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Rheological measurements for characterizing sticky point temperature of selected fruit powders: An experimental investigation

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ABSTRACT

Stickiness is one of the common problems frequently encountered during production, handling and storage of fruit powders with high concentration of low molecular weight sugars. Several techniques and devices were developed in the past to determine the level of stickiness of some of those products. Nevertheless, there is still a need for a simple, more accurate and reliable method. In this study, a new method to quantify and characterize the sticky point temperature (T_s) of fruit powder was explored using a rheometer technique. The rheometer system utilized a serrated parallel plate to hold the samples and was operated in dynamic oscillation mode at a frequency of 1 Hz and a constant strain amplitude of 2%. The samples of a model fruit powder (Refractance Window (RW)-dried mango powder) were scanned from 25 to 95 °C at an increment of 10 °C and a holding time of 180 s for each increment. A *crossover* between the storage modulus (G') and loss modulus (G'') was established and denoted as sticky point temperature of the model fruit powder. Results showed that the sticky point temperature obtained using the new method agreed well with the published data and can be considered as a suitable technique to characterize the sticky point temperature of sugar-rich materials. The procedure for sample conditioning and rheometric measurements to determine the sticky point temperature are straightforward. This new technique can measure the sticky point temperature of fruit powders with a high degree of repeatability and accuracy (SD = 0.58–1.73 °C).

1. Introduction

Stickiness is a major quality index for food powders in traditional food processing operations such as drying, handling, storage, and is also a concern for equipment cleaning and sanitation. Recent academic and industrial interest in thermal pasteurization of low moisture foods to control Salmonella spp., *Cronobacter* spp., and other pathogens (Villa-Rojas et al., 2013) make the study of quality changes in powder products after heat treatments even more relevant. Likewise, stickiness is frequently encountered in food powders with high concentration of low molecular weight sugars, e.g. glucose, fructose and sucrose (Downton et al., 1982; Bhandari et al., 1997). Schubert (1987) reported that the mechanisms that may influence the tendency of particles to stick together include liquid bridging, solid bridging, inter-particle attraction forces and mechanical attraction. Liquid bridging happens when a sufficient amount of moisture is present between particles, while solid bridging is a result of solid diffusion or condensation within the solid matrix, normally at elevated temperatures (Barbosa-Canovas et al., 2005). The appearance of liquid bridges between powder particles allows for the transformation of the material from the glassy to rubbery phase and greatly influences its strength, causing it to be sticky (Ozkan et al., 2002).

Stickiness of materials in amorphous form is also due to water plasticization and its subsequent depression of glass transition temperature during processing and storage (Goula et al., 2007). Thus, stickiness of amorphous materials is influenced by its glass transition temperature. Roos (1993) reported that the sticky point temperature (T_s) of sugars is higher than the glass transition temperature (T_g) at $T_s - T_g$ between 10 and 20 °C. If the temperature of a glassy amorphous material exceeds T_g, then the material is transformed into a rubbery state and may become sticky Roos and Karel (1991a). This implies that stickiness can be prevented if the

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temperature of amorphous material is kept below T_g. Previous experiments on tomato, pineapple and mango powders (Jaya and Das, 2009), and milk powder (Hennigs et al., 2001) showed that T_s and T_g change with moisture content, but the difference between the two temperatures ($T_s - T_g$) was constant at a given water content.

The transformation of glassy amorphous material to rubbery state was associated with the storage modulus (G') and loss modulus (G'') (Rao, 1999). The G' value is a measure of deformation energy stored in the samples during the shear process (elastic behavior), while G'' is a measure of dissipation of energy (viscous behavior) (Mezger, 2006). It has been reported that when the material is in a glassy solid form, the value of G' is expected to be higher than G'', while the materials are transformed into rubbery state or become "liquid-like" when G'' exceeds G' (Rao, 1999). Using the mechanism of liquid-bridging related to elasticity and viscous behavior of the material at the interface, the observed values wherein G' and G'' are crossing at certain temperature can also be characterized as sticky point temperature of powder as a function of water activity and water content.

Several direct techniques to characterize the stickiness of food products have been reported. The oldest published method is the manually-driven propeller method initially developed by Lazar et al. (1956) to measure T_s of spray-dried tomato, orange, mango, and other powders. Brennan et al. (1971) modified the Lazar design by adding a motor-driven impeller, which Hennigs et al. (2001) further improved by connecting the impeller to a data logging system to record electrical resistance output. Ozkan et al. (2002) developed a rotational viscometry technique with an L-shape spindle inserted into the powder to measure T_s. The torque required to rotate the spindle at a given temperature is recorded using a data logging system connected to the viscometer. Kudra and Mujumdar (2002) also reported a semi-automatic sticky-point tester with humidity control. Adhikari et al. (2003) developed an *in situ* method for measuring sticky point temperature consisting of a drying chamber, an image acquisition system and a weighing balance connected to a data logging device. There are other methods reported in the literature, such as the Jenike shear cell method (Jenike, 1964), ampule method (Tsourouflis et al., 1976), fluidization test (Dixon et al., 1999), blow test (Paterson et al., 2001), cyclone sticky test (Boonyai et al., 2004), tack method (Green, 1941), and the contact probe test method (Kilcast and Roberts, 1998; Adhikari et al., 2001, 2003).

However, a critical analysis conducted by Boonyai et al. (2004) suggests that the above measuring devices and techniques are all empirical in nature. They also noted that due to the inaccuracy and difficulty of application of these devices to actual processing and handling operations, there are continuing demands and challenges to develop accurate, simple and easy to use methods to characterize the onset of stickiness of selected food powders. This research was carried out to develop and investigate a new method to quantify and characterize the sticky point temperature (T_s) of fruit powder using a rheometer technique.

2. Materials and methods

2.1. Preparation of samples

Refractance Window® (RW)-dried mango powder was chosen as the model sample for the entire experiment. The powder with water content of 0.039 kg water/kg dry solids was produced using the RW drying process following the procedure described by Caparino et al. (2012). One hundred grams of RW-dried mango powder with particle size ranging from 180 to 350 μm was prepared by slight grinding using a mortar and pestle, and sieving using mesh sizes of 80 and 45 (American Society for Testing and Materials, ASTM) (Barbosa-Canovas et al., 2005). The particle size range selected was based on the approximate size of the serration of the two parallel plate geometries used during the sticky point measurements. The prepared powder samples were put inside aluminum-coated polyethylene bags, flushed with nitrogen gas, heat sealed and kept at −35 °C for use in later measurements.

2.2. Conditioning of samples at different water content

Overall, samples with six different water contents (0.003, 0.022, 0.029, 0.039, 0.048, and 0.066 kg water/kg dry solids) were prepared and used in the experiment. The conditioning of samples was carried out by drying or by water absorption methods. The prepared RW-dried mango powders with water content of 0.039 kg water/kg dry solids served as the starting reference sample prior to conditioning. The samples with water content below 0.039 kg water/kg dry solids were obtained by drying the reference sample, while those above 0.039 kg water/kg dry solids were prepared by conditioning in thin-layers under 100% relative humidity for different times. The sample with water content of 0.003 kg water/kg dry solids was achieved by putting 5 g of the reference sample in a sealed jar containing P_2O_5 solution to avoid mold development and storing for 30 days at room temperature.

The samples with water content of 0.022 and 0.029 kg water/kg dry solids were obtained by placing 2 g of the reference sample in an aluminum pan with approximate thickness of 1 mm and oven-dried at 50 °C for 30 and 60 min, respectively. To obtain samples with higher water contents of 0.048 and 0.067 kg water/kg dry solids, the reference samples were spread in a 90 mm diameter Whatman #2 filter paper and placed on top of a perforated membrane located 20 mm above the water level inside a sealed container (Polylab® Plasticware, Hyderabad, India) and held for 20 and 40 min at room temperature, respectively. The relative humidity inside the container was assumed to be ~100%. To obtain uniform moisture adsorption during conditioning at ~100% RH, an aluminum sample holder with a dimension of 2 × 2 inch and thickness of 2 mm was fabricated. The size of the holder allowed spreading of samples evenly to 2 mm thickness onto the filter paper prior to loading into the desiccator to equilibrate. Apparently, the powder formed a lump of particles or agglomerated after conditioning at ~100% RH, and hence they were dispersed quickly using a stainless spatula, sealed in a small bottle and kept at room temperature for at least 2 h to equilibrate prior to measurement and analysis. Mango powder with water content greater than 0.067 kg water/kg dry solids were not included in the measurement because the material became very sticky at room temperature and were no longer practical to use for the study. We note here that some researchers were able to measure the sticky point temperature of a material up to 0.08 kg water/kg dry solids for higher molecular weight samples such as mango, orange and pineapple powders with added maltodextrin carrier (Jaya and Das, 2009).

2.3. Determination of water content

Water content of mango powder samples was determined using an automatic Karl Fischer titrator (Mettler Toledo, Columbus, OH) following the procedure in the instrument manual. Approximately 1 g of mango powder was put into the titration vessel through a funnel with the aid of a glossy paper. The exact sample weight was measured to 0.1 mg accuracy before delivering the same into the titration vessel. Following titration, the water content was determined automatically via a built-in software program. Measurements were performed in triplicate at room temperature.