# Sources of Odour and Control in a Kraft Pulp Mill

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Adaptability of the kraft process to a wide variety of wood species and the production of high brightness bleached pulp have led to a spectacular rise in acceptability and growth of the krait pulping process. Higher pulp yield and considerable flexibility of the process ensure that this increasing trend will continue for some years.

The two essential operations in a kraft process include pulping and chemical

The sources of odour and the chemical reactions responsible for the release of odoriferous components in the effluent streams from a kraft pulp mill are outlined. The emissions from the vorious sources show that the multiple effect evaporators, the direct contact evaporator and the recovery furnace contribute most to the odour problem; the digester relief and blow gases and condensates, pulp washer vent and to a lesser extent smelt dissolver vent and lime kiln gases account for the minor sources of odour and sulfur loss.

Some of the control methods available for reducing the kraft mill odour include oxidation of black liquor, combustion of non condensable gases from digester, blow tank and evaporators, steam or air stripping of condensate streams and efficient control of the recovery furnace operating conditions. The shortcomings of these techniques are discussed



Mr. Hariharan Veeramani, Assistant Professor, Department of Chemical Engineering, Indian Institute of Technology, Kanpur. recovery, Figures 1, 2 and 3. The active white liquor chemicals, sodium hydroxide and sodium sulfide, effect dissolution of non-cellulosic portions of wood chips with the accumulation of a wide variety of organic and inorganic compounds in the black liquor, Table I. The spent inorganic chemicals from the digestion must be reclaimed and reused for an economic operation of the kraft process. The dissolved wood components support combustion and provide process steam while the recovered inerganic chemicals in the smelt, sodium

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Figure 2 - Kraft pulp: mill - Pulping section - Batch digester.

carbonate and sodium sulfide are utilised in the preparation of white liquor.

The use of sodium sulfide as an active pulping reagent, the chemical responsible for the superiority of the kraft process, contributes to the well-known kraft mill odour problem.

#### **Sources of Odour Formation**

The sources of odour release and the reactions leading to the formation of malodorous compounds are summarised in Table 2.

Sodium sulfide in white liquor may hydrolyze in accordance with equation (1). The hydrolysis is strongly dependent upon pH and temperature. The first dissociation constant of hydrogen sulfide at 25°C corresponds to  $pK_1 =$ 7.05(1); the second dissociation constant is not determined with certainty but is probably around  $pK_2 = 14.0(2)$ . Both sulfide and hydrosulfide ions are probably present at the initial alkalinity of white liquor (pH = 14.0); over the pH range prevailing in a kraft digester and the final black liquor (pH = 11.0) the hydrosulfide ion will be the predominant species.

Dimethylation reactions (2 and 3) lead to the formation of methyl thiol and dimethyl sulfide. Mercaptide ions may hydrolyze to some extent at all pH levels to form methyl thiol, frequently called methyl mercaptan in pulping terminology, reaction (4). Equation (5) represents air oxidation of mercaptide ion. The products of reactions (3, 5 and 6), namely dimethyl sulfide and dimethyl disulfide, are not ionised by the alkaline process liquors and hence their vapour pressure is independent of pH level. These reactions may take place in the digester, blow tank, pulp washer, evaporators and elsewhere in other units, to an extent dependent upon the availability of oxygen.

Carbonation reactions (7 and 8) lead to the loss of hydrogen sulfide and methyl

thiol to the flue gas in the direct contact evaporator. Sodium sulfate added as a make up chemical and sodium thiosulfate resulting from the oxidation of sodium sulfide in the black liquor are reduced to sodium sulfide in the reducing zone of the recovery furnace, reactions (9 and 10). The control of furnace atmosphere determines the release of malodorous sulfur compounds from the recovery furnace (3, 4); oxidation of the reduced forms of sulfur compounds accounts for the presence of sulfur dioxide in the flue gas. Smelt dissolving tank emissions consist mainly of hydrogen sulphide and dusts. The small amount of sodium sulfide present in the lime-mud is carbonated to hydrogen sulfide and oxidised to sulfur dioxide with the excess air used for combustion, reactions (11 and 12). Sulfur is lost to some extent at the pulp washers depending upon washer efficiency (15-25 pounds of salt-cake per ton of pulp) (5) and at the dregs washer.

Sodium losses in a kraft pulp mill are incurred with the pulp leaving pulp

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FIGURE 3: KRAFT PULP MILL CHEMICAL RECOVERY SECTION

washers, smelt dissolving tank, dregs washer and in particulate emissions from the recovery furnace and lime kiln. Efficient pulp washers (5,6) and the use of various types of scrubbers and electrostatic precipitators (7,8) have effectively curtailed the sodium losses from the recovery cycle. In many Fulp mills, this situation has resulted in a disproportionately high loss of sulfur from the system.

The emission of sulfur compounds besides being a source of atmospheric pollution contributes significantly to the loss of the sulfur from the pulp mills. The positions of odour release and sulfur loss are denoted by the symbol 'S' in Figures 1 to 3. The values reported in the literature for the sulfur losses in a pulp mill chemical recovery operation vary considerably and extend over a rather wide range. The actual emissions in a particular mill will depend upon a number of factor such as black liquor pH, sulfidity, influence of

air oxidation, type and capacity of equipment and operating conditions.

Column A in Table 3 represents the sulfur losses in a conventional mill with little or no control of these emissions (9-19). It may be observed from these values that the multiple effect evaporators as well as the direct contact evaporator and the recovery furnace considered together contribute most to the odour problem; digeser relief and blow gases and condensates, pulp washer vents and to a lesser extent smelt dissolver vent and lime kiln gases account for the minor sources of sulful loss and odour.

# Kraft Mill Odour Control Methods :

Techniques currently in use for the control of emission of odorous sulfur compounds and particulate matter from a kraft pulp mill are reviewed by a number of authors (3,4,9,13,15-22). A modern kraft recovery system incorporating some of the recent advances in the control of odour and sulfur loss is shown in figure 4.

The noncondensable gases from the digester, blow gases, pulp washer vent and the multiple effect evaporators may be burned in the recovery furnace, lime kiln or a separate furnace (23,24). In some cases, malodorous compounds in the noncondensable gases as well as the condensates are oxidised with the chlorination effluent from the bleach plant (25-27). Steam stripping (SE-KOR Process) and in some instances air stripping has been proposed to i s o late malodorous compounds from the blow gas condensate and evaporator condensates (15,28,29).

The diffusion washing system of the Kamyr continuous digester shown in Figure 4, allows pulp to be withdrawn at a much lower temperature than with batch digesters (5); this minimises the amount of blow gas released and consequent odour problems at this point.

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Pulp washing in the HI-Heat washing zone of a Kamyr digester and in a diffusion washer over the blow tank, besides completing the entire unbleached pulp wash with a low loss of sodium (11-16 pounds of salt-cake per ton of pulp), eliminates odour release to the atmosphere since the pulp is washed in the complete absence of air (6).

The most significant effort in odour control is the use of an efficient oxidation system which stabilizes hydrosulfide ion in the black liquor according to the following reactions (30).

$$2 \text{ NaHS} + 2O_2 = \text{ Na}_2 S_2 O_3 + H_2 O$$
 (13)

$$2 (CH_3)_{9}S_{9} + 4 OH^{-}$$

$$2 CH_3S^- + H_2O = (CH_3)_2S + HS^- + OH^-$$
(6)

(5)

Black liquor oxidation greatly reduces the problem of hydrogen sulfide and methyl mercaptan loss and odour emanation in the multiple effect evaporator condensates and noncondensable gases. The most significant contribution of black liquor oxidation is the reduction of hydrogen sulfide loss from the direct contact evaporator which otherwise occurs through the carbona tion reaction (7). The more common equipment designs for oxidising black liquor include the asbestos sheet filled tower with concurrent flow of black liquor and air, the Bergstrom-Trobeck unit based on foam formation, the air sparged tank and a tower packed with cross partition rings (31, 32).

Oxidation of black liquor is normally quite efficient and several installations have reported one hundred per cent efficiency of hydrosulfide oxidation. Emission of malodorous compounds varies with the degree of oxidation of black liquor sodium hydrosulfide. Total oxidation of hydrosulfide eliminates hydrogen sulfide, otherwise released in the contact evaporator; the effect of partial oxidation depends upon black liquor pH and the concentration of sodium hydrosulfide (33). The reversal of black liquor oxidation reaction is discussed later.

The recovery furnace emissions depend upon several factors : composition and concentration of black liquor, flow pattern and drop size of black liquor admitted to the furnace, air supply and degree of turbulence and temperature in the different zones. The normal kraft recovery furnace stack gas, upstream from the contact evaporator, will have negligible concentration of malodorous sulfur compounds provided the furnace capacity is not exceeded, sufficient excess oxygen is available and good turbulence and a sufficiently high temperature are maintained in the upper oxidation zone of the furnace (3, 4, 34). Black liquor oxidation apparently has no influence on the quantities of odorous material leaving the furnace. (34). Fine droplets of black liquor spray result in a higher concentration of sulfur dioxide and hydrogen sulfide in the flue gas (3).

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A recent proposal (35) utilises an air contact evaporator. Combustion air is preheated to about  $600^{\circ}$ F by heat exchange with flue gas and subsequently used for evaporating the black liquor (50% solids) to the desired final concentration (63-65% solids). The air leaving the contact evaporator at 260-300°F. is conveyed to the primary and secondary air admission ports of the recovery furnace. Malodorous sulfur compounds released during the evaporation process are destroyed by incineration in the high temperature combustion zones of the furnace.

Feurstein, Thomas, and Brink (36) studied in considerable detail the malodorous products from the combustion of kraft black liquor. The combustion has been characterized by initial endothermic reactions which degrade the organic part of the black liquor into smaller fragments and by the competing exothermic reactions of oxidation and recombination. The latter reactions are responsible for the release of malodorous compounds. These authors are initiating pilot scale work on a two chamber reactor (37). The black liquor charge will be pyrolyzed in the first chamber in the absence of oxygen with proper temperature and residence time to insure production of a simple gase. ous fuel. Combustion will take place in the second chamber with a fuel that burns so hot that it will be virtually impossible for the occurrence of recom-Lination reactions.

Elimination of the direct contact evaporator in recovery boiler design will remove a prime source of odour. Direct contact evaporators are not used in Scandinavian countries (28). The desired final black liquor concentration is achieved by an increased in multiple effect evaporation capacity. The capital cost of this system is high and can be justified only where fuel is expensive. A disproportionately large heat transfer surface is required in the evaporator due to the high viscosity of the black liquor and the low gas temperature necessitates a larger heat exchange surface in the recovery furnace (39,40). The values given in column B, Table 3 represent average sulfur losses reported in the literature for pulp mills practising black liquor oxidation as an integral part of their recovery operations and equipped with some of the odour control methods des-

cribed earlier. A comparison of the values in columns A and B points out the progress made in the control of odorous emissions from a kraft pulp will. Nevertheless, as is common in many instances, these odour control techniques have certain limitations which are outlined in the next section.

#### Limitations of Current Odour Control Methods:

In a Kamyr digester, the steam from the first flash tank containing malodorous sulfur compounds is used for chip presteaming. This recirculation may build up the concentration of nonionised odorous species making it difficult to control their escape. In batch digesters, the desired liquor to wood ratio is usually obtained by the addition of weak black liquor : sodium hydrosulfide, methyl mercaptide and methoxyl content of the black liquor will contribute to the odour forming reactions (2 and 3), (41).

Odourous constituents in the air effluents from the filter washer vent, black liquor oxidation tower and the direct contact evaporator are present in such low concentration that further treatment by condensation, absorption or combustion is not feasible. Maintaining a higher pH level in the black liquor, entering the filter washer and oxidation units will minimise hydrogen sulfide and methyl mercaptan losses. However, the non-ionized odorous compounds the methyl sulfide and terpenes will be stripped out by carrier gas at any pH level of the black liquor. Possible toxic nature of the chlorinated compounds could limit the technique of chlorinating effluent streams. 'The flash tanks and multiple effect evaporators remove the volatile sulfur compounds continuously as the black liquor circulates through the system.

Several authors (30, 42, 44) have shown the partial reversibility of the hydrosulfide oxidation reaction, which may occur in storage tanks and multiple effect evaporators and partially nullify the benefits of oxidation. A second oxidation step, utilizing the strong black liquor storage tank converted to an air sparge tank, is reported to be effective in controlling the emission of hydrogen sulfide from the direct contact evaporator (43).

Douglas et. al. (44) have shown that methyl mercaptan may be formed in

multiple effect evaporators, the amount depending upon the degree of oxidation, black liquor concentration and temperature.

Elemental sulfur formation has been reported (30, 42) with black liquor oxidation at temperatures below 65°C; sulfur goes into solution in the alkaline black liquor according to the reaction.

$$S + 6 NaOH = 2 Na_2S + Na_2S_2O_3 + 3 H_2O$$
 (15)

In full scale operation of commercial direct contact evaporators, Murray and Rayner (33) found the emission of hydrogen sulfide to exceed the predictions of pilot scale experiments. This difference was attributed to the fact that in a commercial evaporator, pH of the black liquor in the evaporator reservoir is always higher than the pH of the liquor on the gas-liquid contacting surface.

The control of sulfur losses and odour from the recovery furnace may be achieved by a proper balance of the furnace operating conditions mentioned earlier (34,45). The emission of hydrogen sulfide from overloaded units increases exponentially with increasing overload since the supply of secondary air must be curtailed to avoid overheating of the furnace (13,46). Maintaining 2.5-3.5 per cent excess oxygen level at the economizer outlet minimize the release of odorous compounds (3,4). It may be concluded from the above discussion that sulfur losses from many effluent positions and the extremely low odour threshold levels (47) of malodorous compounds have continuously posed a difficult problem for the pulp incustry.

#### **Conclusions :**

The problem of recycling odorous sulfur compounds in a continuous digester may be minimized by an application of the SEKOR process (28, 29) to the black liquor extraction flow. Steam stripping black liquor before the flash vessels may isolate methyl sulfides and terpene compounds.

Black liquor oxidation, a major step in current odour control practice is not very efficient unless accompanied by auxiliary equipment for the secondary oxidation of strong black liquor and for controlling emissions from the recovery furnace. Further oxidation or reduction of sodium thiosulfate leads to

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additional sulfur losses, reactions (9 and 12). Black liquor oxidation also affects the heat balance of the mill. The black liquor oxidation reaction is exothermic while the reduction reactions in the recovery furnace are endothermic in nature; the exothermic heat of reaction is partially utilized in evaporating black liquor in the oxidation tower.

The main source of odour in the recovery furnace is the reducing zone; this reductive feature is imperative for recovering sulfur as sodium sulfide from the salt cake added as a make up chemical and sodium thiosulfate resulting from oxidation reactions in the black liquor. A major proportion of the capital and operating costs of black liquor combustion may be attributed to this factor. A considerable amount of savings in the costs of recovery furnace operation and odour control equipment would result if the reduction of sulfur compounds in the recovery furnace can be eliminated.

The removal of sulfur compound from weak black liquor could considerably reduce the emission of odorous constituents from the evaporators and the recovery furnace. Investigations are in progress for the recovery of sodium sulfide directly from weak black liquor by-passing the evaporators and recovery furnace (48, 49).

TABLE 1

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#### REACTIONS

- 1. Carbohydrates :-Hydrolysis of acetyl groups and glucosidic bonds Peeling and stopping reactions
- 2. Lignin-degradation and dissolution
- 3. Resin and fatty acids saponification

### **BLACK LIQUOR CONSTITUENTS**

Inorganics :	NaOH, NaHS, Na <sub>2</sub> CO <sub>3</sub>		
	$Na_2S_2O_3$ , $Na_2SO_4$ , etc.		
Organics :	Carbohydrates		
	Lignin		
	Soaps		
pH :	10.5-13.0		
% solids :	12.0-24.0		
Calorific value	e : 3000-4000 Kcal/kg.		

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#### TABLE 2

## SOURCES OF ODOUR FORMATION AND SULFUR LOSS IN A KRAFT PULP MILL

Source	Major odour forming reactions		
Digester	$H_2S + 2 OH^- = HS^- + OH^- + H_2O$		
	= S <sup></sup> + 2H <sub>2</sub> O	(1)	
	$Lignin-OCH_3 + HS^- + OH^- = Lignin-O^- + CH_S^- + HO$	(2)	
	Lignin-OCH. $\pm$ CH S= $\pm$ Lignin O== $\pm$	(4)	
	$= 12 \operatorname{gmm} - 0 \operatorname{m}_3 + 0 \operatorname{m}_3 = 12 \operatorname{gmm} - 0 + \frac{1}{2} \operatorname{gmm} - 0$	(3)	
	$CH_2SH + OH^- = CH_2S^- + H_2O$	(0)	
	$4 \text{ CH}_{-3}$ $+ 0 + 2 \text{ H}_{-3}$ $+ 120$	(4)	
	$1 \text{ ongo } + 0_2 + 2 \text{ n}_2 \text{ or } = 2 (\text{cn}_3)_2 \text{ s}_2$	(5)	
	$2 \text{ CH}_{2}\text{S}^{-} + \text{H}_{2}\text{O} = (\text{CH}_{2})_{2} \text{S} + \text{H}_{2}\text{S}^{-} + \text{OH}_{2}$	(C) (G)	
Blow tank	Reactions $(1,4,5,6)$	(0)	
Pulp Washer vent	Reactions (1,4,5,6)		
Multiple effect evaporator	Reactions (1,4 to 6); Reactions (2,3) to a small extent		
Direct contact evaporator	$HS^{-} + CO_2 + H_2O = HCO_3 + H_2S$	(7)	
	$CH_3S^- + CO_2 + H_2O = HCO_3^{} + CH_3SH$	(8)	
Recovery	$Na_2SO_4 + 2 C = Na_2S + 2 CO_2$	(9)	
furnace	$ 2 \operatorname{Na}_{2} \operatorname{S}_{2} \operatorname{O}_{3} + 3 \operatorname{C} = 2 \operatorname{Na}_{2} \operatorname{S} + 2 \operatorname{S} + 3 \operatorname{CO}_{2} $ (1) $ \operatorname{S}^{} + \operatorname{CO}_{2} + \operatorname{H}_{2} \operatorname{O} = $	10)	
	$CO_3^{}$ + H <sub>2</sub> S (1)	11)	
	$S + O_2 = SO_2 \tag{2}$	12)	
	Release of $H_2S$ , $SO_2$ and organic sulfides depend upon the degree of control of furnace atmosphere.		
Smelt dissolver	Reaction (1)		
Lime Kiln	Reactions (11, 12) Release of $H_2S$ depends upon the excess oxygen level.		

#### TABLE 3

# EMISSIONS OF SULFUR COMPOUNDS FROM A KRAFT PULP MILL\*

(9,13,15-22)				
Source	A	В		
Digester relief	1.0	0205		
Blow gas	1.0	0.2-0.0		
Washer vent	0.25	0.2		
Oxidation tower	0.20	0.0-0.1		
Evaporator	4-14	0.1		
Contact evaporator and recovery furnace	5-12	4-7		
Dissolver	0 1-0 2	0.05.0.1		
Lime kiln	0.2-0.5	0.05-0.1		

\* expressed as kg. of sulfur per ton of air dry pulp.

A Pulp mill with no controls.

B Pulp mill with odour control equipment.

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