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# Particle Size Effect on Caking in Sucrose

A Thesis presented in partial fulfilment of the requirements for the degree of Masters in Chemical Technology at Massey University

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### - Abstract -

Caking, and the associated loss of flowability, have for a long time been a problem in the sugar industry, causing difficulties during conveying and making the product unacceptable to consumers. There are many factors that are thought to contribute to this problem, including relative humidity, compaction and packing of the crystals, and particle size.

In order to determine if particle size did have an effect, a series of samples containing different sized crystals, and different proportions of fine crystals were created. These samples then had their water vapour sorption isotherms measured by equilibrating samples over saturated salt solutions. Caking tests were also carried out using the friability test and the blowtest. No significant difference was found on either the isotherms or the friability test. The blowtest, however, was found to be much more sensitive to the small differences in caking occurring between samples. It was found that only fines less than 150  $\mu$ m had any effect on caking, and even then, only when they were present in large quantities. In addition, the smaller the particles, the smaller the amount required for caking to occur. For example, the critical water activity for standard sugar was found to be 0.61. For a sample of 100% 212-315  $\mu$ m particles this decreases to 0.55 and for a sample of 0-75  $\mu$ m this decreases even further, to 0.22.

No additional effect was found to be caused by crystal damage, over and above the effect of decreased particle size.

It is proposed this increase in caking in fine particles is due to capillary condensation. The smaller the crystals, or the greater the proportion of fines small crystals present, the more contact points between particles. Between each of these contact points capillary condensation can occur, which means more moisture can be absorbed at a lower water activity, therefore, the amount of water needed for caking to occur is also reached at a lower water activity. This effect is very small, and neither the isotherms nor the friability test was able to detect these changes, but the blowtester was able to.

Some of these fine crystals will originate in the crystallisation process, however many of the fine crystals are a product of attrition. It was found that this was a problem when sugar was conveyed using a screw conveyor, but not when a redler chain conveyor was used. In addition, there were no differences found in the amount of attrition occurring when conveyors are run at less than full loads. It is recommended that in future construction and modification of the plant, chain redlers be considered rather than screw conveyors.

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## - Acknowledgements -

"However high we climb in pursuit of knowledge, we shall see heights above us, and the more we extend our view, the more conscious we shall be of the great immensity that lies beyond" - D.A. Wrangham

My pursuit of caking in sucrose started in Palmerston North, over a year ago. Since then I have spent nine months in Auckland – somewhere I've always vowed I never wanted to live, then a few weeks in Taranaki, before returning to the familiarity of Palmerston North. During this time there were many people who helped me in my pursuit.

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## Chapter 1

## - Project Overview -

#### 1.1 Introduction

Caking in bulk sucrose is a major problem in the sugar industry. Not only are caking problems costly, but they also cause frustrations to customers, and with this, the potential loss of trade. At New Zealand Sugar it has been found that these adverse effects of caking are most severe when bulk sugar is transported to customers some distance from the refinery in Auckland. In extreme cases, 20 tonnes of bulk sugar has set into a single lump, requiring manual intervention to allow sugar to flow.

Recent studies at New Zealand Sugar have quantified the effects of temperature gradients on induced caking in bulk sucrose and it is now known what water activity the sugar needs to be when it is packed and transported in order to avoid caking problems (Billings, 2002). Caking problems can also be minimised by conditioning, the blowing of dehumidified air through the bulk sugar before packing, and also by insulating the walls of the sugar container after packing to minimise temperature gradients (Rastikian & Capart, 1998).

There is anecdotal evidence that fines are instrumental in the caking problem. Some of these fines are produced in the crystallisation process, however most are thought to be the result of attrition as the sugar crystals are conveyed.

#### 1.2 Caking

Caking is the formation of solid lumps from formally free flowing powder. These lumps are caused by temperature gradients in the bulk material. This causes fluctuations in interstitial air relative humidity since warm air holds more moisture than cooler air. Moisture migrates to restore the equilibrium, creating moisture concentration gradients. In areas of high relative humidity, moisture is adsorbed and condenses onto particle surfaces. This causes the dissolution of the particle surface, and the formation of liquid bridges consisting of solution. As the equilibrium changes again these liquid bridges dry out, creating a solid bridge between crystals particles. In addition, capillary condensation caused by surface tension effects may cause caking to occur at a lower humidity.

There have been conflicting reports published in literature as to whether the presence of fine crystals in sucrose increases the potential for caking to occur (Roge & Mathlouthi, 2000, 2003), or whether the bulk sucrose is so soluble that this effect does not exist (Johanson & Paul, 1996).

#### 1.3 Attrition

Attrition can be defined as the unwanted breakage of particles, resulting in a decrease in particle size. This breakage can take two forms – abrasion or fracture. The first of these, abrasion, occurs when edges or corners are removed, resulting in a number of very small particles and a slight decrease in size of the parent particle. Fracture, meanwhile, occurs when the parent particle splits into at least two particles that are of a comparable size.

Damage occurs to crystals when particles hit each other at high velocity, such as in a pneumatic conveyor, or when particles are subject to shear. At the New Zealand Sugar factory crystals are conveyed using both screw conveyors and chain redler conveyors. It is thought that the latter caused more damage, since particles are subject

to shear forces, both between the blades and the casing of the conveyor, and between the blades and other particles.

#### **1.4 Project Objectives**

The aims of this project were as follows:

- To identify if fine crystals have an affect on caking
- To determine what mechanism caking is occurring by
- To identify where in the New Zealand Sugar plant damage to crystals is occurring
- To identify if crystal damage has any affect on caking apart from that related to size

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