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Effect of Temperature and Moisture on Dynamic Viscoelastic Properties of Soybeans

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EFFECT OF TEMPERATURE AND MOISTURE ON DYNAMIC VISCOELASTIC PROPERTIES OF SOYBEANS

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ABSTRACT. *The soybean crushing industry utilizes conditioning in the temperature range of 60–90°C and moisture range of 9–12% to modify the viscoelastic properties of cracked soybeans in order to make them more deformable and to produce thin flakes for oil extraction. Viscoelastic properties of soybean cotyledons, namely G′ and tan δ in the temperature range of 30–120°C and moisture range or 9.5–12.8% were determined using DMTA. In this moisture range, G′ decreased at a rapid rate by 2–4 times due to an increase in conditioning temperature from 30°C to 72.5–81°C. Thus, flake thickness could be substantially reduced by conditioning soybeans up to 72.5–81°C. The tan δ curves showed a peak in the temperature range of 40–50°C, which shifted to a lower temperature by 2–4 °C after a decrease in frequency from 1 Hz to 0.3 Hz. This indicates that the material underwent glass transition in this temperature range. In some experiments a second transition occurred at 95–105°C. However, data was insufficient to conclude that this was a glass transition. In general, G′ values below 10% moisture content were about two times greater than at higher moisture contents, which confirmed that at room temperature below 10% moisture content soybeans existed in a glassy state. Due to low deformability of soybeans in the glassy state, flaking below 10% moisture content would require higher mechanical energy and result in thicker flakes.*

***Keywords**. Viscoelastic, Glass transition, Soybeans, Storage–modulus, Loss–modulus.*

Most biological materials, including soybeans, are viscoelastic. In the soybean crushing industry, soybeans are conditioned through application of heat and moisture before forming them into thin flakes for oil extraction. During conditioning, thermo–hydro effects modify the viscoelastic properties of the material and make them more deformable. Easily deformable soybeans form thinner flakes, from which more oil can be extracted. Our preliminary work determined that the U.S. soybean processing industry might be losing $7–16 million in oil extraction potential because of lack of information on soybean viscoelastic properties in typical operating temperature and moisture ranges (Singh et al., 1999).

Viscoelastic properties depend upon the nature of the material and the history of applied thermal, moisture and mechanical stresses. Viscoelastic properties are determined by quasi–static and dynamic tests. The quasi–static tests determine the stress relaxation or creep behavior of the material over a long time scale, and the dynamic tests determine the storage and the loss moduli of the material over a short time scale (Christensen, 1982). Several workers have performed quasi–static experiments on soybeans (Bilanski, 1966; Paulsen, 1978; Parrish et al., 1982; Bilanski, 1987; Liu et al., 1990; Cenkowski et al., 1991; Maki et al., 1994). Liu et al. (1990) obtained relaxation moduli and the ultimate strength of soybean cotyledons at different moisture and temperature levels. The maximum temperature used in their experiments (55°C) was lower than the range (60–75°C) at which conditioning is performed in the soybean crushing industry. Cenkowski et al. (1991) performed creep and recovery tests on soybean kernels. Bilanski (1987) determined that the apparent modulus of elasticity and deformability of soybeans became constant at moisture content values greater than 13% (all moisture contents are reported on a wet basis unless otherwise noted). The experiments were performed at room temperature. At this temperature, the apparent modulus of elasticity rose sharply as moisture content declined below 9%, showing an inflection point in the region of 7–9% moisture content (Maki et al., 1994). The sharp rise occurred due to formation of the glassy state in the cotyledon cytoplasm at moisture content below 9%. In the glassy state, the force required to cause deformation was very high and a sudden failure, like in brittle materials, was observed (Maki et al., 1994). None of the past studies have determined the viscoelastic properties of soybeans in the narrow moisture range of 9–12% at which soybeans are typically conditioned and flaked in the crushing industry.

Dynamic mechanical testing provides a sensitive method for determining thermomechanical characteristics of the material by measuring the elastic modulus (G′) and loss modulus (G″) as a function of frequency, temperature, or time (*ASTM Standards*, 1995). Tests on viscoelastic solids are performed using DMTA. The dynamic tests are very sensitive to molecular scale transitions taking place in the material.

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Williams (1994) performed some preliminary dynamic tests on soybeans and peas in a temperature range below 50°C and reported test–plots for peas. In the temperature range of 30–40°C, the pea cytoplasm started transiting from the glassy to the rubbery state. He noted that the application of DMTA testing in seed science was quite recent. In the cytoplasm of soybeans the glassy state has been observed due to transition taking place in the disaccharide–water domains at room temperature at moisture contents below 10% (Bruni and Leopold, 1992). This transition was called the hydration–dependent glass transition. In the glassy state, soybeans are hard to deform and the internal stresses have a long relaxation time. As we will show in this paper, upon heating above T_g, soybeans change into a rubbery state at a fast rate up to 72.5–80°C. During transition from a glassy to a rubbery state, a sharp increase in free volume and entropy, and a sharp decrease in viscosity, take place. In DMTA experiments, the glass transition is exhibited through a sudden decrease in G′ followed by a peak in the tan δ function, both of which are frequency dependent.

Glass transition is also observed in solid polymers due to changes in molecular configurations and entanglements with change of temperature (Ferry, 1980). Biological materials also exhibit glass transition. For example, various proteins like ovalbumin, sodium caseinate, casein, gluten, and gelatin showed a peak in tan δ at about 80, 85, 60, 40 and 55°C, respectively, at a moisture content of 15% (Kalichevsky et al., 1993). In polymer mixtures, water acts as a plasticizer. Water is called a "mobility enhancer" because its low molecular weight leads to a large increase in mobility of the polymers, due to increased free volume and decreased viscosity (Slade and Levine, 1991). At higher moisture contents T_g values are lower. At a constant temperature, an increase in moisture causes a decrease in G′. Both temperature and moisture can affect the viscoelastic modulus of the sample.

The overall goal of our study was to determine the viscoelastic properties of soybeans at a temperature and moisture content range used in the soybean crushing industry.

Our specific objective was to determine the storage modulus (G′) and the tan δ values for soybean cotyledons in a temperature range of 30–120°C and a moisture content range of 9–12%.

To determine the viscoelastic properties, DMTA was the method of choice due to its sensitivity in recording macroscale mechanical properties that have a direct correlation to microscale changes taking place inside the material.

Materials and Methods

Composition of several soybean varieties obtained from the 1998 Purdue University Soybean Performance Trials was determined with a Near–Infrared Transmittance (NIRT) Whole Grain Analyzer (Infratec 1229, Foss North America Inc., Prairie, Minn.). The composition of the 'Resnik' variety was representative of varieties grown in Indiana. Thus, this variety was selected for the DMTA experiments. Different constituents of this variety were: protein 37.5%, oil 17.9% (both at 13% reference moisture content basis), and initial moisture content 7%. The soybeans were stored in zip–lock bags at room temperature for about two months before their use in experiments.

Sample Preparation

To separate the seed coat and prepare regular shapes for the DMTA experiments, the soybeans were sanded into cylindrical shapes by using a belt sander equipped with a custom–built sample holding device. The axis of the cylinder was perpendicular to the plane joining the two cotyledons. After grinding, the two cotyledons were split at the joint into two smaller cylinders (diameter 3.5–4.7 mm, thickness 1.5–2 mm). Thus, there was no cleave in any sample. Grinding did not seem to change the properties of the material, because it gently scraped off some material from the outer layers of the whole soybeans without causing large deformations in the shaped cylinders.

The target moisture levels (9, 10, 11, 12%) were achieved by suspending the cylindrical cotyledon samples over different saturated salt solutions in humidity chambers (Lide, 1994). The Chung equation was used to calculate the relative humidity levels required to attain the desired moisture values (ASAE Standards, 1989). Table 1 shows the salt solutions and temperature settings used in the conditioning chamber to attain desired moisture content values.

The salt solutions were selected such that the desired humidity levels were attained at chamber set–point temperatures below 40°C. This was a precaution against causing irreversible changes in the structure of biopolymers due to high temperatures during conditioning and to assure that these changes would occur during the DMTA runs only.

It would have been expensive to use sanded samples to periodically monitor moisture values by the destructive convection–oven method during conditioning. Therefore, whole soybeans were conditioned along with the cylindrical samples. The purpose of conditioning whole soybeans was to roughly estimate the moisture content achieved by the cylindrical samples at a given time. In the DMTA runs only the cylindrical samples were tested. The moisture content of the whole soybeans was monitored every two days using a GAC 2100 Dickey–John moisture meter. When the moisture content of the whole soybeans reached close to the target value, the cylindrical samples were vacuum–sealed and stored in zip–lock bags in a cold room at 4°C. The desired moisture levels were attained in 10–15 days. There was no significant color change of the cotyledons observed due to possible biochemical effects during the conditioning and storage time period. Immediately before performing DMTA experiments, about 15 cylindrical samples of a given moisture level were dried in a convection oven at 103°C for 48 hours to determine the exact moisture content. The average moisture contents attained by the cylindrical samples were 9.5%, 10.5%, 11.5% and 12.8%. The moisture

Table 1. Salt solutions and chamber temperature settings used to obtain desired soybean moisture content values. Relative humidity and actual moisture content were the measured values.

Salt Solution	Relative Humidity	Set Temperature	Target Moisture	Actual Moisture
Sodium bromide	55%	37 °C	9%	9.5%
Ammonium nitrate	60%	28 °C	10%	10.5%
Potassium iodide	66%	40 °C	11%	11.5%
Sodium nitrate	72%	40 °C	12%	12.8%

content of 6–10 samples of each moisture level was also determined at the end of the DMTA runs by the oven drying method to quantify the moisture loss during runs.

DMTA Experiments

The DMTA was calibrated once a week using the standard procedure recommended by its manufacturer (Polymer Laboratories Ltd., Loughborough, U.K). This DMTA unit was designed to perform shear type tests on small samples. An attachment was fabricated to conduct compression type tests. However, this method did not prove successful because during the reverse stroke, the moving part of the attachment did not remain attached to the sample. Attempts to conduct compression tests after gluing the particle to the attachment were made. However, this required application of a small force on the moving part of the DMTA, which could damage the sensitive spring. After each such attempt recalibration of the DMTA was required. Therefore, the compression tests were abandoned after initial trials. New DMTA instruments with auto–tension devices are designed to carry out the compression tests.

About twelve replications of shear type tests were performed on samples at each moisture content. To avoid biases introduced by the testing sequence, the samples were tested in the following order: 12.8, 9.5, 11.5, and 10.5%. About 10 hours before testing samples of a given moisture content, the sample bags were placed at room temperature. Each shear type run required mounting two samples on both sides of the moving part of the attachment. The average values of diameter and thickness of the two samples was entered into the DMTA software. After mounting the samples in the DMTA, they were covered with petroleum jelly (Vaseline™). This helped to minimize the moisture loss during experiments. During preliminary runs, particles slipped at a low normal force in the DMTA attachment and a high normal force squeezed out the oil. Therefore, it was decided to glue the particles to the DMTA attachment using Super Poxy glue (Power Poxy Adhesive Inc., New Berlin, Wisc.). This glue had a strength of 20 N/mm^2 at 25°C, which is 100 times greater than the maximum stress of 0.2 N/mm^2 applied by the DMTA. The maximum allowable temperature for this glue was 122°C. The glue was allowed to cure for 45 minutes before starting a run. An oscilloscope attached to the DMTA was used to monitor for slip conditions. It displayed two moving points indicating deformation and force behavior of the samples during experiments. The force point displayed oscillations with high amplitude in the event of slip. Whenever slip was observed, the experiment was stopped to take corrective measures, or to reject the data entirely. To check the effect of temperature on the viscoelastic behavior of the glue, a test was conducted by directly gluing together the moving and stationary parts of the DMTA attachment. The indicated G' value remained constant over the entire temperature range, which showed that the glue did not undergo any transition. At the end of an experimental run, the DMTA attachment was cleaned with an ethyl alcohol based cleaner.

During a given DMTA run, temperature sweeps were performed while deforming the samples simultaneously at two frequencies (0.3 Hz, 1 Hz). The DMTA applied a sinusoidal deformation of 16 μm (1% strain) to particles and measured their force response. For this strain the behavior of the material was within the linear viscoelastic range. This was further confirmed from the traces of amplitude and force curves observed in the oscilloscope. The deformation and force values were converted into G' and tan δ by equations programmed into the DMTA's microprocessor (DMTA Manual). The temperature was increased from 30°C to 120°C at a rate of 1°C /min. The frequencies were selected after several trial runs: at frequencies less than 0.3 Hz, the DMTA calculations were based upon fewer sampling points, thus more noise was observed; at frequencies greater than 1 Hz the presence of slip was observed and many more samples had to be discarded.

Data Analysis

The DMTA measured G' and tan δ values every 4 seconds. The median values of G' and tan δ measured during the one minute temperature intervals of 1°C were plotted versus the temperature points. In some cases very high fluctuations were observed in the recorded values. Such points were termed as noise and were either deleted or smoothed using the central average scheme. The reason for the noise is not clear. It was observed at all moisture content levels (9.5–12.8%). We hypothesize that the noise resulted from the non–parallel opposite faces of cylindrical samples, which lead to inaccuracies at 1% strain levels. During compression type DMTA tests on peas, Williams (1994) observed that the amount of noise decreased after the samples attained the rubbery stage. It is possible that in the rubbery stage opposite sample faces become more parallel due to greater plasticity of the material.

When calculating the average values of G' and tan δ at each moisture level, the samples were divided into two groups. During DMTA runs about two–third of the samples formed or tended to form two peaks in the tan δ curves. These samples were termed double peak samples. Due to unknown reasons about one–third of the samples did not tend to form the first peak in the tan δ curves. Grouping of these samples with double–peak samples would not have allowed making practical deductions and would have caused errors during averaging over samples. Thus, the single–peak and double–peak samples were analyzed separately.

Discussion of Results

During DMTA runs, a slight loss in moisture was expected due to increase in temperature. The measured moisture loss values for 9.5, 10.5, 11.5 and 12.8% samples were 0.9, 1.1, 0.6 and 1.5 percentage points, respectively. This moisture loss was relatively small because the samples had only 45% exposed surface area. The remaining area was glued to the DMTA attachment. Covering the exposed area with petroleum jelly further helped in reducing the moisture loss. Subsequent results are presented based on the initial sample moisture contents.

Temperature and Moisture Effects

Double Peak Samples

Figure 1 shows the variation of G' and tan δ with temperature for 9.5% moisture samples deformed at 1 Hz. From 30 to 72.5°C, G' decreased at a rapid rate after which the rate slowed. The G' value for samples with moisture content of 10.5, 11.5 and 12.8% decreased at a rapid rate up to 72.5, 81 and 75°C, respectively (figs. 2–4). At lower magnitudes of G′, soybeans were more rubbery and easily deformable. Some soybean processing industries crack and de–hull soybeans before conditioning them. In these industries, conditioners are generally operated in the temperature range of 60–70°C. An increase in conditioning temperature up to 72.5–81°C would be expected to yield thinner flakes. The increased thermal energy required to operate a conditioner at a higher temperature would be somewhat compensated by the reduced mechanical energy required to flake more easily deformable soybeans. At all moisture contents, samples tested at 0.3 Hz showed lower G′ values than those tested at 1 Hz (comparison with 0.3 Hz not shown). This was expected because the elastic component of the viscoelastic materials decreases with decrease in oscillation frequency (Christensen, 1982).

In figures 1–4, the tan δ curve shows a peak in the temperature range of 40–50°C. The peak shifted by 2–4°C to a lower temperature at 0.3 Hz (comparison with 0.3 Hz not shown). Near glass transition the relaxation times for molecular motions are so large that the T_g values may depend on the time scale of the experiment by which it is measured (Gibbs and DiMarzio, 1958). In dynamic tests this is exhibited through shifting of tan δ peaks to lower temperature with decrease in frequency (Christensen, 1982). The rapid change in G′ values with temperature and shift in tan δ peaks with frequency confirmed that the soybean cotyledons transited from the glassy to the rubbery stage in the temperature range of 40–50°C. During glass transition, the temperature at which G′ started decreasing at a fast rate was less than at which the tan δ peak was attained. Therefore, for soybeans at 9.5% moisture content, after undergoing a slope change in the temperature range of 25–30°C, the G′ value was decreasing at a fast rate until the equilibrium was attained at 72.5°C (fig. 1). For samples with 10.5–12.8% moisture content, the equilibrium was attained in a temperature range of 72.5–81°C (figs. 2–4).

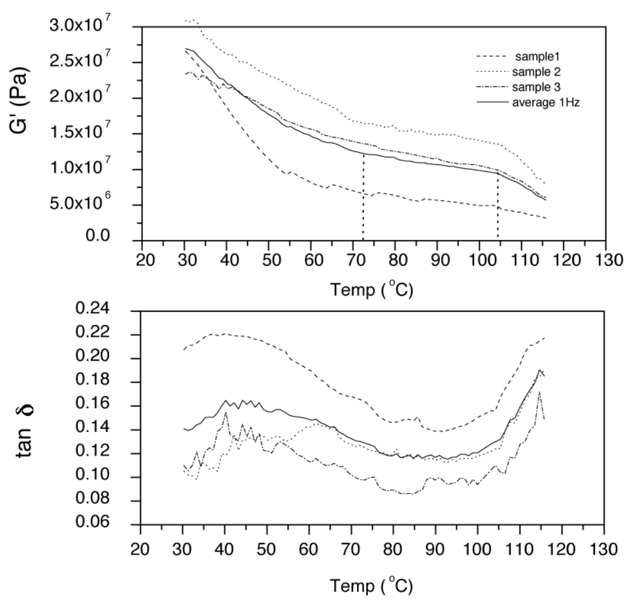

Figure 1. Temperature versus storage modulus (G′) and the ratio of loss modulus to storage modulus (tan δ) for soybean cotyledons with 9.5% moisture content deformed at 1 Hz. Samples formed or tended to form two peaks in tan δ function.

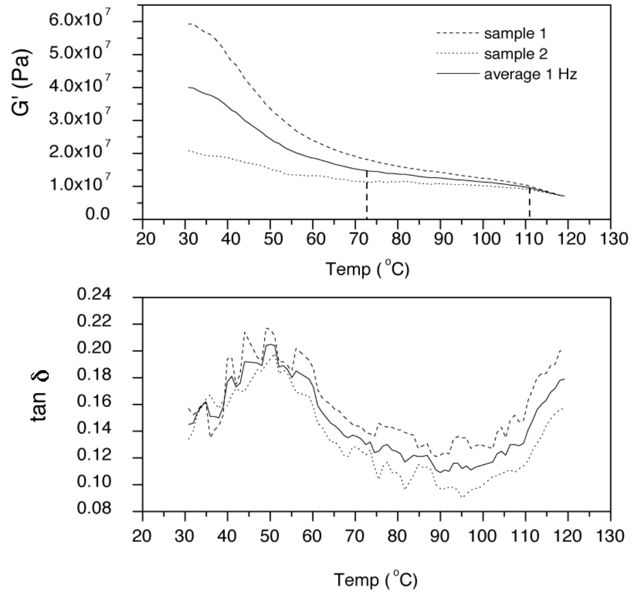

Figure 2. Temperature versus G′ and tan δ for soybean cotyledons with 10.5% moisture content deformed at 1 Hz. Samples formed or tended to form two peaks in tan δ function.

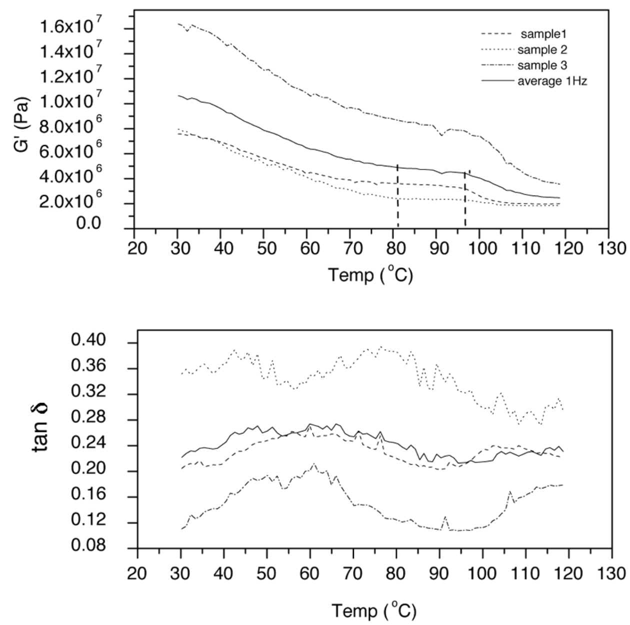

Figure 3. Temperature versus G′ and tan δ for soybean cotyledons with 11.5% moisture content deformed at 1 Hz. Samples formed or tended to form two peaks in tan δ function.

Different techniques used to determine T_g yield different results and it is necessary to make allowances for the measurement principle being used (Mitchell et al., 1991; Kalichevsky et al., 1993). Soybean tissue containing 7–9% or less moisture content exists in a glassy state at room temperature (Bruni and Leopold, 1992; Kalichevsky et al., 1993; Sun and Leopold, 1993; Maki et al., 1994). Bruni and Leopold (1992) conducted experiments using ESR. In the literature, the comparison of the T_g value obtained by the DMTA with the one obtained by DSC, DETA and NMR has been reported (Kalichevsky et al., 1993). But, studies

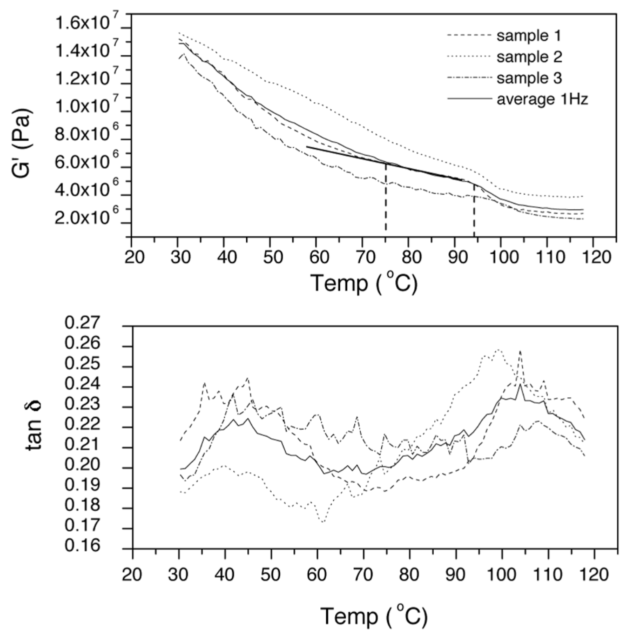

Figure 4. Temperature versus G′ and tan δ for soybean cotyledons with 12.8% moisture content deformed at 1 Hz. Samples formed or tended to form two peaks in tan δ function.

comparing T_{gDMTA} with T_{gESR} have not been performed. Thus, absolute comparison of the T_g range (tan δ peak) of 40–50°C with the room temperature value obtained by Bruni and Leopold (1991) cannot be made. Still, the rapid decrease in G′ values with temperature and shift in tan δ peaks with frequency in the temperature range of 40–50°C and moisture content range of 9.5–12.8% suggested that soybeans must have existed in a glassy state at lower temperature (less than 30°C) and moisture values (less than 9%). Because it is more difficult to deform soybeans in the glassy state, flaking below 10% moisture content would require higher mechanical energy and would produce thicker flakes.

At 9.5% moisture content, the slope of G′ reduced and became linear between 72.5 and 105°C (fig. 1). A similar reduction in slope was observed for samples at higher moisture contents (10.5–12.8%) in the temperature range from 72.5–81°C up to 95–110°C (figs. 2–4). In the soybean processing industry, an increase in temperature from 72.5–81°C up to 95–110°C would not cause much improvement in flake thickness because the deformability of soybeans would decrease at a slow rate.

Above 95–110°C a transition in the slope of G′ was again observed (figs. 1–4). In general the temperature at which the second transition began decreased from 105 to 95°C with increasing moisture content from 9.5 to 12.8%. It seemed that the higher temperature imparted more energy to bound water molecules, thus improving its role as plasticizer. Thus, we know that the transition in this temperature regime was sensitive to moisture content. The samples with 12.8% moisture content showed a second tan δ peak near 103°C (fig. 4), which shifted to a lower temperature after decreasing frequency from 1 Hz to 0.3 Hz (0.3 Hz data not shown).

Biopolymers with higher molecular weights have higher values of T_g (Slade and Levine, 1991). The shift in tan δ peak indicated that in this temperature regime some higher molecular weight component of the soybeans could be undergoing glass transition. However, the data was insufficient to establish the effect of frequency at lower moisture contents (9.5–11.5%). For samples with lower moisture content the tan δ curve rose around 100°C and it would ultimately have formed a peak at some temperature value greater than 120°C. However, the maximum allowable temperature of the glue (122°C) did not allow us to perform experiments beyond 120°C. Thus, the currently available data was not sufficient to support the notion that soybeans underwent glass transition in this temperature range. It could be some other type of transition. It could even be an artifact of the measurement, because at the end of our experiments some volume increase of samples was observed. Therefore, further study is required to establish firmer conclusions on the effect of frequency and volume increase on viscoelastic behavior in this temperature regime.

Some soybean processing companies use the hot–bean dehulling process, where whole soybeans are conditioned at about 11% moisture content and 90°C. These companies are able to obtain thinner flakes than those conditioning cracked soybeans at lower temperature (60–75°C) (Singh et al., 1999). This may be the result of conditioning the soybeans in the proximity of the second transition temperature, which was observed in our experiments to occur at 95–105°C.

Single Peak Samples

The data of samples forming or tending to form only a single peak in tan δ is not shown here. The first peak in tan δ (at 40–50°C) was not clearly visible in these samples. The reason for this behavior is not clear at this time. Research has shown that certain factors (e.g., temperature) lead to accelerated aging of food grains due to which they loose their ability to undergo glass transition (Sun and Leopold, 1993). Soybeans conditioned at 35°C for more than 22 days failed to attain a glassy state below room temperature (Sun and Leopold, 1993). In our experiments about one–third of the samples showed this phenomenon at all moisture contents (9.5–12.8%). It appears that even when the same treatment was given to different samples at each moisture level, some random factors suppressed the glass transition in some of them in the first temperature regime (40–50°C). The effect of such factors has not been reported in the past, thus, further study is necessary to quantify them.

The behavior of the G′ and tan δ functions for single peak samples at temperatures above 100°C was similar to their behavior for double peak samples (figs. 1–4).

Effect of Moisture on Average G′ Values

Figure 5 shows the effect of moisture on average G′ values. In single peak samples the G′ values were greater at 9.5% moisture content than at other moisture values. The G′ values for samples with 10.5–12.8% moisture content overlapped in a wide temperature range. In double peak samples the G′ value for samples with 9.5% moisture content was greater than for samples with 11.5 and 12.8% moisture content. However, the values of 10.5% samples were the largest. Since sample–to–sample variations in G′ values were observed (figs. 1–4), conclusions can only be made in a general sense. The data showed that in general the G′ decreased with an increase in moisture content. The G′ value for 9.5% moisture content soybeans was about two times greater than the value at higher moisture contents. This is consistent with literature values indicating that soybeans

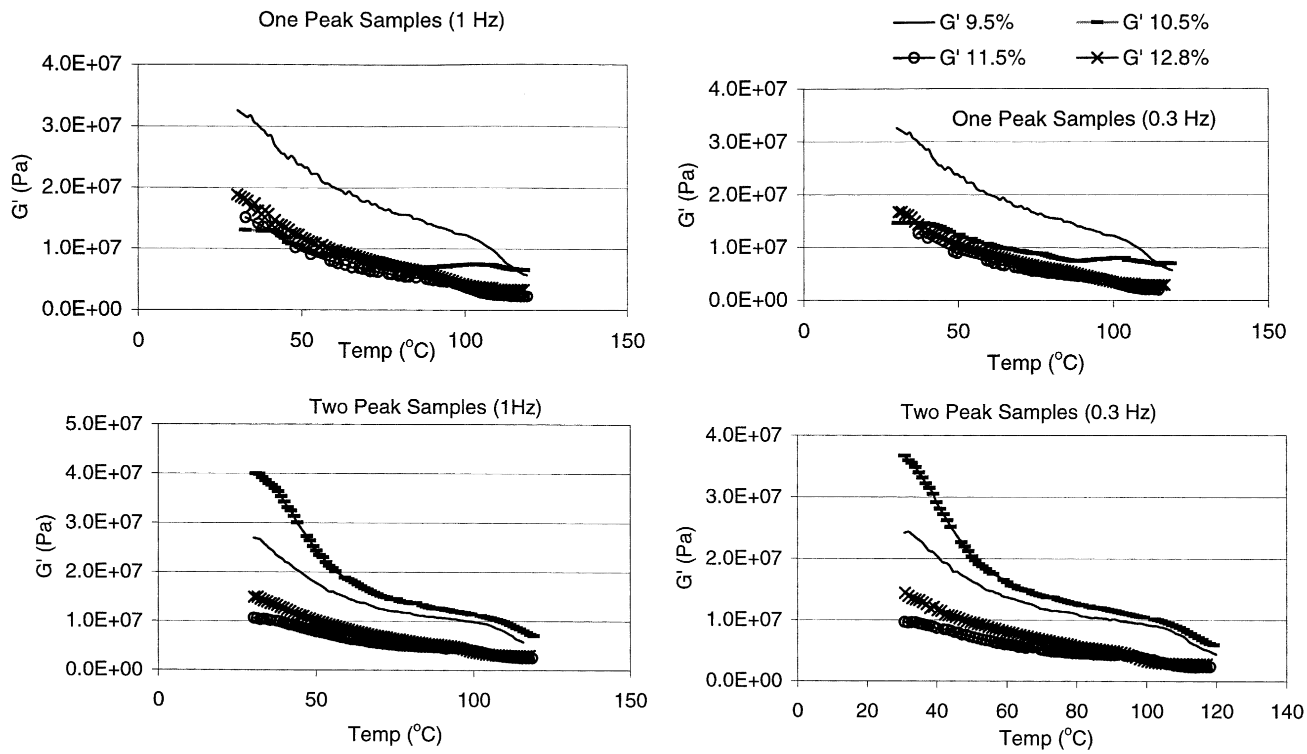

Figure 5. Effect of moisture content on average G′ values of one peak and two peak samples deformed at 1 and 0.3 Hz.

exist in the glassy state below 10% moisture content (Bruni and Leopold, 1992; Sun and Leopold, 1993; Maki et al., 1994). Experiments with more precisely ground samples and a larger number of replications are required to more accurately establish the effect of moisture content on average G′ values.

Suggestions for Experimental Improvement

1. In experiments involving glass transition behavior of anhydrous materials (e.g. soybean, corn, shrimp), it is important that the temperature during conditioning is kept below 25°C. We could not take this precaution (our maximum temperature was 40°C, table 1) because until we were well into our experiments, we did not know up to which temperature the effect of hydration–dependent glass transition remains. Because the aim of our experiments was to determine viscoelastic properties at a relatively higher temperature, we expected that in this range the hydration–dependent glass transition would not play a role, as cytoplasm would already have turned into a rubbery state. In this range we were expecting glass transition effects only due to high molecular weight biopolymers, which undergo transition at a much higher temperature (40–100°C) (Kalichevsky et al., 1993). Therefore, we expected that any changes in mechanical properties due to conditioning up to 40°C would be reversible, which did not prove to be true.
2. To reduce sample–to–sample variation of absolute value of G′, a sample holding device and grinding machine are required that would make the opposite faces of the samples more parallel. Also, a larger number of replications are needed.
3. Data on the viscoelastic behavior of soybean cotyledons at a temperature greater than 100°C may not be needed from a processing point of view. However, if particle behavior in this temperature region were needed, glue with a higher working temperature (to about 150°C) and a shorter curing time (less than 1 hour) would be required. We could not find such glue in the market.

Conclusions

1. In the moisture content range of 9.5–12.8%, a two to four times decrease in G′ was observed due to increases in temperature from 30°C up to 72.5–81°C. In the soybean crushing industries that operate conditioners at 60–70°C, an increase in temperature up to 72.5–81°C would cause a substantial decrease in flake thickness because it is easier to deform soybeans with lower G′ values. The increased energy demand of the conditioner operating at a higher temperature would be somewhat compensated by the reduced energy demand of the flaking roll stands.
2. Further increase in temperature from 72.5–81°C up to 95–105°C would not cause much improvement in flake thickness because the G′ slope initially decreased exponentially and then became linear. Therefore, the temperature range of 72.5–81°C should be considered as the optimum conditioning temperature.
3. The tan δ curves showed a peak in the temperature range of 40–50°C. The peak shifted by 2–4°C to a lower temperature upon decrease in frequency from 1 Hz to 0.3 Hz, which is consistent with the material undergoing glass transition in this temperature range.
4. A second transition temperature was observed in some of our experiments in the temperature range of 95–105°C. This observation warrants additional investigation. Lower flake thickness obtained by companies using the hot–bean dehulling process might be due to conditioning soybeans

in the proximity of the second transition temperature, provided this was a true transition and not an artifact of the measurement.

5. The G′ value for 9.5% moisture content soybeans was about two times greater than the value at higher moisture contents, which was consistent with literature values indicating that soybeans exist in the glassy state below 10% moisture content (Bruni and Leopold, 1992; Sun and Leopold, 1993; Maki et al., 1994). Flaking of soybeans with moisture content less than 10% would require higher mechanical energy and would yield thicker flakes.

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Nomenclature

DETA = Dielectric thermal analysis
DMTA = Dynamic mechanical thermal analyzer
DSC = Differential scanning calorimetry
ESR = Electron–spin resonance
G′ = Elastic (or storage) modulus (Pa)
G″ = Loss modulus (Pa)
NMR = Nuclear magnetic resonance
tan δ = Ratio of loss modulus to storage modulus (= G″/G′) (unitless)
T_g =z Glass transition temperature (°C)