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Measurement of sticky point temperature of coffee powder with a rheometer

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Abstract. Sticky point temperature (Ts) measurement for hygroscopic food and biomaterial powders is traditionally performed with complex glass instruments. This property is used to characterize material stickiness, which substantially affects the flow and physical behavior of powders. In this research study we developed a new methodology to measure sticky point temperature using a rheometer, and validated our Ts data with previously published coffee powder data. Our Ts measurement using a rheometer was performed in two replications. The behavior of Ts as a function of moisture content (%, db) was observed to be non-linear. After 16% (db) moisture content, however, there were no changes in Ts with increases in moisture content. An exponential prediction model for Ts as a function of moisture content was achieved with an R^2 value greater than 0.93; a power law regression model also fitted well, with an R^2 value of 0.97.

Keywords. Caking, coffee, rheometer, sticky point temperature
Introduction

A large amount and varieties of powder are formed industrially everyday and thus, there is a great need of information about their handling, storage, and processing characteristics. Cohesive attraction and frictional resistance developed between particles in a powder when consolidated due to processing and handling must be overcome to make the powder flow efficiently. For proper flowability and discharge of the material the cohesive attractive forces are necessary to determine the minimum hopper opening size of silos. A cohesive arch will prevent the smooth discharge of the materials, and thus overcoming the cohesive force and characterizing the cohesion phenomena involved is very important (Fitzpatrick et al., 2007). Many food powders and food component mixes makes this problem of caking and flowability more difficult due to the complex ingredients present in the food systems. Furthermore, during processing, handling, storage, and distribution of food powders to the final consumers there is a high possibility for the material to experience different environmental conditions, and therefore there is a lot of caking and flowability problems associated with it.

Stickiness is a phenomenon that reflects the propensity of some materials to agglomerate and to adhere to contact surfaces (Kudra, 2002). Stickiness and caking are commonly encountered problems in food powders and other related sugar rich amorphous powders (Wallack and King, 1988). The structural collapse of the product during air drying or freeze drying, or during the storage of dried products, is responsible for the reduction in the volume and porosity, which results in the loss of desirable appearance and volatile components. Powder stickiness and caking are the phenomena related to such structural collapse (Le Meste et al., 2002). It is usually known that this structural collapse, stickiness, and caking of the powders are controlled mainly by the moisture content and it is often time dependent (Le Meste et al., 2002). The mechanism of stickiness and agglomeration of food particles are mostly hypothesized to occur due to intermolecular and electrostatic forces, liquid bridges, solid bridges, and/ or mechanical interlocking of particles (Papadakis and Bahu, 1992). Food powders mostly contains amorphous carbohydrates like lactose from milk and other sources that can undergo physical changes such as crystallization, clumping, sticking, and caking during processing and handling of it (Levin and Slade, 1986). Stickiness property of the powders can also be used as an advantage in making agglomerated food products like instant coffee and milk powders and other related applications (Wallack and King, 1988). For instant coffee, milk, fruit juice powders a further step of agglomeration is often required to enlarge particle size generally from 50-80 µm to 250-300 µm, in order to obtain good ‘instant powder’ properties like wetability, dispersibility, and solubility (Gianfrancesco, 2006).

Apart from usual disadvantage of stickiness and agglomeration is storage and handling of powders, it is also negatively impact on the drying operations. For example, in spray drying operation the stickiness can lead to reduced sensory characteristics due to thermal degradation as well as lower product yields, operating problems, and fire and explosion hazards (Kudra, 2002). There can also other problems due to stickiness and deposition of powders particles on the wall of spray dryers like more frequent instrument shutdowns for cleaning wall deposition and thus, increased cost and processing time (Ozmen and Langrish, 2002). Moreover, in spray drying, stickiness can be a major problem, due to agglomeration of food powders and for heat sensitive products this can lead to overheating and leading to unpleasant sensory characteristics. This problem affects majorly the fruit juices which contains large amount of hygroscopic and amorphous sugars from getting successfully dehydrated (Downton et al., 1982). For fruit juice powders, its inherent hygroscopic and thermoplastic property causes more stickiness and caking in them (Chegini and Ghobadian, 2007). Reduction in the stickiness behavior for fruit juice was studied widely using drying aids or more specific handling
instruments. Maltini and Bertolo (1986) produced peach, pear, and apricot type of powder using vacuum belt industrial dryers. Malt dextrin with DE values of 6, 12, and 19 was used as drying aid for raspberry powder drying operation and to obtain free flowing powder by Bhandari et al., (1993). Gupta (1978) used liquid glucose as the drying aid to produce free flowing orange juice powder. Malto dextrins are widely used to in food components to increase viscosity, retard crystallization, to improve drying characteristics for food encapsulation, and to decrease stickiness and hygroscopicity of dried powders (Roos and Kharel, 1991). It is also being reported that malto dextrin are sometimes used to improve the storage and stability of the frozen foods (Levine and Slade, 1986). Introduction of cold air at the bottom of the drying chamber was studied by Lazar et al., 1956 and Bhandari et al., 1997. However, a limited amount of cold air can only be introduced since the cooling process may increase the humidity and thus, affects the caking of powders. Karatas and Esin (1990) used scraped surface for drying of tomato paste, in order to reduce caking of the product. In some cases may be large towers with low drying temperatures of roughly 30°C and 3% relative humidity was used for spray drying, so that caking on the surface of dryers can be reduced (Hayashi, 1989).

The most commonly used methods to quantify and characterize the stickiness and caking for food powder and other related substances are glass transition temperature and sticky point temperature. White and Cakebread (1966) stated that spray-dried food powders are amorphous solids because the solid particles in this operation are produced in high drying temperatures and very short period of time. Thus, the molecules do not have sufficient time to aligned them and get crystalline. The amorphous is generally referred to as glass or metastable super-cooled liquids, with lower viscosities. When these amorphous solids undergo slight heat treatment it under goes a second order phase transition with more rubbery nature. The caking and lumpiness of agglomerated powders are often related to this rubbery state. Glass transition temperature is generally measured by differential scanning calorimetry (DSC). Several studies with stickiness and glass transition temperature with various food, fruits, and other biomaterial powders have been done extensively and more details could be found in Bhadra et al., 2009; Chuy and Labuza, 1994; Farkas and Farkas, 1996; Fitzpatrick et al., 2007; Fujio and Lim, 1989; Ganesan et al., 2007; Jaya and Das, 2007; Lloyd et al., 1996; Morales and Kokini, 1997; and Roos and Kharel, 1991.

The other parameter, which is widely used to measure and quantify the stickiness and caking food powders, is sticky point temperature (Ts). Sticky point temperature measurement was initially developed by Lazar et al., 1956 and then it was applied by other researchers for measuring stickiness in various food powders (Wallack and king, 1988; Downton et al., 1982). As stated in Lazar et al. (1956), the sticky point temperature of the tomato juice powder was determined empirically by inserting the sample in a water bath temperature and then the temperature of the bath is slowly raised. This powder was stirred intermittently by rotating the propeller at ¼ turn. The sticky point temperature was defined as the bath temperature at which the force or torque necessary to stir the propeller increases sharply. It was found that sticky point temperature (Ts) for tomato juice powder showed an inverse function of moisture content. The most widely accepted definition of sticky point temperature is stated as the combination of temperature and moisture content for which the given mass of the powder is resists movement and is not longer free flowing. In moisture content –temperature co-ordinates, the set of sticky-point temperatures forms the so-called sticky point curve or stickiness curve, which provides a sharp boundary between the sticky region below the curve, and the non-sticky region above the curve (Kudra, 2002). Previous research study with coffee powder by Wallack and King (1988) also reveals such stickiness curve with change in moisture content. A representative diagram (figure 1) of sticky point curves and sticky regions of general food powders is given below.

The traditional method of quantifying sticky point temperature includes use of glass apparatus with the sample inserted in the water bath, where the temperature of the glass tube could be controlled. The sample glass holder has a propeller inserted which is manually controlled by hand to stir the sample, while the temperature is being raised. In order to prevent the moisture
content for the samples with increasing water bath temperature, mercury seals are provided in the glass tube. The temperature at which the force required to stir the sample increase dramatically is called the sticky point temperature, for that corresponding moisture content. More detailed description of this arrangement can be found in Wallack and King (1988) and Lazar et al. (1956). Since its existence in scientific world, sticky point temperature (Ts) measurement was studied thoroughly and lot of variations were applied to quantify it. It was found out that stirrer type of devices were better for sticky point temperature (Pasley et al., 1995). The similar type of manual stirring and glass apparatus was used by Downton et al. (1982) for Ts measurements. In order to avoid tedious method of manual stirring, Brennan et al. (1971) used a motor–driven stirrer. In a study by Hennigs et al. (2001) a DC motor driven stirrer was applied to sample at 38 rpm.

To eliminate the disadvantage of traditional method of measuring Ts, like excessive evaporation, tedious manual stirring operation, fragile glass apparatus, and using carcinogenic mercury compounds. The main objective of this study was to measure the Ts for coffee powder using a semi-automated rheometer. The Ts data measurement in this novel approach is much more sophisticated, fast, and uses a stirrer type device. The Ts data using a rheometer were validated for coffee powder through previously published research work by Wallack and King (1988). This paper also could predict a regression model for Ts as a function of moisture content for coffee powder.

Materials and Methods

Sample Collection

Commercial “Arabica” plantation coffee powder with moisture content 6.500% (db) was procured from the market. The moisture content analysis was carried out using standard AACC method (1995). Similar result of coffee moisture content was found by Ramalakshmi et al. (2007) for “Arabica” plantation variety. Later on, the coffee powder samples were prepared by drying it to 0% moisture content (db) and then adding specified amount of water (for higher moisture contents). The coffee samples were prepared at moisture contents of 4, 6, 8, 10, 12, 14, 16, 20, and 25 % (db), and were stored at room temperature (24 ±1°C). For each moisture content, Ts measurement was performed twice (i.e. n=2).

Traditional method of Ts measurement

The traditional method of sticky point temperature measurement used glass apparatus and a propeller with pointed flat wedge tip. The powder sample is designed to be placed glass tube where the propeller is being inserted and it being stirred mechanically. In order to avoid moisture evaporation mercury seal are being inserted into the glass tube holding the coffee sample. The bath temperature was raised about 1°C every three minutes at temperatures far below the sticky point temperature, and near the sticky point temperature approximately, the rise of temperature is about 1°C every 5 minutes. The heating are was slowly so that the powder temperature remains same as the bath temperature. The propeller was turned manually ¼ every other second. In order to ensure proper contact of the powder of the propeller, the sample tube was tapped periodically. As the bath temperature increased the force required to stirrer the sample, for a particular moisture content increase dramatically. That temperature as which the force required to stir the sample is known as the sticky point for that corresponding moisture content. The detailed and further discussion of the Ts measurement could be obtained from Wallack and King (1988) and Papadakis and Bahu (1992). The diagram for Ts measurement using traditional glass apparatus can be represented as given below in figure

Ts Measurement using a Rheometer
As stated in the objective of our current research study, for our coffee powder sample, we did not use the traditional experimental set up to measure Ts but instead used a Rheometer (Model: Viscoanalyzer, ATS Rheosystems, Bordentown, NJ) with cup and vane tool arrangement for this purpose. The vane tool is a 4 blade stirrer (4/13.5, Model: Viscoanalyzer, ATS Rheosystems, Bordentown, NJ) made of stainless steel with stress coefficient of $4.15 \times 10^4$ Pa/Nm, strain coefficient of 0.93 1/s/rad, and inertia of 2.05x$10^7$ kg mm. The rheometer was arranged in a “bob and cup” arrangement, and vane tool was inserted in the sample cup (CC25, Model: Viscoanalyzer, ATS Rheosystems, Bordentown, NJ) which was regulated with thermostat that increased the sample temperature from 20 to 80°C. The shear rate selected was to be 0.25 1/s and the temperature profile was selected to be every 2°C rise in sample temperature for 3 minutes, for the whole temperature span of 60°C. This selection of the profile time and shear rate was selected after repeated trials with coffee powder samples. A thermostat was attached with the sample cup as shown in the figure 4, which helped in raising the temperature of the coffee powder. The temperature at which the torque showed a dramatic increase in the value is indentified as the sticky point temperature. In order to prevent the moisture loss from the sample during increasing temperature, an aluminum foil coat was provided over the exposed sample cup. Figure 3 represents the pictorial view of the experimental set up used to analyze Ts with rheometer. Figure 4 shows how the stirrer was inserted in the sample cup attached to the rheometer. Figure 5 represents the propeller attachment used with 4 blade having dimensions as 36.79 mm (length) × 6.78 mm (width), the shaft of the propeller was about 105.63 mm in length. The cylindrical sample cup consists of an internal diameter of 26.68 mm, external diameter of 32.58 mm, and height of the cylinder is found to be 65 mm (figure 6). Both the sample cup and the propeller attachment were made of stainless steel. The sample cup shown in figure 6 was filled approximately 75% by the sample material and the vane tool was inserted into the cup.

**Validation of Ts data from Wallack and King (1988)**

For our proposed method to be authentic for coffee samples, we carried out a validation study with coffee powder for the above selected moisture contents as stated in Wallack and King (1988), where the traditional glass apparatus was used. The selected moisture content for our coffee sample is given as 4, 6, 8, 10, 12, 14, 16, 20, and 25% (db) and is plotted against the Ts (°C) to obtain the sticky point curve and to compare the sticky point curve obtained by Wallack and King (1988). Later on statistical regression modeling using Microsoft excel (2003) and using SAS software (SAS Institute, Cary, NC) were carried SAS was performed to obtain the best fit model to predict with higher R² and lower standard error mean for Ts as the function of moisture content, in coffee powder. The results of the validation and the comparison will be discussed later in the results and the discussion sections.

**Results and Discussion**

The sticky point temperature (Ts) curve for coffee powder samples used in this study is represented in the figure 7. In this is procedure we had two replications for each moisture contents, and from our results we can clearly observe that there were close observed data points for Ts curve for both the replications, indicating that our rheometer based Ts measurement was fairly accurate. Figure 8 represents the Ts from Wallack and King (1988) using coffee powder samples. In Wallack and King (1988), the range of moisture content for coffee powder samples was from 4% (db) to 15% (db), however for our coffee powder samples we have used the moisture content ranges from 4% (db) to 25% (db). It was purposely done to understand and evaluate the Ts curve for higher ranges of moisture contents. From figure 7, we could observe that for moisture content higher that 15% (db) there were not much variation in the Ts data, as the moisture content changes. The Ts curve as reported by Wallack and King
Almost similar results in the Ts curve was observed for our coffee samples using a rheometer, as indicated in figure 7. From figure 7, till 5% (db) moisture content there was a linear pattern in the Ts curve and then non linearity was observed for higher moisture contents. After 15% (db) moisture content, we could observe that the Ts curve showed almost flat pattern, suggesting that Ts did not vary much with change in moisture contents (figure 7). In the Ts curve (figure 8) reported by Wallack and King (1988) such flat region in Ts curve could not be seen due to lower ranges of moisture contents.

From figure 7 and 8, we could clearly note that as we increased the moisture content the Ts of the sample decreased, indicating that material will show more tendencies to stick at lower temperature regions and create flow problems. Thus, it was confirmed that the Ts is an inverse function of moisture content. A similar result was observed for spray dried tomato powders by Lazar et al. (1956). Stickiness is a major reason that limits the spray drying of various sugar rich foods. High hygroscopicity of amorphous powder increases the solubility of the sugars with temperature and thus, a lower melting point and glass transition temperature region is observed. This contributes to stickiness of the powder and it could also be evaluated by Ts curve (Bhandari et al., 1997). Presence of sugar molecules in the food powders may keep the product in the liquid syrup state, instead of a complete spray dried powder form which may contributes to stickiness of food powders (Bhandari et al., 1997). Apart from Ts measurement for stickiness in food powders, glass transition temperature (Tg) measurement are also used popularly to characterize stickiness and flowability in food powders as stated earlier. It was found out from previous literature reports that Ts and Tg measurements for stickiness are very closely correlated, and they vary very slightly in assessing stickiness for powder materials (Ozmen and Langrish, 2002). More details of this discussion could be found out in Ozmen and Langrish (2002); Bhadra et al., 2009; Sablani et al., 2007; Roos and Kharel (1990); Adhikari et al., 2001; Chegini and Gobadian (2007); and Werner et al., 2006.

Figure 9 represents the validation of rheometer based Ts curve for coffee powder. We could clearly see that our proposed method of Ts measurement with rheometer showed very close results as reported by Wallack and King (1988), using traditional procedure of Ts measurement for coffee powder samples. This indicates that our current methodology of finding Ts, showed accurate results with more precision. Since in rheometer based Ts measurement, sophisticated computer software generates the torque values, the Ts data had more precision than the traditional glass apparatus method, where there was no automation. Since there were no report of Ts data after 15% (db) in Wallack and King (1988), therefore it was not possible to validate the Ts data for moisture content higher than 15% (db).

After Ts measurement as a function of moisture content and validation of Ts data with Wallack and King (1988) report, we moved one step ahead to obtain a single regression model that will predict Ts for varying moisture content, for coffee powder samples. From table I, we could state that a power law type of regression equation worked best to predict Ts = f(moisture content) with high R² value of 0.97 and low standard error mean (SEM) value of 2.73. The corresponding plot of this regression model for predicted Ts data is illustrated in figure 10, along with the observed Ts values from Wallack and King (1988) and current study with coffee powder samples. However, from table I, we can observe that for polynomial type of equation, the R² is about 0.99 but still this model was not selected for our regression modeling purposes. Polynomial regression equation yielded extremely high negative predicted Ts value for moisture contents higher than 14% (db), during statistical prediction. Due to extremely high predicted Ts values for moisture content >14% (db), the SEM value (689.60) was also high and it was definitely unacceptable. This was probably due to the complicated nature of the polynomial function which provides sinusoidal type of regression curve and thus, make is difficult to predict. Moreover, the polynomial function is long and more complicated than exponential and power law type of regression models. Thus, we finally selected the power law type of equation for
predicted Ts = f(moisture content). This type of regression modeling with Ts = f(moisture content) was not reported in Wallack and King (1988) research paper. However, for glass transition temperature (Tg) (another parameter to measure stickiness), there is a popular Gordon-Taylor model (1952) for predicting Tg = f(moisture content). From table I, we could also note that the exponential model also yielded a $R^2$ value of 0.93 and SEM of 4.70. However, for best fit model we have selected the regression equation which produced higher $R^2$ and lowest SEM values.

**Conclusion**

This research paper could establish a new method of measuring sticky point temperature (Ts), using a rheometer. This method of measuring Ts has some obvious advantages over the traditional method. This procedure is more automated, fast, easy to handle, and precise than the traditional process. Our research could also validate the Ts data over varying moisture contents with previously published paper of Wallack and King (1988), where the traditional method was used. Such innovative research approach will help the food and powder industries to use sticky point temperature (Ts) parameter more often and effectively in future. More validation studies with other food samples should be done to test the effectiveness of this procedure.

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**References**


<table>
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<th>Model</th>
<th>Type of model</th>
<th>$R^2$</th>
<th>SEM</th>
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<th>b</th>
<th>c</th>
<th>d</th>
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<td>$y=ax^{(b)}$</td>
<td>Power law</td>
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<td>$y=ae^{(bx)}$</td>
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<td>107.54</td>
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<tr>
<td>$y=ax^4+bx^3+cx^2+dx+e$</td>
<td>Polynomial</td>
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<td>0.90</td>
<td>-0.92</td>
<td>26.47</td>
<td>77.88</td>
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† Where $y$ is predicted $Ts$; $x$ is moisture content (% db); SEM is standard error of the mean; $a$, $b$, $c$, $d$, and $e$ are the estimated model parameters; $\alpha=0.05$.

Figure 1: Typical sticky region and sticky point temperature ($Ts$) curve description, based on Kudra (2002).
Figure 2: Glass apparatus and experimental set up for Ts measurement, based on Papadakis and Bahu (1992).

Figure 3: Experimental set up to measure the sticky point temperature (Ts) using a rheometer.
Figure 4: View of the stirrer being inserted into the sample cup for measuring sticky point temperature (Ts).

Figure 5: View of the stirrer with 4-blade vane tool used for the Ts measurements.

Figure 6: View of the sample cup used for the Ts measurements.
Figure 7: Sticky point temperature data for coffee powder using the rheometer, as determined in this study.

Figure 8: Sticky point temperature curve for coffee powder, as reported in Wallack and King (1988)
Figure 9: Validation of Wallack and King (1988) coffee powder Ts data using the rheometer.

Figure 10: Modeling of predicted Ts = f (moisture content) using a power law regression equation (model 1, table I) for coffee powder. $R^2 = 0.9703$ and SEM= 2.73