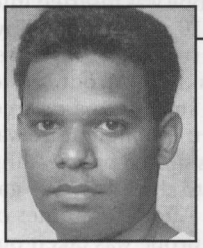
FUME CHEMISTRY, MORPHOLOGY AND DEPOSITION IN A KRAFT RECOVERY BOILER

Fume deposits content and makeup varied across boiler sections

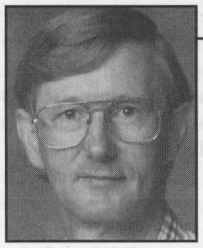
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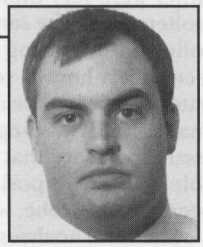
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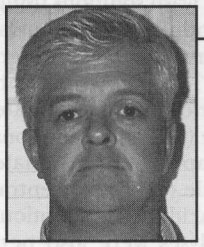
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BECAUSE OF the impact on boiler plugging and thermal performance, the formation of fume in kraft recovery boilers has been the subject of intensive research over the last two decades. Fume originates from sodium (Na) and potassium (K) compounds volatilized from the lower furnace [1,2,3] and from Na and K compounds released during the pyrolysis of black liquor, and char bed burning [3,4,5]. In the upper boiler, fume is thought to deposit on cooler tube surfaces by direct condensation of alkali vapours and/or by thermophoretic transport of materials that have been condensed in-flight [6].

Fume deposits are usually soft and can be removed easily by sootblowers. They start to sinter at about 300°C but at a slow rate [7]. At 500°C, however, fume deposits may sinter in less than an hour, and so become hard and resistant to sootblowing [7]. Problems associated with fume deposition have worsened in recent years, as more and more boilers are operated at higher black liquor solids and with hotter beds.

The chemistry of fume is critically important to boiler fouling, plugging and thermal performance because it greatly affects the deposit melting and sintering behaviour, the dust load recycling within the boiler, as well as SO_2, HCl and particulate emissions. The precipitation process of alkali vapors and the kinetics of chemical reactions leading to fume formation are strongly dependent on temperature. The flue gas temperature in a recovery boiler varies with location, from about 1100°C in the lower furnace, to 900°C at the bullnose elevation, to 600°C at the boiler bank inlet, and to 200°C at the economizer exit. Thus, it is important to understand how fume composition changes with location and to know where the majority of fume forms in the boiler so that the thermal behaviour of fume deposits and the rate of deposition can be better predicted.

The present work was part of the Recovery Boiler Fireside Research Program at the University of Toronto. The project's purpose was to develop a fume chemistry model to predict the composition of fume at various locations in the boiler as a function of black liquor chemistry and boiler operating conditions. The work involved: equilibrium calculations of gas and condensed phase composition and quantity, laboratory studies of vaporization/condensation mechanisms of alkali compounds, and field studies using a specially designed furnace deposition probe to examine the fume composition, morphology and relative deposition rates at various locations in the boiler. This paper discusses the results of a field study conducted at E.B. Eddy Forest Products Ltd., Espanola, ON.

INVESTIGATION

Fume deposition probe: As shown schematically in Fig. 1, the fume deposition probe used in this study consists of three concentric Type 310 stainless steel tubes. The outer tube is used as a protective shell for the deposition surface, which is the outer surface of the centre tube. The deposition surface temperature is controlled by introducing compressed air into the probe at the cold end of the inner tube and circulating the air through the space between the inner and centre tubes. The temperature is measured using four thermocouples evenly

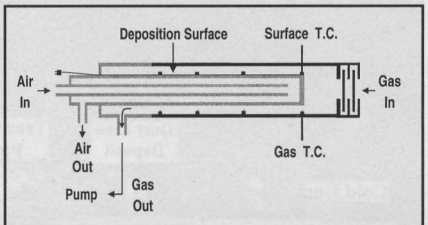


FIG. 1. SCHEMATIC OF THE FUME DEPOSIT PROBE.

spaced along the outside of the centre tube. The gas temperature is recorded by four thermocouples exposed to the gas stream, directly opposite the deposition surface thermocouples. Note that the temperature recorded by the first gas thermocouple is comparable to a suction pyrometer measurement of the gas temperature at the probe location. Flue gas is drawn from the boiler into the probe at a constant flow rate using a vacuum pump. The flue gas had a residence time of approximately one to two seconds in the probe. Carryover particles entrained in the gas are removed by a five-plate impaction screening system attached to the front end of the probe.

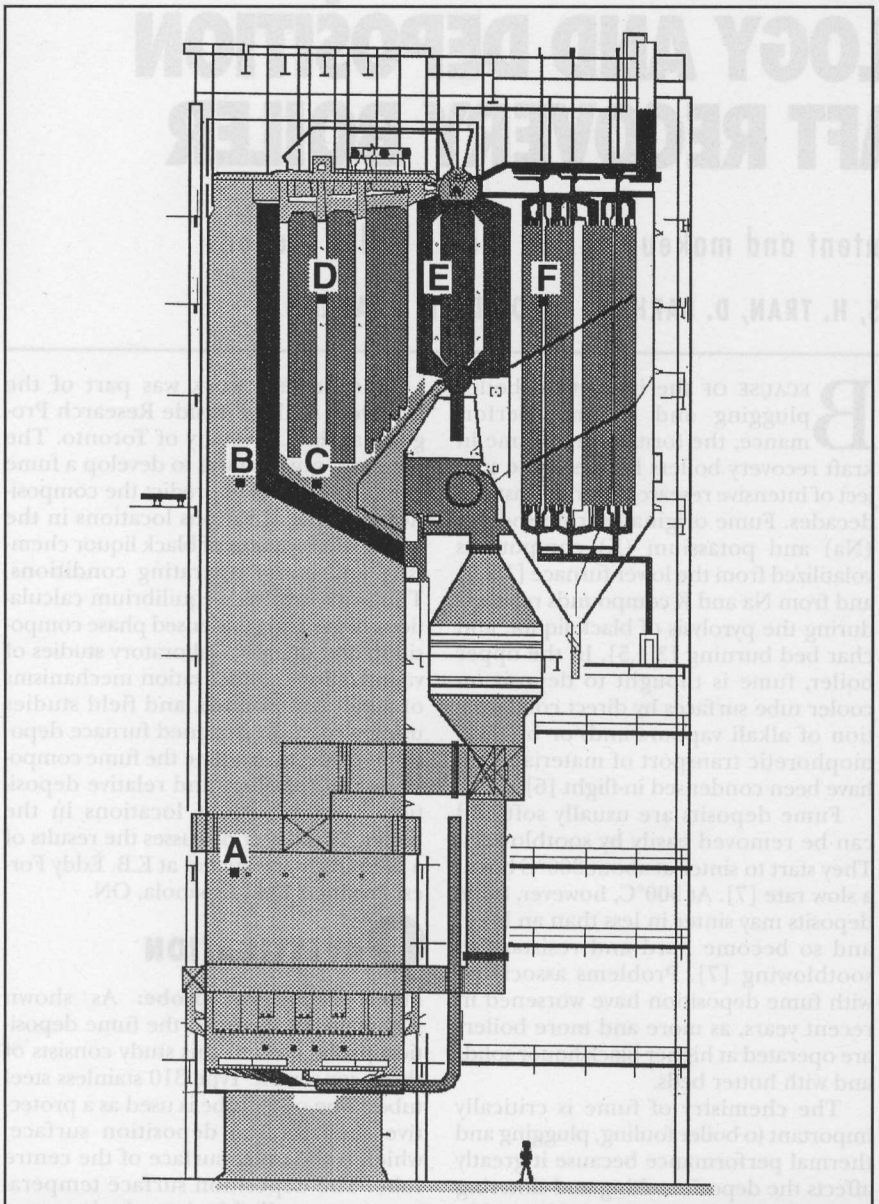


FIG. 2. TEST LOCATIONS IN THE RECOVERY BOILER AT E.B. EDDY FOREST PRODUCTS.

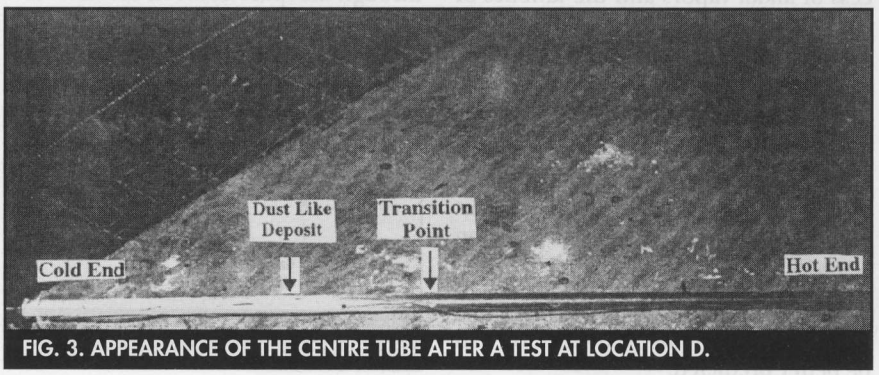


FIG. 3. APPEARANCE OF THE CENTRE TUBE AFTER A TEST AT LOCATION D.

Testing sites and procedures: The recovery boiler at E.B. Eddy Forest Products is a 1982 low odor CE unit designed to burn 1700 tons/day (3.7 million lbs/day) of black liquor dry solids, and to produce 270 tons/hr (600 000 lbs/hr) steam rated at 64 bars (900 psig) and 420°C (790°F). At the time of the investigation, the boiler was operating at 12% above designed capacity, burning 1900 tons/day (94.2 million lbs/day) of black liquor dry solids. The probe was exposed at various locations in the boiler from the lower furnace to the screen tubes near the bullnose, superheater, boiler bank and economizer, Fig. 2. During each test, the probe was inserted into the boiler for 5 to 10 minutes to allow the temperature to reach a steady state. Compressed air was then introduced to cool the deposition surface to a desired temperature. When this temperature was reached, the test was started by activating the vacuum pump to draw the flue gas through the probe. After 10 minutes of gas withdrawal, the probe was taken out of the boiler and the outer tube was removed. The deposition surface was thoroughly examined, photographed, and deposit samples were collected.

Figure 3 shows the appearance of deposits on the outer surface of the centre tube of the probe exposed location D. The back half (cold end) of the probe was covered with a layer of white, dust like deposits, whereas the front half (hot end) had a transparent or solidified molten deposit. The method of deposit sampling differed depending on the type of deposits observed on the probe. The dust-like deposits found on the back half of the probe were carefully scraped off with a spatula, collected and weighed. The scraping procedure, however, was impossible for deposits on the front half, which either were very thin or solidified from a molten state. The sample in this case was collected by rinsing the deposition surface with a known volume of deionized water and saving the resulting solution for analysis. This washing procedure was necessary to ensure that visually unrecognizable molten deposits that might have been on the probe, were also included.

Chemical analyses were performed on all solid deposits and wash solutions using various methods. Sodium (Na^+), potassium (K^+) concentrations were determined by means of Atomic Absorption Spectrometry, chloride (Cl^-), sulphate (SO_4^{2-}) and sulphide (S^{2-}) concentrations by Ion Chromatography, and carbonate ($C0_3^{2-}$) and hydroxide (OH^-) concentrations by Automatic Acidimetric Titration. Morphological analyses were also per-

TABLE I. COMPOSITION OF FUME DEPOSITS IN THE LOWER FURNACE (LOCATION A)

Distance from probe hot end (cm)	Na (wt%)	K (wt%)	Cl (wt%)	SO_4 (wt%)	CO_3 (wt%)	OH (wt%)	$[Cl_2+SO_4+CO_3+(OH)_2]/[Na_2+K_2]$ molar ratio
				Run #1			
0-20	36.9	8.1	2.2	20.6	23.1	9.1	0.99
20-40	36.4	5.1	3.7	26.4	24.3	4.1	1.00
40-60	35.6	7.8	3.4	26.0	19.7	7.5	1.00
60-80	35.6	7.8	3.4	26.0	19.8	7.4	0.99
				Run #2			
0.20	39.1	5.9	2.3	19.3	23.1	10.3	0.99
20-40	37.1	6.4	1.7	24.9	21.6	8.3	1.00
40-60	35.4	8.1	2.9	26.3	20.0	7.3	1.00
60-80	35.7	6.9	1.7	26.6	22.8	6.3	1.00
Average	36.5	7.0	2.7	24.5	21.8	7.5	1.00

TABLE II. COMPOSITION FO FUME COLLECTED AT SCREEN TUBE INLET (LOCATION B)

Distance from probe hot end (cm)	Na (wt%)	K (wt%)	Cl (wt%)	SO_4 (wt%)	CO_3 (wt%)	$[Cl_2+SO_4+CO_3]/[Na_2+K_2]$ molar ratio
0-20	27.7	7.5	1.2	59.9	3.7	1.00
20-40	27.9	7.1	0.8	60.3	3.9	1.01
40-60	29.0	5.5	1.3	61.3	2.9	1.01
60-80	29.3	4.8	0.7	62.4	2.8	1.01
Average	28.5	6.2	1.0	61.0	3.3	1.01

TABLE III. COMPOSITION OF FUME DEPOSITS IN THE UPPER FURNACE

Test	Na (wt%)	K (wt%)	Cl (wt%)	SO_4 (wt%)	CO_3 (wt%)	$[Cl_2+SO_4+CO_3]/[Na_2+K_2]$ molar ratio
B	29.6 ± 1.5	4.8 ± 1.0	1.8 ± 0.7	60.1 ± 0.8	3.7 ± 0.4	1.01
C	28.3 ± 0.6	6.4 ± 1.2	4.8 ± 2.2	57.7 ± 2.9	2.8 ± 1.1	1.02
D	28.9 ± 1.0	7.4 ± 2.1	0.9 ± 0.5	56.2 ± 1.0	6.6 ± 1.5	0.99
E	29.3 ± 0.5	6.8 ± 2.1	2.2 ± 0.2	54.3 ± 1.8	7.4 ± 1.6	0.99
F	31.1 ± 0.6	6.5 ± 0.5	1.4 ± 0.1	44.2 ± 0.1	16.8 ± 1.6	1.00
Precipitator Dust	29.1 ± 0.6	6.8 ± 0.6	1.4 ± 0.1	56.7 ± 0.5	6.0 ± 0.5	0.99

formed on selected solid samples using Scanning Electron Microscopy (SEM).

FUME CHEMISTRY

Lower furnace: Table I summarizes the chemical analysis results of fume deposits collected along the probe length at the black liquor gun elevation, below the secondary air level, Fig. 2, Location A. Two runs were performed at this location. No significant variation in composition along the probe length was found. The deposit, on average, contains 36.5 wt% Na^+, 7.0 wt% K^+, 21.8 wt% CO_3^{2-}, 24.5 wt% SO_4, 2.7 wt% Cl^- and 7.5 wt% OH^-. No S_2^- was found indicating that the deposits were smelt-free. In all samples, the molar ratio of total anions to total cations. i.e. the $[CO_3+SO_4+Cl_2+(OH)_2]/[Na_2+K_2]$ molar ratio is equal or close to one, meaning that that all alkali compounds have been accounted for by the deposit analyses, within the limits of experimental error.

Note that significant amounts of hydroxides were found in all samples. Assuming the anions (CO_3^-, SO_4^{2-}, Cl and OH^-) are bound proportionally to Na^+ and K^+, the total amount of alkali hydroxides can be estimated to be 19.3 wt%, ranging from 10.0 to 25.9 wt% NaOH plus KOH.

The finding of such a high concentration of hydroxides in the lower furnace is intriguing since hydroxides are believed to cause localized corrosion of composite tubes near air ports. The results, however, are not entirely unexpected. Equilibrium calculations of concentrations of gas species in the lower furnace for this boiler show that at a typical bed temperature between 1050°C and 1100°C, Na, K, NaOH and KOH vapors may exist at a combined concentration as high as 3000 ppm. The concentration of SO_2 is less than two ppm, insufficient to convert all the alkali species to sulphates. The CO_2 concentration is about 10%, significantly more than is required for all hydroxides to be converted to carbonates. Because of the slow kinetics of the carbonation of hydroxides [8], particularly in the presence of 10% water vapour at 350°C, the average probe deposition surface temperature, the 10-minute sampling time was insufficient to covert all hydroxides to carbonates.

Upper furnace: Table II shows the results of chemical analysis of fume deposits collected at the bullnose elevation near the entrance of the screen tubes, Fig. 2, Location B. The deposit consisted mostly of, Na_2SO_4, with small amounts of Na_2CO_3, NaCl and potassium salts, but no hydroxides. The composition of deposits was relatively constant along the probe. The $[CO_3+SO_4+Cl_2]/[Na_2+K_2]$ molar ratio was also equal or close to

one, meaning that all alkali compounds in the deposits have been accounted for, to the limits of experimental error.

Note that the residence time of the combustion gas from the black liquor guns to that of the bullnose is usually less than 10 seconds. The absence of hydroxides in deposits indicates that the sulphation and carbonation reactions of NaOH and KOH must occur rapidly. This is plausible considering that the gas atmosphere above the secondary air level is oxidizing; the concentration of SO_x (i.e. SO_2+SO_3) in the gas is higher, >300 ppm, due to the oxidation of H_2S; and the flue gas temperature in the upper furnace is much lower than in the lower furnace, thus it favors the formation of SO_3.

Similar results were obtained at other test sites. Table III summarizes the average composition of deposits collected at all test sites in the upper furnace and the composition of the precipitator dust. At each test site, the composition was consistent along the probe, and no hydroxides were found. The Cl and K contents were relatively constant. The carbonate content (expressed as CO_3) increased from about 3.3 wt% at the bullnose elevation (B and C) to 6.6 wt% in the middle of the superheater region (D), and to about the same value in the generating bank deposits (E) and precipitator dust. In the economizer deposits (F), the carbonate content was unusually high, 21 wt% CO_3, corresponding to about 32 wt% of Na_2CO_3 plus K_2CO_3. It is not known why such a high carbonate content was only found at this location, particularly when the value was double that of the precipitator dust. The sulphate content of the deposits was higher in deposits that have a lower carbonate content.

The increase in the carbonate content of the deposits towards the back end of the boiler could be caused by the depletion of SOx, resulting in more sodium and less sulphur in the flue gas stream. This amount of excess sodium, when compared with sulphur in the flue gas, results in the formation of Na_2CO_3, which in turn decreases the Na_2SO_4 content of the fume deposits.

The molar ratios of Cl_2, $(OH)_2$, CO_3 and $S0_4$ to $[Na_2+K_2]$ at all test sites and precipitator dust are plotted in Fig. 4. The change in fume chemistry throughout the boiler can be seen clearly.

MORPHOLOGY

Figure 5 shows SEM photographs of fume deposits collected in the upper boiler and of precipitator dust. The precipitator dust was an agglomeration of submicron sized particles (0.5μm). Fume deposits collected on the probe surface, however, consisted of slightly larger particles (0.5-0.8μm), which appear to be "fused" and connected to one another. The larger size and fused appearance is clearly shown in fume deposits collected in the economizer (F).

The difference in morphology between fume deposits and precipitator dust is probably because fume deposits in the precipitator dust were formed and solidified inflight. They had little chance to collide with one another until they were collected by the precipitator (<200°C). Fume deposits collected with the probe, on the other hand, consist of particles that may not have completely solidified before depositing on the probe surface or may have sintered after deposition due to the high temperature of the surface (320-500°C). Note that the

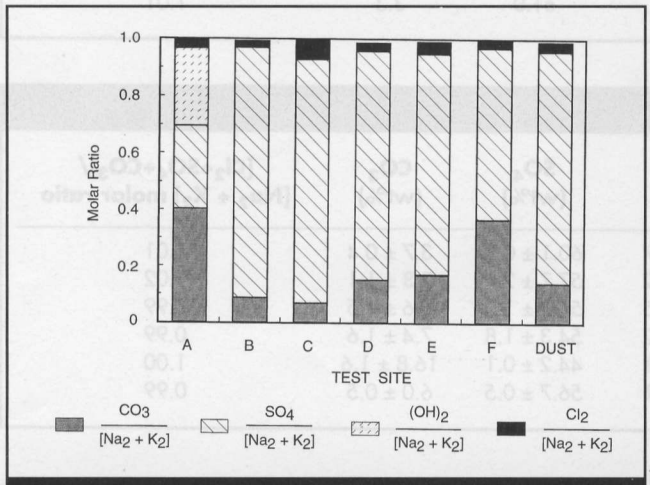


FIG. 4. CHANGE IN FUME CHEMISTRY WITH LOCATION.

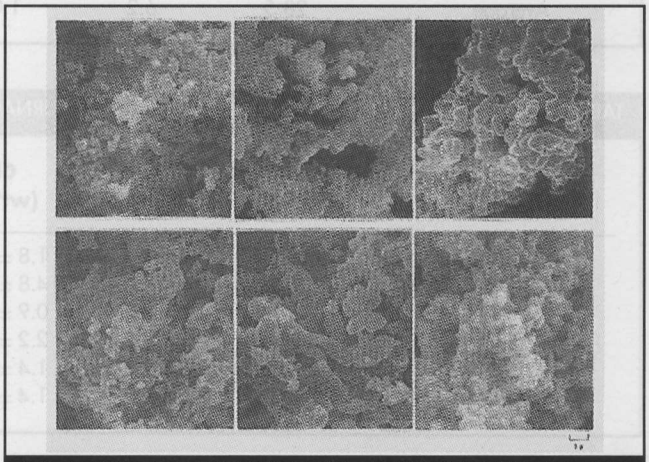


FIG. 5. SEM PHOTOGRAPHS OF UPPER FURNACE FUME DEPOSITS AND PRECIPITATOR DUST.

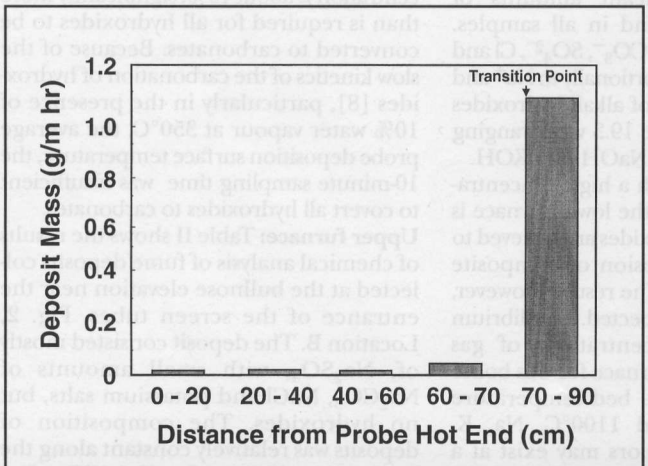


FIG. 6. MASS OF DEPOSITS ALONG THE PROBE.

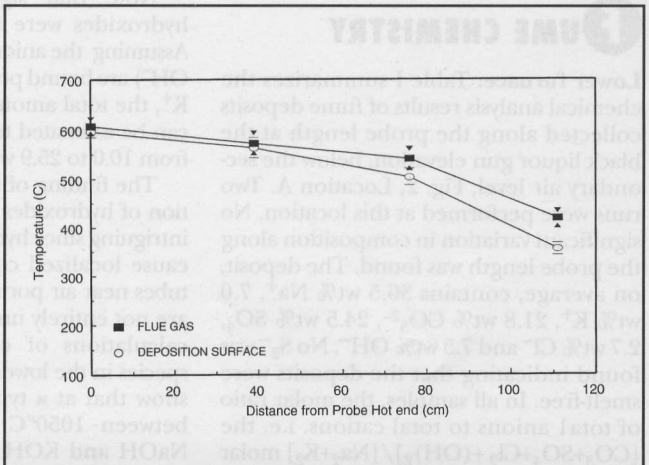


FIG. 7. SURFACE AND GAS TEMPERATURES ALONG THE PROBE.

larger and more fused appearance of the economizer fume deposits, compared with deposits collected at other locations, may be related to its unusually high carbonate content.

Deposition rate: While the composition was relatively constant along the probe, the amount of deposits collected by the probe varied, dependent on the temperature of the deposition surface. Figure 6 shows the amount of deposit collected along the probe at Location D. The amount was significantly less on the front half of the probe than on the back-half. The results were consistent with the appearance of the probe, Fig. 3. The flue gas temperature in the probe, and the deposition surface temperature during this test, are shown in Fig. 7. At the transition point the location on the probe where dust-like deposits started to be observed), the flue gas and deposition surface temperatures were about 570°C and 520°C respectively. Similar results were obtained for an uncooled probe exposed after the entrance to the screen tubes at the bullnose elevation (Location C). At this location, however, the flue gas and deposition surface temperatures at the transition point were somewhat higher, about 640°C and 570°C respectively. In both cases, though deposit mass before and after the transition point was different, the composition of the deposit calculated from the probe-wash solution from the front half of the probe (before the transition point) was the same as that of the dust-like deposits on the back half(after the transition point).

To compare the deposit mass collected at various locations in the boiler, only the data obtained from tests in which the centre tube was uncooled or slightly cooled, was used. This minimizes the effect of surface temperature on the rate of deposition. As shown in Fig. 8, the amount of deposits collected in 10 minutes by the probe was, more or less, the same, 20 mg/m^2, before and after the screen tubes at the bullnose elevation. The amount increased to 40 mg/m^2 in the middle of the superheater, then increased markedly to 140 mg/m^2 in the generating bank (this set of data was obtained with a slightly cooled probe), and reached 150 mg/m^2 in the economizer. This increase in the amount of fume deposits collected in the generating bank and economizer is consistent with the results shown in Fig.6, implying that in this boiler the majority of solid fume starts to form or to precipitate out from the flue gas at a location where the temperature is in the range of 520°C to 640°C. This corresponds to a location between the middle of the superheater (D) and the middle of the generating bank (Location E). This temperature range provides a reasonable estimate of the first melting temperature for the mixture of salts found in the upper boiler. A differential thermal analysis was conducted on the solid deposit obtained at each of the test sites, with the first melting temperature found to be 540+10°C.

Thermophoresis has been speculated to be the main mechanism of fume deposition in recovery boilers. Fine solid particles in the hot gas may be driven to a cool surface by a thermophoretic force, established as a result of the more energetic molecular bombardment on the hot side (facing the gas) than on the cold side (facing the cool surface) of the particles. The magnitude of thermophoresis depends on the gas and particle properties, and the temperature difference between the flue gas and the deposition surface. Based on a theory developed by Gokoglu and Rosner [9], the deposition rate, expressed as a deposited mass, can be expressed as:

$$\text{Deposition Rate} = K\,[T_g - T_w]/T_w$$

where T_g and T_w are the gas and tube wall surface temperatures, respectively, and K is a proportionality constant.

In this study, the deposition rate was obtained by measuring the mass of deposit collected over 20 cm sections of the probe. As shown in Fig. 9, the deposition rate at the location E increases with $[T_g - T_w]/T_w$. Similar results were obtained at other locations in the upper furnace, which suggests that fume deposition is consistent with a thermophoretic mechanism.

CONCLUSIONS

The chemistry, morphology and rate of accumulation of fume deposits in the kraft recovery boiler at E.B. Eddy Forest Products were studied using a fume deposition probe. While there are a number of unexplainable findings, the following conclusions can be made for the recovery boiler at E.B. Eddy Forest Products.

- In the lower furnace, fume deposits contained as much as 26 wt% alkali hydroxides (NaOH plus KOH), besides the usual components, Na_2SO_4, Na_2CO_3, NaCl and potassium salts.
- In the upper furnace, from the bullnose elevation onward, no hydroxides were found. Chloride and potassium contents were relatively constant throughout the boiler. The carbonate content increased, at the expense of the sulphate content, from about 5 wt% as Na_2CO_3 plus K_2CO_3 at the bullnose elevation to 12 wt% in the middle of the superheater region, and remained constant at this location onward, except in the economizer.
- Compared to precipitator dust, fume particles collected on the probe appeared to be larger, "fused" and connected to one another. The morphological difference is presumably due to the fact that fume particles collected on the probe may not have been completely solidified before depositing, or may have sintered rapidly after deposition.
- The majority of solid fume appeared to form at a location close to the entrance of the generating bank where the flue gas temperature is in the range 520 to 640°C.

The deposition rate results are consistent with thermophoresis as a fume deposition mechanism.

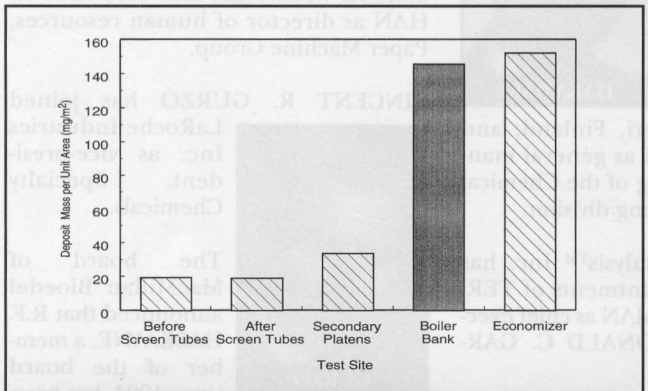


FIG. 8. MASS OF DEPOSITS COLLECTED AT VARIOUS LOCATIONS IN THE BOILER.

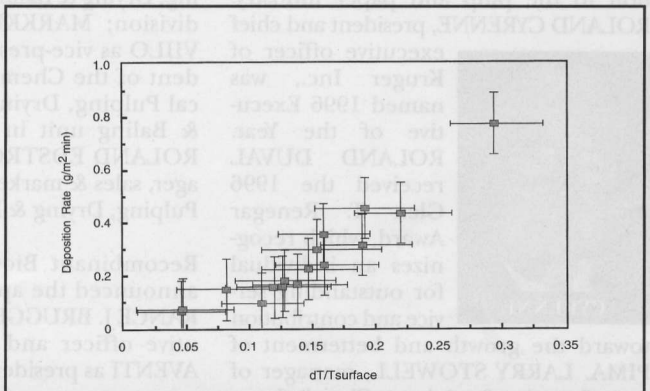


FIG. 9. DEPOSITION RATE FOR TESTS AT LOCATION E.

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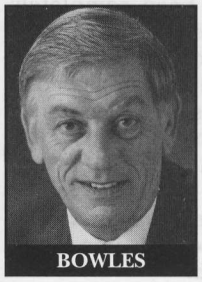
Résumé: Nous avons recueilli des dépôts de fumées à divers endroits dans une chaudière de récupération. Les dépôts de la partie inférieure de la chaudière contenaient 26wt% d'hydroxydes alcalins en plus des sels de sodium et de potassium habituels. Les dépôts de la partie supérieure de la chaudière ne contenaient pas d'hydroxydes et avaient une teneur constante en potassium et en chlorure. La teneur en carbonate augmentait aux dépends du sulfate, de 5 wt% à la section quart de cercle à 12 wt% dans l'aire du surchauffeur. A partir de cet endroit et au-delà, elle demeurait constante, sauf dans l'économiseur, où la teneur en carbonate était inhabituellement élevée. Nous avons noté la formation de fumée solide à l'entrée des parois latérales de l'aire de chauffe. Nos observations sur les taux de déposition étaient conformes au mécanisme de déposition thermophorétique.

Abstract: Fume deposits were collected from various locations of a recovery boiler. Lower furnace deposits contained 26 wt% alkali hydroxides, plus the usual sodium and potassium salts. Upper furnace deposits contained no hydroxide and had constant chloride and potassium contents. The carbonate content increased, at the expense of the sulphate content, from 5 wt% at the bullnose to 12 wt% in the superheater region, and remained constant from this location onward, except in the economizer where the carbonate content was unusually high. Solid fume appeared to forrn at the entrance to the generating bank. Deposition rate results were consistent with a thermophoretic deposition mechanism.

Reference: TAVARES, H., TRAN, H., BARHAM, D., ROUILARD, P., ADAMS, B. Fume chemistry, morphology and deposition in a kraft recovery boiler. *Pulp Paper Can* 97(10):T361-366 (October 1996). Paper presented at the 1995 Int. Chem. Recovery Conference of the Technical Section, CPPA, co-sponsored with TAPPI at Toronto, ON on April 24-27, 1995. Not to be reproduced without permission. Manuscript received April 4, 1995. Revised manuscript approved for publication by the Review Panel, January 11, 1996.

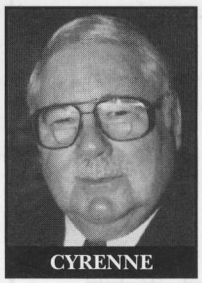
Keywords: RECOVERY FURNACES, DEPOSITS, CHEMISTRY, PLUGGING, DEPOSITION, EMISSIONS, FUMES

Nacan Products Limited has appointed CHRIS BOWLES to the position of divisional vice-president, Adhesives Division.


BOWLES

CAL MOISAN, Standard Paper Box, has been named president of the Canadian Corrugated Case Association for 1996.

The Paper Industry Management Association (PIMA) recently honored some persons for their contribution to the pulp and paper industry. ROLAND CYRENNE, president and chief executive officer of Kruger Inc., was named 1996 Executive of the Year. ROLAND DUVAL received the 1996 Glen T. Renegar Award, which recognizes an individual for outstanding service and contribution toward the growth and betterment of PIMA. LARRY STOWELL, manager of manufacturing for Arizona Chemical, was the second recipient of the PIMA Magazine Vision 300 Award, given annually to the individual who has done the most to raise the image of the paper industry and educate the general public about the paper industry's role in North American business and culture.


CYRENNE

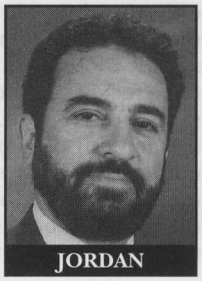
Sunds Defibrator recently made some executive appointments, naming HANNU HAKAMÄKI as president of the Chemical Pulping Systems Business Area; LARS SJÖDIN as vice-president and general manager of the Chemical Pulping, Drying & Baling division; MARKKU VIILO as vice-president of the Chemical Pulping, Drying & Baling unit in Pori, Finland; and ROLAND EDSTRÖM as general manager, sales & marketing of the Chemical Pulping, Drying & Baling division.


HAKAMÄKI

Recombinant BioCatalysis™ Inc. has announced the appointments of TERRANCE J. BRUGGERMAN as chief executive officer and DONALD C. GARAVENTI as president.

GEORGE C. RIGHTMYER has been appointed by Degussa Corporation to the position of director, sales, Peroxygen Chemicals Division.

Beloit Corp. announced a handful of appointments: ALAN JORDAN has been named managing director, Beloit Industrial Ltda, BERNIE STEIN as director, corporate business development, RUSSELL PAOLETT as director of compensation benefits worldwide, and TIM MONAHAN as director of human resources, Paper Machine Group.


JORDAN

VINCENT R. GURZO has joined LaRoche Industries Inc. as vice-president, Specialty Chemicals.


GURZO

The board of MacMillan Bloedel announced that R.F. HASKAYNE, a member of the board since 1994, has been elected chairman of the board effective October 1, 1996.