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Kraft Odour—Causes and Control

VENKOBA RAO G*

ABSTRACT

The main sources of kraft mill odours and atmospheric sulphur losses are reviewed. Eactors affecting the formation and release of odourous compounds at different stages of pulping and chemical recovery are discussed. The methods available for controlling the emissions of these gases from various sections are described. Special emphasis is given on the control of odour gases from the digester side.

Atmospheric pollution by kraft mills is by no means a new problem, but it is becoming increasingly more important. These atmospheric emissions include, in addition to gaseous or odourous components, sulfurdioxide and particulate emissions from recovery furnaces and power plants. The odour problem assumes greater importance than the problems associated with other emissions from the esthetic point of view it has become a necessity to reduce the level of odourous emissions. Especially from the kraft mills located in densely populated areas are faced with relatively undiminished levels of public annoyance and complaints. Such response, can be traced to various factors including the non-linear relationship between the odour intensity and odourant concentrations, varying dispersive capacity for different emission sources, the altered expectations of the population and the ever changing Government rules and regulations. Eventually in such cases, one is driven to the realisation that effective kraft mill odour control not only requires a very high degree of emission reduction, but also virtually complete reliability of the control measures while the nuisance caused by kraft odour is not to be underestimated, the odour components differ favourably from many other air pollutants in other respects.

The main odour causing components are found to be reduced sulfur components like hydrogen sulphide, methyl mercaptan and dimethyl sulfide.

Although high concentrations of hydrogen sulphide are lethal studies carried out so far have failed to indicate that the odour components increase the frequency of respiratory illness^{1'2}. Further kraft odour emissions have neither been associated with adverse affects on plant growth, nor with haze formation³.

The odour threshold levels for all major reduced sulphur compounds from the pulping process is unfortunately low and is of the order of magnitude of 1 ppb (1 microgram/M³). In Table—1 are presented odour threshold levels of these compounds⁴.

Some of the important physical characterics of these four compounds are given in table-2

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12	٦D	LD	

Odour threshold levels of reduced sulphur compounds in kraft pulp mill emissions. Ref : 4

Compound	Odour threshold p p.b.	
il,S	0.7 - 4.7	
il₂S CH₃SH	0.8 - 2.1	
CH ₃ S. CH ₃	1.0 - 3.9	
CH ₃ S S.CH ₃	5.0 - 6.0	

	Physical pr	roperties of kraft mil	l odour compounds	an a
ta a Compound veisions 7.25 las <u>amits stanop</u> otreinfitual a	Boiling point °C K	teat of cumbustion L. Cal/mole	Explosive concentration range in air %	Dissociation constant of ag. solution at 100
nten Hasin an training an Asin a	—61 .8	124	4.3 — 45.5	$K_1 = 2.1 \times 10^{-7}$
CH ₃ SH CH ₃ S-CH ₃ CH S SCH	+ 58 + 338	299 457	2.2 - 9.2 3 9 - 21.8	$K_2 < 10^{-14}$ $K = 4.3 \times 10^{-11}$ Not dissociated
CH ₃ S. SCH ₃	+ 118	530	Not determined	Not dissociated.

TABLE - 2

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From the perusal of the data, it is evident only H_2S and CH_3SH can dissociate in aqueous solution according to equations (1) & (2)

$$H_{3}S \stackrel{K_{1}}{\rightleftharpoons} HS^{-} + H^{+} \stackrel{K_{2}}{\rightleftharpoons} S^{-} + H^{+} \dots (1)$$

$$CH_{3}SH \stackrel{K_{1}}{\nleftrightarrow} CH_{3}S^{-} + H^{+} \dots (2)$$

The dissociation of both compounds exerts an important effect on vapour liquid equilibria, as well as reactivity, the anion representing the commonly reactivespecies. Methyl sulphide and and dimethyl disulphide do not ionise and generally less reactive than the former two compounds.

SOURCES OF ODOUROUS GASES IN THE MILL a) Pulping Section :--

The formation of mercaptan and methyl sulphide under kraft pulping conditions have been carefully studied^{5'6}, one of the important reaction of sulphide ion and hydro sulphide ions (formed by the ionisation of sodium sulphide) with the lignin macro molecule is the demethylation of the methoxyl groups in lignin⁷. This demethylation reaction results in the formation of methyl mercaptan and dimethyl sulphide which can be represented by the following equation.

 $HS^- + CH_3O - Lignin \rightarrow CH_3SH + O - lignin (3)^4$

The mercaptan formed ionizes to mercaphide which in turn reacts with lignin methoxyls forming methyl sulphide. $CH_3S+CH_3O-Lignin \rightarrow (CH_3)_2S+O^--Lignin...(4)$

Since mercaptan is an intermediate in the formation of methyl sulphide, the ratio of former to latter decreases continuously, but it remains wellover one. The mercaptan to methyl sulphide ratio depends also on the pH of the liquor, being higher at pH 11 than 12. In a typical cook approximately 1.2 lb of mercaptan and 0.6 lb of methyl sulphide are formed from ton of dry soft wood².

From hard wood species it is found that more than twice the amont of these two compounds are formed in comparison with soft woods

The activation energies of 'mercaptan and methyl sulphide formation have been found to be 22.5 and 19.0 K Cal/mole respectively⁶ and are considerably lower that of delignification, which has been found to be 31 K. Call/mole ⁸ These demethylation reactions of methoxyl groups by sodium sulphide are examples of straightforward SN₂ displacements being first order with respect to necleophile i.e. hydro sulphide ion or sulphide ion and to the methoxyl concentration⁹.

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Thus these organic sulfer compounds viz. methyl mercaptan and methyl sulphide form part of the digester relief gases and digester blow gases.

b) Other points of release of odourous gases in pulping section :

In addition to digester relief and blow gases, in the pulping section, the other points of odour emanation are washer hood and seal tank vents, where because of low volatality some quantity of methyl mercaptan and methyl sulphide find their way.

c) Release of odours in the Recovery Section (i) In multiple effect evaporators

From their study of evaporation of dilute unoxidised black liquor from hardwoods, Donglass and coworkers¹⁰, found that the emissions from this section contained methyl mercaptan with concentrations exceeding the o lour threshold limits. They found that the dilute black liquor carried into multiple effect evaporators appreciable amount of methyl mercaptan (0.219 gms/gallon or 438 gms/ ton of pulp), but very little demehyl sulphide.

This mercaptan was evidently stroped from the liquor during the evaporation process. A partially axidised (68%) hardwood black liquor carried into evaporator much less residual methyl mercaptan (0.047 gms/gallon or 103 gms/ton of pulp) and during the evaporation of this less of methyl mercaptans was formed.

ii) In Recovery furnace and direct contact evaporator forming part of the furnace

It has been found that the majority or possibly all the unpleasant odour emitted from a kraft recovery boiler is due to the direct contact evaporator $(DCE)^{11^{13}}$. Murray and Raynor¹⁴ have shown that the emission of hydrogen sulphide at the DCE is directly proportional to the sodium sulphide content of the black liquor. However, it is to be added here that the DCE removes 20-30% of the dust and about 75% of the sulphurdioxide in the recovery furnaze flue gas.

Clement and Elliots¹¹ have also found that the boiler operation within sufficient combustion air results in emission of odourous gases from recovery furnace.

In Table—3 are given typical reduced sulphur concentrations from kraft mill sources and in Table-4 typical reduced sulphur gas emission rates from kraft mill sources

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TABLE-3

 Concentration (ppm by volume)

 Emission source
 H₂S
 CH₃SH
 CH₃SCH₃
 CH₃S

 Batch Digesters
 H₂S
 CH₃SH
 CH₃SCH₃
 CH₃S

lypical	Reduced	Sulphur	gas	concentrations fro	om kra	aft pulp	mill sources	
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CH,SSCH, **Batch Digesters** Blow Gases 0-1,000 0-10,000 100-45,000 10-10,000 Relief gases 0-2,000 10- 5,000 100-60,000 100-60,000 **Continuous Digester** 10- 300 500-10,000 1,500-7,500 500- 3,000 Washer hood vent 0-5 0-5 0-15 0---3 Washer seal tank 0- 2 10-50 10-700 1-150 Evaporator hotwell 600-9,000 300-3,000 500- 5,000 500- 6,000 Recovery furnace (after DCE) 0 - 1,5000- 200 0---100 2---95 Smelt dissolving tank 0-75 0---2 0-4 0---3

From: Ref. 15

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TABLE-4	
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Typical reduced sulphur gas emission rates from kraft pulp mill sources

	Emission rate Kg. sulphur per MT of AD pulp				
Emission source	H ₂ S	CH ₃ SH	CH ₃ SCH ₃	CH₃SSCH	
Batch Digester					
Blow gases	0-0.1	0-1.0	0-2.5	0-1.0	
Relief gases	0-0.05	0—0.3	0.05-0.8	0.05-1.0	
Digestes continuous	0—0.1	0.5-1.0	0.05-0.5	0.05 - 0.4	
Washer hood vent	00.1	0.05-1.0	0.05-0.5	0.05-0.4	
Washer seal tank	0—0.01	0—0.01	00.05	0-0.03	
Evaporator hotwell	0.05—1 5	0.05-0.8	0.05-1.0	0 05-1 0	
Recovery furnace (after DCE)	025	0—2	0—1	0-0.3	
Smelt dissolving Tank	0—1	00 8	0-0 5	0-0.3	

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From : Ref 15

CONTROL OF KRAFT ODOUR

Having discussed the causes of odour from kraft pulp mills, the attention is now turned to the review of various methodologies that have been adopted for control of the emission of malodourous gases.

Digester Relief and blow gases treatment

A perusal of the published literature on control of the emission of malodourous gases from digester relief and blow gases show that generally two kinds of strategies are adopted.

a) Scrubbing or treating these gases with liquor containing alkali or with chlorine.

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b) Collection and burning of these gases either in a separate incenerator or in the rotary lime kiln.

Odour control through scrubbing of digester gases :

One of the earliest references of an odour control system based on scrubbing digester gases is provided by the work of Thomas and coworkers, who have described the air pollution abatement at S.D. Warren's Kraft Pulp mill at Westrook¹⁶. In this mill a system was developed in which digester blow gases were passed through primary and secondary deodourising scrubbers, utilising the chlorine stage effluent. As a back up weak hypo bleach liquor was also added to the secondary

unit. However, the non condensible gases (which are responsible for odour) from the relief gases were burnt in the rotary lime kiln.

Another well know odour control system based on scrubbing is the TLT (Troback \rightarrow Lenz \rightarrow Tirado) system which was in operation in the Pena Pobre Mill in the Mexico city¹⁷,¹.

This TLT system involves the condensation of the digester relief gases to remove condensibles and followed by oxidation of the non-condensible first with air and then with gaseous chlorine (Fig.)

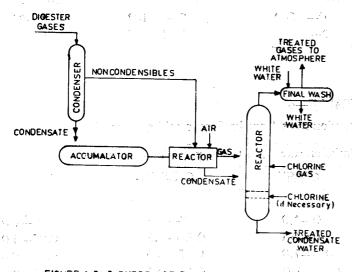


FIGURE 1 TLT SYSTEM OF DIGESTER GAS TREATMENT

This system was in operation at the Loret Y Dena Porbre Kraft mill at Mexico for a considerable period of time and ten years experience in odour control at this mill has been described by Tirado and Gonsalez¹⁸.

In this mill gaseous chlorine was used to have efficient oxidation. A cold chlorination was preferred in order to minimise the explosion risks and also to have a better control on the reaction products. Their experience. further showed that if the temperature and/ or the quantity of chlorine is high enough the mixture burned and exploded violently.

The possibility of using advantageously the residual chlorine in the bleach liquor was also explored by them. It was found by them, because of the low residual chlorine in these effluents it was impractical or impossible to transfer 100% of the chlorine, even with acid effluents i.e. chlorination effluents from which because of its low PH it is more easy to vaporise chlorine. Based on the TLT system, a 97% reduction in the odour index has been claimed.

On account of the practical difficulties in using elemental chlorine, the mill carried out a trial for the absorption of the mercaptan and the other sulphur compounds in white liquor, to make a special cooking liquor. Thus a 'mercaptan tower' was put up by them. Commercial trials were run during a control period of 37 days maintaining a conventional sulfidity of 23 0% and during two independent test periods of 39 and 27 days, during which odourous gases from digesters were scrubbed in white liquor. A 67% reduction in the odour index was observed and the strength charasterstics of the pulp also showed some improvement.

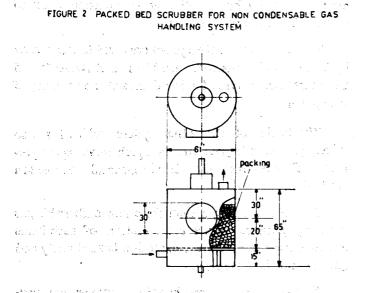
British Columbia Research Council has advocated the disposal of non.condensibles by passing them through co-current oxidation tower¹⁹. The method has been adopted with modifications elsewhere such as Morrum Mill²⁰.

Ruus²¹ has pointed out the high chlorine demand for the oxidation of the sulphur compounds (from 2 to 9 kgs of chlorine per kg of the compound) and the consequent high costs. It would also be pertinent at this point to refer to the work of Kappa and Adams²² who have studied the gas phase chlorination of kraft pulp mill gases, came to the conclusion that gas-phase chlorination of gases from kraft pulp mills (under safe operating conditions) appears to be of limited value as a means of odour reduction. The chlorine oxidised the methyl mercaptan in the gases to dimethyl disulphide but did not change the concentration of hydrogen sulphide or dimethyl sulphide.

Taking congnizance of the fact that both hydrogen sulphide and methyl mercaptan are weak acids, a process was developed by E.T. Venemark²³, which was assigned to Mooch Domsgo A.B., Sweden. This comprises atomising an aqueous alkali solution into a stream of the vapours containing the volatile acidic compounds, like hydrogen sulphide and methyl mercaptan in a volume ratio of alkali solution to vapour of about 0 15 to 1 liter per cubic meter at a rate of at least 5 meters per second at an alkali solution load of at least 250 liters per minute per square meter to form a final alkali solution having a pH of at least 8.

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There is also another reference in literatur i.e. alkaline liquor scrubbers of these digester gases²⁴. It describes the design of a packed bed scrubber for non-condensible gases handling system. The liquied solution employed for scrubbing is either caustic soda or white liquor for return to the chemical make up system or water for subsequent discharge into effluents. A typical scrubber system is given in Fig 2



A very recent report²⁶ describes a stand by wet scrubbing system for controlling the odours from digester relief and blow gases. This system has been installed and operated at Domtar's Cornwall mill at Canada. Sodium hypoch'orite has been used as the scrubbing media. To prevent catalysed decomposition of hypo by metals such as iron, nickel, manganese non-metallic construction maetrials were used throughout the system. The scrubber structure, and the various valve and pump components in contact with the scrubbing liquor were made from glass fibre reinforced plastic, liquor supply and recirculation lines were made up of PVC.

There has also been an interesting report by Teder and Tosmund²⁶ who have shown that methyl mercaptan can be removed with polysulphide or poly theonate.

Odour control by burning of non-condensible malodourous gases

As it is evident from the foregoing description that for odour control scrubbing of non-condensible gases is feasible but not very effective.

The best way was found to be the burning the odorous gases in an incinerator in a lime kiln.

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The digester and evaporator non condensible gases have relatively low volume flow rate and high malodourous sulphur compound concentrations. The gas flow rates for these individual process streams are subject to wide variations among the individual mills, depending on the production rate, process operating variables and degree of condensation in heat recovery systems

The design and operation of system for thermal oxidation of the noncondensible gases (i.e.) combustion of these gases requires measures to prevent explosions. Therefore, a flow equalisation system becomes a necessity before the gases are taken to combustion. Four important factors which must be given utmost consideration while designing the combustion system are —

- 1. Variations in gas flow rate
- 2. Passage of entrained moisture droplets in the burning device
- 3. Flamability limits of odour causing compounds
- 4. Flame propogation speeds as compared to gas flow velocities in piping.

The flammability limits in air for compounds Present in kraft noncondensable gas stream are given in Table—5

TABLE-	
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Flammability limits of compounds present in noncondensable gas from digesters

Compound	Flammability limits concen- tration percentage by volume	
	Lower	Upper
H,S	4.3	45.0
CH ₃ SH	3.9	21.8
H ₂ S CH ₃ SH CH ₃ S CH ₃	2.2	19.7
		· · · · · · · · · · · · · · · · · · ·

From : Ref. 27

Therefore these gases will have to be diluted with air before inceneration to avoid the risk of explosion. The dilution requirements are given in Table—6

TABLE-6

Dilution requirements with air for digester noncondensable gas streams

Gas stream	Volume of dilution reqd. air/gas ratio
Relief only	50/1
Relief and blow	20/1
From : Ref. 28	

The gas velocities in the noncondensable gas piping and the primary air inlet must be greater than any flame propagation speed to prevent damages to process units from possible explosions. Maintenance gas velocities of atleast lmet./sec. (3 feet/sec) at all times and the use of flame arrester degices in the non-condensable gas line should minimise the danger of explosions from excessive flame speeds.

The flame propagation speeds for air-mercaptan mixtures are given in Table-7

TABLE-7

Flame propagation speeds for Air-mercaptan mixtures

Mercaptan	Flame velocity		
concentrations % by volume	Met/sec	ft/sec	
18.0	0.55	1.8	
22.8	0.46	1.5	
23.1	0.40	1.3	
23.7	0.37	1.2	
25.5	0.18	06	
25.7	0.15	0.5	

From Ref. 15

Vapour condensation

The primary purpose of condensers in the digester and evaporator gas handling systems is the heat recovery from the gas stream by water vapour condensation.

The condenser system also removes a portion of the organic vapours such as terpenes which can cause explosion.

Flow equalisation

Two major devices are employed for non-condensable flow equalisation are —

- 1. Vapor sphere
- 2. Floating cover gas holder.

In essence, the vaporsphere is a spherical device with flexible fabric diaphragm attached around the epicentre of the sphere and the diaphragm consists of an Mylar film sandwitched between two layers of cotton convas. The system also contains a counter weigh connected to the diaphragm which moves up and down as the gas flow in and out of the vapor sphere. The diaphragm is weighted to provide a slight positive pressure on the system at all times. Automatic flow controls on the system are used to prevent the damage of the diaphragm and excessive air leakage. A pressure and vaccum relief system prevents damage to the diaphragm. The condenser section and all associated piping to the vaporsphere must be sealed to prevent the possibily of air-leakage and potential explosive mixtures from forming Also the inlet and outlet gas streams must be vented separately for the full flow equalisation effect to obtained from the vaporsphere.

The full details of the system are given in the review by Blosser and Copper²⁹. De Haas and Harsen³⁰ and Coleman²⁸ also provide further details of the working of the system.

The details of the other system which is not so widely prevalent is given the comprehensive report prepared Ekono inc for Environmental Protection Agency¹⁵.

Design of a piping system for a noncondensable gas handling system requires a consideration of such items as materials of construction, explosion hazard safety and gas flow pressure drops.

Non-condensa le gas handling systems are normally constructed in mild steel, but 304 and 316 SS has been used in some applications to inhibit corrosion constructing noncondensable gas piping systing to obtain a minimum velocity of 1 meter/sec (3 ft /sec) is usually necessary to minimise the likelyhood of flame propagation through the pipe. High velocities also result in increased pressure drops through the piping, particularly for lines longer than 30 meters (100 feet). An auxillary fan or a large diameter pipe should be installed to minimise the pressure drop across the system and to allow use of the inlet draft of combustion air systems.

Safety Considerations:

1. Condensate traps to remove water are placed at low points in the piping system at intervals of approximately 15 meters and just upstream of the air inlet.

2. Flame arrestors of the leaf or grid type are commonly added to the non condensable gas line to prevent the passage of any flame fronts to the process units. It is normally to the gas line immediately upstream at the point of introduction to the primary air ducts.

- 3. Two features are added to provide emergency venting of excess gas pressure during possible explosions. Rupture discs are added to the gas lines at approximately 30 meter intervals. These devices have diaphragm discs set to explode at certain bursting pressures.
- 4 To cope up with power failures an emergency vent release is normally placed in the gas line connected to the flame out control for the combustion device,
 - Ayers etal³¹ have described now these malodourous gases have been collected and incenerated in the Mead Paper Mill at Escanaba at Michigan, U.S A.
 - The application of retrofit kraft odour control technology in S.D. Warren & Co, Michigan, where the malodourous gases were collected and burnt in coalfired boilers has been described by Porrit³².

The experiences in the running of the plant for the thermal oxidation of kraft pulping emissions in the Ticonderoga Mill of International Paper Company have been narrated by Connery³⁸.

Control of odour emnating from Recovery Section :

In the earlier part of the review it had been pointed out that when dilute weak black liquor carried into the recovery system significant quantities of dissolved methyl mercaptan which was stripped from that liquor during evaporation. Further, the residual sodium sulphide found in the liquor gave rise to the formation of hydrogen sulphide when it came into contact with carbondioxide of the flue gas in the direct contact evaporator of the recovery furnace.

Thus any process which would convert these two compounds should result in reduction of the evolution of these gases in recovery section.

The black liquor oxidation represents one of the most successful strategies that have been adopted for stabilising such labile sulphur compounds in the black liquor. This in essense, involves, the oxidation of sodium sulphide in the black liquor to sodium thiosulphate, methyl mercaptan into less volatile dimethyl disulphide.

A lot of literature has been published on black liquor oxidation, and it would not be possible to discuss IPPTA Vol 24 No. 3 (Suppl.) Sopt. 1987 all the important aspects of this most useful strategy for odour control in Recovery Section in this review.

Comprehensive reviews on black liquor oxidation both weak black liquor oxidation as well as heavy or concentrated black liquor oxidation have been prepared by Blosser and Cooper³⁴,³⁵.

Normally the concentrated black liquor oxidation is suggested for mills using highly resinous wood because of the weak black liquor of these raw-materials will result in excessive foaming. In heavy black liquor oxidation 3 to 5 times the stoichiometric amount of air is provided for oxidation; retention time of 120 minutes to 150 minutes is given.

However, there are also certain inherent drawbacks of the black liquor oxidation system for controlling odour which are as follows :

- a) Methyl sulphide remain volatile and odourous.
- b) There is a loss of heating value from these oxidations and from the heating of air that comes for oxidation.
- c) The effluent air poses problems of foaming and difficulty with odours of volatile sulphur components it contains.
- d) Sulphur or polysulphide may form through the partial oxidation, later reverting to hydrosulphide ion.

Attempts have been made to overcome the problems of foaming and malodourous exit air by using pure oxygen³⁶. Oxygen consumption was high resulting in bad economies, and further the pulp strength was reduced when oxygen was added prior to blowing.

An existing mill has these options, but in a new mill certain steps can be taken in the initial stages itself so that these problems are eliminated by suitable design. The challanges have been taken up and machinery manufacturers, both Babcock and Wilcox as well Combustion Engineering have comeout designs of low odour recovery boilers. Basically in the revised design the direct contact evaporator, cyclone evaporator in the case of Babcock and Wilcox design or cascade evaporator in the case of the Combustion Engineering design is eliminated by replacing the DCE with additional multiple effect evaporator capacity.

The operating experiences with a "low odour" combustion engineering recovery furnace is described by LO Cicero and Sjoeseth³⁷.

Gommi³⁸ has also reviewed a number of low odour systems for kraft mill recovery units based on combustion engineering design.

Clement and Elliot in their paper have given details of 'Low odour' recovery furnace based on Babcock and Wilcox design¹¹.

Essentially these 'Low odour' recovery boilers were designed for unoxidised black liquors. In this connection it would be pertinent to refer to the work of Walther and Amberg^{12,13} who found that if the black liquor feed to cyclone evaporator were oxidised to a sodium sulphide concentration of 0 3 gpl or less then the TRS emissions from the boiler compared favourably to that of TRS emissions from 'low odour' recovery boiler i.e. with cyclone evaporator. Further they found that actually the direct contact cyclone evaporator can remove 75% of the sulphur dioxide emissions and about 15-20% of the particulate matter. Processing of oxidised black liquor through a conventional recovery liquor will therefore, have twofold advantages.

- (a) There is a considerable reduction in the TRS levels in the emissions.
- (b) the DCE which is present does not generate any hydrogen sulphide and at the same time acts as scrubber for sulphurdioxide and particulate matter. The same has been indicated by Murray as well⁴⁰.

A similar strategy has been advocated by Semke⁴¹ also. He suggests that for control of TRS as well as particulates from the recovery boiler fluegases, it would be better to have black liquor oxidation followed by combustion in a recovery boiler having direct contact evaporator. The oxidation reduces the concentration of labile sulphur compounds there by minimising its formation in DCE, while the DCE acts as a scurubber for particulates (about 20–25% is removed) and sulphur dioxide (70–75% is removed). This would bring down the capacity requirements of Electrostatic Precipitator as well as more life due to less corrosion on account of lesser sulphur dioxide concentration in the flue gases.

There have also been references in literature⁴²,⁴³ where the gases coming out of the electrostatic precipitator, attached to the recovery furnace pass through a scrubber, where both particulates as well TRS emissions are reduced. A well known scrubber of that type is Flakt—MoDo Scrubber which is in operation in a number of mills.

Finally to conclude, it is to be stated that this article is only a brief one, where only important major contributions in this important field have been reviewed. To adopt some of these techniques outlined above it is necessary some modifications will have to be made to suit the local requirements, after detailed study of the emission levels from various sources.

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