Effects of Temperature and Loading Characteristics on Mechanical and Stress-Relaxation Behavior of Sea Buckthorn Berries. Part 3. Relaxation Behavior

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ABSTRACT

Relaxation force properties of single sea buckthorn berries under uni-axial compression were studied. Sea buckthorn berries had a time dependent behavior similar to other viscoelastic materials. The three-term Maxwell model with a maximum relative difference (MRD) of $\leq 5\%$ was chosen as the best fit equation to the relaxation data. For both Indian Summer and Sinensis cultivars, the firmness (as evidenced by the decay forces) decreased with temperature. As berry temperature increased from 4.5 to 34.5°C, F_1 decreased 32.9 and 19.9% for the Indian Summer and Sinensis cultivars, respectively. Sinensis berries were significantly firmer than Indian Summer berries. For each cultivar, a master curve was proposed to determine the relaxation data of the sea buckthorn berries at temperatures between 4.5 and 34.5°C. Deformation level and loading velocity had a decreasing effect on relaxation time, but temperature had an increasing effect.

Keywords: *Sea buckthorn berry, Relaxation force, Rheology.*

INTRODUCTION

Sea buckthorn berries *(Hippophae rhamnoides* L.), like other fruits, are subjected to a series of static and dynamic loads during harvesting, handling, transport, processing, and storage. Such loadings cause damage which decreases quality and increases susceptibility to deterioration during storage. Mechanical damage also causes the fruit to lose large amounts of biologically valuable substances (Blahovec et al. 1995). The nature and extent of damage depends on several mechanical and rheological characteristics of the fruit together with the forces, or loading condition, to which the fruits are subjected.

It is, therefore, important to study mechanical and viscoelastic properties of the fruit such as force relaxation and creep. When basic data are known, processing and handling equipment can be designed for maximum mechanical efficiency and highest quality of the final products.

One of the most important characteristics of fruit is stress (force) relaxation. The stress relaxation determines the elastic and viscous properties of the fruit cell walls (Sakurai and Nevins 1992). The results of the relaxation test are useful for estimating susceptibility to damage, since they measure the rate at which material dissipates stress after being subjected to a sudden force. A slow stress dissipation may damage the fruit during a long period of load application (Sarig and Orlovski 1974).

Although there is a large published work on the viscoelastic properties of grains, fruits and vegetables, information on the force relaxation behavior of the sea buckthorn berry is not available. The objectives of the present work, therefore, were to evaluate the effects of berry temperature, deformation level, and loading velocity on relaxation behavior of sea buckthorn berries.

MATHEMATICAL MODELS

A generalized Maxwell model with 2 or 3 elements is generally used to represent stress relaxation data. In this model, applied force (F(t)) can be used instead of stress. Thus, the force relaxation behavior can be represented by an exponential equation as given below (Sarig and Orlovsky 1974; Husain et al. 1971):

$$F(t) = \sum_{i=1}^{n} F_i (e^{-t/\tau_i}) \qquad \text{(Eq.1)}$$

where τ_1, $\tau_2 \ldots \tau_n$ are the relaxation time constants corresponding to various elements in the Maxwell model, F_1 to F_n are the decay forces, and F(t) is the instantaneous force. The instantaneous force could be replaced by any other decaying parameter such as stress or modulus of elasticity (Pappas and Rao 1989; Waananen and Okos 1992). The Maxwell model is widely used to represent relaxation data. For many foods, three terms involving six constants are sufficient.

Different methods have been used to determine the number of Maxwell elements sufficient to represent the force relaxation behavior of materials. One of the most widely used methods is the successive residual method as described by Mohsenin (1986). Some researchers used the non-linear regression procedure provided in different statistical packages (Bargale and Irudayaraj 1995; Bargale et al. 1994; Sarig 1974; Pappas and Rao 1989).

Research has shown that the effect of a change in temperature on the viscoelastic properties of many materials is similar to the effect of change in the real time scale (Waananen and Okos 1992). In other words, data measured for short periods at several different temperatures can be combined on a single curve (i.e., all relaxation curves can be shifted to a reference curve) that is equivalent to data measured at a fixed temperature over an extended period (Waananen and Okos 1992).

To illustrate the meaning of shifting the curves, let $F_{To}(t)$ be the relaxation function at some reference temperature T_o and $F_T(t)$ be the relaxation function at temperature T. As proposed by several researchers (Balastreire et al. 1978; Cumming and Okos 1983; and Waananen and Okos 1992), one can write:

$$F_{T_\circ}(\log t) = F_T\left[\log_{10} t + f(T)\right] \qquad (Eq.2)$$

where T_o is the reference temperature and f(T) is a shift function (in the logarithmic scale). Assuming that $f(T) = \log_{10} a_T(T)$, then:

$$F_{T_\circ}(\log t) = F_T\left[\log_{10} t + \log_{10} a_T\right] = F_T\,[\log_{10}(t \cdot a_T)] \qquad (Eq.3)$$

Therefore:

$$F_{T_\circ}(t) = F_T(t \cdot a_T) \qquad (Eq.4)$$

The quantity $t{\cdot}a_T$ is called the reduced time or pseudo time (Balastreire et al. 1978). Hence, it has been shown that t units of time at temperature T_o are equivalent to $t{\cdot}a_T$ units of time at temperature T. The shift factor a_T is an inherent property of a given viscoelastic material and must be determined experimentally (Herum et al. 1979). For $T < T_0$, $a_T < 1$; for $T > T_o$, $a_T > 1$; and for $T = T_o$, $a_T = 1$.

By applying the shifting procedure as described above to a series of curves measured at different temperatures, a single “master curve” may be obtained. Thus, the relaxation force at any temperature, T, can be represented by the force at a reference temperature, T_o, over an extended time scale.

MATERIALS and METHODS

Sea buckthorn berries of the cultivars Indian Summer and Sinensis were used in this study. Physical characteristics of the berries are described in Part 1 of this series (Khazaei and Mann 2004). Berries were refrigerated at 6.5°C from the time of harvesting until the time of testing. Indian Summer berries ranged from 7.6 to 8.1 mm (intermediate diameter). “Small” Sinensis berries ranged from 6.8 to 7.2 mm (intermediate diameter) and “large” Sinensis berries ranged from 7.9 to 8.4 mm (intermediate diameter).

Relaxation tests were completed using a texture testing machine equipped with a 250 N load cell and having a precision of 0.001 N. Three sets of relaxation tests were conducted: a) to determine the effect of berry temperature on relaxation behavior, b) to determine the effect of initial deformation on relaxation behavior, and c) to determine the effect of loading velocity on relaxation behavior.

The effect of berry temperature at 4.5, 16.5, and 34.5°C was studied for both Indian Summer and small Sinensis berries. The loading velocity and initial deformation were constant at 0.7 mm/s and 10%, respectively. Each test was replicated 20 times. Berry temperature was modified by immersing the berries in water baths of 4.5, 16.5, and 34.5°C for 15-20 min. Individual berries were removed, drained, and immediately tested.

The effect of initial deformation at 10, 18, and 25% was studied for large berries of the Sinensis cultivar. Tests were conducted at a temperature of 16.5°C and a loading velocity of 0.7 mm/s. Each test was replicated 20 times.

The effect of loading velocity at 0.1, 0.3, and 0.7 mm/s was studied for sea buckthorn berries of the Indian Summer cultivar. These tests were completed at a temperature of 4.5°C and a deformation level of 10%. Each test was replicated 20 times.

For each relaxation test, a berry was placed on its cheek on a flat steel washer in the center of a loadcell, and then compressed between parallel plates with a given loading velocity and a predetermined deformation. Relaxation data were collected for 40 s. Because the actual surface area under an applied force continually changes with compression of intact berries, true stress values are not known. Therefore, force relaxation was evaluated instead of true stress relaxation. The time dependent force data obtained from the relaxation experiments were used to calculate the parameters of the Maxwell model (SIGMAPLOT statistical software was used).

The number of terms in the Maxwell model was selected based on the maximum relative difference (MRD) between the model-predicted values and the experimentally measured values (Bargale et al. 1994; Al-Mashat and Zurit 1993). The model with the number of terms corresponding to an MRD value of $\leq 5\%$ was chosen as the best fit model. The MRD value was calculated based on:

$$\text{MRD} = \max\left\|\left[\frac{\text{measured} - \text{calculated}}{\text{measured}}\right]\right\| \times 100 \qquad \text{(Eq.5)}$$

R^2 values were also calculated for each model. Finally, the relaxation curves were converted to a single master curve by computing the temperature shift factors (a_T) using 16.5°C as the reference temperature. Shift factors were not calculated for the effect of initial deformation or loading velocity.

RESULTS and DISCUSSION

Effect of Temperature on Relaxation Behavior

Relaxation force curves for sea buckthorn berries at different temperatures are shown in Fig. 1. Sea buckthorn berries behave in a similar manner to other viscoelastic materials, therefore, similar mathematical techniques may be employed to define the behavior of the sea buckthorn berry under the influence of external loads.

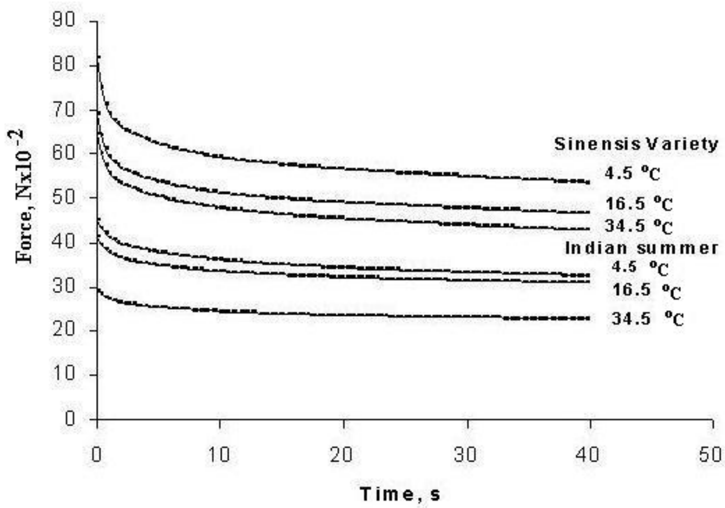

Fig. 1. Effect of berry temperature on relaxation behavior of sea buckthorn berries.

The experimental data were fit to Maxwell models of one, two, or three terms. The MRD values decreased with an increase in number of terms (Table 1). For the one-term model, the MRD values ranged between 18.6 and 27.4%; for the two-term model, the MRD values ranged between 8.3 and 15.5%. The three-term model had MRD values below 2% in all cases with a maximum value of 1.7%. Furthermore, a comparison based on R^2 values revealed that the three-term Maxwell model fit the data the best. Therefore, the three-term Maxwell model with a MRD of $\leq 5\%$ and R^2 higher than 0.990 was chosen as the best fit equation to the relaxation data. For all treatments, the first term of the three-term Maxwell model (F_1) made a major contribution (72.7- 81.8%) to the total decay force (Table 2).

Table 1. Comparison of the Maxwell models with one, two, and three terms for sea buckthorn berries at different temperatures.

Cultivar	Berry temperature (°C)	One-term model		Two-term model		Three-term model	
		MRD (%)	R^2	MRD (%)	R^2	MRD (%)	R^2
Indian Summer	4.5	21.6	0.808	9.8	0.971	0.9	0.997
	16.5	20.2	0.771	9.9	0.959	1.1	0.995
	34.5	18.6	0.755	8.3	0.958	1.7	0.990
Sinensis (small)	4.5	27.4	0.708	15.5	0.931	0.61	0.999
	16.5	25.7	0.715	14.1	0.939	0.71	0.998
	34.5	26.2	0.753	13.9	0.947	0.86	0.999

Table. 2.Effects of berry temperature on parameters for three-term Maxwell model (Eq.1).

Cultivar	Berry temperature (°C)	F_1 ($Nx10^{-2}$)	F_2 ($Nx10^{-2}$)	F_3 ($Nx10^{-2}$)	τ_1 (s)	τ_2 (s)	τ_3 (s)
Indian Summer							
	4.5	35.6	5.3	4.3	435	7.0	0.7
	16.5	33.0	4.2	4.0	588	7.1	0.8
	34.5	23.9	3.0	2.3	769	7.7	0.8
Sinensis (small)							
	4.5	59.4	9.7	12.5	384	5.1	0.5
	16.5	51.5	8.0	9.6	400	4.9	0.5
	34.5	47.6	7.9	8.9	370	5.8	0.5

The fruit tissue dissipates a major proportion of the initially applied force at a relatively slow rate. For instance, for Indian Summer berries at 16.5°C, 80% of the initially induced force was dissipated during the initial 588 s and the remaining force was dissipated during the final 7.9 s (Table 2).

The decay forces, F_1, F_2, and F_3, represent the elastic components in the Maxwell elements, and indirectly, these are the measures of the elasticity of the material being tested (Kajuna et al. 1998). The relaxation time indirectly represents the viscous properties of the cell wall (Sakuria and Nevins 1992). Temperature had an increasing effect on relaxation time (τ_1) (Table 2). Kajuna et al. (1998) reported that time constants decreased progressively as the fruit softened during storage at different temperatures for core samples of both bananas and plantain fruits.

For both Indian Summer and small Sinensis berries, the firmness (as evidenced by the decay forces) decreased with temperature (Table 2). With an increase in berry temperature, the fruit softens and becomes more viscous. The inter-particle friction within the fruit is also reduced at higher temperatures, consequently, less force is required to maintain a specified deformation level. As berry temperature increased from 4.5 to 34.5°C, F_1 decreased 32.9 and 19.9% for the Indian Summer and Sinensis cultivars, respectively.

Using 16.5°C as the reference temperature, relaxation force data at temperatures of 4.5 and 34.5°C (Fig. 1) were shifted horizontally until superposition of the data was achieved (Fig. 2). The mean temperature shift factors (a_T) determined for each temperature using Eq. 4 are given in Table 4.

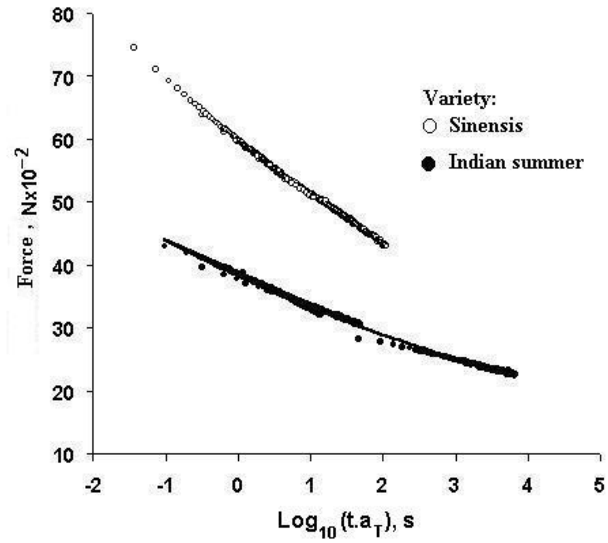

Fig. 2. Force relaxation master curve for sea buckthorn berries at temperatures between 4.5 and 34.5°C, based on a reference temperature of 16.5°C.

Table 4. Temperature shift factors (a_T) obtained for sea buckthorn berries.

Cultivar	Berry temperature (°C)	Temperature shift factor
Indian Summer	4.5	0.3014
	16.5	1
	34.5	145
Sinensis (Small)	4.5	0.1147
	16.5	1
	34.5	2.88

The shift factor functions, determined by nonlinear regression as a function of the difference between test temperature and the reference temperature ($T - T_o$), for Indian Summer and small Sinensis berries, respectively, were:

$$\log_{10} a_T = 0.0741(T - T_o) + 0.0026(T - T_o)^2 \qquad R^2 = 0.99 \qquad \text{(Eq.6)}$$

$$\log_{10} a_T = 0.0572(T - T_o) - 0.0018(T - T_o)^2 \qquad R^2 = 0.99 \qquad \text{(Eq.7)}$$

where T_o is equal to 16.5°C. The master curves shown in Fig. 2 may be approximated as a second-order polynomial curve for the Indian Summer and small Sinensis berries, respectively:

$$F(t) = 38.29 - 5.2811 \cdot \log_{10}(t \cdot a_T) + 0.2994 \cdot [\log_{10}(t \cdot a_T)]^2 \qquad R^2 = 0.996 \qquad \text{(Eq.8)}$$

$$F(t) = 59.804 - 9.0659 \cdot \log_{10}(t \cdot a_T) + 0.4846 \cdot [\log_{10}(t \cdot a_T)]^2 \qquad R^2 = 0.999 \qquad \text{(Eq.9)}$$

where F(t) is force, $Nx10^{-2}$.

Effect of Initial Deformation on Relaxation Behavior

The effect of initial deformation on relaxation behavior for large Sinensis berries is shown in Fig. 3. The parameters for three-term Maxwell models at three deformation levels (10, 18, and 25%) are given in Table 5. Values of maximum relative difference (MRD), and R^2 for one-, two- and three-term Maxwell models are compared in Table 6.

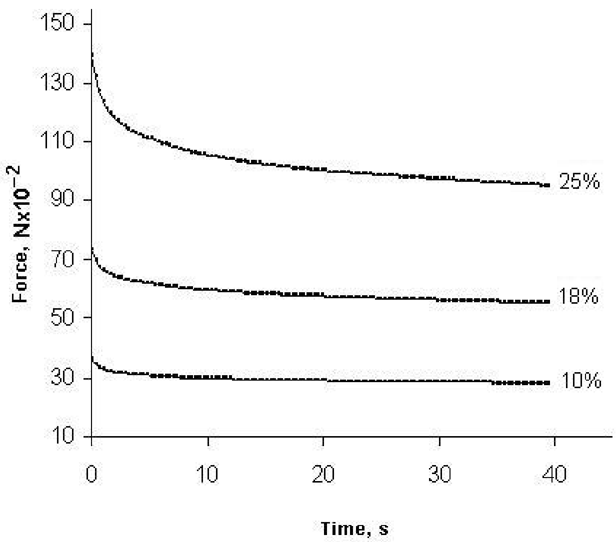

Fig. 3. Effect of initial deformation on relaxation behavior of large Sinensis berries. Loading velocity was held constant at 0.7 mm/s and temperature was constant at 16.5°C.

Table 5. Effects of initial deformation on parameters of the three-term Maxwell model (Eq.1) for large Sinensis berries.

Deformation (%)	F_1 ($Nx10^{-2}$)	F_2 ($Nx10^{-2}$)	F_3 ($Nx10^{-2}$)	t_1 (s)	t_2 (s)	t_3 (s)
10	29.6	31.1	3.5	769	5.5	0.5
18	59.7	7.2	7.0	526.3	5.4	0.5
25	104.6	18.3	16.9	416.7	5.9	0.7

Table 6. Comparison of the Maxwell models with one, two, and three terms for large Sinensis berries at different deformation levels.

Deformation (%)	One-term model		Two-term model		Three-term model	
	MRD (%)	R^2	MRD (%)	R^2	MRD (%)	R^2
10	18.5	0.687	9.9	0.921	1.8	0.980
18	19.3	0.756	9.6	0.955	0.94	0.996
25	25.2	0.743	12.2	0.957	0.51	0.999

Deformation level had a definite effect on the decay force (F_1). As deformation increased from 10 to 25%, the decay force (F_1) increased from 29.6 to 104.6 Nx10^{-2}. Deformation level also had a significant effect on relaxation time (i.e., the first term of the three-term Maxwell model) with relaxation time decreasing with increasing initial deformation (Table 5). At higher deformation levels, the force induced in the fruit is higher than that at the lower deformation levels. Accordingly, when the crosshead is stopped, the force is decreased with a higher speed and this leads to a decrease in relaxation time. Similar results for effect of deformation level on relaxation time were obtained for other agricultural materials (Husain et al. 1971; Zoerb and Hall 1960), however, Bargale and Irudayaraj (1995) reported that the effect of deformation level on relaxation time was consistent and did not show a clear trend.

Effect of Loading Velocity on Relaxation Behavior

The influence of loading velocity (during the loading process) on relaxation behavior of Indian Summer berries was determined for a temperature of 4.5°C (Fig. 4). The Maxwell models describing the relaxation behavior of Indian Summer berries at loading velocities of 0.1 and 0.3 mm/s, respectively, were:

$$F(t) = 35.73^{-t/625} + 5.90e^{-t/15.38} + 4.29e^{-t/1.83} \qquad R^2 = 0.997 \qquad \text{(Eq.10)}$$

$$F(t) = 23.48^{-t/526.3} + 4.23e^{-t/9.89} + 3.61e^{-t/0.75} \qquad R^2 = 0.989 \qquad \text{(Eq.11)}$$

where F(t) is force, Nx10^{-2}. For these models, the values of maximum relative differences (MRD) for one-, two- and three-term Maxwell models were in the range of 22.2-25.7, 9.3-12.3, and 1.5-2.5, respectively. Corresponding values for R^2 were in the range of 0.853-0.810, 0.976-0.967, and 0.997-0.989, respectively. The Maxwell model for a loading velocity of 0.7 mm/s (Fig. 1) was:

$$F(t) = 35.6e^{-t/435} + 5.3e^{-t/7.0} + 4.3e^{-t/0.67} \qquad R^2 = 0.997 \qquad \text{(Eq.12)}$$

The relaxation time constant of the first term of the three-term Maxwell model (τ_1) showed a decreasing trend with an increase in the loading velocity. As the loading velocity increased from 0.1 to 0.7 mm/s, the mean value of τ_1 decreased from 625 to 435 s. At time = 0 s, the forces for loading velocities of 0.1 and 0.7 mm/s were 45.9 and 45.2 Nx10^{-2}, repectively (Fig. 5). These two values are not significantly different. Similar results have been reported by Zoerb and Hall (1960) and by Husain et al. (1971) for other food materials, but Bargale et al. (1994) found that the relaxation time constant, τ_1, increased with increasing loading velocity. Bargale and Irudayaraj (1995) found that the effect of loading velocity on relaxation time was consistent and did not show a clear trend.

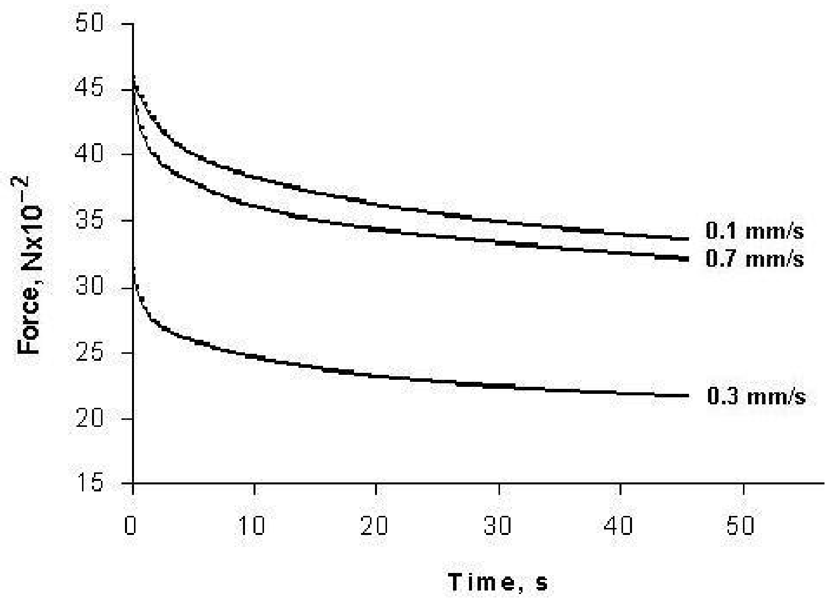

Fig. 4. Effect of loading velocity on relaxation behavior of Indian Summer berries at a temperature of 4.5°C and a deformation level of 10%.

CONCLUSIONS

From this study, the following conclusions were drawn for sea buckthorn berries:

- The sea buckthorn berries had a time dependent behavior similar to other viscoelastic materials.
- The three-term Maxwell model with a maximum relative difference (MRD) of $\leq 5\%$ was chosen as the best fit equation to the relaxation data.
- For both Indian Summer and small Sinensis berries, the firmness (as evidenced by the decay forces) decreased with temperature. As berry temperature increased from 4.5 to

34.5°C, F_1 decreased 32.9 and 19.9% for Indian Summer and Sinensis berries, respectively. The Sinensis berries were significantly firmer than the Indian Summer berries.

- Decay force increased from 29.6 to 104.6 $Nx10^{-2}$ as the deformation level increased from 10 to 25%.
- Deformation level and loading velocity both had a decreasing effect on relaxation time, but temperature had an increasing effect on relaxation time.

ACKNOWLEDGMENTS

The authors would like to thank the Prairie Agricultural Machinery Institute (PAMI) for providing branches with berries for testing and Dr. S. Cenkowski, Department of Biosystems Engineering, University of Manitoba for use of his texture analyzer.

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