

Vaporization from Alkali Carbonate Melts with Reference to the Kraft Recovery Furnace

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This paper examines the processes responsible for fume generation in the kraft furnace. Fume generation under oxidizing conditions was found to be an order of magnitude greater than under strongly reducing conditions. Addition of sodium hydroxide to the sodium carbonate-sulphide melt did not increase the fuming rate. Potassium and chloride levels in kraft furnace fume are lower than values calculated from equilibrium data; however, if the enhanced vaporization of sodium due to oxidizing conditions is taken into consideration, the low potassium and chloride levels in the fume can be explained. Raoult's law gives an accurate description of sodium chloride vaporization from a sodium carbonate-sulphide-chloride smelt under oxidizing conditions.

INTRODUCTION

One of the principal steps in the kraft pulping process is the recovery of the pulping chemicals. In the recovery furnace, black liquor from the pulping process is burned and the residual pulping chemicals are converted to alkali carbonate and sulphide. The burning of black liquor is considerably more complex

than the burning of fossil fuels. The combustion process must be controlled to achieve both the combustion of the organic content of the black liquor and the reduction of the sulphur compounds present to sodium sulphide.

During the burning of the kraft black liquor, a large quantity of fume particles are generated from the vaporization and condensation of inorganic sodium and potassium compounds. These particles are typically 0.25 to 1.0 μm in diameter and are composed principally of sodium sulphate and sodium carbonate. Fume particles form deposits on the heat transfer surfaces, which reduce heat transfer rates, may eventually plug the gas passages, and may lead to the formation of a corrosive environment at the metal surface. Fume that does not deposit in the furnace is captured by an electrostatic precipitator, mixed with the black liquor, and recycled to the furnace.

Borg et al. [1] conducted a study of the origins of sulphur and sodium emissions from a kraft boiler. From electron microscopy studies, two types of particles were identified: those formed by entrainment and burning of black liquor droplets, and those formed from vaporization of inorganic compounds in the lower furnace and condensation of these compounds in furnace gases. From the particle sizes it was determined that the weight percentage of actual carry-over

(burnt liquor particles) was very small (less than 1 wt.% of the total particles) compared to the fume particles. However, the location at which the particles were collected was not identified, and it is well known that carry-over particles, being heavier, tend to drop out of the gas stream before the precipitators. Rzhinshvili and Kaplun [2] also identified both types of particles. They found that the larger carry-over particles were nearly absent from the gas stream beyond the economizer.

Potassium and Chlorine

Important non-process elements in the kraft process are potassium and chlorine. Keitanniemi and Virkola [3] found that the wood chips are the major source of potassium. Seawater transportation of logs can be a significant source of chloride. Karjalainen et al. [4] estimated that the sodium chloride input to a kraft mill from seaborne logs can be 45 lb of sodium chloride per ton of pulp (22.5 g/kg pulp). This is a relatively high level compared to a typical sodium sulphate makeup of 80 lb per ton of pulp (40.0 g/kg pulp). Relatively small amounts of chloride are also introduced into the recovery system with makeup chemicals. A potential source of chloride is the recovery of bleach plant effluent to eliminate a major source of water pollution. Karjalainen et al. [4] estimated that sodium chloride input from bleach plant



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effluent could be as high as 270 lb per o.d. ton of pulp (135 g/kg pulp).

Compared to Na_2CO_3 , potassium and chloride compounds such as NaCl and KCl are relatively volatile in the kraft furnace and the fume is enriched in these elements. Reeve et al. [5] reported potassium enrichment factors [the ratio of $\text{K}/(\text{Na} + \text{K})$ in the fume to $\text{K}/(\text{Na} + \text{K})$ in the white liquor] of 2.4 for inland mills and from 1.1 to 1.6 for West Coast mills. For Finnish mills, Keitaniemi and Virkola [3] reported an enrichment factor of 1.6. For chloride, Reeve et al. [5] reported an enrichment factor (the ratio of Cl/Na in the fume to Cl/Na in the white liquor) of 3.0 for inland mills and from 1.2 to 1.6 for West Coast mills.

The presence of potassium and chloride in the fume can have a marked effect on the operation of a kraft recovery boiler. Potassium and chlorides significantly lower the melting point of deposits in the kraft furnace. Shivgulam et al. [6] reported the addition of sodium chloride to a synthetic kraft smelt lowered its eutectic melting point from 763 to 598°C. The replacement of 5 mol% sodium with potassium further lowered the eutectic melting point to 525°C. Reeve et al. [5] have attributed severe superheater corrosion to the presence of chlorides and potassium in the fireside deposits. Although superheater deposits are primarily formed through carryover, fume would enrich these deposits in potassium and chloride. The accelerated corrosion was thought to result from the lower melting point of the enriched deposit.

Equilibrium Calculations in Recovery Furnaces

Several researchers have used thermodynamic equilibrium calculations to predict the liquid and gaseous species present in the kraft furnace. Bauer and Dorland [7] were among the first to apply this technique to the kraft furnace. Their study predicted that the volatile fuming species in the furnace are Na and Na_2 . Warnqvist [8] also applied equilibrium thermodynamics to the kraft furnace and concluded that, in addition to Na and Na_2 , NaOH is also an important fuming compound. Warnqvist recognized that the equilibrium assumption was somewhat unrealistic, but felt that equilibrium calculations provide an indication of the processes and chemical species present in the furnace.

Recently detailed thermodynamic equilibrium calculations on the inorganics in the kraft furnace have been made by Pejryd and Hupa [9] and Shiang and Edwards [10]. Shiang and Edwards determined that equilibrium calculations predict chloride and potassium enrichment factors greater than 10. They checked these calculations with those of

Pejryd and Hupa and found good agreement.

Shiang and Edwards proposed that the observed enrichment factors were lower than those predicted from equilibrium calculations as a result of carryover dilution. Carryover may dilute the chloride and potassium content in the deposits found in the superheater and front section of the boiler bank. However, there are few carry-over particles in the precipitator dust and the chloride and potassium enrichment factors in the precipitator dust range from 1.1 to 3.0. Therefore, carry-over dilution is not the cause of the lower predicted chloride and potassium enrichment factors observed in the fume.

Oxygen Enhanced Fume Generation

Work by Clay et al. [11] and Cameron et al. [12] has shown that air oxidation of sodium sulphide in a sodium sulphide/sodium carbonate melt can produce large quantities of sodium carbonate fume. The rate of fume generation under oxidizing conditions was found to be considerably greater than the rate under strongly reducing conditions. This phenomenon was unexpected and very difficult to explain in terms of equilibrium treatments.

Recently, Cameron [13] has shown that the large quantity of fume generated during sulphide oxidation is a result of sodium oxidation in the gas phase. Oxidation of sodium vapour near the melt's surface lowers the partial pressure of sodium in the gas phase and increases the rate of sodium vaporization. This mechanism for fume generation will have a major effect on fume generation in the kraft furnace. Therefore, the fume generation rates within the furnace will be significantly different from those predicted by equilibrium treatments.

Objectives

The objectives of this research are to compare the relative vaporization processes present in the kraft furnace and to determine the mechanism responsible for the lower than equilibrium predicted

potassium and chloride enrichment factors.

EXPERIMENTAL

Experimental System

The vaporization experiments were conducted by monitoring the fume produced from alkali carbonate/sulphate/sulphide/chloride melts under various experimental conditions. The experimental system, shown in Fig. 1, consisted of an induction heated reactor, gas meters and fume filter. The reactor vessel was an alumina crucible 6.4 cm in diameter and 10 cm high contained in a stainless steel reactor. The steel retort was heated by an induction heating coil energized by a 20 kW Lepel high frequency power supply. The alumina crucible was heated by thermal radiation from the steel retort. The temperature of the melt was monitored using a chromel-alumel thermocouple. The fume from the reactor was followed by filtering the off-gas and weighing the fume particles. To ensure that this gravimetric method collected all the fume particles in the off-gas, the sodium content of the off-gas after the filter was checked with a flame photometer. Although some fume deposition was observed near the top of the reactor, the weight of this deposit was considerably less than the weight of the fume collected on the filter. Therefore, fume deposition within the reactor system did not significantly affect the measured fume generation rates. The fume generation rates were found to be highly reproducible.

The fume particles collected were typically white spherical particles approximately 0.25 to 1.0 μm in diameter. These particles were composed principally of sodium carbonate, potassium carbonate, sodium chloride and potassium chloride.

Comparison of Fume Generation Rates Under Different Experimental Conditions

The equilibrium treatment of fume generation assumes that the large quantity of fume present in the kraft furnace results from the high temperature and

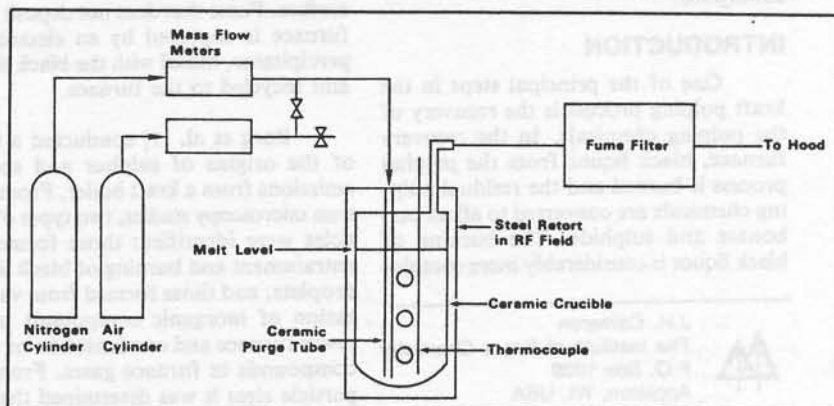


Fig. 1. Experimental system.

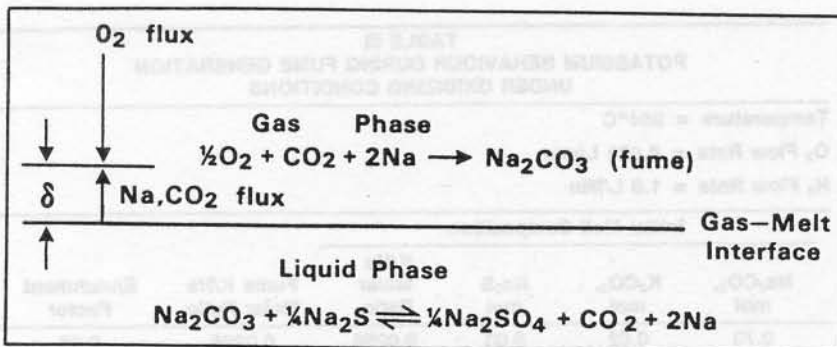


Fig. 2. Fume generation under oxidizing conditions.

TABLE I
TYPICAL FUME GENERATION RATES DURING SULPHIDE OXIDATION

Initial Melt Composition			
Na ₂ CO ₃ = 0.77 mol		Purge = 1.15 L/min at 13.0% O ₂	
Na ₂ S = 0.06 mol			
Temperature = 954°C			
Time, s	Calculated Concentrations		Fume Generation
	Na ₂ SO ₄ mol/L	Na ₂ S mol/L	Rate, mg/min
191	0.010	0.049	21.2
278	0.015	0.044	21.2
452	0.025	0.035	22.5
627	0.035	0.025	23.9
738	0.041	0.019	24.5
933	0.052	0.008	24.1

TABLE II
COMPARISON OF FUME GENERATION UNDER DIFFERENT CONDITIONS

Initial Melt Composition		Temperature = 954°C	
Na ₂ CO ₃ = 0.77 mol		N ₂ Purge = 1.0	
Na ₂ S = 0.03 mol			
Conditions	Fume Generation ± S, mg/min		
• N ₂ Purge Only	0.76 ± 0.02		
• Oxidizing Conditions 0.15 L/min O ₂	22.9 ± 1.5		
• Reducing Conditions			
Carbon Monoxide			
0.03 L/min	0.76 ± 0.005		
0.05	0.80 ± 0.005		
0.10	0.72 ± 0.002		
Hydrogen			
0.02 L/min	0.70	—	
0.05	0.80	—	
0.10	2.0	—	
• Kraft Char Addition			
Carbon Content of Melt, wt. %			
0.49	3.24	—	
0.97	3.8 ± 0.4		
1.9	4.1 ± 0.3		
2.8	3.8 ± 1.7		
3.6	3.4 ± 0.7		
• NaOH Addition, mole added			
0.01	0.60 ± 0.005		
0.10	0.64 ± 0.05		

reducing conditions present in the furnace bed. The major reducing species present in the kraft furnace are carbon, carbon monoxide, and hydrogen. In this study, fume generation rates were measured with various levels of these reducing species present and compared to rates produced under oxidizing conditions.

Fume Generation Under Oxidizing Conditions

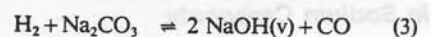
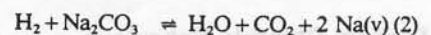
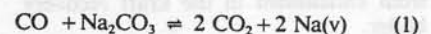
The origin of fume during sulphide oxidation has been described by Cameron [13]. Fume produced during sulphide oxidation results from the oxidation of sodium vapour in the gas phase. The oxidation of sodium produces a sodium sink in the gas phase, reducing the partial pressure of sodium and the mass transfer resistance to vaporization. The vapour sink significantly increases the rate of sodium vaporization. The situation is illustrated in Fig. 2. The relatively high Na vapour pressure results from the equilibrium reaction of sodium carbonate sodium sulphide. The sodium then vaporizes and reacts with carbon dioxide and oxygen to form sodium carbonate.

Shown in Table I are typical fume generation rates during air oxidation of sodium sulphide in a sodium carbonate/sulphide melt. These are presented for comparison with fume generation rates under reducing conditions.

By analyzing the melt during sulphide oxidation, it was determined that sulphide oxidation consumes all oxygen supplied to the melt. The sulphate and sulphide levels in Table I were then calculated from an oxygen balance on the system.

Fume Generation Under Reducing Conditions and with Sodium Hydroxide Present

Fume generation under reducing conditions was studied by adding hydrogen, carbon monoxide or carbon to sodium carbonate-sulphide melts and monitoring fume. These reducing agents may react with Na₂CO₃ to form either sodium or sodium hydroxide as shown in Equations (1) to (4).



Hydrogen and carbon monoxide were mixed with the nitrogen steam and introduced below the melt's surface. The carbon was obtained from the pyrolysis of kraft black liquor. The pyrolyzed char which contained approximately 27% fixed carbon was added to the sodium carbonate and sulphide salts. This mix-

ture was then rapidly brought to the desired temperature.

Equilibrium treatments predict that a major source of fume is the formation and vaporization of sodium hydroxide. This mechanism for fume generation was studied by adding sodium hydroxide pellets directly to sodium carbonate/sulphide melts.

With the exception of the carbon addition experiments, the fume generated during these experiments was sodium carbonate. During the carbon addition experiments, the fume consisted of a mixture of elemental sodium and sodium carbonate.

Table II shows the rate of fume generation under various experimental conditions. Neither the addition of carbon monoxide to the purge stream nor the addition of sodium hydroxide directly to the melt increased the fume generation rate above that observed under a pure nitrogen purge. The addition of hydrogen to the purge and the addition of carbon directly to the melt slightly increased the fume generation rate. In these experiments fume generation under oxidizing conditions was significantly greater than that observed under reducing conditions or with NaOH added directly to the melt.

Potassium and Chloride Behaviour During Fume Generation

Potassium Behaviour During Fume Generation Under Oxidizing Conditions

To determine if fume generated during sulphide oxidation would be enriched in potassium, the potassium/sodium ratio in fume generated during sulphide oxidation was compared to the potassium/sodium ratio in the melt. Shown in Table III are the potassium/sodium ratio in the melt, the potassium/sodium ratio in the fume, and the enrichment factor.

From this table, it is apparent that the fume generated during sulphide oxidation is not enriched in potassium. Therefore, oxidation-enhanced fume generation is not the cause of the potassium enrichment in the kraft recovery boiler.

Sodium Chloride Behaviour in Sodium Carbonate

The behaviour of sodium chloride in sodium carbonate/sulphide melts was studied by adding sodium chloride to sodium carbonate/sulphide melts and monitoring the fume generation rate under both oxidizing and nonoxidizing purges. The chloride/sodium ratios in the melts were chosen to be in the range of typical smelt samples as reported by Reeve et al. [5]. The fume samples collected from these melts were analyzed for

TABLE III
POTASSIUM BEHAVIOUR DURING FUME GENERATION UNDER OXIDIZING CONDITIONS

Initial Melt Composition					
Na ₂ CO ₃ , mol	K ₂ CO ₃ , mol	Na ₂ S mol	K/Na Molar Ratio	Fume K/Na Molar Ratio	Enrichment Factor
0.75	0.02	0.03	0.0256	0.0225	0.88
0.71	0.06	0.03	0.0811	0.0888	1.09
0.67	0.1	0.03	0.143	0.141	0.99
0.37	0.4	0.03	1.0	0.96	0.96
0.1	0.71	0.03	5.46	6.01	1.10

TABLE IV
CHLORIDE ENRICHMENT FROM CARBONATE MELTS

Temp., °C	Melt Cl/Na Molar Ratio	O ₂ Flow Rate, L/min	Fume Cl/Na Molar Ratio	Enrichment Factor
954	0.10	0.0	0.86	8.6
954	0.049	0.1	0.0815	1.66
982	0.049	0.1	0.0759	1.55
954	0.122	0.1	0.221	1.81
982	0.122	0.1	0.175	1.43

TABLE V
CALCULATED AND MEASURED VAPORIZATION RATES OF SODIUM CHLORIDE DURING SULPHIDE OXIDATION IN A CARBONATE MELT

Total Fume Generation, mg/min	Cl Content of Fume, wt. %	Calculated NaCl Vaporization, mg/min	Measured NaCl Vaporization, mg/min
14.6	5.34	1.2	1.28
19.3	4.95	2.2	1.6
16.2	14.1	3.1	3.76
24.9	11.5	4.62	4.71

sodium and chloride and the enrichment factor then calculated. Table IV shows the results of these experiments.

Fume from experiments with no oxygen in the purge gas contained 86% sodium chloride and an enrichment factor of 8.6 was obtained. This enrichment factor compares closely with that of 10 predicted by Shiang and Edwards [10] from equilibrium calculations. Under oxidizing conditions, the rate of sodium vaporization is enhanced by gas phase oxidation of the sodium vapour and the fume enrichment factor is reduced to the range of 1.4 to 1.8, which is near that reported by Reeve et al. [5] for kraft furnaces.

If the vaporization of sodium chloride is an equilibrium process and the melt is an ideal system, the vaporization of sodium chloride may be calculated assum-

ing Raoult's Law [Eq. (6)] and assuming that the gas stream is saturated in sodium chloride.

$$p_{\text{NaCl}}(T) = p^*_{\text{NaCl}}(T) X_{\text{NaCl}} \quad (6)$$

Here, $p_{\text{NaCl}}(T)$ is the equilibrium partial pressure of NaCl at temperature T ; p^*_{NaCl} is the vapor pressure of pure NaCl at temperature T ; and X_{NaCl} is the mole fraction of NaCl in the melt.

The vaporization rates for sodium chloride calculated from Raoult's Law are compared to the actual rates calculated from total fume generation rates and fume composition in Table V.

As shown in this table, the calculated and measured sodium chloride vaporization rates during sulphide oxidation enhanced fume generation are in

TABLE VI
EFFECT OF CHLORIDE ON POTASSIUM ENRICHMENT
DURING SULPHIDE OXIDATION

Melt Composition		Purge Rate				
Na ₂ CO ₃ = 0.565 mol		N ₂ = 1.0 L/min				
Na ₂ S = 0.14 mol		O ₂ = 0.0 to 0.1 L/min				
NaCl = 0.1 to 0.16 mol						
KCl = 0.02 to 0.10 mol						
Temperature 954°C						
Melt		O ₂ Flow Rate, L/min	Fume		Enrichment Factor	
Cl (Na + K)	K (Na + K)		Cl (Na + K)	K (Na + K)	Cl	K
0.119	0.0187	0.0	0.745	0.119	6.3	6.8
0.122	0.062	0.1	0.164	0.10	1.3	1.6
0.119	0.0188	0.1	0.173	0.046	1.5	2.4
0.127	0.0278	0.1	0.16	0.051	1.3	1.9
0.116	0.0129	0.1	0.19	0.026	1.5	2.0

TABLE VII
POTASSIUM AND CHLORIDE
ENRICHMENT FACTORS

Calculated Enrichment Factors		Observed Enrichment Factors	
Cl	K	Cl	K
1.6	1.2	1.3	1.6
1.3	1.2	1.5	2.4
1.3	1.2	1.3	1.9
1.3	1.2	1.5	2.0

zation of the chlorides. The majority of the fume generated is a result of oxidation-enhanced vaporization of sodium and potassium.

close agreement. This indicates that sodium chloride vaporization is an equilibrium process and that its vaporization rate during oxidation-enhanced fume generation can be determined assuming Raoult's Law is valid.

Potassium Behaviour in Alkali Carbonate/Chloride Melts

With no chloride in the melt, there was no potassium enrichment in the fume during sulphide oxidation. To determine the effect of chloride on potassium enrichment, various levels of potassium chloride and sodium chloride were added to the melt and the fume generated during sulphide oxidation was analyzed for potassium and chloride. The results of these experiments are shown in Table VI.

As shown in Table VI, the addition of potassium chloride to the melt results in the fume generated during sulphide oxidation being enriched in both potassium and chloride. As previously shown in Table V, no potassium enrichment was observed when the melt contained potassium but no chloride. Therefore, the potassium enrichment observed in the fume is due to the presence of potassium chloride in the melt.

To determine if Raoult's Law describes potassium chloride vaporization during sulphide oxidation fume generation, the potassium and chloride enrichment factors were calculated based on Raoult's Law and compared to the observed enrichment factors. These calculations are shown in Table VII.

It was previously shown that with no chlorides in the melt no potassium enrichment occurred in fume generated during sulphide oxidation. Since the carbonate portion of the fume in Table VII results from gas phase oxidation-enhanced vaporization, no potassium enrichment should occur in this portion of the fume. Therefore, potassium enrichment factors in Table VII were calculated assuming that all potassium

enrichment resulted from potassium chloride vaporization. The calculated potassium enrichment factor is significantly lower than the observed enrichment factor. This difference results from either potassium chloride having an activity coefficient much greater than one, or from the carbonate portion of the fume also being enriched in potassium due to the presence of potassium chloride in the melt.

Although the sodium and potassium chloride vaporization are responsible for the chloride and potassium enrichment observed in fume generated during sulphide oxidation, the vaporization of these species contributes little total fuming rate. For example, in Table VII less than 20% of the total potassium and sodium in the fume results from the vaporization of the chlorides.

DISCUSSION

It is apparent (from this study) that the fume generation during sulphide oxidation is considerably greater than under strongly reducing conditions. One mechanism that has been proposed for fume generation in the kraft furnace is the formation and vaporization of sodium hydroxide. Equilibrium calculation by Pejryd and Hupa [9] show that the main sodium species in the gas phase are NaOH and Na. Borg et al. [1] also states that sodium is vaporized primarily as NaOH(g) and Na(g). The vaporization of sodium hydroxide was studied by adding sodium hydroxide to the alkali carbonate-sulphide melts and measuring the fuming rate. In these experiments, relatively high levels of sodium hydroxide were added to the melt and no increase in fume generation was observed. Sodium hydroxide is also formed in the melt when hydrogen

REFERENCE: CAMERON, J.H. Vaporization from Alkali Carbonate Melts with Reference to the Kraft Recovery Furnace. *Journal of Pulp and Paper Science*, Vol. 14(4) J76-81 July 1988. Paper offered as a Contribution to the Technical Section, Canadian Pulp and Paper Association. Not to be reproduced without permission from this organization. Manuscript received July 6, 1987; approved by the Review Panel March 25, 1988.

ABSTRACT: This paper examines the processes responsible for fume generation in the kraft furnace. Fume generation under oxidizing conditions was found to be an order of magnitude greater than under strongly reducing conditions. Addition of sodium hydroxide to the sodium carbonate-sulphide melt did not increase the fuming rate. Potassium and chloride levels in kraft furnace fume are lower than values calculated from equilibrium data; however, if the enhanced vaporization of sodium due to oxidizing conditions is taken into consideration, the low potassium and chloride levels in the fume can be explained. Raoult's law gives an accurate description of sodium chloride vaporization from a sodium carbonate-sulphide-chloride smelt under oxidizing conditions.

RÉSUMÉ: Notre article traite des mécanismes responsables de la production de la fumée dans les chaudières des usines de pâtes kraft. L'oxydation de sulfure favorise une production de fumée que est près d'un ordre de grandeur plus élevé que celui qui résulte de conditions de réduction élevée. La soude caustique n'est pas un type de composé chimique hautement volatil dans une masse fondue faite de carbonate de soude et de sulfure. Les facteurs d'enrichissement en-deçà du point d'équilibre, dont font montre le chlorure et le potassium dans les chaudières d'usines kraft, sont sans doute le résultat de la dilution de la fumée engendrée par la vaporisation du sodium sous l'effet incitatif de l'oxydation. La loi de Raoult décrit avec précision la vaporisation du chlorure de sodium dans une masse fondue faite de carbonate de soude, de sulfure et de chlorure au cours de la vaporisation du sodium sous l'effet incitatif de l'oxydation.

KEYWORDS: RECOVERY FURNACES, FUMES, SULFIDES, REACTION MECHANISMS, VAPORIZATION, SODIUM HYDROXIDE, CHLORIDES, THEORIES, CARBONATES, REACTION KINETICS.

is present in the gas phase and, although significant quantities of carbon monoxide were evolved during the hydrogen reducing experiments (indicating the formation of sodium hydroxide), no increase in fume generation was observed. Therefore, sodium hydroxide is not highly volatile in an alkali carbonate-sulphide melt.

One of the major areas of interest in the kraft furnace is the behaviour of the potassium and chloride compounds. Equilibrium calculations on the vaporization rates in the furnace predict a chloride enrichment factor considerably greater than that actually observed. The experimental results presented in this study demonstrate that oxidation enhanced vaporization of sodium can reduce the enrichment factor for chloride to that observed within the kraft furnace. Oxidation-enhanced vaporization of sodium is then the likely mechanism that results in the significantly lower than predicted chloride enrichment factor. The experimental results of this study demonstrate that the sodium chloride vaporiza-

tion rates during oxidation-enhanced fume generation can be predicted by assuming Raoult's Law is applicable.

No potassium enrichment was observed during oxidation-enhanced fume generation when chlorides were not present in the melt. When chlorides were present in the melt, the fume was enriched in potassium and the enrichment factors were similar to those observed in the kraft furnace. Therefore, the potassium enrichment in the kraft furnace is likely enhanced due to the presence of chloride.

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