



# Investigation of the moisture-induced caking behavior with various dietary salts

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## ABSTRACT

Caking of dietary salt is a well-known problem in food industries. In this study, caking behavior of various kinds of dietary salt, including refined salt, sea salt (from South Korea and France), bamboo salt, rock salt, and sodium chloride (used as control group) was investigated. According to our results, these dietary salts had different mineral content and moisture sorption isotherm. The sea salt had more minerals than the other salts. As a result, the deliquescence relative humidity of the sea salt was significantly lower than that of the other dietary salt, which resulted in higher water sorption ability. After salts were stored in two humidity cycle conditions, the change in moisture content, caking strength, passed ratio, and morphology was determined. In the case of sea salt, the highest caking strength, the lowest passed ratio, and lots of crystal bridges caused by caking were observed.

## 1. Introduction

Caking is a detrimental phenomenon wherein powders are transformed into undesirable materials, resulting in not only a loss of quality and functionality but also severe economic losses (Barbosa-Cánovas et al., 2006). Caking usually occurs in powdered materials including foods, pharmaceuticals, fertilizers, inorganic salts, and others. (Fitzpatrick et al., 2010; Lipasek et al., 2012). These powders may experience significant changes in temperature and relative humidity (RH) atmospheres during processing, handling, and storage which can alter their appearance (Fitzpatrick et al., 2010). During handling and storage, dry materials can become caked because of the unwanted agglomeration of particles (Carpin et al., 2017). This results in waste in these industries. Since humidity is the one of the most critical factors in the caking mechanism, it is essential to investigate the behavior of powdered materials and to control the humidity of the environment (Carpin et al., 2017; Mauer and Taylor, 2010).

Recently, many investigations have been examining various caking mechanisms depending on the powder size, types, components, and other factors (Bode et al., 2012, 2015; Carpin et al., 2017; Chen et al., 2017; Fitzpatrick et al., 2010; Lipasek et al., 2012). Powder materials are generally divided into two groups: amorphous powders and crystalline powders. Each group has different caking mechanisms. For instance, caking of amorphous powders is usually caused by glass

transition temperature ( $T_g$ ), viscosity, and elasticity (Hartmann and Palzer, 2011; Silva et al., 2015). On the other hand, caking of crystal powders is related to polymorphic transitions, phase transitions, chemical reactions, and growth of crystallization (Chen et al., 2017). Among many factors, humidity and mechanical caking can occur in both powder types and are the most powerful factors (Barbosa-Cánovas et al., 2006; Fitzpatrick et al., 2010). Humidity cycling, when powders are exposed to high relative humidity conditions followed by low relative humidity conditions, can especially lead the formation of solid bridges between crystalline powder particles. These phenomena usually occur in highly soluble particles, for instance, dietary salts. During exposure to high humidity, liquid bridges containing dissolved constituents form and these bridges are converted to solid bridges as the moisture is vaporized during low humidity conditions. This re-crystallization may cause severe caking in bulk crystalline powders. Fig. 1 shows the process of the caking in crystalline powder. In the present study, six dietary salts were stored in two humidity cycling conditions: one from RH 57%–83% and the other from RH 15%–95%. The differences depending on the range were also investigated. The humidity cycles were chosen to simulate conditions that may be experienced by the powder during handling and storage processes.

Sodium chloride (NaCl) is traditionally a well-known example of a caking material (Bode et al., 2015). Dietary salt mainly consists of NaCl. Since NaCl is an essential component in our body, dietary salt is

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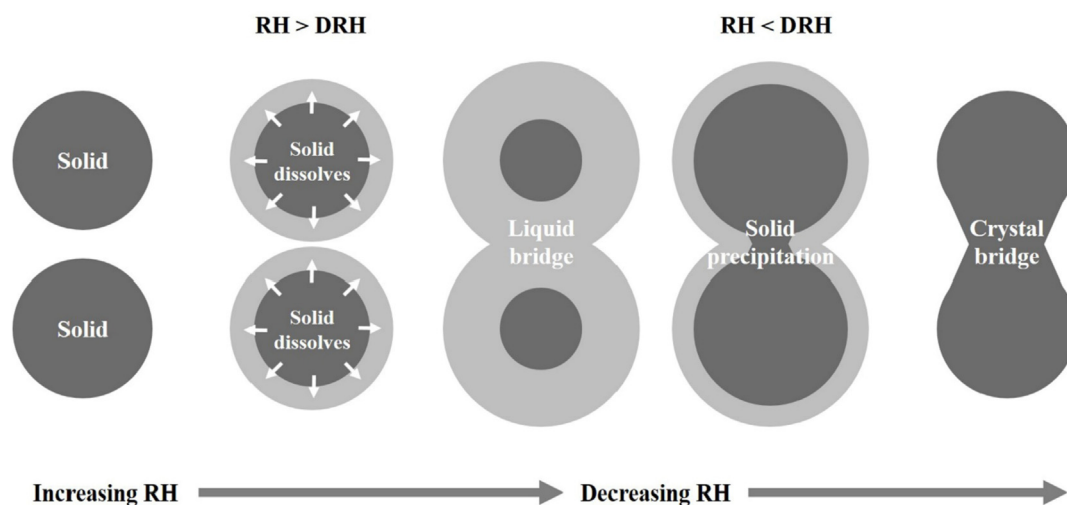


Fig. 1. Processes of moisture induced caking in crystalline powder.

considered one of the most important materials in the food industry (Bode et al., 2012). It is usually produced in large amounts in food industry and stored and transferred very often. In this process, dietary salt can become caked. Nowadays, many kinds of table salts are consumed, including refined salt, sea salt, rock salt, and bamboo salt as representative examples. According to the study of Ha and Park (1998), these various kinds of salts have different contents of minerals like calcium, magnesium, and potassium (Ha and Park, 1998). In other words, various kinds of salts have different compositions. Therefore, in this study, we focused on the fact that different mineral contents can affect the caking behavior of salts. Various dietary salts including refined salt, sea salt (from South Korea and France), bamboo salt, rock salt, and NaCl used as a control were prepared for investigating caking behavior.

The aim of this study was to study the caking behavior and estimate the caking strength of many kinds of dietary salts. We investigated the mineral content and measured water sorption of different kinds of dietary salts. Furthermore, in humid cycling storage conditions that mimic handling conditions, moisture content and caking strength were estimated. In addition, morphology was detected by scanning electron microscope (SEM).

## 2. Materials and methods

### 2.1. Sample preparation

Samples consisted of refined salt, sea salt (South Korea), sea salt (France), bamboo salt, and rock salt, all purchased from a local market, and sodium chloride (NaCl) was used as a control. Sodium chloride was obtained from Duksan, Korea. Samples were pulverized with a commercial grinder for 15 min and sieved with a No. 25 mesh sieve (710  $\mu\text{m}$ ) to obtain a homogeneous particle size. Samples were stored in dry oven at 25 °C and 15% RH. Pulverizing and sieving processes were performed before each experiment.

### 2.2. Mineral content

Mineral content was estimated by inductively coupled plasma optical emission spectrometry (ICP-OES) and ion chromatography. Representative mineral of dietary salts, including sodium (Na), potassium (K), magnesium (Mg), calcium (Ca), chloride (Cl), bromide (Br), sulphate ( $\text{SO}_4$ ), and nitrate ( $\text{NO}_3$ ) were determined. Samples (0.2 g) were dissolved in 10 ml of deionized water and diluted. Solutions were diluted with deionized water before running ICP-OES and the dilution factor was varied from 1:1 to 1:500. The conditions of

ICP-OES were as follows: RF generator power, 1500 W; RF frequency, 40 MHz; Plasma gas flow, 12 L  $\text{min}^{-1}$ ; Auxiliary gas flow, 0.2 L  $\text{min}^{-1}$ ; Nebulizer gas flow, 0.55 L  $\text{min}^{-1}$ , respectively. The conditions of ion chromatography with Metrosep A sup 5–150/4.0 column were as follows: Eluent, 3.2 mM  $\text{Na}_2\text{CO}_3/1.0$  mM  $\text{NaHCO}_3$ ; Flow, 0.7 mL  $\text{min}^{-1}$ ; Volume, 20  $\mu\text{L}$ ; Pressure, 7.6 MPa; Temperature, 20 °C. Linearity was verified with standards of indicated mineral (0, 10, 50, 100, 500  $\mu\text{g L}^{-1}$ ) that were used to establish the calibration curve. In addition, there was no background interference and memory effects were overcome with known concentrations of samples (system blank). The experiment was performed in triplicate.

### 2.3. Moisture sorption isotherm

Moisture sorption isotherms of samples were obtained with a Dynamic Vapor Sorption (DVS) Advantage 1 (Surface Measurement Systems Ltd., London, UK) equipped with a Cahn microbalance. The experiments were performed at 25 °C using a moisture flow rate of 200 standard  $\text{cm}^3 \text{min}^{-1}$ . About 5 mg of samples were subjected to ramping of RH from 10% to 95% in 14 stages of increasing from 5 to 10% RH with water as a solvent. Table 1 shows detailed operation conditions. Equilibrium was considered to be reached if the rate change in mass was less than 0.002%  $\text{min}^{-1}$ . From this experiment, deliquescence relative humidity (DRH) could be detected.

Table 1  
Detail operation conditions of dynamic vapor sorption (DVS).

| Stage | Time (min) | Start % (P/P <sub>0</sub> ) | Stop % (P/P <sub>0</sub> ) | Temp (°C) |
|-------|------------|-----------------------------|----------------------------|-----------|
| 1     | 15         | 10                          | 10                         | 25        |
| 2     |            | 20                          | 20                         |           |
| 3     |            | 30                          | 30                         |           |
| 4     |            | 40                          | 40                         |           |
| 5     |            | 50                          | 50                         |           |
| 6     |            | 55                          | 55                         |           |
| 7     |            | 60                          | 60                         |           |
| 8     |            | 65                          | 65                         |           |
| 9     |            | 70                          | 70                         |           |
| 10    |            | 75                          | 75                         |           |
| 11    |            | 80                          | 80                         |           |
| 12    |            | 85                          | 85                         |           |
| 13    |            | 90                          | 90                         |           |
| 14    |            | 95                          | 95                         |           |

<sup>(1)</sup> P/P<sub>0</sub> means water vapor of sample divided by pure water vapor.

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