



EFFECT OF PROCESSING CONDITIONS ON PROPERTIES OF POMEGRANATE SYRUP

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ABSTRACT

Pomegranate juice and syrup has gained great importance due to its health properties and consequently is now a highly demanded product. Effect of processing conditions on the quality of pomegranate syrup was evaluated. Citric acid, sodium benzoate, arabic gum and pomegranate peel extract were mixed with the pomegranate juice. The juice was concentrated until 42° Brix. Sugar was added to the concentrated juice in different manners (cold method, semi-hot method and hot method) to increase the syrup concentration to 62° Brix. Syrup samples were stored at room temperature for three months. Chemical composition, vitamin C, total phenolic content, antioxidant activity, colour and sensory evaluation of pomegranate syrup were determined during the storage period. Results showed that the total phenolic content (TPC) of pomegranate syrup ranged from 223.33 to 293.33 mg gallic acid/g sample. Antioxidant activity and TPC decreased gradually during the storage period. Obvious changes in L*, a* and b* values of pomegranate syrup were noticed, confirming the degradation of visual colour components of the syrup during the storage period. Sensory evaluation revealed that pomegranate syrup prepared with cold method was the most accepted while that prepared by hot method was the least preferred.

Key words: Pomegranate syrup, pomegranate juice, concentration.

INTRODUCTION

Pomegranate is considered as one of the oldest fruits and one of the earliest to appear in human diet and belongs to the Punicaceae family. It is native to southwest Asia and has been cultivated over the whole Mediterranean region of Asia, Africa and Europe. In particular, its successful adaptation to the Mediterranean climate has produced a wide diffusion in various countries thus originating several local genotypes along the centuries. Pomegranate is widely cultivated throughout Afghanistan, Algeria, Armenia, Azerbaijan, Iran, Iraq, India, Pakistan, Syria, Turkey and Egypt (Hassan *et al.*, 2014). Egypt total production of pomegranate was estimated with 64574 tons in 2011 according to statistics of the Egyptian Ministry of Agriculture (Central Administration of Agricultural Extension, 2014).

Pomegranate fruits can be eaten fresh or in the form of processed products such as single strength juices, jellies, juice concentrates, jams, marmalades, minimally processed pomegranate arils, frozen arils, refrigerated arils, arils in syrup, candied arils, arils in brandy and in vinegar, carbonated beverages, pomegranate wine, and pomegranate syrup, *etc.* The processed products such as juice, concentrate, syrup and jelly were highly acceptable because of their nutritive and dessert qualities and palatability (Dhumal *et al.*, 2014).

Pomegranate fruits are rich source of carbohydrates, minerals, crude fibers, and various active compounds, such as vitamin C, and certain phenolic compounds as punicalagin, ellagic acid, gallotannins and anthocyanins. The natural antioxidants of pomegranate can protect humans against the oxidative stress and reduce consequently of chronic diseases and prevent

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disease progression (Lansky and Newman, 2007; Zaouay *et al.*, 2012).

It has been reported that pomegranate fruits were used in folkloric medicine as a treatment for many diseases such as diarrhea, parasitic worm infections, urinary tract infections and kidney stones (Sudheesh and Vijayalakshmi, 2005). Several additional studies have demonstrated that pomegranate juice and concentrate slow bacterial growth and inhibit bacterium-induced toxins (Braga *et al.*, 2005). The therapeutic effects of pomegranate fruit, peel and juice as powerful antioxidants and anti-inflammatory substances that include polyphenols and tannins has been reported (Aviram and Dornfeld, 2001; Aviram *et al.*, 2002; Kim *et al.*, 2002; Afaq *et al.*, 2005; Gasemian *et al.*, 2006).

The effect of processing, such as pasteurization, clarification, concentration, and dehydration effects on pomegranate juice bioactive compounds have been reported (Mena *et al.*, 2014). Pasteurization has been shown to decrease anthocyanin content based on pomegranate cultivar and anthocyanin type (Vegara *et al.*, 2013). Clarification, using chemical agents and filtration processes, is used to remove cloud from juices to improve colour, flavour, and storage stability. Clarification methods have displayed differences in levels of phenolic removal, for example gelatin showed a greater reduction of total phenolic while polyvinylidene polypyrrolidone (PVPP) had a greater effect on anthocyanin reduction (Alighourchi *et al.*, 2013). Also, microfiltration have a greater reduction in anthocyanin content than the use of chemical agents (Vardin and Fenercioglu, 2003; Alighourchi *et al.*, 2008; Turfan *et al.*, 2011; Anderson *et al.*, 2015).

Despite the great importance of pomegranate derived products, the industrial processing of pomegranate is limited due to peeling difficulties and lack of technological development for industrial processing of pomegranate (López-Rubira *et al.*, 2005).

The aim of this study was to extend the commercial shelf life of pomegranate products by producing pomegranate syrup with best properties and evaluate the effect of processing conditions on the properties of pomegranate syrup.

MATERIALS AND METHODS

Materials

About 50 kg fresh pomegranate fruits (*Punica granatum* L.) of the Manfaloty cultivar grown in Egypt were obtained from El-Obor market. Sugars, citric acid, sodium benzoate and arabic gum were obtained from Zagazig local market.

Preparation of juice

The fruits were washed, damaged fruits were discarded. Each fruit was cut in two halves and arils were manually separated and the remaining carpellar membranes were removed. Pomegranate arils were used for juicing by using juice Extractor (Molenix). The juice was then filtered through cheese cloth (Dhumal *et al.*, 2014).

Preparation of pomegranate peel extracts

Pomegranate peel was dried by hot air at 45°C until constant weight. The dried pomegranate peels were ground into powder using a grinder (Moulinex, France) and sieved through a 60 mesh screen until fine powder was obtained. About 100g of pomegranate peel powder was extracted according to the method of Sanbongi *et al.* (1998) with some modifications. Pomegranate peel powder was suspended in ethanol 70% (W/V) at ratio of 1:10. The mixture was stirred with magnetic stirrer overnight at ambient temperature (30°C) and filtered through Whatman No.1 filter paper. The solvent was evaporated by rotary evaporator (Buchi Waterbath B-480 with Buchi Rotavapor R-124) at 45°C to obtain the crude extract. The yield was 100 ml / 70 g pomegranate peel powder. The extract was kept by freezing.

Preparation of pomegranate syrup

Citric acid (0.3 g/100 g sugar), sodium benzoate (0.1g/100ml), arabic gum (3 g/100ml) and pomegranate peel extract (1 ml / 100ml) were mixed with the extracted pomegranate juice (16° Brix). The juice was concentrated at 60°C by rotary evaporator (Buchi Waterbath B-480 with Buchi Rotavapor R-124) until 42° Brix. The concentrated juice was divided into three batches (A, B and C). Sugar was added to the all syrup batches in different manners (A: cold

method; B: semi-hot method; C: hot method) according to Bassiuny (1994). Cold method: the sugar (105g) was dissolved in the concentrated juice at room temperature until 62° Brix. Semi-hot method: the specified amount of sugar (105g) was dissolved in a quantity of water (equal to 1/3 volume of the concentrated juice) then brought to boiling 100°C. The hot sugar solution was allowed to cool to 45°C. Then the concentrated juice was added to sugar solution to obtain final TSS of 62° Brix. Hot method: the sugar was dissolved in the concentrated juice at 100°C in water bath to obtain pomegranate syrup of 62° Brix. Syrup samples were stored at room temperature for three months.

Analytical methods

The total soluble solids were measured by hand held refractometer (Bellingham and Stanley Ltd., UK). The pH value of juice and syrup was measured using pH meter type 3320 Jenway LTD. (Felsted Danmow Essex CM63 IB, UK). Titratable acidity (as citric acid), total, reducing and non-reducing anhydrous sugars and ash were determined according to the method described in AOAC (2005).

Determination of total phenolic content (TPC)

Total phenolic contents of pomegranate juice and syrup were measured by Folin-Ciocalteu method according to Singleton *et al.* (1999) using gallic acid as standard at wavelength 760 nm. The results were expressed as mg gallic acid/ 100 ml sample.

Determination of free radical scavenging activity (DPPH Assay)

The free radical scavenging activity using the 1,1-diphenyl-2-picrylhydrazil (DPPH) reagent was determined according to Cuendet *et al.* (1997) with slight modifications. Fifty microliters of pomegranate juice or syrup was added to 1 ml of 0.2 mM of methanolic DPPH solution (0.0078 g of pure DPPH (Mw = 394.32 g/ mol) in 10 ml methanol). The solutions were vigorously shaken and left at room temperature in the dark. After 45 min of incubation in dark at room temperature, the absorbance was measured at 517 nm against a blank (methanol). The

inhibition of DPPH free radicals (I%) was calculated as follows:

$$(I\%) = [(A_{517\text{nm}} \text{ blank} - A_{517\text{nm}} \text{ sample}) / A_{517\text{nm}} \text{ blank}] \times 100$$

Colour measurement

Colour values of pomegranate juice and syrup (L*, a* and b*) were measured using Hunter Lab colour analyzer (Hunter Lab Colour Flex EZ, USA) according to Rao *et al.* (2011). The L* value (lightness index scale) ranges from 0 (black) to 100 (white) while, a* value indicates the redness (+a) or greenness (-a*) and the b* value refers to the yellowness (+b) or blueness (-b*).

Sensory evaluation

Pomegranate syrup was reconstituted to the original strength (16° Brix) by water. All samples were subjected to sensory evaluation for colour, taste, flavour and overall acceptability. The juice samples were introduced to 10 panelists (Staff members of Food Science Department, Faculty of Agricultural, Zagazig University, Egypt) immediately after juice preparation. The samples were sensory evaluated using hedonic scale of 1 to 9 point, where (1 = dislike extremely, 2 = dislike very much, 3 = dislike moderately, 4 = dislike slightly, 5 = neither like nor dislike, 6 = like slightly, 7 = like moderately, 8, like very much, 9 = like extremely) (Matta *et al.*, 2005).

RESULTS AND DISCUSSION

Chemical Characteristics of Pomegranate Juice and Syrup

Table 1 shows the chemical composition of pomegranate juice and syrup (cold method). Total soluble solids (TSS) of syrup (62° Brix) was found to be higher than that of juice (16° Brix) due to sugar addition during processing. The pH of syrup (3.26) was lower than that found in juice (3.45) and concentrated juice (3.50) while, total titratable acidity of syrup (2.71%) was found to be higher than that of juice (0.88%). This may be related to the presence of citric acid. The ash content of syrup (2.73%) was found to be higher than that of juice (0.62%) because of the presence of peel. The ascorbic acid content of the juice (68.0 mg/

Table 1. Chemical characteristics of pomegranate juice and syrup

Sample	TSS (°Brix)	pH	Total titratable acidity (% citric acid)	Ash (%)	Ascorbic acid (mg/100ml)	Reducing sugars (%)	Total phenolic content (mg/g)
Juice	16	3.45	0.88	0.62	68.0	12.40	2.57
Syrup (cold method)	62	3.26	2.71	2.73	20.21	56.74	293.33

100 ml) was higher than that of the syrup (20.21 mg/100ml) because of the oxidation and heat effects during the concentration process (Paul and Ghosh, 2012). Logically, reducing sugars of the syrup (56.74%) was found to be higher than that of juice (12.40%). Total phenolic content of the syrup (293.33 mg/g) was found to be higher than that of juice (2.57 mg/g) because of the peels presence which has higher antioxidant compounds. These results are in close agreement with Yousefi *et al.* (2012) and Fazaeli *et al.* (2013).

Effect of Storage Period on Total Phenolic Content (TPC) on Pomegranate Syrup

Total phenolic content of pomegranate syrup was measured during the storage period of 3 months. Table 2 shows that the syrup made by cold method had better total phenolic contents than semi-hot and hot methods. The total phenolic content ranged from 223.33 to 293.33 mg gallic acid/g sample. It was noticed that TPC content decreased gradually during the storage period. These results are in accordance with Chalfoun-Mounayar *et al.* (2012) who measured the total polyphenols content of pomegranate molasses, compared to fresh pomegranate juice, being 252.28 and 79.49 mg of gallic acid equivalent/l, respectively. Total phenolic content revealed that pomegranate syrup prepared with cold method was the highest for the non-use of heat.

Effect of Storage Period on Free Radical Scavenging Activity (DPPH assay)

Antioxidant activity of pomegranate syrup was measured using DPPH method during the storage period and the results are illustrated in

Fig. 1. Antioxidant activity was affected by the storage period and also with the processing method. Gradual decrease in the antioxidant activity of all syrup samples was noticed during the storage period. Similar to the decrease in TPC content, cold method gave the highest antioxidant activity values followed by the semi-hot and hot methods. Yousefi *et al.* (2012) investigated the effect of heating method on the degradation of antioxidant activity and they stated that applying microwave instead of conventional heating method could conserve better the antioxidant activity of pomegranate juice. Fazaeli *et al.* (2013) reported that anthocyanin degradation and consequently decrease in antioxidant activity were more pronounced in rotary evaporation compared to microwave heating method. These results are in harmony with Mousavinejad *et al.* (2009).

Effect of Storage Period on Colour Attributes of Pomegranate Syrup

The colour of fruit juices and concentrates is considered an important factor that attracts the consumer and has a remarkable influence on his acceptance and quality evaluators (Turfan *et al.* 2011). The colour values of the fresh pomegranate juice were 9.25 (L*), 6.46 (a*) and 0.21 (b*). The changes in the colour values (L*, a*, and b*) of pomegranate syrup during the storage period are illustrated in Fig. 2. In general, all colour values were gradually decreased during the storage period. The method of processing significantly affected the colour values of pomegranate syrup. Cold method had the highest pomegranate syrup colour values while, hot method had the lowest. There were obvious changes in (L*, a*, and b*) values, confirming

Table 2. Effect of storage period on total phenolic content of pomegranate syrup

Storage period (week)	TPC (mg gallic acid/g sample)		
	Cold method	Simi-hot method	Hot method
0	293.33	289.73	284.33
3	284.33	271.67	255.00
6	271.67	260.00	243.33
9	263.33	236.67	235.00
12	251.67	231.67	223.33

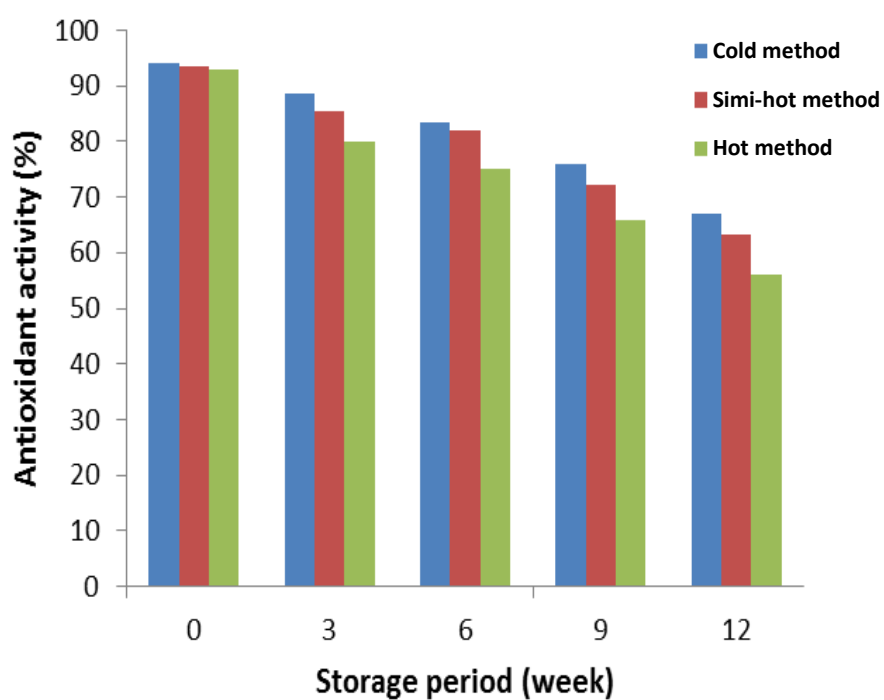


Fig. 1. Effect of storage period on antioxidant activity of pomegranate syrup

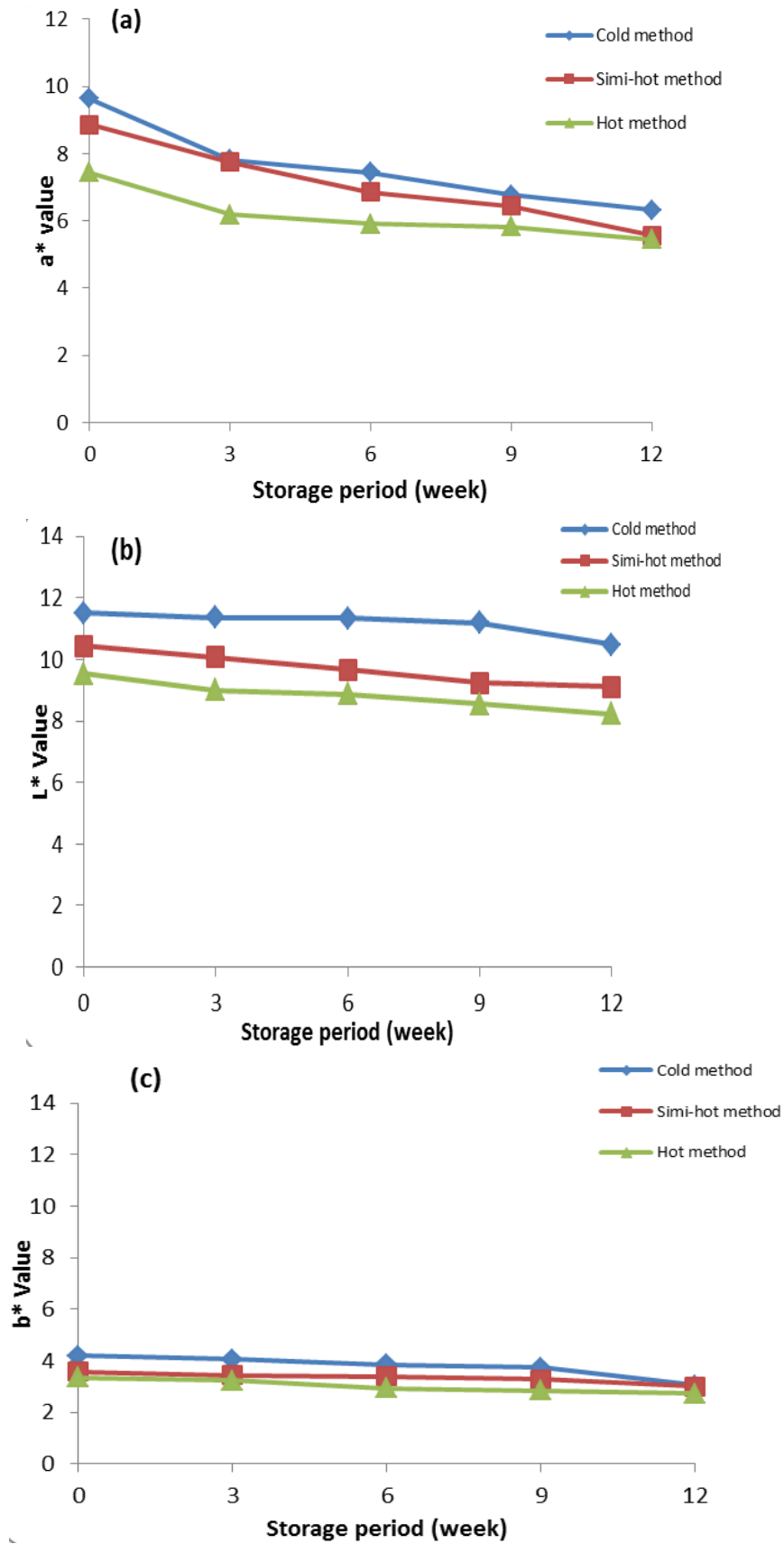


Fig. 2,a,b,c. Effect of storage period on colour values of pomegranate syrup

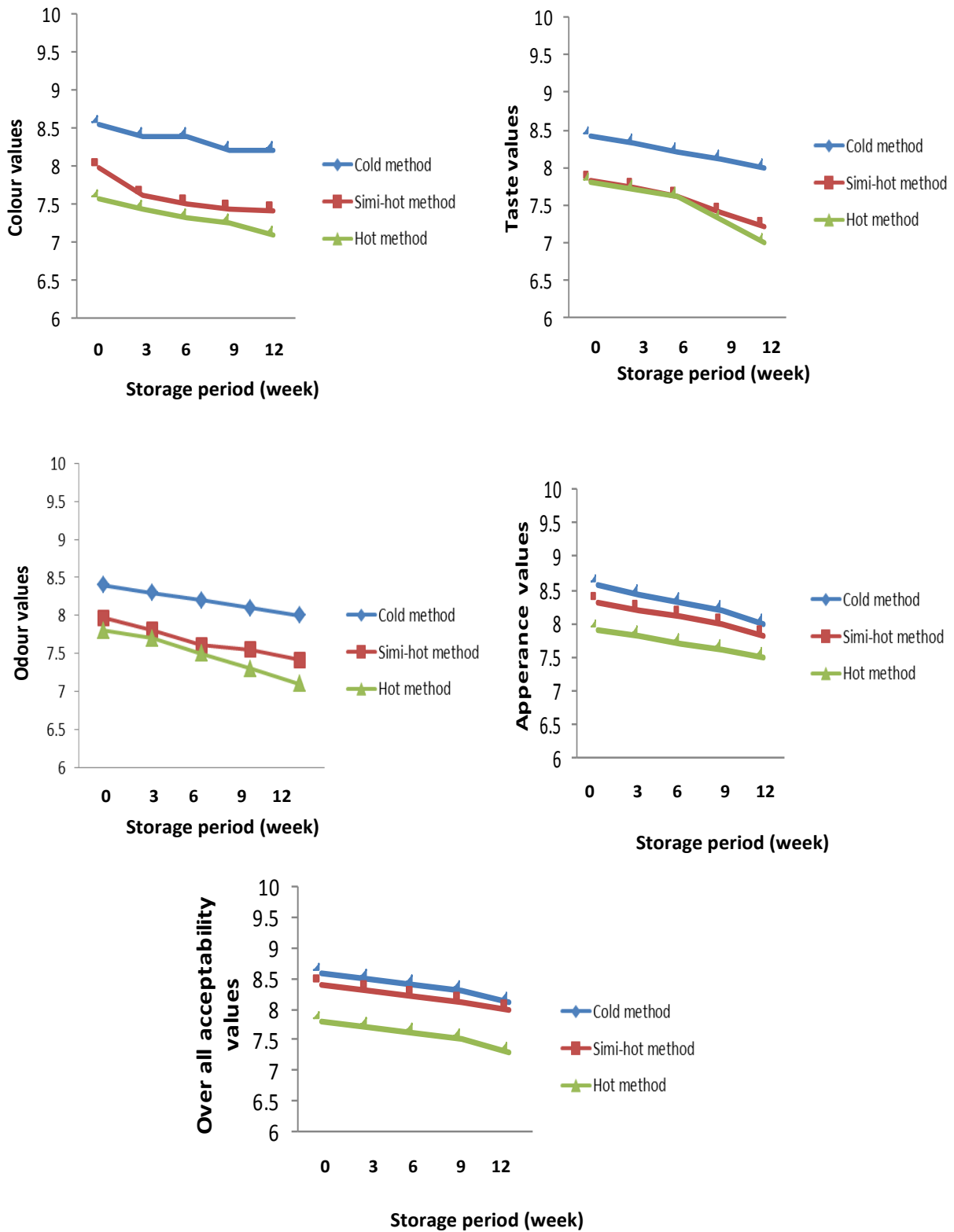


Fig. 3. Effect of processing method on sensory evaluation of pomegranate syrup

the degradation of visual colour components of the syrup during the storage period. Turfan *et al.* (2011) reported that the reduction in a^* value of pomegranate concentrate, as an indicator for the degradation of anthocyanin pigments, normally occurs due to heat damages during thermal processing, nonenzymatic browning reactions and polymerization of anthocyanins with other phenolic compounds. Khajehei *et al.* (2014) found that the a^* value of pomegranate juice which was concentrated by rotary vacuum evaporator and by heating at atmospheric pressure reduced by about 71 and 74% in average, compared to the a value of the initial pomegranate juice, respectively. Colour parameters were decreased at the end of the storage period. These data are in agreement with those obtained by Yousefi *et al.* (2012).

Effect of Storage Period on Sensory Evaluation of Pomegranate Syrup

Sensory evaluation plays significant role in measuring characteristics and acceptability of foods and food products. The results of sensory evaluation (colour, taste, odour, appearance and overall acceptability) of pomegranate syrup immediately after preparation are shown in Fig. 3. Processing method significantly affected the sensory quality of pomegranate syrup. Results of the sensory evaluation by the panelists showed that the pomegranate syrup prepared with cold method was the most accepted while that prepared by hot method was the least preferred. As for colour, cold method had the highest score recorded 8.39 while hot method had the lowest (7.58). Pomegranate syrup prepared with cold and semi-hot methods had higher overall acceptability than that prepared with hot method. Thermal processing and concentration process are the most commonly used preservation technique to extend the shelf-life of juices. However, this process may have adverse effects on sensory and nutritional values of juices (Plaza *et al.*, 2006).

Conclusion

The results of this study indicated that producing pomegranate syrup by the cold method is a promising product from pomegranate juice. It is characterized by highly acceptable colour and antioxidant.

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تأثير ظروف التصنيع على صفات شراب الرمان

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نظراً لأهمية عصير وشراب الرمان من الناحية الغذائية والصحية وزيادة الطلب علي المنتج فقد تم إنتاج شراب رمان وذلك بإضافة كل من حامض الستريك، بنزوات الصوديوم، الصمغ العربي ومستخلص قشور الرمان إلى عصير الرمان ثم تركيز العصير تحت تفريغ إلى ٤٢ بيركس مع إضافة السكر إلى العصير المركز بأساليب مختلفة (الطريقة الباردة، الطريقة نصف الساخنة، الطريقة الساخنة) وذلك لرفع تركيز الشراب إلى ٦٢ بيركس وقد تم تقدير تأثير عملية التصنيع علي جودة شراب المنتج تم تخزين عينات الشراب علي درجة حرارة الغرفة لمدة ٣ شهور ثم بعد ذلك تقدير التركيب الكيماوي، محتوى فيتامين C، محتوى الفينولات الكلية، والنشاط المضاد للأكسدة، اللون والتقييم الحسي لشراب الرمان وذلك خلال فترة التخزين وأظهرت النتائج تراوح محتوى الفينولات الكلية لشراب عصير الرمان من ٢٢٣.٣٣ إلى ٢٩٣.٣٣ مجم حامض الجاليك/جم عينة، كما حدث انخفاض في نشاط مضادات الأكسدة ومحتوى الفينولات الكلية تدريجياً خلال فترة التخزين، وأظهرت معدلات التغيير لقيم b^* , a^* , L^* لشراب الرمان حدوث تدهور لمكونات اللون المرئي للشراب خلال فترة التخزين كما أظهر التقييم الحسي أن شراب الرمان المحضر بالطريقة الباردة كان الأكثر قبولا بينما كان المحضر بالطريقة الساخنة الأقل قبولا.

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