Influence of chlorine and potassium on operation and design of chemical recovery equipment

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ABSTRACT: The main objective of this study is to evaluate the effect of chlorine (Cl) and potassium (K) on the operation and design of equipment in the chemical recovery cycle. Due to stricter environmental regulations, mill closure tends to increase, causing the accumulation of undesirable elements such as Cl and K in the kraft liquor cycle. Total inorganic solids in pulping liquors increase in the presence of Cl and K, which affects the operation of chemical recovery equipment. The objective here is to show the magnitude of these impacts and estimate operational cost differences by using different Cl and K contents in liquors. Pulp mill material and energy balances for each case are used for this purpose, and a modern Brazilian pulp mill served as a base case model. The results show that for one specific range, the solids content in black liquor can be 6.6% higher by increasing the mass percentage of Cl and K in black liquor. This difference reduces the black liquor higher heating value by 6.2% and increases the amount of dry solids to burn in the recovery boiler, also by 6.6%. The evaporation load increases along with steam consumption. This lowers total electricity output by up to 1.6 MW due to reduced flow to the condensing stage of the steam turbine. The balances also demonstrate that some pumping costs can be 12% higher when operating from a low to high concentration of Cl and K in black liquor.

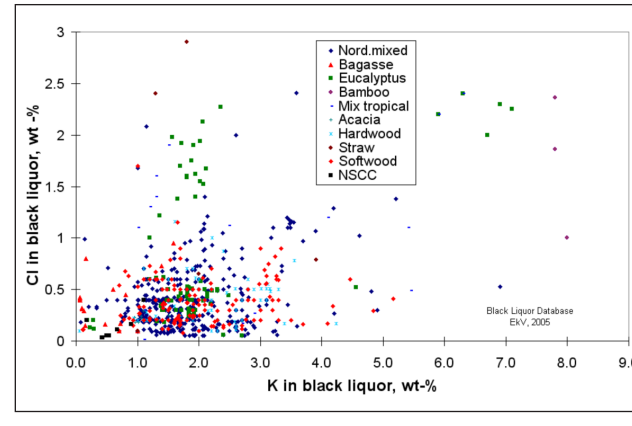
Application: Showing the influence of the Cl and K dead loads on the recovery system could help some pulp mills understand the additional cost acquired and the equipment capacity reduction.

Elements such as chlorine (Cl) and potassium (K) accumulate in the pulp mill liquor cycle. Research on the role of Cl and K in pulp mills is mostly concentrated on their impact to the recovery boiler corrosion and fouling rates, caused by the enrichment of these elements in ash [1]. Cl ions form in pulping liquors dead loads of potassium chloride (KCl) and sodium chloride (NaCl). Potassium acts similarly as sodium, so active (potassium hydroxide [KOH] and potassium sulfide [K_2S]) and non-active (potassium carbonate [K_2CO_3], potassium sulfate [K_2SO_4], etc.) compounds are formed. The influence of white liquor concentration on pulp mill energy consumption was studied by Kojo decades ago [2]. The relevance of K and Cl on the whole pulp mill operation has been raised recently. Two such examples analyzing the effects of dead loads on the recovery system have been studied by Grace and Tran [3] and Saeidi et al. [4].

Cl and K in the liquor cycle

Cl and K inputs to the kraft pulping process include wood, mill freshwater, makeup chemicals, and spent acid from chemical manufacturing. These inputs can vary a lot depending on, for example, whether pulp is processed in coastal or inland mills. The typical net input for chloride in eucalyptus mills varies between 1–3 kg Cl/a.d. metric ton and, for potassium, 2–5 kg K/a.d. metric ton. For softwood mills, these values are, respectively, 0.3–1 kg Cl/a.d. metric ton and 1–2 kg K/a.d. metric ton [5].

Cardoso et al. demonstrated that black liquor in Brazilian eucalyptus mills can contain 1.5 – 2.2 wt% K and 1.9–4.5 wt% Cl [6]. The range probably refers to a coastal forest region due to the high concentration of chlorine found in coastal regions. For the base case, lower values were considered, i.e., about 1.1 wt% for K and 0.5 wt% for Cl. It is important to note that an ash treatment system must then be installed to achieve these lower levels of Cl and K in liquor. **Figure 1** shows a summary of the various types of black liquor found worldwide [7]. Although values up to 8 wt-% for potassium can be found in exceptional cases, the maximum amount that is considered in this work is 3.5 wt% K.



1. Chloride (Cl) and potassium (K) content in black liquor [7].

TARGET MILL

The target mill was a new eucalyptus mill in South America, with a production capacity of 1250000 a.d. metric tons/year [8]. The cooking operation and yields were assumed to remain constant. This implies no significant changes in the bleaching and wood preparation operations. The operating systems that were evaluated included: evaporation, recovery boiler, and causticizing. The cost impacts of liquor and cooling water pumping are also accounted for in this study.

Evaporation

To achieve the efficient removal of metals, dilution water is needed during the pulp washing process. If the amount of inorganics in the weak liquor to be removed from pulp increases, additional dilution is required. Thus, the effective capacity of the evaporator becomes limited as the water carrying extra chemical loads increases. Simply, the total water entering the evaporator is increased [3]. The boiling point rise (BPR) of black liquor is also affected by increased dead load level. The change in the BPR impacts the equipment design and process conditions. However, it is difficult to estimate the magnitude of the BPR change, since black liquor is a complex solution of various organic and inorganic compounds and few rigorous methods exist for predicting the BPR [9]. Taking these difficulties into account, this present work does not evaluate the impacts of dead loads on the boiling point rise. The dry solids flow of black liquor, which is the focus of this section, is not affected by the change in BPR.

Recovery Boiler

The recovery boiler burns black liquor and provides appropriate conditions for the reduction of sulfate ions. In such an environment, the desirable anions, such as S^{2-}, can be reactivated. The reduction efficiency in the recovery furnace determines the amount and content of sodium and potassium sulfate dead loads circulating with the various liquor flows. The increased dead load reduces the black liquor high heating value and, subsequently, reduces the lower furnace temperature. This makes operation more problematic, causing, for example, lower reduction with higher sulfur emission [10]. It is also expected that if the dry solids in black liquor increase, the flow of the virgin black liquor to the recovery boiler furnace will increase in the same proportion. The increase in the flow of dry solids and decrease in the black liquor heating value can leave the heat load (GJ/a.d. ton) to the recovery boiler furnace relatively unchanged.

Causticizing

A white liquor of good quality is essential for the proper operation of the pulping process. Good quality in this case means a high concentration of active ions (OH^- and S^{2-}) and a low concentration of inert ions such as CO_3^{2-}, SO_4^{2-} or Cl^-. Thus, a high causticizing efficiency (CE) is desired to reduce the dead load of sodium carbonate in the liquor cycle. It is known that Goodwin's curve describes the highest achievable causticizing degree. In order to avoid adverse consequences of over-liming, many mills operate below the equilibrium value. They typically try to achieve values about 3% below the theoretical, resulting in causticizing efficiencies between 82%-84% [11]. Mills without an appropriate control system can develop unstable operation with a CE below 80%. Some authors [12,13,14] have researched the influence of inorganic salts on causticizing equilibrium.

Traditional use of total titratable alkalis (TTA) to determine causticizing degree is misleading, as according to Dor-

Variables	Unit	A	B	B1	C1	C
Cl in black liquor	**% wt**	**0.0**	**0.5**	**0.5**	**2.0**	**3.2**
K in black liquor	**% wt**	**0.0**	**0.2**	**1.1**	**3.2**	**3.5**
Dry solids in black liquor	kg/a.d. metric ton	1338.7	1351.1	1355.2	1406.4	1440.6
White liquor AA	gNaOH/l	**138.1**	**138.1**	**138.1**	**138.1**	**138.1**
White liquor TTA	gNaOH/l	156.3	156.6	156.6	157.8	158.8
White liquor TAC	gNaOH/l	157.8	160.5	160.5	170.1	177.6
Causticizing efficiency	%	83.85	83.61	83.60	82.71	81.98
White liquor flow	m^3/h	456.9	456.8	456.6	456.0	455.9
Inorganics in black liquor	kg/a.d. metric ton	402.3	415.1	419.8	473.5	508.8
Total dead loads in white liquor	kg/a.d. metric ton	94.6	107.5	113.3	169.6	205.2
Black liquor HHV	MJ/kg	14.45	14.32	14.28	13.76	13.43
Weak black liquor flow	m^3/h	1235.3	1246.7	1250.5	1297.7	1329.3
Virgin black liquor flow to furnace	tDS/day	4939	4985	5000	5189	5315
Evaporation capacity	t_{H2O}/h	1093	1103	1107	1149	1177
- Steam consumption	t/h	182.2	183.9	184.5	191.9	196.8
- Cooling water flow	m^3/h	11539	11647	11682	12127	12424
Power generation	MW	162.7	162.5	162.4	161.5	160.9

I. Mill balances with fixed active alkali (AA) content in white liquor. All tonnage references are metric.

Variables	Unit	A	B	B1	C1	C
Cl in black liquor	**% wt**	**0.0**	**0.5**	**0.5**	**2.0**	**3.0**
K in black liquor	**% wt**	**0.0**	**0.2**	**1.1**	**3.2**	**3.5**
Dry solids in black liquor	kg/a.d. metric ton	1340.9	1351.1	1355.2	1398.5	1426.3
White liquor active alkali (AA)	gNaOH/l	140.0	138.1	138.1	132.0	127.4
White liquor TTA	gNaOH/l	159.0	156.6	156.6	148.8	143.3
White liquor TAC	gNaOH/l	**160.5**	**160.5**	**160.5**	**160.5**	**160.5**
Causticizing efficiency	%	83.44	83.60	83.60	84.15	84.54
White liquor flow	m^3/h	450.6	456.8	456.6	477.6	494.3
Inorganics in black liquor	kg/a.d. metric ton	404.4	415.1	419.8	465.9	494.7
Total dead loads in white liquor	kg/a.d. metric ton	96.7	107.5	113.3	161.8	191.1
Black liquor HHV	MJ/kg	14.43	14.32	14.28	13.83	13.56
Weak black liquor flow	m^3/h	1229	1239	1242	1282	1308
Virgin black liquor flow to furnace	tDS/day	4939	4985	5000	5160	5262
Evaporation capacity	t_{H2O}/h	1087	1095	1100	1134	1157
- Steam consumption	t/h	181.2	182.6	183.2	189.4	193.4
- Cooling water flow	m^3/h	11474	11562	11597	11971	12210
Power generation	MW	162.8	162.6	162.5	161.7	161.2

II. Mill balances with fixed total anion concentration (TAC) in white liquor. All tonnage references are metric.

ris [15], for example, the causticizing reaction equilibrium constant is a function of the total anion concentration (TAC), i.e., the sum of non-titratable anions (NTA) and TTA. The effect of a larger concentration of Cl in liquor, for example, is to lower the equilibrium constant of the causticizing reaction, thereby reducing the equilibrium causticity and the effective alkali for a fixed TTA. The presence of these undesirable ions, such as NaCl, has an adverse effect on the conversion of Na_2CO_3 to NaOH.

Saeidi et al. have recently observed the influence of some alkali salts on the causticizing equilibrium [4]. They use a thermodynamic model based on experimental data to calculate the phase equilibrium in aqueous solutions of ionic salts. Their results show that the causticizing efficiency (CE) decreases with an increasing amount of chlorine in the liquor. Potassium and sulfate ions caused only a slight impact on the CE. Saeidi et al. have evaluated this change by fixing the TTA for different levels of Cl and K.

MILL BALANCE AND RESULTS

In order to evaluate the influence of Cl and K on certain operational variables, calculations were performed using a detailed pulp mill balance developed in Microsoft Excel format. Since thousands of variables and calculations are involved, tools for tracing the dependent variables become important. This feature makes Excel particularly user friendly for the task. A modern Brazilian pulp mill served as the base case model and different inputs for Cl and K in the dry black liquor were tested.

The cost saving calculations are based on more realistic conditions, where the Cl content ranges from 0.5% to 2.0% and K from 1.1% to 3.2% (wt in black liquor). They are identified as point B1 and point C1. These points represent, for example, a coastal mill with the intention to invest in available technologies for removing Cl and K. The hypothetical case when both Cl and K equal zero is also calculated and is identified as point A. The base case model is represented by point B1. **Table 1** shows some variables at these specific points. Graphical comparisons were done by varying Cl content from 0.5% to 3.0% and K from 0.2% to 3.5% (wt in black liquor). These are identified as point B and point C.

Grace and Tran stated that around 20% of the recovered inorganics that circulate through the cycle are as inactive species [3]. Table 1 shows comparable values with several Cl and K levels. The balance is calculated with regard to the following major assumptions:

- Active alkali (AA) fixed as 138.1 gNaOH/l
- Difference of CE to Goodwin's curve: 3.5%
- Fixed values for sulfidity (32%) and reduction (96%)
- Formation rate of thiosulphate and sulfite compounds are fixed
- Weak black liquor concentration fixed at 15.2%
- No ash treatment for Cl and K removal
- Operation hours: 350 days or 8400 h
- Problems regarding fouling and corrosion are not considered

The assumption in Table 1 of fixed AA is the basis for all the graphical comparisons. Pulp mills normally operate the causticizing plant to target the active or effective alkali. Later, as in **Table 2**, an alternative is evaluated by assuming fixed TAC in white liquor.

Table 1 shows that, with constant AA but different Cl and K content in liquor, the mill operating parameters do change, such as evaporation capacity, black liquor heating value, and

recovery boiler load. Increasing inert load can be seen as the black liquor dry solids flow increases and black liquor heating value decreases. The white liquor flow basically does not change and hence the causticizing capacity is not significantly affected. The white liquor causticizing efficiency is, however, affected.

Effects of Cl and K on black liquor properties

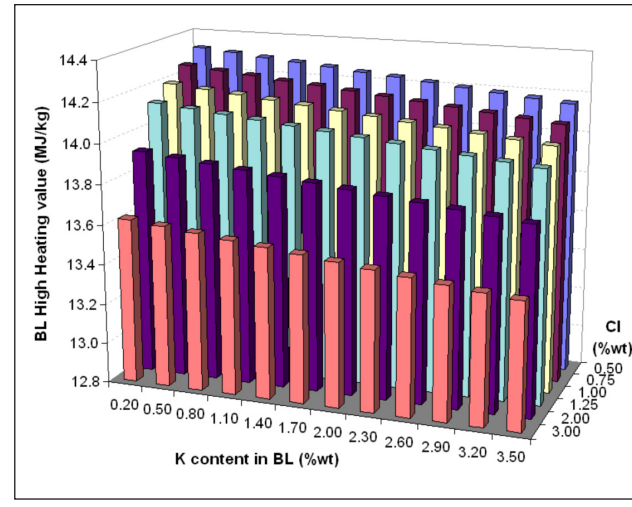
The dry solids in black liquor consist of both organic and inorganic fractions. The organic content remains basically the same but as we increase the Cl and K content, the inorganic content increased from 415.1 kg/a.d. metric ton (point B) to 508.8 kg/a.d. metric ton (point C). This change corresponds to change in dead loads in black liquor ranging from 107.5 kg/a.d. metric ton to 205.2 kg/a.d. metric ton. The total black liquor dry solids formed for the same points ranged from 1351.1 kg/a.d. metric ton to 1440.6 kg/a.d. metric ton (Table 1). The magnitude is in accordance with values referenced by Grace and Tran [3], who have suggested total dead loads ranging from 7% to 15% of the total black liquor solids.

The method used for calculating the black liquor higher heating value [16] takes into account the dead load content. Its correlation with Cl and K can be observed in **Figure 2**. The trends show that an increase in potassium causes a lesser decrease on the black liquor heating values than increasing chlorine content. For a fixed percentage of Cl, the heating value decreases by around 1.2% when K in black liquor increases from 0.2% to 3.5%wt. For a fixed percentage of K, the heating value decreases by around 4.5% when Cl in black liquor increases from 0.5% to 3.0%wt. Because the amount of organics in black liquor remained constant, the heat load to furnace did not change from about 19.4 GJ/a.d. metric ton.

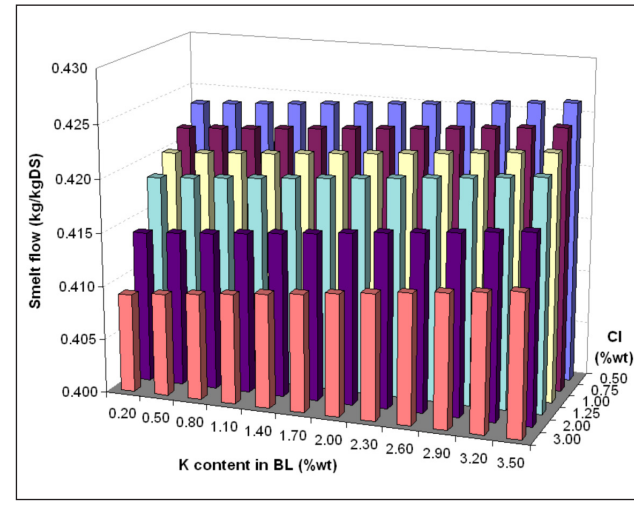
Effects of Cl and K on recovery boiler operation

It is reasonable to assume that the reduction efficiency is not significantly affected by the presence of dead loads. Thus, a fixed reduction efficiency of 96% can be taken as the basis for the balance calculations. **Figure 3** shows the impacts on smelt flow. The change in the smelt flowing to dissolving tank will not significantly affect the mill energy balance. Smelt sensible heat represents around 21% of total heat losses from black liquor combustion. The smelt heat losses are roughly proportional to the mass flow. Because the total smelt mass flow changes only by 5%, the percentage remains roughly the same.

The energy balance results also shows that the steam generation is increased 1% by increasing the potassium content from point B to point C. This can be attributed to the heat absorbed during the reduction of K_2SO_4 to K_2S (9630 kJ/kg). Since reduction heat for Na_2SO_4 to Na_2S is higher (13090 kJ/kg), the net heat for steam production becomes slightly higher. The heats absorbed during Na_2S and K_2S reduction represents around 35% of the total heat losses. The remaining losses refer to heat lost in hot flue gas, smelt sensible heat, and some other minor streams. It is typical for flow instruments to state the accuracy as 1%-2 % of the measured



2. Effect of Cl and K on the black liquor heating value.



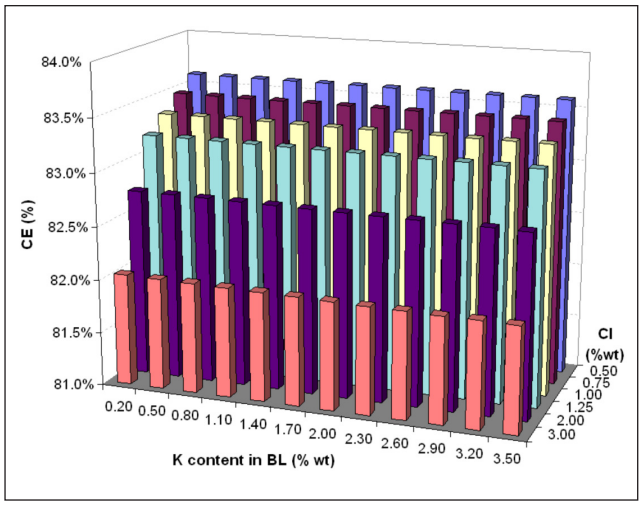
3. Effect of Cl and K on smelt flow.

value. Hence the small change of 1% in the steam generation would not be noted in practical mill operation.

Effects of Cl and K on causticizing efficiency

Dorris states that Goodwin's curve correlates not only TTA, but also with NTA [15]. The difference of 3.5% to Goodwin's curve assumed in this work defines the causticizing efficiency for each alkali level. With fixed AA, the causticizing efficiency decreases with the increasing of Cl, as **Figure 4** shows. At the same time, the total anion concentration (TAC) is increased by 11% from point B to C. When the TAC in white liquor increases, the amount of sodium carbonate will also increase with decreasing CE. This may disturb, for example, the operation of the evaporation plant, especially if combined with a high content of sodium sulphate in black liquor [17]. Table 1 shows that the causticizing efficiency decreases by 1.9% from point B to point C.

According to Saeidi et al., coastal mills are predicted to have a CE 1%–2% lower than that of inland mills [4]. This is due to the Cl concentration in liquors of coastal mills being



4. Effect of Cl and K on causticizing efficiency for fixed AA.

2–4 times higher than those of inland mills. It is important to emphasize that they have considered, for this case, fixed values for the TTA. In practice, if constant TTA is required by pulp mills, the causticizing efficiency will be inevitably lower.

Effects of Cl and K on evaporation load

If the amount of inorganics in the weak liquor to be removed from pulp increases, additional dilution is required. Consequently, the evaporation load increases, along with low pressure steam and cooling water consumption. The balance shows both steam and cooling water consumption increasing by around 4% from point B1 to C1. The differences are equivalent to 7.4 metric tons/h of low pressure steam and 445 m^3/h of cooling water.

Mill balances for fixed TAC

The second set of comparisons is made by performing balances with fixed TAC. These have a fixed amount of alkali (constant TAC) in white liquor. Table 2 thus illustrates how the mill balance would change for the same amount of circulating alkali if the K and Cl would change. The alternative has been evaluated in this work, especially for the causticizing process, because some authors have published valuable data using TTA as variable [4,15]. The change in recovery capacity for evaporation and the recovery boiler is smaller, but the white liquor flow changes. It is clear that to achieve same cooking yield, mills should dramatically change the cooking time. So, in practice, mills operate according to Table 1.

Table 2 shows that potassium causes no visible changes in the CE which, surprisingly, may increase by 0.9% in the presence of chlorine from point B to point C because of the decrease in the TTA content of white liquor.

Effects of Cl and K on pumping cost

One of the aspects studied was the magnitude of change in liquor pumping because of increased K and Cl levels. Four pumping systems are considered in the evaluation: weak black liquor pumping from cooking to evaporation plant (P1), white liquor pumping from causticizing to cooking (P2), green liquor pumping from dissolving tank to causticizing (P3), and cooling water pumping from cooling towers to evaporation plant (P4). Realistic design data were used to better estimate pumping costs and are shown in **Table 3**. The cost saving evaluation is done by using a range from point B1 to point C1. The hydraulic (P_h) and shaft (P_s) power in kW for each pumping are defined as follows:

$P_h = q\,\rho\,g\,h / 3.6\,10^6$

$P_s = P_h / \eta$

q: liquor volumetric flow [m^3/h]
ρ: liquor density [kg/m^3]
h: differential head [m]
η: pump efficiency

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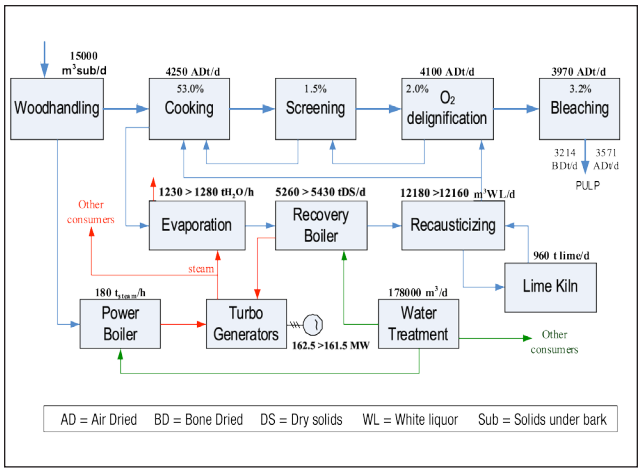
An electricity price of 30 Euros/MWh was used as the basis for cost evaluation. It is assumed that pump efficiencies do not change according to their characteristic curves. Table 3 shows that, from point B1 to C1, the pumping cost increased by 54000 Euros/year for fixed TAC. If AA is fixed, the white liquor (P2) and green liquor pumping systems (P3) will present no significant changes. Even so, the difference in cost for all the pumps reaches 59000 Euros/year. When TAC is fixed, the costs for P2 and P3 become higher, because the AA charge for cooking does not change since the chip flow to the digester remains the same. Thus, the white liquor flow has to increase in order to compensate for its reduced AA. In practice, these cost differences for P2 and P3 and even P1 are not representative for each pulp mill.

Effects of Cl and K on power generation

A modern 1250000 a.d. metric ton/year pulp mill equipped

Description	Unit	WBL, P1	WL, P2	GL, P3	CW, P4
Head	M	60	35	40	40
Pump efficiency	%	78	78	78	78
Change in shaft power AA (TAC)	kW	35.0 (29.0)	1.8 (10.5)	0.7 (9.8)	100.0 (165.3)
Change in pumping cost AA (TAC)	Euros/year	8800 (7700)	453 (2649)	166 (2470)	50000 (41660)
Pumping cost increase AA (TAC)	%	11.8 (9.9)	3.0 (15.9)	1.0 (14.7)	11.9 (10.0)

III. Change in pumping cost for fixed AA and fixed TAC (in parentheses) from point B1 to C1.



5. Schematic diagram of 1.25 million a.d. metric tons/year pulp mill, with design capacities indicated above each block.

with a condensing tail turbine has a potential for generating 162.4 MW of electricity at design point B1. For a fixed AA, the energy balance shows that an increase from point B1 to C1 in steam consumption, mainly by the evaporation process, decreases electricity generation by 0.9 MW. From point B to C, the decrease reaches 1.6 MW. This is caused by the reduction of steam flow to the condensing tail part of the steam turbine. The power change is equivalent, for example, to one modern wind turbine. Since it is typical for pulp mills to sell surplus electricity to the grid, it is interesting to estimate the cost involved. Thus, considering the most realistic range (from B1 to C1) and assuming 30 Euros/MWh in 350 operation days, the cost of 0.9MW is equivalent to about 252000 Euros/year. For a fixed TAC, the cost would reach about 215000 Euros/year.

Pulp mill design

The stream flows and equipment capacities in pulp processing are calculated by assuming cooking yield (53%) and fixed shrinkage percentages. The changes can be seen in **Figure 5** for a fixed AA. Both the fixed TAC and fixed AA cases are also presented in **Table 4**. All inputs are based on actual data typically used for dimensioning pulp mills. References for some values can be consulted online [18].

As Table 4 shows, an increase of K and Cl can significantly increase the required design capacities of recovery departments. Therefore, the predicted level of K and Cl in the pulp mill to be considered is of utmost importance.

CONCLUSIONS

The results of this study show that NPEs like Cl and K affect the operation and capacity of chemical recovery equipment. The detailed pulp mill balance from our Microsoft Excel format deals with complex processes and contains hundreds of inputs and interrelated parameters that are not mentioned in this paper. Although the results do not represent actual measured process values, they show that pulp mills having high concentrations of Cl deserve attention with regard to chemical recovery equipment operation. It is clear from the results that K and Cl do affect departmental sizing and operations costs at pulp mills; thus, decreasing K and Cl will be beneficial. **TJ**

ACKNOWLEDMENTS

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System	Unit	Fixed TAC				Fixed AA			
		B	B1	C1	C	B	B1	C1	C
Recovery boiler	tDS/day	5250	5260	5430	5540	5250	5260	5460	5590
Causticizing	m³WL/d	12180	12180	12740	13180	12180	12180	12160	12160
Evaporation	$_t H_2O$/h	1220	1220	1260	1290	1230	1230	1280	1310

IV. Selected design for studied cases. All tonnage references are metric.

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ABOUT THE AUTHORS

It is well known that dead loads (poor reduction and causticizing degree) cause negative impacts on pulp mill operations. While there was ample research on the impacts of elevated potassium (K) and chlorine (Cl) on corrosion, there were few studies about their role as additional dead load. We realized that it was possible to evaluate the magnitude of these impacts by using real process data in an advanced pulp mill balance that we developed.

The effects of K and Cl on individual pulp mill department operations have previously been studied. Our research expands the previous whole mill operation studies by considering new variables related to potassium and chlorine. Our most difficult challenge in this study was to accurately correlate the non tritable anions with the causticizing efficiency. This was addressed by conducting a literature survey and by consulting companies with practical expertise.

One interesting finding from this study was that the increased content of Cl and K in black liquor can significantly increase the amount of dry solids to be burned in the recovery boiler.

The results from this study show the importance of removing non process elements (NPEs), such as Cl and K, from the liquor cycle. Not only do corrosion costs increase with elevated levels of Cl and K, but significantly higher pulp mill operations costs result as well. Using our research, pulp mills can better estimate the magnitude of impacts from Cl and K on their recovery processes and determine the best levels for these NPEs in their operations.

Hamaguchi

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One of the next interesting areas of study for us will be the influence of NPE on the chemical recovery process when certain amounts of lignin are removed from the black liquor.

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