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Review

Caking of lactose: A critical review

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ABSTRACT

Background: Caking is a recurrent problem in various industries, whether it occurs during the production, storage or transport of powders. Caked powder results in longer processing times and decreased product quality, leading to significant economic loss. Several caking mechanisms have been described in the literature. However, they are often difficult to take into account in an industrial context, given the many parameters which influence the overall caking phenomenon.
Scope and approach: This review describes the three relevant caking mechanisms for food powders in general. Focussing on predominantly crystalline lactose powder, we discuss how each of these mechanisms can explain caking and be prevented in the industrial context. The second part of this paper presents a critical review of the methods used to characterise caking to date.
Key findings and conclusions: The presence of amorphous material and other impurities must be assessed in crystalline lactose powders, as they can trigger amorphous and humidity caking. Particle size distribution is another key parameter requiring control as it can encourage caking through enhancement of particle interactions. In general, preventing caking in food powders can only be achieved by a thorough understanding of the production process and storage conditions. Moreover, the characterisation of caking remains a challenge as most methods published in the literature do not fit the needs of the food industry. The real demand is for a reliable method to predict caking which would be rapid and easy enough to be applied to each batch for quality control.

1. Introduction

Lactose is the principal sugar in milk, constituting about 40% of the dry matter in whole milk and up to 80% in whey (Hobman, 1984). Cheese manufacture yields large quantities of whey as a by-product. Cheese production in 2013 amounted to approximately 21 million tonnes worldwide. The corresponding whey by-product was 186 million tonnes of liquid whey or 12 million tonnes of whey solids (Affertsholt & Fenger, 2013). Because of commercial and environmental concern, the traditional disposal of whey as pig feed or in sewage water has evolved towards further processing of whey (Fox, 2009). Whey can now be ultrafiltered to separate the whey proteins from the lactose and minerals. The most common form of lactose commercially available is produced by slow crystallisation in a cooling tank following various concentration stages. The large crystals obtained are then washed of minerals and other impurities and finally dried in a fluidised bed dryer.

Despite growing consumer concern about lactose intolerance, the food and pharmaceutical industries have found many applications for lactose, mainly in the crystalline form. Indeed, lactose is the main constituent of breast milk, and infant formula manufacture is therefore the most important market for lactose producers, requiring high quality and safety standards. Other food applications of lactose include confectionary and baked goods (Lifran, Hourigan, Sleigh, & Johnson, 2000). Lactose is also commonly used as an excipient and tableting agent in the pharmaceutical industry.

The demand for infant formulae in emerging countries is growing. The global production of lactose powder consequently increased from 947,000 tonnes in 2009 to 1.255 million tonnes in 2013 and is expected to reach 1.5 million tonnes in 2017 (Affertsholt & Fenger, 2013). However, achieving quality requirements in the dairy industry is often complicated by the well-known issue of

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caking of lactose powder. Caking can take different aspects in the powder, can occur at several points during manufacture, transportation and storage and have different causes. As an illustration, caking can be seen as a hard crust of powder blocking a spiral conveyor. However, the same term is used for the formation of both loose and hard agglomerates of various sizes in a bag after storage. A general definition of caking is therefore difficult to formulate. In all cases, the formation of caked powder has a negative impact on product manufacturability and quality. It is thus associated with additional costs as it can stop the production process or require further processing to regain a free-flowing powder. Customer complaints are also common in such a situation, which can affect the supplier financially in the long term. There is consequently an urgent need for greater understanding of the caking mechanisms and for means to control them.

2. The lactose molecule

Lactose is a disaccharide composed of a D-galactose and a D-glucose unit bonded through a β-1,4-glycosidic linkage. Lactose (4-O-ß-D-galactopyranosyl-D-glucopyranose, $C_{12}H_{22}O_{11}$) can occur in α and β forms. As can be seen in Fig. 1, the two forms are stereoisomers, which differ by the spatial arrangement of the hydroxyl group at carbon number 1 of the hemiacetal group. The α-form has the greater optical rotation in the dextro direction (Holsinger, 1988). In solution the rate of the transformation between the α- and β-anomers, called mutarotation, is temperature- and pH-dependent. On the other hand, the ratio at equilibrium depends only slightly on temperature and is not affected by pH (Holsinger, 1997).

Lactose can be found in a crystalline state, an amorphous state or a mixture of both. By definition, crystalline lactose presents a very ordered structure, with the exact shape of the crystal depending on the crystallisation conditions. In amorphous lactose, the lactose molecules are not organised according to a regular lattice. Moreover, lactose is polymorphic, meaning that it can crystallise into different forms. The six currently known forms of lactose are presented in Table 1. The crystalline form α-lactose monohydrate has a different chemical composition due to the inclusion of water in the crystal structure. It is however often presented as a lactose polymorph in the literature (Kirk, Dann, & Blatchford, 2007).

The properties of each lactose form have been summarised by Listiohadi, Hourigan, Sleigh, and Steele (2005b). The most important of these properties in relation to the caking phenomenon are given below.

2.1. α-lactose monohydrate

α-lactose monohydrate is the most common and stable form of lactose under normal temperature and humidity conditions. The crystal structure includes one molecule of water of crystallisation for each lactose molecule. This water molecule is crucial to the structure and stabilisation of the crystal lattice as it links together oxygens of four lactose molecules (Clydesdale, Roberts, Telfer, & Grant, 1997). It also explains the relative non-hygroscopicity (ability to attract moisture from the atmosphere) of α-lactose monohydrate. At 25 °C this form of lactose is stable below 95% relative humidity (RH) (Salameh, Mauer, & Taylor, 2006). The most common crystal shapes for α-lactose monohydrate are tomahawk and prism shapes (Holsinger, 1988) as seen in Fig. 2. The process commonly used in the dairy industry to obtain this lactose crystal form consists of slow cooling of whey permeate concentrate in a crystallisation tank (Hourigan, Lifran, Vu, Listiohadi, & Sleigh, 2013).

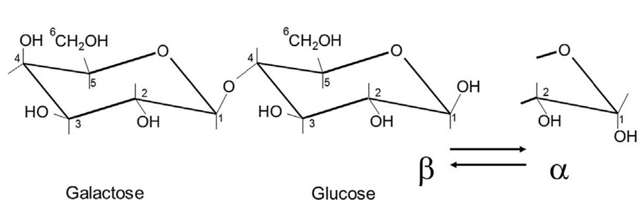

Fig. 1. Lactose structure in α and β forms (Schuck, 2011).

Table 1
Currently known forms of lactose (from Listiohadi et al. (2005b))

Crystalline	Monohydrate	α-lactose
	Anhydrous	Unstable α-lactose
		Stable α-lactose
		β-lactose
		Compound β/α lactose
Amorphous		Mixture of α-lactose and β-lactose

2.2. Stable and unstable anhydrous α-lactose

Stable and unstable forms of anhydrous α-lactose are derived from the previous form upon release of the water of crystallisation from the crystal lattice. In unstable anhydrous α-lactose, the arrangement of the lattice is left unchanged, giving a highly porous and unstable structure. This lactose form is therefore extremely hygroscopic (4% water sorption at 10% RH and 20 °C (Figura & Epple, 1995)) and reforms α-lactose monohydrate upon moisture sorption. In contrast, stable anhydrous α-lactose presents a different crystal structure to that of α-lactose monohydrate. This form is stable at room temperature and relative humidity below 50% (Figura & Epple, 1995).

Both forms can be produced from α-lactose monohydrate by heating at temperatures between 100 °C and 190 °C, preferably under vacuum. It has been shown that unstable anhydrous α-lactose is a precursor of the stable form during thermal dehydration of the monohydrate lactose form (Figura & Epple, 1995). Stable anhydrous α-lactose can also be produced by different techniques using an organic solvent not miscible with water.

2.3. Anhydrous β-lactose

Anhydrous β-lactose is the only form of the β anomer of crystalline lactose. This lactose form is stable below 95% RH at 25 °C and therefore less hygroscopic than anhydrous α-lactose (Salameh et al., 2006). The crystal shapes differ from those of the α anomer. Crystallisation of anhydrous β-lactose from water yields uneven-sided diamonds (Fig. 3) while crystallisation from alcohol leads to curved needle-like prisms (Holsinger, 1988). Different ways of producing anhydrous β-lactose have been reported in the literature, either from a saturated lactose solution or from α-lactose monohydrate. Most of these methods involve heating the saturated solution at high temperature, as lactose preferentially crystallises in the β form at temperatures higher than 93.5 °C (Hudson, 1904). Independently of the method used, obtaining a powder of one anomer completely free of the other appears to be particularly difficult. Commercial anhydrous lactose contains up to 80% β-lactose and is usually produced by roller drying (Hourigan et al., 2013).

2.4. Compound crystals of anhydrous α- and β-lactose

The compound crystals of anhydrous α- and β-lactose are