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Characterization of Tomato Pulp Stickiness during Spray Drying using a Contact Probe Method

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A contact probe test was developed to characterize the surface stickiness of a tomato pulp droplet at various moisture contents and temperatures. To provide tomato pulp samples with different moisture contents, tomato powder produced by a laboratory spray dryer was wetted to seven different moisture levels. The instantaneous tensile force curve was recorded during the probe withdrawal from which the maximum tensile force and other useful information were obtained and cross-examined against images of bonding, debonding, and failure of the material. Generally, at higher moisture contents tomato pulp exhibited cohesive failure followed by semi-adhesive failure, but when moisture content decreased to a certain level, a peak tensile pressure was observed and the failure was adhesive. In addition, higher temperatures shifted the points of adhesive failure toward lower moisture content.

Keywords Adhesion; Cohesion; Glass transition; Spray dryer; Viscosity

INTRODUCTION

Spray drying is a common method used to dehydrate foods and vegetables. A feed liquid or paste is atomized into droplets and contacted with hot air. The drops dry as they fall through the dryer. In a spray-drying process, if the temperature of droplets surface is higher than the surface glass transition temperature (Tg), then the surface is sticky and this leads to the formation of agglomerates or to caking on dryer walls.[1]

Tomato pulp is a typical example of a product that is very difficult to spray dry due to the low glass transition temperature of the low-molecular-weight sugars present. The sugars found in tomato products are mainly dextrose and levulose, with a Tg of 31 and 5°C, respectively.[2] Several measures have been experimented with to cope with the spray drying of tomato pulp.[3,4] In a previous work, an experimental spray dryer was modified for drying tomato concentrate. The modification made to the original dryer design consisted of connecting the dryer inlet air intake to an adsorption air dryer. The modified system was proved advantageous over the standard spray dryer. Preliminary air dehumidification improved not only product recovery but also product properties. The low air humidity leads to an increase in drying rate, which results in a decrease in air temperature and particle surface temperature, and thus in rapid formation of a solid particle surface (skin) with Tg higher than that of a liquid or a semi-liquid surface. In addition, the rapid particulate skin formation decreases the interparticle adhesion, which decreases the powder moisture. These two results contribute to an increase in powder bulk density and solubility.[5,6]

Generally, stickiness is a phenomenon that reflects the tendency of some materials to agglomerate and/or to adhere to contact solid surfaces and can be described in terms of cohesion (particle–particle stickiness) and adhesion (particle–solid wall surface stickiness). Cohesion is an internal property and is a measure of the forces holding the particles together, whereas adhesion is an interfacial property and is a measure of the forces holding the particles to the surface of another material.[7] Stickiness mechanisms have been divided into five major groups, namely intermolecular and electrostatic forces, mobile liquid bridges, immobile liquid bridges, solid bridges, and mechanical interlocking.[8] It has been shown that the major causes of stickiness in amorphous materials are water plasticization of particle surfaces and temperature, which allow a sufficient decrease of surface viscosity for adhesion and cohesion and hence result in interparticle binding and formation of clusters.

For given moisture content, a distinct transition from the non-sticky to the sticky state has been observed when the temperature increases. This transition is called the sticky-point temperature.[9] By varying the moisture content, different sticky point temperatures can be found,

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which form the so-called sticky-point curve when plotted against the moisture content. Combinations of temperature and moisture contents lying above this curve result in incipient material stickiness, while the region below the curve represents non-sticky behavior. This sudden change in the material property may be important information for the operation of a spray dryer, since it is likely that depositions can be reduced when keeping the particles near the chamber walls below this curve at a lower temperature or moisture content.

Various techniques have been developed for the characterization of the stickiness behavior of food materials. These methods are generally based on the material properties, such as viscosity, resistance to bulk shear motion, and glass transition temperature. The first two concepts provide a direct interpretation of the stickiness behavior, whereas the result obtained by the latter measurement concept can be indirectly correlated to stickiness. Therefore, the stickiness characterization techniques may be divided into direct and indirect techniques, and further classification can be made according to the testing mechanism as conventional, pneumatic, and in situ techniques.[10] Direct methods involve measuring shear force, viscosity, optical properties, cohesion, and adhesion of the sample as it changes from a free-flowing to a sticky state as a function of moisture and/or temperature. Conventionally, the stickiness behavior can be measured by means of mechanical stirring, ampule, optical probe, and shear cell, whereas the pneumatic tests generate particle dynamic in an air stream within the test cell.[11–17] The in situ technique, on the other hand, determines development of adhesiveness and cohesiveness of a wet particle surface during moisture removal.[18] Finally, the glass transition temperature technique and the thermal compression are characterized as indirect methods.

The contact probe test is one of the most common methods used for characterization of stickiness. The essential feature of this test is to bring a probe in contact with an adhesive with light contact pressure for a short time and pull away at a fixed speed. The peak tensile pressure is a measure of tack or stickiness. The tack method has been employed to characterize the stickiness of dough, confectionery products, sugars, honey, maltodextrin solutions, and pressure-sensitive adhesives.[12,13,18–20]

The only method used for characterization of tomato products stickiness is the propeller-driven technique, which was first used in 1956 by Lazar et al.[21] to measure the sticky-point temperature of spray-dried tomato powder. The instrument consists of a test tube containing the sample submerged in a heating medium. An impeller embedded in the sample is turned and the sample's temperature is increased at a specified rate. With increasing temperature, the particle surface becomes more viscous compared to that in its free-flowing state, and this produces an increase in force required to stir the sample. However, this method is influenced by the cohesion property of the material, as it is based on the fact that for a given combination of material temperature and moisture content, the mass of the material resists movement and is no longer free flowing. With such a definition, stickiness reflects mostly cohesive forces in the bulk of solids. However, in principle, both cohesive and adhesive properties of the material are important during processing and drying.

Hence, the objective of this work is to characterize the stickiness of tomato pulp during spray drying using a specially designed device based on probe tack test to provide information about both the cohesion and adhesion phenomena.

MATERIALS AND METHODS

Sample Preparation

To provide tomato pulp samples with different moisture contents, tomato powder produced by a laboratory spray dryer was wetted to seven different moisture levels, 12.0, 16.0, 20.0, 34.0, 41.2, 64.0, and 86.0% (wet basis, w.b.). Tomato powder samples of about 2 g ($\pm$0.01 g) were conditioned at 25°C using sulphuric acid solutions to maintain the water activity level between 0.15 and 0.95, according to the sorption isotherm methodology. After equilibrium was reached, samples of about 0.1 g were taken for characterization of material stickiness.

A Buchi mini spray dryer (Model 191, Buchi Laboratoriums-Technik, Flawil, Switzerland) was employed for the spray-drying process. A peristaltic pump pumped tomato pulp to the atomizer and atomization was performed using a two-fluid nozzle (inside diameter 0.5 mm), which used compressed air. The modification made on the original design consisted of connection of the spray dryer inlet air intake nipple with an air drying unit by a flexible plastic air duct. The compressed air was also dehumidified before its supply to the two-fluid nozzle. An Ultrapac 2000 adsorption dryer (Model 0005, Ultrafilter International AG, Haan, Germany) with two desiccant cartridges was used to dry air down to 0.01 g of water per kg of dry air. The atomizer pressure, the feed temperature, and the feed rate were 5 ± 0.1 bar, 32.0 ± 0.5°C, and 1.75 ± 0.05 g/min, respectively, whereas the feed was medium concentrated tomato pulp with a constant total solids mass concentration of 14 ± 0.05%, containing 1.40 ± 0.02% insoluble solids, 5.61 ± 0.07% sugars, 1.53 ± 0.03% acid, 2.20 ± 0.02% protein, and 1.10 ± 0.01% salt. Tomato pulp was spray dried at air inlet temperature of 130°C ($\pm$1°C), drying airflow rate of 22.75 m^3/h ($\pm$0.18 m^3/h), and atomizing agent flow rate level of 600 L/h ($\pm$20 L/h).

Stickiness Testing Device

The stickiness testing device is shown in Fig. 1. It involves a pneumatic linear actuator-probe system connected to

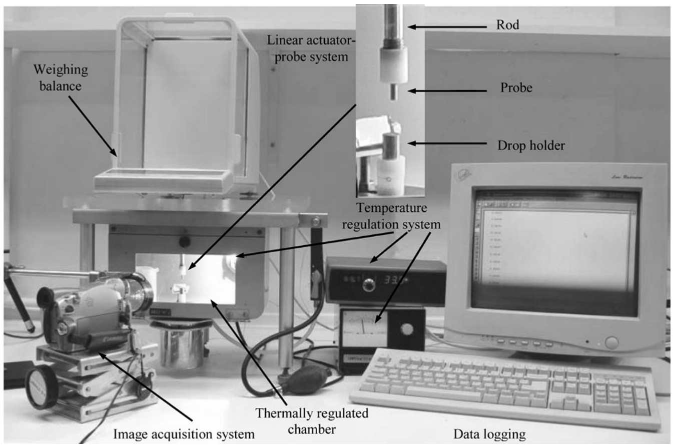

FIG. 1. Stickiness testing device.

an Ohaus weighing balance (Model AP210, Ohaus Corporation, Pine Brook, New Jersey) with data logging provision and a Canon image acquisition system (Model MV530i, Canon, Inc., New York, New York). The linear actuator-probe system is inside a thermally regulated chamber. The probe is a 3 mm (diameter) $\times$7 mm (height) stainless steel rod welded with a 3 mm (diameter) $\times$10 cm (height) stainless steel cylindrical rod. The rod provides sufficient contact pressure to exceed 100 g/cm^2 on the probe surface and when the probe completely rests on the surface of the sample, a contact pressure of $\geq$10 kPa is realized. A wire hook is used to connect the rod with the precision ($\pm$0.1 mg) balance. The readings from the balance are continuously logged to a desktop computer through an RS 232 serial port.

The linear actuator is coupled with a shaft, which carries the drop holder on its top. A 7 mm (diameter) $\times$10 mm (height) stainless steel cylinder with a flat top is used as a drop holder. A cylindrical hole of 3 mm (diameter) $\times$6 mm (height) is bored from its other end and facilitates its placing on top of the actuator shaft. A sample droplet is placed on the sample holder, brought in contact with the probe, and finally withdrawn at controlled speed. The instantaneous tensile force curve is recorded during the probe withdrawal from which the maximum tensile force and other useful information are obtained and cross-examined against images of bonding, debonding, and failure of the material.

Measurement of Stickiness

The properly cleaned drop holder is placed on the actuator shaft and a tomato pulp sample of about 0.1 g is transferred on top of it. The formed drop is hemispherical in shape and about 3.5 mm in radius. The probe is hooked from the balance and its positioning centrally above the sample drop is carried out manually using the optical system. To measure the stickiness, the sample is brought in contact with the probe at a fixed speed of about 50 mm/min and when the drop surface makes a good contact with the probe, it is withdrawn at the same speed. Generally, the probe must make a good contact with the drop surface but should not rupture, break, or splash it, whereas the contact time is 5 s before the probe is withdrawn. When the test is completed, the probe is detached from the balance and both the probe and drop holder are cleaned with a sequence of hot water/cold water/ethanol/cold water and finally wiped dry with lint-free tissue. Since the drop surface is altered after contact with the probe, no drop is used more than once.

In order to examine the influence of temperature on stickiness, the material temperature is varied between 30 and 50°C using a thermal regulation system ($\pm$0.1°C) equipped with an electrical lamp. A special ultrathin (0.1 mm) surface thermocouple dressed with self-adhesive backing on one side to stick on the surface of the stainless steel surface and insulation on the other side is used to drive the thermal regulation system. Because the droplet temperature cannot be practically measured without interfering the sample's behavior during testing, it was assumed the same as the rod's temperature. During measurement, in order to achieve a constant material moisture content, the air humidity is controlled using a saturated salt solution of known relative humidity, according to water sorption isotherms data for spray-dried tomato pulp.

RESULTS AND DISCUSSION

Modes of Failure

Various modes of failure were observed during withdrawal of the probe from the drop surface. Adhikari et al.[18] mentioned that stickiness is a broad term and its use, without specifying its subclasses, becomes somewhat confusing. Thus, description of the modes of failure is instrumental in defining and specifying the stickiness. According to Bhandari and Howes,[17] in the tack test, probe-adhesive failure can occur at the interface, in the inter-phase, or in the bulk of the film or substrate. Interphase failure occurs when a thin layer remains on the probe surface, whereas in bulk failure, film can be seen clinging to the surface of the substrate. Brown[22] used the term "adherence failure," which is not limited to adhesion failure and also includes partial cohesive failure. Pocius[23] reported that cohesive failure, which indicates that the product is stuck to another surface and is influenced by the viscoelastic property of the material, occurs when the adhesive bond is limited by the adhesive material property.

In this study, failure was classified into three major modes, cohesive, cohesive-adhesive, and adhesive. In the cohesive mode of failure, which is presented in Fig. 2a, the breakage takes place within the drop itself and upon separation the probe surface remains completely covered with the residue material. This mode takes place when

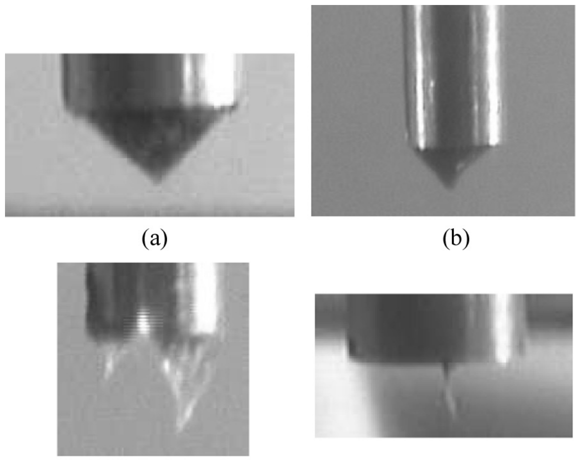

FIG. 2. Modes of failure during withdrawal of the probe from the drop surface (a: cohesive failure, b: cohesive-adhesive failure, cohesive dominance, c: cohesive-adhesive failure, equal dominance, d: cohesive-adhesive failure, adhesive dominance).

the bonding strength between the material and the probe surface is stronger than the cohesive strength of the material. According to Adhikari et al.,[19] in this case the material develops necking, which becomes progressively thinner on further pulling away of the probe and ultimately breaks down, leaving material residue on the probe.

Cohesive-adhesive failure occurs when cohesive and adhesive modes exist together and is the transitional stage between the complete cohesive and complete adhesive failures. This mode can be further classified into three subclasses, one with cohesive dominance, one with equal dominance, and one with adhesive dominance. In the mixed failure with cohesive dominance (Fig. 2b), after complete separation, 5–10% of the probe surface is cleanly separated. Figure 2c illustrates a cohesive-adhesive failure where each of the modes has almost equal dominance and almost 50% of the probe surface is still covered with material residue. Finally, when the cohesive strength of the material increases enough to allow dominance of the adhesive mode and upon complete separation only a small amount of material still remains as a residue on the probe surface, as shown in Fig. 2d, the mixed failure with adhesive dominance occurs.

In the adhesive failure, the probe surface, upon complete separation, is as clean as it was before the contact, whereas a portion of the drop surface might be inflected up by the probe while it is being separated. In this case, separation takes place at the material-probe interface, since the cohesive strength of the material just exceeds the bonding strength at the probe-material interface. Finally, when the material is drier than it is in the case where the adhesive failure is seen, a state of completely non-adhesion is observed.

Effect of Moisture Content on Stickiness

Figure 3 presents the variation of maximum tensile pressure of tomato pulp drops with moisture content at 30°C. Data for tensile pressure represent average values of two replications. The repeatability for pressure expressed as the average standard deviation of the two replications was 25.78 Pa. The tensile pressure required to separate the probe increased at the beginning and then started decreasing as the moisture content decreased. A similar trend was reported by Adhikari et al.,[19] who developed a linear actuator driven testing device for characterization of stickiness of sugar-rich foods. They reported that the rapid rise of cohesive strength of maltodextrin drop associated with the decrease of average moisture content from 1.5 to 1.0 kg water/kg solids may be due to the fact that the outer surface of the drop forms a skin soon after the onset of drying. As the drying progresses, the skin becomes thicker and the outermost layers become glassy, whereas the majority of the drop within remains as viscous solution. After a maximum tensile strength, the drop surface becomes completely non-sticky and the surface has completed its rubber to glass transition. However, in this study the material moisture content is not varied during the measurements, and thus the outer surface of the drop does not form a skin.

Figure 3 shows also the modes of failure of tomato pulp at 30°C. At high moisture contents (41.2–86.0%), the probe

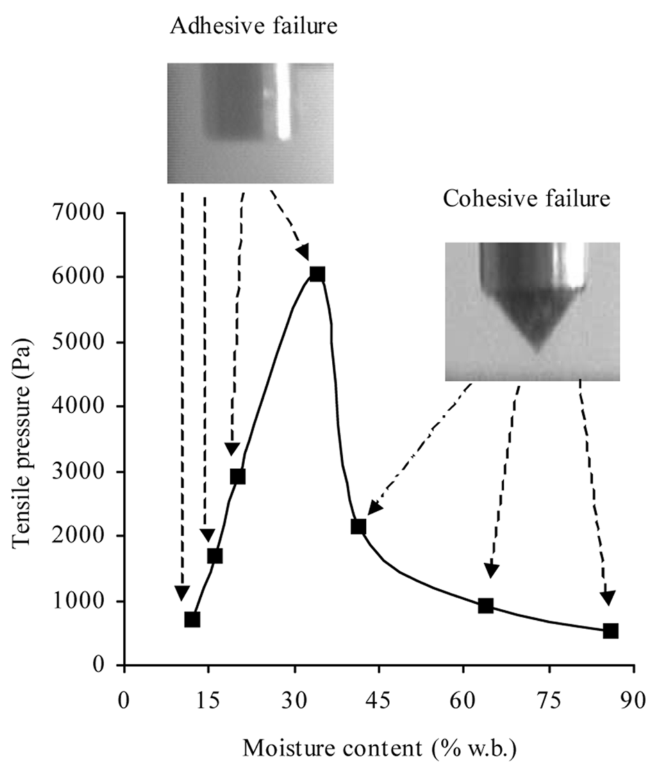

FIG. 3. Effect of moisture content on tensile pressure at 30°C.

tack test showed that the mode of failure was cohesive, indicating that the energy required to create a new surface within the drop was lower compared to adhesive energy at the probe-drop interface. This weak cohesive strength and the resulting cohesive failure represent the worst case of stickiness; thus, it is not possible for tomato pulp to be easily spray dried. This explains our daily experience that if tomato pulp comes in contact with an equipment surface, it easily adheres to it and cannot be removed easily without leaving a substantial amount of residue. Below moisture content of 34.0%, the failure was adhesive. At the peak tensile pressure observed at 34.0% moisture content, the surface of the drop appeared to be soft and thermoplastic and inflection of part of the drop surface was observed when the probe was withdrawn.

Adhikari et al.[18] also reported that maltodextrin solution shows cohesive failure at higher moisture contents, but when moisture content decreases to 0.98 kg water/kg solids, a peak tensile pressure is observed and the failure is adhesive, a trend similar to that obtained in this study. When the drop is further dried, the surface becomes rugged, hard, and completely non-sticky at moisture 0.69 kg water/kg solids. In addition, although sucrose solution exhibits cohesive failure throughout the whole moisture content range, when maltodextrin is added, the drop surface fails cohesively at high moisture contents, whereas when the moisture content decreases to 0.70 kg water/kg solids, the failure is completely adhesive, giving a peak tensile pressure. However, in this study the maximum tensile pressure was observed at a moisture content of 34% or 0.52 kg water/kg solids. This lower moisture content may be explained by the extent of material stickiness. According to Bhandari et al.,[24] the extent of stickiness or the consequence on structural change of a material depends on the difference between the temperature of the product and its glass transition temperature and a small change in Tg may have a major effect on the sticky behavior of the product. Tomato pulp has a lower glass transition temperature compared to maltodextrin and sucrose-maltodextrin mixture. According to Adhikari et al.,[19] the cohesive failure represents the worst case of stickiness, and the stickier the product the points of adhesive failure move toward lower moisture contents.

As the moisture content was lowered from 86.0 to 34.0%, the tensile strength increased. This observation is similar to that obtained by other researchers, who studied the stickiness of fructose, sucrose and honey using a probe tack method.[18,19] They reported that the increase of cohesive strength with increase in solids concentration was lowest for fructose, highest for sucrose, whereas that of honey was between and all of these materials failed cohesively. The tensile strength values of tomato pulp with moisture contents of 86.0, 64.0, and 41.2% were 521.5, 922.8, and 2150.5 Pa, respectively, whereas that of pure water droplets was about 87 Pa. The tensile strength increases with total solids concentration, which indicates that the tomato pulp molecule has affinity for water molecule. The cohesive strength of the water molecule is mainly due to hydrogen bonds, and thus the higher tensile strength of tomato pulp, compared to water, indicates a possible increase in number of hydrogen bonds in the solution. Compared to water, the viscosity of tomato pulp with moisture contents of 86.0, 64.0, and 41.2% is increased by a factor much higher than that corresponding to the tensile strength. Thus, in this case where the drop fails cohesively and the formation of the new surface takes place within the drop itself, the viscosity may not be the dominant factor that controls the surface stickiness.

Below moisture content of 34.0%, it was expected that the tensile strength would increase with solids concentration following the behavior described previously. However, the tensile pressure decreased rather than increasing. Generally, the viscosity of a solution increases with an increase in the solids concentration. Thus, if the viscous force was the dominant factor then the tensile strength of the drop should exhibit an increasing trend as shown by the viscosity. This observation leads to the conclusion that surface forces rather than viscous forces are dominant. However, as it can be seen in Fig. 3, below moisture content of 34.0% the failure was adhesive. Adhikari et al.[25] measured the tensile strength of lactose solutions using a probe tack test and mentioned a similar trend. The tensile pressure decreased rather than increased with solids concentration. However, in that case the failure was cohesive (adhesive stickiness), and thus they concluded that for surface forces to be dominant over viscous forces, a key criterion has to be met, which is: The cohesive strength of the drop should be weak enough to allow the formation of new surface. Therefore, surface tension appears to be a good indicator for the adhesive mode of stickiness. However, in this case the failure was adhesive (cohesive stickiness), and this leads to the conclusion that the effect of moisture content on tensile strength cannot be used as a criterion for deciding if the surface rather than the viscous forces are dominant.

Effect of Temperature on Stickiness

Figure 4 presents the effect of material moisture content on maximum tensile pressure at 35°C. In this case, the cohesive strength increased on increasing the solids concentration and drop failed cohesively until the moisture content decreased to 34.0%, where the failure was cohesive-adhesive with adhesive dominance. When the moisture content decreased to 20.0%, the failure was completely adhesive giving a peak tensile pressure. At 40 and 45°C (Figs. 5 and 6), the variation of stickiness with moisture followed a trend similar to that at 30 and 35°C. However, the failure at moisture content levels of 34.0 and 20%

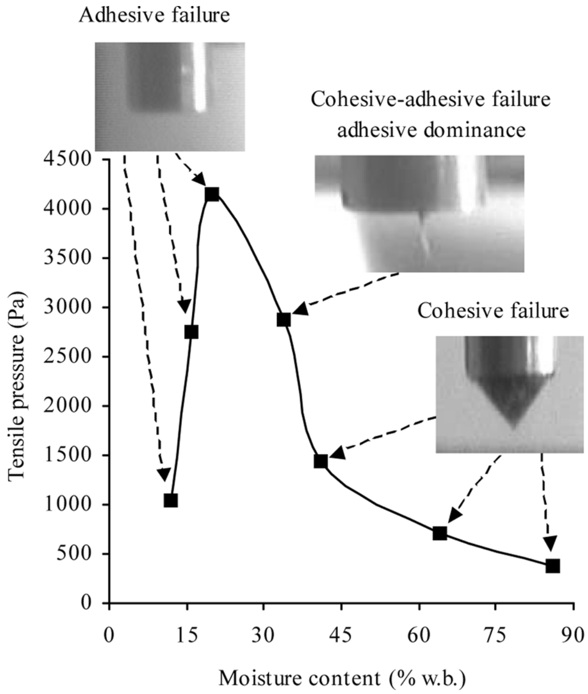

FIG. 4. Effect of moisture content on tensile pressure at 35°C.

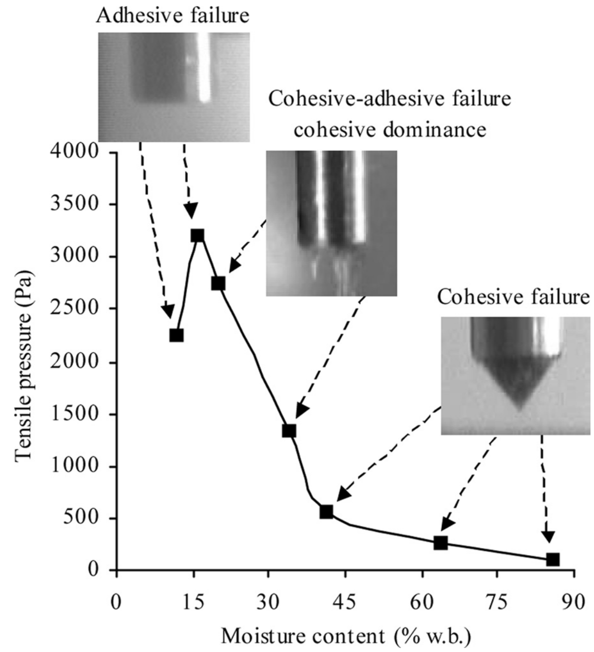

FIG. 6. Effect of moisture content on tensile pressure at 45°C.

was adhesive-cohesive with cohesive dominance, and a peak tensile strength was observed at a moisture content of 16.0%, where adhesive failure took place. Finally, at 50°C (Fig. 7) cohesive failure occurred throughout, which implies that the cohesive strength of the drop surface never attains a value at which the adhesive failure at probe-drop interface could have taken place. In addition, at this temperature, the curve does not present a peak tensile strength and the tensile pressure increases by increasing solids concentration throughout the whole moisture content range.

Figure 8 summarizes the effect of temperature on stickiness. It can be concluded that higher temperatures shift the points of adhesive failure toward lower moisture content.

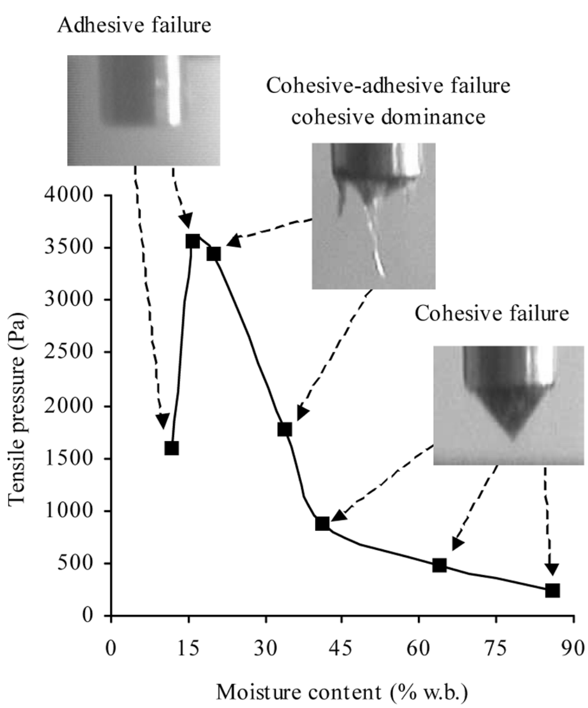

FIG. 5. Effect of moisture content on tensile pressure at 40°C.

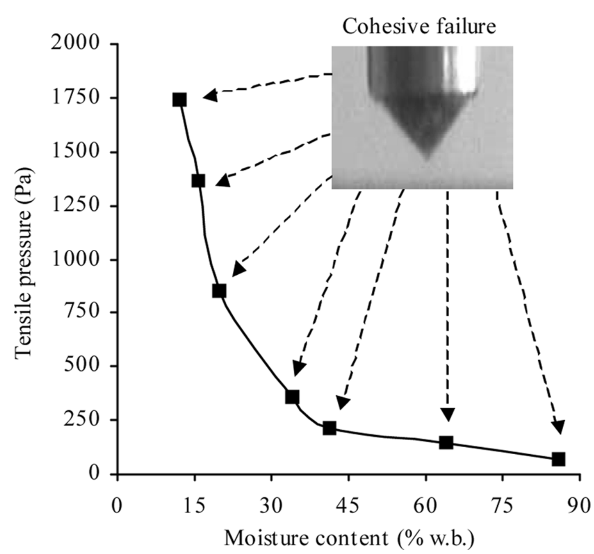

FIG. 7. Effect of moisture content on tensile pressure at 50°C.

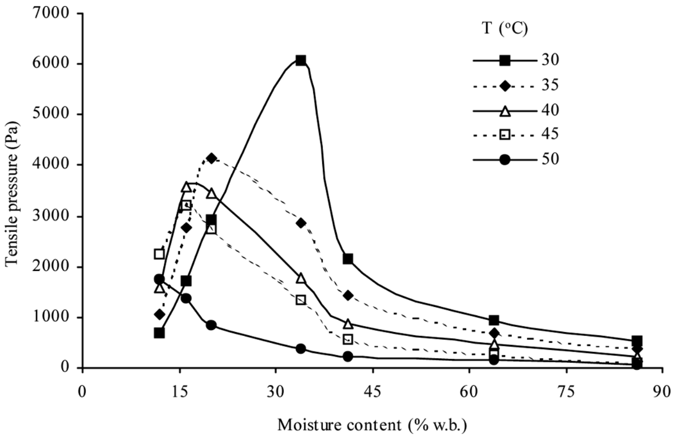

FIG. 8. Effect of temperature on tomato pulp stickiness measured by the probe tack test.

Hence, it could be said that operating at higher temperatures worsens the stickiness behavior of tomato pulp drying. This observation is similar to that obtained by Adhikari et al.,[18] who characterized stickiness of maltodextrin solutions at 63 and 95°C. In that case, the variation of stickiness with moisture at 95°C was found similar to that at 63°C, and the only difference was that the adhesive failure and the point of non-adhesion were observed at moisture contents of 0.79 and 0.53 kg water/kg solids, respectively, which were much lower than their corresponding values at 63°C. Addition of maltodextrin to sucrose solutions gives similar results. It was found that maltodextrin shifts the points of adhesive failure and non-adhesion toward higher moisture contents.[19] These effects of temperature and maltodextrin on the variation of maximum tensile pressure with moisture content explain why two of the measures adopted to cope with the spray drying of tomato pulp are operation at lower temperatures and addition of high-molecular-weight additives, such as maltodextrin.[2,24]

In addition, as can be seen in Fig. 8, at high moisture contents the tensile pressure decreased by increasing temperature. This observation means that the cohesive strength decreases with an increase in temperature, implying that the higher temperatures favor cohesive failure and hence would not be conducive to spray drying. This temperature effect may be due to the increased mobility of molecules and decreased interparticle force of attraction at the higher temperatures. The decreased viscosity at higher temperatures also decreases the cohesive strength.

SUMMARY

A stickiness testing device was fabricated, tested, and used to characterize the stickiness of tomato pulp. Tomato pulp samples of different moisture contents were used and the effect of temperature was studied. Tomato pulp droplet exhibited cohesive, adhesive, or semi-adhesive stickiness depending on pulp moisture content and temperature. Generally, adhesive stickiness was exhibited when tomato pulp had high moisture content and the cohesive strength increased as the total solids content increased, followed by semi-adhesive failure and completely adhesive failure when the moisture content decreased to a certain level. In addition, the higher the droplet temperature, the more intense the cohesive failure, as higher differences between the temperature of a material and its glass transition temperature favor adhesive stickiness.

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