good dewatering properties if a sufficient amount of Al is present. Nowadays, a considerably more common reason for poor filtration properties of lime mud seems to be the accumulation of complex Al-Si compounds. Many of them, besides high water retention, give lime mud (but not reburned lime) a characteristic green color; some results suggest that they can even contribute to ring formation. If such symptoms are observed, the recommendation is for the mill to identify and, if possible, decrease Al and Si input to the lime cycle. The possible measures can include decreasing the Al and Si input with biosludge combusted in the recovery boiler or with biofuels to the lime kiln, finding ways to improve Al-Si separation with green liquor sludge, or switching to better quality makeup.

Lime makeup

Surprisingly, low-quality makeup, especially if rich in Si, has recently been identified as a source of problems in the lime cycle for many mills. An extreme case includes a situation in which the lime cycle capacity is temporarily or permanently undersized for the adequate white liquor production and the mill is forced to supplement the missing lime flow with cheap, easily available makeup of reburned lime. The problems arising due to NPE accumulation may be so severe that investing in increasing the lime cycle capacity becomes desirable. Thus, the recommendation for a pulp mill

experiencing problems in the lime cycle is to start with examining the composition of its makeup and, if a high NPE content is found, calculating the total costs of switching to a cleaner alternative.

Biofuels in the lime kiln

Biofuels can, in certain circumstances, constitute a significant source of NPE to the lime cycle. When a decision regarding switching to a biofuel is made, the composition of the biofuel should be analyzed and the potential effects of the extra NPE input carefully investigated, preferably with the help of process simulation. A new fuel may significantly change the combustion conditions in the lime kiln, such as the flame length and temperature profile, which may potentially decrease or aggravate the NPE-related problems.

Purging lime kiln ESP dust

Certain NPE, especially P and some heavy metals (e.g., cadmium and lead), sometimes tend to become enriched in lime kiln dust relative to lime and lime mud. Earlier, P and possibly Si were believed to do so consistently; however, the newest results suggest that this tendency may vary between mills and depend on the total level of NPE in the lime cycle and possibly the details of lime kiln operation. We recommend that the mill analyze its lime kiln dust several times and if it becomes certain that problematic NPE is enriched there, then dust can be purged instead of lime mud. This will allow removal of problematic NPE with lower calcium losses.

ABOUT THE AUTHORS

The problems connected to NPE accumulation in the liquor and lime cycle were brought to our interest by our industrial partners. Our purpose was to summarize, verify, and update the previous knowledge in this field, including the common rules of thumb used by mill operators. We found that many earlier results still held, while some had to be revised due to changed conditions in modern mills, such as adjustment of Mg/Al ratio for better separation of green liquor sludge.

We found large variation in the literature data regarding NPE concentrations, which we addressed by carefully choosing data for comparison and trying to explain the differences. Another challenge was obtaining repeatable, representative data during the sampling campaign, which we addressed by carefully designing the sampling procedure.

The problems related to NPE in the kraft process are very complex and deserve a holistic approach, as even small changes in one process stage may have large repercussions in other parts of the mill, such as the effects of biofuels in the lime kiln on white liquor quality. We also found that the influence of silicon is often larger than commonly acknowledged.

This work may benefit mills generally, by putting a



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Ahlroth

greater attention on the NPE aspects of their recovery cycle, and more specifically, by applying some of the recommendations we present.

The findings of this study should further be verified against more, and more recent, data from modern kraft pulp mills. A logical next step would be to refine the existing methods for dealing with NPE-related problems and finding new ones.

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Solubility of NPE in white liquor

According to the traditional assumptions, solubility of most problematic NPE in white liquor should be limited and any excess should easily be precipitated in and separated together with lime mud. However, high concentrations of certain NPE, even if still below the solubility limits, may contribute to serious problems in other process stages. A classic example includes the formation of hard, extremely difficult to remove aluminosilicate scales in evaporation if the concentration of Al and Si in white liquor is high. When facing such a problem, the mills should control whether the lime makeup quality is adequate and if separation of Al and Si in green liquor sludge can be improved.

Another interesting and potentially disturbing phenomenon is transfer of NPE from biofuel in the lime kiln to white liquor via lime. As few mills consistently use NPE-rich biofuels, very little data regarding such phenomenon are available. However, there are some indications that the concentration of Si and Al and transition metals Mn and Fe in white liquor may be increased this way. If such liquor is oxidized and used as an alkali source in delignification and bleaching, the transition metals may contribute toward decomposition of bleaching chemicals and the pulp quality may be compromised. **TJ**

ACKNOWLEDGEMENTS

The authors acknowledge the financial support by the mill members of Cluster Chemical and Energy Recovery 2012-2014 and Innventia AB, and give special thanks to the mills that contributed to the sampling campaign.

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