# CONDITIONING AND CAKING EXPERIMENTS ON REFINED SUGAR

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**Abstract**

The behaviour of refined sugar during conditioning and caking experiments in the laboratory was investigated. Tests were performed on a small scale in test tubes as well as in mini-silos and in both cases moisture loss was monitored and caking tests were performed to ensure that the sugar had been adequately conditioned. The information obtained was applied in the construction of the conditioning silo at Tongaat-Hulett Refinery as well as in the planning of the conditioning plant to be constructed at Noodsberg refinery. These will enable refined sugar to be transported in bulk to bottlers, canners and other bulk sugar consumers.

## Introduction

In order to distribute refined sugar in bulk form, the caking properties of freshly manufactured sugar had to be considered. Sugar manufactured in Natal, loaded into a 45 ton rail tanker and transported 600 km to the Witwatersrand, where the majority of consumers are concentrated, will cake severely if any temperature changes are experienced on the journey. Because of this a conditioning or curing period must be undergone by the sugar before it can be transported in bulk. In 1979 the construction of a conditioning silo at Tongaat-Hulett South African Refineries (HULREF) was considered. Before the design was finalised the HULREF sugar was subjected to investigatory tests on a laboratory scale to establish its behaviour on conditioning as well as the minimum conditioning time required.[1, 5]

The first full season of bulk sugar transport to Germiston commenced during 1982 and by mid 1983 it was apparent that a second conditioning silo would be required to meet the increasing demand for bulk refined sugar. Noodsberg (NB) was considered to be a suitable location for the second conditioning plant and thus NB refined sugar was subjected to similar tests to see how it would behave when conditioned in the laboratory.[3]

The properties of refined sugar under the conditions of tests devised to ascertain behaviour patterns when subjected to the conditioning process on a laboratory scale are discussed in this paper.

### *Moisture*

It is necessary to discuss the form in which the moisture is present in the sugar. Rodgers and Lewis[6] differentiate between three categories of moisture.

**Free moisture** is the more or less dilute sugar solution surrounding all sugar crystals leaving the centrifugals. This moisture is easily removed by conventional driers.

**Bound moisture** is moisture on the surface of the crystal and is more difficult to remove, requiring the sugar to be maintained under dry conditions for a comparatively long time as in the sugar conditioning process. This moisture is also called migratable moisture and is the form of moisture which is said to be responsible for caking. The bound moisture is formed when sugar is dried rapidly in a drier. A certain amount of moisture is removed from the outside of the crystal (free moisture) but the sugar present in the outside layer then crystallizes in an amorphous layer which traps the remaining quantity of outside moisture to form the bound moisture. The rate of release of this moisture is determined by the rate of crystallization of sucrose because the amorphous sugar recrystallizes on the crystal and the moisture becomes available for evaporation. The rate of release increases proportionally with an increase in temperature. Caking of refined sugar occurs when this bound moisture migrates through the total mass of sugar and collects in the coldest areas especially if the temperature in these areas subsequently rises thus displacing the collected moisture again.

**Inherent moisture** consists of pockets of sugar solution which have become trapped during the growth of the crystal. It is possible that this moisture may diffuse through the crystal to the surface, but at an extremely slow rate.

## Experimental Procedures

### *Test tube conditioning and caking tests*

The test tube method of conditioning recommended by Purchase[5] was modified as follows. The tubes had 28 mm internal diameters and were 200 mm long. Each was fitted with a rubber stopper through which two copper tubes 6 mm in diameter were passed. One long tube reaching down to 15 mm from the bottom of the test tube carried dry air at 40°C into the test tube. Another short copper tube ending just above the sugar level allowed the air to pass out. These test tubes were then immersed in a 40°C water bath up to the lip to maintain the sugar temperature at 40°C. This apparatus enabled 10 test tubes of sugar to be conditioned simultaneously. Air entering these test tubes was dried by passing through a column of silica gel and warmed by passage through 3 metres of coiled copper tubing immersed in the same water bath.

Sugar was collected after the drier at the refinery in insulated containers to prevent temperature drop during transport of samples. After samples were taken for Karl Fischer moisture determination the rest of the sugar was transferred to test tubes for conditioning and the initial mass was noted. At 24 hour intervals, during conditioning, the test tubes were removed, dried thoroughly and the mass noted to track actual mass loss. After a predetermined conditioning time the sugar was subjected to caking tests.

An unconditioned sample of the same sugar underwent the caking test at the same time to serve as a control. At the end of the caking tests the tubes were emptied and checked for caking.

Caking tests were done in the same test tubes used for conditioning. The stopper containing copper tubes was substituted with another stopper to seal the tube completely. The test tube was immersed in a water bath to a depth of 20 mm and subjected to one or two cycles each of cold and warm temperatures. The cold cycle was always applied first and consisted of 15 hours at 5°C. The warm cycle consisted of 9 hours at 40°C or 60°C.

### *Experimental conditioning silos*

Once the conditioning time had been established on the small scale it was tested on the larger scale. The two pilot silos constructed by Bruijn[1] and shown in Figure 1 were used. Each silo was 250 mm in diameter, 1 000 mm high and had a capacity to hold 40 kg of sugar resting on a fine wire mesh screen. The contents of the silos were maintained at a constant temperature

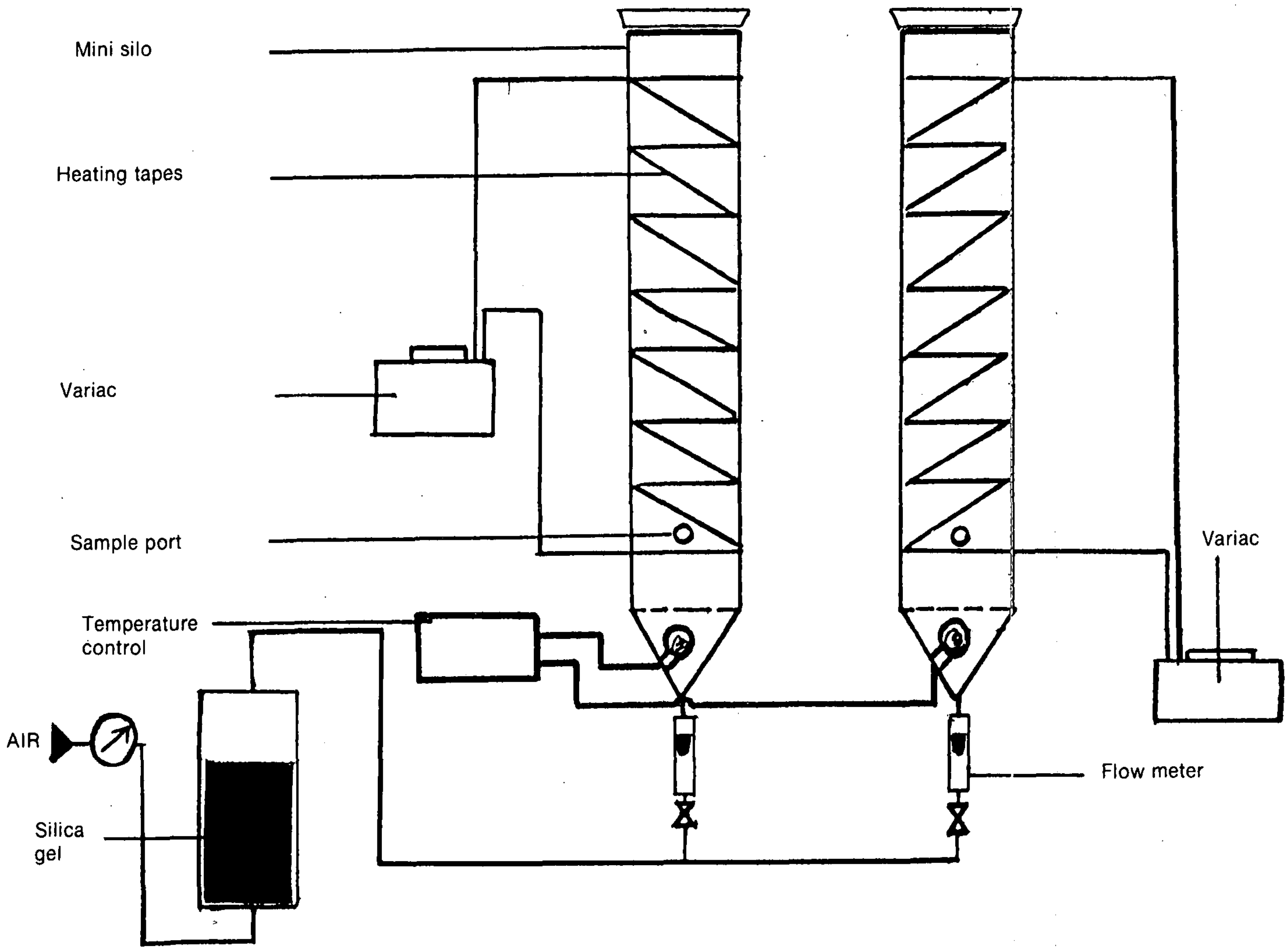


**FIGURE 1** Mini conditioning silos

by controlled heating of the vessel walls, which were wrapped externally with silicone rubber heating tapes. Each silo had a thermometer fitted in the centre and an opening at the bottom for sampling. Compressed air, dried over silica gel and preheated to the required temperature by means of a tungsten bulb, was introduced at the bottom of each silo. The air flow rate was maintained at 2 ℓ $min^{-1}$ per silo which is the equivalent of 50 ℓ $min^{-1}$ per 1 000 kg of sugar, a typical figure used for commercial installations.

Sugar was collected after the drier at the refinery. It was transported in polystyrene boxes to prevent excessive temperature drop. The sugar was transferred to the silos and conditioned for the required time. A sample was taken immediately and thereafter at 24 hour intervals from the sample port at the bottom of the silo for Karl Fischer moisture determinations.

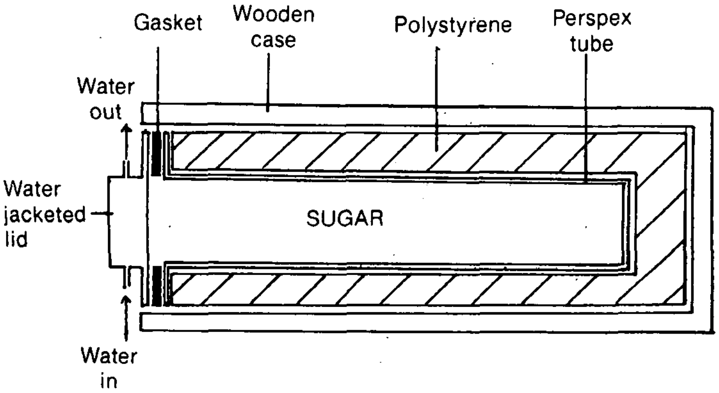


**FIGURE 2** Railway truck simulator

*Caking tests*

The specially constructed railway trucks that transport the conditioned sugar have a diameter of 3 m and a nominal capacity of 50 tons. Because of the large differences between day and night temperatures, especially inland, the most likely place for the sugar to cake would be while in these railway tankers. To establish the potential caking characteristics in transit the small scale simulator of a railway truck previously used by Bruijn *et al*[2] was used (see Figure 2).

This consisted of a 200 mm diameter perspex tube sealed at one end. All sides except the open end were insulated with a 50 mm polystyrene layer and this was encased in a wooden box. The open end was fitted with an airtight metal lid through which water could be circulated. This simulator, which held approximately 40 kg of sugar, was 1 400 mm long which is half the diameter of the railway truck used. The closed end of the tube represented material in the centre of the truck while that in contact with the metal lid represented the sugar in contact with the wall of the truck. By adjusting the temperature of the water flowing through the hollow lid it was possible to subject the sugar to extreme temperature gradients. Hot conditioned sugar was transferred quickly to the caking box and packed tightly. The lid was carefully sealed in position. Cold water (5°C) was circulated through the lid for 15 hours to simulate extreme night time conditions followed by warm water (40°C or 60°C) for 9 hours to simulate extreme day time conditions. After 24 hours the box was opened and the contents were checked for caking.

This large scale simulator was found to be cumbersome and difficult to seal thus a smaller scale caking box was designed

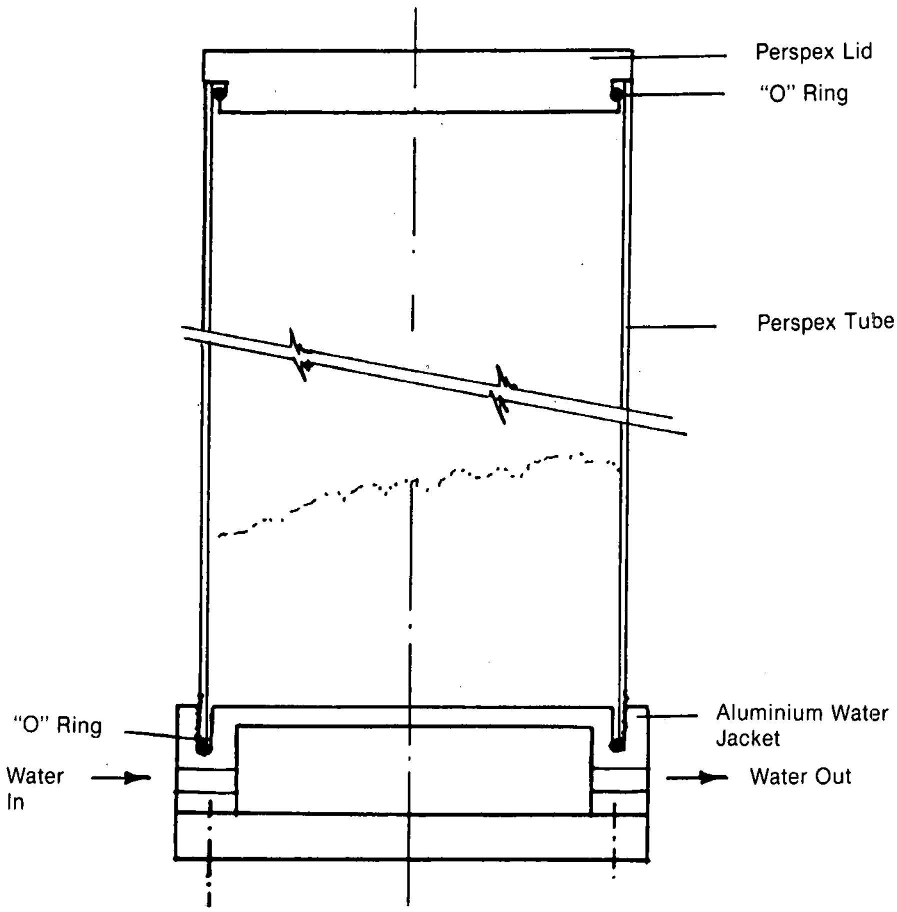


**FIGURE 3** Small-scale caking box

and constructed. This consisted of a perspex tube 400 mm long and 140 mm in diameter. This tube was closed at the bottom end with a hollow aluminium lid through which water was circulated. The sugar was poured in from the top of the tube which was sealed with a perspex lid with an 'O' ring (see Figure 3). The box was then covered with a tight fitting polystyrene cover for insulation. This caking box was much easier to seal and its size made it much easier to handle.

*Moisture determination*

Drying the weighed sugar sample in an oven at 105°C for three hours (SASTA Laboratory Manual[4]) removes only the free moisture and possibly some of the bound moisture. This is therefore not a true indication of the actual moisture in the sample especially when the sugar is high in conglomerates which have been found to trap more moisture than individual crystals. For this reason the findings of this report are based mostly on a moisture content determined by an automatic Karl Fischer titrator using the formamide method of Bruijn *et al.*[2] The formamide dissolves the crystals thus determining the total moisture present.

*Grain size and conglomerate count*

Grain size was determined according to the method in the Laboratory Manual for S.A. Sugar Factories.[4] The standard conglomerate count method described by Bruijn *et al.*[2] was modified as follows to include the larger grains which would previously not have been counted.

The sample was well mixed. A small sample was taken and viewed under a binocular microscope. A section of 100 crystals was marked for viewing. In evaluating the crystals, twinned cyrstals, clusters, star shapes and occluded crystals were taken as conglomerates.

*Equilibrium relative humidity*

The equilibrium relative humidity (ERH) of unconditioned and conditioned refined sugar was determined by weighing sugar samples before and after exposure to atmospheres of known relative humidities (RH) at 20°C. These known RH atmospheres were created in desiccators containing saturated solutions of various salts. The following RH conditions were used: 35, 45 56, 63, 76 and 86%.

## Results and Discussions

*Test tube conditioning and caking Tests*

Test tube conditioning silos were massed every 24 hours. These masses were used to calculate the percent mass loss based on an initial Karl Fischer moisture determination. Graphs were plotted of mass loss against conditioning time. Figure 4 shows three typical runs each consisting of five samples. It can be seen that in most cases a large proportion of the moisture is lost after 24 hours after which the curves flatten.

The caking tests performed on the test tube silos showed however that there was still caking after 24 hours, rarely after 48 hours, but never after 72 hours.

The test tube conditioning silos proved to be a good indication of what would happen on a larger scale.

*Experimental conditioning silos*

These were sampled 24 hourly during conditioning and graphs of Karl Fischer moisture content against conditioning time were plotted (see Figure 5). These graphs show three individual conditioning runs. These curves show a sharp drop in moisture content after 24 hours and thereafter a flattening of the curves. Caking tests in the railway truck simulator showed that sugar conditioned for 24 and 48 hours caked but that sugar conditioned for 72 hours did not.

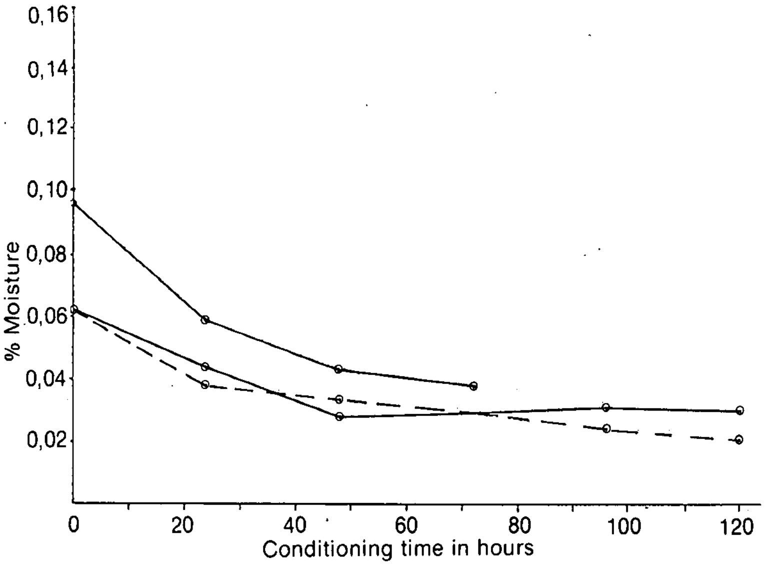


**FIGURE 4** Test tube conditioning

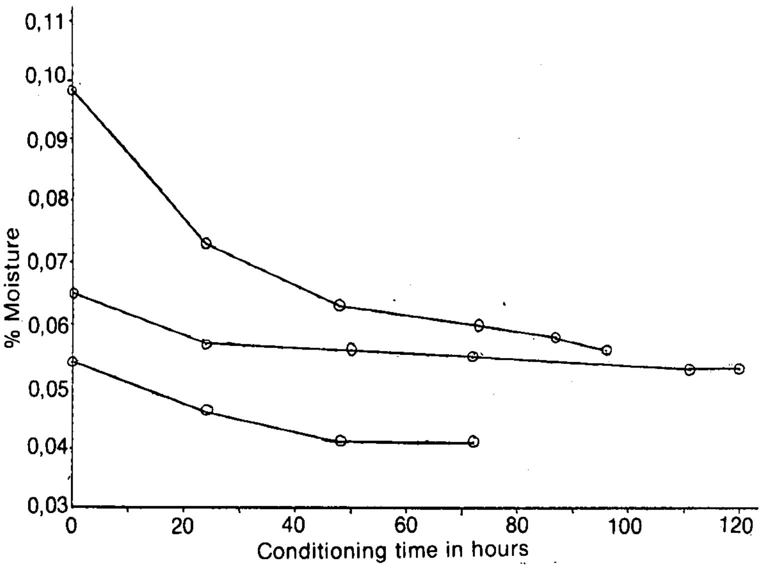


**FIGURE 5** Model silo conditioning

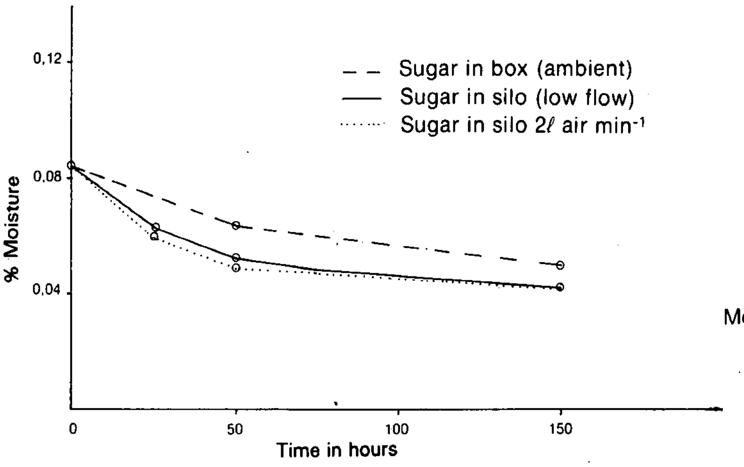


**FIGURE 6** Drying rate during conditioning with different rates of air flow

*Effect of temperature*

Purchase[5] compared the rate of conditioning at 40°C and at 50°C and found that although the sample at 50°C dried faster during the initial 48 hour period the difference was not statistically significant for the number of trials done (see Table 1).

**TABLE 1**

**Effect of temperature on conditioning rate**

| | 40°C | 50°C |
|---|---|---|
| Number of trials | 10 | 4 |
| Mean moisture loss after 48 hrs | 0,029% | 0,034% |
| Mean moisture content after 96 hrs | 0,051% | 0,040% |

The moisture content after 4 days was significantly lower at 50°C than at 40°C thus indicating that lower moisture contents are achieved at higher conditioning temperatures (see Table 1).

*Air flow*

Purchase[5] experimented with air flow and performed the following test. A sample of sugar was divided equally between the experimental conditioning silos and the air flow through one silo was decreased considerably. Results showed that this had no apparent effect on the drying rate and even sugar in a polystyrene box at ambient temperature with a loose fitting lid dried at the same rate but to a higher final moisture content (see Figure 6).

*Grain size and conglomerate count*

It was found that the sugar with a larger portion of grains above 1 700 $\mu$m and a high coefficient of variance did not condition easily. Purchase[5] used test tubes to study the rate of drying of various fractions of sugar. Figure 7 shows that the larger fractions contained more moisture than the smaller grains and continued to release moisture long after the others had stabilised. Conglomerate counts showed these larger particles to be mainly conglomerates indicating that it is these larger conglomerated particles that determine the rate of conditioning.

*Caking of unconditioned sugar*

Unconditioned sugar was always used as a control in test tube caking tests and it was always found to cake in the tube. Unconditioned sugar was placed in the railway truck simulator and subjected to the temperature changes of the caking test. A hard cake 50 mm thick was found at the opening of the tube. Unconditioned sugar was also tested in the new small caking boxes. When subjected to a hot cycle followed by a cold cycle a sticky layer was found in contact with the alluminum base showing that the moisture had been attracted to the cool area.

When a cold cycle was followed by a hot cycle a 30 mm hard cake was formed in contact with the metal base indicating that moisture had been attracted causing a stickiness and then driven away causing the crystals to be 'glued' together into a solid mass.

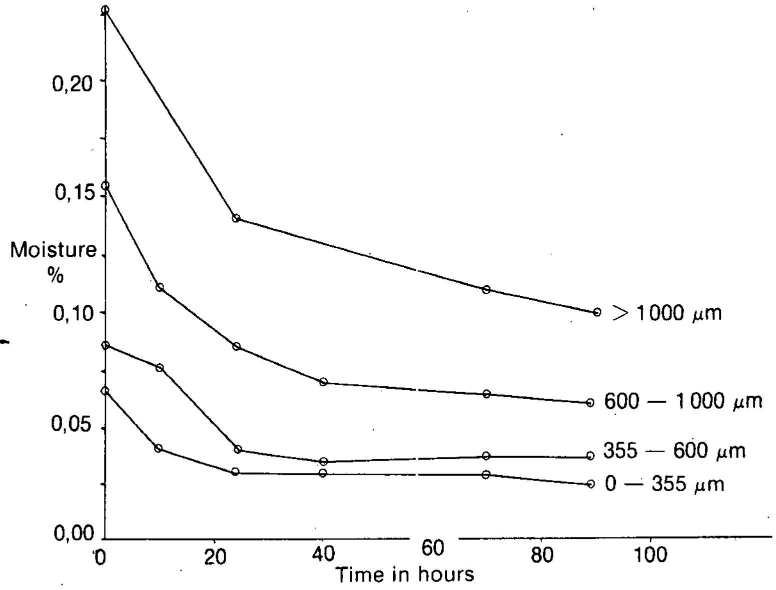


**FIGURE 7** Drying rates during conditioning of various particle size fractions

*Potential movement of residual moisture on storage of conditioned sugar*

If residual moisture in conditioned sugar were to be released slowly during storage over a period of time it could cause caking by accumulating in the coolest areas. To check this, conditioned sugar was stored in sealed plastic containers for 6 to 8 weeks and then subjected to caking tests after this period. In none of these cases did the sugar cake.

*Equilibrium relative humidity*

The percentage loss or gain was calculated and plotted against the percent relative humidity (RH). The point at which no moisture is lost or gained is the equilibrium relative humidity (ERH) which was found to be 76% for both conditioned and unconditioned sugar. This means that if the RH is above 76% (at 20°C) the sugar will absorb moisture from its surrounding atmosphere whereas if the RH is below 76% the sugar will lose moisture to the atmosphere.

*Mixing of conditioned sugar of different moisture content*

If, for example, conditioned sugar from two different origins were sent to Germiston, a situation could occur where sugars of different moisture contents could be stored together in the same silo. It was thus necessary to determine whether such sugars, which do not cake individually, could be induced to cake in various mixtures.

Two conditioned sugars of the following moisture contents were layered in test tubes in eight different combinations.
Conditioned sugar No. 1, Karl Fischer moisture = 0,039%
Conditioned sugar No. 2, Karl Fischer moisture = 0,046%

Test tubes 1 to 8 were subjected to caking cycles and the contents were inspected for caking at the end. None of the samples caked.

## Conclusions

Both NB and HULREF sugar were sufficiently conditioned after 72 hours in order not to cake when subjected to various caking tests. All HULREF and NB sugar conditioned to a Karl Fischer moisture content within the 0,04% to 0,06% range and did not release sufficient of this moisture on storage for six to eight weeks in sealed containers to cause caking.

It is apparent from the tests performed that temperature and air flow do not increase conditioning rate significantly but do influence the final moisture content obtained. Larger grains and conglomerates were found to contain more moisture which was given up more slowly on conditioning thus indicating that the greater their presence in the sugar the longer the time required to condition such sugar.

New equipment for testing the behaviour of refined sugar was developed as the tests progressed. The experimental silos still remain an excellent means of conditioning 40 kg of sugar whereas the cumbersome railway truck simulator was replaced by the smaller caking boxes. The test tube conditioning appartus gives a good indication of how the sugar will behave in the larger scale tests.

On the basis of the tests described in this paper the conditioning silo was constructed at HULREF and plans for a second silo at NB are underway.

## Acknowledgements

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