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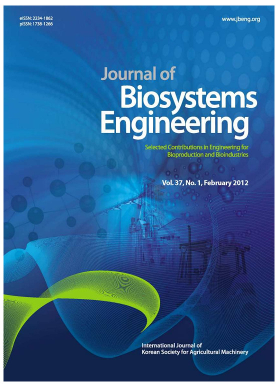

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Measurement of Dynamic Compressive Properties of Apples using the Oscillatory Test

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Abstract

Purpose: This study performed the oscillatory test using the texture analyzer to characterize the viscoelastic behavior of apples such as the storage modulus (E'), the loss modulus (E''), the complex modulus (|E*|) and the energy dissipated per cycle (W_{diss}). **Methods:** The sinusoidal deformation with the frequency of 1~10 Hz and the maximum displacement of 0.1 mm were applied to the flesh tissues of Fuji, Golden Delicious and Red Delicious apples. The Lissajous figure was used to measure the phase angle(δ) between stress and strain curve. **Results:** Trigger force was critical to the measurement of the phase angle. E', E'', |E*| and Wdiss were measured using the Lissajous figure and the phase angle. The complex modulus of Golden Delicious apple was significantly lower than those of Fuji apple and Red Delicious apple. **Conclusions:** Apple flesh was exhibiting more elasticity at low frequency, and more viscosity at high frequency. Dynamic compressive properties of Fuji apple were similar to those of Red Delicious apple but significantly different from those of Golden Delicious apple.

Keywords: Dynamic mechanical test, Loss modulus, Phase angle, Storage modulus, Texture analyzer

Introduction

Mechanical properties of fruits affect the consumer's sensory perception and the design of processing equipment. Once the range of sensory acceptability is determined, fruit processing equipment are selected and controlled enough to distinguish between an acceptable and unacceptable product, based on a texture analysis.

As fruits have viscoelastic attributes, it is important to analyze elastic components and viscous components. There are several tests to obtain these parameters such as compression test, tension test, shear test, bending test, dynamic mechanical test and so on. A simple compression test gives mechanical properties such as bioyield strength and strain (bioyield force and deformation), ultimate strength and strain (rupture force and deformation), apparent elastic modulus (tangent modulus, secant modulus or initial tangent modulus), resilience, toughness, etc. A hysteresis test is used to measure loss work, hysteresis loss and degree of elasticity. These studies were not concerned in dynamic tests for identifying viscoelastic parameters (Kim et al., 1992; Kim et al,, 1999; Kim, 2000; Kim, et al., 2003).

Viscoelastic materials have a relationship between stress and strain which depends on time or frequency. The oscillatory compression test is one of dynamic mechanical tests, and is useful in evaluating dynamic compressive properties such as the storage modulus (E'), the loss modulus (E''), the complex modulus ($|E^*|$), the tangent δ, and the dissipated energy (W_{diss}), etc. The complex (elastic) modulus, expressed by $E^* = E' + jE''$ where $j^2 = -1$, contains sufficient information about the viscoelastic characteristics of the biological tissues (Zhang, 2005).

Dynamic testing instrument may be divided into two general categories: controlled rate instruments where the deformation (strain) is controlled and stress is measured, and controlled stress instruments where the

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stress amplitude is fixed and stress is measured. Both produce similar results (Steffe, 1996).

Oscillatory test is referred to as small-amplitude oscillatory test because small deformations must be employed to maintain linear viscoelastic behavior (Steffe, 1996; Gunasekaran, 2001). Small-amplitude deformation tests, generally involving strains of less than 3% (1 μm ~ 10 mm), permit evaluation of microstructural changes at the cellular level and facilitate characterizing time-dependent or viscoelastic properties. The small-amplitude oscillatory test is a nondestructive technique, based on sinusoidal strain and stress data. The test, in principle, applied a sinusoidal stress to measure the strain produces the same material properties within the linear viscoelastic region (Gunasekaran, 2001).

In a dynamic experiment a sinusoidal strain is applied to a material and the resulting sinusoidal stress is measured, or vice versa. The resulting stress has the same frequency as the applied strain, but its phase is different because of relaxation mechanisms associated with the material. The viscoelastic properties of the material can be therefore obtained by measuring the phase shift (δ) of the stress. The phase shift shows whether a material is viscously or elastically dominated.

This study focused on evaluating viscoelastic behavior of apples using the texture analyzer. The oscillatory test was performed to assess dynamic compressive properties of the apple flesh, such as the phase angle, the storage modulus, the loss modulus, the complex modulus and the dissipated work. This paper described how to calculate the phase angle between stress and strain curve because it was essential in investigation of dynamic mechanical properties.

Materials and Methods

Materials

Fruit samples were selected from three varieties of Fuji apple, Golden Delicious apple and Red Delicious apple for the dynamic compression testing. Samples were purchased at market places near the University of Missouri, USA and then kept in laboratory conditions of about 20℃ and 60% RH for 6~12 hours before tests were conducted.

Apple flesh slices, 16~18 mm in thickness, were removed from a whole apple in an axial direction using specially designed slicing apparatus, and then the specimen sampler was used to make cylindrical flesh tissues of Φ19.5 mm and L13.5 mm in a perpendicular direction of the flesh slice. Length and diameter of a cylindrical sample were measured by a standard digital caliper (resolution: 0.01 mm, model: EC16, Tresna, USA), and weight was measured by an electric microbalance of platform type (resolution: 1 μg, max. scale: 10 g). A density of specimen was calculated from specimen sizes and weight as shown in Table 1. The test used ninety apples (three varieties × thirty apples) and each apple has 2 replicates in the test.

Experimental setup

The compression test is considered as a traditional method for measuring mechanical properties of foods and agricultural products. When a mechanical testing is done using universal testing machines like *Instron* and *Texture Analyzer*, the shape, sizes and head speed of a probe as well as the shape and sizes of fruit sample should be described clearly (Kim et al., 1999; Kim, 2000; Kim et al., 2003). This study performed the oscillatory test using Texture Analyzer (TA+D*i*, Texture Technologies Corp, New York, USA) with a top load cell of 490 N (50 kg_f).

Table 2 shows crosshead speed and sampling rate of the oscillatory test. The probe of the texture analyzer had a flat surface tip and its diameter was 2.54 cm. The loading rate of the crosshead was adjusted according to the experimental frequency level of 1 Hz to 10 Hz. Sampling rate is restricted by a memory size of the texture analyzer. Head speed of the probe was from 2.5 mm/min to 25.0 mm/min, within the range of loading rate specified by the

Table 1. Physical properties of the samples for the oscillatory test

Variety of apples[1]	Item	Weight (g)	Length (mm)	Diameter (mm)	Density[2] (kg/m^3)
Fuji	Mean	3.49	13.6	19.6	852.7
	(SD)	(0.12)	(0.37)	(0.07)	(19.5)
Golden delicious	Mean	3.37	13.7	19.5	825.3
	(SD)	(0.08)	(0.25)	(0.12)	(17.6)
Red delicious	Mean	3.36	13.6	19.5	829.9
	(SD)	(0.15)	(0.34)	(0.16)	(29.0)

[1] n = 30 for each variety, [2] Density = Weight / (π/4 × Diameter^2 × Length) / 10^6

ASAE Standards 2002. Sampling rates depended on the frequency such as 400 points/sec for 1 Hz and 40 points/sec for 10 Hz. It means the higher frequency, the lower sampling rate and the lower resolution in time domain.

The texture analyzer set to travel to a target distance of ±0.1 mm, and it started to record the force response of the sample to the deformation imposed, from trigger point. The trigger force was 0.1 N and critical to the measurements as discussed later.

Maximum value of the sinusoidal deformation was 0.1 mm, less than strain of 0.8%. A strain of 0.8% is close to a level needed to bruise apple tissue. It is known that tissue failure occurs at around 1% strain, and tissue damage occurs at about 0.1% strain (Pitts et al., 1997). While working with the sinusoidal deformation, the probe moved in contact with the sample tissue during one half of a sinusoidal cycle, and then traveled off the apple tissue for the other half cycle.

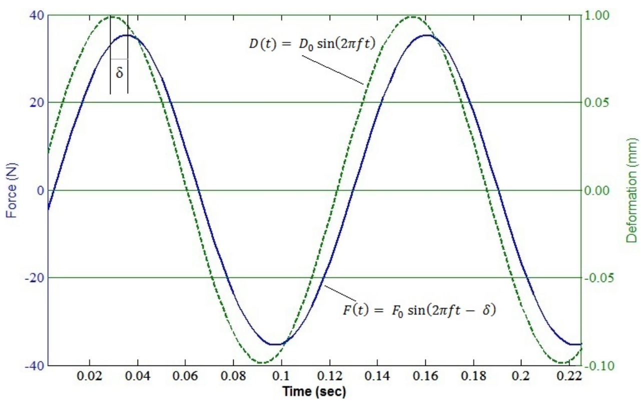

Figure 1. Typical sinusoidal curves of the force (normalized) and the deformation in time domain. δ is the phase angle. f is the frequency, and D_0 and F_0 are amplitudes of deformation and force, respectively.

In Figure 1, δ is the phase difference in time domain between force and deformation curves and it is very important parameter in the dynamic mechanical analysis as described later.

Dynamic mechanical parameters

Force and deformation values in time domain were converted to comma-separated values (CSV) file to be easily readable in Matlab Ver. 7 (MathWorks, Inc., USA) and Microsoft Office Excel 2007. Programs were developed with Matlab for processing the data and analyzing dynamic mechanical parameters.

The time duration (period, T) between a maximum point and the next maximum point in the cyclic curve of force and deformation is used to calculate the frequency of the sinusoidal curve by $f = 2\pi/T$. Frequency of force curve is same to that of deformation curve, if the material is linear viscoelastic.

The oscillatory test is based on sinusoidal strain and stress data within the linear viscoelastic region. Strain level of less than 3% is considered acceptable to assure linearity (Gunasekaran, 2001). Maximum deformation of this experiment was 0.1 mm that is less than strain of 0.8%.

The phase angle can be determined directly by the time difference between the strain curve and the closest stress curve. The problem is a resolution of the phase angle. For example, 1 Hz signal had 400 data points with resolution of 0.9°/point while a resolution of 10 Hz signal was 9.0°/point. The higher frequency of signals, the less a resolution of the phase angle was. This study obtained the phase difference using the Lissajous figure. In the Lissajous

Table 2. Specifications of the oscillatory test

Test order	Frequency		Average head speed[1) (mm/min)	Sampling rate		Period of half cycle[2) (sec)
	Hz	rad/sec		point/cycle	degree/point	
1	1	6.3	2.5	400	0.90	0.500
2	2	12.6	5.0	200	1.80	0.250
3	3	18.8	7.5	133	2.71	0.167
4	4	25.1	10.0	100	3.60	0.125
5	6	37.7	15.0	66	5.45	0.083
6	8	50.3	20.0	50	7.20	0.063
7	10	62.8	25.0	40	9.00	0.050

1) Crosshead speed varied with the sinusoidal deformation in time domain.
2) The probe was in contact with the sample for one half cycle of the sinusoidal deformation, whereas it moved off the sample during the other half cycle.

figure, a resolution of the phase angle is a little less sensitive to variation of signals rather than the method using time difference measurement (Lake, 2004). A graph of stress versus strain data is ellipse in shape for a linearly viscoelastic material, as illustrated in Figure 2. I In the Lissajous figure, the forward movement from -0.1 mm to +0.1 mm plotted an upper half ellipse of the diagonal, whereas the backward movement from +0.1 mm to -0.1 mm plotted a lower half ellipse. Points *A*, *A** and *F* in Figure 2 might not be in experimental data. This study determined these points using the linear fitting with data points neighboring to zero stress and zero strain.

From the Lissajous figure, the phase angle (δ), the storage modulus (*E*'), the loss modulus (*E*'') and the complex elastic modulus (|*E**|) are given by equations (1)~(4), respectively.

$$\delta = sin^{-1}(A/C) \quad (1)$$

Where, *A* : The vertical thickness of the ellipse

C : The full height (ε_{max}) of the Lissajous figure

$$E' = E/C \quad (2)$$

$$E'' = F/C \quad (3)$$

$$|E^*| = D/C = \sigma_{max} / \varepsilon_{max} \quad (4)$$

Where, *D* : The full width (σ_{max}) of the Lissajous figure

E : The horizontal thickness for the full height (ε_{max}) of the Lissajous figure

F : The horizontal width of the ellipse

Using the magnitude of the complex elastic modulus

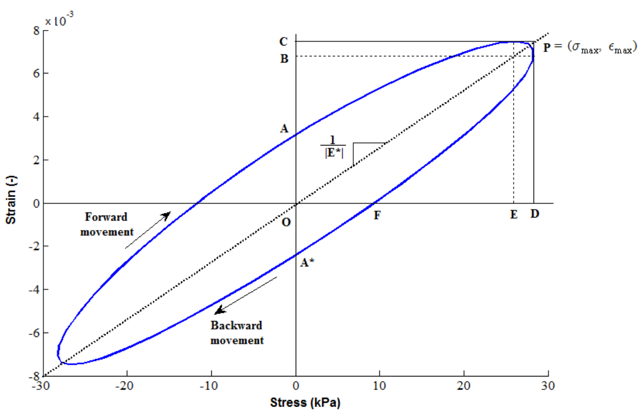

Figure 2. Lissajous figure due to stress (σ) response to strain (ε) that is sinusoidal in time. ($\overline{OA} = \varepsilon_{max} \sin \delta$, $\overline{OB} = \varepsilon_{max} \cos \delta$, $\overline{OC} = \varepsilon_{max}$, $\overline{OD} = \sigma_{max}$, $\overline{OE} = E' \varepsilon_{max}$, $\overline{OF} = E'' \varepsilon_{max}$).

defined by equation (4), the storage modulus and the loss modulus are determined by equations (5) and (6). Mathematically, equations (5) and (6) are equal to equations (2) and (3), respectively.

$$E' = |E^*| \cos \delta \quad (5)$$

$$E'' = |E^*| \sin \delta \quad (6)$$

The energy dissipated (W_{diss}) during cyclic loading and unloading on the apple flesh was corresponding to the area enclosed by *A-P-F-A**-*A*.

Results and Discussion

Phase angle

Figure 3 indicates that a stress curve moved to the right against a strain curve along with the frequency. Figure 4 shows the width and area of the ellipse increased as the frequency increased. It results in increasing the vertical thickness of the ellipse, $\overline{OA}$ in Figure 2. It is obvious from two figures that the phase angle increased with increasing the frequency.

Figure 5 shows averaged values and standard deviations of phase differences between the strain and stress data of apple fleshes (*n* = 30 for each variety) in the range of 1 Hz to 10 Hz. Phase differences had the lowest value at 2 Hz for Fuji apple and Red Delicious apple and 3 Hz for Golden Delicious apple.

When the cause (the strain) applied to measure the response of the effect (the stress), the effect generally lags behind the cause. It means that the stress curve lags the strain curve. Nevertheless, the stress leaded the strain at the low frequency of 1 Hz or 2 Hz, as illustrated in Figures 3 and 5. This study suggested this behavior was related to the trigger force. The trigger force in this experiment was 0.1 N, corresponding to 1.5% of maximum forces that was the mean of all observations. This trigger value might be a little high. High trigger force induces underestimation of strain because it delays to start to record the force and deformation data. Even after the probe is already in contact with the surface of a material, deformation data yet is recording negative values while the force data is already recording positive values. It results that the effect (stress) leads the cause (strain). Conversely, premature trigger force causes the probe to oscillate up to shallow

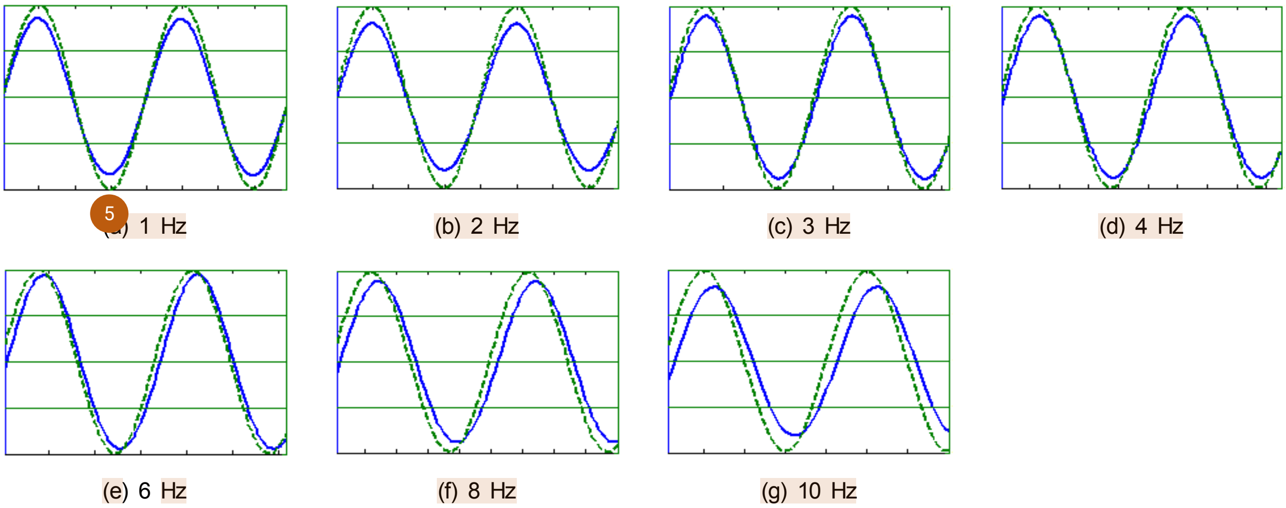

Figure 3. Plots of strain (dashed) and normalized stress (solid) data in time domain (x-axis) according to the frequency.

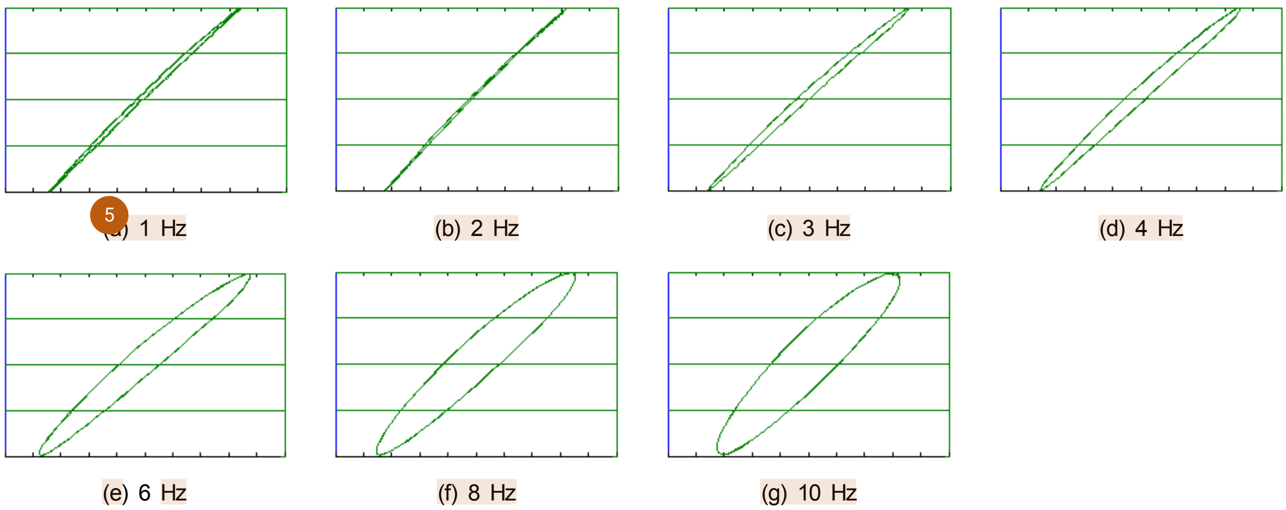

Figure 4. Trend of Lissajous figures with stress (x-axis) and strain (y-axis) according to the frequency. A plot at each frequency corresponds to a stress-strain curve in Fig. 3.

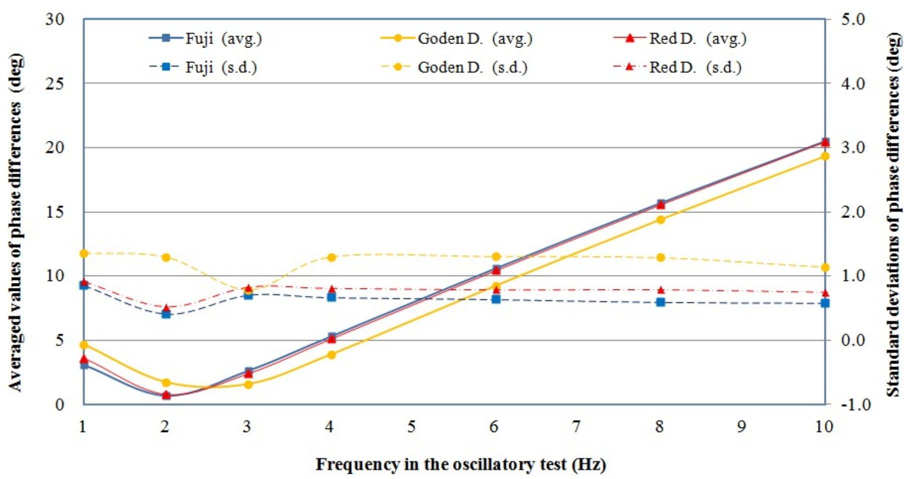

Figure 5. Averaged values (avg.) and standard deviations (s.d.) of phase differences between the strain and stress data of apple fleshes according to the frequency.

depth inside a material or at different initial loading distances.

As well as the setting value of trigger force, variations of the height of the cylindrical specimens might also affect the trigger point. It implied to need a profound investigation about the experimental data at frequencies of 1 Hz and 2 Hz.

Golden Delicious apples had slightly smaller averaged values and larger standard deviations in phase lags than those of other apples, as shown in Figure 5, but there was no statistical differences among the apple varieties at p = 0.05 from Duncan's multiple range tests.

Storage, loss and complex moduli of apple flesh

Figure 6 shows the storage modulus and the loss modulus of the apple flesh according to the frequency. The storage modulus of apple flesh ranged 3.55~3.75 MPa for Fuji and Red Delicious apples and 2.91~3.02 MPa for Golden Delicious apple. The storage modulus of apple flesh by this test was much higher than the apparent elastic modulus reported at some papers (Kim et al., 1999; Kim, 2000; Kim et al., 2003). It means that the storage modulus measured by the small-deformation test was based on a different approach, comparing with the simple compression test.

As the phase angle increased with increasing the frequency, the loss modulus increased consequently with the increase of the frequency. As shown in Figure 6, the loss modulus of the apple flesh was more prevalent at high frequency than at low frequency, whereas the storage modulus was almost same at the frequency of 1~10 Hz.

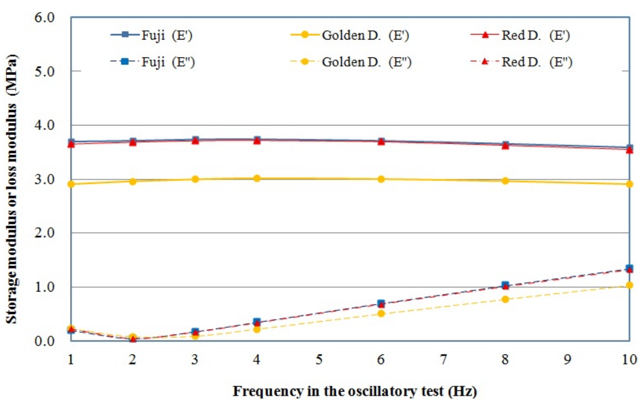

Figure 6. Averaged values of storage modulus (E, solid line) and loss modulus (E', dashed line) of apple fleshes according to the frequency.

They mean that the apple flesh was exhibiting more elasticity at low frequencies, and more viscosity at high frequencies. Therefore the apple tissues may follow the Kelvin-Voigt model which has a spring in parallel with a dashpot (Mohsenin, 1986). If a loss modulus increases to be high at short period, a material is apt to failure with fast contact rather than slow contact, even with a same contact force.

Table 3 shows the complex modulus of apple tissues according to the frequency obtained by equation (4), and results of Duncan's multiple range tests. The complex modulus of Golden Delicious apple was highly significant lower than Fuji apple and Red Delicious apple. Variations of the complex modulus were almost same at the frequency of 1~10 Hz. By definition of the complex modulus, variations of the complex modulus results from variations of the storage modulus and the loss modulus.

Table 3. Complex modulus of the apple flesh obtained by equation (4)

Frequency	Mean of the complex modulus (MPa)		
	Fuji	Golden Delicious	Red Delicious
1 Hz	3.708 (0.5468) a	2.915 (0.7247) b	3.655 (0.8210) a
2 Hz	3.717 (0.5295) a	2.963 (0.7356) b	3.682 (0.8320) a
3 Hz	3.747 (0.5327) a	2.998 (0.7325) b	3.710 (0.8383) a
4 Hz	3.763 (0.5281) a	3.023 (0.7367) b	3.728 (0.8366) a
6 Hz	3.781 (0.5256) a	3.044 (0.7449) b	3.754 (0.8503) a
8 Hz	3.797 (0.5299) a	3.063 (0.7534) b	3.764 (0.8664) a
10 Hz	3.826 (0.5354) a	3.084 (0.7742) b	3.790 (0.8812) a

1) Numbers in parenthesis : standard deviation (n = 30)
2) Same letters in a row : not significant at p = 0.01

Dissipated energy of apple flesh

Table 4 shows the dissipated energy of apple tissues according to the frequency and results of Duncan's multiple range tests. The dissipated energy of apple tissues increased with increasing the frequency. The dissipated energy of Golden Delicious apple was lower than those of Fuji and Red Delicious apples, as the loss modulus.

The dissipated energy is supposed to be small because the maximum deformation in the oscillatory test was only 0.1 mm, while the probe was loading and unloading on the cylindrical apple tissues. Dissipated works measured by the simple compression test (Kim et al., 1992; Kim et al., 1999) were roughly 2~4 kJ/m^3 at the deformation of 1.5 mm (7.5% strain). There was a great difference in the dissipated energy between the oscillatory test and the simple compression test. It means that two tests were based on different approaches. Further study is expected to investigate the comparison of mechanical properties between the oscillatory test and the simple compression test.

Table 4. Dissipated energy while the probe was working on the apple flesh

Frequency	Mean of the dissipated energy (J/m^3)		
	Fuji	Golden Delicious	Red Delicious
1 Hz	10.3 (3.03) a	11.3 (1.97) a	11.4 (3.11) a
2 Hz	3.4 (1.38) a	4.1 (2.68) a	3.2 (1.58) a
3 Hz	16.5 (3.95) a	8.5 (4.85) b	15.3 (5.58) a
4 Hz	31.4 (5.87) a	19.9 (7.91) b	30.2 (8.30) a
6 Hz	60.8 (10.56) a	43.2 (13.31) b	59.2 (14.06) a
8 Hz	88.9 (15.08) a	65.8 (18.67) b	87.1 (20.18) a
10 Hz	115.0 (19.75) a	86.8 (23.75) b	113.1 (25.86) a

[1)] Numbers in parenthesis : standard deviation (n = 30)
[2)] Same letters in a row : not significant at p = 0.01

For viscoelastic materials, the dissipated energy is closely related to the loss modulus. The linear regression model was developed as equation (7), regardless of the frequency and the apple variety (R^2 = 0.9882).

$$W_{diss} = 0.08626\,E'' - 1.0533 \qquad (7)$$

Where, W_{diss} : Dissipated energy per cycle (J/m^3)
E'' : Loss modulus of apple tissues (kPa)

Conclusions

This study performed the oscillatory test using the texture analyzer with the frequency of 1~10 Hz and maximum deformation of 0.1 mm to measure the dynamic compressive properties of apples.

The phase angle between stress and strain curve was essential in the oscillatory test for investigation of viscoelastic properties of a material. This paper described the method using the Lissajous figure of the experimental stress-strain data to calculate the phase angle. Trigger force in collecting force-deformation data was critical to the measurement of the phase lag (δ).

The storage modulus (E'), the loss modulus (E''), the complex modulus (|E*|) and the energy dissipated per cycle (W_{diss}) were measured using the Lissajous figure and the phase angle.

The storage modulus of each apple flesh was almost same at the frequency of 1~10 Hz, with ranges of 3.55~3.75 MPa for Fuji and Red Delicious apples and 2.91~3.02 MPa for Golden Delicious apple.

The loss modulus of the apple flesh was more prevalent at high frequency than at low frequency. The complex modulus of Golden Delicious apple was highly significant lower than Fuji apple and Red Delicious apple. The apple flesh tissue was exhibiting more elasticity at low frequencies, and more viscosity at high frequencies. It means that the apple tissues may follow to the Kelvin-Voigt model which has a spring in parallel with a dashpot.

The energy dissipated by cyclic loading and unloading on the apple flesh was closely related to the loss modulus with the correlation coefficient of R = 0.9941.

Dynamic compressive properties of Fuji apple were similar to those of Red Delicious apple but significantly different from those of Golden Delicious apple.

The further study is expected to compare a simple compression test and a hysteresis test with the oscillatory test in evaluation of the mechanical parameters.

Conflict of Interest

No potential conflict of interest relevant to this article was reported.

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