Determination of viscoelastic properties of dates (Mazafati Variety)

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Abstract: The knowledge about rheological properties of agricultural fruits can be significantly helpful in design optimization and fabrication of the appropriate processing machines. In this study, the viscoelastic characteristics of date (Mazafati variety) were investigated. Experimental tests were established at six moisture content levels (7.5, 9.0, 10.8, 12.8, 17.59 and 25.5%d.b.) and temperature of 45ºC, which contributed to obtain a suitable model. To achieve the desired moisture levels, various saturated salts were used. Experimental data of creep tests were fitted into Burger four-element model. The results indicated that, creep factor inclined when moisture content increased, meanwhile, its minimum and maximum values were 0.08 mm and 0.26 mm at the moisture contents of 7.5% d.b. and 25.5% d.b., respectively. Consequently, constant values related to the model, were determined and correlated with the moisture contents of the products using non-linear regression analysis. Analysis of variances and using Tukey test (P_{value}<0.01) on creep parameters revealed that moisture content had significant effects on all firmness values except the primary strain. Moreover, the moisture content showed a great impact on the delay time. According to the results, the rheological properties of date are highly dependent on its moisture content.

Keywords: viscoelastic properties, Mazafati, date, creep test, moisture

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1 Introduction

Rheological properties assessment of agricultural products is a basic study to investigate the relationship between product structure, texture, chemical compositions, change of product behavior during different processes. These characteristics are fundamental indices for determining the product maturity, shelf life, and processing conditions. Date as a conventional tropical fruit, contains the highest percentage of carbohydrate, including fructose and glucose. In addition to carbohydrate, the date fruits have remarkable amounts of protein, crude fiber, pectin, tannin, minerals, and vitamins (Sidhu, 2006). Due to the characteristics of date, this product has become an important commercial fruit for producer countries.

Owing to increasing importance of food production by the advent of food scarcity, the processing technology requires more knowledge and researches on food material properties. For this reason, the rheological properties play an important role in the quality attributes of processed foods. The material properties highly depend on temperature which is a key element for rheological behavior analysis (Rao and Steffe, 1992). Having physicochemical and structural properties values as functions of temperature and moisture content of a particular variety, is essential for optimum date processing designs. Date palm is becoming a crucial important commercial crop in the producing countries where a significant increase in the yield can be achieved by adopting advanced biotechnological approach. In addition, date processing industry has not been developed widely so far (Ahmed and Ramaswamy, 2006).

The results of several experiments have been documented well, which mostly reported the viscoelastic behavior of different fruits and vegetables. However, it has been demonstrated that creep compliance tests can provide more information than stress relaxation tests (Mohsenin,

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1996). Researchers reported that the combination of generalized Kelvin model with Maxwell model could lead to the best presentation of the creep data obtained from some produce like apples, potatoes, and cheeses (Datta and Morrow, 1983). Furthermore, the Burgers model has been yielded under static creep condition using numerical studies of rheological model and has contributed to estimate the values of the properties of apple, potato, pear and other produce (Mohsenin, 1996). The main advantage of the creep compliance tests over the stress relaxation tests is that analysis can be facilitated using the Burgers model, which results in independent predictions of a larger number of rheological parameters and elastic, viscoelastic and viscous flow characteristics (Sherman, 1966).

Figure 1 depicts a creep curve, related to wheat flour dough (Bockstaele et al., 2011). In another study, grain dust showed similar behavior to that of the four element Burgers model (Chang and Martin, 1983). The behavior of the four elements Burger model is illustrated in Figure 1 and is formulated as the following expression (Mohsenin, 1996).

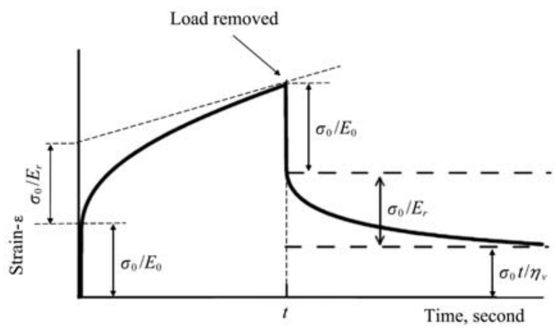

Figure 1 Typical creep and recovery curve in a viscoelastic material exhibiting instantaneous elasticity, retarded elasticity and viscous flow

$$\varepsilon(t) = \sigma_0 \left[\frac{1}{E_0} + \frac{1}{E_r}(1 - e^{-t/T_{ret}}) + \frac{t}{\eta_v} \right] \quad (1)$$

where, σ_0 is constant stress in creep test (MPa); E_0 is instantaneous modulus at zero time (MPa); E_r is retarded modulus (MPa); T_{ret} is retarded time (s); t is time (s) and η_v is Newtonian viscosity (MPa s).

Equation (1) is derived from a Kelvin model connected in series with a spring and a dashpot element. Eissa et al. (2012) used the four element Burgers model and the following equation as shown in Figure 2 to determine the rheological constants of the model (K_1, K_2, C_1, and C_2) and their relations with the fruit (pears) parameters.

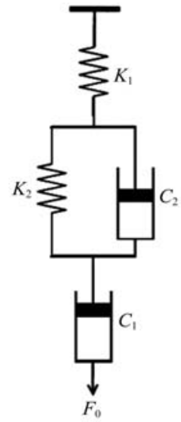

Figure 2 Creep of the Four-element model (Burger model)

$$d(t) = F_0 \left[\frac{1}{K_1} + \frac{1}{K_2}(1 - e^{-t/T_{ret}}) + \frac{t}{C_1} \right] \quad (2)$$

where, $d(t)$ is deformation as a function of time t (mm); K_1 and K_2 are instantaneous and retarded elasticity (N s mm^{-1}).

Several studies have indicated that the viscoelastic nature of the agricultural and food materials can be analyzed by rheological models (Bagley and Christianson, 1987; Bargale et al., 1994; Davis et al., 1983; Hamann 1992; Pappas et al., 1988; Purkayastha et al., 1985). For example, the rheological characteristics of fresh and cooked potatoes were reported by using creep tests (Alvarez et al., 1998; Purkayastha et al., 1985). Moreover, the changes in the parameters of the creep models depending upon the storage time and the temperature was reported in the above studies. The rheological parameters provide more details of the food structure behavior during unit operation procedures (Guerrero and Alzamora, 1998; Holdsworth, 1993). Considering the consumer demand for high quality foods, it would be necessary to monitor the food rheological properties during the processing operations, which may affect the overall acceptability (Nindo et al., 2007). Date fruits have high popularity in the word, especially in the Middle East, Mediterranean region, and North Africa. Viscoelastic properties of eight date cultivars in two stage of maturity have been studied by Hassan et al. (2005), while the dates' moisture content was about 19% on wet basis (w.b.). They pointed that the date fruit exhibited viscoelastic properties. Iran as the second largest date producer has 162998 hectares cultivation area and its production is1083720 (ton h^{-1}) (Ehteshami et al., 2017).

However, due to improper processing of this product, Iran has not received appropriate position in the world market (Anonymous, 2010b). Mazafati variety in the domestic market has attained the most popularity of date. Mazafati fresh date fruit as a semi-dry variety has dark red to black-oriented color and contains high value of moisture content, therefore its behavior can change during different process. The main problem for this variety is its delicate nature with high tendency to deteriorate due to the high equilibrium relative humidity.

The main objective of the present study is to evaluate the viscoelastic characteristics of date fruit under creep test by determination of elastic modulus and retardation time at different moisture content levels.

2 Materials and methods

2.1 Experiment test design and Methodology

Dates (dark red to black-oriented color in Rutab stage) were harvested from the date trees grown in Bam, Iran. Fresh samples were kept at 4°C with relative humidity of 50% to 65%. Pitted dates were then used for the experiments. The compressive creep tests were used to determine the date viscoelastic behavior. The creep test was conducted using texture analyzer (SANTAM, STM-20) device, while the speed of the moving jaw was set to 10 mm s^{-1}. During the experiments, dates were placed into two parallel plates. Samples at each moisture content level were subjected to a step load of 1.7 N for 300 s. The moisture content was measured gravimetrically using an oven which was set to 70°C. The moisture content of the harvested dates or fresh stage (FS) material was found to be 28%d.b. The dimensions of date were measured with a caliper. The average length of the major axis (longest dimension) was (36±1) mm, and minor axis (16±1) mm (width), and normal axis (15±0.8) mm (thickness). The average mass of the fruit was measured as (12.14±1.00) g. Pitted dates (cut into half in longitudinal direction) were stored in air-sealed glass jars. To meet the conditions of equilibrium moisture content of the samples, the dates were exposed to supersaturated salts. In order to provide supersaturated solution, following salts were used: LiCl, NaCl, $NaNO_2$, KNO_2, $MgCl_2$, and $KC_2H_3O_2$. Relative humidity ranging from 0.11% to 0.75% was measured for these salts. The supersaturated conditions were maintained by keeping a crystal layer of salt at the bottom of beaker containing salt solution. This situation was achieved by adding salt until a layer of crystals was observed. The salt was added time to time during the equilibration period when the salt crystals were dissolved by water adsorption from the sample.

2.2 Theoretical consideration

The creep deformation is a combination of three types of deformation: instantaneous elastic, retarded elastic and viscous deformation. In this study, the creep data were used to model the Burgers model. As the Figure 3 shows, the Burgers model is the mathematical expression of the combination of mechanical analogues (springs and dashpots) which represents the viscoelastic behavior. The Burgers model is originally designed for the modeling of linear viscoelastic properties, but the model is also a promising tool for examining creep recovery curves.

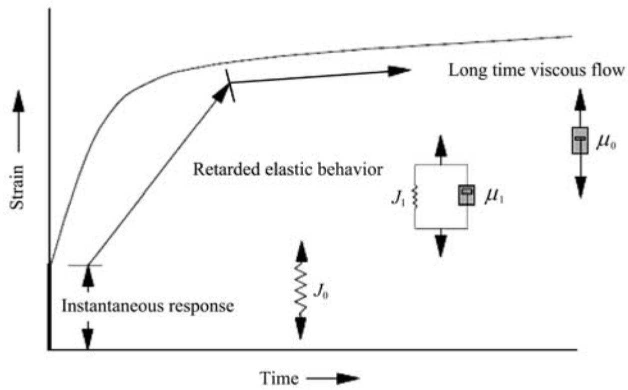

Figure 3 Creep curves for Burger model parameter

$$\varepsilon(t) = \sigma_0 / E_0 + \sigma_0 / E_r (1 - e^{-t/T_{ret}}) + \frac{\sigma_0}{\eta_v} \quad (3)$$

$$J(t) = J_0 + J_1(1 - e^{-t/T_{ret}}) + t / \eta_v \quad (4)$$

where, $J_0 = 1/E_0$ is called Primary creep compliance and $J_1 = 1/E_r$ called retarded creep compliance (Mohsenin, 1996).

2.3 Statistical analysis

The data analysis was obtained using nonlinear regression (Levenberg Marquadt) method in SPSS software (Version 16.0 for windows, SPSS Inc.). The models were verified by a new set of collected date and using criteria such as percent of average relative error (*ARE*%), root mean square error (*RMSE*), mean bias error (*MBE*), and coefficient of determination R^2 (Alirezaei et al., 2013; Zare et al., 2006).

$$ARE = \frac{100}{N} \times \sum \frac{|Y - Y'|}{Y} \quad (5)$$

$$MBE = \frac{1}{n} \times \sum (Y - Y') \quad (6)$$

$$RMSE = \frac{100}{N} \times \sqrt{\sum \left(\frac{Y - Y'}{Y} \right)^2} \quad (7)$$

$$R^2 = \frac{\sum (Y' - \bar{Y})^2}{\sum (Y - \bar{Y})^2} \quad (8)$$

where, Y is te experimental data; Y' is the modeled value; $\bar{Y}$ is the sample mean and N is the number of observations.

3 Results and discussions

3.1 Equilibrium moisture content

Mean equilibrium moisture content (*EMC*) was determined for date fruit in six different levels of relative moisture content, as shown in Table 1.

The results showed that the *EMC* increased with growth in the relative moisture content due to different vapor pressures between the material and the ambient.

Table1 Laboratory measured value for equilibrium moisture content (absorption) at 45°C

Relative moisture content, %	74.5	60.1	45	31.1	19.8	11.2
Equilibrium moisture content, % d.b.	25.5±0.2	17.6±0.1	12.8±0.04	10.8±0.1	9.0±0.4	7.5±0.4

3.2 Isotherm graph

As Figure 4 illustrated, water absorption had a slight raising trend before water equilibrium relative humidity reached 50%, and consequently graph shot up. In fact sugar in forms of crystal material has a bit of water activity due to its low bonding strength with water molecules, in comparison with solvent sugar. These results are satisfactory with those conducted by Jayas and Mazza (1991).

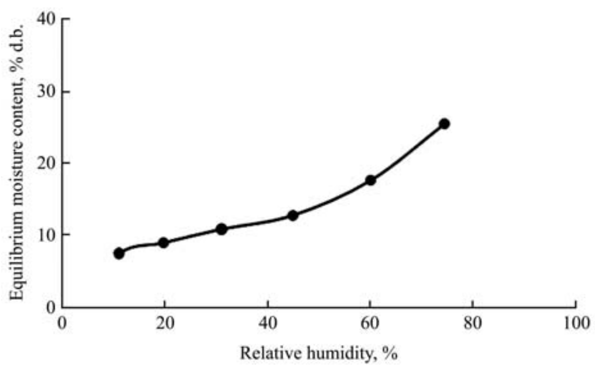

Figure 4 Isotherm graph for Mazafati date at T=45°C

3.3 Rheological properties of Date fruit

3.3.1 Instantaneous module of elasticity

The results showed that there was no significant effect on instantaneous module of elasticity as a result of different moisture contents. However, the raising trend shows the transition of date texture to paste form (Figure 5).

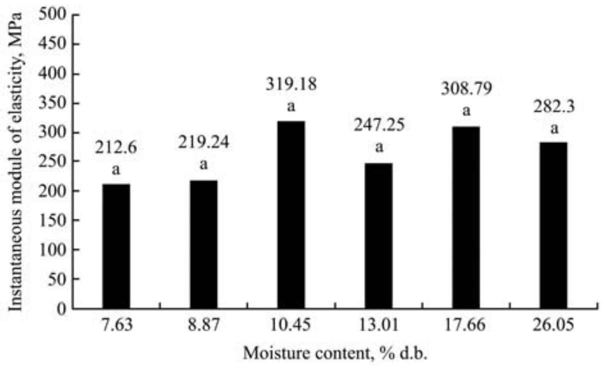

Figure 5 Moisture content's effects on instantaneous module of elasticity

3.3.2 Retardation module of elasticity:

As Figure 6 showed, the moisture content had a significant effect on retardation module of elasticity (P-value<0.01). While moisture content increased, the fruit texture became softer, deformed considerably over a given time and changed from elastic to plastic form. The analysis indicated that, the retardation module of elasticity decreased as moisture content increased. Water absorption is high as far as the existence of polar sides in protein networks is considerable. In addition, the hydrocolloids diminishing leads to the reduction of the firmness value. Furthermore, the formation of more extensive chains will contribute to higher water holding capacity. (Campbell et al., 2009)

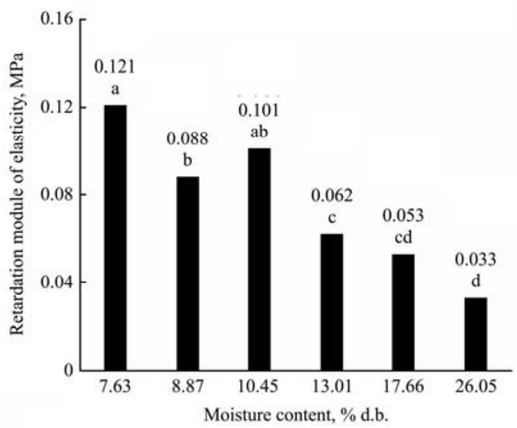

Figure 6 Moisture content's effect on retardation module of elasticity

3.3.3 Retardation time

Since retardation time shows opposite trend compared to the retardation module of elasticity, when the retardation module of elasticity decreased, the retardation time increased. This fact implies that the temperature has more effects on retardation time in comparison with the moisture content (Figure 7&8).

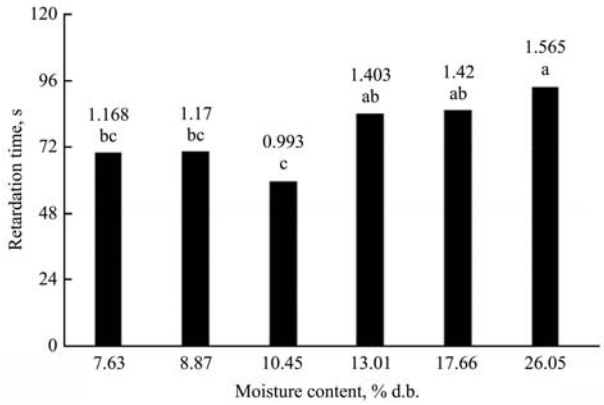

Figure 7 Moisture content's effect on retardation time (P-value<0.01)

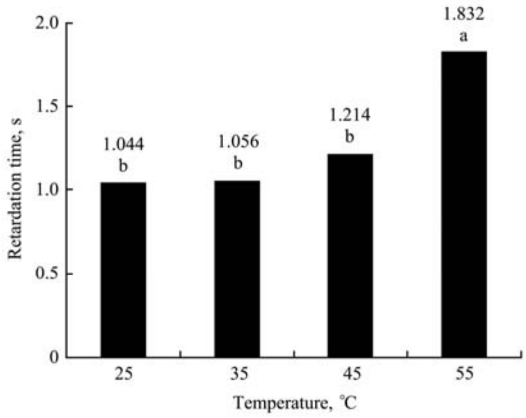

Figure 8 Temperature effect on retardation time (P-value<0.01)

3.3.4 Viscous deformation and viscous coefficient

Figure 9 and 10 provided that as moisture content increased, the viscosity decreased. This can be attributed to higher solubility and the quantity of total solids in the lower moisture contents. Viscosity of most materials and fluids decreased as moisture content increased and the reason is the changes of solvent molecular bonding forces.

3.4 Moisture content's effect on creep test

The results illustrated that if the moisture content increased, the creep test values grow (Figure 11-12). Steward (1968) reported that water would retain by molecular bonding forces which can impact the viscoelastic properties, he also noted that there were considerable effects in lower moisture content.

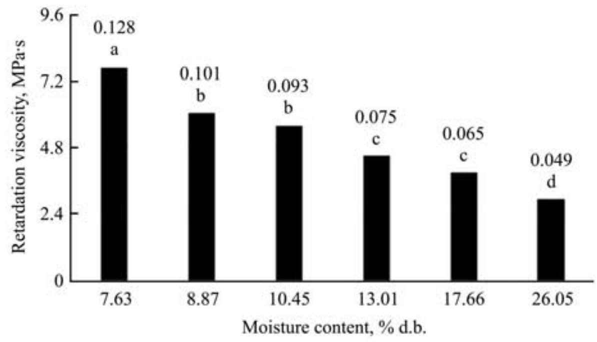

Figure 9 Moisture content's effect on viscous deformation (P-value<0.01)

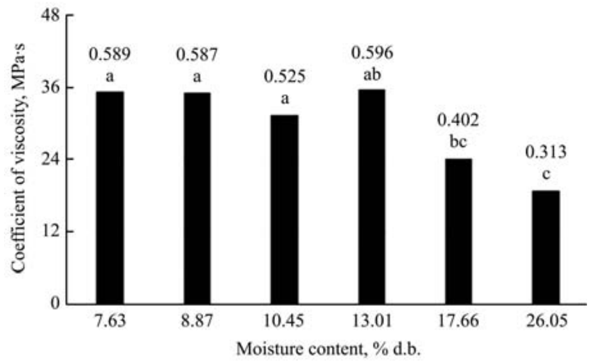

Figure 10 Moisture content's effect on viscous coefficient (P-value<0.01)

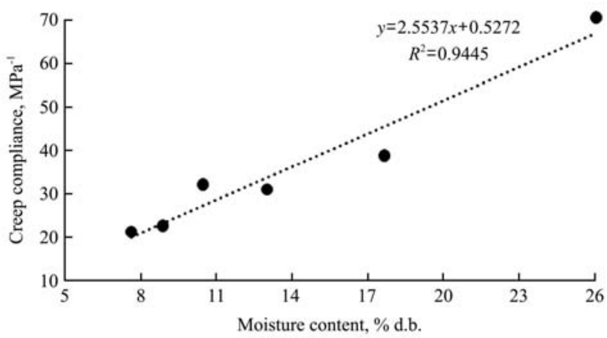

Figure 11 Moisture content's effect on creep compliance

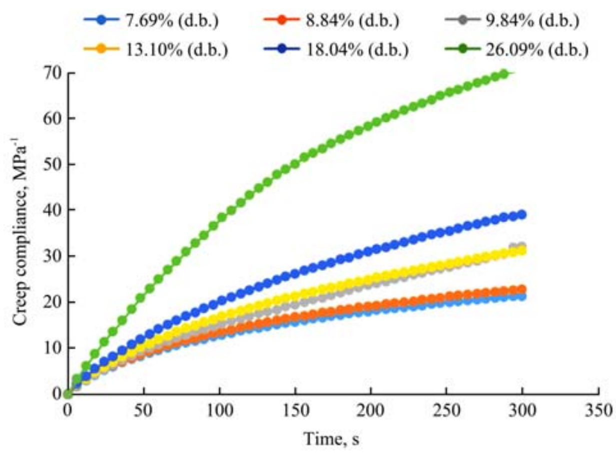

Figure 12 Time's effect on creep compliance at $T = 45^oC$

3.5 Four elements model (Burger model)

By using nonlinear regression for experimental data as shown in Figure 13, some statistic parameters were derived as Table 2 provided.

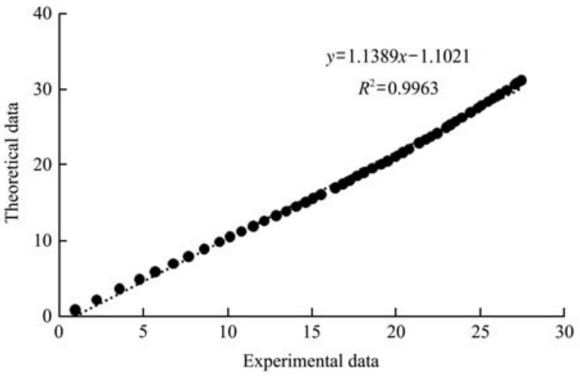

Figure 13 Correlation between experimental data and theoretical data for T = 45°C and moisture contents (M.C.) of 10.45%d.b.

Table 2 Regression results for four elements model at different moisture contents

M.C. (d.b. %)	R^2	*RMSE* %	*ARE* %	*MBE* %
7.6	0.99	0.48	4.86	0.46
8.9	0.99	0.48	4.75	0.47
10.5	0.99	1.72	6.44	-1.28
13.0	0.99	2.01	8.7	1.86
18.1	0.99	0.37	2.64	0.34
26.1	0.99	1.26	2.97	1.1

3.6 General equation of creep test

Using SPSS software, all creep test data were fitted into a principal graph. This consisted of 80% of experimental data to form a nonlinear relationship and 20% of data for creep model validation.

A general equation as function of time, temperature and moisture content, was derived as:

$$J = 0.008115 + [(1.134 lnT^4 + 0.119M^2)(1-(Exp(-t/0.059T + 1.052 lnM^2)))] + [4t^{0.1}/(0.145T + 0.675M^{0.01})] \quad (9)$$

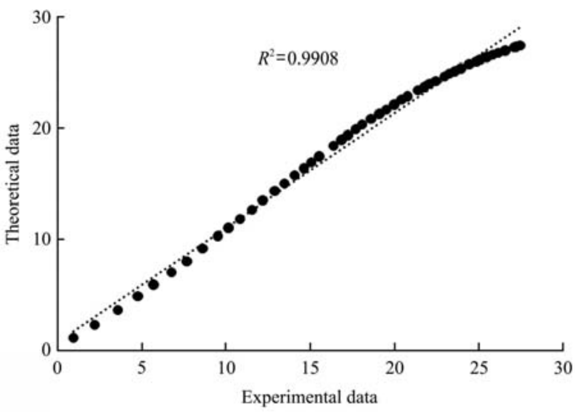

Figure 14 Correlation between experimental data and theoretical data from general model at T=45°C and M.C. of 10.45%d.b.

In equation above, T (°C), M (%d.b.) and t represented temperature, moisture content and time, respectively. It must be added that this relation has been regressed for any moisture content. Figure 14 shows correlation of experimental data and predicted value for a typical condition at T=45°C and date moisture content of 10.45% d.b.

4 Conclusions

Creep test shows that date fruit, like other agricultural products, has viscoelastic properties. Subsequently, some viscoelastic properties such as instantaneous module of elasticity, retardation module of elasticity and retardation time have been determined. The results showed that the moisture content had the dominant effect on defined properties. Thus, the decrease of moisture content would decrease the retardation module of elasticity and increase the retardation time. Finally, a general equation based on creep test data for date fruit (Mazafati variety) has been derived at 45°C and different levels of moisture content, which can represent fruit behaviors under different time levels, depending on temperature and moisture content.

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Nomenclature

C_1 Free viscous element, (N s mm^{-1})

C_2 Retarded viscous element, (N s mm^{-1})

$d(t)$ Deformation at time (t), (mm)

E_r Retarded modulus, (MPa)

E_0 Instantaneous modulus or modulus at zero time, (MPa)

F_0 Constant force, (N)

$J(t)$ The compliance at time (t), (MPa^{-1})

J_0 Instantaneous compliance, (MPa^{-1})

J_1 Retarded compliance, (MPa^{-1})

K_1 Instantaneous elasticity, (N mm^{-1})

K_2 Retarded elasticity, (N mm^{-1})

T_{ret} Retarded time, (s)

T Time, (s)

$\varepsilon(t)$ Strain at time (t)

σ_0 Constant stress in creep test, (MPa)

η_v Newtonian viscosity, (MPa s)