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# SOMEWHAT DRY . . . A NEW LOOK AT THE CONDITIONING OF REFINED SUGAR

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## Abstract

Conditioning of refined sugar comprises slow drying to a moisture content where the sugar is unlikely to cake during storage or transit. Conditioning mechanisms are explained briefly and 'conventional wisdom' is examined. Pilot plant conditioning test work is described, in which the effects of variations in conditioning temperature and sugar quality were investigated. Observations are made with respect to factors affecting the 'conditionability' and caking propensity of refined sugar, and comments are given on the methods of testing for caking. A simple model of the conditioning process is derived from the pilot plant data, and conclusions are drawn on the time required for acceptable conditioning. Initial moisture content is shown to be the main determinant of the shape of the conditioning curve, and it is concluded that conditioning is highly dependent on the way in which the sugar is dried between centrifugation and conditioning.

## Introduction

The process of conditioning is routinely employed by refineries to reduce the residual moisture in refined sugar to a point where the sugar is unlikely to cake during storage or transit. It is a capital intensive process due to the long residence times required, Hulett Refineries currently operating at a minimum of 72 hours. It was with the objective of finding options for reducing this time requirement that an investigation into the conditioning process was undertaken. The scope of the examination included the use of the results of a pilot plant study to attempt to clarify and quantify, for local conditions, the widely accepted conceptual model of the conditioning process.

## The Concepts of Caking and Conditioning

### *Refined sugar moisture*

Rodgers and Lewis (1963), and many authors hence, have differentiated between three forms of moisture in refined sugar:

- *Free moisture*: A dilute solution forming a thin film on the surface of the crystals when they are discharged from the centrifugals. This moisture is relatively easily (and rapidly) removed in driers.
- *Bound moisture*: Also known as migratable moisture, a concentrated solution on the surface of the crystals, associated with and trapped by amorphous sugar. This low permeability amorphous shell is formed during initial drying of the sugar, when moisture release is too rapid to allow the sucrose to crystallise on the crystal surface. This moisture is the greatest cause of caking, and must be removed by conditioning.
- *Inherent moisture*: Moisture that is trapped within the crystals and released only by dissolution or grinding. There is no evidence of the migration of this moisture, and it is believed to play no part in caking.

### *Caking*

Caking is a phenomenon in which sugar crystals give up bound moisture, resulting in supersaturation at the crystal surface and consequent crystallisation. At points of contact between crystals this surface crystallisation causes intercrystalline bridging. The sugar then ceases to be free-flowing and is referred to as 'caked'.

In practice, caking usually occurs due to a change in the relative humidity of the air in contact with the sugar, due commonly to temperature gradients. This may be illustrated by considering a warm mass of sugar cooling down at its boundaries. As the interstitial air cools down, its relative humidity rises beyond the Equilibrium Relative Humidity of the sugar, and the sugar absorbs moisture. This absorption lowers the partial pressure of water at the boundaries, and moisture migrates to these regions from the warm centre. Sugar at the boundaries thus undergoes surface dissolution as it continues to absorb moisture. It therefore either becomes progressively damper, or, more typically, warms up again as ambient conditions change, gives up surface moisture, recrystallises and consequently cakes. In the warm regions, the relative humidity has dropped, so moisture on the crystals evaporates, causing surface crystallisation and, possibly, additional caking.

### *Conditioning*

Sugar conditioning, therefore, should aim to remove any remaining free moisture and reduce the bound moisture content of a refined sugar to the point where it will not cake. This is done, in practice, by exposing the sugar to low-humidity air in conditioning silos for extended periods. However, as Bagster (1970) emphasises, it is not possible to make a non-caking sugar; given sufficiently adverse conditions, any sugar will cake. Researchers that proclaim a sugar to be 'conditioned' after a stipulated time do so at their peril. That appellation should strictly only be applied to a sugar from which all bound moisture has been removed, leaving only inherent moisture (a very lengthy process, if at all achievable). Conditioning is at all times a practical compromise, aiming only at obtaining sugar that is, in the words of this paper's title, 'somewhat' dry. A sensible conditioning objective is that offered by Bruijn *et al.* (1982), that the moisture should be reduced 'to the point where no serious caking will occur when the sugar is subjected to temperature (or humidity) gradients slightly in excess of those expected in practice'.

### *Analysis*

Residual moisture in sugar may be determined in one of four ways:

1. Oven drying for 3 hours (as detailed in the SASTA Laboratory Manual).
2. Oven drying for several days.
3. Karl Fischer titration using methanol (as detailed by Bennett *et al.*, 1964).
4. Karl Fischer titration using formamide (method as above).

Method 1 measures free moisture, and perhaps part of the bound moisture. Methods 2 and 3 measure free plus bound moisture, as methanol washes, but does not dissolve the crystals. Method 4 measures free, bound and inherent moisture, as formamide dissolves the crystals.

Methods 1 and 4 are routinely employed at South African refineries. Method 4 is accepted as the standard method for determination of the effectiveness of the conditioning process, as it takes into account all of the water present in the sugar. It could be argued, however, that method 3 would be more appropriate, as inherent moisture does not cause caking, and variations in the ratios of inherent to bound moisture are not known.

An accurate measure of the caking propensity of sugar is even more problematic. Bruijn *et al.* (1982), Excell (1984) and Ramphal (1989) have reported the use of a number of standard tests to measure this property. Two of these were used in this work:

- *The test tube test*: Samples were taken in test tubes (150 mm long and 20 mm ID) which were then stoppered, and the lower 20 mm of the tube was immersed in a water bath, which was set at 10°C for 2 hours and then at 40°C for two hours. The test tube was then removed and the sugar was poured carefully on to a flat surface. If the sugar flowed freely and contained no lumps, the test was negative. The presence of any lumps or any signs of sugar adhering to the walls of the tube constituted a positive result.
- *The 'large scale' test*: Two test units were borrowed from the SMRI, each consisting of a perspex cylinder, closed at the bottom by a hollow aluminium disc and at the top by a perspex lid fitted with an O-ring. Each column was filled with approximately 7 kg of sugar, closed, and then covered with a polystyrene insulating cylinder. Water at 5°C was then passed through the aluminium base for 2 hours, followed by water at 40°C for 2 hours. The sugar was then poured out in the same way as in the test tube tests, and any lumping or adhesion observed.

While the effectiveness of conditioning should in fact be assessed according to a sugar's caking propensity, the above tests are both subjective and qualitative. It was therefore necessary to rely on residual moisture (in conjunction with caking test results) as an indication of the condition of the sugar.

## Conditioning Rate Theory

Figure 1 represents a portion of sugar crystal surface undergoing conditioning. Three processes may be considered to be taking place:

- The crystallisation of sucrose from the supersaturated film on to the main crystal lattice and the migration of sucrose molecules towards the crystal surface. This may be viewed as a 'liberation' of water molecules through film dilution.
- The migration of water molecules from the film across the amorphous layer by molecular diffusion.
- The interphase transfer of water molecules into the interstitial air by evaporation.

The rate of conditioning (moisture loss) is governed at any time by the slowest of the three processes. Mikus and Budicek (1986) have shown that the evaporative process is at all times an order of magnitude faster than the other two mechanisms, and that it may therefore be neglected when considering conditioning behaviour. A model of the conditioning process has been formulated, which yields the following conclusions:

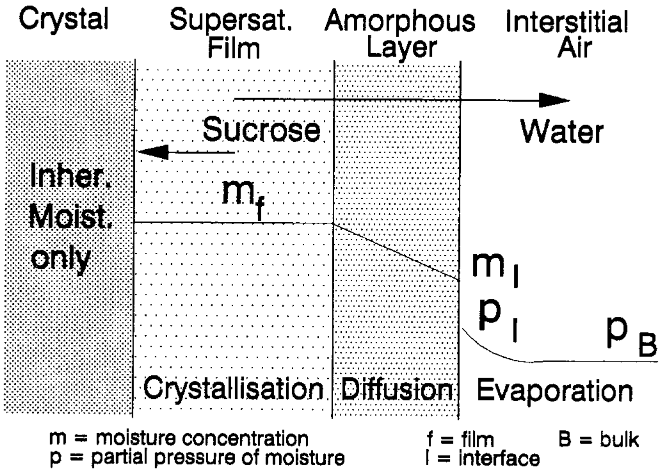

**FIGURE 1** The conditioning process at a portion of sugar crystal surface.

- Crystallisation is the rate limiting process throughout conditioning, but this statement may easily be misconstrued. The conditioning rate is in fact equal to the rate of diffusion (not crystallisation), but crystallisation limits the diffusion rate by its effect on the concentration of moisture in the surface film, which determines the driving force for diffusion.
- Conditioning may be approximated by a modified exponential function:

  $$M_B = K \, . \, e^{-f(t)}$$

  where $M_B$ = bound moisture
  t = conditioning time
  K = a constant

  The function f(t) = k.t for pure diffusion, but because of the influence of crystallisation, prediction of f(t) requires a more sophisticated model than has been developed. For the purposes of this work, therefore, f(t) has been estimated by best fit to empirical data.
- Bound moisture tends to zero as time tends to infinity. For practical purposes, this means that sugar moisture will approach the inherent moisture given sufficient conditioning time.

## Experimental Method

### *Pilot plant equipment*

Figure 2 is a diagram of the conditioning pilot plant. The two stainless steel columns were water jacketed and each held 25 kg of sugar, supported on a fine mesh screen at the base of each column. Compressed air from the laboratory mains was passed through a water trap and an oil filter and then split into parallel streams. Each passed through a rotameter and was dehumidified through a perspex column packed with about 1 litre of silica gel. Each stream was then heated to the conditioning temperature by passing it through 4 metres of 5 mm ID copper tubing coiled in a water bath. The air then entered each column at the bottom of an inverted cone at the column's base. After passing through the mesh screen and the column of sugar, the air exited to atmosphere via holes in each column lid. Air supply pressure was maintained at 200 kPa(g) by means of a regulating valve, and this was monitored by means of a pressure gauge before the rotameters. Air flow was controlled to 1,25 litres/min per silo (equivalent to 50 litres/min per ton of sugar).

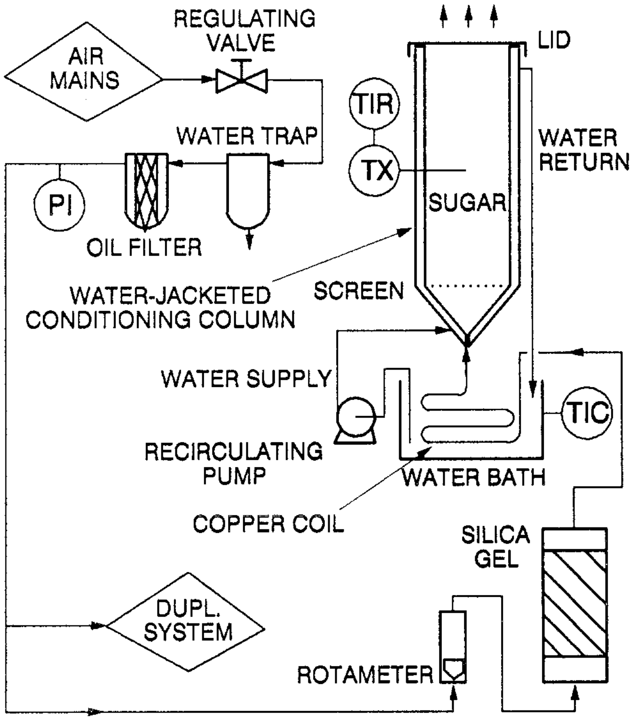

**FIGURE 2** The conditioning pilot plant.

Each water bath was controlled at the conditioning temperature for that run, and the water was circulated through the water jacket to maintain the walls of the conditioning column at a constant temperature. The sugar/interstitial air temperature was measured by probes inserted half way up each column, and was recorded on a chart recorder, while thermometers in the water baths were used to monitor the water temperature. Samples were taken at a stoppered port near the bottom of each column.

### *Procedure*

Mixed sugar samples were taken at Hulett Refineries after the sugar scale which weighs all sugar leaving the rotary cascade drier station. First and third boiling sugar samples were taken at the discharges of the driers that process these grades. Samples were collected in polystyrene cool-boxes (each accommodating 25 kg) and their temperatures measured by means of a digital thermometer, before being transported to the pilot plant laboratory.

The pilot plant water bath, water circulation and air flow were started and the plant was allowed to reach operating temperature and stabilise well in advance of the commencement of each test. Immediately on arrival, the sugar samples were transferred to the columns, temperature recording was started and sampling was begun. The calibration of the temperature probes and the water bath temperature controllers was checked during each run using a digital thermometer. The relative humidity of the exiting air was checked periodically by placing an hygrometer on top of each column of sugar beneath the lid. Each sample was conditioned for a minimum period of 72 hours, and some of the runs were continued for up to 150 hours.

Sixteen conditioning tests (eight parallel column runs) were performed. The first five parallel runs each involved the conditioning of two sugar samples of differing quality under identical conditions. The last three runs each comprised the conditioning of two similar sugar samples (despite using subsamples of the same large sample, initial moistures differed) at different conditioning temperatures.

### *Sampling and analysis*

In each 72 hour period, 7 samples were taken at 8 and 16 hour alternating intervals, to accommodate day shift sampling only. The initial sample comprised samples for oven moisture, Karl Fischer moisture (formamide) and the test tube caking test. All subsequent samples were analysed for Karl Fischer moisture (formamide), and an additional sample was taken each morning for the test tube caking test. Samples for oven or Karl Fischer moisture determinations were taken in 35 ml polyvials, which were filled to the top to exclude air, and stoppered. The stoppers were then sealed in place with Sellotape.

At the end of each run, a further sample was taken for Karl Fischer (methanol) analysis, and the conditioned sugar was subjected to grain size analysis [Mean Apperture (MA) and Coefficient of Variance (CV)], reducing sugars analysis and conglomerate count. For the last 8 tests, an additional large scale caking test was performed at the end of each run. The conglomerate count analyses were performed by the SMRI, all others being carried out in-house.

## Results

### *Raw data*

Table 1 presents a summary of the Karl Fischer moisture analyses and caking test results for the 16 pilot plant tests. Inherent moistures were obtained as the difference between the formamide and methanol analyses on the last sample. Bound moisture may be calculated by subtracting inherent moisture from total moisture at any stage in the conditioning. Caking behaviour is indicated in the table by the time in hours at which the last sample was taken which yielded a positive test tube caking test followed by the time at which the first sample was taken which yielded a negative test. In the large scale test done at the end of the run, a 'soft' result was a cake that crumbled easily when touched, while a 'hard' result was a cake that showed some resistance to breaking as well as some adhesion to the vessel. It should be noted that all large scale tests performed showed some caking.

### *Effect of sugar quality*

No relationships were found to exist at statistically significant levels between the main quality parameters for the sugar samples (MA, CV, sugar grade, conglomerate count and reducing sugars) and the shape of the conditioning curves, the final moistures reached or the caking behaviour. This does not corroborate the findings of Bruijn *et al.* (1982) and Excell (1984) who reported that larger particles (particularly with higher conglomerate counts) were the main determinants of conditioning rate, taking longer to condition. It is noteworthy, however, that both authors also reported that the coarser fractions tested had higher initial moistures than the finer fractions.

### *Effect of initial moisture*

The most significant determinant of both the shape of the conditioning profile and the final moisture content reached after 72 hours was the initial Karl Fischer moisture of the sample. Figure 3 illustrates this phenomenon for three typical tests (each set of data is shown with its best fit curve, as discussed later). Higher initial moisture sugars showed higher initial conditioning rates (as might be expected), but also resulted in higher final moistures. The initial moistures did not, however, correlate well with any of the other sugar properties.

**Table 1**

**Karl Fischer moisture analyses and caking test results**

| Test no. | Temperature (°C) | Total moisture (%) after | | | | Inherent moisture (%) | Cake test results +/− (h) | Large scale cake test |
|---|---|---|---|---|---|---|---|---|
| | | 0 h | 24 h | 48 h | 72 h | | | |
| 1 | 45 | 0,048 | 0,047 | 0,031 | 0,032 | 0,015 | 20/40 | not done |
| 2 | 45 | 0,064 | 0,060 | 0,041 | 0,037 | 0,020 | 24/44 | not done |
| 3 | 55 | 0,059 | 0,053 | 0,039 | 0,041 | 0,021 | 0/23 | not done |
| 4 | 55 | 0,076 | 0,054 | 0,051 | 0,043 | 0,022 | 40/65 | not done |
| 5 | 50 | 0,099 | 0,062 | 0,054 | 0,046 | 0,021 | 23/47 | not done |
| 6 | 50 | 0,061 | 0,044 | 0,036 | 0,032 | 0,020 | 15/39 | not done |
| 7 | 48 | 0,081 | 0,057 | 0,048 | 0,041 | 0,036 | 21/45 | not done |
| 8 | 48 | 0,053 | 0,044 | 0,036 | 0,032 | 0,029 | 21/45 | not done |
| 9 | 48 | 0,075 | 0,051 | 0,040 | 0,037 | 0,021 | 0/15 | +ve hard |
| 10 | 48 | 0,095 | 0,071 | 0,058 | 0,055 | 0,039 | 0/15 | +ve soft |
| 11 | 42 | 0,079 | 0,055 | 0,049 | 0,041 | 0,025 | 0/20 | +ve soft |
| 12 | 48 | 0,073 | 0,053 | 0,044 | 0,043 | 0,025 | 0/20 | +ve soft |
| 13 | 42 | 0,087 | 0,058 | 0,054 | 0,046 | 0,034 | 0/23 | +ve hard |
| 14 | 48 | 0,078 | 0,060 | 0,051 | 0,046 | 0,030 | 0/23 | +ve hard |
| 15 | 42 | 0,087 | 0,065 | 0,053 | 0,048 | 0,024 | −/0 | +ve soft |
| 16 | 48 | 0,083 | 0,063 | 0,052 | 0,047 | 0,027 | −/0 | +ve soft |

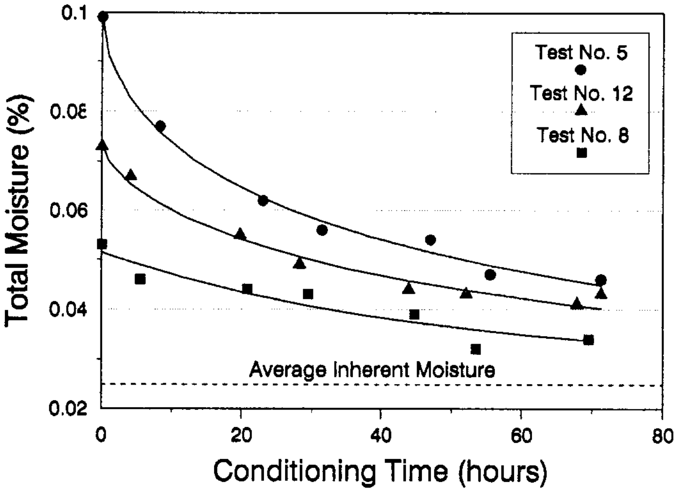

**FIGURE 3** The effect of initial moisture content on conditioning.

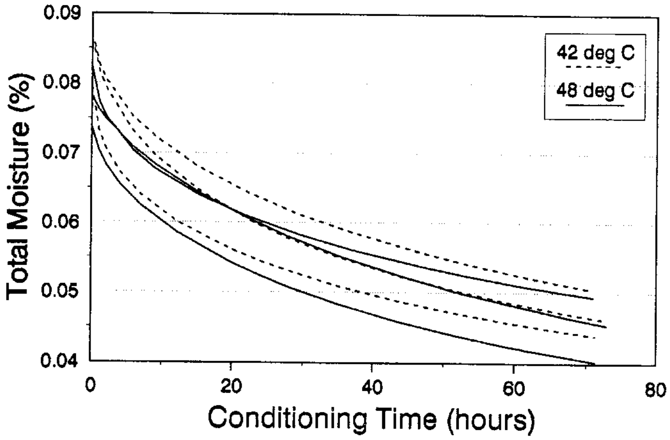

**FIGURE 4** The effect of temperature on conditioning.

*Effect of temperature*

Stachenko et al. (1966) found that increased conditioning temperature improved conditioning rates during the first 24 hours, but had no subsequent effect. Figure 4 shows the best fit curves for the last 6 tests, to allow comparison between conditioning at 42°C and 48°C (best fit curves rather than raw data are used to aid clarity). While it may be argued that the higher temperature appears to enhance conditioning rate slightly, this difference is not statistically significant.

The temperature of the sugar samples when loaded into the pilot columns ranged from 39°C to 56°C, which resulted in an initial temperature difference between sugar and air of up to 14°C (on average 6°C). The sugar typically took between 8 and 18 hours to reach conditioning temperature. A relationship was not evident, however, between initial conditioning rate and initial sugar temperature.

When the pilot columns were emptied after each test, observations were made of any lump formation or caking that had occurred in the columns themselves. This phenomenon was found to occur particularly with sugar that was loaded into the column at a lower temperature than the conditioning air, and the severity of the caking increased with greater temperature differentials. Ramphal (1989) observed the same result with sugar at 32°C and air at 40°C. An explanation may be moisture migration in the column due to temperature gradients, but a similar problem was not encountered with sugar that was loaded hotter than the conditioning air.

*Application of the model*

Various functions of the form predicted by the conditioning model were compared with experimental data, and a good fit was obtained using the relationship:

$$f(t) = K \,.\, t^{N} \qquad \text{where } N \leqslant 1$$

yielding the following general conditioning correlation:

$$M_T = M_{Bi} \,.\, e^{-K.t^N} + M_H$$

Where $M_T$ = total moisture (%)
$M_{Bi}$ = initial bound moisture (%)
$M_H$ = inherent moisture (%) (1)
t = conditioning time (h)
K = a constant ($h^{-N}$)
N = a constant $\leqslant 1$

The values of K and N varied from test to test, and appeared to be specific to the sugar quality and conditions used. Figure 5 illustrates how the shape of the function varies with changes in K and N, for constant $M_{Bi}$ and $M_H$. Table 2 lists the K and N values for the 16 tests, with their corresponding non-linear regression $R^2$ value to indicate closeness of fit.

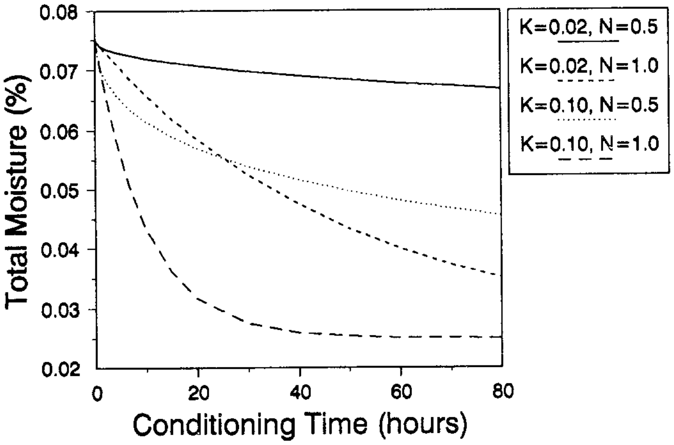

**FIGURE 5** The dependence of the conditioning profile on factors K and N.

Table 2

**Factors K and N and non-linear regression $R^2$ values**

| Test No. | K | N | $R^2$ |
|---|---|---|---|
| 1 | 0,01 | 1,00 | 0,77 |
| 2 | 0,02 | 0,88 | 0,70 |
| 3 | 0,08 | 0,52 | 0,78 |
| 4 | 0,10 | 0,52 | 0,88 |
| 5 | 0,11 | 0,55 | 0,98 |
| 6 | 0,07 | 0,65 | 1,00 |
| 7 | 0,09 | 0,68 | 0,92 |
| 8 | 0,02 | 1,00 | 0,78 |
| 9 | 0,11 | 0,56 | 0,99 |
| 10 | 0,35 | 0,25 | 0,72 |
| 11 | 0,13 | 0,50 | 0,96 |
| 12 | 0,08 | 0,64 | 0,97 |
| 13 | 0,09 | 0,64 | 0,98 |
| 14 | 0,04 | 0,78 | 1,00 |
| 15 | 0,08 | 0,57 | 0,98 |
| 16 | 0,10 | 0,53 | 0,97 |

K and N clearly have an inverse relationship, but attempts to identify a significant relationship with specific sugar quality or operating parameters were unsuccessful. There is some indication that the value of N increases with increasing conglomerate count, but the relationship is not statistically significant. Once again, the only clear correlation that emerged was with initial moisture. In general terms, the higher the initial moisture, the lower the value of N and the higher the value of K.

To allow the prediction of K and N and therefore of conditioning curves from initial sugar moistures, the following correlations were obtained:

$$K = 16{,}8\ M_{Ti}^2 \text{ and}$$

$$N = 1{,}35 - 9{,}42\ M_{Ti}$$

where $M_{Ti}$ = initial total moisture (%)

Caution is advised in the use of these correlations, however, as they are relatively poor fits ($R^2 = 0{,}39$ and $0{,}50$ respectively) and are intended to provide a rough prediction only. They are also limited to Hulett Refineries sugars having initial moisture contents between 0,048% and 0,099%.

## Discussion

### *Required conditioning time*

Estimates in conditioning literature of the theoretical time requirement to condition refined sugar to an acceptable moisture level range from 36 to over 100 hours, while existing installations around the world operate at between 24 and 72 hours. It is clearly not possible, on the basis of the pilot plant work presented here, to make a sensible assessment of required conditioning time according to caking test results. It is therefore necessary to specify an 'acceptable' moisture content, below which caking problems are not anticipated.

Only three positive test tube caking tests were obtained on samples with total moisture below 0,05%, and all were after less than 40 hours of conditioning. If this moisture level is thus accepted as the conditioning 'target', a required conditioning time may be estimated from the pilot plant tests. A total moisture content of 0,05% was exceeded after 56 hours of conditioning in only one test (a sugar which had not reached 0,05% even after 72 hours - Bruijn *et al.* (1982) made mention of similar 'difficult' sugars). A conditioning time of 56 hours therefore appears to be a minimum acceptable requirement for Hulett Refineries sugars. A plant scale test is currently in progress in which one of the conditioning silos at Hulett Refineries is being operated with a sugar residence time of 56 hours for a period of two weeks.

### *Caking propensity determination*

The results of this work suggest that neither the test tube test nor the large scale test simulates reality sufficiently accurately to be used as an assessment of conditioning plant performance. The large scale test is too sensitive, giving a positive result on all samples, while the test tube test was in some cases negative for samples with moistures as high as 0,087%. The development of an intermediate test may address this problem. In an attempt to provide a more quantitative result, a trial is currently under way using the large scale test, in which the mass of the caked sugar is taken on emptying and used as a relative measure.

### *Conditionability*

The variation of the factors K and N in the conditioning equation (1) suggest that each sugar sample has an innate property that may be termed 'conditionability', which affects its conditioning behaviour. When the dependence of the factors on initial moisture is viewed in conjunction with the model of the conditioning process, it may be concluded that the conditionability of a sugar depends on the nature and thickness of the supersaturated film and amorphous layer on each crystal surface.

This deduction points to the initial drying process as the main determinant of conditioning behaviour, as it is during drying that the amorphous layer is formed and the supersaturated film is trapped. The conditionability of the sugar entering the conditioning silo may therefore be linked directly to the way it has been dried. This conclusion has been corroborated by Noodsberg Refinery staff (Getaz, personal communication), who have observed that conditioned sugar moisture varies seasonally, although ambient conditions should, in theory, affect drying but not conditioning. Improvements in the drying station will therefore translate into improvements in conditioning, but care should be taken in the way in which these improvements are achieved. More rapid drying (for example drying at higher temperature) may produce a drier sugar (oven moisture), but would be likely to promote the more rapid formation of a thicker amorphous layer, trapping more bound moisture and having an adverse effect on the subsequent conditioning rate.

## Conclusions

A general model of the conditioning process has been developed and used to interpret pilot plant conditioning data. A minimum residence time of 56 hours in the conditioning pilot plant was required in order to reduce the residual moisture of most Hulett Refineries sugars to below 0,05%. The effect of sugar quality and temperature on conditioning was equivocal, while the initial moisture of each sugar sample had the greatest effect on its conditioning behaviour. The way in which bound moisture is trapped on the sugar crystal during the drying process was thus identified as the chief determinant of the sugar's conditionability.

## Acknowledgements

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