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A Review of Issues Related to Green Liquor Quality, and Approaches towards Improved Clarification

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Abstract

The quality of green liquor has a domino effect throughout the entire liquor preparation and recausticizing system in a kraft pulp mill. Poor quality green liquor impacts not only causticizing efficiency, but also lime mud characteristics, which in turn impact the quality of reburned lime and white liquor quality. The situation is exacerbated when economics drives mills to push pulp production beyond the original design capacities of their recausticizing systems.

Environmental drivers are setting in motion a trend towards increased closure of pulp mill and bleach plant water circuits in kraft mills. More and more, industry efforts are being focused on ways in which to integrate filtrates from the bleach plant and pulp mill into the chemical recovery system, and towards solving inherent problems in doing this. Increased pulp mill water closure makes it imperative to get good control of green liquor quality, since a marginal quality issue can very rapidly become magnified and degenerate into a serious problem due to limited purge points in closed systems. The build-up of non-process elements very rapidly increases the dead load in the liquor system, the negative impact of which is very quickly manifested in the form of poorer pulp quality.

This paper seeks to examine the issues related to green liquor quality, with particular reference to recent trends in the industry towards mill water closure. The paper also discusses approaches towards addressing this issue, with a review of some new technologies and liquor processing approaches.

Keywords

Kraft Pulp Mill, Green Liquor, Clarification, Dregs, Mill Closure, Pulp Quality, Bleach Plant Filtrates, Non-process Elements, Purge Points, Chemical Recovery, White Liquor Quality, Lime Quality.

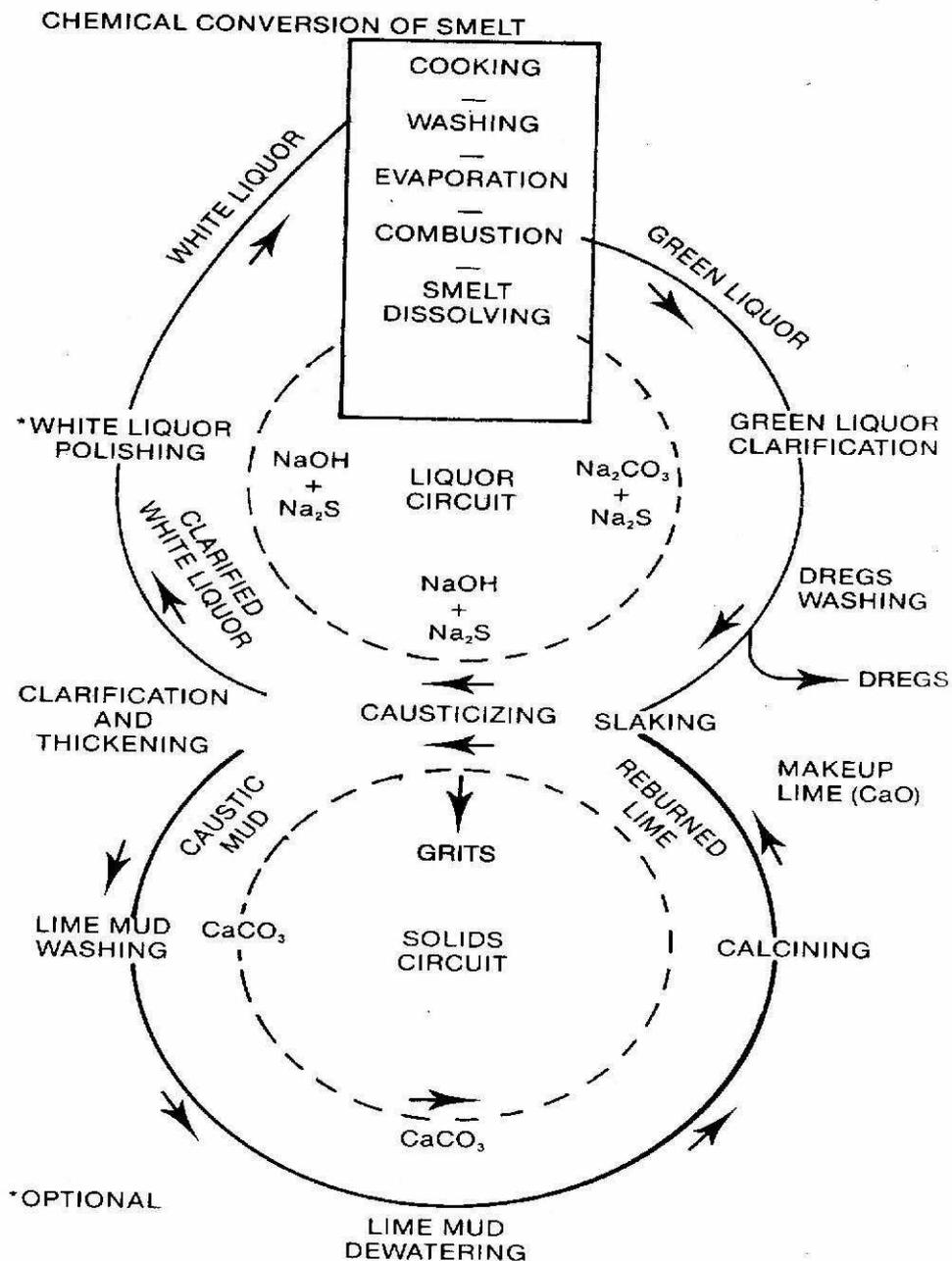
Fábrica de celulose kraft, licor verde, clarificação, dregs, fechamento de circuito, qualidade da polpa, filtrados da planta de branqueamento, pontos de purga, recuperação química, qualidade do licor branco, qualidade de cal.

A well-designed and operated recausticizing system is essential to maintaining pulp and paper quality. This is because the quality of pulp produced is dictated by the quality of white liquor used in cooking, which in turn is only as good as the quality of green liquor from which it is generated. In the regeneration of white liquor, it is essential to start with good control of the quality of green liquor. No matter how good the design of the white liquor system may be, it cannot compensate for loss in quality of green liquor. Green liquor quality has a domino effect on all downstream operations, starting with causticizing efficiency and white liquor clarification to lime mud quality and lime kiln operations. The next section examines in more detail some of these inter-relationships.

Fundamental Issues in Green Liquor Quality

Figure 1 is a schematic of the liquor and lime cycle in a kraft pulp mill [1]. Green liquor is constituted by dissolving molten smelt from the recovery boiler with weak wash from the dregs washer and the lime mud washer in a dissolving tank. Green liquor mainly consists of sodium carbonate and sodium sulfide. Sodium carbonate is converted to sodium hydroxide in the recausticizing cycle, resulting in white liquor comprised of sodium hydroxide and sodium sulfide, the active cooking chemicals in the kraft pulping process. Lime mud, mainly calcium carbonate, is separated from white liquor in the white liquor clarifier, and processed in the lime cycle (called "solids circuit" in the figure below). In this cycle, lime mud is washed, thickened and burnt in the lime kiln to form calcium oxide (reburnt lime), which is sent to the slaker, where it reacts with water to form slaked lime, or calcium hydroxide. Slaked lime is the agent responsible for causticizing of green liquor. It reacts with sodium carbonate in the green liquor to form sodium hydroxide and calcium carbonate. White liquor, comprised of sodium hydroxide and sodium sulfide, is clarified and used for cooking, thus circulating in the liquor circuit.

Figure 1. Liquor and Lime Cycles in a Kraft Pulp Mill [1]



In addition to sodium carbonate and sodium sulfide, green liquor contains impurities that need to be removed to prevent problems downstream in the liquor cycle, and in the production of pulp. The nature of the problems caused is dictated by the types of impurities found in the liquor. The most commonly found impurities are unburnt carbon from incomplete combustion of black liquor in the recovery boiler, and silica originating from wood. Called dregs, these are usually in the form of suspended solids, that are traditionally removed in the underflow from the green liquor clarifier following the dissolving tank. Green liquor from the dissolving tank usually contains about 800 to 1200 mg/l (ppm) of dregs [1,2], with possible excursions up to 3000 mg/l during upsets. Clarified green liquor is considered to be of acceptable quality for downstream operations at a concentration of 100 ppm suspended solids or less. If not reduced to acceptable levels, dregs can hamper lime mud settling characteristics, resulting in less than desirable white liquor clarity. Needless to say, poor quality white liquor negatively impacts pulp quality. White liquor that hasn't been well-clarified contains elements and compounds that do not take part in pulping reactions, and constitute a dead load on the system. The consequences of a higher dead load being carried around in the liquor system are (a) higher pumping costs, and (b) more opportunities for problems in process equipment.

The underflow from the green liquor clarifier usually has a concentration of 10% solids (dregs). The large amount of liquor carried in this stream contains valuable sodium that needs to be recovered. The dregs are therefore washed on a dregs filter, from where the filtrate is returned to the dissolving tank. Washed dregs are normally disposed of to a landfill. This represents an important purge point in the mill.

In addition to unburnt carbon and silica, non-process elements are also found in green liquor and need to be controlled. They circulate in the liquor and lime cycle, and are carried over with pulp. If not controlled to acceptable levels, they can build up to significant levels in the liquor and lime cycles, causing serious operational problems. Additionally, carry-over with pulp negatively impacts both pulp and paper quality, and can cause operational problems on the paper machine.

Trends towards increased water system closure in mills has made it vitally important to have suitable "kidneys" for control and removal of non-process elements. Green liquor clarification or filtration is an important kidney. Some of the issues related to non-process elements, strategies for their control, and where green liquor quality control fits into the scheme of things, will be examined in the following sections.

Non-Process Elements

There are two categories of non-process elements that are found in liquor systems – those that are soluble in the liquors, and those that are insoluble. Soluble non-process elements include Cl, K, Si and Al, which tend to accumulate in the liquor cycle. Insoluble elements include Ca, Ba, Mg, Mn, P, and Fe, which accumulate in the lime cycle [3,4].

Non-process elements, or NPEs, as they are commonly called, and methods for their control, are commanding increasing attention as mills look for ways to close up their bleach plants and mill water systems. Concepts for bleach plant closure include schemes that involve taking bleach plant filtrates counter-currently through the pulp mill, which carry with them a number of the non-process elements cited above.

Most of the non-process elements enter the pulp mill with wood. Other sources include the chemicals and water used in the mill. While the types of elements and their concentrations depend on the particular species and geographic location from where the wood originated, Table 1 lists a typical profile of non-process elements for a softwood, and the amount typically carried over in bleached pulp [4].

Table 1. Profile of Non-process Elements in Incoming Wood and Bleached Pulp [4]

Element	NPEs in Incoming Wood (g/ADMt Bleached Pulp)	NPEs in Bleached Pulp (g/ADMt Bleached Pulp)
Al	12 – 35	2 – 5
Si	2 – 25	1 – 5
P	50 – 100	2 – 5
Mg	75 – 250	10 – 40
Ca	500 – 1000	50 – 100
K	300 – 800	--
Cl	300 – 800	--
Mn	50 – 200	< 1
Cu	1 -- 2	0.1 – 0.3

These elements cause different types of problems in different areas of the mill. Some of these are:

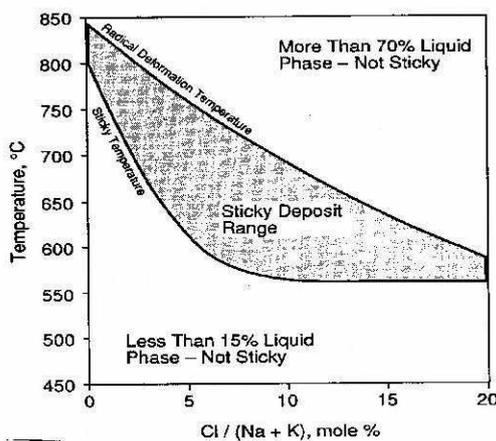
1. Cl and K

(a) Boiler Tube Deposits and Pluggage

Cl and K are known to lower the sticky temperature of dust and fume carryover particles in a recovery boiler, which can cause stubborn pluggage of the passages in the boiler tube bank. The carryover particles are mainly inorganic particles from the smelt. These particles are usually molten at the entrance to the superheater section, where the temperature is above 850°C, but solid at the generating bank exit, where the temperature is usually below 600°C. What this means is that between these two regions, the particles go through a transition from molten to solid, and pass through a regime of stickiness.

The sticky temperature is defined as that temperature at which there is just enough of a molten (liquid) phase to make the inorganics sticky. This is usually the temperature at which about 15 to 20% of the material is molten. The radical deformation temperature is the temperature above which there is enough of a liquid phase that the material is free-flowing. At this temperature more than 70% of the material is in the liquid phase. Consequently, there will not be a deposit /pluggage problem in the region of the recovery boiler where the temperature is below the sticky temperature or above the radical deformation temperature. Deposit formation and pluggage are a problem in the regime between these two temperatures. This is illustrated in Figure 2 [1].

Figure 2. The Temperature Regime where Sticky Deposits are Formed [1]



The concentrations of Cl and K have a significant impact on the sticky temperature. As the Cl/(Na+K) ratio increases from 0 to 5 mole %, the sticky temperature drops steeply anywhere from 200 to 300°C, depending on the K/(Na+K) ratio. This is shown in Figure 3 [1]. This means that as the concentrations of Cl and K build up, regions of the boiler that were below the sticky temperature, and thus free of deposit problems, can now fall in the sticky deposit regime between the sticky temperature and radical deformation temperature. This results in deposit formation on boiler tubes and pluggage of the passages.

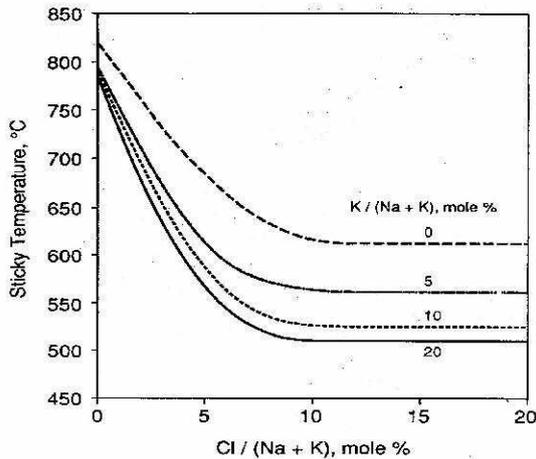


Figure 3. The Effect of Cl and K Concentrations on Sticky Temperature [1]

(b) Corrosion

Corrosion is a major problem commonly encountered in closed mill water systems. Both K and Cl are known to cause corrosion at neutral and acid pH conditions.

2. Ca, Ba, Si and Al

These elements are primarily responsible for scale deposits on process equipment and fouling of heat transfer surfaces. Scaling problems occur when either (a) soluble elements become insoluble as their concentration rises, or (b) they form insoluble salts such as carbonates, sulfates and oxalates, which precipitate out. Scale formation is extremely sensitive to pH, temperature and concentrations of the elements involved, since these are factors that dictate solubility behavior of inorganic compounds. Oxalic acids generated as lignin breakdown products during the bleaching process react with Ca to form calcium oxalate scale. Calcium oxalate, calcium carbonate and barium sulfate scales are common occurrences in bleach plants with tight filtrate circuits, which provide the opportunity for build-up of temperature and concentrations of Ca and Ba [5]. Calcium carbonate scale is a common problem in evaporators as well. Silica causes a significant increase in black liquor viscosity as liquor is concentrated in the evaporators, resulting in processing problems. Further, Al and Si are also responsible for sodium aluminosilicate scale which is extremely stubborn, and causes severe reduction of heat transfer capacity in evaporators due to its low thermal conductivity [1].

3. Mn, Cu and Fe

These metal ions are detrimental to the stability of various bleaching chemicals. They are known to be responsible for the catalytic decomposition of hydrogen peroxide under alkaline conditions, and also cause ozone decomposition. Such reactions also result in free radical formation, which negatively impact pulp viscosity.

Strategies for Control and Removal of NPEs

Strategies for control and removal of NPEs are based on the individual behavior of the elements, such as whether they are soluble or insoluble in alkaline liquors, whether they accumulate in the liquor cycle or lime cycle, whether they can be precipitated out by changes in pH etc. An in-depth discussion of these strategies is beyond the scope of this paper. A brief summary of possible purge points and control measures is described here.

1. Cl and K

These elements tend to be enriched in the electrostatic precipitator dust. In a survey of 17 mills, Tran *et al.* [6] compared the enrichment ratio of Cl and K at various points (ratio of concentration in the liquor or deposit to concentration in the as-fired liquor), and found it to be highest in the precipitator dust. Consequently, dust purge is a means to control these elements. The technologies available to achieve this have been addressed at length in a number of papers [3,6,7,8]:

- (a) BFR™ Chloride Removal Process: This technology developed by Champion exploits the higher solubility in water of sodium/potassium chloride as compared to sodium sulfate. The precipitator dust is completely dissolved in water, and then evaporated/concentrated to form sodium sulfate crystals. The sodium sulfate crystals are separated from sodium chloride which remains in solution by washing over a filter. The chloride-containing filtrate is disposed of, and the sulfate

crystals returned to the mix tank.

- (b) Other technologies that are being developed or being commercialized include Precipitator Dust Recovery (PDR) by Eka Chemicals, Precipitator Dust Purification (PDP) by Paprican and ProSep Technologies, a Japanese technology for potassium removal and electro-dialytic technologies [3].

2. Al and Si

These elements, being soluble in alkaline liquors, accumulate in the liquor cycle. Methods to remove them include lowering the pH to precipitate them, followed by filtration or sedimentation. High silica content in black liquor from non-wood pulping processes has been a particularly intractable problem. Desilication methods proposed for handling non-wood liquors in traditional recovery systems include lowering the pH of black liquor to around 9.8 -- 10.5 followed by filtration. It has been claimed that such a process can remove anywhere from 88% to over 90% of silicon with little co-precipitation of lignin [3,9]. Removal of Al requires lowering the pH to 7. Technologies such as the Netfloc® System developed jointly by Kemira and MoDo for the purpose of lowering the amount of extractives in pulp by addition of polyethylene oxide (PEO) to filtrates are also claimed to be an effective kidney for metal removal [10,11]. The pitch sludge that is formed as a result of interaction of extractives with PEO can apparently be removed by flotation. Trials run with Mixed Tropical Hardwood have been reported to result in a 91% reduction in Si and a 94% reduction in Al in the filtrate [11].

3. Ca, Ba, Mg and Mn

These elements are insoluble in alkaline liquors. Consequently, it should be possible to naturally purge these with the dregs from green liquor filtration or clarification. It has been proposed that for integration of acid bleach plant filtrates in the recovery system, these elements, which are in dissolved form in such acid filtrates, be precipitated and filtered out through contact with lime mud, lime or some other alkali source prior to integration [3,4].

Green Liquor Clarification and Filtration

As stated previously, the primary purpose of green liquor clarification has been to remove suspended solids, called dregs, from green liquor that adversely affect lime mud settling and other downstream operations. The size of these particles can vary over a wide range, from fairly large, hydrated inorganic particles to very fine carbon particles. These dregs particles are difficult to settle, whether it be due to their fineness, or due to the low density of the larger particles. Consequently, removal of dregs with traditional sedimentation clarifiers requires considerable green liquor residence time. This is achieved by designing the clarifier such that green liquor entering the clarifier rises at a certain design rate, called rise rate, allowing time for the particles to settle. The poor settling characteristics of these particles requires a low green liquor rise rate. Because of this, green liquor clarifiers tend to be rather large units, with a typical residence time of 12 hours. The side benefit of this is that they provide for green liquor storage.

However, one of the major drawbacks of clarifiers is the fact that once a clarifier of a certain size has been installed at a mill for a given production rate (throughput of green liquor flow), it is very difficult to increase the production rate (green liquor flow) due to the rise rate requirement to ensure good settling. Though settling aids can accelerate the settling rate of dregs particles to a certain extent, the increased production that can be realized through such measures is not significant.

More recently, different types of filter designs have been proposed to address some of these drawbacks [13]. Filters have the advantage that they do not occupy a large area, and have considerable turndown capability. However, blinding and plugging of the filter media can be a problem, and the operating costs can be high. Some designs claim to avoid this by inducing turbulence at the filter medium surface. Filters have been shown to be able to produce clarified green liquor with a lower solids content than clarifiers, typically around 20 to 25 mg/l (ppm).

As is evident from the discussion above, green liquor clarification or filtration also plays an important role in the control of non-process elements, and represents a vital kidney in the mill. A considerably significant amount of alkali-insoluble elements such as Ca, Ba, Mg and Mn are removed with the dregs from a green liquor clarifier or filter. Filters have also been shown to be effective in removing heavy metal ions such as Cu and Fe [12]. The partitioning of various non-process elements between dregs and clarified green liquor is presented in Table 2 [15].

Table 2. Partitioning of NPEs between Dregs and Clarified Green Liquor [15]

Element	% in Solution in Green Liquor	% in Dregs
Mg	< 3	> 97
Mn	< 6	> 94
Fe	< 11	> 89
Al	92	8
Si	99.5	0.5

Filter Designs for Green Liquor Filtration

Following is a brief description of some of the types of filters used for green liquor filtration.

1. Cross-Flow or Falling Film Filters

In this design, the filter consists of a vessel with vertical filter media. Green liquor is introduced at the top of the vessel, from where it is distributed to flow as a film on the surface of the filter media. A pressure differential is created across the filter media by the introduction of compressed air into the vessel. This provides the driving force to force the green liquor across the filter media. The clarified green liquor that passes through the media is collected at the bottom of the filter elements, from where it is sent to downstream operations. Unclarified liquor, along with dregs, that stay on the outside surface and do not pass through the media, is collected at the bottom of the vessel, from where it is recirculated back to the top of the vessel along with fresh unfiltered green liquor. The dregs are concentrated in this fashion and periodically withdrawn and sent to the dregs washer. The filter media have to be periodically flushed with water.

2. Cassette Filters

In cassette filters, the filter elements are tubular. Rather than compressed air, the pressure generated by the green liquor feed pump is used to drive the liquor across the filter media. The clarified liquor is collected from the inside of the filter media. Dregs build up on the outside surface as a cake, causing an increasing pressure drop across the filter. At a pre-determined pressure drop setting, the vessel is allowed to drain, raising the concentration of the dregs cake up to 30 to 50% solids. At this point, the filter elements are backwashed with high pressure water, knocking the cake off and discharging it to a dregs tank, from where it is conveyed to the dregs washer. Blinding and tearing of the filter medium cloth are common problems.

3. Tube Filters

Though originally designed for white liquor clarification, some mills have reported reusing these filters for green liquor filtration with good results [12]. The principle of operation is similar to the cassette filter, though the design and control schemes are different. Here too, the liquor is forced through the filter media by the pressure of the feed pump, and a cake of solids builds up on the outside surface. The cake is dropped off the media by a flow reversal to the feed tank, rather than the application of flushing water. When used for white liquor clarification, the cake consists of lime mud, which drops to the bottom of the filter vessel. Proper control of this level of lime mud in the vessel is critical to smooth operation. In green liquor filtration applications, the solids removed as a cake off the filter consists of dregs. In this case, it is the level of dregs in the vessel that would have to be controlled. Problems arise if the level of solids collected rises beyond the elevation of the white liquor (or green liquor, as the case may be) feed line to the filter.

4. Pressure Disk Filters

Disk filters operate on a principle similar to lime mud precoat filters. However, disk filters use pressure rather than vacuum to drive the liquor across the medium, and disks are used in place of drums. A precoat of lime mud is used for cake buildup. These have been mostly used in white liquor clarification, though they have been proposed for green liquor filtration as well.

Most of these filters require a subsequent dregs washing step for good recovery of sodium from the dregs. However, because of the fact that the dregs cake formed in filters have a higher solids concentration (30% to 50%) than dregs from a clarifier (typically around 10% solids), less sodium is carried in liquor with the dregs, which places less of a burden on the dregs washer to recover this valuable sodium.

Mill Installations and Experiences

A number of mills have installed cross-flow falling film filters for green liquor filtration. Ahlstrom has 12 mill installations of their X-Filter, which is a cross-flow filter [14]. Results from operations of such a filter at the Louisiana-Pacific mill in Samoa, CA as reported in the literature are presented in Table 3 below [16].

Table 3. Green Liquor Filtration Results Using an X-Filter at Louisiana Pacific, Samoa, CA [16]

Element	Unfiltered Green Liquor (mg/l)	% Reduction after Filtration
TSS	963.4	97
Ca	565.1	47
Si	286.6	17
Ba	8.3	52
Mn	14.4	88
Mg	32.4	86
Fe	17.3	79
Cu	1.0	59
Al	24.1	39
Pb	2.9	21

Green liquor filtration results from a commercial X-Filter operation as reported by Ahlstrom [15] are presented in Table 4 below.

Table 4. Green Liquor Filtration Results from a Commercial X-Filter Installation [15]

Element	Green Liquor Feed (mg/l)	After Filtration (mg/l)
Zn	7.54	<0.04
Fe	29.4	0.4
Mn	50.8	0.66
Mg	184	0.05
Cu	0.74	0.03
Ca	176	6.2
Al	13.3	1.4
Si	115	94.5
P	38.6	36.8

The Stora Cell mill in Norrsundet, Sweden has reported reusing existing pressure tube filters operating on white liquor for green liquor filtration [12]. These filters replaced their existing green liquor clarifier when they decided to install new pressure disk filters for white liquor clarification. The green liquor clarifier was reused purely for clarified green liquor storage. Figures 4 and 5 illustrate NPE removal achieved by the pressure tube filters [12]. Their conclusion was that the filters were effective in removing NPEs that are insoluble, and not very effective at removing those that were in soluble form.

Figure 4. Amount of NPEs Before and After Green Liquor Filtration [12]

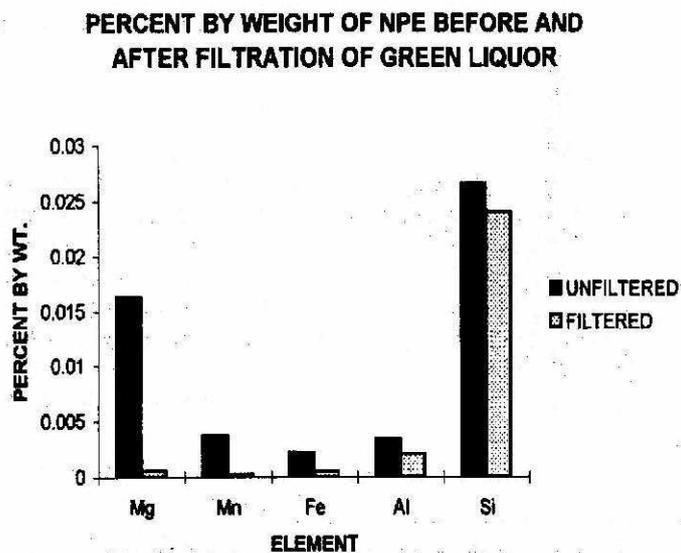
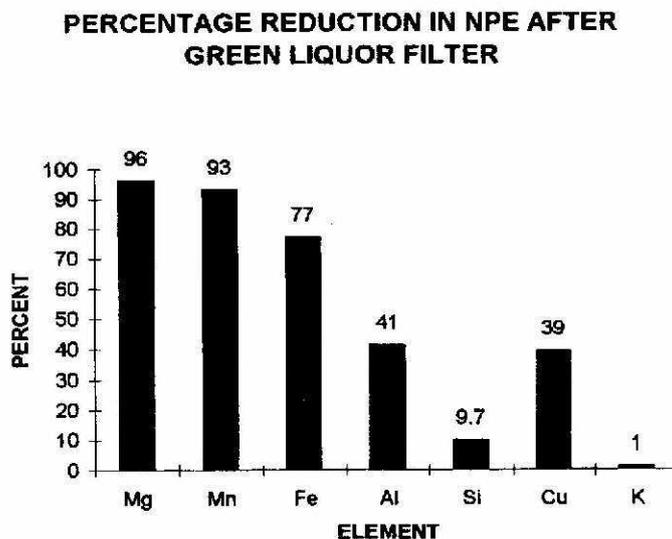


Figure 5. Percent Reduction of NPEs across Pressure Tube Filter [12]



Conclusions

Green liquor quality has a significant impact on the performance of the rest of the recausticizing system, as well as on pulp and paper quality. In addition to the need for removing unburnt carbon and silica found as dregs in all green liquor systems, green liquor clarification is an important kidney for the purging of non-process elements. The trend towards increased mill system closure makes it all the more important to have efficient green liquor clarification systems. Filters are a viable alternative to sedimentation-type clarifiers for green liquor clarification. Operating experience gathered so far indicate that they are also effective at removal of non-process elements, which is an important consideration as mills move towards increased system closure. More importantly, filters allow mills with overloaded clarifiers to increase production by providing increased green liquor throughput. Operating issues, such as blinding and plugging of filter media, tearing of filter cloth, proper controls, and high operating costs, are being worked on and improved designs being developed.

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