ISSN 1110-0419

Annals Of Agric. Sc., Moshtohor,
Vol. 43(4):1723-1733, (2005).

PHYSICAL, CHEMICAL CHARACTERISTICS AND RHEOLOGICAL BEHAVIOR OF DATE PULP

BY

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ABSTRACT

A fine hemogenated date plup was produced and the method used was recorded as a Patent No. 22040 (2002). Sewi and Shamia date fruits were used for this purpose. The processing of date fruits into pulp changed the chemical characteristics of the fruits. Where total and reducing sugars, tannins, hydroxy methyl furfural were increased, while tannins, protein, crude fiber, total phenols and pH values were decreased.

Rheological characteristics were measured by Brookfield Digital Viscometer at temperatures of 5 – 70 °C. Three rheological models (Bingham plastic, IPC paste and Power Law) were used to calculate the relationship between shear stress and shear rate. The date pulp behaved as a non-Newtonian fluids (pseudoplastic behavior) at different TSS contents. The effect of temperature on viscosity was very well correlated with the Arrhenius equation (r^2 > 0.92). Activation energy for the viscous flow was in the range of 9969.80 – 18568.55 KJ/ Kmole and 10248.43 – 12676.97 KJ/ Kmole in case of Sewi and Shamia date pulp, respectively, at the same concentration. Shamia date pulp had lower 10 RPM viscosity values than Sewi date pulp and consequently had higher flow rate.

Key words: date fruit, pulp, rheological parameters, food industries

INTRODUCTION

Date palm (*Phoenix dactylifera* L.) is one of the oldest cultivated tree crops and one of the important fruit trees in some countries in the world, especially in Islamic and Arabian countries.

Egypt lies in the first largest date producer among world countries. Egyptian production represents about 18% of the total production of date in the world, which reaches about 6.2 million Tons (FAO, 2002).

Date fruits pass through four distinct stages of ripening; Kimri, Khalal, Rutab and Tamr; which are used to represent the immature green, the mature full colored, the soft brown and the hard raisin-like stage of development,, respectively. Date cultivars are classified on the basis of the texture of the ripe fruit into three generally accepted categories soft, semi-dry and dry types (Selim *et al.*, 1970 and Barreveld, 1993).

Date fruits are good source of carbohydrate, fibers, vitamins (A, B1, B2 and niacin), and minerals (potassium, calcium, selenium, fluorine and iron) and it contain a fair amount of protein, copper, magnesium, sulphur and phosphorus (Nixon and Carpenter, 1978; Yousif *et al.*, 1982a; Khatab *et al.*, 1983; Nour and Magboul, 1985; Barreveld, 1993; Ahmed *et al.*, 1995 and El-Samahy and Co-Workers, 1998-2001).

Most of produced dates are consumed directly at their Khalal, Rutab and Tamr stages, with little or no further processing. Recently, the date producing countries gave more attention to the improvement and development of date processing. Many products such as date syrup, vinegar, alcohol and liquid sugar are successfully marketed (Yousif *et al.*, 1987). Several studies have been carried out to incorporate dates in different food products such as bakery items, date bars, beverages, jam, jelly, sheets and date kutter (Yousif *et al.*, 1982b; Sumainah and Al-Nakhal, 1984; Al-Ogaidi *et al.*, 1986; Yousif *et al.*, 1996; Mostafa *et al.*, 2002 and El-samahy *et al.*, 2003).

Processing date fruits into date paste is a way to preserve the fruits and to reduce transportation and storage costs. It also, resulted in an availability of the fruit paste during the whole year and could open new outlets for date fruits. Date paste is rich in sugars, dietary fiber, minerals and trace elements. Thus, incorporation of dates into other food items could contribute significantly to their nutritive value (Yousif *et al.*, 1991).

Generally traditional methods are used to prepare date paste, especially from the soft or semi-dry varieties. The traditional methods include washing, peeling and destoning of soft date fruits or washing, destoning, soaking in tap water for semi-dry date fruits and then macerated using meat grinder and the fineness of the product can be regulated by the use of different size holes in the discs (Mustafa *et al.*, 1986; Barreveld, 1993 and El-Dieb, Samia, 2002). The date paste produced by these methods is unhomogenated and coarse.

Rheology of foods is crucial for improving the quality of food products and as many problems encountered in manufacturing including mixing, pumping and atomization during spray drying. Most concentrated structured fluids exhibit strong viscoelastic behavior at small deformations and their measurement is very useful as a physical probe of the microstructure (Ahmed and Ramaswamy, 2005) and in processing equipment design, products development, storage and transportation and quality control of juices and purees (Oomah *et al.*, 1999). The rheological behavior of fruit juices and concentrates is influenced by their composition, especially type of fruit and the treatment performed in its technological process (Ibarz *et al.*, 1996). In addition, factors such as temperature and concentration influence rheological properties of these products (Rao *et al.*, 1984; Khalil *et al.*, 1989; Ibarz *et al.*, 1992 and 1994). Presence of pectin substances or/and suspended solid particales cause non-Newtonian behavior of juices and concentrates.

Ahmed and Ramaswamy (2005) investegated the viscoelastic properties and visual color degradation kinetics of date paste at selected temperatures. They found that complex viscosity (η^*) of date paste decreased with temperature and followed a power law type relationship with frequency. They added that date paste was classified as weak gel based on the overall mechanical spectra. Variation of color during thermal processing of date paste followed the first-order reaction kinetics.

The objective of the present work was to produce a fine homogenated date pulp from second quality semi-dry and dry date varieties and evaluate the chemical and physical characteristics, minerals and rheological behavior of the resultant pulp.

MATERIALS AND METHODS

Materials

Sewi; semi-dry variety; date fruits at Tamr stage were obtained from a private orchard at Fayoum Governorate. Shamia; dry variety; date fruits were purchased from the local market at Aswan Governorate at Tamr stage.

Methods

Preparation of date pulp

Sewi or Shamia date fruits were sorted, washed, destoned, and then the date pulps were produced according to the Patent No. 22040 of El-Samahy *et al.* (2002).

Methods of analyses

Moisture content, total soluble solids (°Brix), pH value, reducing and total sugars, crude protein, crude fiber, ash, and tannins were determined according to AOAC (1990). Contents of some minerals (Na, K, Zn, Cu, Mn, Pb and Hg) were determined according to AOAC (1990) by wet ashing and using Atomic Absorption Spectrophotometer (Varian AA20). Total phenols were determined by using Folin-Ciocalteu reagent as described by DevChoudhury and Goswami (1983). Color index at 420 nm and hydroxymethyl furfural were determined as described by Ranganna (1977).

Rheological properties of produced pulps were carried out by the Brookfield Digital Rheometer model DV-III+. The Brookfield small sample adapter and Sc4-14 spindle were used. The data were analyzed by using the Bingham plastic, IPC paste and Power Law mathematical models to provide a numerically and graphically analysis of the behavior of data sets (Hegedusic *et al.*, 1995). These models are:

$\tau = \tau_o + \eta\gamma$ $\eta = KR^n$ $\tau = K\gamma^n$, respectively;

where: τ = sheer stress (N m^{-2}), τ_o = yield stress, shear stress at zero shear rate (N m^{-2}), η = plastic viscosity (mPa.s) for Bingham and 10 rpm viscosity (mPa.s) for IPC paste, γ = shear rate (sec^{-1}), K = consistency multiplier (mPa.s) for IPC paste and K = consistency index (mPa.s) for Power Law, R = rotational speed (rpm), n = shear sensitivity factor for IPC paste and flow index for Power Law. Activation energy and the effect of temperature on viscosity were calculated using Arrhenius-type equation as mentioned by Ibarz *et al*., (1996). The equation is:

$\eta = \eta_\alpha \exp(E_a/RT)$, where: η is the viscosity, η_α is a constant, E_a is the activation energy, R is the gas constant and T is the absolute temperature in °K.

Statistical analysis

The analysis of variance (ANOVA) and LSD were performed as described by Ott (1984).

RESULTS AND DISCUSSION

Physical and chemical characteristics of date fruits and pulp

Physical and chemical composition of Sewi and Shamia date fruits and their pulps are shown in Table (1). It was noticed that, processing date fruits into date pulp changed the most physical and chemical characteristics of date fruits. The protein, crude fiber, ash and total phenols contents decreased. The pH values decreased from 7.26 and 5.73 to 5.33 and 5.02 for Sewi and shamia date fruits and their pulps, respectively. This may be attributed to the liberation of organic acids from fruit tissues (Hassan and El-Sheemy, 1989). A marked increase was observed in the contents of total and reducing sugars. Where, total sugars increased from 85.94 and 75.63% for Sewi and Shamia fruits to 87.91 and 90.86% for their pulps, respectively. Also, reducing sugars increased from 45.18 and 45.52% before processing to 66.13 and 53.17% after processing of Sewi and Shamia fruits, respectively. This may be due to the hydrolysis of polysaccharides (such as pectin, crude fiber) to simple sugars during processing. The hydroxymethyl furfural (HMF) contents were increased in the produced pulps than in fruits. This may be due to the sugar carmalization and non-enzymatic browning occurred during process. Color index value of the processed Shamia pulp was significantly higher than that of Sewi date pulp. This may be attributed to the higher concentration of Shamia pulp (45° Brix) than Sewi pulp (35 °Brix), processing conditions and to a little bit to the higher concentration of HMF of Shamia fruits.

Regarding the contents of some elements, the present data indicated that K contents significantly decreased from 568.36 and 669.67 mg $100g^{-1}$ for Sewi and Shamia date fruits to 530.38 and 625.42 mg 100 g^{-1} for their pulps, respectively. The Na, Zn, Mn and Cu contents slightly decreased by processing.

From the obtained results it could be concluded that, processing date fruits to pulp have no negative effects on the nutritional values of date fruits. But, it converts the fruits to pleasant and untraditional form to consume it. The produced pulp could be used as an intermediate food product in various food formulation such as functional and baby foods, pulpy juices, biscuits, extrudates, sheets and ice cream (Abd El-Hady *et al*, 2002 and Youssef, 2004).

Table (1): Physical and chemical*♀ characteristics and some elements content of date fruits and its pulps

Components	Sewi date		Shamia date	
	Fruit	Pulp	Fruit	Pulp
Moisture content, %	47.14[b]	62.10[a]	11.20[b]	52.30[a]
TSS, °Brix	nd	35.00	Nd	45.00
pH value	7.26[a]	5.33[b]	5.73[a]	5.02[b]
Total sugars, %	85.94[b]	87.91[a]	75.63[b]	90.86[a]
Reducing sugars, %	45.18[b]	66.13[a]	45.52[b]	53.17[a]
Non-reducing sugars, %	40.76[a]	21.78[b]	30.11[b]	37.69[a]
Crude protein, %	3.31[a]	3.04[b]	3.78[a]	3.67[a]
Crude fiber, %	2.84[a]	2.71[b]	2.76[a]	2.54[b]
Ash, %	2.18[a]	2.05[b]	2.34[a]	1.70[b]
Total phenols, %	0.80[a]	0.47[b]	0.82[a]	0.88[a]
Tannins, %	0.38[b]	0.43[a]	0.36[b]	0.75[a]
Color index, (O.D. at 420nm)	nd	0.536	nd	0.822
HMF, (mg $100g^{-1}$)	1.85[b]	55.19[a]	2.10[b]	66.71[a]
Elements, (mg $100g^{-1}$)				
K	568.36[a]	530.38[b]	669.67[a]	625.42[b]
Na	80.12[a]	75.75[a]	67.58[a]	64.46[a]
Zn	0.20[a]	0.19[a]	0.36[a]	0.34[a]
Mn	0.66[a]	0.62[a]	0.99[a]	0.94[a]
Cu	0.62[a]	0.59[a]	0.42[a]	0.39[a]
Pb	-	0.00	-	0.00
Hg	-	0.00	-	0.00

*Calculated on dry weight basis, nd = not determined, ♀ Means of triplicates
Means having the same letter within each property for the same variety are not significantly different at $p \leq 0.05$

Rheological behavior of date pulps

The rheological behavior of Sewi and Shamia date fruit pulps were studied at different total soluble solids and temperatures within the range 5–70°C. The obtained results are recorded in Table (2) for Sewi pulp and in Table (3) for Shamia pulp.

From the given data measured at different temperatures, it appeared that the relationship between shear stress – shear rate was non-linear, which related to non-Newtonian behavior. These pulps were pseudoplastic fluids due to a complex interaction between soluble pectin, organic acids and soluble solids as described by Rao (1987).

Tables (2) and (3) include the rheological properties of Sewi and Shamia date pulps, respectively. The consistency index (k) mPa.s^n, flow index (n), plastic viscosity (η) mPa.s, yield stress (τ_0) N m^{-2}, viscosity at 10 RPM (mPa.s), shear sensitivity (N_1), and confidence of fit (%) were calculated at 15, 25 and 35° Brix for Sewi pulp and at 15, 25, 35 and 45° Brix for Shamia pulp.

Table (2): Rheological parameters of Sewi date pulp determined at different TSS, temperatures and models

TSS	Temp. °C	Power law K	Power law n	Power law Confi-dence, %	Bingham η	Bingham T_0	Bingham Confi-dence, %	IPC paste 10 RPM viscosity	IPC paste N_1	IPC paste Confi-dence, %
15° Brix	5	60.5	0.49	95.2	29.1	2.64	94.9	195.0	0.51	95.2
	10	54.6	0.49	94.8	23.7	2.15	94.6	158.8	0.51	94.8
	30	49.2	0.43	90.8	17.3	2.13	90.5	154.7	0.57	90.8
	50	42.4	0.43	87.5	12.6	1.67	87.0	118.4	0.57	87.5
	70	34.4	0.40	84.4	8.3	1.27	83.7	90.1	0.60	84.4
25° Brix	5	1776	0.33	96.5	434.1	35.0	95.6	6425	0.67	96.5
	10	1628	0.24	89.5	249.3	32.3	89.4	6220	0.76	89.5
	30	1525	0.22	92.7	186.4	25.5	93.1	5163	0.78	92.7
	50	1366	0.18	89.9	122.4	18.9	90.9	4077	0.82	89.9
	70	1275	0.07	87.9	26.5	16.5	87.9	3747	0.93	87.9
35 °Brix	5	6826	0.38	93.7	2398	164.5	93.1	28713	0.62	93.7
	10	6628	0.37	92.6	1973	142.1	91.9	24831	0.63	92.6
	30	6390	0.27	89.4	1137	127.5	89.2	24069	0.73	89.4
	50	5977	0.21	86.4	673.4	106.5	86.2	21255	0.79	86.4
	70	5495	0.19	83.7	530	86.7	83.9	17852	0.81	83.7

K= Consistency index (mPa.s^n), n= Flow behavior index (dimensionless), η= Plastic viscosity (mPa.s), τ= Yield stress (Nm^{-2}), 10 RPM viscosity, (mPa.s), N_1= Shear sensitivity (dimensionless)

The consistency index values increased with increasing the total soluble solids and decreased with increasing temperature. The consistency index values were 60.5, 1776 and 6826 mPa.s^n for 15, 25 and 35° Brix of Sewi pulp and 20.8, 567.1, 6758 and 48043 mPa.s^n for 15, 25 35 and 45° Brix of Shamia pulp at 5 °C, respectively. While, the values were 6826, 6628, 6390, 5977 and 5495 mPa.s^n for 35° Brix Sewi pulp and 48043, 31116, 22591, 18451 and 14014 mPa. s^n for 45° Brix Shamia pulp at assayed temperatures, respectively.

The flow index (n) values decreased with increasing temperature and total soluble solids. These results are in agreement with those obtained by Crandall *et al.*, (1982); Bahlol, (2000) and Juszczak and Fortuna (2003). Ibarz and Pagan (1987) found that the consistency index (k) and flow index (n) decreased as the temperature rose. Also, K increased with increase in soluble solids content, whereas (n) tended to decrease.

Concerning plastic viscosity (η) and yield stress (τ_0), results showed that η and τ_0 values decreased with temperature increase, while it increased with increasing total soluble solids. Where, it decreased from 2398 mPa.s and 164.5 N m^{-2} for 35° Brix Sewi pulp at 5 °C to 530 mPa.s and 86.7 N m^{-2} at 70 °C, respectively. And it increased from 29.1 mPa.s and 2.64 N m^{-2} for 15° Brix Sewi pulp at 5°C to 2398 mPa.s and 164.5 N m^{-2} for 35° Brix Sewi pulp at the same temperature. The same trend was observed for Shamia pulp (Table, 3). The obtained data are similar to that reviewed by Ahmed and Ramaswamy (2005) who found that complex viscosity (η*) of date paste decreased with temperature increase.

Table (3): Rheological parameters of Shamia date pulp determined at different TSS, temperatures and models

TSS	Temp. °C	Parameters for different models								
		Power law			Bingham			IPC paste		
		K	n	Confi-dence, %	η	T_0	Confi-dence, %	10 RPM viscosity	N_1	Confi-dence, %
15° Brix	5	20.8	0.65	98.0	21.3	0.99	97.2	75.9	0.35	98.0
	10	17.5	0.62	96.8	19.4	1.02	95.9	75.7	0.38	96.8
	30	17.5	0.56	92.9	13.0	0.90	92.6	65.7	0.44	92.9
	50	16.9	0.47	87.3	8.56	0.88	87.3	63.8	0.53	87.3
	70	16.7	0.42	81.1	4.78	0.67	80.3	46.6	0.58	81.1
25° Brix	5	567.1	0.43	93.3	270.3	15.3	92.4	2560	0.57	93.3
	10	462.7	0.44	92.8	234.7	12.8	91.9	2120	0.56	92.8
	30	334.0	0.40	79.9	127.5	9.55	77.9	1463	0.60	79.9
	50	323.3	0.37	77.0	92.3	6.01	73.6	1205	0.63	77.0
	70	171.2	0.29	79.0	58.7	6.94	78.2	821.9	0.71	79.0
35° Brix	5	6758	0.30	97.7	1424	138.8	95.6	25602	0.70	97.7
	10	6216	0.31	97.6	1409	130.5	95.6	23890	0.69	97.6
	30	4339	0.30	90.0	921.4	90.6	89.5	16481	0.70	90.0
	50	3464	0.24	84.2	475.6	64.9	84.1	12163	0.76	84.2
	70	2916	0.22	79.9	297.1	53.5	79.1	9880	0.78	79.9
45 °Brix	5	48043	0.23	98.3	16665	527.9	98.1	125735	0.77	98.3
	10	31116	0.17	96.7	14089	418.6	94.5	98614	0.83	96.7
	30	22591	0.18	88.9	7972	238.5	87.0	72754	0.82	88.9
	50	18451	0.13	84.6	3977	198.7	82.6	55431	0.87	84.6
	70	14014	0.08	76.8	3568	162.9	73.5	39387	0.92	76.8

K= Consistency index (mPa.s^n), n= Flow behavior index (dimensionless), η= Plastic viscosity (mPas), T_0= Yield stress (N/m^2), 10 RPM viscosity, (mPa.s), N_1= Shear sensitivity (dimensionless)

Regarding viscosity at 10 RPM, it also increased with the increment of total soluble solids and decreased with temperature increasing. The values were 195.0 and 75.9 mPa.s at 5°C for 15° Brix Sewi and Shamia pulp, respectively, increased to 28712 and 25602 mPa.s for 35 °Brix Sewi and Shamia pulp at the same temperature, respectively. And it decreased to 17852 and 9880 mPa.s for 35° Brix Sewi and Shamia pulp at 70 °C, respectively. The viscosity is a function of temperature and the dissolved solid concentration. As the temperature is increased, the intermoleculer distances increase and therefore the viscosity will decrease for this main reason (Bayindirli, 1992). Shear sensitivity (N_1) values increased by increasing temperature and total soluble solids. These results are in agreement with those found in many literatures for fruit juices and purees.

Generally, it could be noticed that, rheological parameters; K, η, τ_0 and 10 RPM viscosity; for Shamia date pulp were lower than those for Sewi date pulp at the same total soluble solids content and temperature. This may be referred to the type of fruits and their composition (Ibarz *et al.*, 1996). The Sewi pulp had higher reducing sugars and crude fiber contents (66.13 and 2.71%, respectively) than Shamia pulp (53.17 and 2.54%, respectively) as shown in Table (1). Presence of these components cause significant effects on rheological behavior of fruit juices and purees (Guerrero and Alzamora, 1998).

Effect of temperature on the viscosity of date fruit pulp

The change in apparent viscosity of date pulps with temperature can be described by an Arrhenius – type equation. The parameters of this equation (activation energy E_a and viscosity constant η_α) are shown in Table (4).

Table (4): Parameters of the Arrhenius model for the effect of temperature (5 – 70°C)on Sewi and Shamia date pulps

Date pulp, °Brix	Activation energy, E_a (KJ/Kmol)	Constant, η_α (mPa.s)	Coefficient of correlation (r^2)
Sewi, 15	9969.80	-0.365	0.92
25	10428.75	-0.608	0.85
35	18568.55	+4.591	0.98
Shamia, 15	10248.33	-0.875	0.95
25	11776.52	+1.464	0.95
35	12676.97	+3.131	0.98
45	14556.85	+4.782	0.98

* Data calculated for apparent viscosity at 100 RPM, except for Shamia 45 °Brix at 20 RPM

The activation energy (Ea) values were 9969.80, 10428.75 and 18568.55 KJ/Kmol for 15, 25 and 35° Brix Sewi pulp, and ηα values were –0.365, -0.608 and 4.591 mPa.s, respectively. While, the Ea values were 10248.33, 11776.53, 12676.97 and 14556.85 KJ/Kmol for 15, 25, 35 and 45° Brix Shamia pulp and ηα values were –0.875, 1.464, 3.131 and 4.782 mPa.s, respectively. These results are in agreement with those obtained by Crandall *et al.*,(1982) and Vitali and Rao (1984a,b), they reported that Ea increased with increasing pulp content and total sugars.

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الخصائص الطبيعية- الكيميائية والريولوجية للب البلح

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تم تصنيع لب ناعم ومتجانس من البلح، وقد تم تسجيل الطريقة المستخدمة في التصنيع كبراءة اختراع برقم ٢٢٠٤٠ لسنة ٢٠٠٢م. استخدم لهذا الغرض صنفين من البلح هما السيوي والشامية.

أدت عملية التصنيع الي حدوث بعض التغيرات في الخصائص الكيميائية حيث ازدادت نسب كل من السكريات الكلية والمختزلة و الهيدروكسي ميثيل فيرفيورال، وانخفضت نسب كل من البروتين، الألياف الخام، الفينولات الكلية وكذلك رقم الحموضة.

تم تقدير الخصائص الريولوجية للب الناتج عند درجات حرارة من ٥– ٧٠°م باستخدام جهاز بروكفيلد. وتم تحليل البيانات المتحصل عليها باستخدام ثلاث انماط من المعادلات لتوضيح العلاقة بين اجهاد القص ومعدل القص هي: بنجهام، **IPC paste** وقانون الأس) وتم تقدير الثوابت الريولوجية ذات الأهمية التطبيقية في التصنيع الغذائي واتضح من خلالها ان لب البلح يسلك السلوك غير النيوتيني (البلاستيكي الكاذب مع اجهاد خضوع) عند درجات التركيز المستخدمة.

كما أمكن دراسة تأثير درجة الحرارة علي اللزوجة باستخدام معادلة أرهينيوس. وأوضحت النتائج المتحصل عليها من خلال المعادلة أن للحرارة تأثير واضح علي لزوجة اللب الناتج حيث كانت قيمة r^2 أكبر من ٠,٩٢. وتراوحت قيم طاقة التنشيط من ٩٩٦٩,٨٠ – ١٨٥٦٨,٥٥ كيلو جول/ كيلو مول و ١٠٢٤٨,٤٣ – ١٢٦٧٦,٩٧ كيلو جول/ كيلو مول في حالة لب البلح السيوي والشامية علي التوالي عند نفس التركيز. كذلك وجد أن لزوجة لب الشامية عند ١٠ لفات أقل منها عما في البلح السيوي عند نفس التركيز.