

# SOME NOTES ON THE FUNCTIONING OF SULPHUR TOWERS

By P. J. LAUBSCHER:

The tendency in industry is to replace batch processing of materials by continuous processing, because the continuous processes have many advantages over the former. In any process it is always our aim to keep within narrow limits to the optimum conditions that have been determined for the process. This is especially true for the juice clarification process in the sugar industry, where only small deviations from the optimum pH values and temperatures can be allowed.

The sulphur tower is used for the continuous sulphuring of limed mixed juice, and the object of the present investigation was to find out what variations from the optimum pH we get in juices sulphured by them. In the second place, the efficiency of the sulphur dioxide absorption was determined by analysis of the gas entering and leaving the tower. The tests done were simple and they were done only at three factories, but it is believed that they served the purpose of the investigation. The effect of variations in the pH of juice sulphured by the tower upon the clarification process was not investigated.

## Design and Operation of the Sulphur Towers.

All three sulphur towers worked on were more or less similarly constructed. Their dimensions are given in Table 1. The towers are square and the walls are made of wood. Inside, they have a system of trays through which the juice falls. These consist of parallel, horizontal-running, wooden bars or boiler tubes. The tubes or bars of consecutive trays run in perpendicular directions. On top of the towers are chimneys through which the exhausted gas is blown by a steam or air injector.

Limed mixed juice enters at the top of the tower just below the injector and falls on a device which spreads it. The gas enters at the bottom through a slit, 1 inch to 2 inches wide and in length 1 foot shorter than the width of the tower, in the bottom of the 8-inch pipe bringing in the gas from the sulphur burner.

The sources of  $\text{SO}_2$  are rotary sulphur burners from which the gases pass to a combustion chamber for the completion of the combustion. For cooling, the gas pipes pass through water, but at one factory the gas was cooled by passing for a fairly long distance through air. In the factory B (see Table 1), the gases also pass through a scrubber to remove any sublimed sulphur.

TABLE I.

	Factory A (2 towers)	Factory B	Factory C
Height of tower	45'	25'	40'
Width of tower	4' 6"	4'	3' 6"
Dimensions of chimney	4' 9" x 9" x 7"	—	—
Injector ...	$\frac{1}{2}$ " steam pipe	$3\frac{1}{2}$ " air pipe	1" steam pipe
Trays—			
lower half	2" tubes 1" apart	—	2" tubes $\frac{1}{2}$ " apart,
top half	2" tubes 1" apart	—	2" wooden rods 2" apart
Distance between trays	2'	2'	3'
Dimensions of burner	12' 6" x 1' 8"	8' x 1' 8"	6' 6" x 2'
Dimensions of combustion chamber ...	3' 6" x 3' 6" x 8'	4' x 4' x 3' 9"	4' x 4' x 9'
Cooling ...	9' water	5' water	air
Tons mixed juice handled per hour ...	117	56	100

**Note:** Factory B uses an air injector just below the neck of a Venturi tube of 5 inches minimum diameter.

At Factory A the sulphur burner is hand-fed and at such a rate that  $\pm 250$  lbs. of sulphur is burnt per hour, the operator judging the time for feeding from the flame he sees through a slit between the furnace and the combustion chamber. The lime in mixed juice is so regulated that the pH at which the sulphured juice comes out is  $\pm 7.3$ . Indicator paper is used for this purpose; the checking of the pH and the adjusting of the lime valve is done more or less once in 10 minutes.

At Factory B, sulphur is fed mechanically and continuously to the burner. The lime added is so regulated that the juice, after sulphuring, has a pH of 7.6 to 8. This test is done more or less once every five minutes, with a suitable indicator.

The practice at factory C is to lime to 11 pH, which is done with the help of a Beckman pH meter. The burner is hand-fed and the sulphuring is regulated by adjusting the steam flow through the injector when necessary. The pH aimed at in this factory is 5.9.

From the sulphur towers the juice flows into tempering tanks, where phosphoric acid is added, usually to give a  $\text{P}_2\text{O}_5$  content of 300 mg. per litre, and the pH is then tempered with lime to 7.3 to 7.6; the juice is then settled and filtered, after which the pH is 7.0.

### Experimental.

The mills' test laboratories do pH determinations on hourly composite samples of sulphured juice and, as these showed only small variations, the determination was not repeated. pH determinations were done on samples of sulphured juice obtained from the trough bringing the sulphured juice to the tempering tanks; such samples were collected at intervals of 5 and 1 minutes. The determinations were also done on samples collected from each tempering tank when filled up before any phosphoric acid or lime was added. For the determination of the pH of the cooled samples a Beckman pH meter was used.

The apparatus for the determination of the SO<sub>2</sub> content of the gas entering and leaving the tower was a flask of 1 litre  $\pm$  capacity fitted with a rubber stopper through which pass the stem of a dropping funnel reaching to the bottom of the flask, and a short glass tube connected with a rubber tube to a rubber bulb pump which draws the gas sample.

With the tap of the dropping funnel open, gas is pumped through the flask till all air is displaced; then the tap of the dropping funnel is closed. More gas is then pumped into the flask to get the gas at a pressure well above atmospheric when the connecting rubber tube is shut with a pinch cock. After cooling to room temperature, the excess gas is allowed to escape slowly, leaving the flask filled with gas at room temperature and at atmospheric pressure.

A known volume of a standardised N/10 iodine solution is now washed into the flask through the dropping funnel and shaken up with the gas. After making sure that all the SO<sub>2</sub> has reacted with iodine, the excess iodine is titrated against a standard N/10 sodium thiosulphate.

For the gas entering the tower this method was found very satisfactory, but for the gas leaving the tower it was less suitable, because gas after passing through the juice is saturated with water vapour at a high temperature which condenses in the flask, and, due to the high solubility of SO<sub>2</sub> in water, one can expect that the determined SO<sub>2</sub> by this method, for the gas leaving is high. It was found, however, that the SO<sub>2</sub> content of the gas leaving was so low that a great error in this determination does not seriously affect the resulting calculated efficiency.

The efficiency of the tower can be calculated from the SO<sub>2</sub> percentages in the gas entering and leaving the tower by making use of the formula:—

$$\text{Per cent. efficiency} = 100 \frac{(100p_1 - 100p_2)}{p_1(100 - p_2)}$$

where  $p_1$  = percentage pressure due to SO<sub>2</sub> in gas entering tower,  $p_2$  = percentage pressure due to SO<sub>2</sub> in gas leaving tower.

Seeing that this test on the efficiency runs only for a few minutes, it does not take into account sulphur lost through stoppages and mechanical losses.

### Results.

Figure 1 is a graphic representation of the pH values of the juice versus time at the factories A, B and C. For A and B, samples were collected every five minutes and for the curves C<sub>1</sub> samples were collected every one minute. C<sub>2</sub> are the pH's of samples collected from each tempering tank when filled up. (The time required for a tank to fill up was from 6 to 7 minutes).

Table 2 below gives the results of the efficiency determinations on the towers:—

TABLE 2.

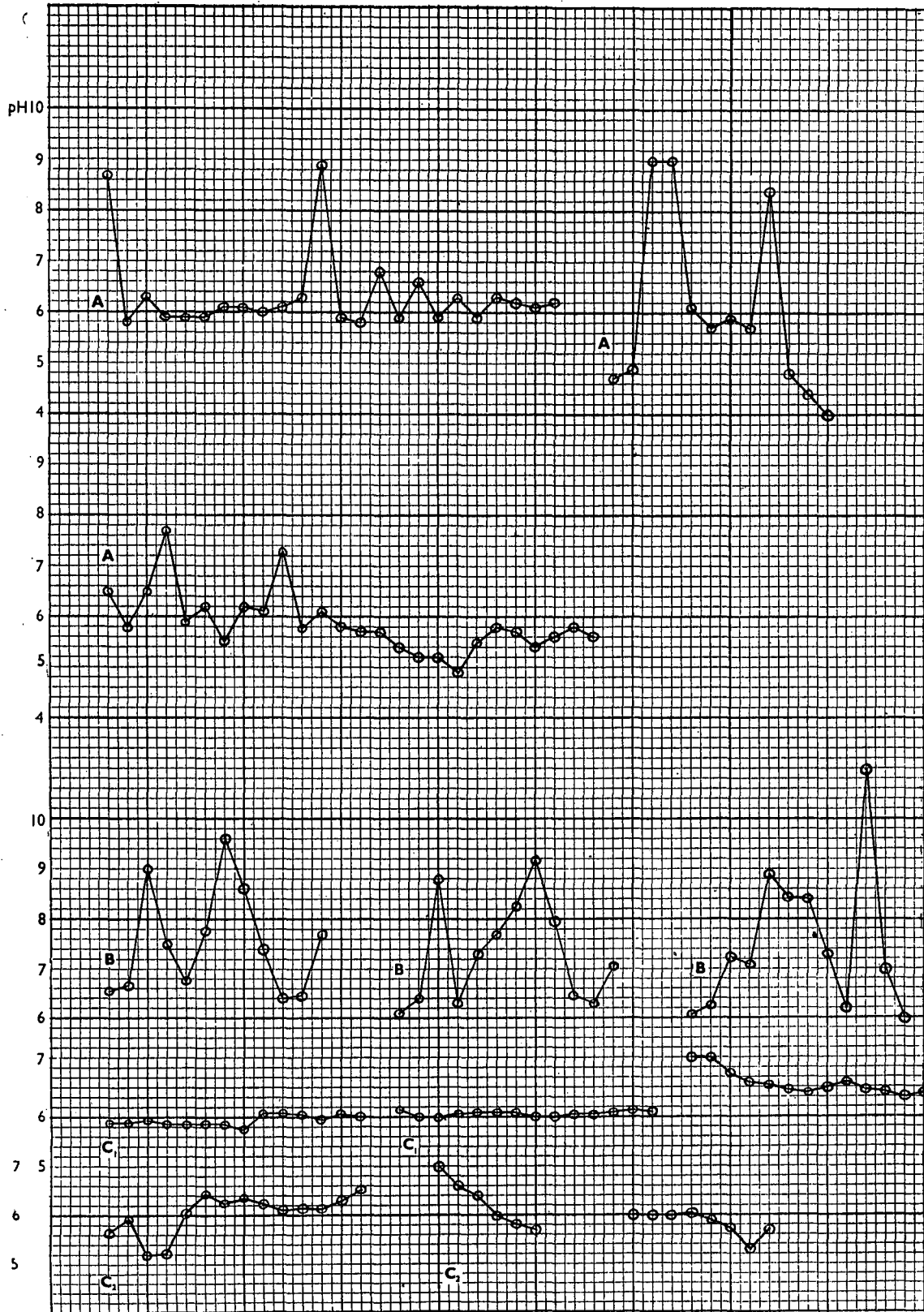
Factory.	Per cent. SO <sub>2</sub> in gas entering.	Per cent. SO <sub>2</sub> in gas leaving.	Efficiency per cent.
A ... ..	6.88	0.23	96.8
A ... ..	6.53	0.73	89.4
B ... ..	11.12	0.03	99.77
B ... ..	12.97	0.02	99.87
B ... ..	12.62	0.02	99.86
C ... ..	10.7	0.01	99.9
	7.88	0.01	
	7.2		

### Discussion of Results.

The fluctuations in the pH of the juice from the sulphur tower, from whatever pH is aimed at, is rather high and undesirable for good work. Fluctuations in the pH of the sulphured juice can be due to one of the following causes:—

- (1) Changes in the rate of flow of (a) juice or (b) gas through the tower.
- (2) Variations in the SO<sub>2</sub> content of the gas entering the tower caused either by (a) or irregular feeding of sulphur to the furnace.
- (3) Variations in the lime content of the juice going to the tower.
- (4) Variations in the properties of the juice. Semi-quantitative experiments by Mr. Dymond showed up to 100 per cent. variations in the amount of lime required to produce the same pH rise in equal quantities of juice.

It is interesting to note that for the curves C<sub>2</sub> (samples taken from successive tempering tanks) we get greater variations than for C<sub>1</sub> (one-minute samples).



A & B pH OF SAMPLES TAKEN AT FIVE MINUTE INTERVALS  
 C, pH OF SAMPLES TAKEN AT ONE MINUTE INTERVALS  
 C<sub>1</sub> pH OF SAMPLES TAKEN FROM SUCCESSIVE TEMPERING TANKS

The large excess of air in the sulphur dioxide air mixture seems undesirable, because excess oxygen favours sulphur trioxide formation and the thorough contact of the hot juice with oxygen may be harmful to the juice.

#### Summary.

The variations in pH of juices sulphured by the sulphur towers at three factories were determined.

The efficiencies of the sulphur towers were determined by analysis of gas entering and leaving the towers.

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The PRESIDENT said that the subject dealt with was very important in all sugar growing countries, but particularly in South Africa, where it seemed impossible to make even raw sugar without the use of sulphur dioxide.

Mr. DUCHENNE noted that the author had recorded an efficiency of 99.9 for factory C. This was Umfolosi, where he was process manager. He considered the actual efficiency much less than 99, for he had found the overall absorption to be about 75 per cent. by measuring the sulphites in the juice before entering, and after leaving, the sulphur tower and the sulphur dioxide produced. It was necessary to measure the sulphites in the juice entering the tower for those factories which had Oliver-Campbell filters and which returned the filtrate to the raw juice.

Mr. ELYSEE referred to the author's remarks about a large excess of air in the gas entering the tower being undesirable, because of the excess oxygen favouring sulphur trioxide formation. With the rotary furnaces now in use with secondary and even more combustion chambers, a large amount of heat was evolved which might well give too high a temperature, thus leading to dissociation of the sulphur dioxide with subsequent formation of sulphur trioxide. He had found that after working for a while, more and more sulphur was required to reach a certain sulphur dioxide content in the juice.

Furthermore, he had had to abandon water-cooling of the pipes conveying the hot gas, as any attempt to cool them led to rapid corrosion, and having to replace a corroded pipe was very inconvenient in mid-season. His experience was that when the pipes corroded and water entered, the factory recovery was lowered by about 2 per cent. This had occurred about four times. A loss of 2 per cent. in recovery in a factory was very serious, and unfortunately when this loss was determined it was too late to change a pipe and it had to remain until the following week-end. This meant the loss in recovery continued for another week.

Mr. CHIAZZARI said that there was no mention in the paper of the temperature of the juice entering the tower. He was of the opinion that the absorption of sulphur dioxide was affected by the temperature of the juice.

Mr. LAUBSCHER regretted that he had not recorded the juice temperatures, but in any case he did not think that a variation in this of ten degrees would have a noticeable effect on efficiency.

Mr. DYMOND thought that the possibility of using liquid sulphur dioxide should be further explored, for it had many advantages. Enquiries he had made had resulted in his calculating that the cost of using the liquid gas would be approximately the same as the cost of the sulphur and the burning of it.

Dr. DOUWES-DEKKER said he had discussed the question of liquid sulphur dioxide with the manufacturers, and had come to the conclusion that the cost would be much higher, and that at the moment it was not possible to consider using liquid sulphur dioxide.

He wished to ask Mr. Elysee what kind of pipes he employed, and if it were not usual in Natal to use lead pipes.

Mr. ELYSEE replied that his experience had shewn him that the excessive heat produced with his sulphur-burning plant would melt lead pipes.

The pipes he had used were of cast-iron, 8 inches in diameter, the vertical ones being 25 feet long, and the horizontal ones 20 feet. Both were water-jacketed.

The horizontal pipe corroded first and he eventually abandoned the use of a pipe in this position and used a brick conduit instead.

Dr. DOUWES-DEKKER drew attention to the fluctuation shown in the graphs. In the first curve, where the average was about 6.5 pH, the variation was from 9 pH to 4 pH. This seemed rather unsatisfactory, and he thought the best way of pH control should be thoroughly studied.

Mr. DUCHENNE agreed that this was most important. During the last three years he had been experimenting with, and applying a pre-liming pH of above 10.0, using a pH recorder. This pH had caused a greater reduction of impurities and colouring matter. He hoped to put in a "Foxboro" automatic pH meter, recorder and controller to control also the pH of the juice leaving the sulphur tower this season.

In reply to a question by Dr. Douwes-Dekker, he stated that at his factory new sulphiting apparatus had been installed, but the machine had to be used in connection with a sulphur dioxide cooler. As the latter was still in the experimental stage, he could not give any figures as to the efficiency of the

machine. This apparatus was of the "Quarez" type, and had a six-jet injector set over a tailpipe 25 feet high. He hoped to lose less sulphur dioxide with this device.

Mr. RAULT considered figures of over 99 per cent. efficiency for a sulphur tower to be rather high, and in view of the difficulty of testing waste gases he thought testing the juice for sulphur dioxide to be a more practical method. For a factory where the sulphur tower was only 25 feet high the efficiency seemed very high, as this would seem to indicate that a tower 40 or 50 feet high was unnecessary.

As far as the variation in pH of juice coming out of the sulphur tower was concerned, it should be remembered that this was not a final stage of chemical treatment of juice. This was always brought to the desired end-point by lime and phosphoric acid. There is no evidence that liming first and sulphuring after produces very different results from sulphuring first and liming after, when the final point and the total quantity of lime used is the same.

Dr. DOUWES-DEKKER considered that it should be our aim to adhere strictly to detail in any method of juice clarification. He did not think it was enough for only the end-point to be correct. The carbonation process was one of the best-studied processes, and we knew exactly the required temperature and pH limits during the juice treatment.

Mr. PHIPSON said it was practically impossible to measure sulphur tower efficiency in a factory where the Oliver-Campbell filtrate was returned to the pre-liming tank, unless the weight of the filtrate was accurately known.

As far as juice temperatures were concerned, he related that during the last season he had found that there was a drop in purity from mixed juice to syrup in the early stages of the season. At that time the raw juice was heated to 140°F. When the temperature was reduced to 130°F., the purity of the syrup was increased to as high as one degree above that of the mixed juice.

Mr. DUCHENNE was glad to note that so many factories in South Africa were paying attention to the pH of the primary liming. This was not done in the past. At Umfolozi they were pioneers in this work. Many colloids and other impurities were precipitated during the primary liming, but he was of the opinion that much of that good work was nullified by the subsequent neutralisation.

He was glad to see that the efficiency of the sulphur tower, as used here, was so high. The overall efficiency could not be measured by the tower, however. What also counted was the sulphur dioxide

in the juice compared with the amount of sulphur burned, and he considered that, to determine the efficiency, testing of the juices for sulphites was a good method. By this method he had found the overall efficiency to be about 75 per cent.

Mr. RAULT thought that the Natal Estates towers gave an efficiency of about 90 per cent.

Mr. ELYSEE wondered if one got greater efficiency from a longer tower than from a shorter one. The shorter tower certainly gave more flexibility. At Amatikulu the concentration of the gas could be regulated and he had noticed that incrustations in the tower depended upon the concentration of the gas.

He did not return filtrate through the tower, and thus the efficiency of the latter could be easily calculated.

The PRESIDENT enquired if the author had any analytical figures to illustrate the amount of sulphur trioxide formed during the burning of the sulphur.

Mr. LAUBSCHER replied that he did not have such figures, but since the reaction  $2\text{SO}_2 + \text{O}_2 \rightleftharpoons 2\text{SO}_3$  is exothermic when going from left to right, low temperatures favour the formation of sulphur trioxide. It is also clear from the above reaction that a high oxygen content of the gas mixture favours sulphur trioxide formation.

Mr. DYMOND considered that work should be done to determine the conditions under which nodules of calcium sulphite were formed, as compared with those under which crystals were formed.

Mr. CARTER asked what the final effect of cooling the gas was. He had found that at times he could not get any juice to clarify satisfactorily, and had resorted to sulphuring first and liming afterwards. He would revert again to the usual process but might have to change the pH. Certain juices caused great difficulty and he did not find that the standard process would work equally well with all juices.

Mr. ELYSEE stated that when he cooled the sulphur dioxide gas he could easily control the sulphur dioxide content of the sugar to below 70 parts per million. Without cooling he could not do that.

The PRESIDENT said it was noticeable that recently some factories tended to have a sulphur dioxide content of sugar above the standard laid down some years ago. The question of this sulphur dioxide content might rise again and we should be prepared to meet it.

Mr. DUCHENNE enquired why it was that sugar was allowed the very low sulphur dioxide content of

70 parts per million, while dried fruits could be exported with 3,000 to 6,000 parts per million.

The PRESIDENT thought that the sugar industry was placed under a severe handicap as far as the sulphur dioxide content of its main product was concerned. In some foodstuffs it was not used in the

manufacturing process but was added as a preservative. Naturally, if the sulphur dioxide content was found to be high, suspicion was cast on the quality of the original material. That was not the position as regards sugar, where sulphur dioxide was merely a residue of manufacture and not added subsequently, but it was doubtful if the authorities always realised that fact.