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Some physiochemical properties of pomegranate jams enriched with black rosehip and black mulberry extracts

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Abstract

In this research, 5 different pomegranate jams were produced with the addition of anthocyanin-based extract obtained from black mulberry and black rose hips by classical and ultrasonic extraction methods and without the addition of anthocyanin-based extracts (control). The jams are stored at room temperature (20±2 °C) for 60 days, ensuring that the storage is 0., 20., 40. and 60. color, total dry matter, water soluble dry matter, pH, titration acidity, reduced sugar, sucrose, total sugar, HMF. The total dry matter, water soluble dry matter, ash, reduce sugar and HMF were found to be higher compared to the control sample of pomegranate jams to which the extract was added. In addition, at the end of the storage period of all samples, there was a decrease in total dry matter, water soluble dry matter, sucrose and total sugar amounts compared to the beginning. L, a, b, C and H values increased. L, a, b, C and H values of pomegranate jams to which extracts obtained by ultrasonic method were added were found to be higher compared to pomegranate jams to which extracts obtained by classical method were added. HMF amount in pomegranate jams to which extracts obtained by ultrasonic method were added were found to be lower than in the control group and pomegranate jams to which extracts obtained by classical method were added. As a result, it can be said that black mulberry and black rosehip extracts can be used to improve the color of pomegranate jam and other foods.

Keywords: Pomegranate (*Punica granatum* L.) jam; Black rosehip (*Rosa pimpinellifolia* L.); Black mulberry (*Morus nigra* L.); Extraction; HMF; Physicochemical properties

1. Introduction

Pomegranate (*Punica granatum* L.) is a perennial herb belonging to the Punica genus of the Punicaceae family of the order Myrtales [1]. Pomegranate, which is grown in tropical and sub-tropical climate zone, is grown in Turkey, Iran, Afghanistan, South Asia, West Asia, South Caucasus, Anatolia, Mediterranean and Middle East [2]. Pomegranate is grown in the Mediterranean, Aegean and South Eastern Anatolian Regions of Turkey in areas up to a maximum of 1000 meters above the coast [3].

Pomegranate fruit consists of three parts: 50% edible juicy seed (grain), 30% non-edible outer skin and 20% non-edible white inner lamella [4]. Approximately 30% of the fruit weight of the pomegranate consists of water and the shell (pericarp) contains the inner membrane. Approximately 3% of the pomegranate seed is made up of the kernel part, and about 20% of the kernel is made up of fat [5]. The edible part of the pomegranate fruit contains a significant amount of organic acids, sugars and phenolic compounds [6]. 100 grams of pomegranate seed contains 79% water, 18% carbon hydrates, 1.1% protein, 0.9% fat, 100 grams of grain gives 70 kcal of energy [7], contains organic components such as citric acid (1 g / L) and vitamin C (7 mg / L) [6;8]. The water-soluble dry matter of pomegranate juice varies between 13.3-16.9%, pH between 0.93-4.6% and titration acidity between 0.25-3.17% [9].

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Pomegranate fruit is consumed fresh and processed into jam, marmalade, liqueur, syrup, jelly, compote and fruit juice. During the processing of pomegranate into these products and the storage of the products, anthocyanins are degraded depending on various factors such as temperature, oxygen, UV light, pH [10]. Therefore, in the production of various foods, color intensity is increased by adding natural or artificial colorants and the food is made attractive in terms of color [11].

Stability and coloring power of artificial colorants are stronger than natural colorants. However, since the negative effects of artificial colorants on health have been determined in toxicological studies conducted in recent years, natural colorants have begun to be used in the coloring of foods [12]. Black mulberry (*Morus nigra*) and black rosehip (*Rosa pimpinellifolia* L.) fruits are among the richest fruits in terms of anthocyanins, which are natural color pigments [13].

In the literature review, no study was found in which coloring matter was added to pomegranate jam. In this study, it was aimed to add black mulberry and black rosehip extracts, hence anthocyanins, which are natural color substances, in order to increase the color intensity of pomegranate jam. For these reasons, in this research, 4 different anthocyanin-based extracts were produced from black rose and black mulberry fruits using acidified ethanol: water (v/v; 50:50) solvent by classical and ultrasonic extraction methods and added to pomegranate jam. Periodic changes in some physicochemical properties of jams stored at room temperature (20±2 °C) for 60 days were determined during storage (on the 0th, 20th, 40th and 60th days).

2. Material and methods

2.1. Material

In the research, pomegranate (Hicaznarı) fruit was obtained from the market of Erzurum province in Turkey, black mulberry was obtained from Erzurum province Uzundere and Tortum districts, and dried black rosehip fruit was obtained from herbalists in Erzurum province. The peel parts of the pomegranate fruits are thoroughly washed so that there is no dirt and soil left. Then the pomegranate peels were cut with a knife so that the grains would not be damaged and the pomegranate seeds were removed.

2.2. Method

2.2.1. Preparation of black mulberry and black rosehip extracts

Black mulberry and black rosehip extracts were obtained by ultrasonic and classical methods. For this purpose, 50 g of dried black mulberry and black rosehip fruits were weighed and 50 ml of acidified ethanol solution was added to them. The prepared mixtures were crushed in a blender (Waring) for 2 minutes and the mixtures were taken into 200 ml flasks and their mouths were closed with parafilm. Then, it was shaken for 2.5 hours in an ultrasonic water bath (35 °C) a for extraction with ultrasonic extraction method and in a shaking water bath at 60 rpm and 35 °C for extraction with conventional method. Then, they were filtered through four layers of cheesecloth, ordinary filter paper and Whatman No: 1 paper, respectively. After filtration, the solvent in the extracts was removed with the help of a rotary evaporator at 60 °C and 210 rpm. The extracts in the balloon were stored at -20 °C until jam production.

2.2.2. Jam production

The flow chart of pomegranate jam production is given in Figure 1.

2.3. Physicochemical Analysis

L, a and b values of jam samples were measured with Conica Minolta Colorimeter (Chroma Meter, CR-400, Minolta-Konica, Japan) device, C and H values were calculated using a and b values [14;15]. The total dry matter and ash of samples by the standard AOAC method [16], pH by the OHAUS Starter 3100, USA model pH meter, Titrated acidity was diluted with 10 g of homogenized sample in 25 mL of distilled water and titrated with 0.1 N sodium hydroxide solution until pH reached 8.1, the amount of water-soluble dry matter by the Abbe Refractometer (Carl Zeiss), the reducing sugar, sucrose, and total sugar content by the volumetric Lane-Eynon method and the amount of HMF via spectrophotometrically were determined [17].

2.4. Statistical analysis

The obtained data were subjected to correlation and variance analysis using IBM SPSS Statistics 25 package program. According to the Duncan Multiple Comparison Test, the importance of the difference between the groups was determined with a confidence limit of 0.05.

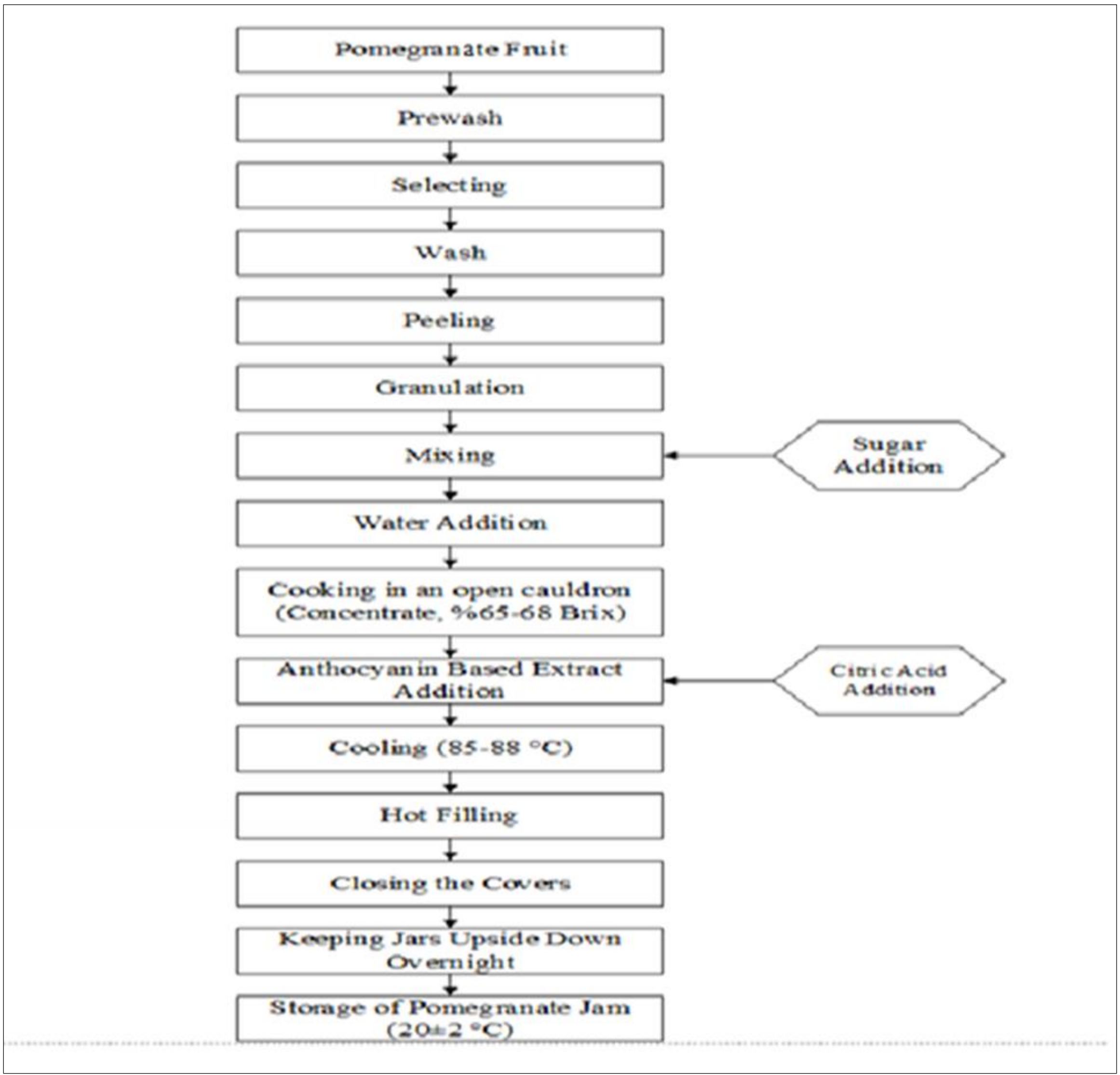

Figure 1 Pomegranate jam production flow chart [18]

3. Results and discussion

Table 1 shows changes in the color values of jams produced from pomegranate fruit. It was found that the fruit variety and extraction method had a statistically significant effect on a, b and C values in jam samples at $p<0.01$ level. It was determined that the a, b and C values of the samples with the addition of extract decreased compared to the control, and this decrease was greater with the addition of black mulberry extract. That is, the addition of fruit extract reduced the red color of jam samples. The addition of the extract obtained by the ultrasonic method caused the a, b and C values of the jams to be higher compared to the addition of the extract prepared by the classical method (Table 1). Ekici [19] reported that the passage of material through the cell wall is easier in the ultrasonic method than in other methods. The storage time had a statistically significant ($p<0.01$) effect on the L a, b and C and H values of the jam samples (Table 1). It was determined that the L value of the jams increased on the 20th day of storage and decreased again on the 40th and 60th days, that is, the color gradually darkened. The a and b values of the jams did not change from the baseline on the 20th day of storage, but increased gradually on the 40th and 60th days. C and H values were found to be the highest on the 60th day of storage (Table 1). This is due to the non-enzymatic browning reaction that takes place during storage [20].

The H values of the jam samples increased during storage. It has been stated that the H value also increases due to the increase in the yellow color value by the breakdown of anthocyanins during storage [14;21].

Herrmann [22] reported in his research that color pigments undergo oxidation and disintegrate as a result of applied heat treatments. Wicklund *et al.* [23] determined that the color quality of strawberry jams stored at 4 °C was better than those stored at 20°C in their study. He stated that this situation is due to the fact that heat treatments applied to anthocyanin-rich fruits cause significant changes in color values by breaking down anthocyanins [24]. Hager *et al.* [25] stated that during the processing and storage of raspberries, an increase in color values occurs in proportion to the degradation of anthocyanins.

Table 1 Changes in the color values of jams produced from pomegranate fruit

	N	L	a	b	H	C
Kind of Fruit (KF)						
Black rosehip	24	21.84±4.05	10.21±1.79^{b}	5.88±1.47^{b}	29.84±4.37	11.81±2.13^{b}
Black mulberry	24	22.34±4.34	9.20±20.43^{c}	5.24±2.00^{c}	28.95±4.72	10.63±3.03^{c}
Control	12	23.85±1.69	11.06±0.79^{a}	6.41±1.13^{a}	29.90±3.19	12.80±1.17^{a}
Significance		NS	**	**	NS	**
Extraction Method (EM)						
Classic	24	21.84±4.29	8.95±2.44^{c}	5.30±1.80^{c}	30.10±3.76	10.42±2.97^{c}
Ultrasonic	24	22.33±4.11	10.46±1.58^{b}	5.83±1.73^{b}	28.70±5.16	12.02±2.07^{b}
Control	12	23.85±1.69	11.06±0.79^{a}	6.41±1.13^{a}	29.90±3.19	12.80±1.17^{a}
Significance		NS	**	**	NS	**
Storage Time (ST)						
0. day	15	20.81±5.26^{c}	9.04±1.81^{c}	4.58±1.29^{c}	26.51±3.89^{c}	10.16±2.11^{c}
20. day	15	24.56±1.41^{a}	8.80±1.72^{c}	4.94±0.91^{c}	29.42±3.15^{b}	10.11±1.87^{c}
40. day	15	22.71±3.32^{b}	10.46±1.72^{b}	6.12±1.21^{b}	30.35±4.03^{ab}	12.15±1.91^{b}
60. day	15	21.68±3.67^{bc}	11.6±1.72^{a}	7.29±1.78^{a}	31.71±4.45^{a}	13.73±2.28^{a}
Significance		**	**	**	**	**
ST x KF		**	*	**	**	**
ST x EM		**	**	**	NS	**
KF x EM		NS	**	NS	*	**
ST x KF x EM		**	**	**	**	**

* indicates p<0,05, ** indicates p<0,01, NS non-significant differences p>0,05; The averages shown in the same column with the same letter are statistically indistinguishable from each other.

Table 2 shows changes in the proximate composition of jams produced from pomegranate fruit during storage. The fruit variety was statistically significantly effective on the amount of water-soluble dry matter and titration acidity of jam samples at p<0.01 level and significantly effective on the amount of reduced sugar at p<0.05 level, while it was not effective on other characteristics (p<0.05). It has been determined that the amount of water-soluble dry matter of jam with the addition of black mulberry extract is higher than other jams. It was found that the titration acidity of the control sample and jams with the addition of black mulberry water-soluble dry matter was higher than that of jams with the addition of black rosehip. It has been determined that the reduced sugar content of the jam containing black rosehip extract is higher than the others. The extraction method was statistically significantly effective on the amount of water-soluble dry matter of jam samples at the $p<0.01$ level and on the amount of HMF at the $p<0.05$ level, while it was not effective on other characteristics ($p>0.05$) (Table 2).

Storage time of jams, total dry matter, water-soluble dry matter, total ash, pH, titratable acidity, total sugars, reducing sugars, sucrose on the amount of statistically very significant ($p<0.01$) while effective in the amount of HMF did not effect ($p<0.05$) (Table 2). Cemeroğlu [18] stated in their study that the desired pH range for the formation of a good gel in jams depends on the amount of dry matter in the jams. Accordingly, the dry matter between %68-72 in the jam pH 3,0-3,3, %72-75 located between jams in 3,1-3,4 between %75-85 and in the jam 3,2-3,5 should be found between stated. In our study, the pH in jams with a dry matter content of 64-77% was determined between 3.4 and 3.6, and [18] showed a similar value in their study.

When Table 2 is examined, it is seen that the amount of sucrose and total sugar decreases during storage, and the amount of reduced sugar increases. During storage, sucrose undergoes inversion, albeit at a low level under room conditions, increasing the amount of reduced sugar. The reason for the fluctuation in the total amount of sugar is due to the sugar added during the cooking process [26]. It is believed that the change in the total amount of sugar during storage is due to Maillard reactions. Maillard reaction occurs as a result of the interaction of amino acids with reduced sugars found in the environment in heat-treated foods [27;28].

Altan [29] determined that the amount of sucrose decreases with the inversion caused by the effect of heat treatment. It is believed that the reason for the decrease in the total amount of sugar during storage is due to non-enzymatic browning reactions [30]. Karataş and Şengül [31] reported in their study that the change in temperature and acidity during storage affects the amount of sucrose. Zor and Şengül [32] determined the amount of sucrose at the beginning of the storage of jams as 20.24% in his study.

During storage, an increase in the pH values and titration acidity amounts of jam samples occurred. Buckow *et al.* [33] determined that when they stored pasteurized blueberry juice at 4, 25 and 40 C, the pH decreased from 3.0 to 2.85 and then remained stable. They explained this situation by the increase of degradation products of anthocyanins, such as phenolic acids. One of the most important ways to prevent the development of microorganisms is the high amount of dry matter [34]. In our study, jam samples are unsuitable for the development of microorganisms, since the amount of dry matter during storage is between 66.28% and 74.38%. While the titration acidity value of all samples decreased on the 20th day, it increased on the other days during storage. Due to the dissolution and deterioration reactions occurring in the products during storage, the free carboxyl groups and free hydrogen ions in the product increase, it causes an increase in the titration acidity value in the products. In addition, the decrease in the total amount of anthocyanin during the storage period increases the titration acidity [35]. It is stated that the changes in the titration acidity and pH values of the jam samples during storage cause fluctuations in the total phenolic substance amounts [35]. Crystallization is an undesirable situation in terms of jam quality and can be prevented when approximately 30-35% of the total sugar in jams is reducing sugar [36]. The total ash amount was directly related to the mineral substances contained in the foods, and the ash amount of the samples increased during the storage period (Table 2). The amount of ash can be changed by chemical and physical means. Some mineral substances can be oxidized with oxygen to high amounts [37].

The amount of HMF in the jam samples differed statistically significantly ($p<0.05$) according to the extraction method. While the amount of HMF in the jams with extracts prepared by the classical method was the highest, the amount of HMF in the plain pomegranate jam sample was the lowest. Burdurlu and Karadeniz [27] stated that the amount of HMF would increase due to the heat treatment of jams, the formation of brown pigments during cooking, the Maillard reaction and the breakdown of sugars in acidic conditions. In addition, the pH value of the control sample being lower than the other jam samples is one of the reasons affecting the amount of HMF. Yılmaz [38] determined the amount of HMF in jam samples by HPLC and reported that the amount of HMF in jam samples varied in the range of 5.32-18.24 mg/kg. He said that although the sugar amount of the jam sample used as a control was higher than the low-calorie jam samples, the amount of HMF was lower. In addition, the fact that the pH value of the control sample was lower than the other jam samples indicated that it was one of the reasons affecting the amount of HMF. He reported that the amount of HMF was low due to the lower temperature and shorter cooking time of the control sample. Factors affecting the formation of HMF are the amount of carbohydrates in the food, pH, total acidity, temperature, water activity, storage, metallic containers, monosaccharides such as glucose and fructose, and the presence of amino groups. In addition, disaccharides and many polysaccharides can hydrolyze to simple sugars and initiate HMF formation [39]. In our study, the amount of HMF fluctuated during storage. The reason for this is thought to be due to the reasons mentioned above.

Table 2 Some physicochemical properties of pomegranate jams

	N	Dry Matter	Water Soluble Dry Matter	Ash	pH	Titration Acidity	Reduce Sugar	Sucrose	Total Sugar	HMF
Kind of Jam (KJ)										
With black rosehip extract	24	69.39±6.45	64.40±0.72[c]	1.26±0.8	3.55±0.04	0.53±0.06[b]	48.49±8.84[a]	9.52±7.12	68.03±7.50	10.87±4.35
With black mulberry extract	24	71.19±6.95	66.13±0.83[a]	1.15±0.83	3.56±0.04	0.54±0.06[a]	47.64±8.34[ab]	10.69±8.63	69.57±10.19	9.73±3.23
Control (without extract)	12	68.10±2.95	64.62±0.32[b]	0.89±0.53	3.54±0.06	0.54±0.05[a]	46.68±6.42[b]	14.57±9.03	76.59±13.15	8.69±4.09
Significance		NS	**	NS	NS	**	*	NS	NS	NS
Extraction Method (EM)										
Classic	24	71.72±8.55	65.72±1.06[a]	1.21±0.86	3.55±0.04	0.53±0.06	48.07±9.16	10.45±7.60	69.52±8.50	11.03±3.78[a]
Ultrasonic	24	68.86±3.77	64.81±1.10[b]	1.20±0.77	3.56±0.04	0.53±0.06	48.05±8.01	9.76±8.24	68.08±9.38	9.57±3.82[ab]
Control	12	68.10±2.95	64.62±0.32[c]	0.89±0.53	3.54±0.06	0.54±0.05	46.68±6.42	14.57±9.03	76.59±13.15	8.69±4.09[b]
Significance		NS	**	NS	NS	NS	NS	NS	NS	*
Storage Time (ST)										
0. day	15	74.38±1.94[a]	65.95±1.23[a]	0.35±0.07[c]	3.54±0.04[c]	0.57±0.01[a]	34.52±1.31[c]	23.31±3.69[a]	82.37±8.12[a]	10.12±2.88
20. day	15	66.28±1.98[c]	64.68±0.85[c]	1.10±0.46[b]	3.52±0.05[c]	0.45±0.02[c]	50.16±2.87[b]	9.26±5.58[b]	69.17±10.06[b]	10.02±5.30
40. day	15	70.83±10.32[ab]	64.96±0.89[b]	1.49±1.01[ab]	3.56±0.02[b]	0.53±0.02[b]	52.92±1.90[a]	5.06±2.73[c]	63.31±4.79[c]	9.53±3.39
60. day	15	67.91±2.36[bc]	64.95±0.94[b]	1.62±0.45[a]	3.59±0.04[a]	0.58±0.01[a]	53.54±2.24[a]	6.36±2.45[c]	66.59±5.29[b]	10.25±4.01
Significance		**	**	**	**	**	**	**	**	NS
ST x KJ		NS	**	NS	**	NS	**	**	**	*
ST x EM		*	NS	NS	*	NS	**	**	**	**
KJ x EM		NS	**	NS	*	NS	NS	NS	NS	**
ST x KF x EM		NS	**	NS	NS	NS	NS	**	**	**

* indicates $p<0,05$, ** indicates $p<0,01$, NS non-significant differences $p>0,05$; The averages shown in the same column with the same letter are statistically indistinguishable from each other

4. Conclusion

Pomegranate, black rosehip and black mulberry fruits are fruits rich in antioxidants, anthocyanins, flavonoids, phenolic acids, organic acids, vitamins and minerals. According to the results of the research, it was determined that the addition of black mulberry extract and black rosehip extract prepared by classic method increased the total dry matter, water soluble dry matter, ash, reduce sugar and HMF in jams. In addition, it was determined that total dry matter, water soluble dry matter, sucrose and total sugar decreased when the jams were stored at 20±2 °C for 60 days.

Compliance with ethical standards

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Disclosure of conflict of interest

All authors hereby declare no conflicting interest.

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