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ABSTRACT

Sticky point temperature (Ts) is a parameter that quantifies stickiness of food and biological powders. It is traditionally measured using glass instruments. In this study we developed a new methodology to measure sticky point temperature using a rheometer, and we successfully used it to determined Ts for coffee powder samples. The behavior of coffee Ts as a function of moisture content (%, db (i.e., dry basis)) was observed to be non-linear, but after 16% (db) moisture content, there were no changes in Ts with further increases in moisture content. An exponential prediction model for Ts = f(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. Rheometry was shown to be a viable and convenient means to determine Ts for various coffee powders.

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

INTRODUCTION

A large variety of powders are generated industrially and thus there is a great need for information about their handling, storage, and processing characteristics. Cohesive attraction and frictional resistance developed between particles in a powder when consolidated (due to handling and storage) must be overcome to make the powder flow efficiently, otherwise cohesive arches will prevent the smooth discharge of the materials. For proper flowability and discharge of powder materials, knowledge of cohesive attractive forces is necessary to design the minimum

size of hopper openings. Thus, overcoming cohesive forces and characterizing cohesion phenomena is very important (1). Food powders and food component mixes make the problem of caking even more difficult due to the complex ingredients present in these systems. Furthermore, during processing, handling, storage, and distribution of food powders to final consumers, there is a possibility for the material to experience variable environmental conditions (i.e., temperatures), thus there is a high probability of caking and flowability problems.

Stickiness is a phenomenon that reflects the propensity of powders to agglomerate and to adhere to contact surfaces (2). Stickiness and caking are commonly encountered problems in food powders and other sugar-rich powders which are amorphous in nature (3).. Powder stickiness and caking are phenomena related to this structural collapse (4). Often structural collapse, stickiness, and caking of powders are strongly influenced by moisture content, and these behaviors are often time dependent (4).

The mechanisms of particle stickiness and agglomeration are mostly due to intermolecular and electrostatic forces, liquid bridges, solid bridges, and/or mechanical interlocking of particles (5). Food and biological powders often contain amorphous carbohydrates that undergo physical changes such as crystallization, clumping, sticking, and caking during processing and handling (6). Although these changes are not desirable during storage, stickiness of powders can sometimes be an advantage when making agglomerated food products like instant coffee, milk powders and other related applications (3). For example, for instant coffee, milk, and fruit juice powders, 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 such as wetability, dispersibility, and solubility (7).

In addition to the usual disadvantage of stickiness and agglomeration in storage and handling of powders, they can also negatively impact drying operations (8, 9, 10). Malto dextrins are widely used in food components to increase viscosity, to reduce crystallization, to improve drying characteristics, to decrease hygroscopicity, and to decrease stickiness of dried food powders (11). The most commonly used method to quantify and characterize stickiness and caking potential of food and biological powders is the glass transition temperature (Tg) (Bhadra et al. (12); Chuy and Labuza (13); Farkas and Farkas (14); Fujio and Lim (15); Ganesan et al. (16); Jaya and Das (17); Roos and Kharel (11))

Apart from Tg, sticky point temperature (Ts) is also used to measure and quantify the stickiness and caking of powders.. Sticky point temperature measurement was initially developed by Lazar et al. (18), and then it was applied by other researchers for measuring stickiness in various food powders (3, 9). As stated in Lazar et al. (18), the sticky point temperature of tomato juice powder was determined empirically by inserting the sample vessel into a water bath, and then the temperature of the bath was slowly raised. The powder was stirred intermittently by rotating a propeller ¼ turn at a time. The sticky point temperature was defined as the bath temperature at which the force or torque necessary to stir the propeller increased sharply (i.e., a critical point). It was found that sticky point temperature for tomato powder showed an inverse relationship with moisture content. In order to prevent moisture loss from the samples during testing, mercury seals were provided in the glass tube. More detailed descriptions of this arrangement can be found in Wallack and King (3) and Lazar et al. (18).

The most widely accepted definition of sticky point temperature is the combination of temperature and moisture content for which the given mass of the powder resists propeller movement and is no longer free-flowing. A representative diagram of sticky point curves and

sticky regions of general food powders is given in Figure 1. In moisture content vs. temperature coordinates, the sticky point (or stickiness) curve provides a sharp boundary between the sticky region below the curve, and the non-sticky region above the curve (2). Figure 1 shows that the granular non-sticky particles (below the lower boundary Ts curve) can be converted to sticky mass with increase in moisture content and increase in temperature. Further increase in moisture content or temperature would ideally convert the sticky material to liquid state, crossing the upper boundary Ts curve. Previous research studies with coffee powder by Wallack and King (3) also revealed a stickiness curve with a change in moisture content. For Wallack and King (3) data, the lower boundary of the Ts curve (the non-sticky discrete particles), and the sticky region were considered as the sticky point curve.

Since its inception, sticky point temperature has been studied for various powder products, and it has been determined that stirring devices work best (9). In order to avoid tedious manual stirring, Brennan et al. (19) used a motor–driven stirrer. In a study by Hennigs et al. (20), a DC-motor driven stirrer was applied to a sample at 38 rpm. To eliminate the several disadvantages of the traditional Ts method, such as excessive evaporation, tedious manual stirring, fragile glass apparatus, as well as using dangerous mercury compounds, the traditional method of Ts measurement should be modified with a more sophisticated approach. Thus, the objectives of this study were: (i) To develop a new method to measure sticky point temperature (Ts) using a semi-automated rheometer. (ii) To validate vis-à-vis the coffee powder sticky point temperature (Ts) data obtained by using the new rheometer method with the previously published research of Wallack and King (3). (iii) To developed a regression model for predicting sticky point temperature (Ts) as a function of moisture content.

MATERIALS AND METHODS

Sample Collection and Preparation

Commercial Arabica plantation coffee powder, with an initial moisture content of 6.5 % (db), was procured from market (Walmart Stores, SD). Moisture content analysis was carried out using an AACC standard method (21). Similar results for coffee powder moisture content were found by Ramalakshmi et al. (22) for Arabica variety. The coffee variety that was used by Wallack and King (3) is not reported clearly. Hence, we could not match the coffee variety of the test samples with Wallack and King (3).The coffee powder samples were then prepared for experimentation by drying to about 0% moisture content (db) (this was achieved by drying the coffee powder for 8 hours at 50˚C), and then adding amounts of water to achieve specific moisture contents of 4, 6, 8, 10, 12, 14, 16, 20, and 25 % (db). After moisture adjustment, the samples were stored at room temperature (24 ±1˚C). For each moisture content, Ts measurement was performed twice (i.e. n = 2).

Ts Measurement

The traditional method of sticky point temperature measurement used a glass apparatus and a propeller with pointed, flat wedge tips. Ts measurement using the traditional glass apparatus is shown in studies carried out by Papadakis and Bahu (5) and Wallack and King (3). The powder sample was placed in a glass tube where the propeller was inserted and then mechanically stirred. The glass tube was then placed in a water bath. In order to avoid moisture evaporation, a mercury seal was used. The water bath temperature was then raised about 1˚C every 3 min (at

temperatures far below the sticky point temperature but for temperature near the actual sticky point temperature the water bath temperature rise was about 1˚C every 5 min). Heating was done slowly so that the powder temperature remained in equilibrium with the bath temperature. The propeller was generally turned manually ¼ revolution every other second. In order to ensure proper contact between the powder and the propeller, the sample tube was tapped periodically. At some points, as the bath temperature increased the force required to stir the sample drastically increased (i.e., a critical point was reached). The particular temperature (for a given moisture content) at which the force required to stir the sample increased is known as sticky point temperature (Ts). Detailed further discussions of Ts measurement can be obtained from Wallack and King (3) and Papadakis and Bahu (5).

Although never done before, to measure Ts in this study, we used a rheometer (Viscoanalyzer, ATS Rheosystems, Bordentown, NJ) with a cup and vane tool arrangement. The vane tool was a 4 blade stirrer (model 4/13.5, Viscoanalyzer, ATS Rheosystems, Bordentown, NJ) made of stainless steel with a stress coefficient of 4.15 x 10^4 Pa/N·m, strain coefficient of 0.93 1/s/rad, and inertia of 2.05x 10^7 kg·mm. The vane tool was inserted into the heated sample cup (model CC25, Viscoanalyzer, ATS Rheosystems, Bordentown, NJ). Figure 2 provides a pictorial view of the experimental set up used in this study. Figure 3 illustrates the 4 blade vane attachment, with dimensions of 36.79 mm (length) × 6.78 mm (width) (the shaft of the propeller was 105.63 mm in length) as it is being inserted into the cylindrical sample cup (which had an internal diameter of 26.68 mm, external diameter of 32.58 mm, and height of the 65 mm). Both the sample cup and the vane attachment were made of stainless steel. The sample cup was filled approximately 50% full with the sample material, and then the vane tool was inserted into the cup.

The sample temperature was regulated with a thermostat that increased the cup temperature from 20 to 80°C. The shear rate used was 0.25 1/s, and the increasing temperature rate was 2°C rise in every 3 min (or 0.67 °C/min), with an entire temperature span of 60˚C. This combination of temperature, time, and shear rate was selected after preliminary trials with the coffee powder samples (data are not shown). As the traditional method, the temperature at which the torque showed a dramatic increase in value was identified as the sticky point temperature. Torque is a measurement for twisting forces or tendency for force to rotate an object on axis.

Data Analysis

Statistical regression modeling and analysis using SAS software (SAS Institute, Carry, NC) were performed to obtain the best fit model to predict Ts as a function of moisture content. Graphs were produced using MS Excel (v. 2003) software.

Validation of Ts Data

To examine the validity of rheometry as an appropriate means to quantify sticky point temperature, we compared our results with that of Wallack and King (3), where the traditional glass apparatus was used to measure coffee powder sticky point temperature.

RESULTS AND DISCUSSION

The sticky point temperature (Ts) curve for coffee powder samples used in this study is presented in the Figure 4. We had two replications for each moisture contents and can clearly observe that the Ts readings were very close for both the replications. This indicates that our

rheometer-based Ts measurement procedure was fairly precise. Figure 4 presents the Ts data from Wallack and King (3) superimposed with our data. For Wallack and King, the range of moisture contents for their coffee powder samples was from 4% (db) to 14% (db). However, for our coffee powder samples, moisture contents ranged from 4% (db) to 25% (db). This was purposely done to understand and evaluate the Ts curve for a higher range of moisture contents. The Ts curve as reported by Wallack and King (3) (Figure 4), showed a fairly linear pattern until ~ 7% (db) moisture content, and then there was a non-linear decrease in the Ts values as the moisture content increased up to 14 % (db). Almost similar results were observed for our coffee powder samples, as indicated in Figure 4. From Figure 4 we also observe that for moisture contents higher than ~15% (db) there was little change in the resulting Ts, and as the moisture content increased the curve remained almost unchanged with less decrease in Ts values..

From Figure 4 , we clearly note that as the moisture content of the coffee powder increased, the Ts decreased, which indicates that the coffee powder has greater tendency to stick (at the lower temperature regions) and create flow problems. This typical Ts curve confirms that for low moisture contents (<15% (db)) if only the temperature is raised high above 40°C, then the particle–particle cohesion takes place which triggers stickiness in coffee powder. On the other hand, for moisture contents (>15 % (db)) particle cohesion was observed for temperature between 30 to 40°C. Ts decreased significantly for lower moisture levels (<15% , db) because increase in water would facilitate more in liquid mobile bridge formation which is the main cause of particle cohesion, and stickiness at lower temperatures. Thus, it was confirmed that Ts is an inverse function of moisture content. A similar result was observed by Lazar et al. (18) for spray dried tomato powders. Stickiness is a major constraint that limits the spray drying of various sugar-rich foods. High hygroscopicity of amorphous powders increases the solubility of the sugars with temperature, and therefore a lower melting point and glass transition temperature

region is observed, contributing to stickiness in powders. The presence of sugar molecules in food powders may keep the product in the liquid/syrup state, instead of a complete dried powder form, which may further contribute to powder stickiness (18).

It has been shown in previous reports that Ts and Tg (glass transition temperature) are very closely correlated, and both can be used to assess stickiness of powder materials (8). More details can be found in Ozmen and Langrish (8), Roos and Kharel (23), Adhikari et al. (24), and Werner et al. (25). Figure 4 presents the validation of rheometer-based Ts curve for coffee powder vis-à-vis Wallack and King's data (3). We can clearly see that the data produced by our method showed very close results to those of Wallack and King (3). The rheometer-based Ts measurement used sophisticated computer software and generated the torque values automatically, so the rheometer method resulted in higher precision than the traditional glass apparatus method, where there was no automation involved in the procedure.

After examining coffee powder Ts as a function of moisture content, and validating our Ts data with that of Wallack and King (3), we moved one step further, and obtained a single regression model that could predict Ts for all moisture contents. As shown in Table I, all models worked well, but a power law of regression equation was best to predict Ts = f(moisture content), with an R^2 value of 0.97 and very low standard error of the mean (SEM) value of 2.73. The corresponding plot of this regression model for predicted Ts vs. observed Ts is illustrated in Figure 5. From Table I, we observe that for the polynomial regression equation, the R^2 was high (0.99), but this model was not selected as optimal due to the extremely high SEM value. Although not examined in this study, for glass transition temperature (Tg), the Gordon-Taylor model (26) can be used to predict Tg = f(moisture content).

CONCLUSIONS

This study establishes a new method for measuring sticky point temperature (Ts) using a rheometer. This method has several obvious advantages over the traditional method. This procedure is more automated, fast, easy to handle, more precise than the traditional process, and does not rely on a glass apparatus or mercury seals. Our research also validates Ts data over varying moisture contents with previously published research on coffee powder. This innovative approach should help the food and powder industries to measure Ts more efficiently.. More validation studies with other food samples should be done to test the effectiveness of this procedure.

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Table 1: Regression modeling results for predicted Ts = f(moisture content).†

			Model Performance		Parameters Estimates				
Model		**Type of model**	**R^2**	**SEM**	**A**	**b**	**c**	**d**	**e**
1	$y=ax^{(b)}$	Power law	0.97	2.73	248.12	-0.77			
2	$y=ae^{(bx)}$	Exponential	0.94	4.70	107.54	-0.09			
3	$y=ax^4+bx^3+cx^2+dx+e$	Polynomial	0.99	689.60	-0.03	0.90	-0.92	26.47	77.88

† 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; α=0.05.

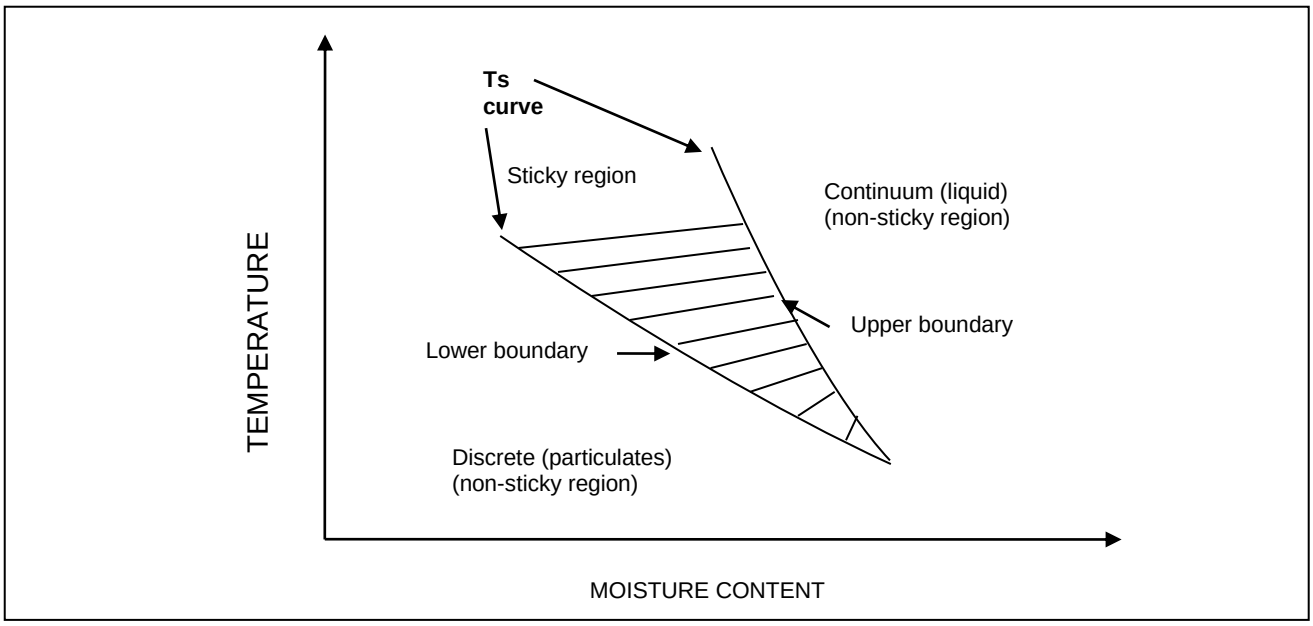
Ts
curve
Sticky region
Continuum (liquid)
(non-sticky region)
Upper boundary
Lower boundary
Discrete (particulates)
(non-sticky region)
TEMPERATURE
MOISTURE CONTENT

Figure 1

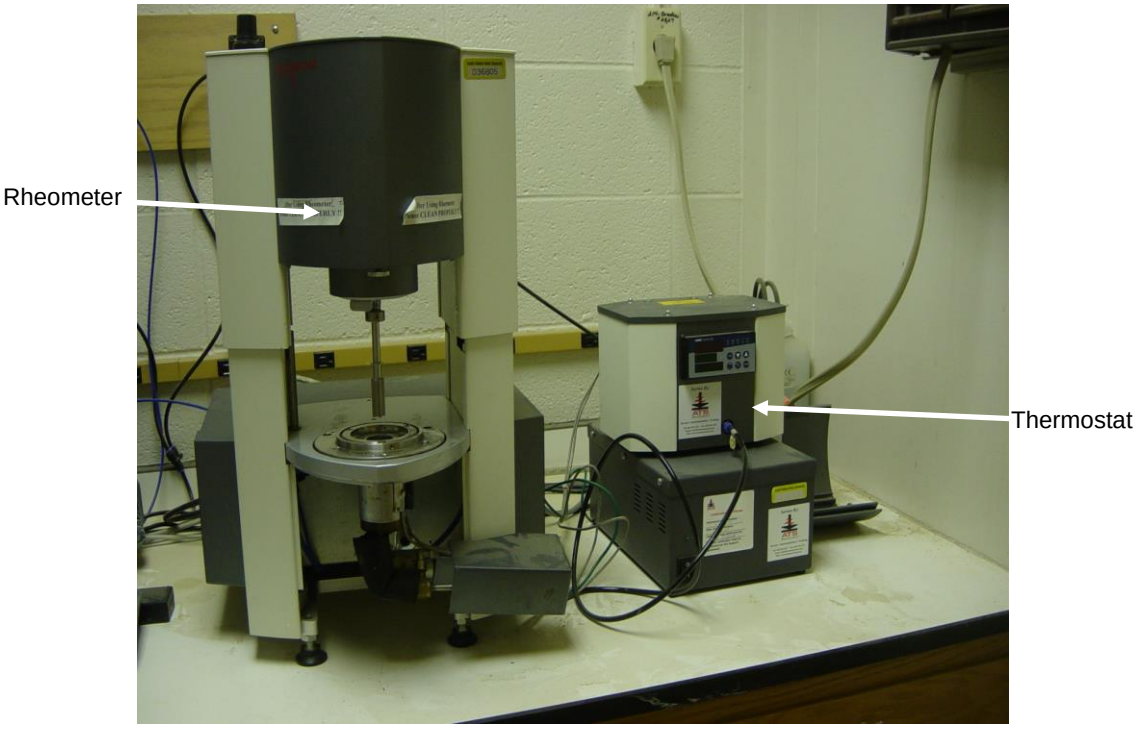

Figure 2

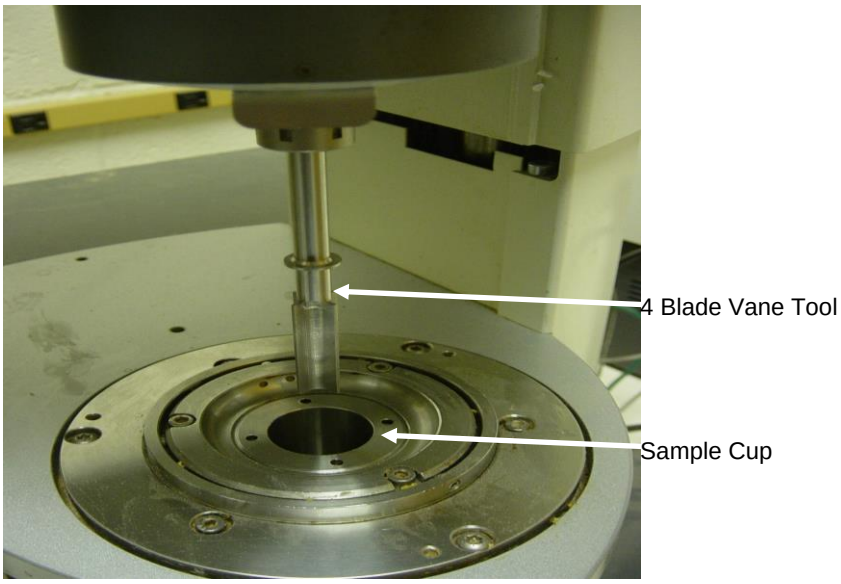

Figure 3

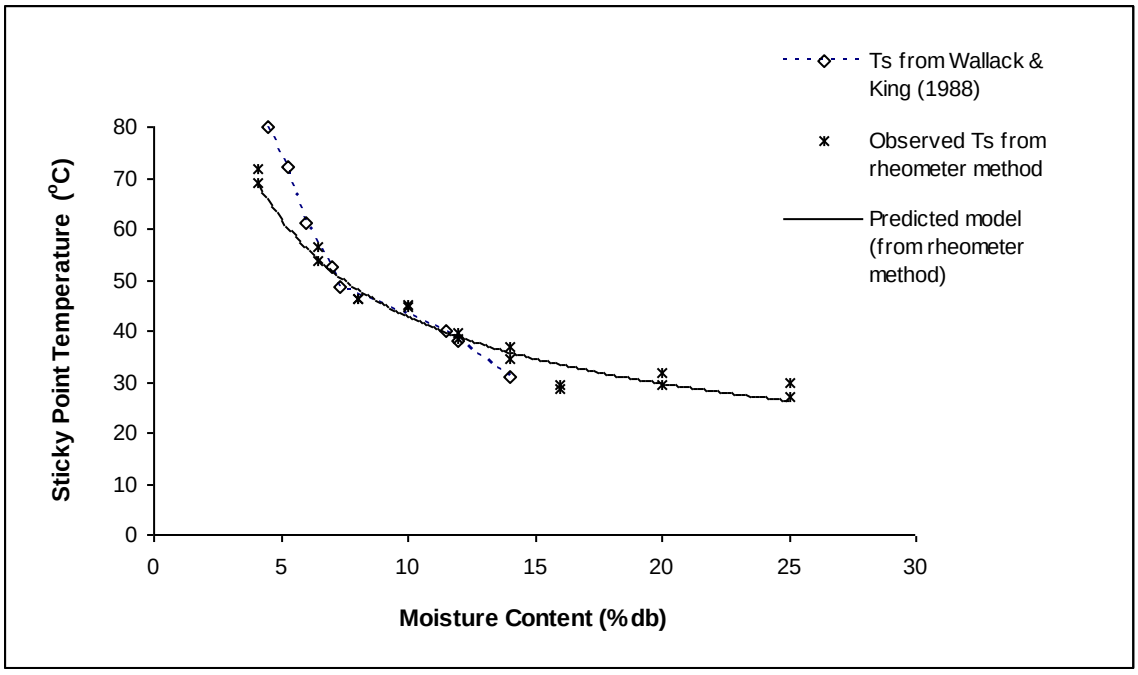
Ts from Wallack & King (1988)
Observed Ts from rheometer method
Predicted model (from rheometer method)
Sticky Point Temperature (°C)
Moisture Content (%db)
0
10
20
30
40
50
60
70
80
0
5
10
15
20
25
30

Figure 4

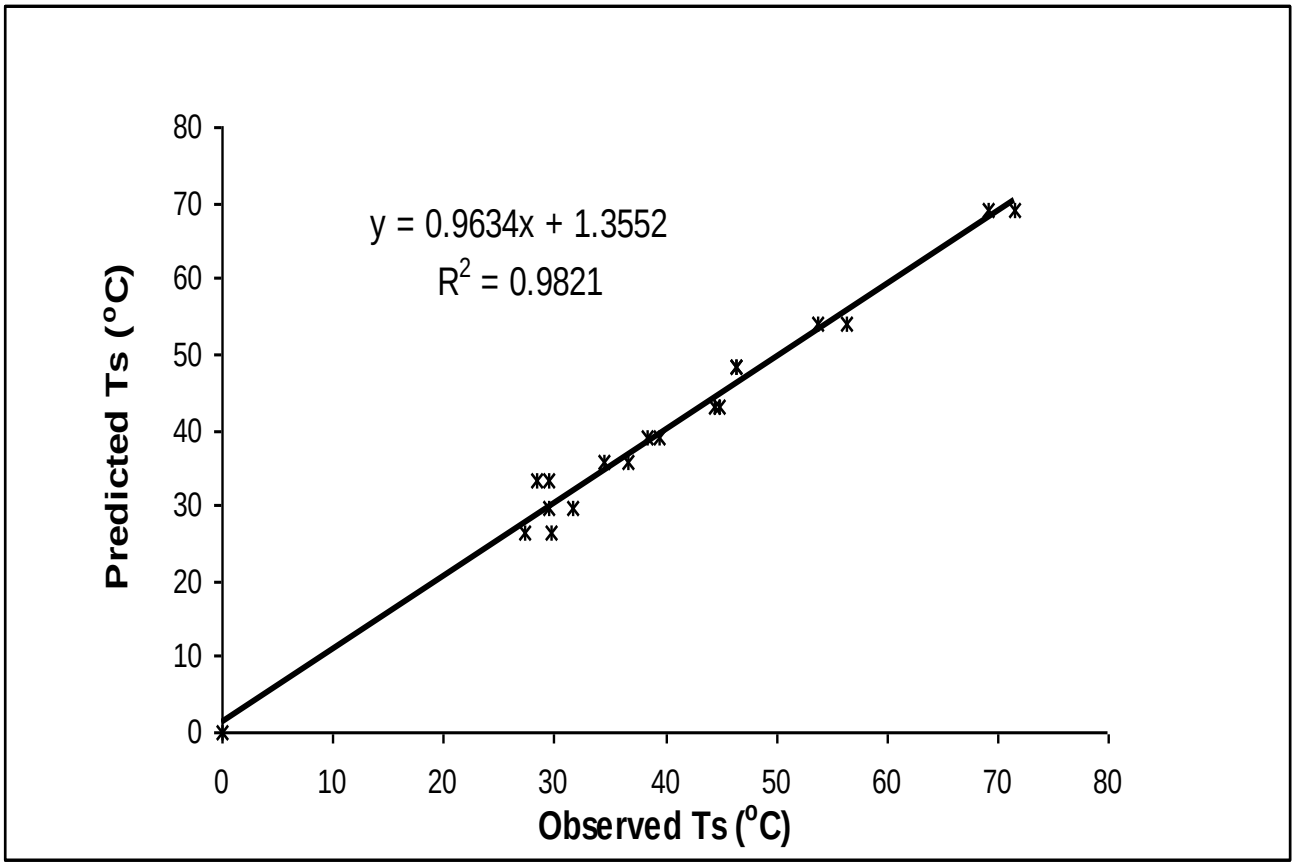
y = 0.9634x + 1.3552
R2 = 0.9821
Predicted Ts (°C)
Observed Ts (°C)
0
10
20
30
40
50
60
70
80

Figure 5

Figure Captions

Figure 1: Typical sticky region and sticky point temperature (Ts) curves for food products, based on Kudra (2). For this study, non-sticky (discrete particles), lower boundary (Ts curve), and sticky region is considered for measurement purposes.

Figure 2: Experimental set up used in this study to measure the sticky point temperature (Ts) with a rheometer.

Figure 3: View of the 4 blade vane tool being inserted into the sample cup for Ts measurement.

Figure 4: Comparison of Wallack and King (3) coffee powder data with Ts data obtained using the rheometer in this study. Predicted Ts= f (moisture content) using a power law regression equation (model 1, Table I), $R^2 = 0.97$, SEM= 2.73.

Figure 5: Relationship between observed Ts and predicted Ts for coffee powder.