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# The practical implications of temperature induced moisture migration in bulk lactose

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#### ABSTRACT

The caking of bulk lactose during transportation is an on going global problem. This paper establishes the limits of water activity that must be achieved before the lactose is bagged off to ensure that temperature gradients encountered during cooling or transportation do not cause caking in the bags. This information is summarised in a graph that can be used to predict the final water activity of the product that must be achieved in order for the product not to cake due to known imposed or anticipated temperature gradients. A water activity of below 0.3 is recommended for meeting the typical temperature gradients encountered during transportation if there is no amorphous lactose present on the surface.

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#### 1. Introduction

Caking of lactose after bagging or during transportation has been an ongoing problem for the dairy industry. The reasons for caking in sucrose have been well documented with the mechanism being identified as "humidity caking", whereby powder exposed to high relative humidity air adsorbs water from the air which collects between the powder particles as a liquid due to capillary condensation and subsequently forming liquid bridges. The water dissolves some of the surface of the sugar and when conditions change and the water evaporates, a solid bridge is created between the particles, causing a solid cake to form (Bagster, 1970a,b; Bruijn et al., 1996; Irani et al., 1959; Leaper et al., 2003, 2002; Ludlow and Aukland, 1990; Mathlouthi and Roge, 2001; McGinnis, 1971; Meade, 1963; Moss et al., 1933; Nelson, 1949; Nichol, 1990; Pancoast and Junk, 1980; Pennington and Baker, 1990; Roge and Mathlouthi, 2000, 2003; Scoville and Peleg, 1981).

The humidity of the air in a bag of powder is a function of both the moisture content of the product and the temperature of the powder in contact with the air. The air will generally be in equilibrium with the moisture content of the powder as described by the moisture sorption isotherm. When a temperature gradient is imposed on the bulk sugar, either by the diurnal changes in environment or when the sugar is packed out hot and is put into the warehouse to cool, the moisture within the bag will move from

the hot region to the cold region. Fig. 1 shows how this happens taking the case where hot powder is bagged off and then placed in a cooler environment so that the outside of the bag starts to cool down. When this happens, the relative humidity of the air near the surface increases, as the ability of the air to hold water decreases with decreasing temperature. This in turn leads to an increase in the amount of water that will be adsorbed onto and between the particles, as shown by the isotherm for lactose (as an example sugar) in Fig. 2. As the water moves from the air to the particles it reduces the absolute humidity of the air near the surface, thus setting up a moisture gradient in the air between the hot region and the cooler region. This moisture gradient leads to mass transfer by diffusion from the middle of the bag to the outside. As moisture moves away from the hot middle section, it lowers the relative humidity of this air, which in turn leads to desorption of more moisture from the powder in the centre of the bag.

Through this mechanism, if enough water is present in the powder when initially bagged off and if the temperature gradient is high enough, then the relative humidity of the air will be high enough to cause capillary condensation between the particles causing liquid bridging and dissolution of part of the surface of the particles. As the centre of the bag cools down, eventually the moisture will start to redistribute back through the bed, so that when the temperature of the powder is uniform, the moisture will also be evenly distributed throughout the powder bed. This will result in the liquid bridges that had formed due to the locally high relative humidities, recrystallising to form solid bridges. Thus a temperature gradient can significantly alter the distribution of adsorbed

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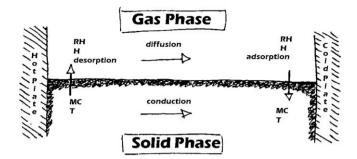


Fig. 1. Mechanism of moisture movement induced by a temperature gradient.

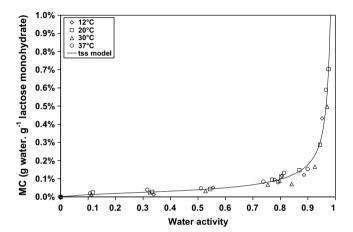


Fig. 2. Sorption isotherm for lactose with fitted third stage sorption model (TSS). Bronlund and Paterson (2004).

moisture within a bag of powder leading to significant amounts of caking and/or lumping.

This process has been mathematically modeled by Bronlund and Paterson (2008) and Leaper et al. (2002) confirming that the moisture movement within crystalline powder beds can be predicted. In order for these models to be useful, it is necessary that the relative humidity conditions that lead to significant levels of caking be established. Leaper et al. (2003) used a tensile tester to measure the strength of particle bridging that occurred when the relative humidity was cycled between 70% and 20% as a function of the number of cycles. No work was found in the literature relating the strength of caking formed from the humidity caking mechanism for lactose.

The aim of this paper is to present the work establishing the relative humidity conditions needed to cause significant levels of caking in bulk lactose and to use the mathematical model presented in Bronlund and Paterson (2008) to predict what temperature gradient and initial lactose water activity are needed for these conditions to be induced within the bag.

In order to do this a method needed to be found that could measure the caking strength of powders that had been exposed to various levels of relative humidity and then dried out and the strength of the particle–particle bounds found. Ideally, the method chosen had to be able to be used in the experimental apparatus used to validate the mathematical model which consisted of a large donut polystyrene ring filled with lactose and subjected to a temperature gradient. Details of the validation work are given in Bronlund and Paterson (2008). The literature reports numerous methods including tensile strength methods as summarised by Schubert (1975) and Kawashima (1991), shear test and cohesion methods as reviewed by Hasegawa et al. (1985)

and compaction and compressibility tests (Abdel-Ghani et al., 1991; Hollenbach et al., 1982, 1983; Moreyra and Peleg, 1981; Riepma et al., 1992; Scoville and Peleg, 1981). These methods were all unsuitable for measuring the caking strength of the powder within the validation equipment, which required a measurement devise that could measure the caked strength of powder within a bed inside a 1200 mm diameter ring. Penetrometry has been used in the field of soil science to characterize soil systems. McLaren and Cameron (1990) and Baker and Mai (1982) used a multi-point penetrometer to measure the strength of the surface layer of soil. In this case the size of the probe was close to the particulate size, thus ensuring that the inter-particle forces were being measured, rather than an overall compression strength.

A multi-point penetrometer was developed to test the caking strength as it could be used on individual samples of powders equilibrated under different relative humidity conditions as well as being suitable for measuring the caking strength of the lactose in the validation rig.

#### 2. Materials and methods

All lactose samples were alpha lactose monohydrate, supplied by Lactose New Zealand Co. as manufactured. Typical size distributions for the special dense, 100, 200 and 300 mesh lactose grades used are presented in Table 1.

The penetrometer developed consisted of a probe made of thirty two 1 mm diameter stainless steel pins pressed into a Perspex disc in a regular circular pattern to a constant depth. Each pin was filed flat to ensure a known contact area. The probe was mounted on the arm of a stripped down beam balance (as shown in Fig. 3) which, through the counterweight system allowed the probe to be set up so that it touched the surface of the powder to be tested in a weightless manner. Weights of water were then incrementally added to provide the downward force until such time as breakthrough of the surface was achieved. This force divided by the area of the pins was taken as the strength characteristic of the bulk powder.

Samples of lactose crystals were placed in plastic Petri dishes 75 mm in diameter and 5 mm deep, leveled with a straight edge and the dishes placed over saturated salt solutions at 18 °C in sealed chambers to give known relative humidity environments. Table 2 gives the salts used with their equilibrium relative humidity. The samples were left for 1, 10 and 50 days for equilibration and the strength of the liquid bridges determined using the multi-point penetrometer immediately after removal from the equilibration chambers. For the solid bridge strength measurements, samples that had been first equilibrated for 50 days over the saturated salt solutions were ambient air dried (RH 50–70%) for 7 days before testing.

**Table 1**Particle size distributions of the as supplied lactose samples: weight fraction passing given size

Size (µm)	Special dense	100 mesh	200 mesh	300 mesh
20	-	-	-	0.24
30	-	0.1	0.3	0.48
40	0.04	0.13	0.43	0.67
50	0.07	0.17	0.56	0.78
60	0.1	0.22	0.63	0.86
80	0.16	0.3	0.84	0.93
90	0.2	0.35	0.88	0.94
100	0.24	0.4	0.91	1
200	0.63	0.8	1	1
300	0.85	0.94	1	1

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