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Caking of crystals: Characterization, mechanisms and prevention

Mingyang Chen^a, Songgu Wu^a, Shijie Xu^a, Bo Yu^a, Mohannad Shilbayeh^d, Ya Liu^a, Xiaowen Zhu^e, Jingkang Wang^{a,b,c}, Junbo Gong^{a,b,c,*}^a School of Chemical Engineering and Technology, Tianjin University, Tianjin, China^b Key Laboratory Modern Drug Delivery and High Efficiency in Tianjin University, Tianjin, China^c The Co-Innovation Center of Chemistry and Chemical Engineering of Tianjin, Tianjin, China^d Faculty of Engineering, Western University, London, ON N6G 1G8, Canada^e Tianjin University, Tianjin, China

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ABSTRACT

Caking of crystals leads to a great deal of negative impact on the quality of products leading to extra cost to restore the products to its original state before caking. It is therefore an undesired phenomenon that should be investigated and inhibited. This review considers particle issues (particle science and technology) in a crystallization view (crystallization science and engineering). 1. Characterization. Caking indices and the relevant characterization methods are classified. Their advantages and disadvantages are summarized. Particularly, potential novel characterization methods, e.g. image analysis technique and its application in analysis of caking ratio, are discussed. 2. Mechanisms. Caking process is organized into three stages: moisture sorption, then liquid bridge, and finally crystal bridge. The mechanisms are reconsidered with recent studies (e.g. mass transfer, adhesion effect of liquid bridge, kinetic of crystal caking) and crystallization science (e.g. nucleation and growth theory, polymorphism, and phase transition). 3. Prevention. Based on particle and crystal engineering, prevention of crystals caking is discussed through controlling ambient conditions (relative humidity, temperature, liquids screening, and pressure) and improving anticaking property of crystals (particles size, particles shape, impurity, and anticaking agent). A potential method of simulating crystal caking behavior based on discrete element method (DEM) and caking mechanisms is presented. With the aid of simulation, the enhanced design of particle and crystal engineering would be available, which could be a key to control these factors systematically.

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1. Introduction

In the crystallization field, most crystalline products are preferred to be produced as single particles with good flowability and stability. These crystals are easy to process prior to commercialization. Caking of crystals often occurs after a distinct drying step, e.g. transport, storage and some drying processes. It can be very problematic due to unusable or additional equipment being required for de-caking [1]. Quite often 2–10% of the product is lost during this process [2,3]. To make matters even worse, the quality of products after the process of de-caking sometimes differs significantly from the original ones, which may cause a series of problems in the following processes and commercialization. Hence, in most cases, caking of crystals is undesired and should be prevented. However, most studies focus on the amorphous powders in the food engineering field instead of crystals. Caking of amorphous powders is generally caused by glass transition, a variation of viscosity

and elasticity [4–9], which is significantly different in comparison with the caking mechanisms of crystals [1,5,10–13]. The relevant mechanisms of amorphous powders therefore cannot be adapted to crystals. In addition, polymorphic transitions, phase transitions, chemical reactions, and the debate of nucleation and growth of crystallization complicate the study of crystals caking. Hence, there remains a strong need to consider particle science and technology with crystallization science and engineering to overcome these issues (Fig. 1). As a foundation, particle science and technology provides basic means of characterization, particle–particle interactions and particle system engineering. Furthermore, the crystallization science and engineering contains nucleation and growth theory, polymorphism, phase transition, crystal engineering, etc., which may give crystals caking a profound view on mechanisms and the prevention methods.

This review focuses on the caking of crystals, especially during transport and storage. In the following sections, characterization, mechanisms, and prevention of crystals caking are summarized and discussed with the aid of recent studies. The motivation of novel caking indices and devices in particle technology are discussed in the section of characterization. The mechanisms of crystals caking are reviewed from

* Corresponding author at: School of Chemical Engineering and Technology, Tianjin University, Tianjin, China.

E-mail address: Junbo_gong@tju.edu.cn (J. Gong).

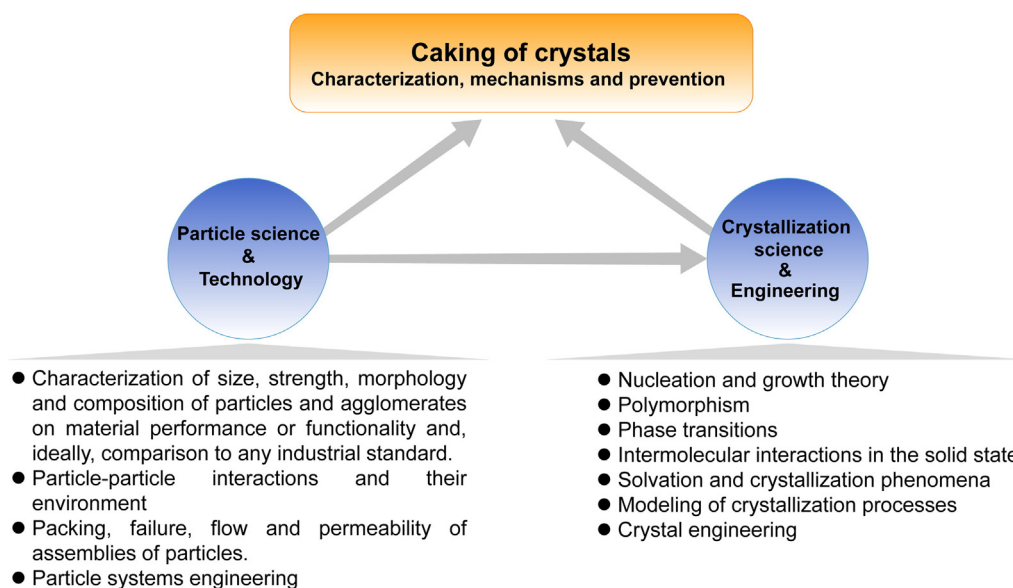


Fig. 1. Solution of crystal caking combined particle science and technology with crystallization science and engineering.

the viewpoint of crystallization science. The polymorphism, phase transitions, chemistry reactions, nucleation and growth theory are considered to discuss the possible research points. Based on particle and crystal engineering, and the idea of “quality by design”, the factors for caking prevention are summarized systematically.

2. Characterization

Several indices (Fig. 2) are proposed in characterizing crystal caking. Caking strength and compressibility are classified as mechanical indices. Caking strength is used to characterize the force breaking the cake [13, 14]. Compressibility shows the shrinkage of the specimen during caking, where the pressure is related to the extent of powder caking [15].

Aspect ratio, circularity, convexity, sphericity and bluntness are morphological indices. They characterize the shape and structure of a particle. Aspect ratio (AR) is the ratio between the major axis and minor axis of the ellipse encircling the object [16]. Circularity is the ratio of the object's projected area to the square of the perimeter of the object [17]. Convexity is the perimeter of the convex hull of the object divided by its perimeter [16,18]. It presents how spiky a particle is. Sphericity is the ratio of the surface area of a sphere having the same volume as the particle to the actual surface area of the particle [18,19]. Bluntness describes how sharp or round the fine convex structures on the particle surface are [20]. In general, agglomerates present irregular shapes. These morphological indices of agglomerates differ a lot from the primary individual particles, so sorting and filtering on these indices will quickly identify the level of caking.

Statistical indices include caking ratio, mechanical indices distributions, and morphological indices distributions. Caking ratio is to calculate the percentage of caked particles, which characterizes the degree of caking [21–23]. Distributions of mechanical and morphological indices are statistical data from a mass of individual particles, which show comprehensive views on the whole sample.

Caking test methods have been developed based on these caking indices. Most available approaches tend to inform the operator whether a product has caked or not. A highly desirable feature of a caking test is the ability to diagnose whether caking will occur in the future. The common and potential caking test methods (Fig. 2) for these caking indices are introduced below. Mechanical indices are common in literature. The conventional methods of these indices are summarized in Section 2.1. Morphological indices are less common than mechanical ones but could be utilized directly for caking due to the improvement of

techniques in recent years. The novel techniques for these indices are discussed in Section 2.2. Most of the statistical indices are not common yet but can be valuable. The potential techniques of these indices and some potential ideas to obtain more precise and repeatable data are discussed for further applications in Section 2.3.

2.1. Mechanical indices

Many researchers focused on the mechanical indices in the past years due to good development of testers. These testers are easy to establish. Some are even commercial, e.g. shear testers, which motivate the investigation about mechanical indices, especially caking strength. Some groups have also developed devices that control humidity, temperature, pressure, and location of particles to fit more specific situations. These testers are summarized in Table 1.

2.2. Morphological indices

Particle morphology is one of the most important aspects in particle science and technology. Caked particles have more complex morphological characteristics, which is valuable to investigate. So far, utilizing atomic force microscopy (AFM) technology has become the tendency to investigate particle surface details in nanoscale. With the aid of AFM, Bode et al. [43,44] observed step pinning of NaCl crystal caused by the anticaking agents, i.e. ferrocyanide and iron (III) meso-tartaric acid. The AFM showed that the step heights are only one atomic layer (2.8 Å). Then, a blocking mechanism for crystal growth of sodium chloride crystals by ferrocyanide was suggested. Stellacci et al. [45,46] developed the high-resolution amplitude-modulation atomic force microscopy (AM-AFM) to improve the precision of AFM significantly. The images could even show a single molecule or atom. In their subsequent work, this technology demonstrated that the stearic acid molecules tend to act as “pinning points” on the calcite's surface and slow down the crystal's restructuring kinetics [47]. These experiments utilizing AFM have offered some possible ideas for anticaking agent. Besides, AFM also provides both morphological microstructure and interactions. This advantage could provide insights into caking mechanisms. However, using AFM is costly, time consuming [48], and limited to the study of massive particles system. For this problem, optical and scanning electron microscopes could be adopted. Previously, image analysis based on optical and scanning electron microscopes primarily depends on manual measurement of numerous photographs [49,50]. However,

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