



A Study on Changes in Some Physicochemical Properties, Volatile Compounds, Sugar, and Organic Acid Contents of Grape Juice During Molasses Production

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Abstract

This study aimed to investigate both qualitative and quantitative changes in the taste and aroma components of grape juice during the production of traditional molasses (pekmez). For this purpose, the changes in some physicochemical properties, such as sugar, organic acid, and volatile compound contents, in fresh Verdani grape (*Vitis vinifera* L.) juice (FGJ) were evaluated in the production of its traditional molasses. After the production, the total soluble solid (TSS), titratable acidity (TA), glucose, fructose, malic, citric, and succinic acid concentrations were increased ($P<0.05$) with the rising concentration. However, the level of glucose and fructose in TSS decreased by 4.67% and 11.78%, respectively ($P<0.05$), based on their degradation. Similarly, as the major organic acids, the rates of tartaric and malic acids in the TSS were decreased by 73.91% and 67.25%, respectively. These reductions raised the pH value of molasses ($P<0.05$). In addition, the majority of volatile compounds in FGJ disappeared after the production of molasses, whereas some volatile furans were formed in significant amounts.

1. Introduction

Grape (*Vitis vinifera* L.) is one of the most consumed fruits worldwide. The annual amount of grapes produced in the world is approximately 78 million tons. Türkiye is the sixth largest country in the world, with an annual grape production of 4.2 million tons, depending on its suitability for growing conditions [1]. This fruit is mostly consumed fresh in the country, but it is also used in the production of its juice, wine, raisins, vinegar, and certain traditional foods, including molasses and orcik [2]. In 2021, 50.6% of grapes produced in Türkiye were table varieties, 39 percent were raisin varieties, and the rest were wine varieties [3]. In addition to these uses, it is estimated that 10-20% of the total amount of grapes harvested in Türkiye is used in the production of molasses, although the exact amount is unknown [4].

Molasses is a traditional Turkish food and has been produced in Anatolia for a long time, from certain fruits such as grape and mulberry [5]. It is a nutritious food in terms of its high sugar, mineral, and organic acid contents. Moreover, molasses is also used to prepare various desserts due to its typical taste, consistency, and caramel color [6]. The quality of molasses types generally depends on their physicochemical properties, and the composition of their sugars, organic acids, and volatile compounds, as well as their non-enzymatic browning levels. Also, there are some phenolic and flavonoid components in both grapes and molasses that can function as bioactive substances [5]. These properties are affected by the variety, ripening degree, and growing conditions of the grape used [7], but also depend on the production method used [4], [8].

In Turkey, molasses is produced in two ways: traditional and industrial (vacuum) methods. The

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main difference between these two productions is the concentration method followed by the deacidification and clarification of fresh grape juice (must) during the production process. In the vacuum method, the must of grapes is concentrated under a vacuum at a lower temperature (60-70 °C), whereas in the traditional method, the concentration is done by cooking the must in a wide and shallow copper cauldron for a longer time. On the other hand, in some regions of Turkey, the deacidified and clarified must of grapes is concentrated by laying it thinly on the trays and keeping them under the sun, as an alternative traditional method. The molasses obtained in this way is called 'gün pekmezi' or 'gün balı' (like-honey) [2]. Mardin, a province located in the southeast of Turkey, is a region where traditional molasses is widely made. Mardin has the second-largest area of vineyards in the country [3]. In this region, around 30 different local varieties of grapes are grown [9].

There are several studies on the general physicochemical properties of grape molasses. Some of these studies were carried out on molasses whose varieties are known, while [2], [10], some of them were conducted on molasses whose grape variety is not known [11]. Studies have also been conducted to determine the imitation and the adulteration [12]; the rheological properties [13], [14]; changes in the amount of hydroxymethylfurfural [15], [16]; and bioactive properties [17], [8] of the kinds of molasses. As a result of the literature research, it has been seen that the studies comparing the main quality characteristics of some grape varieties with their traditional molasses are limited. Therefore, the purpose of this study is to evaluate the changes in some physicochemical properties, volatile compounds, sugars, and organic acids of *Verdani* (Werdani) grape during its production of traditional molasses.

2. Material and Method

2.1. Material

The mature grapes of a variety of Mardin (*Vitis vinifera* L. cv. *Verdani*) (red) used in this study were collected from a vineyard (37°24'12.4"N 41°16'35.2"E) in Gelinkaya village of Midyat district, Mardin, Türkiye. Approximately 100 kg of the bunches of this grape were harvested (28 October 2019) and transferred to the house where molasses is made by the traditional method, locally called 'mahsere'. The manufacturing of molasses started early the next day. Meanwhile, about one liter of squeezed FGJ obtained from the variety used was stored at -18 °C until analysis.

2.2. Production of Molasses

In this study, the sweet molasses type with a liquid consistency was produced, as specified in the Turkish Food Codex for grape molasses [18]. It was produced by the traditional method (Figure 1). First, the rotten or damaged berries were removed from the bunches, and then the bunches were washed with potable water. After that, the bunches were placed in porous bags and crushed with foot (by using clean plastic boots) on a specially designed sloped concrete floor to collect the squeezed juice into a cauldron. After that, the grape juice (must) obtained (~83 L) was mixed with molasses soil (1.7 kg) with high a CaCO₃ (80-90%) content, then boiled for 10 minutes and also rested for 2 hours to deacidify and clarify it. After this period, the must was taken into an open cauldron (85x25 cm) without removing the sediment and boiled using wood fire for 3.5 hours till it reached a Brix of 70. The foams that formed on the edge of the cauldron during boiling were taken continuously. In addition, the foams adhering to the wall of the cauldron were periodically cleaned with a clean cotton rag during this time. The temperature of the molasses during boiling ranged between 101-112 °C. At the end of production, the obtained molasses was cooled, and a sufficient amount of its samples, taken into plastic tubes, was stored at -18 °C until analysis.

2.3. Physicochemical Analyses

The dry matter (DM) content of samples was determined according to the AOAC (2000) method [19]. The amount of TSS (°Brix) in samples was determined using a digital refractometer (HI 96801, Hanna Instruments Inc., Woonsocket, RI, USA) at 22 °C. The amount of TA was determined potentiometrically by the titration method [20]. The pH value of the samples was measured with a digital pH meter (HI 2211, Hanna Instrument Inc., Limena, Italy).

2.4. Sugar Analyses

The sugar (glucose, fructose, and sucrose) contents of the samples were determined by an HPLC (Waters e2695, Waters, Milford, MA, USA) system by adapting the IHC (2002) method to molasses [21]. A 1 g of sample was dissolved in 10 ml of deionized water and centrifuged at 5000 rpm (2300 g) (Hettich Mikro 220 R; Tuttlingen, Germany) for 5 minutes at 4 °C. The supernatant liquid was passed through a 0.45 µm porous PVDF filter and injected into HPLC. The separations and detection were performed by a YMC-Pack Polyamine II (250x4.6 mm, 5µm) carbohydrate

column and a Refractive Index detector (Waters, USA), respectively. The acetonitrile-water (80:20) mixture was used as the mobile phase with an isocratic flow. The furnace temperature was set to 25 °C, the flow rate to 1.2 ml min.⁻¹ and the injection volume to 20 µl. The

quantitation of sugars was made with calibration curves obtained from commercial standard (Sigma-Merck) solutions prepared at different concentrations, and the results were expressed as g 100 g⁻¹.

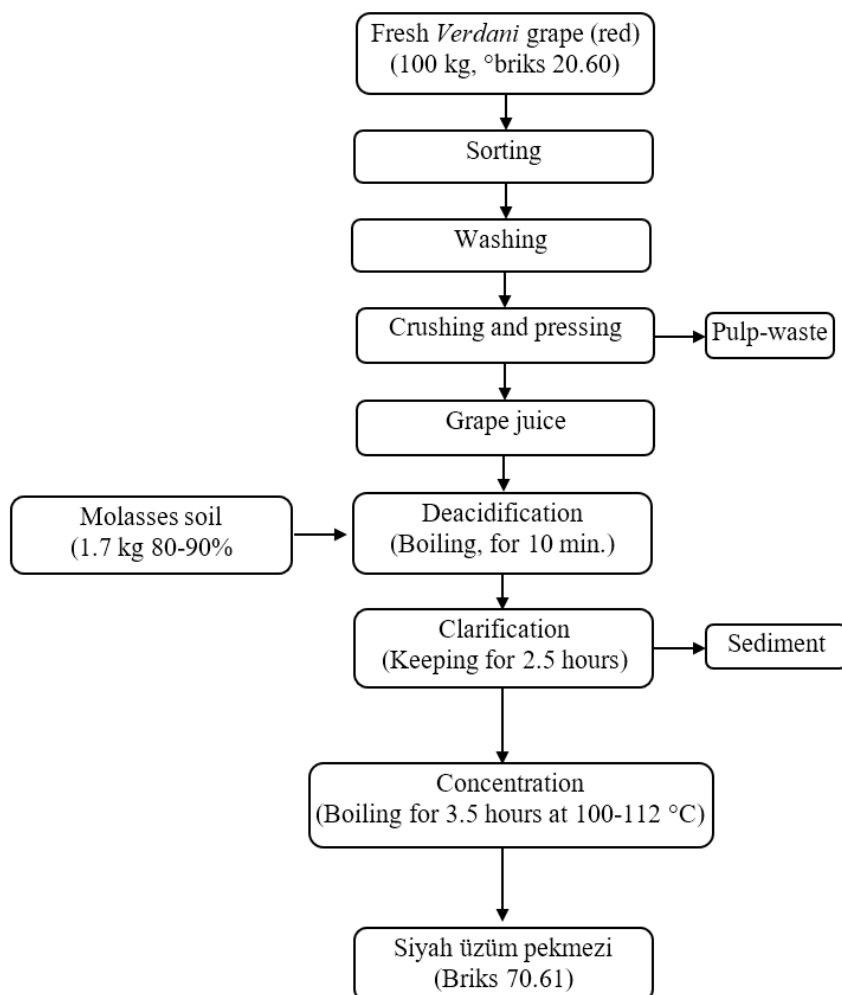


Figure 1. The flow chart of production of the traditional grape molasses

2.5. Organic Acid Analyses

Organic acid (tartaric, citric, malic, and succinic acids) contents were determined by an HPLC (Waters e2695) system [22]. A 2 g of molasses was weighed (10 g for FGJ) and the volume was adjusted to 50 ml with deionized water. Then, the mixture was homogenized, and centrifuged at 5000 rpm for 20 minutes. The supernatant was passed through a 0.45 µm porous PVDF filter and injected into HPLC. The HPLC system was equipped with an Atlantis dC18 (4.6x250 mm, 5 µm,) column at 25 °C and a PDA detector (photodiode array) (Waters) set at 210 nm. 20 mM NaH₂PO₄ solution acidified with H₃PO₄ until pH 2.7 was used as an isocratic elution. The flow rate of the mobile phase was 0.8 ml min.⁻¹, and the injection volume was 10 µl. The quantification of

organic acids was performed with calibration curves obtained from solutions prepared at different concentrations from the commercial standards (Sigma-Merck) of each compound, and the results were expressed as g kg⁻¹.

2.6. Volatile Compound Analysis

Volatile compounds were analyzed by gas chromatography-mass spectrometry (GC-MS) using the Solid Phase Micro-Extraction (SPME) according to Korkmaz et al. (2020) [23].

2.7. Statistical analyses

The statistical differences between the means of the data were determined by the independent sample test

(t-Test) ($P < 0.05$). This statistical analysis was carried out by SPSS (SPSS 16.0 for Windows, IBM, New York, USA) software package.

3. Results and Discussion

3.1. Changes in Physicochemical Properties

DM, TA, and TSS contents and pH values of the FGJ and molasses of the Verdani grape are given in Table 1. TSS ($20.60 \pm 0.15\%$) in the FGJ of the grape used was similar to that of 6 different Brazilian grape varieties [22], whereas it was higher than that of other grape varieties of the same country [24]. Additionally, Dölek (2017) found that TSS and TA in Verdani grape ranged from 17.0% to 19.8% and from 0.61% to 0.78% [25]. The main physicochemical properties such as TSS, DM, TA, and pH in grape varieties are primarily affected by their genetic variability, growing conditions, and ripening degree [26].

TSS and TA increased to $70.61 \pm 0.65\%$ and to $0.921 \pm 0.010\%$, respectively ($P < 0.05$), with the increase in the concentration of molasses during production. The increase in TA level in molasses compared to its level in FGJ was due to the increase in DM during molasses production. It was found that the amount of TA per dry matter decreased by 30.63% during molasses production ($P < 0.05$). This is mostly possible because the main organic acids in the grape are neutralized during the deacidification treatment. Also, it is reported that long-term cooking in molasses production can cause some losses in organic acid [27].

Table 1. Values of quality parameters of the fresh grape juice and the molasses sample

Quality parameters	Fresh grape juice	Molasses
Dry matter (DM) (%)	21.13 ± 0.65^b	74.81 ± 0.37^a
pH	4.23 ± 0.01^b	5.09 ± 0.02^a
Titrateable acidity (TA) (%) [*]	0.375 ± 0.014^b	0.921 ± 0.010^a
Total soluble solid (TSS) (°Brix, %)	20.60 ± 0.15^b	70.61 ± 0.65^a

^{*}g Tartaric acid 100 g⁻¹, TA: Titrateable acidity, ^{a-b} Means with different lowercase in some rows were significantly different between samples ($P < 0.05$).

The increase (by 20.33%) ($P < 0.05$) in pH value at the end of production was the result of this decrease in TA. Türkben et al. (2016) found that the TSS, TA, and pH values of molasses samples made by the traditional method from 14 different grape cultivars ranged between 66.30%-80.27%, 0.27%-1.81% and 3.59-5.23, respectively [28]. These characteristics of molasses types produced from grapes can vary

depending on the concentration method or the production conditions as well as the grape variety used. On the other hand, the molasses obtained in this study was classified as sweet and liquid molasses in terms of pH and TSS [18].

3.2. Changes in Sugar Content

The composition of the sugar and organic acid in fruits has critical effects on their taste and flavor properties and therefore on consumer perception [29]. The sugar and organic acid contents of the samples are presented in Table 2. As the main sugars in grapes, glucose (9.57 ± 0.50 g 100⁻¹) and fructose (9.97 ± 0.41 g 100 g⁻¹) contents in FGJ were found to be close to each other. Their sum (19.55 ± 0.12 g 100 g⁻¹) accounted for 92.47% of the DM in the FGJ. The amount of sucrose was negligible in both FGJ and molasses. Aubert and Chalot (2018) reported that the glucose and fructose contents in six different mature grape varieties varied between 7.58-9.37 g 100 g⁻¹ and 7.91-10.21 g 100 g⁻¹, respectively [30].

Table 2. Sugar and organic acid contents of the fresh grape juice and the molasses

Component	Fresh grape juice	Molasses
Sugar (g 100 g⁻¹)		
Glucose	9.57 ± 0.50^d	32.30 ± 0.29^a
Fructose	9.97 ± 0.41^d	31.14 ± 0.32^a
Total sugar	19.55 ± 0.12^d	63.44 ± 0.17^a
Fructose/glucose	1.04 ± 0.01^a	0.96 ± 0.02^a
Organic acid (g kg⁻¹)		
Tartaric	4.06 ± 0.05^a	3.75 ± 0.06^a
Malic	1.38 ± 0.03^a	1.60 ± 0.10^a
Citric	0.09 ± 0.00^b	0.45 ± 0.03^a
Succinic	0.20 ± 0.00^a	0.35 ± 0.02^a
Total organic acid	5.72 ± 0.07^b	6.15 ± 0.05^a

^{a-b} Means with different lowercase in some rows were significantly different between samples ($P < 0.05$).

In the molasses sample, glucose and fructose contents increased to 32.30 ± 0.29 g 100 g⁻¹ and 31.14 ± 0.32 g 100 g⁻¹, respectively, due to the concentration process during production. However, as a point to be noted, it was found that during the conversion of FGJ into molasses, there were decreases in the amounts of both glucose (4.67%) and fructose (11.78%) based on their percent in DM ($P < 0.05$). The fructose/glucose ratio decreased partially ($P > 0.05$) as a result of the decrease in the amount of fructose being higher than that of glucose. The higher decrease in fructose during the molasses production could be explained by the greater participation of this sugar in Maillard and/or caramelization reactions compared to glucose [31].

Türkben et al. (2016) reported that the amounts of glucose and fructose in 14 different traditional molasses from different varieties of grapes ranged from 27.57 g 100⁻¹ to 41.11 g 100⁻¹ and from 22.34 g 100⁻¹ to 34.69 g 100 g⁻¹, respectively [28]. The sugar content of different molasses types is mostly affected by the duration of the concentration process.

3.3. Changes in Organic Acid Content

Tartaric and malic acids are the main organic acids in grape varieties and the total amount of the two accounts for more 80% of the total organic acid contents in many grapes [24]. It was found that the sum of (5.44±0.08 g kg⁻¹) of tartaric (4.06±0.05 g kg⁻¹) and malic (1.38±0.03 g kg⁻¹) acids in the FGJ corresponded to 94.93% of the total organic acid content (Table 2). In a previous study, it was reported that the tartaric, malic, and citric acid contents in 11 different cultivars of grapes were between 4.98-7.48, g l⁻¹, 1.43-3.40 g l⁻¹ and 0.03-0.164 g l⁻¹, respectively [7]. In another study, it was found that tartaric and malic concentrations in 6 different varieties of grapes ranged between 4.3-6.2 g l⁻¹ and 1.5-2.9 g l⁻¹, respectively [30]. The organic acid content in grapes depends on genetic characteristics, growing conditions, and maturity level [24].

Tartaric, malic, citric, and succinic acid contents in the molasses sample were found to be 3.75±0.06 g l⁻¹, 1.60 ± 0.10 g l⁻¹, 0.45 ± 0.03 g l⁻¹ and 0.35±0.02 g l⁻¹, respectively. The amount of major organic acids in FGJ decreased during the production of molasses, probably due to the deacidification process, which precipitated tartaric and malic acids as salts of calcium tartrate and calcium maleate, respectively. Based on the ratios of tartaric and malic acids in the DM, it was also found that the initial contents of tartaric and malic acid were decreased by 73.91% and 67.25%, respectively (P<0.05). In addition, some of the reductions in organic acid amounts could be due to the boiling treatment during production [27]. It was reported that the reductions in malic, citric, and tartaric acid of a grape molasses type produced by the traditional method were 9.19%, 24.61%, and 3.89%, respectively [32].

3.4. Changes in volatile compounds

The profile of volatile compounds in fruits and vegetables has a wide diversity and affects their aroma characteristics [33]. The volatile compounds identified in FGJ and molasses are given in Table 3. In the FGJ sample, a total of 28 different volatile compounds were identified in different groups:

terpenoid (6), aldehydes (7), alcohols (10), ketones (2), ester (1), acid (1) and other (1).

Terpenoids and various aldehydes, alcohols, and ketones formed by lipoxygenase (LOX) enzyme activity were the most common volatile compounds in FGJ, both quantitatively and qualitatively. Terpenoids in grapes are generally responsible for their characteristic floral odor [30]. D-Limonene (152.91 ±5.07 mg kg⁻¹), β-linalool (11.40±0.58 mg kg⁻¹), carvone (43.02 ±4.62 mg kg⁻¹) and o-cymene (10.65±0.49 mg kg⁻¹) compounds were found as the most common terpenoids in FGJ. Hexanal (14.05±2.27 mg kg⁻¹), (E)-2-hexenal (27.81±8.94 mg kg⁻¹), 1-hexanol (178.92±9.21 mg kg⁻¹), (Z)-3-hexenol (11.68±0.43 mg kg⁻¹) and (E)-2-hexenol (50.04±2.69 mg kg⁻¹), which have fresh leaf grassy aroma, were found to be other compounds with higher amounts in the FGJ sample. Most of these volatile compounds, known also as C6-compounds, are formed by the LOX activity of certain unsaturated fatty acids. These compounds have also been found as predominant volatile compounds in different grape varieties in previous studies [34, 35]. Apart from these, hexyl phenylacetate (23.11±2.05 mg kg⁻¹), nonanal (15.13±1.48 mg kg⁻¹), ethanol (11.35±2.06 mg kg⁻¹), 6-methyl-5-hepten-2-one (13.36±0.26 mg kg⁻¹) and nonanoic acid (36.59±4.11 mg kg⁻¹) were found to be other important volatile compounds in FGJ.

The composition of volatiles in FGJ changed markedly during its conversion into molasses. The majority of the number (20) of compounds responsible for freshness and fruity odors in FGJ disappeared during the production of molasses, probably because of their degradation and/or volatilization during the production processes [36]. Conversely, 11 new compounds responsible for typical grape molasse were formed in the molasses. In particular, some volatile furans were formed abundantly during molasses production. As the newly formed furans, the concentration of furfural, 2-acetylfuran, 5-methylfurfural, dihydro 2 (3H)-furanone, 2-furanmethanol and methyl 2-furoate in molasses were 319.16±36.61 mg kg⁻¹, 24.53±2.92 mg kg⁻¹, 21.93±3.95 mg kg⁻¹, 7.28±2.24 mg kg⁻¹, 16.12±0.53 mg kg⁻¹, 6.11±1.53 mg kg⁻¹, respectively. Among these, only the amount of furfural corresponded to 57.37% of the total amount of volatile compounds in the molasses sample. These furans are responsible for the characteristic caramel, roasted-cooked and coffeelike aromas of grape molasses. They can be formed by Maillard reactions [32], Strecker degradation [23] and caramelization reactions [37] during the cooking process in production. Various volatile furan compounds have

also been found in other molasses types made from various grapes [38], [32].

Table 3. Volatile compound contents in the fresh grape juice and the molasses (mg kg⁻¹)

No	Compound	RI*	Fresgh grape juice	Molasses
	Total terpenoid		231.65±5.66^a	4.39±1.38^b
1	D-Limonene	1231	152.91±5.07 ^a	nd
2	1,8-Cineole	1233	7.33±2.01 ^a	nd
3	γ-Terpinene	1268	6.35±0.98 ^a	nd
4	o-Cymene	1287	10.65±0.49 ^a	nd
5	β-Linalool	1553	11.40±0.58 ^a	4.39±1.38 ^b
6	Carvone	1760	43.02±4.62 ^a	nd
	Total aldehyde		84.60±9.77^a	31.71±7.38^b
7	2-Methylbutanal	979	5.57±0.19 ^a	6.16±1.91 ^a
8	3-Methylbutanal	983	4.71±0.55 ^a	7.71±1.23 ^a
9	Hexanal	1121	14.05±2.27 ^a	nd
10	(E)-2-Hexenal	1245	27.81±8.94 ^a	nd
11	(Z)-2-Heptenal	1344	8.69±1.47 ^a	nd
12	Nonanal	1410	15.13±1.48 ^a	nd
13	Phenylmethanal	1546	8.62±0.24 ^a	nd
14	Phenylethanal	1666	nd	10.52±1.95 ^a
15	2,4-Dimethylbenzaldehyde	1840	nd	7.31±2.28 ^a
	Total alcohol		329.31±8.78^a	17.07±5.34^a
16	Ethanol	997	11.35±2.06 ^a	2.31±0.28 ^b
17	3-Methylbutanol	1230	28.17±2.53 ^a	nd
18	1-Hexanol	1366	178.92±9.21 ^a	nd
19	(Z)-3-Hexenol	1396	11.68±0.43 ^a	nd
20	(E)-2-Hexenol	1418	50.04±2.69 ^a	nd
21	1-Heptanol	1464	3.91±0.37 ^a	nd
22	2-Ethylhexanol	1497	16.98±2.47 ^a	5.81±0.61 ^a
23	2-Heptenol	1520	8.17±1.29 ^a	nd
24	1-Octanol	1565	10.69±0.56 ^a	nd
25	2-Tridecanol	1723	nd	1.47±0.10 ^a
26	β-Phenylethanol	1928	8.76±0.44 ^a	8.94±2.58 ^a
	Total cetone		19.87±1.49^a	9.87±2.85^a
27	3-Octanone	1277	6.51±0.88 ^a	nd
28	Hydroxyacetone	1323	nd	9.87±2.85 ^a
29	6-Methyl-5-hepten-2-one	1357	13.36±0.26 ^a	nd
	Total ester		23.11±2.05^a	nd
30	Hexyl phenylacetate	1666	23.11±2.05 ^a	nd
	Total acid		36.59±4.11^b	109.77±16.96^a
31	Acetic acid	1475	nd	51.92±5.61 ^a
32	Nonanoic acid	2184	36.59±4.11 ^a	49.51±1.08 ^a
33	Decanoic acid	2271	nd	8.34±2.27 ^a
	Total furan		nd	395.15±46.66^a
34	Furfural	1484	nd	319.16±36.61 ^a
35	2-Acetylfuran	1524	nd	24.53±2.92 ^a
36	5-Methylfurfural	1592	nd	21.93±3.95 ^a
37	Dihydro 2(3H)-furanone	1653	nd	7.28±2.24 ^a
38	2-Furanmethanol	1671	nd	16.12±0.53 ^a
39	Methyl 2-furoate	2040	nd	6.11±1.53 ^a
	Miscellaneous		3.31±0.63^a	3.56±0.34^a
40	2,3-Bütandiol	1557	3.31±0.63 ^a	3.56±0.34 ^a
	Total		728.17±18.44^a	556.27±61.30^a

*RI: Retention index calculated on DB-HeavyWax column, nd: not detected, ^{a-b} Means with different lowercase in some rows were significantly different between samples (P<0.05)

4. Conclusion and Suggestions

In this study, the changes in physicochemical properties, sugar, organic acid, and volatile compound content of the *Verdani* grape variety were evaluated for the first time during the production of its molasses by the traditional method. The initial TSS, TA, sugar, and organic acid contents increased depending on the rising concentration during the processing of FGJ into the molasses. However, the results showed that the method of traditional molasses production caused significant losses in the amounts of glucose, fructose, tartaric acid, and malic acid, indicating that this method can reduce their portion in the total dry matter after the molasses production. On the other hand, the data showed that the most of volatile compounds in FGJ disappeared during the conversion into its molasses, and also indicated that the traditional manufacturing method of grape molasses can lead to the generation of volatile furans in large amounts. Based on the results of this study, an evaporation under vacuum or open natural

conditions instead of the long-term and high-temperature cooking process in the traditional method can be more suitable for the concentration process in molasses production. In future studies, the effects of this production method on hydroxymethylfurfural (HMF) and bioactive properties in grapes should be investigated.

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Statement of Research and Publication Ethics

The study is complied with research and publication ethics

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