

ANALYSIS OF SCALE FROM SOME SOUTH AFRICAN SUGAR MILLS

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Abstract

Several samples of evaporator scale collected during the 1993 and 1994 milling seasons were analysed using x-ray diffraction, x-ray fluorescence, scanning electron microscopy and optical microscopy. The results were evaluated in terms of current knowledge of evaporator fouling and similar trends were found to those published in the literature. From these results, typical evaporator scale profiles could be proposed for Southern African mills, depending on whether hydroxyl apatite is formed. Nevertheless each mill has its own characteristics. FX displayed a distinctly high silica content in its early effect scale while calcium aconitate was identified as a major phase in scale from NB. Microscopic examination found that scale was formed in layers. These analytical techniques were used to evaluate the effect of additives to the process streams on scale formed.

Introduction

The fouling of evaporators results in a number of problems including a reduction of the capacity of the evaporator station, cleaning costs, and sucrose loss through inversion due to increased residence times. It is clear that an essential step in combatting fouling is a thorough understanding of the scale compositions and the mechanisms of formation. Thus an examination of local scale was undertaken with the following objectives:

- To examine the chemistry and mineralogy of the scale to understand the mechanisms of formation and removal so that techniques of prevention and removal, as well as process changes, can be evaluated.
- To identify differences between mills in terms of geographical location, seasonal changes and operating conditions.
- To develop a database of scale analysis to identify long term trends.

Review of current knowledge

To ensure an effective examination of the scale and evaluation of the results, a literature survey of evaporator fouling in the cane sugar industry was undertaken. This survey included an examination of local evaporator scale analyses available prior to the commencement of this project. The main findings of this review with regard to factories using defecation were (Walthew, 1994):

The main inorganic components are silica, calcium, magnesium and phosphate. There is evidence that the removal of inorganic scale components, especially calcium, dramatically reduces fouling during evaporation of sugar solutions (Thompson, 1993) and for this reason the chemical analysis of scale is primarily focused on the inorganic components.

The organic compounds are not always well defined, being either amorphous material (no crystalline structure) or precipitated salts of organic acids. Specific compounds of interest have been identified:

- Calcium phosphate is found in scale in two forms. The most common one is as an amorphous calcium phosphate (ACP). While this is nominally " $\text{Ca}_3(\text{PO}_4)_2$ " it is found to have a calcium to phosphate ratio of between 1,3 and 1,7. Also widely reported is a crystalline hydroxyl apatite (HAP) ($\text{Ca}_{10}(\text{OH})_2(\text{PO}_4)_6$). The precipitation of calcium phosphate, which starts in the clarifier, is characterised by slow kinetics and therefore phosphate is predominantly found in the earlier effects. It is generally believed that HAP is formed from ACP through the action of time and temperature. Magnesium and iron can substitute partially for calcium to produce a mixed phosphate. Phosphate is extracted from the cane, although it is sometimes added. Calcium is added as lime during clarification and magnesium originates either from the cane or from the lime used.
- Sugar and other organic material are trapped in the bulky phosphate precipitate and are degraded through the action of heat on the tube walls. Consequently amorphous organic material also decreases as the evaporator effect increases.
- Organic acids are extracted from the cane and some may also be produced by the degradation of sugar during processing. Oxalic acid is the most troublesome from a fouling aspect. Although iron, and magnesium, if present, will form oxalates, the most important compound found in studies of evaporator scales from both the cane and the beet industries is calcium oxalate which is found in two forms, weddellite ($\text{CaC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$) (calcium oxalate dihydrate (COD)), and whewellite ($\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$) (calcium oxalate monohydrate (COM)) which is the thermodynamically stable compound at higher temperatures. The conditions which favour the formation of COD or COM are not clear, and of limited interest since both appear equally difficult to remove. Oxalates are found mainly in the later effects of the evaporators.
- Silica is usually precipitated in the later effects (and in some cases in the pans) as amorphous silica or poorly crystalline calcium silicate. The factors influencing the precipitation of silica during the evaporation of sugar juice are not well established. It is generally believed that the precipitation follows polymerisation and coagulation and that these processes are promoted at high temperature (Iler, 1979). Silica can be introduced to the juice through amorphous silica contained in the fibre of the plant, which is taken into solution during the extraction process, or through the reuse of sweetwater which has been passed through a diatomaceous earth filter, or through the dissolution of silica sand or silt carried in with the cane. The dissolution of quartz sand is not expected to be significant in view of the poor solubility displayed by quartz in water (Iler, 1979).

Analytical methods

The methods used for analysis were X-ray fluorescence (XRF), X-ray diffraction (XRD), optical microscopy and scanning electron microscopy (SEM).

XRF is carried out by irradiation of a solid sample with a beam of primary X-rays. This serves to excite each element in the sample to emit X-ray signals of characteristic wavelengths which are separated and measured. In this way accurate and rapid bulk chemical analysis can be obtained for a range of inorganic elements. This technique can be combined with scanning electron microscopy (SEM) to provide semi-quantitative analysis of spots or small areas of a particular phase within a complex matrix.

Prior to carrying out XRF analysis the sample is ashed at 1 000°C which gives a loss on ignition (LOI). This gives some indication of the "volatile" material and in conjunction with other analysis can confirm the presence of certain compounds. A guide to the expected LOI can be seen in Table 1.

XRD, based on the Bragg principle that X-rays of known wavelength are diffracted by crystalline materials, is used to supplement chemical analysis. Each crystalline phase has a characteristic pattern which is unique, depending on its chemistry and crystal symmetry. The diffraction pattern can usually be identified by referring to the database of the Joint Committee on Powder Diffraction Standards (JCPDS). In this way it is possible to detect and identify the presence of particular crystalline phases (both organic and inorganic). The term "phase" is one commonly used in mineralogy to refer to the particular crystal structure in which a compound is found. For example XRD can distinguish between COM and COD although the chemical compositions are similar. Although XRD can give quantitative information, through standardised measurement of the relative heights of peaks produced by the diffractogram, in the absence of standards it is more commonly used to indicate which phases (species or compounds) are major (high concentrations) and those which are minor (low concentrations). Non-crystalline or poorly crystalline materials cannot be analysed since these are amorphous to X-rays, and are recorded as a raised background "hump" or "halo" rather than as a sharply defined peak.

To enable the examination of scale by microscopic methods, samples were set in resin and made into polished sections for examination using reflected light microscopy and scanning electron microscope (SEM). Thin sections were prepared for examination using transmitted light.

Scale sampling

Samples were taken from a number of mills during the 1993 and 1994 seasons during normal cleaning procedures

and were dried at 110°C, crushed and subdivided for analysis by XRF and XRD. There are several possible sources of error due to sampling:

- In cases where mechanical cleaning takes place in an aqueous environment, some dissolution of scale components by water may take place. The solubilities of the species of interest are however low, and losses by this route should be negligible.
- Where sampling takes place after chemical cleaning, there is a danger of residual caustic leaching. Although work at the Sugar Milling Research Institute (SMRI) has indicated that this should not be significant at the low temperatures involved, the difference between caustic leached and unleached samples could be significant in terms of calculated total percent, since the organic fraction can be reduced in the former case.
- If residual sugar is present, it can ferment in transit and produce acids which will attack some components in the scale.
- Because of the nature of the scale removal operation, some fine scale material will be lost during the flushing operation. Thus it is assumed here that there is no difference in composition between particles of varying sizes.
- The point from which the sample was taken might influence the composition. This aspect is examined below.

Results

Overview

The results of the XRF and XRD analyses taken together generally confirmed the trends identified in the review with respect to amorphous organic material, phosphate, silica and oxalates. However it was evident that the evaporator scale profile varied according to whether HAP was formed. In the cases where no HAP formed the profile followed that shown in Figure 1, which is a simplified "ideal" case. It should be noted that the actual concentrations can vary quite significantly in some cases from the typical case presented, but the general trends remain similar.

Where HAP was formed in the evaporators it was found that the calcium content was increased significantly, especially in the first effects. High phosphate was also found in samples containing HAP, and the relative concentrations of silica and oxalate were reduced as a consequence. A typical concentration profile for a mill in which HAP is present in evaporator scale is shown in Figure 2.

Table 1

LOI figures calculated for various compounds

Compound found in scale	Form after ashing	Estimated LOI
Silica $\text{SiO}_2 \cdot x\text{H}_2\text{O}$ $x = 0,5 - 3$ For this study $x = 1$	SiO_2	$18/78 = 23\%$
$\text{MgO} \cdot \text{H}_2\text{O}$	MgO	$18/58 = 31\%$
Amorphous calcium phosphate (ACP) Assumed composition of $\text{Ca}_{1,5}(\text{PO}_4) \cdot \text{H}_2\text{O}$	$\text{PO}_{2,5}(\text{P}_2\text{O}_5) + \text{CaO}$	$46/173 = 27\%$
Hydroxyl apatite (HAP) $\text{Ca}_{10}(\text{OH})_2(\text{PO}_4)_6$	$(\text{CaO})_{10}(\text{P}_2\text{O}_5)_3$	$18/1004 = 2\%$
Whewellite $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$ calcium oxalate monohydrate	CaO	$90/146 = 62\%$
Weddelite $\text{CaC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ calcium oxalate dihydrate	CaO	$108/164 = 66\%$
Organic	$\text{CO}_2 \uparrow$	100%

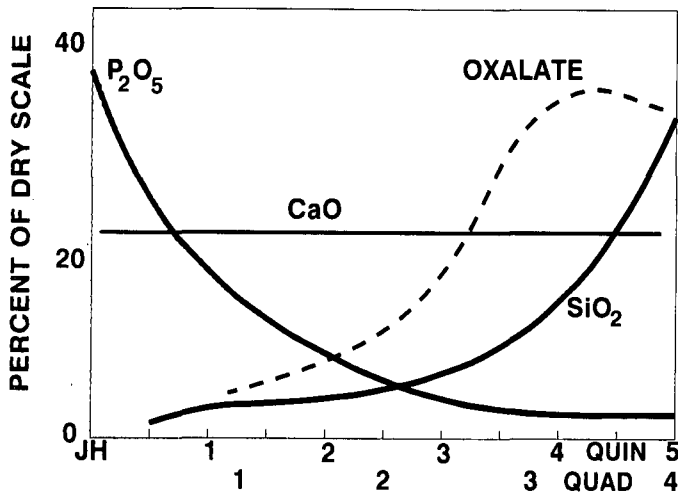


FIGURE 1 Typical profile for mills examined where HAP is not formed.

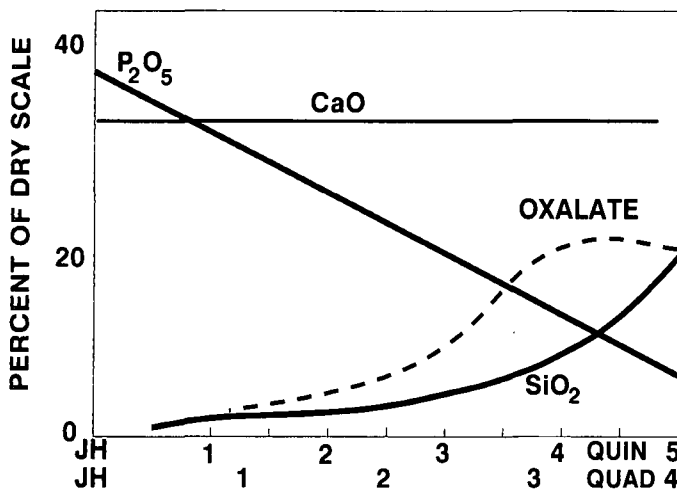


FIGURE 2 Typical profile for mills examined where HAP is formed.

A confirmation of the predominant presence of HAP was seen in the LOI figures which were significantly reduced especially in the earlier effects. Typically LOI values of less than 25% for scale containing HAP were obtained against figures above 40% for scale without HAP. The reason for this difference can be seen from the estimated mass losses shown in Table 1. In scale from the later effects the LOI values are less likely to provide a valuable indication since the formation of oxalate tends to increase the volatile fraction.

Phases formed

The phases identified by XRD in various samples are shown in Table 2. The abbreviations and terminology used are:

- Phosphate: Hydroxyl apatite (HAP)
- Amorphous calcium phosphate (ACP)
- Oxalate: Calcium oxalate monohydrate (COM)
- Calcium oxalate dihydrate (COD)
- Aconitate: Calcium aconitate (CAC)
- Silica: Amorphous silica (AS)
- Quartz (sand) (QTZ)

Table 2

XRD Analysis of scale samples

Quads Mill	Date	Effect	Phosphate		Oxalate		CAC	Silica	
			HAP	ACP	COM	COD		AS	QTZ
DL	Aug 93	1	o	X	o	o	x	o	o
DL		4	o	o	o	X	o	X	o
NH	Dec 93	1	X	o	o	o	o	o	o
NH		2	X	o	x	o	o	o	o
NH		3	X	o	X	o	o	o	o
NH		4	X	o	x	o	o	o	o
UC	July 94	1	o	X	o	o	o	o	tr
UC		2	o	X	tr	o	o	o	o
UC		3	o	x	x	X	o	o	o
UC		4	o	o	X	X	o	o	o
UK	July 94	1	o	X	o	o	o	o	o
UK		2	o	X	o	x	o	o	o
UK		3	o	o	o	X	o	o	o
UK		4	o	o	o	X	o	X	o
SZ	Dec 93	1	o	x	x	o	o	o	X
SZ		5	o	o	X	x	o	X	o
UF	July 93	4	o	o	o	x	o	X	o
UF	94	1	X	o	o	o	o	o	o
UF		2	X	o	tr	o	o	o	o
UF		3	X	o	x	o	o	o	o
UF		4	X	o	x	o	o	x	o
UF		5	X	o	x	tr	o	x	o
FX	Dec 94	1	x	X	o	x	o	X	X
FX		2	o	o	o	o	o	X	o
FX		3	o	o	o	x	o	X	o
FX		4	o	o	X	X	o	X	o
FX		5	o	o	X	x	o	X	o
NB	Aug 93	3	tr	o	o	x	X	o	o
NB	July 94	3	tr	o	tr	tr	X	x	o
NB		4	o	o	tr	tr	X	x	o
PG	July 93	1	X	o	o	tr	o	o	o
PG		2	X	o	o	x	o	o	o
PG		3	X	o	o	X	o	o	o
PG		4	X	o	o	X	o	o	o
PG		5	X	o	o	X	o	o	o
ME	Nov 93	1	o	o	tr	o	o	o	X
ME		2	o	x	X	o	o	o	x
ME		3	o	o	X	X	o	o	tr
ME		4	o	o	x	X	o	o	tr
ME		5	o	o	tr	X	o	o	o

X = Major phase; x = Minor phase; tr = Trace; o = Not detected

The approximate concentrations are classed as major, minor, trace or not detected, according to the following key:

- Major : X
- Minor : x
- Trace : tr
- Not detected : o

It should be noted that the silica in all cases was found to be predominantly amorphous. Where quartz is reported in Table 2 this occurs as sand particles and did not contribute to the fouling, i.e. was distinctly separate from the "scale" particles.

The range of compounds formed suggests that methods of scale prevention and cleaning employed at one mill may not be as successful at another. Most mills appear to have a certain characteristic. This is summarised in Table 3.

Table 3
Characteristics of scale from various mills

	Mill/ Diffuser	4/5 effect	
PG	D	5	HAP found with COD.
UF	D	5	HAP found with COM in 1994 only. In 1993 high silica and COD.
FX	D	5	Very high silica in all effects. 1993 samples lower in silica.
DL	D	4	'Typical' profile for all components, weak CAC peak.
NB	M	5	High aconitate; COD + COM found.
UC	D	4	Low in crystalline phases and high amorphous organic compounds throughout. Mix of COD and COM.
ME	M	4	'Typical' profile for all components, relatively high chloride.
SZ	D	5	'Typical' profile; equal mix of COD and COM.
UK	D	4	Very strong COM and COD.
NH	M	4	HAP and COM only.

Geographical trends

The data in Table 2 suggest that HAP is predominant in scale samples from the northern regions (NH, PG, UF) if NH is considered as a northern mill. Where HAP was found in scale from one of the evaporator vessels it was usually found in all scales from that particular mill. In the cases of NH and UF, HAP is also found with COM rather than the dihydrate. Conventional thinking holds that HAP and COM may result from longer residence times and higher temperatures, since both are essentially dehydrated forms of their precursors, and it would be logical that these two should appear together. However it is unlikely that residence time is a function of geographical location, or that all vessels in an evaporator set would have sufficiently long residence times. This suggests that another factor, such as catalytic effects of small amounts of other chemicals, or seeding effects, may be involved. Generally there seems to be no common denominator which appears to promote either COM or COD or to explain why some mills make only one phase while others make a mixture of COM or COD. Alternatively these groupings may simply be coincidental.

Anomalies

Two mills showed distinct differences from the typical scale concentration profiles presented above. The first was FX which had a very high silica content in its first and second effect evaporator scales. Although the silica content decreased in the 1994 season, concentrations of silica in the first two effects were still higher than those from other mills. This is shown graphically in Figure 3 which shows the silica concentration profiles for FX scale from 1993 and 1994 compared to an average for the other mills sampled. It is noteworthy that this trend was observed in evaporator scale analysed in 1984 where FX scale from the second effect was

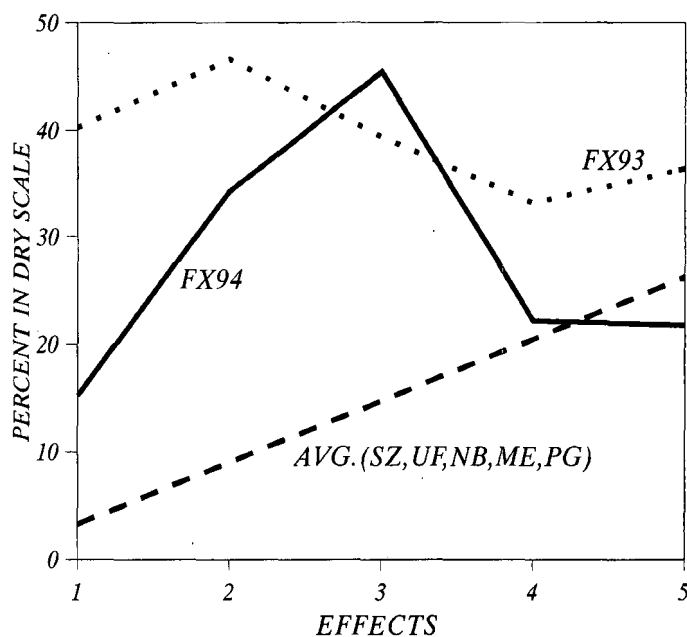


FIGURE 3 Silica in dry scale samples from FX taken during 1993 and 1994 in comparison with other mills (SZ, UF, NB, ME and PG) which are presented as an average.

also found to contain high levels of silica relative to other samples taken prior to this survey (Walthew, 1994). UF was also found to have a high silica content in scale samples during the 1993 season but in the 1994 season the silica concentration corresponded with the typical profile.

As a first approach towards understanding the high silica content the analyses of clear juice from a number of mills, including FX and UF, were examined. Analysis of clear juice composites collected from PG, UF, FX, NB and SZ during the 1993 season did not show the silica concentration of UF or FX clear juice feed entering the respective evaporator station to be any higher than that from the other mills sampled.

Since it is likely that the driving forces behind the formation and precipitation of silica are supersaturation and temperature, it is instructive to note the differences, if any, between the brix changes across the evaporator trains of the mills under examination. Most mills have very similar profiles but two stand out as having a heavy load on the first and second effects, namely NB and FX. This is shown in Figure 4 which, for simplicity, shows the brix of the juice leaving each evaporator for FX and NB relative to the average which was calculated using figures of all mills from which scale samples were taken, excluding NB and FX. The brix profiles do not explain the high silica in FX scale adequately since first to third effect NB scales contain less than 5% silica compared with 30 to 50% silica found in FX first and second effect scale samples. Furthermore the silica levels in the scale from the first two effects at FX are about the same as those found more usually in the final effects at other mills.

The second unusual discovery was that NB scale was found to contain a high level of calcium magnesium aconitate (CAC). Since no corresponding pattern match to the x-ray diffractogram could be found in the official JCPDS index, a sample of calcium magnesium aconitate was made and the pattern was found to correspond with that obtained from the NB third effect sample from 1993, and that of third and fourth effect samples from 1994. High levels of CAC in scale have not been reported previously and only one other mill

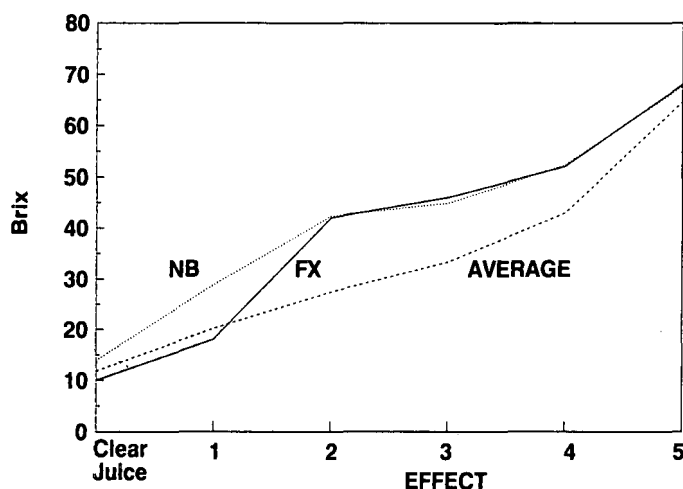


FIGURE 4 Brix (concentration) profiles showing the differences between FX, NB and the other mills (TR, ME, PG, UF and SZ) which are presented as an average.

during this survey was found to contain aconitine. This was DL and the amount was very small, as shown in Table 2. Interestingly, samples from UC were found to be quite different, containing relatively little crystalline material, and no aconitine at all.

Consistency of scale samples

To assess the variation of the scale composition with respect to the point from which samples are taken, samples were obtained from SZ and NB at various points in the vessels. Samples from SZ were taken from the first effect (Kestner) tubes and top tube plate, and from the third effect (Roberts) tubes and top tube plate. At NB samples were taken from the tubes, top tube plates and bottom tube plates from the third and fourth effect evaporators (Roberts). In all cases very little variation in the scale composition was found. However visual inspection of the NB evaporators suggested that significantly more scale was formed at the bottom of the tubes than at the top, even though the compositions were virtually identical. Where parallel evaporator trains are used samples from the A and B phases were also found not to vary substantially.

To assess the effect of the time of the year on scale composition, samples were taken at UK from July to November during the 1994 season. No significant change could be identified over this period. Samples taken from FX, UF, DL, NB and SZ at different times in the same season were also found to have consistent analyses. This is an unexpected result since it is commonly felt that scale formed later in the season is more difficult to handle. These tests suggest that rather than scales at different times of the year being of different compositions, it may simply be that greater quantities are formed later in the season. However significant differences were found between scale samples collected during the 1993 and 1994 seasons from FX and UF. These are shown in the silica profiles shown in Figure 3.

Microscopic examination

To obtain an idea of the way in which the scale components are arranged, optical and SEM examinations were undertaken. A typical SEM photomicrograph is shown in Figure 5 of a polished section or "plug" of scale which was removed from the third effect at SZ during the 1993 season. This shows the oxalate crystals surrounded by the amorphous material. Spot and area analyses of the amorphous compounds established that certain components occurred to-

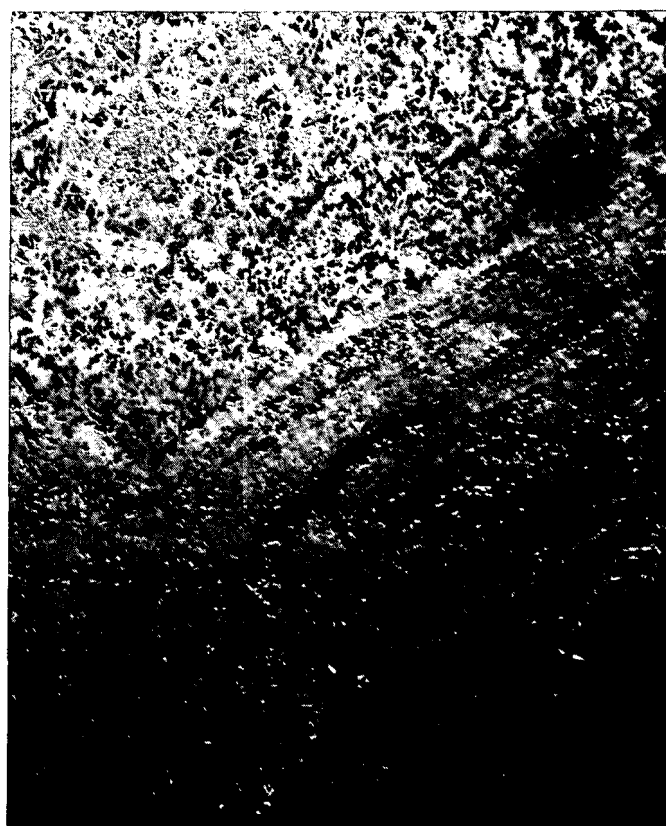


FIGURE 5 SEM photomicrograph (backscatter image) showing crystalline material (light areas) in a matrix of amorphous silica and organic material. Note also the layers formed. Magnification 30x.

gether, namely silica and calcium; and phosphate, calcium and amorphous organic material. Silica was also found to have been precipitated alone with little occluded impurities. These observations suggest that the calcium silicate, silica, and calcium phosphate are formed as colloidal particles in suspension before being deposited around the crystalline material (oxalate). This suggests that suspended solids are as likely to deposit in the evaporators as are dissolved solids. It was also found that the scale is formed in distinct layers each rich in a particular phase, which can also be seen in Figure 5. Examination of thin sections established that the organic material was strongly concentrated in certain layers, and where COM and COD were both present these too were formed in layers. This suggests that during the evaporation process conditions occur periodically which favour a particular compound's deposition, such as "dryout", surges in steam pressure or pH changes. The concentration of certain components in layers implies that where chemical cleaning, using caustic, is used it will attack the organic and silica rich layers, but may not readily penetrate the oxalate rich layers, preventing efficient attack and cleaning. In such cases alkali/acid cleaning may be more effective.

Case study: effectiveness of antiscalant

As a demonstration of the usefulness of the above analytical techniques these methods were used to evaluate the effect of process changes made at NB and UC during the 1994 season. In both cases samples of scale were taken after "normal" operation and were analysed to establish the typical scale composition. Following antiscalant trials by UC and NB over a period of two weeks, samples were again

taken and analysed. At both mills two commercial antiscalant products were used, dosed at 10 ppm (g/ton) into the clear juice and third effect.

Following the antiscalant trials at NB, SO₂ was added to the juice for a two week period at the rate of 200 ppm to improve colour and viscosity. Scale samples were also taken to evaluate the effect, if any, of SO₂ on the scale formed.

Results of UC trials

Analysis of the UC "normal" samples (Table 2) showed relatively little crystalline material to be present, but it could be established that the phases present were ACP in the initial effects and COM and COD in the later effects. After running with antiscalant addition, scale samples analysed showed the same phases present. Of significance is that by comparing the XRD peak heights between the two sets of scale it was estimated that the amount of crystalline oxalate deposited was reduced by about 30% through the use of the antiscalant. The operating staff at UC considered the antiscalant effective in that higher crush rates could be maintained during the test period than were normally expected, although no HTC data were recorded.

Results of NB trials

When run under "normal" conditions, the third and fourth effects were found to contain high levels of CAC with smaller amounts of COM and COD. When antiscalant was used the CAC peaks were reduced by about 30% and only COM was found in very small amounts, *i.e.* it appears that the antiscalant prevents the formation of COD. However SO₂ appears to be even more effective at reducing the amount of crystalline CAC deposited since the relative peak heights were reduced by about 60%. As for the antiscalant, SO₂ appears to inhibit the formation of COD and a small relative increase in the amount of COM was noted. XRF analysis showed that amorphous silica was increased in the second, fourth and fifth effects when both antiscalant and SO₂ were used, the influence of the latter being greater than the former. XRF analysis of treated scale thus confirmed XRD findings of reduced calcium phases, with increased SiO₂/CaO in bulk chemistry. Similarly the additives were found to reduce the amount of magnesium in the scale samples, again SO₂ being more effective than antiscalant. The explanations for these changes are not straight forward but do suggest that small changes in the chemical composition of the juice can have marked effects on the scale formed. In addition it seems that changes in chemical composition brought about by the antiscalant addition were even greater when SO₂ was used. In spite of these well defined effects detected by XRD, no change in the ease of cleaning of the evaporators was noted by the process staff in either of the tests where additives were used. Since ease of cleaning (scale softening), or a clear reduction in the quantity of scale formed, is ultimately necessary to consider an antiscalant effective, the test was considered unsuccessful. Nevertheless the analysis carried out showed its sensitivity to the changed conditions and permits an extension of this work or a consideration of alternative treatments. These results suggest that to some extent the antiscalant was effective at controlling the crystalline deposits and thus presumably scale hardness. It does not how-

ever indicate a change in the rate of deposition or the quantity deposited, and where the fouling is extensive the effect of even a large reduction in the crystallinity may well be swamped by the total mass of scale formed.

Conclusions

This work suggests that the evaporator scale composition in South African mills generally follows trends found in mills using defecation in other parts of the world. It should be noted that this survey has thus far taken place during two drought years and future results may show different trends, although the results generally confirm other scale analyses carried out on samples from mills using defecation prior to this study.

The scale composition follows the trends reported in the literature in that "soft", usually amorphous, scales containing mostly ACP and organic compounds form in the first effect. In later effects harder more crystalline oxalates are formed. In mills where HAP is present the phosphate and calcium concentrations of the scale are generally higher, and the LOI at 1 000°C lower, than in mills where ACP is formed. Nevertheless, most mills seem to display some uniqueness. It can be tentatively proposed that HAP is formed predominantly in northern mills.

Notable exceptions to the "normal" scale are found at FX where high levels of silica were found in all effects and NB where calcium aconitate was identified in large quantities. These two unusual findings are presently not satisfactorily explained.

Microscopic examination of scale shows that the components are deposited in layers which are each rich in a particular component. This suggests that certain layers may be resistant to chemical attack and retard chemical cleaning operations. Within the amorphous phase it seems that silica, calcium silicate, and calcium phosphate are deposited as discrete particles suggesting that the precipitation pathway is via the formation of inorganic "polymers" in solution which are deposited when they reach a critical size.

The analytical methods used appear sufficiently sensitive to enable the effect of process changes to be evaluated.

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