# REPLACEMENT OF SULPHUR BURNER WITH LIQUID SULPHUR DIOXIDE SYSTEM AT THE MALELANE REFINERY.

[1]M MOODLEY, [1]DJ BEKKER, [1]PJ PIENAAR AND [2]R PILLAY

[1]*Transvaal Sugar Limited, PO Box 47, Malelane, 1320*
[2]*Kynochem (Pty) Ltd, Moddercrest, High Street, PO Modderfontein, 1645*

## Abstract

Malelane has a conventional carbonatation/sulphitation refinery. The major proportion of the suspended solids is removed at the carbonatation station. The liquor leaving the carbonatation station is then treated with sulphur dioxide gas which has been produced on site by burning crude sulphur. This system of producing sulphur dioxide had a number of disadvantages, *viz.* inefficient conversion of elemental sulphur, inconsistent liquor pH and leakage of gas into the refinery. A new system, which uses sulphur dioxide gas from a one ton cylinder, has been successfully implemented. The process installation, the control philosophy and some of the problems experienced are described. The cost of the new system is also discussed.

*Keywords:* liquid sulphur dioxide, fine liquor, sulphitation, Malelane

## Introduction

During the past two years there has been a great effort at the Malelane refinery to improve the quality of refined sugar. Of concern was the inconsistent fine liquor pH, which would affect the quality of the refined sugar produced.

One of the quality specifications for refined sugar is residual sulphur dioxide. The pH of the fine liquor from which the sugar is crystallised has an influence on this parameter. Laboratory sulphitation tests done by the SMRI (Lionnet and Moodley, 1991) showed that the sulphur dioxide content of the liquor depends on the pH of the liquor. Test results (Figure 1) indicated that the lower the pH of the fine liquor, the greater the sulphur dioxide in the liquor.

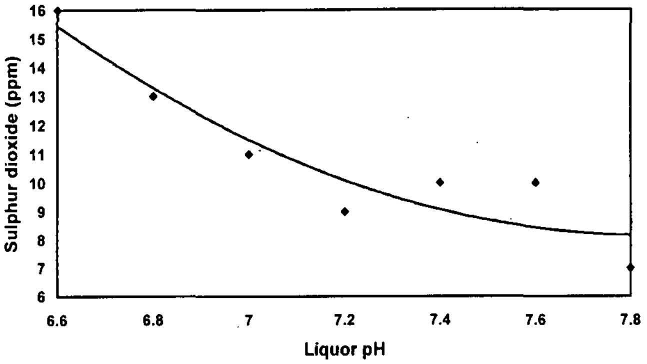

**Figure 1.** Results of sulphitation tests showing the relationship between sulphur dioxide content and pH of liquor.

In addition, for pH values below 7, increasing quantities of soluble calcium bisulphite ($Ca(HSO_3)_2$) are formed instead of insoluble calcium sulphite ($CaSO_3$) (Honig, 1953). On the other hand if the liquor pH is too high, incomplete precipitation of the calcium would occur. This would then cause the ash in the refined sugar to increase. To produce refined sugar with acceptable sulphite and ash levels, the pH of the fine liquor should therefore be between 7,0 and 7,2.

Results of a refinery survey done by the Sugar Milling Research Institute (SMRI) (Moodley and Hastie, 1994) at Malelane showed that the pH of the fine liquor was lower (6,6) than it should have been. The SMRI team also noted that there was a strong odour of sulphur dioxide in the refinery. A further survey done by the SMRI (Moodley, 1994) at Malelane showed the fine liquor pH to be too high (8,0). Because of the erratic movement of the fine liquor pH from low to high, the SMRI recommended that Malelane consider upgrading the sulphitation station. The Noodsberg refinery has successfully implemented a system using liquid sulphur dioxide (Sanders and Getaz, 1992). It was therefore recommended by the SMRI that Malelane evaluate this option.

A study done by Stolz (1994) concluded that it was not economical to implement the liquid sulphur dioxide system at the Malelane refinery and recommended that a new sulphur burning system be installed. In the study Stolz assumed that 100% of the generated sulphur dioxide gas was absorbed by the liquor. In practice less than 50% of the gas that is generated is absorbed. This is due mainly to gas being lost to the atmosphere.

Based on the recommendation of Stolz (1994), a new sulphur burning process was installed. Although the system was an improvement on the old burning process, the following problems were encountered:

- Inefficient conversion of elemental sulphur to sulphur dioxide gas.
- Leakage of sulphur dioxide gas into the refinery.
- Blockage of venturi.
- Inconsistent hourly fine liquor pH.

Due to the above factors and despite the increase in costs, it was decided to replace the sulphur burning system at the Malelane refinery with a liquid sulphur dioxide.

## Experimental

For the liquor and refined sugar solution (30° brix) pH measurements, the samples were first cooled in the laboratory to 20°C and the pH values were then measured. The sulphur dioxide content of both the liquor and refined sugar were measured by the Rosaniline method (Anon, 1985).

## Discussion

*Description of the sulphur burning system*

Sulphur dioxide is produced by burning crude sulphur which is added manually at regular intervals to the burner. The generated gas is drawn into the clear liquor through a venturi. The venturi effect results in an induced draft across the burner, increasing air flow and therefore the combustion rate of the sulphur. The liquor then flows into the sulphited liquor tank. From there the liquor can be pumped to the filters or it can be recirculated through the venturi, depending on the pH of the liquor. When the liquor pH is high (>7,0), the speed of the recirculation pump is automatically increased and more liquor is pumped through the venturi, thus increasing air flow across the burner and absorbing more sulphur dioxide. When the liquor pH is low (<7,0), no liquor is pumped through the venturi and no sulphur dioxide is absorbed. A flow diagram of the system is given in Figure 2.

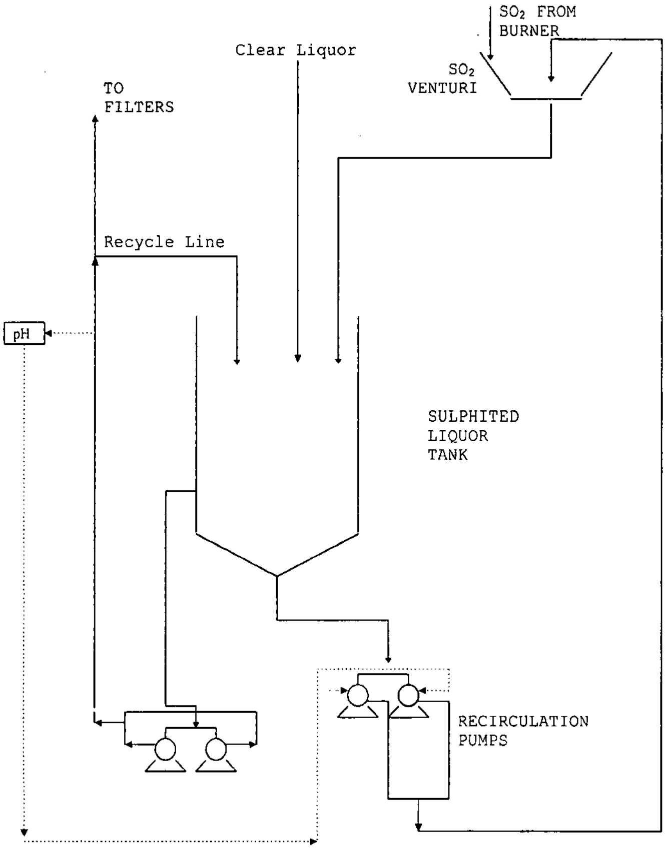

**Figure 2.** Sulphitation station at Malelane.

*Problems experienced with the sulphur burning system*

Although the average daily pH of the fine liquor was fairly consistent, the hourly liquor pH was erratic. There was a constant odour of sulphur dioxide in the refinery, due to the inefficient burning process and also to leakage of sulphur dioxide when the refinery stopped. On several occasions the venturi clogged with unburnt sulphur, which required a refinery stop to clean the venturi. Mixing efficiency of the venturi was also inadequate.

*Description of the liquid sulphur dioxide system*

In this system, the sulphur dioxide is purchased in one ton cylinders. The gas is dosed from the cylinder into the liquor line through the Hastings mass flow controller which operates on a unique thermal electric principle and eliminates the need for pressure and temperature compensation, thereby allowing the controller to be calibrated directly in mass units. It has a built-in automatic metering solenoid valve which allows the controller to dose the correct mass of sulphur dioxide proportional to the output signal from the pH controller. The signal from the flow controller can also be used to total the consumption of the gas thus giving an indication as to when the cylinder should be empty.

The one ton sulphur dioxide cylinder is connected to a manifold via flexible hosing. There are two valves that isolate the supply of gas into the system, i.e. one on the cylinder and one on the manifold). The sulphur dioxide liquid is normally stored at a pressure of about 450 kPa (gauge). An electric blanket placed around the cylinder in operation supplies energy to the liquid sulphur dioxide, and this causes the liquid to flash into the gaseous form. There is a vent line and a pressure gauge on the manifold. Prior to changing from an empty to a full cylinder, the operator has to vent the system.

The manifold and the 12 mm delivery line to the liquor are constructed of 316 stainless steel. The sulphur dioxide pressure is regulated to a pressure of 200 kPa prior to entering the solenoid valve of the Hastings mass flow meter. The flow meter will dose 0-18 kg/h sulphur dioxide depending on the pH of the sulphited liquor. A non-return valve is installed in the line before the dosing point to prevent liquor from entering the gas line.

The sulphited liquor then flows into the sulphited liquor tank. The pH of the liquor is measured prior to the liquor entering the tank. The pH signal is then transmitted to the mass flow meter where the dosage of gas is controlled (Figure 3).

*Problems experienced with the new system*

Several problems were experienced during the commissioning phase. Freezing of the gas in the one ton cylinders when the ambient temperature was low was prevented by the installation of the electric blanket. Blockages of the non-return valve were alleviated by a modification to the dosing point. When the mass flow meter clogged with tiny dirt particles, the instrument was cleaned and the problem did not occur again. The pressure regulator also becomes clogged with dirt particles and therefore has to be cleaned periodically.

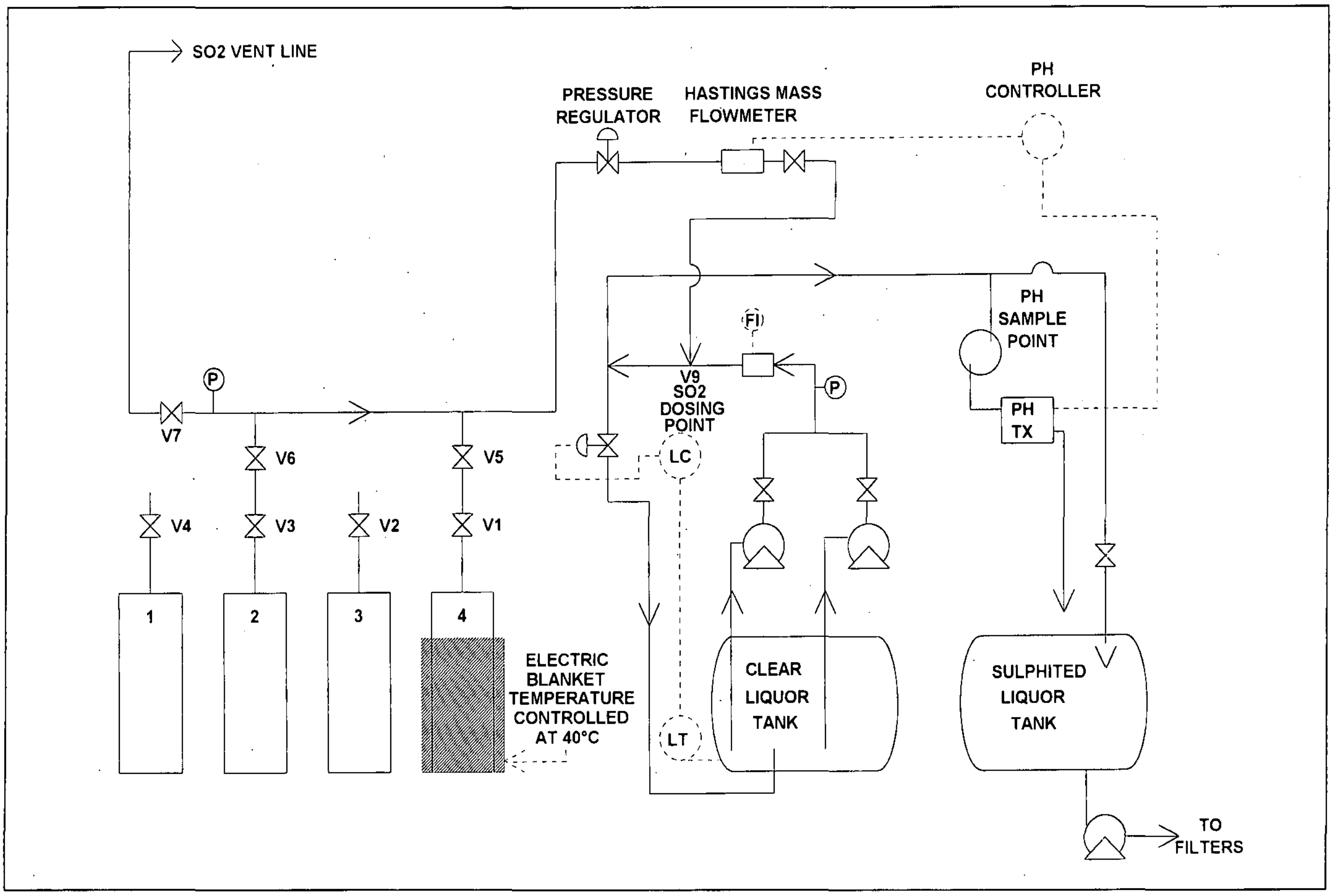

**Figure 3.** Liquid sulphur dioxide system at Malelane.

## Results

### *Liquor pH*

Variation in the sulphur dioxide gas production from the burner caused the pH of the liquor to vary considerably. The results for a typical day are illustrated in Figure 4, showing that the fine liquor pH was very erratic. This would have an adverse effect on the quality of the refined sugar. The liquid sulphur dioxide system was installed and commissioned during October 1996. The pH of the fine liquor during the commissioning period is shown in Figure 5. The change from crude sulphur burning to liquid sulphur took place after 26 hours.

Results indicate that when the sulphur burner was in operation, the fine liquor pH was erratic and varied from 5,3 to 7,9. The pH control improved with the dosing of sulphur dioxide from the cylinder. The flow of gas was more consistent, with the result that the liquor pH did not vary widely.

### *pH of the refined sugar solution*

The wide variation of the fine liquor pH caused by the inconsistent supply of gas from the burner also resulted in the pH of the refined sugar solution being too high (Figure 6). The liquid sulphur dioxide system was commissioned during week 27. Due to improved control, the pH of the refined sugar solution was reduced to around 7.

A decrease in refined sugar pH would have a positive effect on sugar colour. The turbidity of the refined sugar would also decrease. The residual sulphur dioxide in refined sugar as analysed by the SMRI on a weekly basis for the past four years is plotted in Figure 7. The results indicate that there has been a definite decrease in sulphur dioxide in refined sugar produced by the Malelane refinery due to the improvement of the pH control at the sulphitation station.

### *Installation cost*

The cost of the complete installation was about R41 000.

### *Gas absorption*

With the commissioning of the liquid sulphur dioxide system the consumption of gas dropped from 270 to 85 ppm on refined sugar (Appendix 1). This meant that 69% of the gas generated during the burning process was lost to the environment. Apart from being a waste of gas, this posed a safety threat to refinery personnel.

### *Chemical costs*

Installation of the liquid sulphur dioxide system has lowered the cost of dosing sulphur dioxide by 11%.

## Conclusions

The installation of the liquid sulphur dioxide dosing system has improved the quality of refined sugar at the Malelane refinery, due to the consistent pH of the fine liquor.

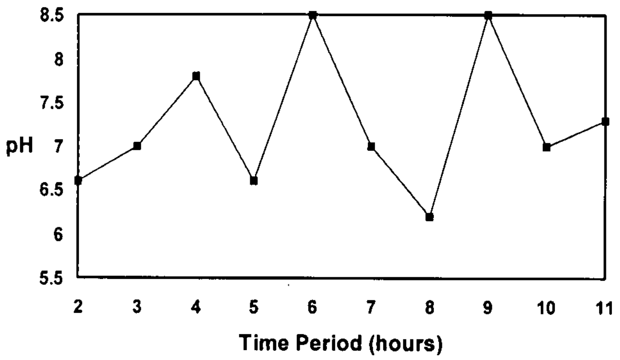

**Figure 4.** Variation in fine liquor pH over a typical 11-hour period.

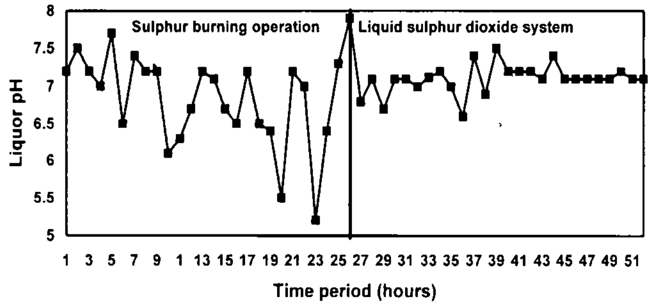

**Figure 5.** pH of fine liquor during commissioning of system.

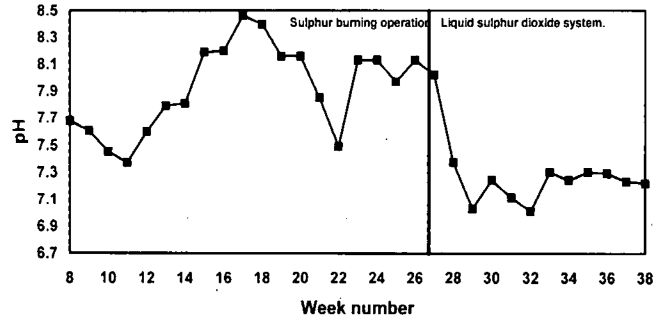

**Figure 6.** pH of refined sugar solution.

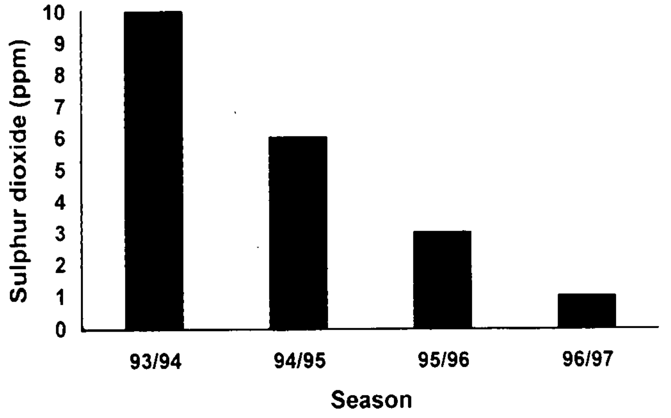

**Figure 7.** Residual sulphur dioxide in refined sugar, 1993-94–1996-97.

The improved dosing system decreased consumption of sulphur dioxide gas by 69%, and effected a saving of 11% in dosing costs.

No odour of sulphur dioxide is present in the refinery, due to the improved control system and non-leakage of gas. When the refinery stops, the gas flow controller immediately stops dosing sulphur dioxide to the liquor. Mixing between gas and liquor has also improved.

Full scale tests have shown that liquid sulphur is an economically viable option when compared with the sulphur burning process.

## Acknowledgements

Many people from TSB have contributed in large measure to this project. The authors would like to thank I Singh, P Hattingh and P Gouws for assisting with this project. The help received from all the refinery operating and maintenance personnel in making this project a success is highly appreciated. Thanks are also due to TSB management for permission to publish this paper. The advice and assistance received from GRE Lionnet of the SMRI is also appreciated.

## REFERENCES

Anon (1993). Laboratory Manual for South African Factories (including Official Methods). *S Afr Sug Technol Ass*: 436 pp.

Honig, P (1953). *Principles of Sugar Technology* Vol. 1. Elsevier Publishing. pp 601-613.

Lionnet, GRE and Moodley, M (1991). Phospho-sulpho flotation tests. Sugar Milling Research Institute Technical Note No. 28/91.

Moodley, M (1994). Refinery survey at Malelane. Sugar Milling Research Institute Technical Note No. 17/94 (RC).

Moodley, M and Hastie, A (1994). A report on a preliminary survey of the Malelane Refinery. Sugar Milling Research Institute Technical Report No. 1694 (R/C).

Sanders, SS and Getaz, MA (1992). Sulfo-phosflotation clarification at Noodsberg. Illovo Sugar Ltd Technical Conference, Durban. February 1992.

Stolz, N (1994). The feasibility of liquid sulphur dioxide. TSB Internal Report. 11 pp.

## APPENDIX 1

## Stoichiometry of sulphur dioxide and milk of lime

$$S + O_2 \rightarrow SO_2\,(g) \tag{1}$$

$$SO_2\,(g) + H_2O \rightarrow H_2SO_3 \tag{2}$$

$$Ca\,(OH)_2 + H_2SO_3 \rightarrow CaSO_3 + 2H_2O \tag{3}$$

The following comments can be made regarding the above equations:

- 32 grams of crude sulphur produces 64 grams of sulphur dioxide gas (equation 1).
- 64 grams of sulphur dioxide are needed to react with 74 grams of calcium hydroxide.

### *Gas absorption*

During the period when the burner was in operation the consumption of crude sulphur was 135 ppm on refined sugar. This is equivalent to 270 ppm of sulphur dioxide gas on refined sugar.

With the commissioning of the liquid sulphur dioxide system, the sulphur consumption decreased to 85 ppm.