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SOME PHYSICAL, CHEMICAL AND RHEOLOGICAL CHARACTERISTICS OF WATERMELON JUICE, CONCENTRATE AND SYRUP

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ABSTRACT

Watermelon is a rich natural source of lycopene, a carotenoid of great interest because of its antioxidant activity and potential health benefits. Watermelon is consumed mainly in its whole or fresh-cut form. There are little works on processing of watermelon into different products. Our objective was to produce watermelon juice, concentrate and syrup and study the physical, chemical and rheological properties of these products.

The results showed that watermelon juice had a low acidity and high lycopene (2.75 mg $100g^{-1}$) content and non-Newtonian flow behavior. Concentration process led to decrease the lycopene content to 1.37 mg $100g^{-1}$ for 23.0 °Brix concentrate, but it increased with concentration to 3.03 mg $100g^{-1}$ for 56.0 °Brix concentrate. The acidity, pectins and color index value of watermelon juice significantly increased with concentration. Concentration process and syrup production had a pronounced effect on the color attributes of the products. Watermelon concentrates had non-Newtonian flow behavior (pseudoplastic). Plastic viscosity, consistency coefficient and viscosity at 10 rpm values increased with concentration, while E_a decreased.

Keywords: Watermelon juice, concentration, rheological properties, physical properties, chemical properties, syrup, lycopene.

INTRODUCTION

Watermelon is highly nutritious and packed full of the phytochemical lycopene. It's one of the few foods that contain it in large amounts. Watermelons contain as much or more lycopene than tomatoes, but have been little studied as a source of lycopene (Perkins-Veazie *et al.*, 2003). The mean lycopene concentration of watermelon (4868 µg 100 g^{-1}) is about 40% higher than the year-round mean for raw tomato (3025 µg 100 g^{-1}) (Holden *et al.*, 1999). Also, watermelon is an excellent source of vitamin C and a very good source of vitamin A notably through its concentration of beta-carotene (Cho *et al.*, 2004).

Lycopene is a red pigment that occurs naturally in certain plant and algal tissues. In addition to giving watermelon his color, it is also thought to act as a powerful antioxidant. Lycopene scavenges reactive oxygen species, which are aggressive chemicals always ready to react with cell components, causing oxidative damage and loss of proper cell function. Scientists have found that lycopene in the diet correlates with reduced incidence of certain types of cancer such as prostate cancer, breast cancer, endometrial cancer, lung cancer and colorectal cancer (Burney *et al.*, 1989; Helzlsouer *et al.*, 1989; VanEenwyk *et al.*, 1991; Giovannucci *et al.*, 1995 and Edwards *et al.*, 2003). Lycopene levels in fat tissue have been linked with reduced risk of myocardial infarction and heart attack (Kohlmeier *et al.*, 1997 and Klipstein-Grobusch *et al.*, 2000).

The nutritional composition of watermelon was reported by Gebhardt and Thomas (2002). The mean values (g 100 g-1) were: 92 water, 7.3 carbohydrates, 0.7 protein, 0.3 fat and 0.3 ash. The mean values for some minerals and vitamins (mg 100 g-1) were: 8.0, 0.2, 2.0, 116.0, 9.0, 0.2, 0.08, 0.02 for Ca, Fe, Na, K, ascorbic acid, niacin, thiamine, riboflavin, respectively and 366 IU for vitamin A.

There are many watermelon cultivars widely grow in Egypt such as Giza 1, Giza 21, Aswan hybrid and many seedless hybrids. The annual production reached about 1.5 million tons (FAO, 2005).

The consumption of watermelon has risen steadily in recent years; it increased about 60% between 1980 and 2000. However, currently it is consumed mainly in its whole or fresh-cut form. There is a need to develop processing methods for watermelon juice/ concentrate that have minimal effect on physico-chemical characteristics, lycopene degradtion and sensory quality (Siddiq *et al.*, 2005).

However, studies on the processing of watermelon are still limited. The aim of the present work was to study some of physical, chemical and rheological properties of watermelon juice, concentrate and syrup and standpoint the effect of processing on these properties.

MATERIALS AND METHODS

1. Materials

Ten ripe watermelon fruits (*Citrullis lanatus* cv. Aswan hybrid) were purchased from a local market at Ismailia governorate, Egypt. The fruits weighted about 5-8 Kg.

2. Methods

2.1. Preparation of watermelon juice, concentrate and syrup

The fruits were washed and manually peeled and cut into small pecies, then pulped (Moulinex blender, type 741, France) and the seeds were screened and removed using a clean muslin cloth.

The produced juice was divided into three parts. The first part was immediately analyzed for physical, chemical and rheological properties.

The second part was concentrated using the rotary evaporator (Rotavapor, BUCHI, Laboratoriums-Technik AG, Switzerland) at 50-55 °C under vacuum to obtain watermelon concentrates of 23.0, 40.0 and 56.0 °Brix.

The third part was used to produce the watermelon syrup according to the steps included in the Egyptian Standard (1986) for syrup production and the final concentration was 57.0 °Brix.

2.2. Methods of analyses

Total soluble solids (°Brix), pH value, titratable acidity (as citric acid) and fractions and total pectin contents were determined according to AOAC (1990). Color index at 420 nm and lycopene content were determined as described by Ranganna (1977). Carotenoids were assayed and calculated according to the method described by Wettestein (1957).

2.3. Color attributes measurements

Color attributes; lightness (L^*), redness (a^*) and yellowness (b^*) were performed using a Minolta Color Reader CR-10 (Minolta Co. Ltd., Osaka, Japan). The color intensity (C^*) was calculated as $C^* = (a^{*2} + b^{*2})^{0.5}$. Furthermore, the hue angle (h_{ab}) was calculated as $h_{ab} = \tan^{-1}(b^*/a^*)$, where $h_{ab} = 0°$ for a red hue and $h_{ab} = 90°$ for a yellow hue (RØrå and Einen, 2003). Whiteness Index (WI) was expressed as:

$$WI = 100 - [(100 - L^*)^2 + a^{*2} + b^{*2}]^{0.5}$$ (Bolin and Huxsoll, 1991).

2.4. Rheological measurements and activation energy calculation

Rheological properties of watermelon juice, concentrate and syrup were carried out by the Brookfield Digital Rheometer model DV-III+. The Brookfield small sample adapter and $Sc_{4\text{-}14}$, $Sc_{4\text{-}21}$ spindles were used. The data were analyzed 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:
$\tau = \tau_o + \eta\gamma$, $\eta = KR^n$ and $\tau = K\gamma^n$, respectively; where: τ = shear 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.*, (1996a). The equation is: $\eta = \eta_\alpha \exp(E_a/RT)$, where: η is the viscosity, $\eta\alpha$ is a constant, E_a is the activation energy, R is the gas constant and T is the absolute temperature in °K.

3. Statistical analysis

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

RESULTS AND DISCUSSION

1. Physical and chemical properties of watermelon juice, concentrates and syrup

Results presented in Table (1) showed the physical and chemical properties of watermelon juice and its products (concentrate and syrup). It was noticed that, the total soluble solids (TSS) of watermelon juice was 7.0 °Brix. The watermelon juice had 5.28 pH value, 0.056% total acidity (as citric acid), 0.955% total pectin, 0.41 mg L^{-1} and 2.75 mg 100 g^{-1} of carotenoids and lycopene, respectively. The color index value of watermelon juice was 0.105. Perkins-Veazie *et al.* (2003) found that the watermelon cultivars exhibited a range of lycopene values, with seeded and seedless red fleshed types having 3.6 to 7.8 mg 100 g^{-1} lycopene. Storage of whole or cut melons for 2 to 10 days reduced lycopene by 6 to 10%. Also, Edwards *et al.* (2003) found that the watermelon juice collected from six samples of fruits contained 2.6 mg lycopene, 0.32 mg β-carotene, 0.12 mg phytoene and 0.06 mg phtofluene per 100g sample.

Table (1): Physical and chemical properties of watermelon juice, concentrates and syrup (wet weight basis)

Property	Watermelon				
	Juice	Concentrate 1	Concentrate 2	Concentrate 3	Syrup
TSS (°Brix)	7.0^{e}	23.0^{d}	40.0^{c}	56.0^{b}	57.0^{a}
pH value	5.28^{a}	4.82^{b}	4.74^{c}	4.70^{c}	3.60^{d}
Acidity, % (as citric acid)	0.056^{e}	0.388^{c}	0.484^{b}	0.621^{a}	0.223^{d}
Pectins, %					
Total	0.995^{d}	3.190^{c}	4.338^{b}	5.116^{a}	nd
Water-soluble	0.237^{d}	0.822^{c}	1.338^{b}	1.630^{a}	nd
Oxalate-soluble	0.567^{d}	2.028^{c}	2.356^{b}	2.662^{a}	nd
Acid-soluble	0.191^{d}	0.340^{c}	0.644^{b}	0.824^{a}	nd
Color index (O.D. at 420 nm)	0.105^{d}	0.314^{b}	0.534^{a}	0.617^{a}	0.201c
Carotenoids (mg L^{-1})	0.410^{d}	0.490^{c}	0.630^{b}	0.841^{a}	0.339^{e}
Lycopene (mg 100 g^{-1})	2.75^{b}	1.37^{d}	1.72^{c}	3.03^{a}	1.81^{c}

Means of triplicates **nd= not determined**

Means having the same letter within each property are not significantly different at $p \leq 0.05$

Production of fruit juice concentrates offers important advantages. They can be used as ingredients in many products such as ice cream, fruit syrup, jellies, fruit juices blends and carbonated beverages. Furthermore, fruit juice concentrates have a higher stability than juices, because of their low water activity (Giner *et al.*, 1996). As a result of concentration process, all the studied parameters significantly changed. The pH value significantly decreased from 5.28 for watermelon juice to 4.82, 4.74 and 4.70 for watermelon concentrates having 23.0, 40.0 and 56.0 °Brix of TSS, respectively. While, the total acidity content significantly increased from 0.056% for the juice to 0.621% for 50.5 °Brix concentrate.

Concerning the total pectin content, the results in Table (1) showed that the total pectin content gradually enhanced by concentration. It significantly increased from 0.955% for the juice to 3.190, 4.338 and 5.116% for concentrates containing 23.0, 40.0 and 56.0 °Brix of TSS, respectively. Pectin fractions (water-soluble, oxalate-soluble and acid-soluble) content significantly increased by concentration. The pectin fractions content must be taken into account during processing since their polymer interactions play the major role in the flow behavior of fruit products (VanBuren, 1991). Especially, pectin extracted with hot chelating agents (oxalate-soluble fraction), where they are presumed to be present in the form of a calcium pectate gel (Selvendran and O'Neill, 1987).

The development of color is an evident and extremely important indicator of the extent of the advanced Maillard reaction (Nursten, 1986). The colors produced range from pale yellow to very dark brown, depending on the type of food and extent of the reaction (Morales and VanBoeckel, 1999). The color index value of watermelon juice significantly extended by concentration. It increased from 0.105 for the juice to 0.314, 0.534 and 0.617 for concentrates containing 23.0, 40.0 and 56.0 °Brix of TSS, respectively.

As a result of concentration process, carotenoids content significantly increased. While, the lycopene content decreased from 2.75 mg 100 g^{-1} juice to 1.37 mg 100 g^{-1}conccentrate having 23.0 °Brix of TSS. Then it significantly increased by concentration to 40.0 and 56.0 °Brix (1.72 and 3.03 mg 100 g^{-1}, respectively). Siddiq *et al.* (2005) found that the heating of watermelon juice produced by hot macerating at 50 °C for 30 min reduced the lycopene contents by 18%. Moderate processing did not produce carotenoid modifications, but a prolonged heating conduces to total destruction of epoxycarotenoids. It was shown that under prolonged heating conditions only the most sensitive carotenoids are highly destroyed (Granado et al., 1992; Khachik et al., 1992 and Delgado-Vargas et al., 2000).

Production of watermelon syrup (57.0 °Brix) led to reduce the pH value (3.60) and increase the total acidity content (0.223%) of resultant syrup than that for the juice as a result of adding citric acid during process (Table, 1). The color index value of resultant watermelon syrup (0.201) was higher than that for the juice (0.105). This may be referred to the Maillard reaction or sugar carmalization during processing (Friedman, 1996). Also, watermelon syrup contained lower lycopene content (1.81 mg 100 g^{-1}) than the juice. This may be due to the effect of heat treatment during processing the syrup.

2. Color attributes of watermelon juice, concentrates and syrup

Color considers an important sensory and quality attribute of foods. Maintenance of naturally colored pigments in thermally processed and stored foods has been a major challenge in food processing (Ihl *et al.*, 1998).

Results recorded in Table (2) showed that the color attributes values of watermelon juice were: lightness (L^*), 27.1; redness (a^*), 10.0; yellowness (b^*), 6.0; color intensity (C^*), 11.66; hue angle (h_{ab}), 30.96 indicating a reddish color of watermelon juice and 26.17 Whiteness Index (WI).

During concentration process, the L^* values significantly decreased from 27.1 for the juice to range of 26.0-26.8 for the three produced concentrates. This may be due to the non-enzymatic browning. The concentration process led to increase the a^*, b^* and C^* values. This wasssociated with decreasing of h_{ab} values from 30.96 for the juice to 21.41-17.35 for the three concentrates, which indicating a more reddish color of these concentrates. Whiteness Index (WI) indicates the development of white surface discoloration. The higher WI scores the more severe the white discoloration. Results presented in Table (2) showed that the WI value significantly decreased by concentration. It decreased from 26.17 for the juice to 23.69 for concentrate containing 23.0 °Brix of TSS. Then, it non-significantly decreased to 22.82 by concentration to 56.0 °Brix.

Production of watermelon syrup led to a reduction in the all color attributes values of resultant syrup than those for the juice. Where, the L^*, C^*, h_{ab} and WI values decreased to 23.3, 8.01, 24.32 and 22.88, respectively. There are many factors that govern the degradation of color and pigment during thermal processing of food products. Those include non-enzymatic and enzymatic browning, process conditions and duration and temperature of storage. Special care must be taken to produce food that retains a bright, attractive color during food processing (Meyer, 1987). Change in color during thermal processing may therefore be used as a tool to evaluate the product quality.

Table (2): Color attributes of watermelon juice, concentrates and syrup

Product	L^*	a^*	b^*	C^*	h_{ab}	WI
Juice	27.1^a	10.0^b	6.0^c	11.66^b	30.96^a	26.17^a
Concentrate (23.0 °Brix)	26.8^a	20.4^a	8.0^a	21.91^a	21.41^c	23.69^b
Concentrate (40.0 °Brix)	26.4^b	20.3^a	7.3^b	21.57^a	19.78^{cd}	23.24^b
Concentrate (56.0 °Brix)	26.0^b	20.8^a	6.5^c	21.79^a	17.35^d	22.82^b
Syrup	23.3^c	7.3^b	3.3^d	8.01^c	24.33^b	22.88^b

Means of 5 readings
Means having the same letter within each property are not significantly different at $p \leq 0.05$
L^* = lightness, 0 = black, 100 = white; + a^* = red, -a^* = green; +b^* = yellow, -b^* = blue; C^*= color intensity; h_{ab}= hue angle; WI= Whiteness Index

3. Rheological properties of watermelon juice, concentrates and syrup

Knowledge of rheological properties of non-processed fluids such fluid fruits before and after concentration, is necessary for pipeline transport, processes design and product development. Even though, many studies and research work have been conducted on rheology of fruit fluids, still there are a lot of non-processed materials without characterization of their flow response.

Table (3) includes the rheological properties of watermelon juice and its products. As the viscosity is depending upon the intermolecular distances and when the TSS increase, the intermolecular distances decrease, it is normally to watch the increment in the plastic viscosity and/ or 10 rpm viscosity by increasing the concentration. Also, yield stress is related to the existence of the reticulated structure, which is generally due to the interaction between colloidal particles or the formation of links between the long chain molecules. Thus, the plastic viscosity (η) mPa.s, yeild stress (τ_o) N m^{-2}, consistency coefficient (k) mPa.s and viscosity (mPa.s) at 10 rpm values gradually increased by concentration of watermelon juice (Table 3). They increased from 1.48 mPa.s, 0.00 N m^{-2}, 0.05 mPa.s and 0.78 mPa.s for the juice to 41.1 mPa.s, 0.40 N m^{-2}, 6.17 mPa.s and 52.9 mPa.s for 56.0 °Brix concentrate, respectively. While, the flow index (n) values decreased from 1.18 to 0.93 for the same samples. This means that the samples exhibited non-Newtonian behavior. These results are in agreement with those obtained by Ibarz and Pagan (1987), Bahlol (2000) and Juszczak and Fortuna (2003). They found that the consistency coefficient (k) increased with increasing the soluble solids content, whereas flow index (n) tended to decrease. Suarez-Quintanilla *et al.* (2003) found that the consistency coefficient and yield stress of watermelon juice increased by the concentration. But, it had a weak influence on the flow index, which indicated an insignificant change in velocity profiles inside the geometry.

The plastic viscosity, consistency index and 10 rpm viscosity values of watermelon syrup were: 37.5, 4.00 and 38.6 mPa.s, respectively. The production of watermelon syrup did not affect the yield stress value of the juice, but it decreased the flow index value (0.98) as a result of increasing the TSS content.

Table (3): Rheological properties (at 20 °C) of watermelon juice, concentrates and syrup

Product	Plastic viscosity (mPa.s)	Yield .stress (N m^{-2})	*Consistency* coefficient (mPa.s)	Flow index	10 rpm viscosity (mPa.s)
Juice	1.48	0.00	0.05	1.18	0.78
Concentrate (23.0 °Brix)	2.99	0.00	0.22	1.05	2.50
Concentrate (40.0 °Brix)	9.58	0.02	1.04	0.99	10.10
Concentrate (56.0 °Brix)	41.1	0.40	6.17	0.93	52.90
Syrup	37.5	0.00	4.00	0.98	38.60

4. Effect of temperature on the viscosity of watermelon juice, concentrates and syrup

The change in apparent viscosity (at 10 rpm) with temperature (5-70 °C) can be described by Arrhenius-type equation. The results presented in Table (4) showed the Arrhenius equation parameters, activation energy (E_a) and viscosity constant (η_α) for watermelon juice, concentrates and syrup. The E_a of watermelon juice was 49464.03 KJ/Kmol.°K and the η_α is 8.7×10^{-10} mPa.s. The activation energy of flow has been related to some fundamental thermodynamic properties of the Newtonian fluids. For example ΔE_a has been found to be approximately equal 1/3 or 1/4 the heat of vaporization, depending on the shape and binding of liquid molecules (VanWazer *et al.*, 1963).

Table (4): Parameters of Arrhenius equation of watermelon juice, concentrates and syrup (temperature range 5-70 °C)

Product	E_a (KJ/Kmol °K)	η_α (mPa.s)	Coefficient of correlation (r^2)
Juice	49464.03	8.7×10^{-10}	0.98
Concentrate (23.0 °Brix)	36044.59	1.14×10^{-8}	0.93
Concentrate (40.0 °Brix)	32500.99	6.34×10^{-8}	0.92
Concentrate (56.0 °Brix)	22154.55	1.13×10^{-3}	0.99
Syrup	22912.82	4.33×10^{-3}	0.96

The concentration process led to decrease the E_a and increase the η_α values of the resultant concentrates. The E_a values decreased from 49464.03 KJ/Kmol.°K for the juice to 22154.55 KJ/Kmol.°K for concentrate containing 56.0 °Brix of TSS. While, the η_α values increased from 8.7×10^{-10} to 1.13×10^{-3} mPa.s for the same samples. Production of watermelon syrup led to decrease the E_a and increase the η_α values for the syrup than those for the juice. Ibarz *et al.* (1996 b,c) reported that E_a increases with sugar content and decreases with pulp content. On the other hand, the η_α increases with increasing in total solids and pectin content (Manohar *et al.*, 1990).

From the obtained results, it can be found that the effect of temperature on the viscosity of watermelon juice, concentrates and syrup was very well correlated with the Arrhenius equation, where the correlation coefficient (r^2) values range was between 0.92-0.99.

In conclusion, watermelon juice exhibited low acidity content, high lycopene content and non-Newtonian flow behavior. Concentration process and syrup production from watermelon juice had a pronounced effect on most quality properties of the products.

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بعض الخصائص الطبيعية، الكيميائية والريولوجية لعصير ومركز وشراب البطيخ

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يعتبر البطيخ من المصادر الطبيعية الغنية بصبغة الليكوبين الهامة جدا نظرا لتأثيرها المضاد للأكسدة وفوائدها الصحية العديدة. يستهلك البطيخ عادة في صورة طازجة غير مصنعة ويوجد القليل جدا من الأبحاث عن تصنيعه. لذا استهدف هذا البحث دراسة امكانية انتاج عصير، مركز وشراب البطيخ ودراسة بعض الخصائص الطبيعية، الكيميائية والريولوجية لهذه المنتجات.

أوضحت النتائج انخفاض حموضة عصير البطيخ مع ارتفاع محتواه من الليكوبين (٢,٧٥ مجم ١٠٠ جم$^{-1}$) كما أن سلوك انسيابه يتبع السلوك غير النيوتيني. أدت عملية التركيز الي خفض محتوى الليكوبين الي ١,٣٧ مجم ١٠٠ جم$^{-1}$ للمركز 23.0 °بركس ولكنه يزداد بزيادة التركيز الي ٣,٠٣ مجم ١٠٠ جم$^{-1}$ للمركز 56.0 °بركس. ازدادت قيم الحموضة، المحتوى من البكتين ومعامل لون العصير بزيادة التركيز. أدي التركيز وخطوات صناعة الشراب الي حدوث تغيرات واضحة في خصائص لون هذه المنتجات.

كان سلوك انسياب مركزات البطيخ غير نيوتيني (زودوبلاستيكي). ازدادت قيم اللزوجة البلاستيكية، معامل القوام واللزوجة عند ١٠ لفات بزيادة التركيز بينما انخفضت طاقة التنشيط.

يعتبر هذا العمل بداية لدراسة امكانية انتاج العديد من منتجات البطيخ، وتحسين جودة هذه المنتجات والمحافظة علي المكونات الهامة بها وتقيمها حسيا في المستقبل.