

OFF-CROP REFINING AT MALELANE

M MOODLEY, PM SCHORN AND I SINGH

Transvaal Sugar Limited, PO Box 47, Malelane, 1320

Abstract

The refining period of the Malelane (ML) factory had to be extended due to the importation of raw sugar from the Komati (KM) factory which was commissioned in the 1993-94 season. This sugar is stored and refined during the off-crop period. Malelane has, therefore, been refining sugar during the off-crop period for the past three seasons, 16 068, 67 651 and 20 000 tons of raw sugar being processed during the 1993-94, 1994-95 and 1995-96 seasons, respectively. This paper traces the processes that have been implemented and the modifications that have been made to refine this sugar. Some of the processing problems encountered as well as some performance figures are discussed.

Keywords: refining, off-crop, Malelane, jet 4

Introduction

Due to the importation of raw sugar from the KM mill, it was decided to operate the ML refinery for a period of 50 weeks, as compared with the 40 weeks that the raw house operates. This meant that the refinery would run for approximately 10 weeks during the off-crop period. The main advantage of lengthening the refining period is better utilisation of plant equipment. The disadvantages are higher energy costs (burning of coal) and the processing of the refinery jet 4. During the season, the jet 4 from the refinery is mixed with raw syrup and then processed further in the raw house. The jet 4 produced is approximately 9% (on brix) of the incoming raw sugar.

Typically, in a central refinery, the final jet is sent to the recovery house where it is further processed. Three boilings are carried out and the purity of the refinery exhaust molasses is about 60.

Laboratory crystallisation tests done by Morel du Boil (1985) have shown that the addition of refinery exhaust molasses to a pure sucrose solution caused extensive elongation of the sucrose crystal. In addition, the crystal growth rate was retarded. Morel du Boil (1985) has also shown that most of the crystal elongation properties in a refinery were confined to the oligosaccharide fraction.

The oligosaccharides present in the VHP sugar crystal are transferred to the refinery. They are concentrated throughout the refinery, thus adversely affecting crystallisation rates (Morel du Boil, 1995). This has been confirmed at the ML refinery where the boiling times of the lower grade refinery massecuites increased progressively. Furthermore, even after further attempts of exhaustion with up to nine batch boilings, refinery exhaust molasses purities could not be lowered below 74. This represents a significant loss of sucrose.

ML therefore decided to investigate alternative methods of sucrose recovery from jet 4 and to investigate ways of decreasing the refinery exhaust molasses purity.

A joint venture with the Sugar Milling Research Institute (SMRI) was initiated to investigate sucrose recovery by ion exclusion chromatography. Pilot plant work by Thompson *et al.* (1994) and Peacock (1995) on ML jet 4 has shown that about 90% of the sucrose in the feed jet 4 stream could be

recovered by ion exclusion chromatography, with good separation of the oligosaccharide fraction. This approach is viewed as a long term project by ML. In the short term, several options have been investigated and implemented to minimise the loss of sucrose in refinery exhaust molasses.

The storing and handling of the raw sugar at ML has been described by de Jager (1995). The plant and structural changes that have been implemented at the refinery to carry out off-crop refining and the initial off-crop boiling cycles have been described by Schorn (1995). Analyses were done to optimise the off-crop refining process. Weekly samples of jet 4 were analysed and colour and oligosaccharide surveys were made. Refinery exhaust molasses storage was investigated by analysing sub-samples of the stored molasses at regular intervals.

The oligosaccharides were measured at the SMRI using analytical ion exclusion high performance liquid chromatography (HPLC) with refractive index (RI) detection. Colour measurements were conducted at 420 nm, using 0,45 micron membranes for filtration at pH 7. Crystal colour determinations were conducted on sugar samples affinated according to the ICUMSA method. Glucose, fructose and sucrose were measured using gas chromatography.

Results

Analysis of jet 4

The average results of the weekly jet 4 samples are given in Table 1 and indicate that there is a significant concentration of oligosaccharide in the jet 4 stream.

Table 1
Jet 4 analytical results

| Analysis | Results |
|------------------------------|---------|
| Sucrose purity | 91,17 |
| Colour | 6768 |
| Glucose (%) | 0,52 |
| Fructose (%) | 0,40 |
| Oligosaccharides (ppm on bx) | 6388 |
| Magnesium (ppm on bx) | 443 |
| Sodium (ppm on bx) | 219 |
| Potassium (ppm on bx) | 2625 |
| Calcium (ppm on bx) | 2531 |

1993-1994 off-crop

The initial boiling scheme is given in figure 1. In this boiling system, it was decided to try to store the lower jets (5th, 6th, 7th, 8th and 9th) separately in only one tank. This posed a problem because it was difficult to manage the system and mixing of jets occurred. The purity of the refinery exhaust jet that was pumped out to final molasses was around 74. Modifications were thought necessary for the 1994-95 season.

1994-1995 off-crop

In this season it was decided to store some of the jets separately in different tanks. The boiling system is given in Figure

- 1 The 5th, 6th, 7th, 8th and 9th Masseccutes are boiled in pan 5
- 2 Tank 2 is used to store jet 5, jet 6, jet 7, jet 8 and refinery exhaust molasses

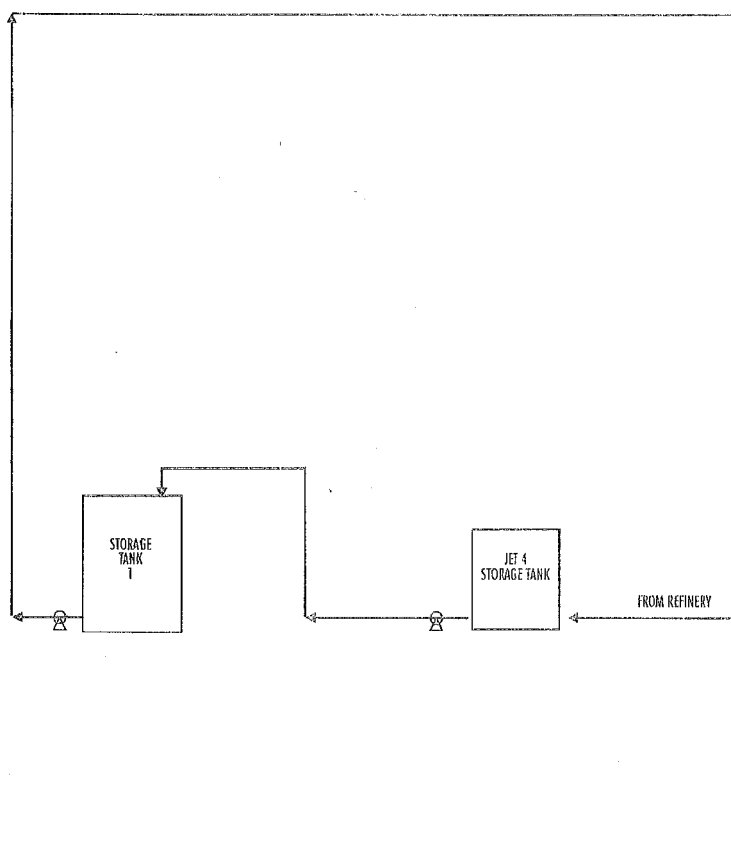


FIGURE 1: 1993-1994 off-crop refining

2. In this system the jet 5 was stored in one tank and the other jets (6th, 7th, 8th and 9th) in another tank. Here again it was difficult to manage the system. Mixing of jets occurred and although nine masseccutes were boiled, the exhaust molasses was still high (around 70).

During the season, several tests were carried out to investigate ways in which the loss of sucrose in refinery exhaust molasses could be minimised and to optimise the boiling programme. These tests are described below.

Effect of oligosaccharides on masseccute boiling times

To assess the effect that oligosaccharides have on masseccute boiling times, samples of feed and masseccutes were sent to the SMRI for analysis. The respective masseccute boiling times were recorded. The oligosaccharide results as reported by Morel du Boil and Walford (1995) have been plotted against masseccute boiling times in Figure 3.

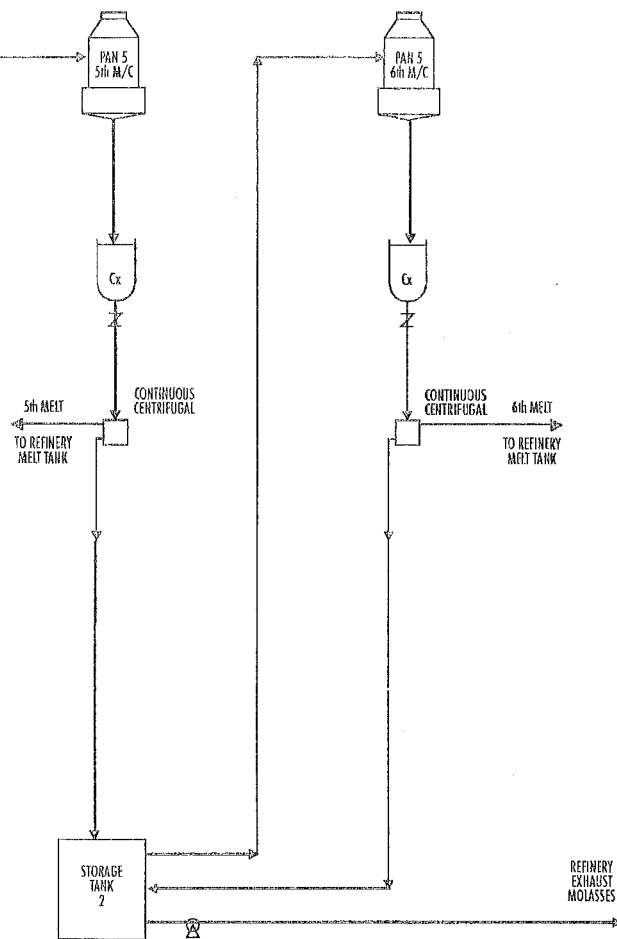
The equation is as follows:

$$M \# = 3,73 + 0,000506 \times \text{Oligosaccharide (ppm on brix)}$$

(n=7, r=0,97)

- Time (in minutes) taken to boil 1 m³ of masseccute

From Figure 3 it can be seen that there is a direct relationship between the time taken to boil 1 m³ of masseccute and the level of oligosaccharides. The boiling time for the 7th masseccute is three times longer than that for the 1st masseccute.



Chemical treatment of jet 4

Samples of chemically (sulphitation, activated carbon and enzymes) treated jet 4 were boiled in the SMRI pilot pan. The objective of this work was to investigate whether some of the impurities which affect crystallisation could be removed. The results (Davis, 1995) show that sulphitation and activated carbon are beneficial in terms of colour and gum reduction. The enzymes tested were not effective in removing the impurities that adversely affect masseccute boiling times. In addition, sulphitation can be used to remove calcium, which is necessary, if ion exclusion separation is to be used.

Storage tests on refinery exhaust molasses

The results of the laboratory storage tests done on refinery exhaust molasses are given in Figure 4, and show that it is quite stable for an extended period of time. The fructose/glucose ratio changed significantly. This is expected as fructose is less stable than glucose.

As a result of these encouraging results it was decided to store refinery exhaust molasses on a trial basis in the plant. Five hundred and fifty tons of various refinery streams, at 70° brix were stored in the factory for a period of 42 days. The results show that exhaust molasses (70° brix) can be stored for a long period of time without a significant loss in sucrose. During the start of the crushing season, the stored molasses was exhausted in the raw house. The final molasses purity for week one was an average of 32. The impurities in the refinery

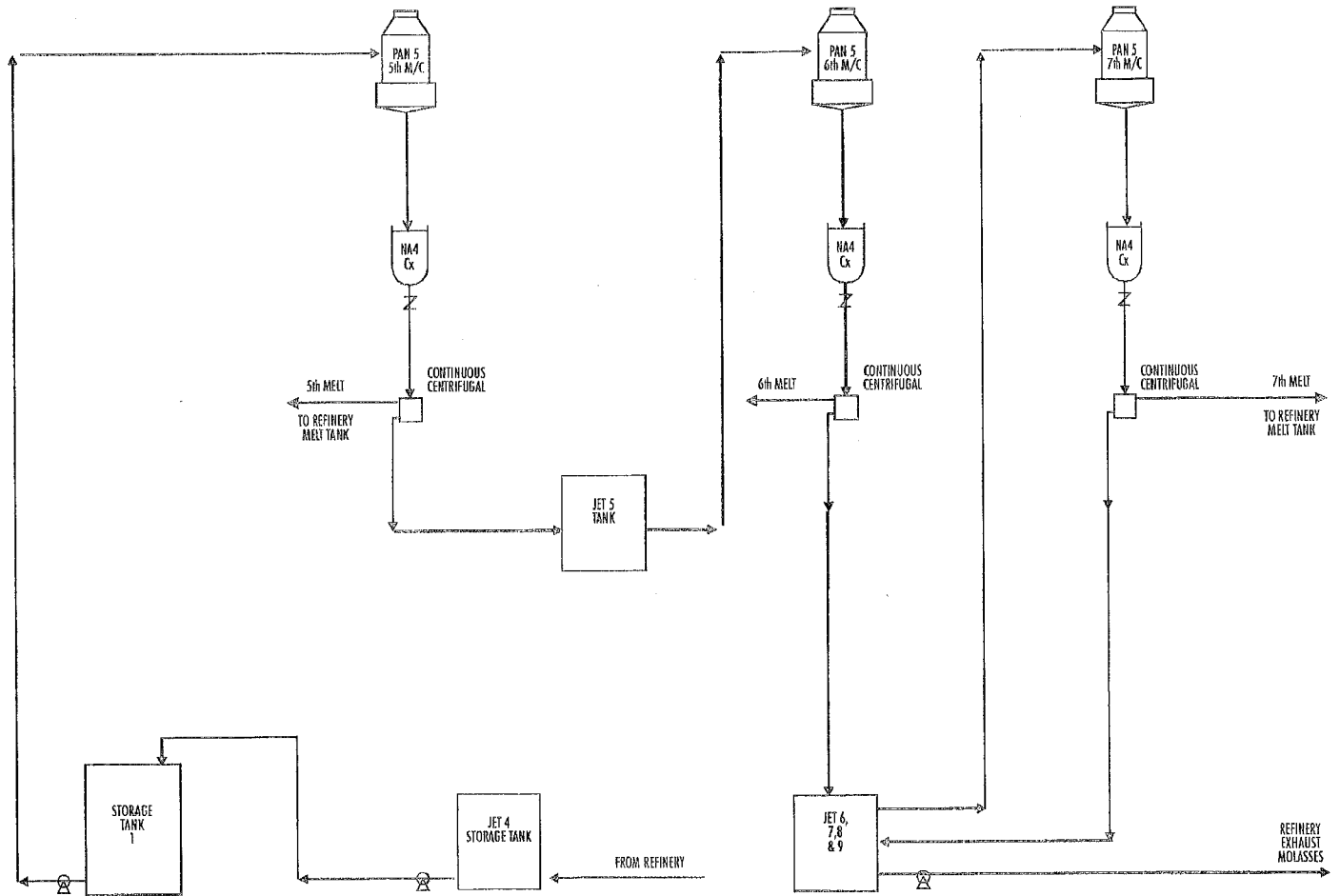


FIGURE 2: 1994-1995 off-crop refining

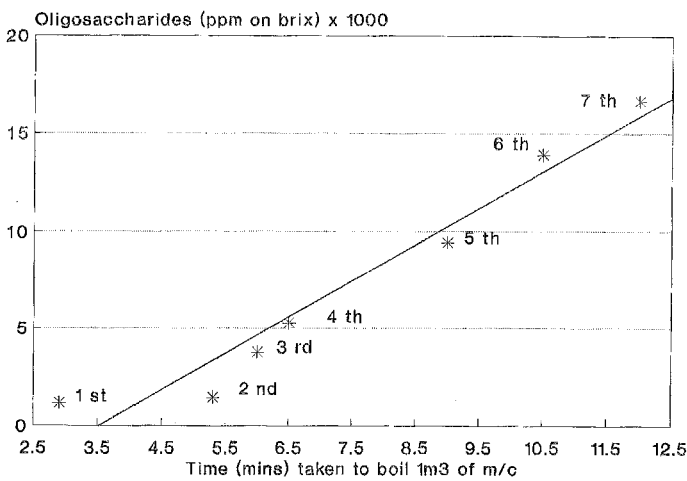


FIGURE 3: Effect of oligosaccharides on boiling times

exhaust molasses will have an effect on crystallisation in the raw house but it is difficult to quantify.

Colour profile across refinery boilings

The various boilings were monitored and the feed and sugar streams were sampled. The sugar samples were affi-

nated at the SMRI and then analysed. The results which are plotted in Figure 5 indicate that the colour transfer decreases with the lower grade refinery boilings. These tests will be repeated during the next off-crop.

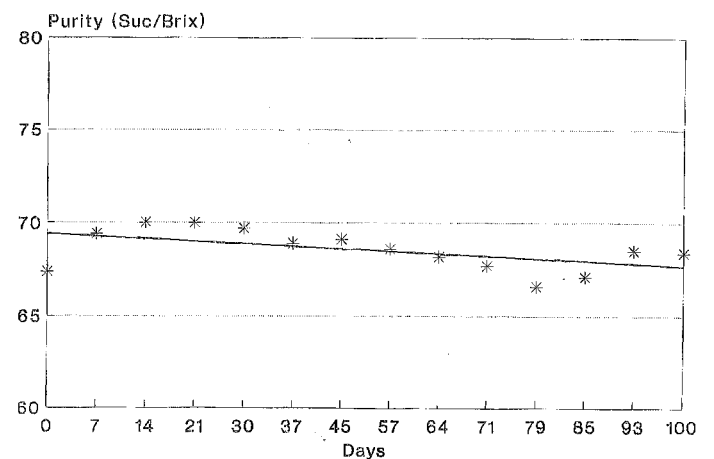


FIGURE 4: Exhaust molasses storage tests

1995-1996 off-crop

From the encouraging results obtained when exhaust molasses was stored both in a pilot plant and on a full scale basis,

it was decided that in the following season, all the exhaust molasses would be stored for processing during the season following the off-crop.

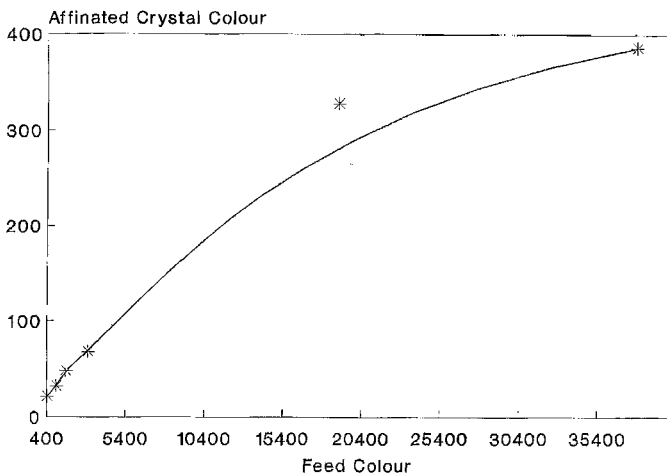


FIGURE 5: Boiling colour profile

Rein (personal communication, 1995) suggested that ML investigate the use of the C-continuous pan to boil lower grade refinery massecuites. It was decided to pursue this option and boil the 7th massecuite in the continuous pan. As a result the boiling system was modified. This is represented in Figure 6. In this system the jets are completely separated. It was also decided to store the exhaust molasses in the raw house clarifiers until the crushing season started. During the crushing season the refinery exhaust molasses will be further exhausted in the raw house.

Continuous pan operation

The initial off-crop period lasted for one month. The continuous pan thus had to be commissioned within a relatively short period of time, but the start-up and operation of the pan went very smoothly. Fears of encrustation on baffle elements within the pan did not materialise.

The pan operation was identical to that for its duty as a normal C-continuous pan. Sixth jet was fed to the first 2 to 6 compartments and the remaining compartments were held on water. During this period the operation of the pan had to be stopped a number of times, due to insufficient feed material as the pan is somewhat oversized for the refinery low grade massecuite. The continuous pan was operated for a period of about 18 days. At the end of this period only about 1 to 2 mm of sugar encrustation was evident.

Maintaining the required ratio of jet 6 and seed was the main operational problem. The existing, variable speed, seed pump as designed for C-massecuite was too large in this application. Reduction of the motor frequency was limited to 10 Hz, at which speed motor cooling became a problem. The analytical results for the lower grade refinery boilings are given in Table 2.

The high 7th massecuite exhaustion is due to the fact that the C-continuous pan was used to boil the massecuite. A 25-30 unit drop in purity was achieved. The higher exhaustion achieved in the continuous pan relative to the batch pan can be attributed to a number of factors. Firstly, due to the low boiling head improved circulation can be achieved in the continuous pan. Secondly, the viscosity of the 7th refined massecuite seems to be lower than the viscosity of C-massecuite, therefore better circulation can be achieved and hence improved crystallisation. Thirdly, the continuous

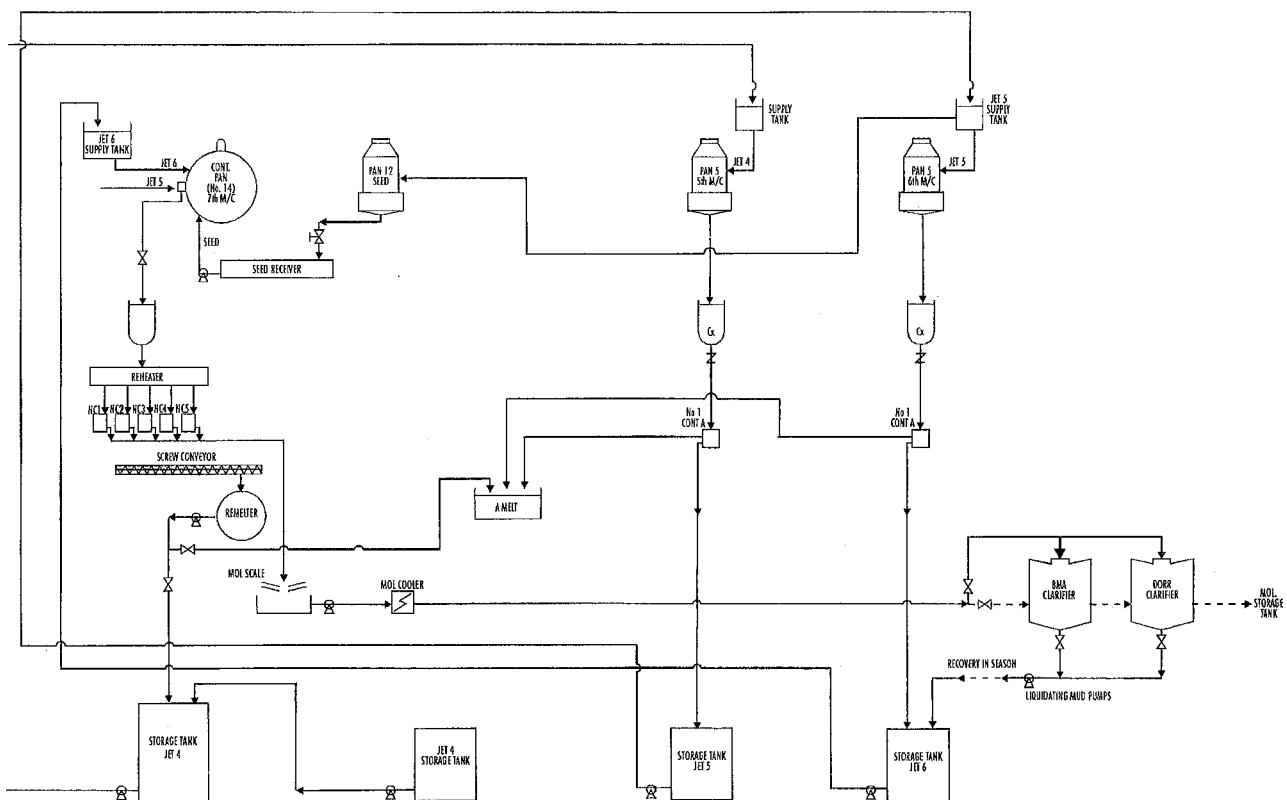


FIGURE 6: 1995-1996 off-crop refining

¹ Dr Peter Rein, Tongaat-Hulett Sugar Ltd, Private Bag 3, Glenashley, 4022

pan is far too large for the present duty and longer retention times were achieved. This would help to improve exhaustion.

Table 2
Average analyses and exhaustions of low grade boilings

| Steam | Brix % | Purity | Exhaustion | ICUMSA 420 colour |
|------------------|--------|--------|------------|-------------------|
| 4th jet | 65 | 92 | | 3728 |
| 5th massecuite | 92 | 91 | 52 | |
| 5th melt | 60 | 98 | | 400 |
| 5th jet | 72 | 83 | | 19457 |
| 6th massecuite | 92 | 84 | 51 | |
| 6th melt | 60 | 98 | | 969 |
| 6th jet | 75 | 72 | | 28665 |
| 7th massecuite | 92 | 71 | 65 | |
| 7th sugar | 99 | 93 | | 12000 |
| Exhaust molasses | 78 | 46 | | 99000 |

Samples of 7th massecuite taken before the reheater were nutschted in the laboratory. The massecuite exhaustion is plotted in Figure 7.

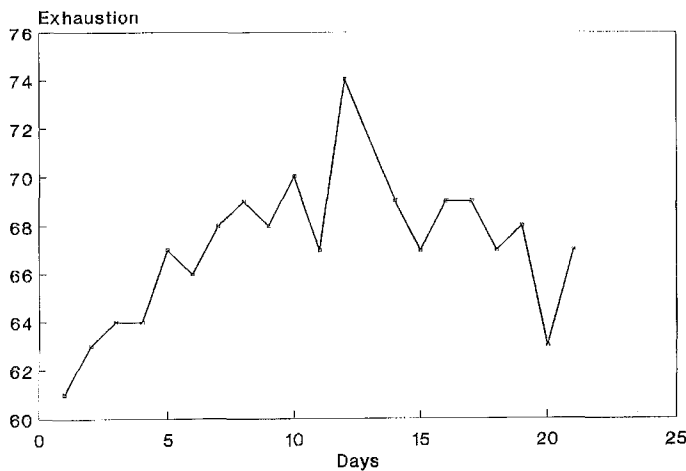


FIGURE 7: 7th massecuite exhaustion

Refinery molasses exhaustion

A comparison of the molasses produced during the season with that from the off-crop refining is listed in Table 3. A sample of the refinery exhaust molasses has been sent to the SMRI for boiling down tests. The average daily exhaust molasses purity for the off-crop period is plotted in Figure 8.

Table 3
Comparison of refinery molasses and final raw house molasses

| Analysis | Final raw house molasses | Refinery molasses |
|--------------------------|--------------------------|-------------------|
| Dry solids | 76,6 | 77,0 |
| Pol/sucrose ratio | 0,88 | 0,99 |
| (F+G)/ash ratio | 1,5 | 2,8 |
| True purity | 35,1 | 47,7 |
| Target purity difference | 3,7 | 20,2 |

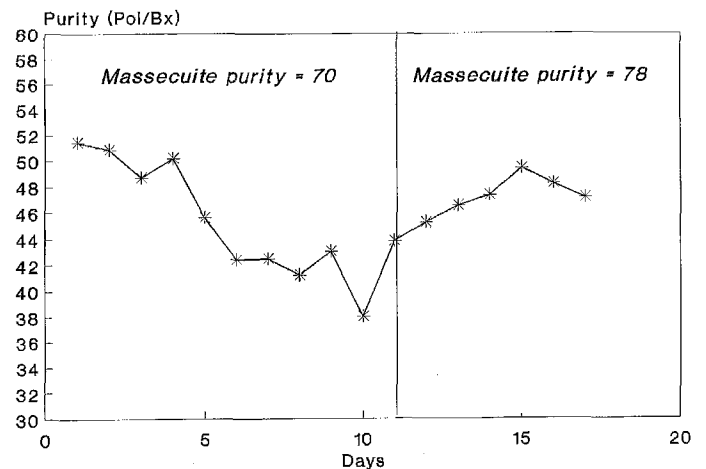


FIGURE 8: Daily exhaust molasses purity

The initial refinery exhaust molasses purity was around 50 and during the pan optimisation period the molasses purity dropped to around 38. It then began to rise. This was due to operational problems in the refinery. Firstly, jet 4 instead of jet 6 was used to make the seed. Secondly, some mixing of jets 5 and 6 occurred. These operational problems caused the massecuite purity to increase from 70 to 78.

Sugar quality

The colour results of the various streams are given in Table 4. The results indicate that low refined sugar colour can be produced from a melt colour of 1173. The average decolourisation obtained in the refinery is around 60%. First sugar colours measured by the SMRI were as low as 15.

Table 4
Colour of refinery streams

| Stream | ICUMSA 420 colour |
|-------------------|-------------------|
| Melt | 1173 |
| Carbonated liquor | 512 |
| Fine liquor | 468 |
| Jet 4 | 3728 |
| Refined sugar | 40 |

Summary

Pilot plant tests have shown that refinery exhaust molasses can be stored at high brix (70) for an extended period of time. Full scale tests have confirmed the laboratory findings.

It is estimated that, during the 1994-95 season, R260 000 worth of additional sugar was recovered by storing refinery exhaust molasses, and exhausting this molasses further during the season.

The continuous pan has been used successfully to boil 7th massecuites, and allows the number of boilings in the refinery to be reduced from nine to seven. This has major benefits in terms of energy usage and undetermined loss.

By operating the continuous pan and storing the refinery exhaust molasses, it is estimated that ML has recovered R410 300 worth of additional sugar as compared with last season. For a normal off-crop refining season [65 000 tons

Very High Pol (VHP)], this would amount to about R1,4 million. Assuming that a 35 refinery exhaust molasses purity can be achieved during the off-crop then R1,7 million worth of additional sugar can be achieved.

A number of areas need to be further investigated:

- The continuous pan will be optimised with regard to seed quality, seed rate and massecuite quality.
- Colour balances have shown that if the fifth sugar is added to the refined sugar, the colour of the refined sugar will increase by 10%. It would be of benefit to combine the fifth sugar with refined sugar.

Investigations will also be done to evaluate whether the 5th and 6th massecuites could be boiled in the continuous pan.

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