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Food Chemistry 76 (2002) 437–441

Food Chemistry

www.elsevier.com/locate/foodchem

Changes in physical and chemical properties during pomegranate (*Punica granatum* L.) fruit maturation

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Received 27 March 2001; received in revised form 17 July 2001; accepted 17 July 2001

Abstract

Physicochemical studies of pomegranate fruits (*Punica granatum*) variety Taifi, including total seed juice extracted from unripe, half-ripe and full-ripe stages are reported. Edible portion of pomegranate (57.51% of total fruit wt.) comprised 63.58% of juice and 36.21% of seeds. Fresh juice contained 84.57% moisture, 14.1% sugar, 1.05% protein and 0.33% ash. Total protein, ascorbic acid, fat and phenolic compounds in seeds were 4.06, 0.23, 0.15, 2.92%, respectively. The pH of the fruit increased with the advance in maturity, whereas ripe fruits were significantly less acidic than green unripe and half-ripe fruits. Ripe fruits had a higher percentage of glucose (53.5%) than fructose (46.6%). Polyphenols were lower in full-ripe fruits than unripe. The amounts of K, Na, Mg and Ca were highest among other minerals in the fruit. Cu, Zn and Ca contents were higher in seeds, whereas K, Na and Fe were higher in juices.

Keywords: Pomegranate; Seed; Juice; Maturity; Ascorbic acid; Polyphenols

1. Introduction

Pomegranate (*Punica granatum* L.) is one of the important commercial fruits in Saudi Arabia and generally very well adapted to the Mediterranean climate. It is cultivated in the southwestern region (Taif area) of Saudi Arabia and in many other Mediterranean countries (Kumar, 1990; Naser, 1983). Its cultivars also contribute to the reduction of the risk of desertification. The fruit is consumed directly as fresh seeds as well as fresh juice which can also be used in beverages for jellies, and flavouring and colouring agents (Ewaidah, 1987; Hodgson, 1917; La Rue, 1969). The edible part of the fruit contains considerable amounts of acids, sugars, vitamins, polysaccharides, polyphenols and important minerals. Some chemical changes during ripening on storage, (pigmentation, sugars, lipids and fatty acid composition) have been reported (Gil, Garcia-Viguera, Artes, & Tomas-Barberan, 1995; Hernandez, Melgarejo, Tomas-Barberan, & Artes, 1999; Melgarejo & Artes, 2000; Melgarejo, Salazar, & Amoros-A, 1995). Mustafa and Hobani (1993) also reported physical dimensions of different cultivars in Saudi Arabia. Due to the influence of environment and degree of maturation and cultivar differences on nutritional values of the fruit, more work is required. The aim of the present investigation was to determine the physical and physicochemical changes during different stages of maturation. A knowledge of changes in mineral contents would be very useful for determination of the fruit quality.

2. Materials and methods

2.1. Sample collection

Fresh pomegranate fruits (*Punica granatum* L.) of Taifi cultivars, from the Agricultural Research and Experimental Station of College of Agriculture, KSU, Riyadh, Saudi Arabia, were selected. Fruits were picked manually at different stages after fruit set. The fruits were classified into three different maturities, based on the subjective evaluation of the texture of the fruit and skin colour.

1. Green: hard texture and green colour.
2. Half-ripe: firm texture and light green colour.
3. Ripe: soft texture and with reddish colour.

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0308-8146/02/$ - see front matter
PII: S0308-8146(01)00301-6

2.2. Physical characteristics

Twenty fruits of each stage were individually analyzed for physical characteristics. The procedure of Mohesenin (1970) was adapted for the physical measurments. Fruits were weighed in the air on a balance of accuracy of 0.001 g. The weight density of the fruit was obtained by the ratio of weight to volume. Length and diameter of the fruit were measured with a vernier calliper and volume by a liquid displacement method. The measurement of length was made on the polar axis of fruit, i.e. between the apex and stem. The maximum width of the fruit, as measured in the direction perpendicular to the polar axis, is defined as the diameter.

2.3. Proximate analysis

Moisture content, protein, fat and ash percentage at each stage of fruit ripening were determined according to AOAC (1990) and colour was measured by using a Lovibond Tintometer (model E, England). Soluble solids were determined with a refractrometer (Erma, Tokyo). Results were reported as degree Brix at 21°. For titrable acidity, the samples were homogenized and 10 g of each sample were accurately weighed into a beaker; 40 ml distilled water were added and pH of the sample was recorded. The resulting mixture was titrated with 0.1 N NaOH to pH 8.1, monitored with a pH meter (Corning Ltd., England). Acidity was expressed as meq/100 g sample. Minerals were determined using an atomic absorption spectrometer (Perkin-Elmer, USA). All results were expressed on a fresh weight basis.

2.4. Ascorbic acid

Ascorbic acid was determined by employing the method described by Ruck (1963). Thirty-gramme portions of homogenized sample were blended with 100 ml of 0.4% oxalic acid for 2 min in a Waring blender. The blended mixture was made up to 500 ml with 0.4% oxalic acid and filtered. Filtrate (20 ml) was titrated with standard 2,6-dichlorophenol indophenol. Results were expressed as mg per 100 g on a wet weight basis.

2.5. Sugars

Glucose, fructose and sucrose were determined by HPLC (Schimadzu model, LC-10 AD, Japan), using a refractive index (RTP-6A) detector, on a 30 cm Shimpack LC-NH2 column. Sample preparation and chromatographic procedure were conducted as described in AOAC (1995) and total sugar was calculated by summation of individual sugars.

2.6. Fatty acid analysis

Fatty acid methyl esters (FAMEs) were prepared, following the procedure described by Metcalf, Schmitz, and Pelka (1966). Aliquots of lipid extract (20 mg) were saponified with 1.5 ml methanolic KOH (0.5 N) solution by refluxing for 10 min at 85°. After the addition of 4 ml BF3-etherate, the sample was boiled for 5 min. The FAMEs were extracted from a salt-saturated mixture with petroleum–ether (40–60 °C). The esters were separated by GC (Scimadzu 17 A, Japan) fitted with a capillary column (Suplecowax 10, 30 m, 0.32 id. 0.5 μm film thickness). Helium was used as a carrier gas at inlet pressure 1.2 kg/cm^2. All FAMEs were run in duplicate and compared with standard samples.

2.7. Polyphenols determination

Polyphenols were determined following the method described by Price and Butler (1977). A 60 mg portion of fruit juice was shaken manually for 60 s with 3 ml of methanol in a test tube. The mixture was filtered and the

Table 1
Physical properties of pomegranate fruits at different stages of ripening

Parameter	Stages			
	Unripe fruits[a]	Half-ripe fruits[a]	Full-ripe fruits[a]	Total (mean)
Length (L) (cm)	6.61±0.29a	6.76±0.28a	6.55±0.28a	6.64±0.31
Diameter (D) (cm)	3.54±0.15a	3.61±0.25a	3.67±0.22a	3.61±0.21
Length/diameter (L/D)	1.87±0.12	1.87±0.13	1.79±1.84	1.84±0.11
Volume (cm^3)	126.74±20.02a	161.02±48.61a	156.74±29.86a	148.16±36.82
Densities of fruit	1.29±0.2a	1.20±0.1ab	1.38±0.2b	1.29±0.16
Fruit weight (g)	163.51±22.48a	193.82±49.44ab	216.50±42.88b	191.27±44.16
Skin weight (g)	48.34±412.21a	54.43±9.0ab	69.01±15.33b	58.92±17.36
% of skin	29.56	28.08	31.87	29.84
Seed weight (g)	90.01±34.03a	111.95±31.37ab	129.27±28.21	110.41±34.10
% of seed	55.05	57.77	59.71	57.51
Juice weight (g)	59.99±17.01a	68.76±20.93ab	81.03±16.72a	59.93±19.60
% of juice (whole)	30.57	30.32	32.88	31.21

[a] Means of 20 fruits in each row followed by different letters are significantly different ($P<0.05$); ±, standard deviation.

Table 2
Physicochemical and compositional properties of pomegranate (*Punica granatum* L.) fruit juice

No.	Parameters	Stages			
		Unripe fruits[a]	Half-ripe fruits[a]	Full-ripe fruits[a]	Total
1	Red	20.64±0.78a	12.0±3.13b	10.42±0.13b	14.35±5.50
2	Yellow	5.84±2.45a	7.16±0.14a	2.96±2.5b	5.32±2.15
3	Blue	1.42±0.53b	1.96±0.32a	0.12±0.05c	1.17±0.96
4	Moisture %	86.27±0.47a	83.79±0.22b	83.65±0.4b	84.57±14.74
5	pH	3.39±0.07b	3.48±0.11ab	3.57±0.2a	3.48±0.09
6	° Brix index	16.4±0.16a	16.3±0.19b	16.9±0.07b	16.5±0.32
7	Acidity (titrable)	25.1±1.03a	21.2±2.18b	19.5±0.11b	21.9±2.88
8	Protein (%)	0.97±0.03a	1.15±0.14b	1.03±0.03b	1.05±0.09
9	Fructose (g/100 g)	6.58±0.08a	6.44±0.08a	6.66±0.28a	6.56±0.11
10	Glucose (g/100 g)	7.56±0.03a	7.26±0.09b	7.72±0.28a	7.51±0.23
11	Total sugars	14.1±0.08b	13.7±0.15c	14.6±0.49a	14.1±0.45
12	Ratio G/F	1.13	1.13	1.16	1.14±0.01

[a] Means of 20 fruits in each row followed by different letters are significantly different ($P<0.05$); ±, standard deviation.

Table 3
Chemical composition of pomegranate (*Punica granatum* L.) fruit seeds

No.	Parameters	Stages			
		Unripe fruits[a]	Half-ripe fruits[a]	Full-ripe fruits[a]	Total
1	Moisture %	79.45±0.96a	80.66±3.39a	77.72±3.36a	79.28±2.89
2	Protein (%)	3.99±0.4a	3.74±0.89a	4.45±0.68a	4.06±0.26
3	Fat (%)	0.2±0.0a	0.01±0.0a	0.25±0.3a	0.15±0.10
	Saturated	18.6±3a	17.9±3.6a	16.4± 2a	17.5±1.02
	Unsaturated	81.6±4.26a	82.1±2.1a	84.6±2.5a	82.8±17.96
4	Ascorbic aid (mg/100 g)	0.26±0.07a	0.25±0.14a	0.18±0.32a	0.23±0.11
5	Phenolic compounds (mg/100 g)	3.65±0.17a	3.22±0.72a	1.90±0.57a	2.92±0.19

[a] Means of 20 fruits in each row followed by different letters are significantly different ($P<0.05$); S.D., ± standard deviation.

tube was quickly rinsed with 3 ml of methanol. The filtrate was mixed with 50 ml water and analyzed within an hour. To 1 ml of filtrate, 3 ml of 0.1 M $FeCl_3$ in 0.1 NHCl and 3 ml of 0.008 M K_4 Fe $(CN)_6$ were added. The absorbance of the colour, developed after 30 min at 30 °C, was read at 720 nm. A standard curve was prepared, expressing the result as catechin equivalent, i.e. amount of catechin (mg/ml) which gave a colour intensity equivalent to that given by polyphenols after correction for the blank.

2.8. Statistical analyses

Data were analyzed statistically (ANOVA) using analysis of variance (Steel & Torrie, 1980) and differences among the means were determined for significance at $P<0.05$ using Duncan's multiple range test and the system programme SAS (1982).

3. Results and discussion

Physical properties of pomegranate fruit of each stage showed no statistical differences ($P<0.05$) in length, diameter or volume. However, differences were obtained in total weight, seed content and densities. These values increase from unripe, through half-ripe to full-ripe fruit, being maximum at this stage (Table 1). The edible part of the three stages comprised 55.05, 57.77 and 59.71%, respectively. It was observed that, on average, the seeds, skin and internal lamella of the three stages constituted 57.51, 29.83 and 12.65% of the weight of whole fruit, respectively. The edible portion of fruit (57.51% of total fruit weight) was 63.58% of juice and 36.21% of seed waste, which is comparable with results of Kumar (1990). Overall, the waste material was calculated as 68.74%, comprising, seeds, outer skin, and inner lamella, which was similar to the 66% reported by Veres (1976). In this study, the full-ripe fruit had the maximum seed content (59.71%), and showed a significant difference from green and ripe fruit. These differences could be attributed to metabolic changes during ripening (Narain, Bora, Holschuh, & Vasconcelos, 1992).

Physicochemical and chemical parameters of pomegranate juice are presented in Table 2. Red, yellow and blue colours of fruits showed a significant difference from unripe to full-ripe fruit juices. Most notable changes were detected in blue colour which decreased

Table 4
Ash (%) and some minerals (mg/100 g) found in pomegranate (*Punica granatum* L.) juice and seeds

No.	Parameters	Stages			
		Unripe fruits[a]	Half-ripe fruits[a]	Full-ripe fruits[a]	Total
Seed	Ash	0.46±0.09a	0.43±0.1a	0.47±0.21a	0.45±0.02
1	Cu	0.03±0.01a	0.03±0.01a	0.04±0.01a	0.03±0.01
2	Fe	0.84±0.12c	1.27±0.06b	1.88±0.14a	1.33±0.52
3	Zn	0.20±0.01b	0.30±0.19b	1.26±0.67a	0.59±0.50
4	Mg	9.87±0.45a	10.2±0.95a	11.9±2.18a	10.6±1.09
5	P	7.37±2.33b	3.91±0.36b	7.49±0.29a	6.26±2.03
6	Na	37.8±10.3a	44.5±5.95a	95.7±2.17a	59.3±31.6
7	Ca	38.2±9.27a	31.4±10.2b	59.3±8.88	43.0±14.5
8	K	309±9.27a	209±14.5b	243±21.7a	253±51.1
Juice	Ash	0.29±0.03b	0.38±0.01a	0.32±0.02b	0.33±0.01
1	Cu	0.06±0.01b	0.07±0.04a	0.07±0.00a	0.07±0.01
2	Fe	2.37±0.01a	1.99±0.00b	2.21±0.01c	2.19±0.19
3	Zn	0.22±0.00b	0.24±0.00ab	0.300±0.0c	0.25±0.04
4	Mg	7.39±0.44a	6.34±0.03b	5.13±0.05c	6.29±1.13
5	P	5.16±0.08a	6.96±0.56c	6.25±0.04b	6.12±0.91
6	Na	79.2±2.2b	76.9±0.144b	72.1±0.12a	76.1±3.59
7	Ca	26.9±0.44a	23.3±0.27a	24.5±0.23b	24.8±1.85
8	K	285±7.53c	302±5.44a	333±15.8b	307±24.4

[a] Means of 20 fruits in each row followed by different letters are significantly different ($P<0.05$); ±, standard deviation.

significantly with ripening due to increased pigmentation. Such changes in colour of pomegranate juice have been previously reported with ripening of the fruit (Ben-Arie, Segel, & Guel fat-Reich, 1984; Gil et al., 1995).

Moisture content varied significantly from green-unripe to full-ripe fruit juices while no difference was observed in seeds. On average, the pomegranate fruit juice and seeds contained 84.57% and 79.28% moisture, respectively (Tables 2 and 3). El-Nemr, Ismail, and Ragab (1992) reported a higher moisture value (85.4%) in fresh juice.

The pH of the pomegranate juice increased with maturity, being a maximum 3.57 at the full-ripe stage. pH characterized the acidic taste of juice (Cemeroglu, Artik, & Erbas, 1992). The titrable acidity and juice percentage decreased with the advance in maturity. Ripe fruits were less acidic (19.5) than both green-unripe (21.2) and half-ripe (25.1), respectively. The soluble solids (° Brix index) content (16.90) was the highest in full-ripe fruits. The Brix/acid ratio increased from 0.66 for unripe to 0.87 for ripe fruits, which is a 48.5% increment.

The mean protein contents of the juice and seeds were (Table 2) 1.05 and 4.06%, respectively (Tables 2 and 3). There were no significant changes on ripening. Similar values for the juice were reported by Morton (1987); however, El Nemr et al. (1992) reported 13.2% protein in seeds, which is higher than our result. Ripening changes of protein have been previously reported in other fruits (Al Khalifa & Dilshad, 1998; Marin & Cano, 1992). The average value of ascorbic acid of seeds (edible portion) for all the three maturities was observed 0.23 mg/100 gm (Table 3) which is similar to that found by Al Kahtani (1992). The ascorbic acid content decreased significantly with advance in maturity, being 0.26 for green-unripe, 0.25 for half-ripe and 0.15 mg/100 gm for full-ripe fruit. On average, a low percentage of fat (0.15%) was obtained, and with no significant difference at all three maturities. Results are comparable to those of Melgarejo, Salazar, and Artes (2000). Results revealed that when pomegranate fruit attained ripeness, the total and individual sugars reached maximum levels (Table 2). As a whole, total sugars and glucose showed a significant difference, whereas no significant difference was observed in fructose. Sucrose was not detected at any stage. Ripe fruit had significantly more reducing sugars 14.6 g/100 g than green-unripe 14.1 g/100 g. This finding is quite similar to the finding of Al-Kahtani (1992) and Saxena, Manan, and Berry (1987). However, Melgarejo et al. (2000) reported more fructose than glucose.

The results of analysis of ash and minerals in pomegranate fruit are shown in Table 4. Ash contents of juice and seeds were 1.05 and 0.45%, respectively, and these values were close to those values reported by Morton (1987). The overall composition of minerals varied markedly among the three ripening stages.The amounts of potassium, calcium and sodium were determined to be highest in both juice and seeds. From an examination, it is obvious that potassium is the most abundant element in fruit, followed by Na and Ca. The other elements, in descending order by quantity (mg/100 g) were Mg, P, Zn, Fe and Cu and their respective values in the fruit were similar to the value reported by Chauhan, Pundir, and Singh (1991). The exception was iron, which was reported to be 21.1 mg/100 g as compared to

1.33 in seeds and 2.91 mg/100 g in juice. The variation could originate from the pomegranate variety, and agro-climatic as well as environmental conditions. The study provides important data for calorific and compositional changes of the fruits (e.g. sugars, ascorbic acid and minerals, respectively) at different stages of ripening, emphasizing that pomegranate fruit can be a good source of nutrients. More studies of physical and chemical relationship among different varieties need to be undertaken.

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