Carbohydrate Polymers 79 (2010) 520–525

Contents lists available at ScienceDirect

Carbohydrate Polymers

journal homepage: www.elsevier.com/locate/carbpol

Dynamic viscoelastic properties of sweet potato studied by dynamic mechanical analyzer

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ARTICLE INFO

Article history:
Received 27 July 2009
Received in revised form 24 August 2009
Accepted 27 August 2009
Available online 3 September 2009

Keywords:
Sweet potato
Viscoelastic property
Dynamic mechanical analysis

ABSTRACT

The relaxation, creep, temperature-dependence and frequency-dependence characteristics of sweet potato roots were evaluated using a dynamic mechanical analyzer (DMA). The sweet potato was cut into rectangular to meet the testing requirements and wrapped with sealing film or aluminum foil to prevent water loss. The temperature scanning tests were carried out at 2 °C/min and 10 °C/min in the temperature range of 30–100 °C, and the frequency sweep tests were conducted in a range of 50–0.1 Hz. The regression results suggested that 5-element Maxwell model described relaxation behavior better for consisting of two relaxation times; the creep behavior matched the Burgers model well, and changes in creep parameters were observed after each creep cycle. The temperature scanning tests revealed that starch gelatinization was only obtained when the temperature increased at 2 °C/min. A resonance frequency was detected both in 3-point bending and compression deforming clamps.

1. Introduction

Sweet potato (*Ipomoea batatas* L. (Lam.)) is widely grown in many countries, especially in China and in Southeast Asian countries (An, Frankow-Lindberg, & Lindberg, 2003). The sweet potato is comprised of 60–80% of water and 10–30% of starch (Zhang, Christopher, & Harold, 2002), while it also contains a variety of nutrients, such as dietary fiber, carotenoid, vitamins, lysine and mineral elements etc., thus sweet potato is extensively used in food industry, light chemical industry, and feed industry. Recent studies show that sweet potato has brilliant anti-cancer and antioxidant effect as well (Teow et al., 2007; Tian & Wang, 2008).

Food can be regarded as some kind of complex polymer. Different food matrix shows different mechanical properties in different viscoelastic regions: the glass-like region, where the material shows a rigid and brittle character and the modulus is relatively high; the glass-transition region, where the storage modulus of the material decreases remarkably; the rubber-like region, where the material shows a high-elastic property; and the terminal region, where the material flows like liquid (Le Meste, Champion, Roudaut, Blond, & Simatos, 2002). The dynamic mechanical analyzer, DMA, has been widely used to determine the structural and mechanical changes in different kinds of matrix, such as starch film (Zhou et al., 2009), dental composite resins (Mesquita, Axmann, & Geis-Gerstorfer, 2006), cheese (Del Nobile et al., 2007a), chocolate (Tremeac, Hayert, & Le-Bail, 2008), frozen dough (Laaksonen & Roost, 2000), meat (Del Nobile, Chillo, Mentana, & Baiano, 2007b), rice kernels (Chen et al., 2007).

In order to determine the viscoelastic behavior of a food matrix, the transient and dynamic tests can be performed by DMA. The most typical transient tests are represented by creep-recovery and stress relaxation experiments (Del Nobile et al., 2007b). The relaxation and creep-recovery tests, which are quite common in the life cycle of materials, are both especially useful for studying materials under very low shear rates or frequencies, under long test times, or under real use conditions (Menard, 1999a).

The Kelvin model, Maxwell model and Burgers model are the most commonly used models to describe viscoelastic behaviors of the matrix (Del Nobile et al., 2007b; Mohsenin & Mittal, 1977) under static load. The Kelvin model, consisting a spring and a dashpot in parallel, represents the start point for the development of mechanical analogs describing the creep behavior (Del Nobile et al., 2007b). The Maxwell model, consisting of a Hookean spring and a Newtonian dashpot in series, is suitable for understanding stress relaxation data, but not able to express the equilibrium of stress; while the generalized Maxwell model, consisting of several Maxwell elements in parallel with a spring, can describe stress-equilibrium behavior better (Steffe, 1992). The Burgers model, consisting of a spring, a dashpot and a Kelvin component in series, can show the instantaneous elastic deformation, delayed elastic deformation and the character of viscous flow at the same time when

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doi:10.1016/j.carbpol.2009.08.035

Nomenclature:

σ	stress
ε	strain
E'	storage modulus
E''	loss modulus
E_1, E_2	elastic modulus
E_c	equilibrium elastic modulus
τ_1, τ_2	relaxation time
τ_r	retardation time
η	viscosity

the material is under the external force. Burgers model is one of the most used rheological models to describe the creep behavior.

DMA also gives the information of various transitions in a polymer as temperature changing. Dynamic mechanical properties of polymers can represent their molecular motion, which has a close relationship with the condensed, chain structures of polymers (Zhou et al., 2009). The thermal transitions in polymers can be explained by theory of free volume (Champion, Le Meste, & Simatos, 2000; Chen et al., 2007; Menard, 1999b), according to which, there is free volume (defined as the space a molecule has for internal movements) existing between molecules. When temperature rises, the free volume of the chain segment of a polymer increases, and its ability to move in various directions also increases. This increased mobility makes the food matrix softened and thus results in a greater compliance (or lower modulus).

Apart from the free volume for molecular movements, the water molecules in the food matrix also have a close relation to the viscoelastic properties of the materials (Chen et al., 2007; Lievonen & Roos, 2002). Water acts as one of the most effective plasticizers for biopolymers. Furthermore, the viscoelastic properties of a food matrix vary with the different amounts of water content in it. Thus, it is of great necessity to keep the water content constant by means of wrapping or coating to materials (Zhou et al., 2009).

The frequency scanning test by DMA gives information of materials' melting property and this test was usually done to the liquid samples. Many thermal analysts and material experts use DMA to study the temperature, time or strain rate (Mulliken & Boyce, 2006; Yi, Boyce, Lee, & Balizer, 2006) dependence of the matrix's mechanical property and neglect the frequency dependence of it. For a dynamic test conducted by DMA, the loading frequency played a vital role to the materials' mechanical response. The low frequency range is where viscous or liquid behavior predominates; when the frequency is relatively high, the material will act in an elastic way and behave stiff. The change caused by increasing in frequency is similar to that by decreasing in temperature (Menard, 1999c). Thus frequency dependence of the materials' viscoelastic property may also be explained by the molecular or chain movements in the free volume theory.

In this study, the relaxation, creep-recovery, temperature and frequency scanning behaviors of sweet potato were tested by DMA. The aim of this work was to model the relaxation and creep-recovery behaviors of the sweet potato sample, and characterize the temperature and frequency dependences of its viscoelastic properties.

2. Materials and methods

2.1. Materials

The cultivar of the sweet potato used in this research was grown in Hebei province and harvested in October. Usually the sweet potato was stored in cellar for months before use or distribution. After the sweet potato was purchased from the market, it was preserved in refrigerator with the temperature set at 4 °C. Before carrying out the experiments, the sweet potato was taken out of the refrigerator and left in the air for more than 20 min until the temperature inside the sample is the same as the ambient. The initial water content of the sweet potato was about 75(±2)%, which was determined by dehydrating it at 105 °C in a drying oven (Luda Laboratory Instruments, Shanghai) for more than 24 h (Del Nobile et al., 2007a).

2.2. Preparation of sample

In order to prevent the water loss, the samples were wrapped with sealing film or aluminum (Al) foil (Chen et al., 2007; Pereira & Oliveira, 2000). It should be noted that the sealing film does not change significantly the magnitude of mechanical parameters of sweet potato samples, while the Al foil does; and the sealing film cannot bear the heat while the Al foil can. Considering the above factors, in the compression mode the sweet potato slices were coated with the Al foil with its top and bottom surface exposed to the compression plates (Fig. 1); and in the 3-point bending mode the samples were wrapped with sealing film totally (Fig. 2). To meet the testing requirements of the instrument, the sample was shaped by a blade to a cuboid with size of about 10 mm × 10 mm × 10 mm in the compression mode, and a rectangle slice of about 7 mm × 10 mm × 35 mm in the 3-point bending mode. The length of the bending sample is about 5 mm longer than the clamp, so that the sweet potato chip can be bent properly. The dimensions of each sample were measured by an electronic digital caliper (PRO-MAX, Fowler, USA).

2.3. Dynamic mechanical analyzer (DMA)

The mechanical behavior of the sweet potato was measured by the Dynamic Mechanical Analyzer (DMA, Q800, TA Instruments, New Castle, USA) using 3-point bending or compression clamps.

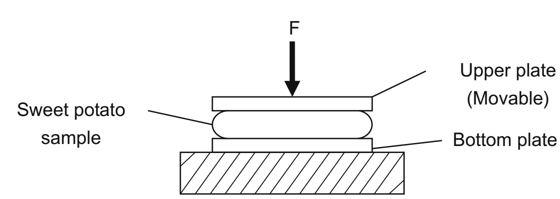

Fig. 1. The loading sketch of sweet potato sample in compression clamp.

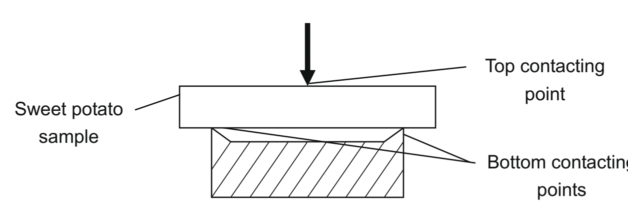

Fig. 2. The loading sketch of sweet potato sample in 3-point bending clamp.

The testing mechanics of the DMA can be simply described as applying an oscillating force to a sample and detecting the material's response to that load (Zhou et al., 2009). Before conducting the experiments, all instrumental calibrations were done following the TA instrument Manual (TA Instruments, 2004).

2.3.1. Stress-relaxation analysis

In a stress relaxation experiment, a constant deformation (strain) is instantaneously applied to the material and the resulting force (stress) is measured as a function of time. In previous literatures, the stress-relaxation tests were mostly conducted by compressing the sample and the experimental data fitted the generalized Maxwell model well (Del Nobile et al., 2007b). However, the bending mode was not employed extensively. Therefore, in this paper the 3-point bending clamp was also applied to conduct relaxation test and determine whether or not the bending results fit the generalized Maxwell model.

In this test, the sample was kept at a constant strain of 1% to make it relax for 5 min in a 3-point bending clamp; while in a compression mode, the sample was kept at a constant strain of 5% for 10 min. During these experiments, the furnace did not work and the temperature inside the furnace was kept at about 23 °C. Both the relaxation tests were replicated three times.

2.3.2. Creep testing analysis

Menard (1999a) mentioned that the repeating of creep-recovery could mimic real-life conditions fairly, thus in this article the creep-recovery cycle was repeated three times to see the mechanical behavior of the sample after fatigue. In a creep test a constant force (stress) is instantaneously applied to the material, and the resulting deformation (strain) is measured as a function of time; after a certain period of time (creep time), the stress is removed and strain recovery of the sample is also measured. In these experiments, the constant stress was set at 0.1 MPa, and the creep time and the recovery time were both made 5 min for the two clamps. Both the creep tests were replicated three times.

2.3.3. Temperature sweep tests

In temperature sweep tests, a dynamic deformation with amplitude of 30 μm was applied on the sweet potato sample in the compression clamp at a frequency of 1 Hz, and then the viscoelastic properties of the samples, such as storage modulus, loss modulus and loss factor (*tan delta*), were measured when the temperature increased from 30 °C to 105 °C. In addition, two heating rates, 2 °C/min and 10 °C/min, were used to determine the effect of different heating rates on the viscoelastic properties of sweet potato. The temperature sweep tests were replicated more than three times for both heating rates.

2.3.4. Frequency sweep tests

The frequency sweep tests were conducted by holding temperature and deformation constantly and scanning across the frequency range of interest. The chewing frequency of human is about 10 Hz, and the vibrating frequency of the agricultural products in the transportation vehicles or the production line varies from about 1 to 50 Hz; thus in this test, the frequency range was set at 50–0.1 Hz, the dynamic deformation of 30 μm was applied in both the 3-point bending and the compression mode. The frequency sweep tests were replicated more than three times for both clamps.

2.4. Data analysis

The results from DMA measurements were analyzed using TA Instruments Universal Analysis 2000 Software Version 4.3A (TA Instruments, USA). The regression analyses of stress-relaxation and creep data were carried out by SPSS software Version 10.0 (SPSS Inc., Chicago, IL, USA). The statistical analyses were done by the SAS® Version 9.0 (SAS Institute Inc., Cary, NC, USA). Analysis of significant variation with the Duncan-test was used to estimate the significant differences among the interested parameters at a probability of 5%.

3. Results and discussion

3.1. Stress-relaxation analysis and modeling

When a constant load was applied to materials, different kinds of relaxation behaviors can be observed with materials of different viscoelastic properties: ideal elastic materials, which do not relax; ideal viscous materials, which show a relaxation instantaneously; viscoelastic solids, which gradually relax and reach an equilibrium stress greater than 0; whereas for viscoelastic fluids, the residual stress vanishes to zero (Menard, 1999a; Steffe, 1992). In this test, the stress of sweet potato sample relaxed gradually and reached to a certain level at last, which illustrated that sweet potato was a viscoelastic material.

The stress relaxation curves of two clamps showed similar trends of relaxation behavior. These curves seemed to consist of two contributions: the first part describes the short-time response of the food matrix and is narrow and high; the second one describes the long-time response of the food matrix and is wide and low.

As mentioned above, the relaxation behavior of polymers can be described by the generalized Maxwell model. Here we applied two models for the regression analysis: the 3-element and 5-element Maxwell model.

For the 3-element model, the equation of modulus against testing time can be expressed as:

$$E(t) = E_1 \exp\left(-\frac{t}{\tau_1}\right) + E_c \tag{1}$$

As for the 5-element model, the expression can be given as:

$$E(t) = E_1 \exp\left(-\frac{t}{\tau_1}\right) + E_2 \exp\left(-\frac{t}{\tau_2}\right) + E_c \tag{2}$$

where, $E(t)$ is the elastic modulus at any time t *(s)*, E_1 and E_2 (MPa) are the elastic modulus for each Maxwell component, τ_1 and τ_2 *(s)* are relaxation times, and E_c (MPa) is the equilibrium elastic modulus. Furthermore the relaxation time has its real physical meaning: when the specimen was imposed with a constant strain, the relaxation time equals to the time that the stress decreased to $1/e$ or 36.8% of the initial stress.

For Eq. (1), there is only one relaxation time (τ_1) in the 3-element model, while the 5-element model contains two relaxation times (τ_1 and τ_2). We have mentioned that the relaxation curve of sweet potato includes two parts: short-time response and long-time response. Therefore we suspect that the 3-element model may not be able to describe the relaxation behavior of the sample as precisely as the 5-element one does.

As it is known, $E = \sigma/\varepsilon$, where σ and ε are the stress and strain of the sample respectively, and E is the modulus. Here in this test, ε = 1% = 0.01, thus $E = 100\sigma$. In this way, $E(t)$ against time data can be obtained to conduct the regression analysis by SPSS software, the parameters of these two models as well as the standard deviations were presented in the Table 1.

From Table 1, we can see that the relaxation times regressed by 3-element Maxwell model are about 27.81 s for the 3-point bending test and 62.47 s the for compression test, while the two groups of relaxation times by the 5-element model were: 4.55 s and 71.95 s, 7.71 s and 230.72 s, respectively. The interrelations among

Table 1
The relaxation parameters for 3-point bending and compression clamps.*

Parameters		E_c (MPa)	E_1 (MPa)	τ_1 (s)	E_2 (MPa)	τ_2 (s)	R^2
3-Point bending	3-Element model	4.09 ± 0.64	9.38 ± 1.14	27.81 ± 4.28			0.953
	5-Element model	9.99 ± 2.37	9.09 ± 1.20	4.55 ± 0.37	2.40 ± 0.28	71.95 ± 4.44	0.998
Compression	3-Element model	0.40 ± 0.01	0.80 ± 0.17	62.47 ± 1.56			0.918
	5-Element model	0.92 ± 0.23	0.73 ± 0.17	7.71 ± 2.06	0.31 ± 0.04	230.72 ± 30.21	0.996

* Values represent the means and standard deviations; $n = 3$.

the two groups of modeling parameters for both clamps are similar, thus we can suppose that the two tests show the same relaxation behaviors. The R^2 of the 5-element model is a little higher than the 3-element model; therefore the relaxation behavior of sweet potato fitted the 5-element model better.

3.2. Creep testing analysis and modeling

Generally considering the creep behaviors of sweet potato by two clamps, the creep deformation curve of the sample consists of three stages: the first stage shows the beginning of elastic deformation in a very short time; the second stage shows the creeping behavior (known as delayed deformation as well), in which the strain rate is declining under the constant loading stress; the third stage shows the strain recovery of the sample after external forces was removed, and quite small amount of strain remained at last.

As mentioned above, the creep behavior of food matrix can be described by the 4-element Burgers model. The creep expression of the Burgers model can be expressed as below:

$$\varepsilon = \frac{\sigma}{E_0} + \frac{\sigma}{E_r}\left(1 - \exp\left(-\frac{t}{\tau_r}\right)\right) + \frac{\sigma}{\eta}t \quad (3)$$

where, σ (MPa) is the constantly applied stress at any time t *(s)*, E_0 and E_r (MPa) the elastic modulus for each spring in the model, η (MPa s) the viscosity of the dashpot component, and τ_r *(s)* the retardation time of the Kelvin component. The retardation time τ_r, similar as the relaxation time in relaxation test, is a measure of the time required for the spring of the Kelvin component to recover to the 63.2% $(1-1/e)$ of the initial strain when retarded by the dashpot. The four parameters of Burgers model for each creep cycle were reported in the Table 2.

Changes in the retardation time τ_r and viscous term η were detected after each cycle for both clamps. The τ_r and η changed slightly in the former two cycles and no significant difference ($p < 0.05$) was observed in parameters between the second and third cycle; the strain curve of each creeping cycle did not change significantly after the first one, which can be explained more objectively and clearly by the regression results in Table 2. The interrelations among the modeling parameters of both clamps are similar, thus we can suppose that the two clamps exhibit the same creep-recovery behaviors.

3.3. Temperature sweep tests

Polymers change both physically and chemically during the heating process: when the temperature is low, materials present glass-like properties and show brittleness; when the temperature is high, a glass-transition behavior is likely to happen, and then materials appear more like rubber and show flexibility (Champion et al., 2000; Le Meste et al., 2002). The storage modulus E', loss modulus E'' and loss factor (*tan delta*) data of sweet potato samples in each heating pattern were presented in Fig. 3.

Fig. 3(a) shows the temperature scanning curve in the compression clamp when the heating rate is set at 2 °C/min. From Fig. 3(a), we can see that E', E'' and *tan delta* changed significantly during the heating process: firstly, the E' and E'' decreased with the increasing of temperature, which is so-called softening in the common sense; E' and E'' stopped decreasing when the temperature reached about 74 °C, and a modulus peak and a transition of *tan delta* appeared in temperature ranges of 83–85 °C and 84–91 °C, respectively. Thus there was a delay between the modulus peak and *tan delta* transition, which was also the mentioned by Zhou et al. (2009). Fig. 3(b) shows the temperature scanning curve of the sample in the compression mode when the heating rate is 10 °C/min. As can be seen in Fig. 3(b), the magnitudes of the E', E'' and *tan delta* all decrease significantly during the entire heating process.

The softening of the material can be explained by the molecular movements with free volume theory. The molecular motion of the polymer chains is immobilized at lower temperature, which makes the material glassy and brittle. When heated it, sufficient heat is supplied to the polymer, molecular motion is increased and molecules can slide against one another, thus the free volume of the chain segment is enlarged and its mobility in various directions also increases. This increased mobility in either side chains or small groups results in a greater compliance (or lower modulus) of material, thus a softening behavior takes place.

The existence of water in food matrix affects the viscoelastic properties of samples when temperature is increased. The main components of sweet potato material are water and starch. Therefore during the heating course, the two are likely to react with each other and the phenomenon of starch gelatinization may take place. The gelatinization temperature of pure sweet potato starch tested by DSC is about 60.8–78.5 °C (Luo, Gao, & Yang, 2004). Since the peak of *tan delta* appeared at about 84–91 °C, this may attribute to the consequence of gelatinization of sweet potato starch.

Table 2
The creep parameters for 3-point bending and compression clamps.#

Parameters		E_0 (MPa)	E_r (MPa)	τ_r (s)	η (MPa s)	R^2
3-Point bending	Cycle 1	26.84 ± 1.01^A	141.26 ± 12.35^A	11.15 ± 0.38^A	77589 ± 6884^A	0.97
	Cycle 2	30.26 ± 1.20^B	168.11 ± 14.37^A	8.23 ± 0.45^B	96539 ± 6636^B	0.97
	Cycle 3	30.51 ± 1.50^B	176.38 ± 15.20^A	7.27 ± 0.88^B	101655 ± 6031^B	0.97
Compression	Cycle 1	1.79 ± 0.10^A	12.32 ± 1.03^A	6.08 ± 0.44^A	4624 ± 176^A	0.98
	Cycle 2	2.11 ± 0.56^B	17.03 ± 0.45^B	5.63 ± 0.58^{AB}	5325 ± 302^B	0.98
	Cycle 3	2.06 ± 0.07^B	17.67 ± 0.58^B	4.75 ± 0.41^B	4934 ± 232^{AB}	0.98

Values represent the means and standard deviations; $n = 3$. Values in a column with different superscripts were significantly different ($p < 0.05$).

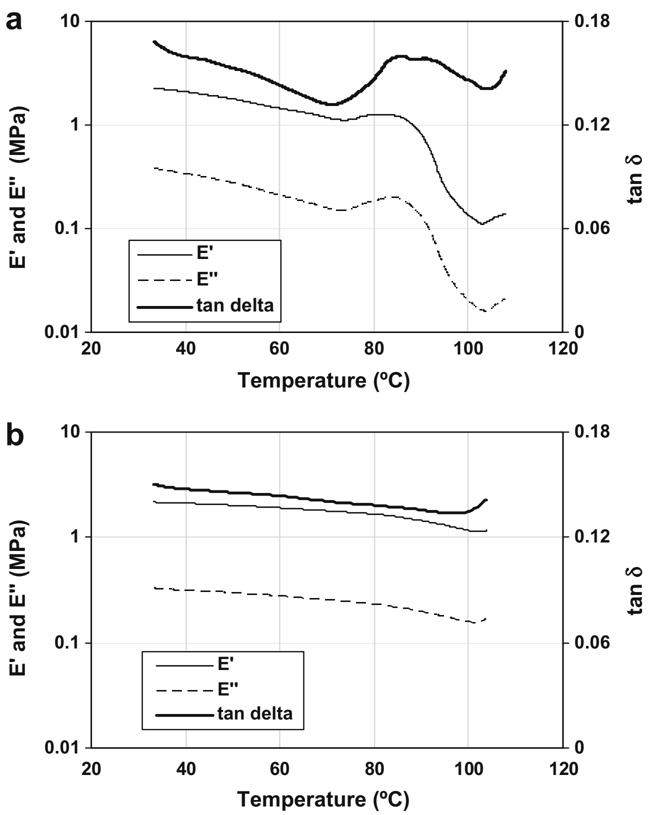

Fig. 3. Temperature scanning curves in different heating-up rates with a constant deformation of 30 μm and loading frequency of 1 Hz in the compression clamp. (a): 2 °C/min, strain: 0.29%; (b): 10 °C/min, strain: 0.27%.

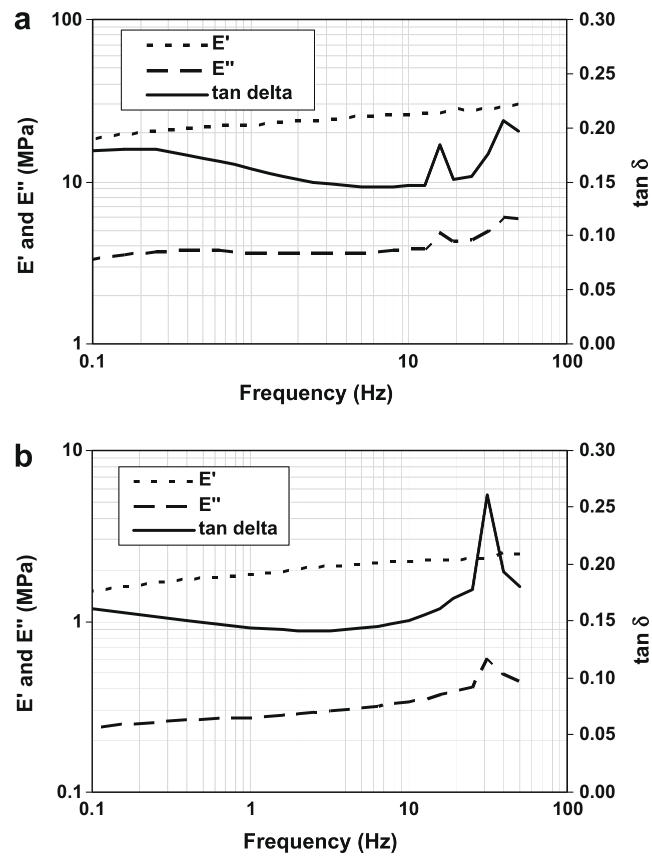

Fig. 4. Frequency scanning curves. (a): 3-point bending mode with a constant deformation of 30 μm at ambient temperature; (b): compression mode with a constant strain of 0.27% at ambient temperature.

However, there is an offset between the two temperature ranges. Apart from starch and water, the sweet potato also contains other components, such as proteins, fibers, vitamins, etc.; we suppose that the coexistence of these components may shift the gelatinization temperature to some extent.

Comparing these two temperature scanning curves, a significant difference can be observed: there is a remarkable turning point in the former (2 °C/min, Fig. 3(a)) curve which is not existed in the later one (10 °C/min, Fig. 3(b)). The experimental conditions in both tests were exactly the same except for the heating rate, so we could conclude that: if the temperature rises too fast, there is not enough time for the structural transition, thus one is not likely to see the thermal-reaction changes inside the material but the original textural information of the tested sample during the heating process.

3.4. Frequency sweep tests

The mechanical properties of food matrix changed textually with the loading frequency, which was called the frequency dependence as well. The E', E'' and *tan delta* data as a function of frequency (in logarithmic plotting) were represented in Fig. 4.

The frequency sweep curves of the sweet potato sample showed similar profiles in different deforming modes. As can be seen in Fig. 4, both E' and E'' decreased as the frequency decreased; *tan delta* decreased sharply when the frequency was decreasing above some certain frequency (turning frequency), and then increased when the frequency was lower than that; and noticeable cuspidal peaks appeared in both E'' and *tan delta* profiles at the same frequency. The turning frequency of *tan delta* was 5 Hz in 3-point bending mode and 1 Hz in the compression mode; the cuspidal peaks of E'' and *tan delta* was 15.67 Hz in the 3-point bending clamps (Fig. 4(a)) and 31.66 Hz in the compression clamp (Fig. 4(b)), thus it is probable that the 15.67 Hz and 31.66 Hz are either the natural resonance frequencies of the sample or one of its harmonics (Menard, 1999c) in 3-point bending mode and compression mode, respectively. And it should be noted that the frequency dependence of sweet potato is closely related to the deforming method and may be dependent on the size of the sample too.

As a conclusion, both E' and E'' of the sweet potato samples decreased as the frequency decreased, and the elastic characteristic dominated significantly during the entire frequency range for the *tan delta* was much less than 1. In the low frequency range, the materials had the time to relax and respond and exhibited comparatively soft and low modulus; while in the high frequency range the materials did not have the time to relax and exhibited solid and high modulus. Comparing to the effect of temperature on the viscoelastic behavior of sample, frequency may have a reverse effect; that is, the effect of increasing in temperature equals to that of decreasing in frequency. A resonance frequency of the sample was detected at 15.67 Hz in the 3-point bending mode and 31.66 Hz in the compression mode in this test.

4. Conclusions

The sweet potato, which contains water, carbohydrates, protein, fiber and some other components, exhibits viscoelastic behavior. The 3-point bending and compression deforming modes showed the similar relaxation and creep behaviors for the sweet potato samples. The regression results of relaxation tests in 3-point bending clamp showed that the relaxation behavior of the sweet potato can be represented well using 5-element Maxwell model. The creep-recovery tests revealed that 4-element Burgers model fitted experimental data of both clamps well, and changes in creep parameters were not significant after the first creeping cycle. The gelatinization of sweet potato starch was detected in the temperature range of about 84–91 °C when the temperature increased at 2 °C/min, and it is possible that one is not likely to see the thermo-chemical or structural change but the softening behavior if the temperature changes comparatively fast. In the low frequency range, the materials had the time to relax and response, thus exhibited low modulus; while in the high frequency range the materials did not have the time to relax and exhibited comparatively high modulus. All of the information above may provide a rough theoretical guide to reduce the injury of sweet potatoes in the following transport, storage or processing procedures.

Acknowledgements

Supported by Program for New Century Excellent Talents in University of China (NCET-08-0537), National Natural Science Foundation of China (30800662), Science and Technology Support Project of China (2009BADA0B03), and High Technology Research and Development Program of China (2006AA10256-02).

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