

MANAGEMENT OF NON-PROCESS ELEMENTS IN EUCALYPTUS KRAFT PULP MILLS

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SUMMARY

Accumulation of non-process elements (NPEs) in Kraft pulp mill process streams is becoming increasingly important as mills move toward a higher degree of system closure. Most of the NPEs are introduced into the pulp mill with the wood chips. Depending on the wood raw material, degree of recycling of filtrate streams, and the process technology applied, NPEs may cause severe production related problems in the pulp mill. The most important NPEs are calcium (Ca), potassium (K), chlorine (Cl), manganese (Mn), aluminum (Al), silicon (Si), phosphorus (P), and iron (Fe). Eucalyptus pulp mills are known to have such high levels of NPEs that special measures have to be applied in order to gain a high availability of the processes.

If the level of NPEs in process streams is too high, troublesome scales may appear in several places. A high NPE load has also a negative impact on the limekiln energy consumption. High levels of K and Cl in black liquor cause fouling and corrosion and limit the production of steam in the recovery boiler.

There exist some natural kidneys for NPEs in the recovery cycle. The main routes out from the process, excluding the effluent streams, are green liquor dregs, grits, lime mud, and recovery boiler precipitator (ESP) ash. From these flows NPEs can be safely taken out.

KEYWORDS

Eucalyptus, kraft pulp mill, non-process elements, scaling, corrosion

INTRODUCTION

Non-process elements (NPEs) are present in all kraft pulping processes. NPEs are defined as those elements that are not essential to the chemical process. Although, potassium (P) and chlorine (Cl) take actively part in the process, they generally are also considered as NPEs. By reducing the effluent flows from kraft pulping and closing the chemical cycles have both led to the increase in the NPE concentrations. The minimization of the fresh water usage increases the levels of NPEs in the cycles, too.

Problems associated with the build-up of NPEs in the recovery area, include scaling in the black liquor evaporators, fouling and the corrosion of heat transfer surfaces in the recovery boiler, together with various other operating problems during the preparation of pulping liquors (Allison et al., 2000). Accumulation of NPEs in the pulping process can also induce an increased use of bleaching chemicals during later stages, and problems with filtration.

Several different NPEs exist in the pulping process. However, only few are present at significant levels. The most important NPEs are calcium (Ca), potassium, chlorine, manganese (Mn), aluminum (Al), silicon (Si), phosphorus (P), and iron (Fe).

The effects of NPEs on Kraft mill operation have been extensively studied (Keitaanniemi and Virkola, 1982; Frederick, 1984; Erickson and Holman, 1986; Richardson et al., 1998; Jemaa et al., 1999; Taylor and Bossons, 2006). These include increased lime kiln fuel use, reduced filtration efficiency, reduced lime mud settling and increased scaling of heat exchangers. Potassium and chlorine influence the thermal properties of fireside deposits in the recovery boiler. Therefore, it is important to control the levels of NPEs in order to gain a high accessibility of the processes.

Eucalyptus wood is known to have relatively high contents of chloride, potassium, and calcium. Although the published figures indicate that the concentrations of the NPEs in the eucalyptus chips may vary a lot, it appears that their chloride contents are typically somewhat higher than usually found in softwood pulping raw materials.

This inevitably results in moderate or high chloride contents in black liquor. Therefore, several eucalyptus kraft pulp mills have adopted a separate removal process for chloride and potassium. However, albeit the high levels of Ca in eucalyptus, no specific system has been exploited for calcium removal.

The objective of this paper is to evaluate the sources and routes of NPEs and their effects on the pulping process in eucalyptus-based kraft pulp mills. This helps to better understand and control the effects of non-process elements.

EFFECTS OF NON-PROCESS ELEMENTS ON THE PULPING PROCESS

NPEs can be divided into two different groups according to their behavior in the chemical cycles. The first group (Ca, Mg, Mn, Fe) contains those NPEs that form insoluble or sparingly soluble compounds in green and white liquor and can be almost completely removed from the process by natural purges such as green liquor dregs, grits, and lime mud. The second group, including Cl, K, Al, Si, and Ba are soluble in conditions in the recovery cycle and will therefore tend to accumulate in the pulping process.

The effects of NPEs are widely scattered throughout the entire pulping process. However, most of the harmful effects can be seen in the recovery cycle. Table I summarizes the problems that NPEs may cause in the pulping process.

Table I: The effects of different NPEs on the pulping process.

NPE	Digester	Bleaching	Evaporators	Recovery boiler	Causticizing	Lime kiln
K, Cl				Plugging and corrosion		
Ca	Scaling of heating surface and screens	Scaling of wash equipment	Scaling of heating surfaces			
Mg					Poor lime mud filterability, plugging of filters	High dead load in lime cycle
Si			Sodium aluminum silicate scaling		Poor lime mud filterability, plugging of filters	
Al			Sodium aluminum silicate scaling			
Mn, Fe		Peroxide decomposition				High dead load in lime cycle
P						High dead load in lime cycle
Ba		Scaling of wash equipment				

When an equilibrium level, where the intake and purges of the NPEs are in balance in the cycles, is reached no specific problems from NPEs are likely to appear. The concentration level, at which the NPE related problems may appear, varies from mill to mill. The maximum allowable levels of NPEs in different parts of the process depend on the input level and the degree of closure of the chemical cycles.

INTAKE AND KIDNEYS OF NON-PROCESS ELEMENTS IN THE PULPING PROCESS

NPEs enter the pulp mill with the wood raw material, make-up chemicals, and mill process water (Sharp, 1996). The NPEs that enter the kraft mill with the wood are largely extracted from the fiber during cooking and bleaching stages. The fate of these extracted metal ions depends on a combination of factors, including concentration of organic and inorganic compounds in the process streams and the chemistry of these NPEs.

Table II shows the NPE content of different eucalyptus species. As can be seen, considerable variations exist between different eucalyptus species. The most remarkable variations can be found in the Ca content. Calcium

content in these particular species varied from 510 mg/kg up to 2700 mg/kg in dry wood. Potassium and chlorine content varies between 410-900 mg/kg and 240-460 mg/kg in dry wood, respectively.

Table II: NPE values of different eucalyptus species (mg/kgds).

	A	B	C	D	E	F
Na	82	200	160	240	170	110
K	900	500	540	640	410	610
Al	nd	nd	nd	nd	nd	nd
Ca	850	510	1900	1000	1000	2700
Fe	30	48	32	18	22	39
Mg	270	110	310	210	76	790
Mn	38	56	71	53	27	68
Cl	460	430	300	340	240	440
Si	15	8.7	7.9	8.2	5.5	4.7
P	88	270	45	51	34	87

nd = below the detection limit.

Many of the NPEs have natural purges in the system. The amount of NPEs can effectively be reduced to acceptable levels by using these purges. Some alkali-insoluble elements (e.g. green liquor dregs) in the liquor cycle can be purged with existing purge streams. However, the removal of alkali-soluble elements, such as potassium and chlorine will require other purge methods (Frederick and Adams, 1999).

In the following, a short description is given about the routes in and out of the most common NPEs appearing in the chemical cycles.

Aluminum (Al)

Aluminum enters with the chips, make-up lime, process water, and biosludge, if it is burned in the recovery boiler. Al is found both in liquor and lime cycles. The main route out from the liquor cycle is green liquor dregs. Some aluminum is also purged with pulp.

The solubility of aluminum is affected by temperature, pH, and the presence of other cations. The chemistry of Al in solutions is very complex due to the many forms it can exist in. However, one of the major factors that affect Al solubility is the Mg/Al- ratio in green liquor. Ulmgren (1987) has shown that the formation of magnesium aluminum compound called hydrotalcite ($Mg_6Al_2CO_3(OH)_{16} \cdot 4H_2O$), is crucial for Al precipitation and removal.

As the aluminum level increases, it will finally reach a saturation limit and Al precipitates in the recovery cycle as aluminosilicate scales on the evaporator heat transfer surfaces.

Barium (Ba)

Most of the barium encountered in the mill enters with the wood. The majority of the barium present in the pulping process exits the digester as barium carbonate ($BaCO_3$) and much of this precipitate should be trapped in the fibers. Barium carbonate dissolves in the bleach plant when the pH drops below 7. If barium and sulfate concentrations are high, barium begins to precipitate as barium sulfate ($BaSO_4$).

Barium is difficult to control because a mill cannot avoid this carbonate-to-sulfate transition using common bleaching technology. Improved brown stock washing helps by removing sulfur compounds that will otherwise oxidize to sulfate in the D_0 stage. The mill can also reduce sulfate by controlling the use of sulfuric acid in the bleach plant. The D_0 pH can be increased if the mill does not have problems with calcium oxalate (CaC_2O_4).

Calcium (Ca)

The main sources of Ca are wood and make-up lime. Calcium is a process element in the lime cycle and if good clarification and filtration occurs, calcium will be confined to the lime cycle. The content of calcium in the eucalyptus chips is apparently in the same level than in other typical wood pulping raw materials, but its behavior in pulping and recovery may differ because of different calcium-binding organic black liquor compounds.

Calcium is soluble in acidic conditions and will dissolve in acidic C, D, Z, Q, and A stages. The main problem in the recovery cycle with calcium is the scaling in the evaporation plant. Heating black liquor to high temperatures (so-called deactivation process) makes Ca insoluble and may reduce scaling in the evaporators significantly.

Chlorine (Cl)

Chlorine enters the system with wood, make-up chemicals, bleaching chemicals, process water, biosludge, and spent acid. Since chlorine is totally soluble throughout the pulping process, the biggest purging routes out are different effluent streams. However, a strong enrichment of chlorine in the recovery boiler precipitator ash will rapidly raise the levels in the liquor cycle, if additional purges are not used.

The effects of high chlorine contents in the liquor cycle are first seen in the recovery boiler. Chlorine changes the melting properties of the ash by lowering the first melting temperature. Therefore, fouling tendency of recovery boiler heat transfer surfaces is increased. In addition to this, chlorine has a strong impact on superheater corrosion (Salmenoja, 2000).

Eucalyptus is known to have higher chloride content than Scandinavian softwood or hardwood. Therefore, eucalyptus kraft pulp mills have naturally higher chloride levels in the liquor cycles and almost invariably all the mills are also equipped with a separate precipitator ash treatment system for chloride purging.

Iron (Fe)

The main inputs of iron are wood, make-up lime, process water, and from steel as the result of corrosion of the process equipment. Iron is insoluble in alkaline conditions and can be removed by green liquor dregs and grits.

The solubility of iron is low in alkaline conditions and therefore iron can be purged with green liquor dregs and grits. Bleaching effluents are also a route out from the mill. Iron seems to be quite harmful in the processes, but it may promote ring formation in lime kiln and cause increased chemical consumption in the bleaching state.

Magnesium (Mg)

Magnesium enters the mill mainly in wood, make-up lime and as magnesium sulfate ($MgSO_4$) into the oxygen delignification stage. The solubility of Mg in the liquor cycle is quite low and magnesium tends to separate almost entirely with the green liquor dregs, grits, and lime mud. However, this necessitates efficient green liquor clarification or filtration.

Magnesium causes difficulties especially in filtering due to magnesium hydroxide ($Mg(OH)_2$) formation. Magnesium hydroxide is gelatinous and easily plugs up the filters. Magnesium hydroxide has also poor settling properties in green liquor clarification.

Manganese (Mn)

The main sources of manganese into the system are wood and process water. Manganese is detrimental to the hydrogen peroxide bleaching stage, catalyzing the decomposition of peroxide. Manganese can be removed by acidic washing and chelation (Wohlgemuth et al., 2003). Manganese does not pose any significant problems in the recovery area. A high level of carryover from green liquor clarification and filtering may color the lime, but it is usually only an esthetic nuisance.

Phosphorous (P)

Phosphorous enters the mill in wood and biosludge. An increased content of P considerably increases the dead load of the limekiln and thus have a negative impact on the energy consumption. It also serves as a nutrient load in effluents.

Phosphorous can be purged out with the lime. Phosphorous is enriched in the precipitator ash from lime kiln and can be purged out with the ash.

Potassium (K)

The main source of potassium is wood. Potassium acts like chloride in the recovery cycle and are also very soluble in the entire process. Therefore, potassium can not be purged by the chemistry, since all potassium salts are soluble.

Potassium is also enriched in the precipitator ash, although slightly less than chlorine, and can be purged with the ash. Dedicated ash treatment systems remove also potassium along with chloride. Potassium changes the melting behavior of the ash by further lowering the first melting temperature. However, potassium is not typically causing fouling problems in the recovery boiler, but is strong contributor in superheater corrosion. A high potassium level together with high chlorine content in black liquor is detrimental to the recovery boiler superheaters.

Silicon (Si)

The main sources of silicon are wood, process water, make-up lime, and silicon-based defoamers. Typically, silicon is distributed throughout the liquor and lime cycles, and is found at all possible purge places. Silicon is problematic, since there are no specific points where it can be removed easily. However, sometimes Si is accumulated in the liquor and lime cycles and can be removed there. Silicon can cause scaling in the evaporator train in the form of aluminosilicates. An increased content of Si considerably increases the dead load of the limekiln and thus have a negative impact on the energy consumption.

The solubility of silicon is affected by other cations. Magnusson et al. (1979) has shown that the solubility of Si decreases with calcium. In the liquor cycle silicon is also coprecipitated with aluminum forming sparingly soluble aluminosilicates. Addition of magnesium can also coprecipitate silicon (Vehmaan-Kreula, 2000).

ENRICHMENT OF NON-PROCESS ELEMENTS IN THE PULPING PROCESS

One of the key elements in successful NPE management in a kraft pulp mill is well functioning natural purges in the process. NPEs may have significant effects on the availability of the pulping process if their levels increase on a too high level. The maximum allowable level depends on the particular NPE and the process.

Water soluble NPEs are purged with effluent streams. Other natural NPE routes out include green liquor dregs, grits, and lime mud. Green liquor dregs act as a purge only if green liquor clarification or filtration is working properly. Especially, aluminum level in the recovery cycle is greatly dependent on green liquor clarification and filtration, since green liquor dregs seem to be the only route out from the process. If this route is blocked, aluminum levels will rapidly rise up to unacceptable levels (Taylor and Bossons, 2006). A good way of checking the green liquor clarification and filtration quality is the lime mud or burnt lime color. Lime mud and burnt lime are easily discolored by NPE carryovers.

The only way to control K and Cl levels is to purge them with the ESP ash. Water soluble K and Cl can also be purged via effluents, but if the degree of closure of the chemical cycles in the mill is high, they are rapidly enriched in the process. Dissolving ESP ash is an efficient way to purge Cl and K, but results in considerable sodium and sulfur losses. To minimize chemical losses, new processes have been developed to selectively remove Cl and K (Tran and Earl, 2004; Gonçalves et al., 2008).

Enrichment of Sparingly Soluble NPEs

The chemistry of NPEs is the same for all kraft pulp mills and dictates how they behave in the process. The NPE level in a mill is dependent on its process configurations and operation principles. A critical level where specific NPE may cause process upsets is also mill specific, and is therefore difficult to preset. However, previous experiences may help to avoid the most cumbersome problems.

Figures 1-5 show the levels of sparingly soluble NPEs Al, Si, P, Mg, and Mn in lime mud of four kraft pulp mills. According to the Figure 1, aluminum level is usually below 500 mg/kgds, except in Mill B, where the level is around three times higher. This is mainly due to poor settling of Al in the green liquor clarifier, which was encountered during the sampling period. This type of problem is typically related to hydrotalcite formation.

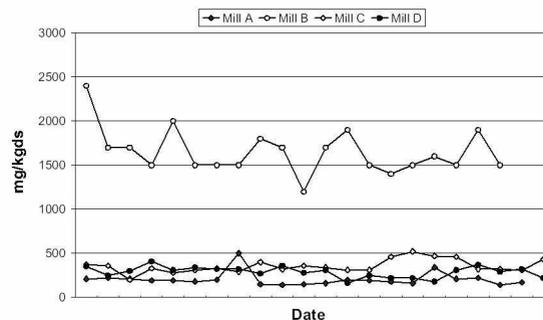


Figure 1: Aluminum content of lime mud in four kraft pulp mills.

Lime mud Si content varies slightly more, but seems to be around 1000 mg/kgds, on the average. Mill C seems to have somewhat higher content of Si in the lime mud. The reason for a higher Si content in lime mud in Mill C is somewhat ambiguous.

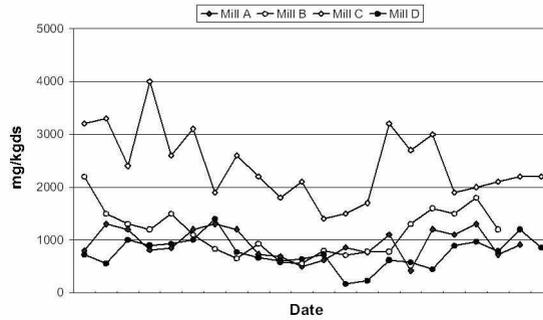


Figure 2: Silicon content of lime mud in four kraft pulp mills.

The variation of phosphorous in the mills is larger than with the other NPEs. The level in lime mud seems to be between 2500 and 7500 mg/kgds. However, no special problems due to P have been encountered at the mills.

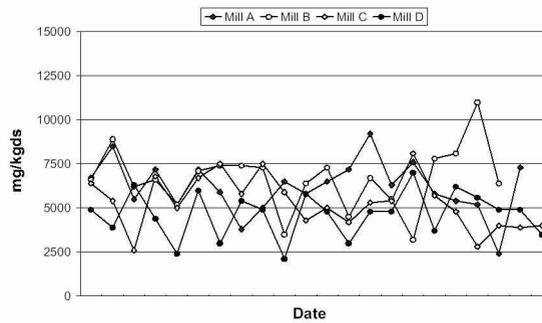


Figure 3: Phosphorous content of lime mud in four kraft pulp mills.

The variation of Mg seems to be on the same level as with P. The level of Mg in lime mud ranges from 2500 up to 7500 mg/kgds. Albeit Mill B has a high level of Al in lime mud, magnesium level is not higher than in the other mills.

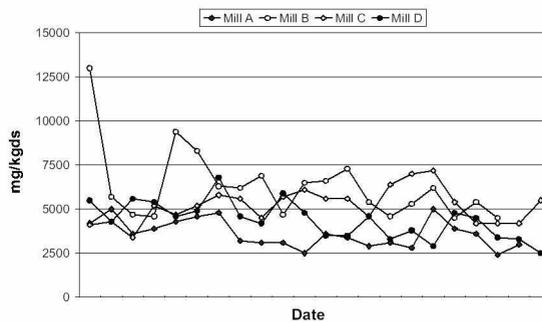


Figure 4: Magnesium content of lime mud in four kraft pulp mills.

Manganese levels in lime mud are usually below 500 mg/kgds. Increased manganese level in white liquor can discolor lime mud and burnt lime. In Mill C, which has a slightly higher Mn content in lime mud, the burnt lime color differs from the other mills.

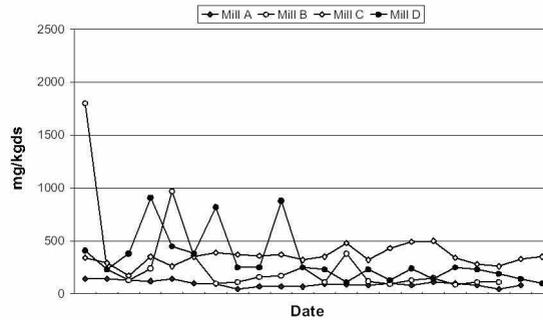


Figure 5: Manganese content of lime mud in four kraft pulp mills.

Continuous follow-up of the NPE trends in different chemical cycles helps to effectively diminish the number of unscheduled shutdowns. Changes in process conditions can be immediately seen in NPE trends and preventive measures can be started in time.

Enrichment of Chlorine and Potassium

Chlorine and potassium are both enriched in the recovery boiler ESP dust and from which it can be effectively removed. Typical enrichment factors for K and Cl in kraft pulp mills are typically around 1.5 and 2.5, respectively. High levels of K and Cl in the liquor cycle will, in the long run, cause fouling in the superheater area and may even totally plug the flue gas passages. Potassium and chlorine are also the biggest contributors to superheater corrosion (Salmenoja, 2000).

Potassium and chlorine affect the melting behavior of the ash deposits. The melting behavior will dictate the fouling and corrosion propensity of the deposits. A high Cl content will increase the amount of melt in the deposits, and a high K content will lower the FMT of the deposits. The FMT is the temperature at which the first liquid (molten) phase appears when the sample is heated. A low FMT generally increases the probability for superheater corrosion and a high Cl content in the ash lowers the sticky temperature and increases the fouling propensity of the ash. A more detailed description of deposit melting behavior is given by Tran (1997).

Typical level of potassium in the ESP ash in Scandinavian mills is shown in Figure 6. The enrichment of potassium into the ESP ash is mainly dependent on lower furnace temperature, which in turn is a function of dry solids content and load. Average K content in the ESP ash is around 50 g/kgds which corresponds to 5% in weight. In Mill B the potassium content in ESP ash is only 2.5%, on the average. Potassium content in the ESP ash is also on the same level in eucalyptus kraft pulp mills.

Chlorine content in the ESP ash is found in Figure 7. Chlorine enrichment into the ESP is mainly dependent on the lower furnace temperature and the sulfur dioxide (SO₂) content in flue gases. If the SO₂ level in flue gases is detectable then the enrichment decreases, since SO₂ drives chlorine in to the gas phase by sulfating solid sodium chloride (NaCl) to sodium sulfate (Na₂SO₄).

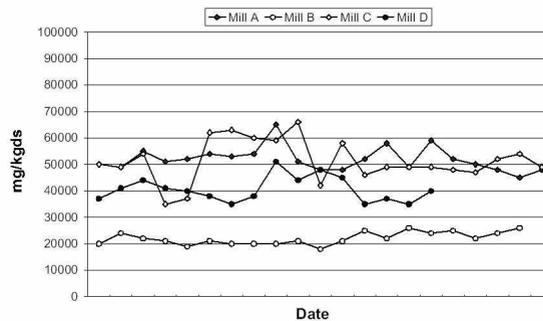


Figure 6: Potassium content in the ESP ash in four kraft pulp mills.

According to Figure 7, average Cl content seems to be ca. 5 g/kgds or 0.5% in the ESP ash. Chlorine value also seems to vary significantly due to the changes in the pulping process and in the lower furnace of the recovery boiler. Eucalyptus mills seem to have considerably higher Cl content in the ESP ash.

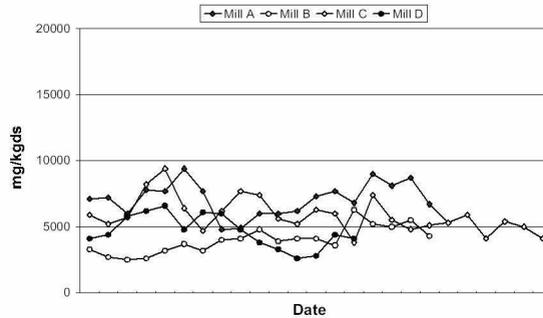


Figure 7: Chlorine content in the ESP ash in four kraft pulp mills.

MANAGEMENT OF NON-PROCESS ELEMENTS IN EUCALYPTUS KRAFT PULP MILLS

Due to the rapid enrichment of non-process elements into the chemical cycles in a modern eucalyptus kraft pulp mill, natural and artificial purges must both be applied. Sparingly soluble NPEs are generally purged with the natural purges, but soluble Cl and K must be purged by other measures.

Since Cl and K are enriched in the ESP ash, efforts have been put to purge them from the ESP ash. Due to environmental reasons and high Na and S losses, direct purging of ESP ash may be limited. Therefore, dedicated purging systems have been developed. Most of the processes are based on the solubility of sodium and potassium compounds (Johansson, 2005).

When a new mill is started up, all the NPEs seek an equilibrium level where the inputs and outputs are in balance. The rate of NPE build-up depends on the degree of closure in the chemical cycles and is thus mill specific. Therefore, it is hard to predict the final equilibrium level beforehand.

Figure 8 shows one example of the rate of build-up of K and Cl in the ESP ash after the start-up of an eucalyptus kraft pulp mill. After around six months from the start-up, steady state levels of K and Cl were reached. The reached level of K and Cl was 60 mg/kgds (6%) and 40 mg/kgds (4%), respectively.

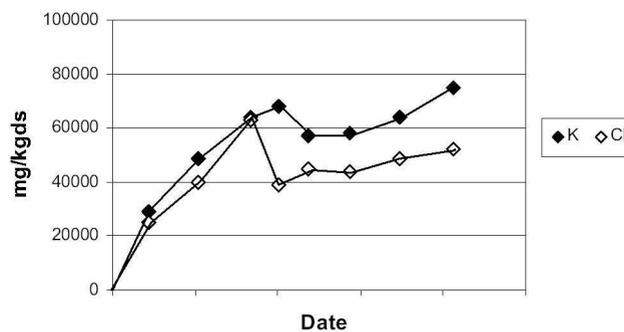


Figure 8: Rate of build-up of K and Cl in the ESP ash.

The potassium level is close to softwood mill levels (Figure 6), but the chlorine content in the ESP ash is around an order of magnitude higher than in softwood mills (Figure 7). The high Cl and K contents prevent the use of high steam outlet temperatures without a dedicated Cl and K purging system.

CONCLUSIONS

Effective NPE management is a must in eucalyptus kraft pulp mills. Most of the NPEs can be purged via natural routes including green liquor dregs, grits, and lime mud. With these routes, levels of low-solubility NPEs can be kept on an acceptable level. Typically, the levels in eucalyptus and softwood kraft pulp mills are close to each other.

However, chlorine levels are significantly higher in eucalyptus pulp mills than in softwood mills. Therefore, dedicated purging measures must be applied. High Cl concentrations in the process flows can affect the availability of the recovery boiler.

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