

# PROGRESS AND DEVELOPMENT IN MELT-SULPHITATION REFINING AT UMFOLOZI

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Having produced a paper some years ago on the refining process as introduced at Umfolozi in 1959-60, it is perhaps not out of place to review what is considered development of the operation technique and the results thereby obtained.

In the earlier presented paper it was stated that sulphur was admitted through a nozzle in the burner and being sprayed on to a splashtile. The instructions were specific as to the required pressure, the angle of the cone of sprayed sulphur and the aperture of the nozzle for a given quantity of molten sulphur.

Experiencing difficulties in keeping the nozzle free from clogged-up dirt contained in the sulphur, a straight tube with a slightly tapered bore down to about  $\frac{1}{4}$  in. has replaced the nozzle and resulted in

complete absence of failures since, i.e. the  $\text{SO}_2$  production has become very steady, vide fig. 1 and fig. 2, and there is practically no more sublimation in coolers.

Sublimation can be caused not only by irregular  $\text{SO}_2$  production, due to unsteady supply of sulphur, but also by low combustion temperatures. These can occur when insufficient sulphur is pumped into the burner, as in the case of a clogged nozzle, but also because of cold air used for the combustions. This is especially the case if the  $\text{SO}_2$  production is below its minimum rating for the plant, which was said to be 250-300 lbs. of sulphur per hour. To overcome this the makers of the plant advised to pre-heat the compressed air by passing it through a jacket around the first part of the atmospheric cooler.

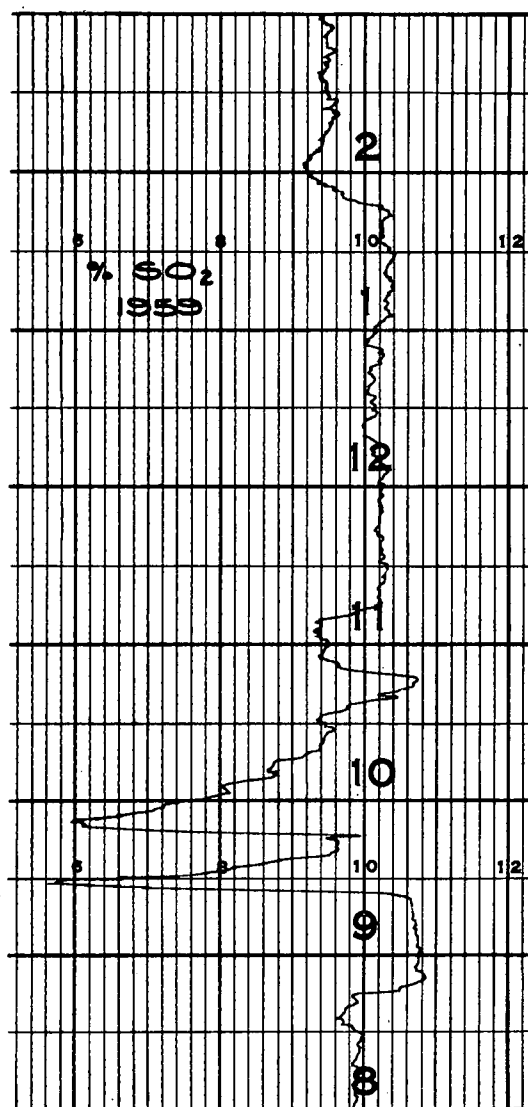


FIG. 1

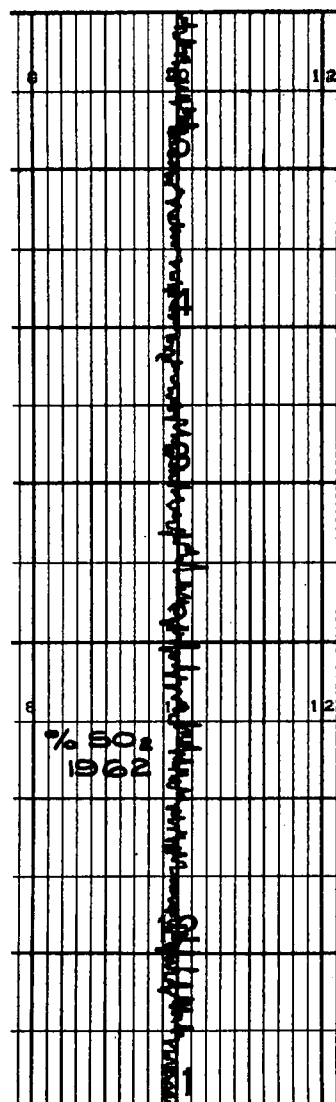


FIG. 2

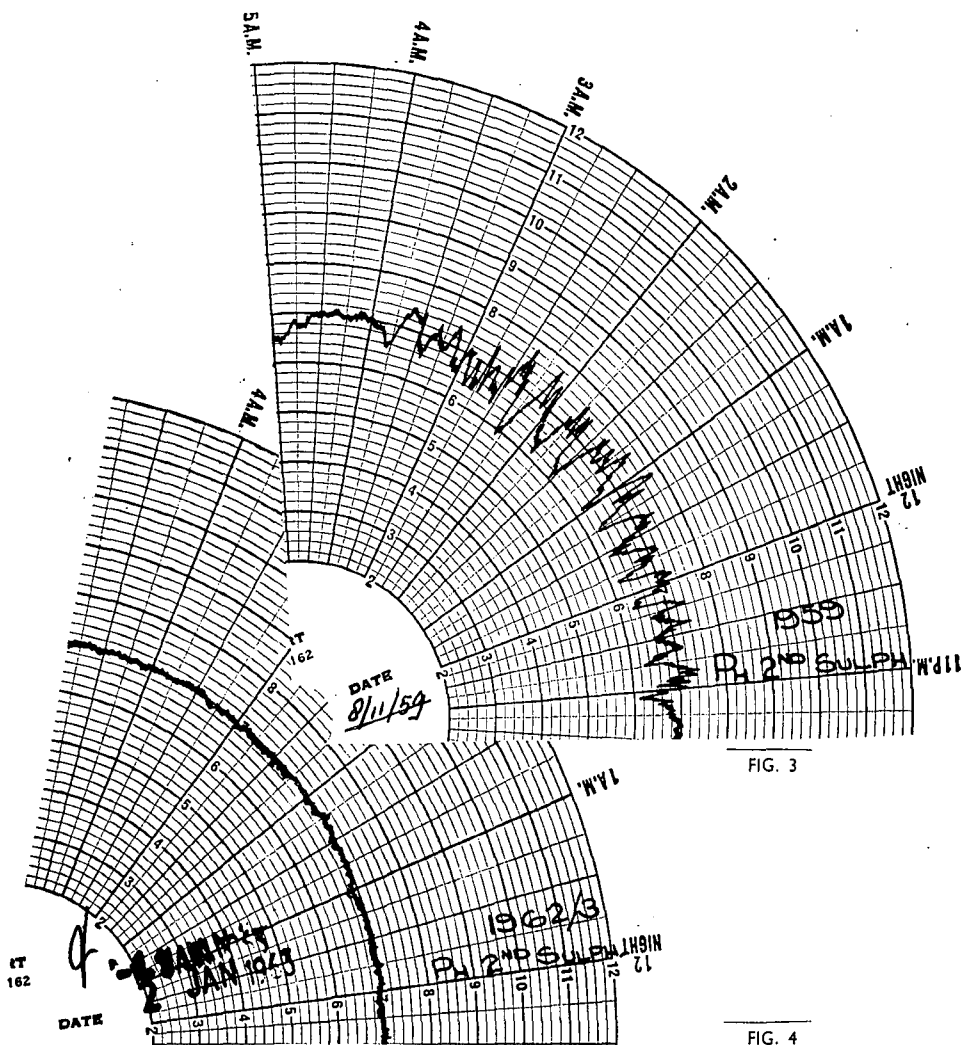


FIG. 3

FIG. 4

The temperature of the gas leaving the burner rose by 70-100°C and observations through the sightglass in the combustion chamber showed a marked improvement over the original arrangement.

The secondary cooler consisting of Karbate elements could not be cleaned effectively without losing many hours. They were subject to breakage and expensive. Therefore they were successfully replaced by cast-iron elements, which are easily replaced and cleaned.

Distribution of SO<sub>2</sub> gas in the required proportions for the individual vessels of the Continuous system has been improved by installing effective Foxboro flowmeter-recorders.

Tests have shown that it is not necessary to apply a 70+30 ratio of SO<sub>2</sub> distribution to the 1st and the 2nd vessels respectively. Perhaps experience elsewhere led to this ratio being prescribed, but in the literature we find nothing to support this. To the contrary, we read in Honig "Principles of Sugar Technology", Vol. I, page 635, that "the action of a high pH on the juice for a somewhat longer time is considered favourable to the precipitation of non-sugars".

Also, it can be considered that high pH will be less harmful in a high concentration than in a low density material and that therefore decomposition of reducing sugars will occur to a lesser extent in the high brix melt with which we operate, i.e. about 68-70° Bx.

Thirdly, it was observed that a larger dose of lime and SO<sub>2</sub> could improve the colour of liquors obtained from darker affinated sugar. As a definite lapse of time is required for the completion of the reactions and for the formation of coarse crystalline precipitate and the absorption and adsorption of various non-sugars, we considered it advisable to add the maximum quantities of lime and sulphur in the primary vessel while maintaining a high pH which, however, should not be so high as to cause colour formation in the liquors. It was found that a pH above 8.9 resulted in no apparent improvements. In the secondary vessel only so much SO<sub>2</sub> is used as proves enough to bring down the pH to a steady level of about 6.9-7 pH. Only a very small quantity of milk of lime is therefore added in the 2nd vessel. This can thus be considered as a correcting vessel to make the final adjustment in the pH of the liquor.

It was found that the dispersion of milk of lime and SO<sub>2</sub> in the juice left much to be desired when relying

on the agitation caused by the ebullition of the gas mixture and by the external circulator of the sulphitation vessel. The lime being added in the suction of the circulator (pump) caused the impeller to be coated with a precipitate to the extent of forming one solid body which badly influenced the quantity of liquor which could be displaced. That, in turn, caused the flow of milk of lime in the piping to stagnate and irregularities in pH were unavoidable, as a time lag was created in the pH correction.

Agitators consisting of two propellers mounted on a shaft were fitted to the vessels to create an immediate dispersion of the milk of lime, the addition of which has been arranged directly into the bulk of the liquor inside the vessel.

Originally the agitator shafts were fitted horizontally and although the working was quite effective, the leaking glands made it necessary to fit the shafts vertically, suspended from the top of the vessel. The opposite pitch of the two propellers on the same shaft causes a very fierce turbulence whereby the contact of lime and juice is intensified and the reaction with the now quickly dispersed gas is speeded up. This leads to a reduction in the fluctuation of the signal to the pH controller. The charts obtained give an illustration of the difference in results (figs. 3 and 4).

A certain amount of colour in the liquor was observed even following intensified sulphitation. Test with apoxiresin paints as an internal surface coating of various units in the plant indicated that rust was the cause to a large extent. As the paint was found to come off in sheets on corroded and hard-to-clean surfaces, fibre-glass coating was successfully tried out.

The above changes have had considerable effect on the results since 1959, when the refinery was commenced.

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**Dr. Douwes Dekker** (in the chair) said the paper was of interest to all who were considering applying, or were applying, the sulphitation system of refining raw sugar. So far there were not many refineries using the system but he felt that it could be used with advantage to a larger extent than at present.

He asked the author about the effect of high pH on the melt and the filtrability of mud. He also asked if it were so that the destruction of reducing sugars in a high concentration of melt was low and how the author arrived at this conclusion.

**Mr. Thumann** replied that in high concentration there was less ionisation and this led to his conclusion that there would, under such circumstances, be less chance of decomposition and formation of colour from reducing sugars.

**Mr. Rault** pointed out that in carbonation refineries, especially with the use of low CO<sub>2</sub> content flue gasses, the pH of the liquor had to be kept rather high in order to allow a better gas absorption and use a sufficient amount of clarifying matter.

He wanted to know the effect of colour and reducing sugar destruction by this practice.

**Mr. Thumann** replied that in the carbonation refineries the temperature was much lower.

**Mr. J. D. Alexander** said that it was found in the central refinery after affining and the first carbonation, if reducing sugars were destroyed, trouble was experienced later in the process. If reducing sugars were destroyed this led to more troublesome after-products. Colour formation and more calcium products resulted, which caused more trouble than the reducing sugars from which these were formed.

**Mr. Hulett** asked how the pH was controlled and what sort of fibre glass lining was used in the tanks.

**Mr. Thumann** said the pH control consisted of a Leeds and Northrops controller with a "splitter-box" for dosing the lime and the sample was taken from the discharge of the tank. A pressure head was thus provided and the inlet of lime was well controlled as could be seen in Figure 4, showing this last season's chart, which compared so much more favourably with Figure 3. The lack of such a head had led to trouble previously as too much lime was left in the long milk of lime inlet pipes and it took a lot of time before this could mix with the liquor, causing a certain time lag at the pH controller. He did not know what type of fibre-glass lining was used.

In reply to Mr. Hulett he said that when the process was stopped the plant was kept in circulation and lime added a short while until all of the circuit registered a pH of 7.0 and this required the use of only one valve. Usually, however, if processing stopped for a short time this circulation was kept going until processing started again.

As the sketch showed, the gas could be discharged to atmosphere when the process was stopped, as could the blower feeding air to the burner. The burner kept hot for a couple of hours so it was easy to start again.

**Mr. Ashe** said the sketch showed the vessels were in two parts. Corrosion took place above the overflow box and there was more below this point as the rest of the tank was full of liquor. The gland on the circulating pump caused trouble and its leaking meant the whole tank had to be emptied. It was therefore proposed that it would be better, in any future installation, to put a co-axial pump at the top of the pipe leading to the splitter-box instead.

The design of the pH electrode vessel was very important. The liquor had to be led through the bottom and discharged from the top. If the inlet was at the side some stagnation occurred causing sedimentation which led to the irregularity shown in Figure 3.

**Mr. Boic** asked if with a low density, liquor, as in juice sulphitation, a similar coating of stirrers would occur. He also wanted to know if high speed of the impellers would not cause a break-up of the floc.

**Mr. Thumann** replied that in the melt sulphitation the Brix of the liquor was about 69 deg. while the lime was about 7 deg. Be. When the entrance of the lime was where the raw liquor inlet is shown in the sketch, a build-up on the stirrers was experienced. It was found necessary to have a deep pitch on the impellers. The stirrers revolved at only 50 r.p.m. so there was no danger of breaking up the floc, but at the same time the turbulence in the area between the impellers was ample to ensure proper mixing.

**Mr. Ashe** said it was found necessary to feed the lime at a higher point than that of the liquor, other-

wise the supply was irregular leading to over-liming and under-liming alternately.

**Mr. Chiazzari** said that in the carbonation refining process at Gledhow, to avoid a high pH, it was found necessary to admit the lime, gas and liquor simultaneously.

**Dr. Douwes Dekker** asked if there was any data showing the amount of SO<sub>2</sub> lost, to which Mr. Thumann replied that while no figures were available, the loss was apparently very slight. No smell of SO<sub>2</sub> was noticeable, although some loss due to the formation of SO<sub>2</sub> was shown by the white appearance of the emission from the chimneys above the vessels.