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EVALUATING THE VISCOELASTIC OF BARHI DATES AT THREE MATURITY STAGES

Abdullah M. Alhamdan[1]; Hussain M. Sorour[2]; Mahmoud A. Younis[3]; & Diaeldein O. Abdelkarim[4]
[1]Chair of Dates Industry & Technology; King Saud University; P. O. Box 2460; Riyadh 11451
[2]Chair of Dates Industry & Technology; King Saud University; P. O. Box 2460; Riyadh 11451
[3]Chair of Dates Industry & Technology; King Saud University; P. O. Box 2460; Riyadh 11451
[4]Chair of Dates Industry & Technology; King Saud University; P. O. Box 2460; Riyadh 11451

Abstract

The viscoelastic characteristics of dates at three stages of maturity were evaluated using a stress relaxation test. Three stress relaxation models (Generalized Maxwell, Peleg and Nussinovitch) were fitted to the experimental data. The initial imposed stress of dates at the Rutab stage declined by 99% at the Khalal stage; in contrast, the initial stress of dates at the Tamer stage was higher than that of dates at the Rutab stage. The Maxwell model was better to predict the experimental data, followed by the Peleg and Nussinovitch models. The best predictions of the three models were for dates at the Tamer stage, followed by those at the Rutab and Khalal stages of maturity.

Key words- *Date cultivars; Stress relaxation; Maturity stages.*

1. Introduction

The fruit of date palm trees (*Phoenix datylifera L.*) is the most important fruit in Saudi Arabia, with a production of 1,008,105 tons. Approximately 24.38 million trees growing in different regions of the country fill a total cultivated area of 155,118 ha (Ministry of Agriculture and Water, 2012). Mechanical tests, such as the compression–relaxation test, have been used to correlate textural attributes to physical parameters through the analysis of force–deformation curves. Rheological measurements are also important for designing processed foods and in understanding the structure of food materials and processed food products (Lewickian and Lukaszuk, 2000; Krokida et al., 2001 and Boudhrioua et al., 2002).

Stress relaxation data for food products are important because they provide information regarding phenomena such as the staling of cereal products (Limanond et al., 2002). Limanond et al., (2002) modeled the kinetics of corn tortilla staling using stress relaxation data and found that a seven-element Generalized Maxwell model fit the data better than three- and five-element models. The compression stress relaxation data obtained for potatoes exposed to a pulsed electric field were modeled using a five-parameter generalized Maxwell model (Finca and Dejmek, 2003). Krokida et al. (1998) determined the viscoelastic properties of carrots and potatoes following air-drying experiments using stress relaxation tests of uniaxially compressed samples. The experimental data were modeled using a two-term Maxwell model, which showed that the relaxation behavior of the samples was not affected by the deformation rate but was sensitive to the moisture content.

Lewicki and Lukaszuk (2000) observed that the relaxation rate of apple specimens subjected to convective drying increased with decreasing moisture content. Kumar and Neelakantan (1993) studied the stress relaxation behavior of polyacetal-polyurethane blends in detail. In their experiments, the rate of loss of the relaxation modulus was found to be a non-linear function of time. Other study by Othan et al. (2004) found that the stress relaxation behavior of banana fiber-reinforced polyester composites was dependent on the amount of banana fiber. The rate of stress relaxation was found to be maximal during the initial stages of relaxation. Incorporation of fiber reduced the rate of stress relaxation, and the greatest reduction was observed in the case of composites with the highest fiber load. The stress relaxation test is a widely accepted method for determining dough behavior because it is easy to perform and uses a simple texture-measuring instrument. In stress-relaxation experiments, the test specimen is compressed to a predetermined level of strain that is kept constant, during which the viscoelastic material shows a trend of declining force/stress as a function of time (Yadav et al., 2006).

El Oudiani et al. (2009) reported that the best model to explain the viscoelastic behavior of *Agave americana* L. fibers was the modified Generalized Maxwell model. Nicoleti et al. (2004) evaluated the viscoelastic behavior of persimmons that were dried at different air temperatures and velocities. The air temperatures and velocities of drying were varied according to a second-order central composite design, with the temperature ranging from 40 to 70°C and air velocity ranging from 0.8 to 2.0 m/s. After drying, the persimmons were equilibrated at four different water activities: 0.432, 0.576, 0.625 and 0.751. The rheological behaviors of the dried and conditioned persimmons were studied using uniaxial compression–relaxation tests. The following three different rheological models were fitted to the experimental relaxation curves: the Maxwell model, the Generalized Maxwell model and the Peleg and Normand model. Based on the root mean square of the residuals, the generalized Maxwell model showed the best fit; therefore, a regression analysis was applied to obtain the response surfaces of the model parameters.

Mechanical models or analogues have been developed to predict different patterns of viscoelasticity. The Maxwell model has frequently been used to interpret the stress relaxation data obtained for a viscoelastic material. A Maxwell element consists of a spring and a dashpot arranged in series. The model contains n Maxwell elements and a spring in parallel (Sandoval, 2009). The Generalized Maxwell model can be written as follows:

$$\sigma(t) = \sum_{i=1}^{n} C_i \left(e^{-\left(t/\tau_i\right)}\right) + \sigma_e \quad (1)$$

Additionally, two other models have been successfully applied to biological materials; the first is the Peleg model (Peleg and Normand, 1983) and the second is the Nussinovitch model (Nussinovitch, 1989). Stress-relaxation data can be interpreted in terms of normalized stress using the Peleg model and fitted to a linear equation (Peleg and Pollak, 1982). The equation can be written as follows:

$$\sigma(t) = \sigma_0 - \sigma_0 \left(\frac{abt}{1+bt}\right) \quad (2)$$

The constant (a) represents the level at which the stress begins to decay during relaxation. Consequently, at (a = 0), stress never relaxes (e.g., for a solid material, such as rubber), whereas at (a = 1), the stress relaxes to the value of (0) after the elapse of infinite time (e.g., for liquids). The constant (b) represents the stress relaxation rate (decay rate), whereas its inverse (1 / b) represents the time required to reach the level of (a / 2). For the case of (b = 0), the stress never relaxes. For viscoelastic materials with low values of (b), relaxation slows, but it accelerates for materials with higher values of (b) (Peleg and Pollak, 1982).

In the Nussinovitch model, the relaxation time constants (τ_i) become steady at 10, 100, and 1,000, and can be presented as follows:

$$\sigma(t) = \sigma_0 \left(A_1 + A_2 e^{\left(-t/10\right)} + A_3 e^{\left(-t/100\right)} + A_4 e^{\left(-t/1000\right)}\right) \quad (3)$$

Knowledge of the various mechanical properties of date fruits and other agricultural crops is important because it provides the engineering data required to design various processing machines, structures, processes and controls; to analyze and determine the efficiency of a machine or an operation; to develop a new consumer product; and to evaluate and retain the quality of final products.

The objectives of the current study were the following:

1- To obtain data describing the stress relaxation characteristics of four date cultivars at the Khalal, Rutab and date (Tamer) stages of maturity;
2- To investigate the effect of the maturation stage on stress relaxation properties; and
3- To find the best model to describe the obtained stress relaxation data.

2. Materials and Methods

2.1. Sample preparation

Barhi dates, were used in the experiments at three stages of maturity [Khalal, Rutab and date (Tamer)]. During the Khalal stage of maturity, the color of the fruit changes from green to yellow, or to red in some cultivars, the moisture content decreases to 55% on a wet weight basis and the tannins begin to precipitate and lose their astringency. The Rutab stage follows the Khalal stage of maturity. During this stage, the moisture content of the fruits of some cultivars decreases to approximately 20% on a wet basis. The date stage follows the Rutab stage of maturity. During this stage, the moisture content of the fruits of some cultivars decreases to approximately 7% on a wet basis. The dates were obtained from the educational farm of King Saud University. The dates were immediately sorted to remove the damaged fruits and maintained for less than 24 h in cold storage at 5°C. The moisture content of the flesh of the dates was determined using AOAC procedures (AOAC, 1995), in which the samples were dried at 70°C for 48 h under a vacuum of 200 mmHg (Vacutherm model VT 6025, Heraeus Instruments, D-63450, Hanau, Germany). The water activity was measured using an Aqua-Lab instrument (Model CX-2T, readability: 1 mg, Decagon Devices Inc., Washington).

2.2. Instrumentation

A texture analyzer (TA-HDi, Model HD3128, Stable Micro Systems, Surrey, England) with a 75-mm-diameter disk plunger (# P75) was used to conduct the uniaxial compression tests. The texture analyzer was interfaced with an IBM-compatible PC. The software package used was Texture Expert Exceed version 2.05, which was supplied by the same company. This software enabled data to be acquired in Excel files. The gradient of the curve between any two specified locations, the area under the curve and other parameters can be determined using the software. All of the experiments were conducted at room temperature (23°C). The instrument was calibrated with forces of 50–100 kN to a linearity greater than 1%. The contact area between the surface of the plunger disk and the surface of each tested fruit was determined experimentally. The plunger disk surface was covered with a piece of white paper and the upper surface of the horizontally oriented fruit was gently pressed against an ink stamp, and then the plunger was allowed to contact the fruit surface. After the contact area was traced on the white paper and scanned, specially developed software that accurately measures the scanned surface area was used to determine the contact area.

2.3. Selection of stress relaxation models to simulate the stress relaxation data

A non-linear regression analysis (Levenberg–Marquardt) in the SPSS (Statistical Package for the Social Sciences) statistical software package (SPSS for Windows, 1999, release 9.0, SPSS Inc., Chicago, Illinois, USA) was utilized to find the constants for the stress relaxation models.

Several criteria were used to evaluate the fit of the different selected models to the experimental data. The following statistical parameters were also used to evaluate the respective results:

Standard deviation of difference (SD)

$$S_D = \sqrt{\frac{\Sigma\left(\sigma_{Exp} - \sigma_{Prd}\right)^2 - \left[\left(\sigma_{Exp} - \sigma_{Prd}\right)\right]^2/N}{N-1}} \quad (4)$$

The average percentage error (%E)

$$E = \frac{100}{N}\sum\frac{|\sigma_{Exp}-\sigma_{Prd}|}{\sigma_{Exp}} \quad (5)$$

The modeling efficiency (EF)

$$EF = \frac{\sum(\sigma_{Exp}-\sigma_{Exp_{mean}})^2-\sum(\sigma_{Pre}-\sigma_{Exp})^2}{\sum(\sigma_{Exp}-\sigma_{Exp_{mean}})^2} \quad (6)$$

Where:

σ_{Exp} = the experimental stress value (kPa);
σ_{Pre} = the predicted stress value (kPa);
$\sigma_{exp\ mean}$ = the mean value of the experimental stress (kPa); and
N = the number of data points.

A model is considered to be good when the coefficient of determination (R^2) and modeling efficiency (EF) are high and the standard deviation (SD) and average percentage of error (E) are low.

Stress relaxation test

Individual date fruits were uniaxially compressed at an across-head speed of 1.5 mm/s. The contact surfaces were oriented parallel to the compression surfaces during loading. Before applying the tested force or stress, the deformation (4 mm) rate and the time (5 min) in which the relaxation was measured were maintained constant at all preformed tests. A random sample of 10 fruits of each cultivar at each stage of maturity was taken for the compression tests, and 120 runs were conducted.

3. Results and Discussion

The mean values of the moisture content for the Barhi dates at three maturity stages are shown in Figure. 1. This figure shows that the moisture content at the Khalal stage is greater than that at the Rutab or date stage. The moisture content ranged from 65.4% (w.b.) for *Khalal stage* dates to 6.9% for Tamer stage, whereas at the Rutab stage, the moisture content was 29.7 % (w.b.).

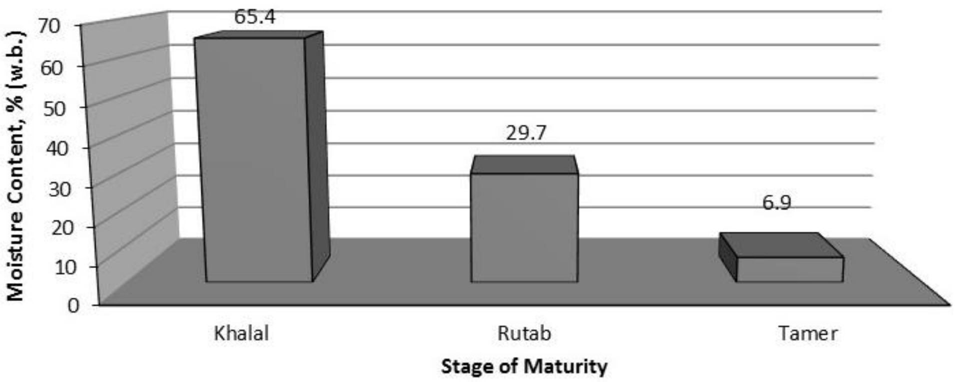

Figure 1: Moisture content of Barhi date at three stages of maturity

Figure 2 shows the initial stress of Barhi dates at the Khalal, Rutab and Tamer stages. This figure shows that the highest stress value at time zero (σ_0) of Barhi was observed for the *Khalal stage* (2708.9 kPa) followed by the *Tamer* stage (27.63 kPa). These viscoelastic values reflect the firmness and cohesiveness of the structural tissues during the maturity stages. Figure 2 also shows the initial stress at the Rutab stage of Barhi. The *Barhi* was the least firm at the Rutab stage. There was a considerable decline in the imposed initial stress (σ_0) for the *Barhi* dates between the Khalal stage, when the value was 2708.9 kPa, and the Rutab stage, when it was 6.9 kPa (a decrease of 99.75%). The reason for this considerable decline in (σ_0) is that major changes in structural tissues occur during maturation from the Khalal to the Rutab stage. The most notable changes are in the type of sugar as a result of the enzymatic conversion of sucrose to fructose and glucose during the maturation process. The changes in pectin content resulting from the activity of pectinase enzymes lead to a softer date structure at the Rutab stage than at the Khalal stage, despite the reduced moisture content at the Rutab stage. The moisture content at the Khalal stage was 62.6%, whereas it was 29.7% at the Rutab stage (wet basis). The soft and supple structure of the dates during the Rutab stage renders them susceptible to shape distortion by imposed mechanical stresses. Figure 2 also, shows the initial stress at the Tamer stage for the four date cultivars. Higher initial stress (σ0) was observed in the Tamer stage dates relative to the Rutab stage dates for Barhi dates.

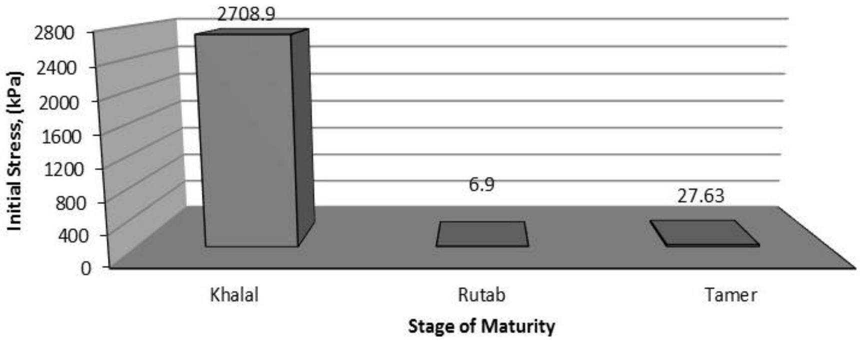

Figure 2: Initial stress of Barhi date at three stages of maturity

The three models used to describe the stress relaxation characteristics of Barhi fruits investigated at the Khalal, Rutab and Tamer stages, were the Generalized Maxwell, Nussinovitch and Peleg models. Preliminary statistical tests were conducted to determine the optimal number of terms for each model. Three terms were best for the Maxwell model, whereas the terms represented by Eqs. (2) And (3) were adequate for the Nussinovitch and Peleg models. The constants for Barhi date at the Khalal, Rutab and Tamer stages in each model were determined using a non-linear regression method (Levenberg–Marquardt); the results are shown in Tables 1, 2 and 3. Based on the coefficient of determination (R^2) and the modeling efficiency (EF), standard deviation (SD) and average percentage of error (%E) values, the three-term Maxwell model most closely represented and predicted the experimental results. The coefficient of determination for Barhi dates for three maturity stages obtained using the three models ranged from 0.998 to 0.853, this means that all the models could satisfactorily describe the stress relaxation characteristics. However among the three studied models, the Maxwell model showed the highest Average coefficient of determination (R^2) of 0.975, 0.990 and 998 and efficiency (EF) of 0.874, 0.986 and 0.995 for Rutab, Khalal and Tamer stage respectively and the lowest standard deviation (SD) of 0.26, 0.347 and 32.9 for Tamer, Rutab and Khalal respectively and the lowest average percentage of error (%E) of 0.05, 0.052 and 0.404 for Tamer, Khalal and Rutab stage respectively.

Table 1: Statistical results from Generalized Maxwell model and the constants of model.

Stage	σ_e (kPa)	c_1 (kPa)	c_2 (kPa)	C_3 (kPa)	τ_1 (s)	τ_2 (s)	τ_3 (s)	R^2	EF	SD	E
Khalal	1207.4	590.5	400.5	427.9	0.9	12.1	133.8	0.990	0.986	32.9	0.052
Rutab	1.6	0.5	2.9	1.1	69.3	1.3	41.3	0.975	0.874	0.347	0.404
Tamer	7.8	5.9	9.7	3.3	9.1	0.7	94.3	0.998	0.995	0.260	0.050

Table 2: Statistical results from Nussinovitch model and the constants of model.

Stage	A_1	A_2	A_3	A_4	R^2	EF	SD	E
Khalal	0.34	0.2	0.11	0.15	0.853	-0.063	288.0	0.605
Rutab	0.41	0.26	0.23	0.25	0.882	-15.8	4.0	5.807
Tamer	0.207	0.287	0.074	0.1	0.908	0.536	2.5	0.680

Table 3: Statistical results from Peleg model and the constants of model.

Stage	A	B	R^2	EF	SD	E
Khalal	0.53	0.16	0.935	0.922	78.2	0.122
Rutab	0.77	0.35	0.969	0.923	0.270	0.327
Tamer	0.703	0.369	0.988	0.988	0.411	0.117

As expected, the predicted values for the imposed stress after complete relaxation (at equilibrium, σ_e) based on the Maxwell model (Table 1) were higher at the Khalal stage of 1207.4 (kPa) than at Tamer and Rutab stage of 7.8 and 1.6 (kPa) respectively.

The values of the stress relaxation constants, C_i, for the three terms of the Maxwell model were highest for the *Barhi* dates at the Khalal stage of maturity, whereas the lowest values were exhibited by Tamer and Rutab stages. This result indicates that the elastic components of the three-term Maxwell model were highest for Khlal stage, followed by the Tamer and Rutab stages. Plots of the experimental and the predicted stress relaxation over time are shown in Figure 3.

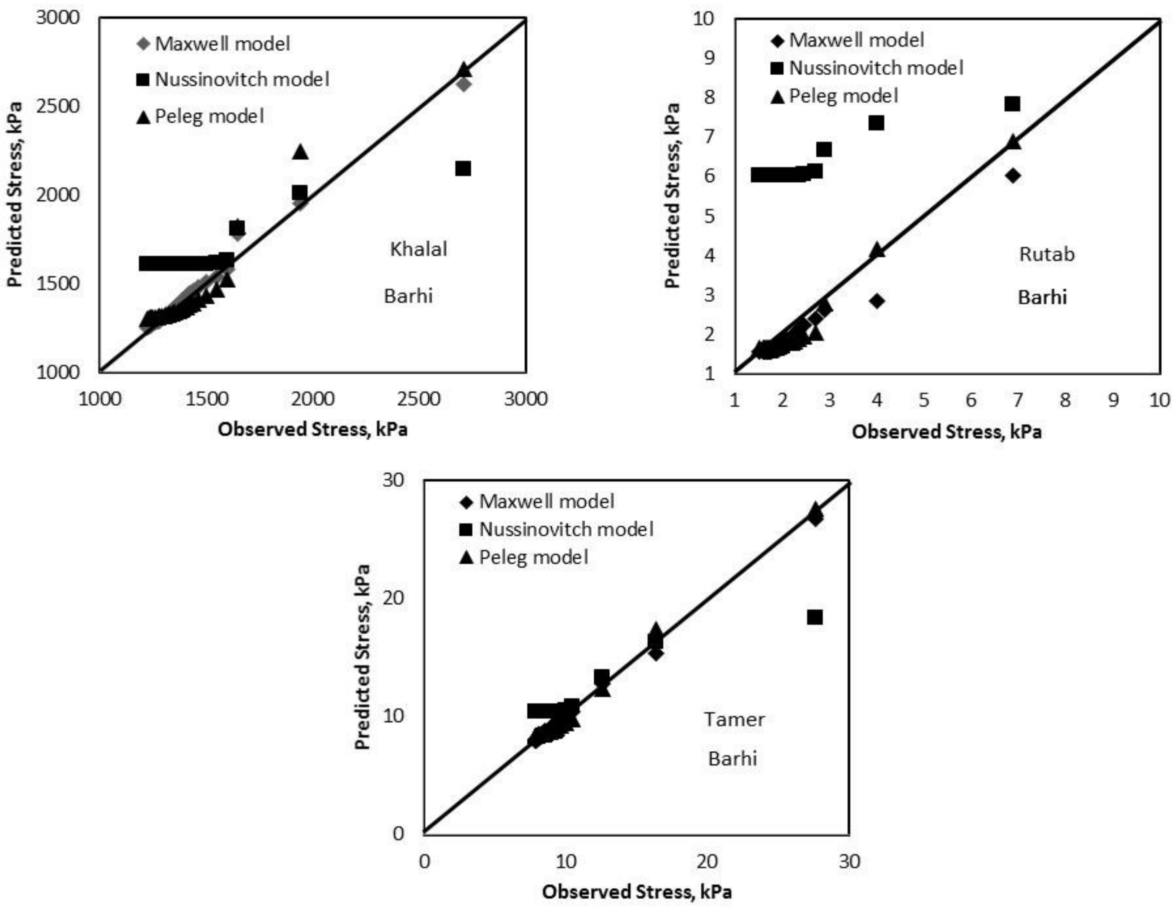

Figure 3: Observed and predicted stress of dates for different models at maturity stages

As shown in this figure, the Generalized Maxwell model provided good agreement between the experimental and predicted data compared with the other two models. In addition, Table 1 and Fig. 3 show that the best predictions of the three models were for the Tamer stage, followed by the Rutab and Khalal stages of maturity.

4. Conclusions

The initial stress of dates at the Rutab stage declined by 99% when they reached the Khalal stage because of the major changes in their structural tissues that occurred during maturation from the Khalal (balah) to the Rutab stage. The initial stress at the Tamer stage was greater than that at the Rutab stage. Three stress relaxation models (Generalized Maxwell, Peleg and Nussinovitch) were fitted to the experimental data. The Maxwell model provided the best predictions of the experimental data, followed by the Peleg and Nussinovitch models. The best predictions of the three models were for dates at the Tamer stage, followed by dates at the Rutab and Khalal stages of maturity.

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