

# PRACTICAL OBSERVATIONS IN THE USE OF PHOSPHORIC ACID AND ACTIVATED CARBON AS AN AID TO COLOUR REMOVAL IN REFINING

By G. JONES

*Transvaalse Suikerkorporasie Bpk, Malelane*

## Abstract

An attempt was made to enhance colour removal in the raw house and refinery under the abnormal conditions of the 1983/84 season so as to maintain an acceptable colour quality in the final refined sugar product. Phosphoric acid was added to the mixed juice to improve the defecation process in the raw house while activated carbon was used in the carbonatation process to increase colour absorption.

## Introduction

The process used for raw sugar manufacture at ML is the conventional hot-liming method followed by a 3-boiling system using B-magma as a footing for A-massecurite. The A-sugar quality is entirely colour orientated as 100% refining is practised. This is done using the carbonatation/sulphitation process, followed by a 4-boiling system. The sugars from these four stages are blended at the centrifugal station to give a final product of acceptable quality.

An important factor of such a refining process is that a more-or-less consistent colour removal can be achieved although the colour input may vary considerably.

Consequently experience has shown that when the A-melt colour exceeds a certain point, the fourth sugar will probably have to be rejected due to too high a colour, with the resulting loss in recovery and factory thermal efficiency.

The 1983/84 crushing season brought with it new factors which greatly increased the possibility of severe colour problems. Although the major cause was poor cane quality due to the drought, a further factor was introduced when ML undertook to refine 22 600 tons of Swaziland raw sugar. This contract necessitated that the refinery be run at maximum capacity for almost the entire season and there was also some doubt as to the anticipated quality of this sugar.

In the light of these developments it was decided to attempt to maintain optimum colour removal by:

- (a) improving mixed juice clarification by maintaining optimum phosphate levels through the addition of phosphoric acid. This was also tried and reported by Carter;<sup>1</sup>
- (b) increasing colour absorption in the carbonatation process through the addition of powdered activated carbon to the A-melt.

## Analytical Procedure

Many of the analyses required to monitor the above processes were not done on a regular basis in the mill laboratory. Furthermore, colour analyses of the various products were not comparable and this necessitated a standard procedure to be adopted and implemented. To meet these requirements using existing laboratory facilities the following changes were made:

- (a) Sub-samples of the Central Board's hourly mixed juice sample were composited over four hours and analysed for phosphate content using the standard SASTA procedure.<sup>2</sup>
- (b) Addition of phosphoric acid was made after weighing and hourly catch samples of this juice were analysed for  $P_2O_5$  content as in (a).

- (c) Sub-samples of the hourly clear juice sample were composited over four hours and analysed for phosphate content as in (a) and (b) and for colour according to the ICUMSA 420 method.
- (d) Two-hourly samples of the A-melt and fine liquor were analysed for colour according to the ICUMSA 420 method.
- (e) Finally, all refined sugars were analysed for colour also according to the ICUMSA 420 method.

It must again be stressed that the collection and sampling of these products were done by the existing laboratory shift staff working under a revised procedure. This should be borne in mind when reviewing these results for comparison with similar trials at other factories.

## Experimental Procedure

The additional analyses were started in week 14 of crushing and by week 20, sufficient data had been gathered on the prevailing conditions to be used as a standard for comparison.

Phosphoric acid was then added to the mixed juice to maintain a 300 to 320 ppm  $P_2O_5$  level in the juice. A diaphragm-type dosing pump was used and the undiluted acid was added into the buffer tank after the scale. The feed of the juice into this tank caused sufficient turbulence to ensure adequate mixing of the acid and the juice. A sample point on the outlet of the pumps removing the juice from this tank was used to take catch samples to reveal the increased  $P_2O_5$  content. The addition of acid continued until the end of the season.

The problems of excessive colour in the refined sugar did not appear as early as had been expected probably because:

- (a) it is common practice at ML to stop using B-magma for A-massecurite footing when A-sugar colours start to rise and may affect the colour of the refined sugar. This was done in week 21 and for the rest of the season, B-magma was not used as footing;
- (b) ML was under contract to refine raw sugar from Swaziland. Although it was initially felt that this raw sugar would present a colour problem, it was in fact of excellent quality and helped to improve colour in the mill. This contract was completed in week 25.

Discussions with suppliers of activated carbon and consumers in similar industries revealed that a powdered type would be most suitable. It was decided to add this carbon at the raw sugar melting stage to ensure adequate retention time for colour absorption prior to filtration. These retention times in practice worked out to be

- from melting to lime addition — 20 minutes
- from carbonatation saturators to filtering — 55 minutes.

During filtration, the carbon was removed together with the calcium carbonate and finally formed part of the wasted refinery sludge. This had no adverse effect on filter performance.

The feeding device for the powdered carbon consisted of a supply bin of 0,17 m<sup>3</sup> capacity mounted above a variable speed vibrator feeder. An adjustable door fed the desired quantity of the powder from the bin onto the feeder. After leaving the feeder, the carbon dropped into a tundish fitted with water

sprays and was washed into the feed of the A-sugar melter. Two problem areas were revealed: the first being that the angle of the sides of the bin were not steep enough and disrupted the flow and secondly, that any air movement at the feeder resulted in powder being blown away in every direction.

After the initial run it was decided to dose the carbon by hand directly onto the wet sugar in the screw conveyor feeding the melter. The advantage was that the powder adhered to the wet sugar and consequently very little was lost in the air. Although not tested, the best method of feeding would probably be an enclosed variable speed screw from a supply bin which would dose the carbon directly into the side of the wet sugar conveyor feeding the melter.

Laboratory trials with various carbon powders under simulated conditions indicated that 0,05 to 0,1% on brix in A-melt would reduce colour to an acceptable level. The carbon chosen was one having a density of 360 to 380 gm/l and a surface area of 950 to 1 100 m<sup>2</sup>/gm.

### Results of Phosphoric Acid Addition

The data collected until week 20 and three weeks thereafter, i.e. after all analytical changes had been carried out are shown in Table 1. The improvement which had been brought about in the clarification process by the higher P<sub>2</sub>O<sub>5</sub> content, unfortunately did not lead to an improvement in the clear juice colour. However, the improvement in the settling and handling quality of the mud was evident and there was an immediate improvement in the pol % filtercake (see Table 1).

TABLE 1  
Data before and after addition of acid

Period	Phosphate content (ppm) of juice		Clear juice colour @ 420 nm	A-melt colour @ 420 nm	Pol % cake
	Mixed	Clear			
Before addition of acid up to week 20	253	33	22 100	1 730	2,01
After addition of acid					
week 21	275	42	21 500	1 770	1,62
22	318	34	23 400	1 705	1,62
23	320	26	24 600	1 867	1,58

As mud handling has been a persistent problem at ML, it was decided to continue the acid addition so as to minimize this problem and to monitor its influence on clear juice colour. The average results for the rest of the season are also shown in Table 2. The pol % cake continued to improve markedly but clear juice colour progressively deteriorated because of the poor quality of cane.

TABLE 2

Average clarification data with acid addition for the latter portion of the 1983/84 season

Period	P <sub>2</sub> O <sub>5</sub> content (ppm)		Clear juice colour @ 420 nm	A-melt colour @ 420 nm	Pol % cake
	Mixed	Clear			
Weeks 24 - 36	282	23	30 380	2 096	1,45

### Results of Addition of Powdered Activated Carbon

After the completion of the Swaziland sugar contract, the colour of the A-melt began to increase and in week 26, the first addition of powdered carbon was carried out. The relevant data prior to this, is shown in Table 3.

TABLE 3

Refinery data prior to addition of carbon

Period	Colour @ 420 nm		% colour removal
	A-melt	Fine liquor	
To date week 25	1 772	742	58,1

The dosage rate was initially set for 0,05% on brix in A-melt but was increased to 0,1% when there was no improvement. At the higher rate, the colour removal was substantially better and the results for weeks 26, 27 and 28 are also shown in Table 4.

TABLE 4

Refinery data after addition of carbon

Week no.	Colour @ 420 nm		% colour removal
	A-melt	Fine liquor	
26	1 550	572	63,1
27	1 967	652	66,8
28	2 010	759	62,2

During this run, it became obvious that higher A-melt colours would require considerably more carbon and a second set of laboratory trials were done using A-melt colours of up to 3 080 units. These tests revealed that at the higher A-melt colour, 0,4 to 0,5% carbon on brix in melt was required to produce a fine liquor colour of acceptable quality. These results are shown in Table 5 together with costs based on the actual cost of R3,00 per kg of carbon for Malelane.

TABLE 5

Carbon concentration, colour removal and cost

Carbon concentration	Units colour removed	Cost/ton refined sugar
0,1% on brix in A-melt	400	R 3,50
0,3% on brix in A-melt	1 200	R10,50
0,5% on brix in A-melt	1 800	R17,50

From week 29 onwards, the A-melt colour averaged 2 500 units which would have required about 0,3% carbon on brix to give an acceptable liquor colour. As this was not economically acceptable, carbon was used only in times of severe colour problems and then only for short periods.

On this basis, carbon was used again during the Christmas-New Year period and in the final week of the season. On both occasions, however, there was no improvement in colour removal. Although there are many reasons for this, the very poor cane quality caused by the heavy rains must have been a major contributor.

### Conclusion

The addition of phosphoric acid to mixed juice to improve the clarification process and thereby the colour of the clear juice did not have the desired results. It did however result in a mud which settled better in the clarifier and was easier to handle at the filter station. These factors resulted in a significant decrease in the pol % filtercake.

The use of powdered activated carbon to increase colour removal in the carbonation stage of the refining process, proved to be successful and can be used in times of short term increased A-melt colour. However, because of the cost involved dosage quantities must be kept within economic limits, and this depends on the degree of remelting.

### REFERENCES

1. Carter, G. G. (1966). Phosphoric acid as an aid to clarification, and observations on liming techniques and mud volumes. *Proc S Afr Sug Technol Ass* 40: 171-180.
2. SASTA Laboratory manual for South African Sugar Factories 2nd edition 1977.