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Determination of Changes in Volatile Aroma Components, Antioxidant Activity and Bioactive Compounds in the Production Process of Jujube (*Ziziphus jujuba* Mill.) Vinegar Produced by Traditional Methods

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Abstract: Jujube has anticancer, diabetic, antimicrobial, anti-inflammatory, cardiovascular, gastrointestinal and immune system effects. In this study, jujube juice, jujube wine and jujube vinegar were investigated in terms of chemical composition, phenolic contents, organic acid contents, volatile compound contents, and antioxidant activity. Antioxidant activity and total phenolic content of jujube vinegar produced by traditional methods were found to be higher than those of jujube juice and wine. Protocatechic acid, chlorogenic acid, phydroxybenzoic acid, caffeic acid, epicatechin, and syringic acid were detected in jujube vinegar. Moreover, oxalic acid, malic acid, tartaric acid, formic acid, ascorbic acid, lactic acid, acetic acid and some other organic acid components were determined in jujube vinegar. Volatile aroma compounds such as ester, aldehyde, alcohol, terpene, acid, and ketone were determined in jujube samples. It was seen that the antioxidant activity and bioactive compounds of jujube vinegar were very rich, and jujube vinegar, which is an alternative product with a high potential produced from jujube fruit, is an important product for the food sector due to its long shelf life. This research is the first detailed study in which the antioxidant activity and bioactive compounds determined during the production stages of jujube vinegar (jujube juice, wine, and vinegar) were evaluated in detail.

Keywords: jujube vinegar; bioactive compounds; volatile compounds; phenolic content; antioxidant activity; organic acid content; TEAC; chlorogenic acid; acetic acid; ethyl acetate

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1. Introduction

Jujube (*Ziziphus jujuba* Mill.) is a deciduous tree or large shrub, belonging to the *Rhamnaceae* family. Its unripe fruits are green, and its ripe fruits can be of different colors, from yellow or red to brown. It is primarily planted for edible fruit, however, it is also used as a medicinal agent. The homeland of the jujube plant is China. It is also grown in Russia, the Middle East, India, Southern Europe, Anatolia, and North Africa [1,2]. Jujube is grown intensively in the Western and Southern provinces of Turkey [3].

In the prescription prepared by Ibn Sina, the jujube plant was used as a cough suppressant, laxative, blood pressure reducer, digestive disorder remedy, and in the treatment of stomach ulcers [4]. In China, the ripe and dried fruits of the jujube fruit, called the "fruit of life", have been used as an aphrodisiac, laxative, and antidote [5]. Recent studies have shown that jujube has anticancer, antidiabetic, antimicrobial, anti-inflammatory, sedative, laxative, cardiovascular, gastrointestinal, and immune system effects, and that phytochemicals in the jujube plant are effective in the treatment of these diseases [2,6–11].

Jujube fruit has high nutritional value. Its nutritional content comprises carbohydrates, proteins, fats, vitamins, minerals, and phenolic compounds [12]. The jujube plant contains galactose, rhamnose, mannose, glucuronic acid, arabinose, glucose, fructose, sucrose, and sorbitol sugars [13]. It has been reported that the diabetic effect of jujube fruit prevents the excessive increase in blood glucose by reducing the glucose uptake of the cells during the glucose absorption stage and delays the diffusion of glucose through the dialysis membranes [9]. Also, the jujube plant is recommended for the treatment of diabetic patients due to its antioxidant capacity and significant inhibitory effect on alpha-amylase [14].

Jujube fruit contains calcium, potassium, bromine, rubidium, lanthanum, magnesium, sodium, iron, zinc, and manganese in terms of mineral substances; retinol, thiamine, riboflavin, niacin, pyridoxine, cyanocobalamin, and ascorbic acid in terms of vitamins; caffeic acid, ferulic acid, p-hydroxybenzoic acid, chlorogenic acid, gallic acid, protocatechuic acid, vanillic acid, p-coumaric acid, ellagic acid, and cinnamic acid in terms of phenolic acids; and procyanidin B2 epicatechin, quercetin-3-O-rutinoside, quercetin-3-O-galactoside, kaempferol-glycosyl-rhamnose, catechin, epicatechin, and rutin in terms of flavonoids [13,15,16]. Keleş [17] determined the organic acid content of jujube fruit to be 84.72 mg/100 g malic acid, 52.35 mg/100 g citric acid, 78.73 mg/100 g succinic acid, and 51.94 mg/100 g ascorbic acid. Vithlani and Patel [18] defined jujube vinegar as a functional vinegar due to its high-level antioxidant activity. The bioactive compounds of jujube fruit are associated with its health benefits. Therefore, jujube fruit is regarded as a potential alternative functional food product. Today, there is an increase in the demand for natural and traditional products. It has been reported that the antioxidant activity and bioactive compound levels of vinegar produced by the traditional methods (surface culture production) were very high [19]. The biochemical reactions that occur in the traditional method of vinegar production are described in two stages. In the first stage, yeast converts sugar to ethanol in an anaerobic environment, and in the second stage, acetic acid bacteria converts ethanol to acetic acid in an oxygenated (aerobic) environment [20,21]. The present study aimed to determine the antioxidant activity, volatile aroma components, organic acid and phenolic compounds of jujube vinegar produced by traditional methods, and to evaluate the potential of jujube vinegar produced from jujube fruit. The scientific studies on this subject are limited, therefore, important information will be provided for those who want to industrially produce jujube vinegar.

2. Materials and Methods

2.1. Chemicals

Chemicals were obtained from Supelco Co. (Bellefonte, PA, USA), Sigma-Aldrich Co. (St. Louis, MO, USA), and Merck Co. (Darmstadt, Germany). The wine yeast culture (*Saccharomyces cerevisiae* strain, ConFermUni V yeast) was supplied from Eaton's Begerow® Product Line Co. (Nettersheim, Germany).

2.2. Materials

Ripe jujube (20 kg) was obtained from the province of Isparta, Turkey, to be used in the production of jujube wine and vinegar. Also, two-year-old jujube vinegar produced by the traditional methods was provided for the vinegar production phase.

2.3. Preparation of Inoculums

Activation of yeast (*Saccharomyces cerevisiae*) prepared for use in wine production was carried out according to Özen et al. [22].

2.4. Production of Jujube Vinegar

Jujube vinegar was produced by the ethanol and acetic acid fermentation stages according to the traditional vinegar production technique [21]. The production flow diagram is given in Figure 1. Jujube fruits were harvested from the jujube plant. Jujube (*Ziziphus jujuba* Mill.) fruits were selected from non-bruised, intact fruits. The fruits were carefully rubbed and washed. The fruits were cut into pieces and filled into jars and the jars were filled with sterile tap water, and their lids were closed. The jars were agitated from time to time without opening their lids. The jars were kept at +10 °C for one week. Jujube juice (JJ) was obtained by filtering the mixture with cheesecloth. The °Brix was adjusted to

12 °Bx by adding sugar to the JJ. Then, alcohol fermentation was initiated by adding yeast (*Saccharomyces cerevisiae*) to the JJ. Alcohol fermentation was carried out at 25 °C for 30 days. At the end of the period, jujube wine (JW) was obtained, which was both a by-product and a raw material for the second stage of the fermentation. For acetic acid fermentation, aged vinegar was inoculated (1/3 ratio). Acetic acid fermentation lasted 60 days at 25 °C for jujube vinegar (JV). The jujube vinegar production was repeated in two parallel, and three replications. All analysis were carried out in two parallels.

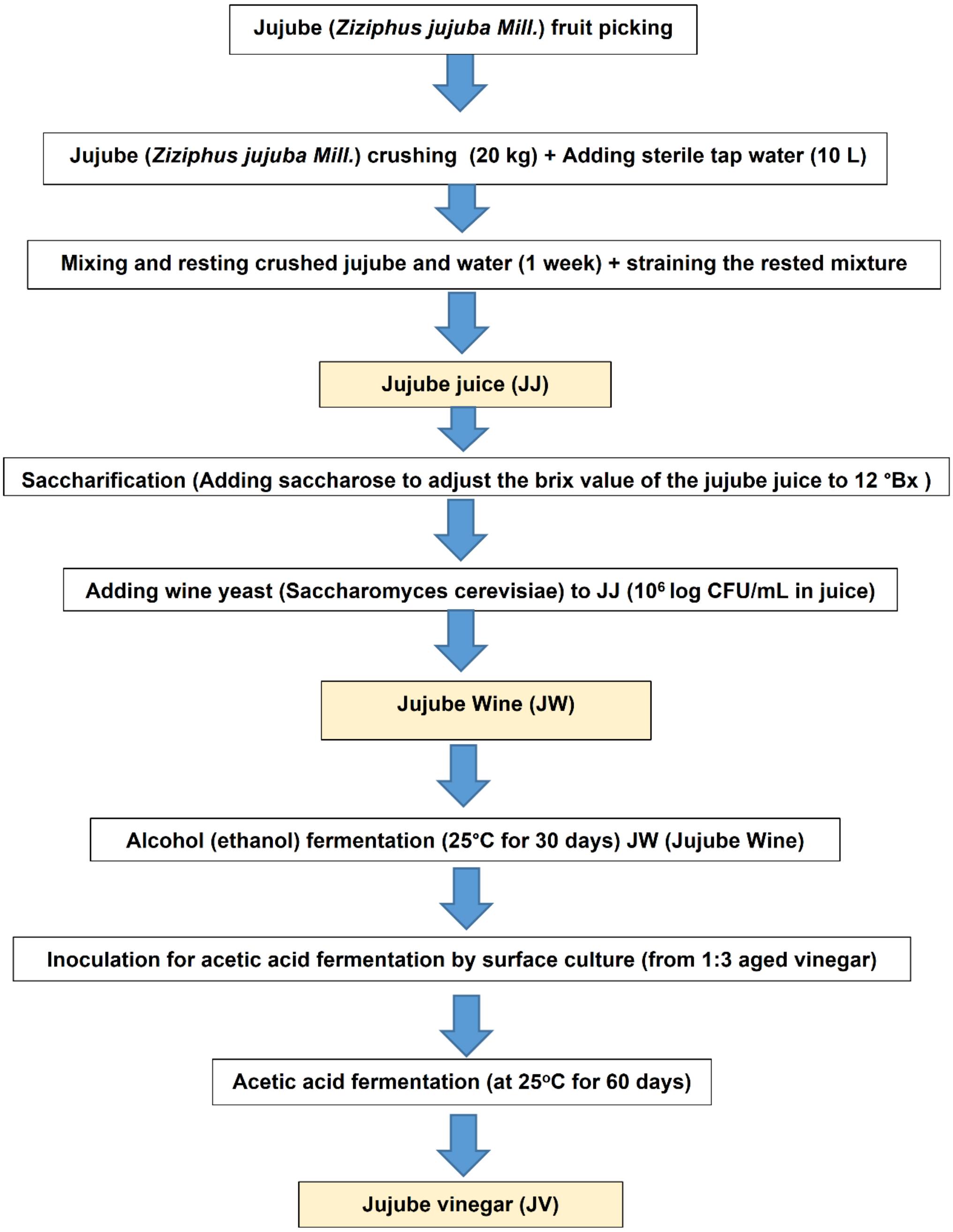

Figure 1. Jujube wine and vinegar production flow chart.

2.5. Proximate Composition Analysis

Chemical analysis applied to the samples were determined according to AOAC [23]. Total titratable acidity (TTA), total dry matter (%) and pH analysis were performed. TTA value was determined as lactic acid (%) in the fruit juice and wine samples and as acetic acid (%) in the vinegar sample. The Abbe refractometer (Bellingham Stanley Limit 60/70 Refractometer, UK) was used to determine the total soluble solids (TSS, °Brix).

2.6. Organic Acid Compound Analysis

Organic acid compound (OACs) analysis was performed using an HPLC device. The device was used effectively at the Innovative Technologies Application and Research Center (YETEM) at Süleyman Demirel University. The OACs of the samples were determined by the method decscribed by Özdemir and Budak [24]. HPLC instrument (Shimadzu SIL–20AC, Scientific Instruments, Inc., Tokyo, Japan) consists of a UV-VIS detector (SPD-10Avp), a pump (LC-20AT projection), a gas separator (DGU-20A5 projection), a column oven (CTO-10AS vp), an LC-20AT projection system controller, and a column (Technochroma TRACER EXTRASIL ODS(2) (250 × 4.6 mm) 5µ TR-016059). The supelco C18 solid phase cartridge was used in the analysis and the injection volume of the device was 20 µL [24,25].

2.7. Phenolic Compound Analysis

Phenolic compound analysis were performed using an HPLC device at the Innovative Technologies Application and Research Center (YETEM) at Süleyman Demirel University. The phenolic compounds of the samples were determined according to Özdemir and Budak [24]. The HPLC instrument (Shimadzu SCL-10A, Scientific Instruments, Inc., Tokyo, Japan) consists of a CTO-10Avp colon oven, an SPD-M 10A vp DAD detector (λmax = 278 nm), a SIL–10AD vp autosampler, an LC-10ADvp pump, a DGU-14A degasser, and a column (Agilent Eclipse XDB-C18 (250 × 4.60 mm) 5 µm; GL Sciences Inc., Southern California, CA, USA) [24,26]. The injection volume of the device was 20 µL. Process steps in HPLC are given in Table 1.

Table 1. HPLC program for phenolic compound analysis.

Time (Min)	Methanol (%; Pump B)
0.10	7
20.00	28
28.00	25
35.00	30
50.00	30
60.00	33
62.00	42
70.00	50
73.00	70
75.00	80
80.00	100
81.00	7

2.8. The Total Phenolic Content and Antioxidant Activity Analysis

The total phenolic content (TPC) of the samples was determined by the Folin–Ciocalteau method [27], while the antioxidant activity (TEAC) was determined by the 2,2′-azinobis (3-ethylbenzothiazoline)-6-sulfonic acid (ABTS) method [28]. The units of analysis of the samples were expressed as mg GAE/L for the total phenolic content and as mmol TE/L for TEAC analysis.

2.9. Volatile Aroma Component Analysis

The volatile compounds of the samples were performed using a gas chromatography-mass spectroscopy device at the Innovative Technologies Application and Research Center (YETEM) at Süleyman Demirel University. The gas chromatography-mass spectroscopy adopted the solid-phase microextraction (SPME) procedure (GC-MS-QP2010 SE; Shimadzu GC-2010 Plus Capillary, Scientific Instruments, Inc., Tokyo, Japan) [29] and modifying the method by Özen et al. [22]. Injection mode, injection temperature, column oven temperature, and mobile phase (helyum) flow rate were split (ratio; 10.0), 250.0 °C, 40 °C, and 1.50 mL/min, respectively. A Restek Rx-5Sil MS column (30 m × 0.25 mm, 0.25 µm) was used. Regarding the GC program procedure, the column oven temperature was kept at 40 °C for 2 min. Then, it reached 250 °C with an increase of 4 °C/min and was kept at this temperature for 5 min. Total time was 59.50 min. Volatile compounds were absorbed into a fused silica SPME fiber (CAR/PDMS Stable Flex, Supelco, Bellefonte, PA, USA) and analyzed based on the schedule outlined above. Compounds were identified using linear retention indices (LRIs) and by the Wiley, Nist, Tutor, and FFNSC libraries in the MS instrument [24], with component results presented in percentiles.

2.10. Statistical Analysis

The statistical analysis were performed using IBM SPSS v. 22.0 (SPSS Inc., Chicago, IL, USA) and calculating using the One-Way ANOVA test. The Duncan test was used for statistically significant results ($p < 0.05$) between fruit juice, wine, and vinegar samples. Also, the *t*-student test was carried out to reveal the statistical differences between the samples.

3. Results and Discussion

3.1. Chemical Contents of Jujube Juice, Wine, and Vinegar

Total titratable acidity (TTA), pH value, total dry matter (%), and total soluble solids (°Brix), of the JJ, JW, and JV samples are presented in Table 2.

Table 2. Chemical contents of jujube juice (JJ), jujube wine (JW) and jujube vinegar (JV).

Sample	pH-Values	Total Dry Matter (%)	Total Soluble Solids (°Brix)
JJ	3.71 ± 0.05^{a}	11.98 ± 0.04^{a}	12.00 ± 0.01^{a}
JW	3.55 ± 0.04^{b}	4.05 ± 0.66^{b}	6.10 ± 1.27^{b}
JV	3.24 ± 0.01^{c}	2.17 ± 0.49^{b}	4.75 ± 0.35^{b}

JJ; jujube juice, JW; jujube wine; JV; jujube vinegar. The results were given as mean ± standard deviation. Different lowercase letters in the same column indicate significant differences between the samples ($p < 0.05$).

The total soluble solids content (TSS) of the jujube juice was set to 12.00 °Brix at the onset of the fermentation, as described by Kim et al. [30]. In our research, it was observed that fermentations started a 12.00 °Brix in the jujube vinegar process and decreased to 6.10 °Brix at the end of the ethanol fermentation, and to 4.75 °Brix at the end of the acetic acid fermentation. The °Brix value of jujube juice was found to be considerably higher than the °Brix value of wine and vinegar samples ($p < 0.05$). Because of this, the sugar in the fruit juice was used by yeasts in alcohol fermentation and turned into ethyl alcohol [31]. Total dry matter showed a similar trend as the total soluble dry matter in this jujube sample. The total dry matter value of the JJ sample decreased from 11.98% to 4.05% in the JW sample and by 2.17% in the JV sample ($p < 0.05$). Pashazadeh et al. [32] emphasized that the total dry matter in vinegar content is based on water-soluble dry matter. In different studies, it has been stated that the total soluble solids value of jujube fruit is 8.1 (°Brix) [33] and the total soluble solids value of four different Spanish jujube fruits is in the range of 14.6–18.4 (°Brix) [13].

Total titratable acidity (TTA) values of JJ, JW, and JV samples were determined to be 0.64, 1.66, and 6.35 g/100 mL, respectively. ($p < 0.05$). Total acidity increased significantly in acetic acid fermentation ($p < 0.05$). Organic acids, and especially acetic acid content, that occur in acetic acid fermentation increases the acidity of vinegar. Vinegar samples are expected to contain at least 4% acid [19]. The pH values of jujube juice, wine and vinegar decreased significantly during the fermentation process. Changes in the pH and TTA values showed similarity in the opposite direction. However, the increase in TTA value in acetic acid fermentation was observed significantly more than the decrease in pH value. It has been stated that the changes in pH value depends on the hydrogen ion concentration [24,34].

3.2. Organic Acid Compound Profiles of Jujube Juice, Wine, and Vinegar

The organic acid content of vinegar varies according to the fruit chosen, the ripening time of the fruit, the type of fermentation, the fermentation period, and the production method of the vinegar [35]. Organic acid contents of JJ, JW, and JV samples are presented in Table 3.

Table 3. Organic acid contents (ppm) of jujube juice (JJ), jujube wine (JW) and jujube vinegar (JV).

Organic Acid Compounds	Jujube Juice (JJ)	Jujube Wine (JW)	Jujube Vinegar (JV)
Oxalic acid	56 ± 0.01 ^{b}	57.2 ± 0.02 ^{b}	75.1 ± 0.04 ^{a}
Tartaric acid	2232.9 ± 20.4 ^{a}	2075.4 ± 24.2 ^{a}	993.5 ± 18.0 ^{b}
Formic acid	865.4 ± 2.40 ^{a}	677.9 ± 3.24 ^{b}	538.5 ± 4.52 ^{c}
Malic acid	919.4 ± 5.65 ^{a}	918.8 ± 8.86 ^{a}	655.2 ± 6.42 ^{b}
Ascorbic acid	95.5 ± 2.02 ^{b}	323.3 ± 12.4 ^{a}	57.3 ± 1.04 ^{b}
Lactic acid	7737.8 ± 64.0 ^{b}	13,231.4 ± 96.2 ^{a}	2030.1 ± 20.4 ^{c}
Acetic acid	2198.5 ± 14.6 ^{b}	2651.2 ± 21.4 ^{b}	46,375.3 ± 62.4 ^{a}
Citric acid	391.1 ± 5.40 ^{b}	315.2 ± 4.86 ^{b}	530.7 ± 6.20 ^{a}
Succinic acid	794.3 ± 4.20 ^{b}	2288.1 ± 16.2 ^{a}	843.4 ± 8.20 ^{b}
Fumaric acid	n.d.	n.d.	3.2 ± 0.01 ^{a}

JJ; jujube juice, JW; jujube wine; JV; jujube vinegar. The results were given as a mean ± standard deviation. Different lowercase letters in the same column indicate significant differences between the samples ($p < 0.05$). Abbreviations: n.d., not detected.

The main organic acids in jujube juice were lactic acid, tartaric acid, and acetic acid followed by lower levels of malic acid, formic acid, succinic acid, citric acid, ascorbic acid, and oxalic acid. Organic acid values of fresh jujube fruit were determined to be 206.7 mg/100 g DW malic acid, 198.9 mg/100 g DW citric acid, and 14.8 mg/100 g DW succinic acid [36]. In another study, malic acid (219.83 mg/100 g), citric acid (108.14 mg/100 g), succinic acid (188.97 mg/100 g), and ascorbic acid (89.63 mg/100 g) were determined in organic acid results of jujube fruit [17]. Ascorbic acid is present in plant tissues bound to glucose [37]. During the jujube extraction, hygienic conditions and an oxygen-free environment were created, and yeast and mold formation were prevented. However, lactic acid formation was observed as a result of lactic acid fermentation during jujube extraction and alcohol fermentation. Ascorbic acid is released as a result of the biosynthesis of glucose in ethanol fermentation. The ascorbic acid value was determined to be 323.3 mg/L in the JW sample. Fumaric acid was not detected in the JJ or JW samples. Oxalic acid, malic acid, tartaric acid, formic acid, ascorbic acid, lactic acid, acetic acid, citric acid, succinic acid, and fumaric acid were detected in the vinegar samples. The tartaric acid, malic acid, and formic acid levels decreased during the fermentation period whereas the oxalic acid and acetic acid levels increased. It was determined that the citric acid value decreased during the alcohol fermentation, whereas it increased during the acetic acid fermentation. Xiang et al. [38] have reported that citric acid concentrations decreased during alcohol fermentation. The malic acid values of the jujube juice and wine samples were 919.4 and 918.8 mg/L, respectively. The malic acid content of the jujube vinegar sample was determined to be 655.2 mg/L. It was determined that malic acid value decreased during acetic acid fermentation. Sanarico et al. [39] have reported that citric acid, malic acid, oxalic acid, and lactic acid values decreased, and this result was associated with the oxidation by acetic acid bacteria (*Acetobacter* spp. and *Gluconobacter* spp.). The highest organic acid value in jujube vinegar was acetic acid. The acetic acid value of JV was 46,375.3 mg/L. This was an expected result since vinegar contains high levels of acetic acid.

It has been stated that organic acids in foods can be of fermentation origin as well as raw materials, processing, and aging conditions. Organic acids are formed as a result of hydrolysis, biochemical metabolism and microbial activity during fermentation. Organic acids affect the sensory and microbiological properties of fermented products [40].

3.3. Phenolic Compound Profiles of the Jujube Juice, Wine, and Vinegar

The jujube juice, wine, and vinegar were evaluated for phenolic compounds gallic acid, protocatechuic acid, catechin, p-hydroxybenzoic acid, chlorogenic acid, caffeic acid, epicatechin, syringic acid, vanillin, p-coumaric acid, benzoic acid, eriodictiol, quercetin, and campherol (Figure 2). Phenolic components that were not detected in the samples were ferulic acid, sinapinic acid, o-coumaric acid, rutin, hesperidin, rosmarinic acid, cinnamic acid, and luteolin. Gallic acid, catechin, syringic acid, p-coumaric acid, benzoic acid, eriodictiol, and quercetin were not detected whereas protocatechuic acid, p-hydroxybenzoic acid, chlorogenic acid, caffeic acid, epicatechin, vanillin, and campherol were detected in the JJ samples. Gallic acid, catechin, p-coumaric acid, eriodictiol, and quercetin were determined to be 2.1, 1.4, 0.1, 0.2, and 1.6 mg/L in the jujube wine samples, respectively. p-hydroxybenzoic acid and campherol values of the jujube wine were determined to be higher than those of the jujube juice ($p < 0.05$). Albeit statistically not significant, the chlorogenic acid, epicatechin, and vanillin values of jujube wine were determined to be higher than those of the jujube juice ($p > 0.05$). Some phenolic acids found in fruit and flower groups are dependent on fruit sugar. The use of sugar in the environment by wine yeast (*S. cerevisiae*) in alcohol fermentation releases phenolic acids bound to sugar [41].

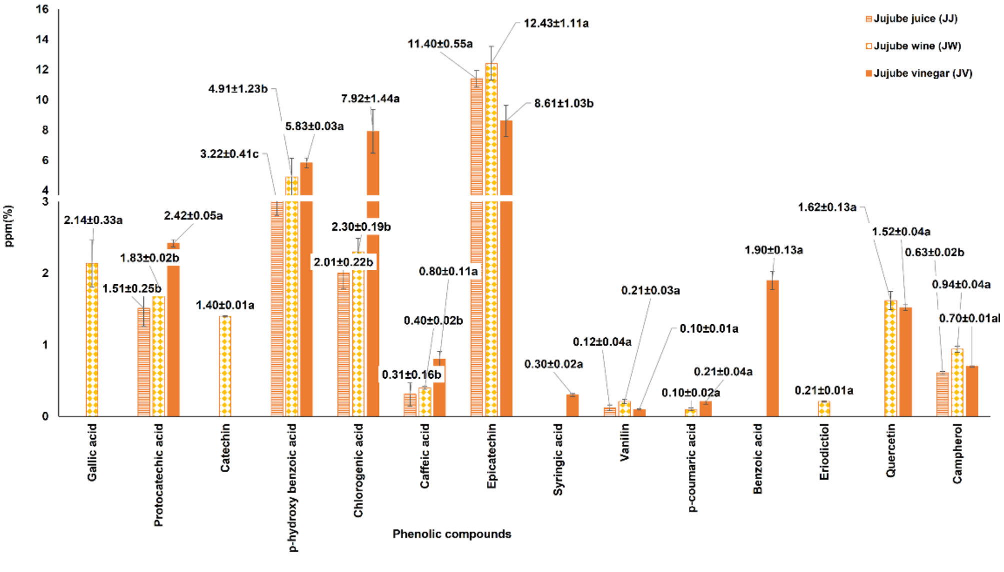

Figure 2. Phenolic compounds of jujube juice (JJ), jujube wine (JW), jujube vinegar (JV) (ppm). Different letters in the figure indicate significant differences between samples.

Gallic acid, catechin, and eriodictiol values of the jujube wine sample were also determined at very low concentrations whereas they were not detected in the JV sample. Yıkmış et al. [42] examined phenolic compounds in jujube vinegar samples and determined caffeic and ferulic acids as the most dominant phenolic compounds. It was found that some phenolic compounds in the environment such as gallic acid decreased since fermentation with oxygen takes place during acetic acid fermentation in vinegar formation [43]. Protocatechuic acid, chlorogenic acid, p-hydroxybenzoic acid, and caffeic acid values showed a gradual increase during the alcohol and acetic acid fermentations. The increase in these phenolic acids were significant in the vinegar example ($p < 0.05$). Laranjinha et al. [44] have stated that chlorogenic acid had a positive effect on health, especially on cardiovascular diseases. It has been explained that this effect occurs by inhibiting LDL oxidation. Also, it has been reported that p-hydroxybenzoic acid has antimicrobial, antialgal, antimutagenic, antiestrogenic, hypoglycemic, anti-inflammatory, anti-platelet aggregation, nematicidal, antiviral, antioxidant, etc. effects. Some 4-hydroxybenzoic acid derivatives have a direct effect on cancer molecules, inhibit acetic acid-induced edema, and are used in the treatment of sickle cell disease [45]. Protocatechuic acid has pharmacological effects in humans, both naturally and as a synthesized compound, by showing anti-inflammatory and antioxidant activity. In particular, the intake of polyphenols and flavonoid compounds in natural foods is important for health [46]. Caffeic acid and its derivatives were found to have antioxidant, anti-inflammatory, and anticarcinogenic activities in in vitro and in vivo studies [47].

3.4. Total Phenolic Content and Antioxidant Capacity of Jujube Juice, Wine, and Vinegar

The antioxidant activity (TEAC) and total phenolic substance (TPC) results of samples were shown in Figure 3A,B.

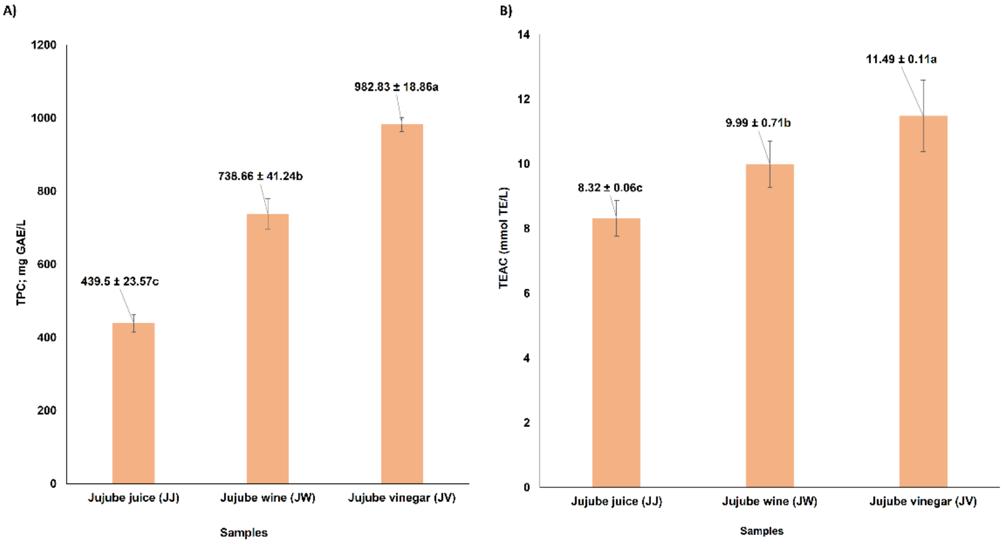

Figure 3. Total phenolic substance (**A**); and antioxidant activity (**B**) of jujube juice (JJ), jujube wine (JW) and jujube vinegar (JV). Different letters in the figure indicate significant differences between samples.

Examining the total phenolic content and antioxidant activity, there was a close relationship in the samples of jujube juice and jujube vinegar. Sorting the TPC and TEAC values from high to low, it was determined to be jujube vinegar, jujube wine, and jujube juice ($p < 0.05$). The TPC value was 982.83 mg GAE/L while the TEAC value was 11.49 mmol TE/L in the JV samples.

The total phenolic content increased with the fermentation process. Because, as a result of the use of sugar by wine yeast in the environment during alcohol fermentation, phenolic compounds that are dependent on sugar in the raw material are released and increase the phenolic compound values [48]. Vithlani and Patel [18] have stated that there was an increase in the flavonoid and flavonol contents during the fermentation stage of jujube wine and vinegar, and this increase increased the antioxidant activities of DPPH and ABTS. In the literature, the total phenolic content of jujube fruits was determined to be in the range of 275.6−541.8 mg GAE/100 g [36], while the total phenolic content of fermented jujube juice was determined to be 2663 μg mL^{-1} [49]. The ABTS scavenging activity of blackened jujube vinegar and red jujube vinegar was determined to be 0.52–0.18 mg Trolox/mL, respectively [50].

The TEAC values of different kinds of vinegar, apple cider vinegar, sour cherry vinegar, peach vinegar, hawthorn vinegar, and pomegranate vinegar were determined to be 11.90 mmol TE/mL, 8.14 μmol TE/mL, 8.05 mmol TE/L, 13.01 mmol TE/L, and 23.56 μmol TE/mL, respectively [22,34,35,51]. The high antioxidant and total phenolic content of the product obtained from the jujube fruit, which has a very short shelf-life, suggested that jujube vinegar is a valuable product.

3.5. Volatile Compound Profiles of Jujube Juice, Wine, and Vinegar

As for the aroma-related volatile compounds of the samples, the main compounds were regarded as those higher than the mean value of the percentage distribution of compounds within the sample.

In the study, a total of 42 volatile compounds including 19 esters, 9 aldehydes, 7 alcohols, 5 terpenes, 1 acid, and 1 ketone were determined. Examining the JJ (32 compounds), JW (22 compounds), and JV (19 compounds) samples together (Table 4 and Figure 4), in the JJ sample, the total ester ratio had the highest value with 66.4%, followed by the alcohols, aldehydes, and terpenes with 3.58% and 22.2%, and 1.65%, respectively. In another study, a total of 46 volatile compounds, including aldehydes, alcohols, alkenes, ketones, esters, and acids were detected in the aroma component analysis of fresh jujube fruit [52].

Table 4. The changes in volatile compounds associated with aroma during fermentation in the production of jujube vinegar.

Compounds		JJ (Jujube Juice)		JW (Jujube Wine)		JV (Jujube Vinegar)	
		Area	Percent (%)	Area	Percent (%)	Area	Percent (%)
Acid	**Acetic acid**	n.d.	n.d.	n.d.	n.d.	45,730,907	60.28 ± 4.42
	Total						**60.28± 4.42**
Ketone	**Diacetyl**	n.d.	n.d.	n.d.	n.d.	359,304	0.47 ± 0.06
	Total						**0.47± 0.06**
Aldehyde	**2-Hexenal-(E)**	104,820	0.22 ± 0.05	n.d.	n.d.	n.d.	n.d.
	Acetaldehyde	152,524	0.31 ± 0.07 ^{a*}	n.d.	n.d.	52,781	0.07 ± 0.01 ^{b}
	Benzaldehyde	n.d.	n.d.	n.d.	n.d.	187,505	0.25 ± 0.09
	Phenylacetaldehyde	806,141	1.66 ± 0.42 ^{b}	6,665,457	10.92 ± 1.68 ^{a}	118,527	0.16 ± 0.08 ^{c}
	Decanal	n.d.	n.d.	20,651	0.03 ± 0.01	n.d.	n.d.
	Heptanal	54,258	0.11 ± 0.04	n.d.	n.d.	n.d.	n.d.
	Hexanal	418,309	0.86 ± 0.09	n.d.	n.d.	n.d.	n.d.
	Nonanal	117,790	0.24 ± 0.05 ^{a}	57,545	0.09 ± 0.02 ^{b}	n.d.	n.d.
	Octanal	86,303	0.18 ± 0.06 ^{a}	76,958	0.13 ± 0.03 ^{a}	n.d.	n.d.
	Total		**3.58± 0.48** ^{b}		**11.17± 0.64** ^{a}		**0.48± 0.24** ^{c}
Alcohol	**Ethanol**	10,242,552	21.14 ± 6.84 ^{b}	25,782,301	42.24 ± 4.62 ^{a}	411,775	0.54 ± 0.14 ^{c}
	2-Methyl-1-butanol	104,230	0.22 ± 0.02 ^{b}	1,812,897	2.97 ± 0.86 ^{a}	310,241	0.41 ± 0.18 ^{b}
	Isoamyl alcohol	216,157	0.45 ± 0.12 ^{b}	3,630,717	5.95 ± 1.28 ^{a}	488,869	0.64 ± 0.34 ^{b}
	1-Hexanol	110,839	0.23 ± 0.01	n.d.	n.d.	n.d.	n.d.
	1-Amino-2-propanol	n.d.	n.d.	791,642	1.30 ± 0.02	n.d.	n.d.
	Phenethyl alcohol	n.d.	n.d.	80,222	0.13 ± 0.01	n.d.	n.d.
	Myristyl alcohol	75,148	0.16 ± 0.03	n.d.	n.d.	n.d.	n.d.
	Total		**22.2± 2.92** ^{b}		**52.59± 3.64** ^{a}		**1.59± 0.02** ^{c}
Ester	**2-Methylbutyl acetate**	n.d.	n.d.	218,994	0.36 ± 0.06 ^{b}	2,330,967	3.07 ± 1.23 ^{a}
	Isoamyl acetate	8,850,960	18.27 ± 3.24 ^{a}	2,014,436	3.30 ± 1.24 ^{c}	7,205,463	9.50 ± 1.86 ^{b}
	3-Hexenyl acetate	128,237	0.26 ± 0.03	n.d.	n.d.	n.d.	n.d.
	Isobutyl acetate	n.d.	n.d.	n.d.	n.d.	765,346	1.01 ± 0.07
	Phenethyl acetate	1,676,095	3.46 ± 0.16 ^{b}	846,233	1.39 ± 0.48 ^{c}	6,352,412	8.37 ± 2.02 ^{a}
	Ethyl acetate	18,808,097	38.82 ± 2.86 ^{a}	6,350,651	10.40 ± 2.11 ^{b}	8,589,076	11.32 ± 4.01 ^{b}
	Hexyl acetate	631,438	1.30 ± 0.15 ^{a}	178,995	0.29 ± 0.04 ^{b}	n.d.	n.d.
	Methyl acetate	n.d.	n.d.	n.d.	n.d.	93,706	0.12 ± 0.01
	Propyl acetate	71,829	0.15 ± 0.01	n.d.	n.d.	n.d.	n.d.
	Ethyl phenylacetate	n.d.	n.d.	n.d.	n.d.	77,701	0.11 ± 0.02
	Ethyl benzoate	63,951	0.13 ± 0.03 ^{a}	114,907	0.19 ± 0.02 ^{a}	n.d.	n.d.
	Benzyl acetate	57,490	0.12 ± 0.02 ^{a}	n.d.	n.d.	78,551	0.10 ± 0.03 ^{a}
	Ethyl butyrate	n.d.	n.d.	58,451	0.1 ± 0.01	n.d.	n.d.
	Ethyl capronate	333,686	0.69 ± 0.24 ^{b}	3,134,463	5.14 ± 2.01 ^{a}	n.d.	n.d.
	Ethyl decanoate	597,681	1.23 ± 0.14 ^{b}	3,013,764	4.94 ± 1.94 ^{a}	n.d.	n.d.
	Ethyl laurate	166,749	0.34 ± 0.04 ^{a}	270,479	0.44 ± 0.21 ^{a}	n.d.	n.d.
	Ethyl caprylate	711,830	1.47 ± 0.04 ^{b}	3,972,062	6.51 ± 0.42 ^{a}	77,700	0.10 ± 0.01 ^{c}
	Ethyl palmitate	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
	Ethyl propanoate	78,836	0.16 ± 0.28	n.d.	n.d.	n.d.	n.d.
	Total		**66.4± 1.82** ^{a}		**33.42± 1.02** ^{b}		**33.70± 1.17** ^{b}

Table 4. *Cont.*

Compounds		JJ (Jujube Juice)		JW (Jujube Wine)		JV (Jujube Vinegar)	
		Area	Percent (%)	Area	Percent (%)	Area	Percent (%)
Terpene	**α-pinene**	476,664	0.98 ± 0.26	n.d.	n.d.	n.d.	n.d.
	α-Thujene	59,049	0.12 ± 0.01	n.d.	n.d.	n.d.	n.d.
	β-Phellandrene	177,922	0.28 ± 0.09	n.d.	n.d.	n.d.	n.d.
	γ-Terpinene	53,957	0.11 ± 0.08 ^{a}	n.d.	n.d.	47,871	0.06 ± 0.01 ^{b}
	p-Cymol	78,263	0.16 ± 0.07 ^{a}	n.d.	n.d.	53,470	0.07 ± 0.02 ^{b}
	Total		**1.65± 0.36 ^{a}**		**n.d.**		**0.13± 0.01 ^{b}**

* Statistical analysis was made according to percentages. The lowercase letters a–c denote the statistical difference between the JJ, JW, and JV samples. Bold background compounds are each sample, and therefore more effective. Abbreviations: n.d., not detected.

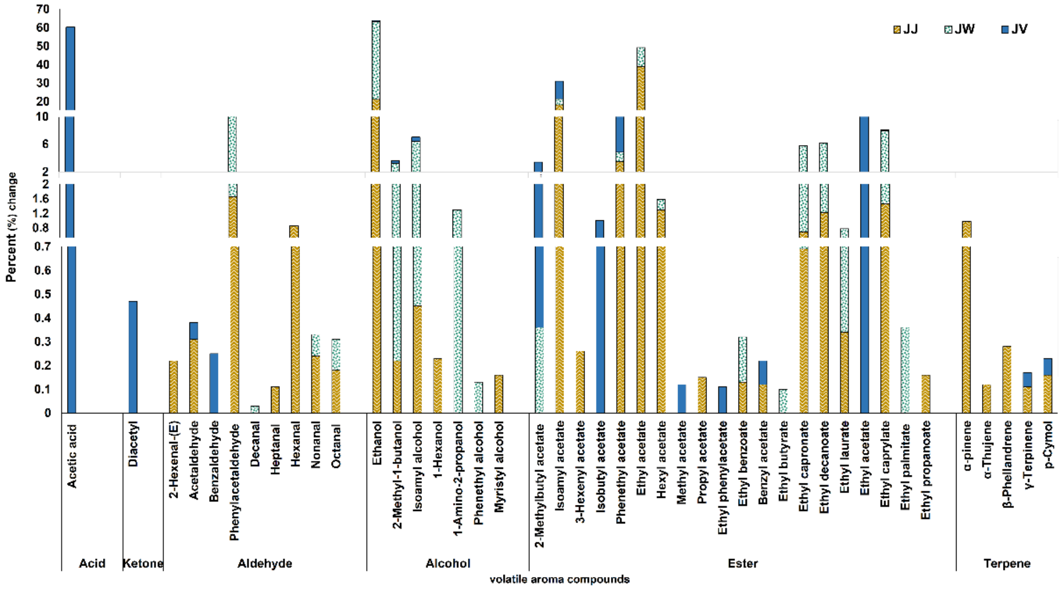

Figure 4. Volatile compounds associated with aroma of jujube juice (JJ), jujube wine (JW) and jujube vinegar (JV) (percent %).

In the present study, the total ester compound content of jujube fruit was found to be the highest. On the other hand, in the JJ sample, ethyl acetate with 38.82%, ethanol with 21.14%, isoamyl acetate with 18.27%, and phenethyl acetate with 3.46%, had high percentages. The difference between the values of these compounds was statistically significant both among each other and according to all the determined compounds ($p < 0.05$). The ethyl acetate is associated with aromatic, brandy, and grape flavors, while the ethanol is associated with alcohol flavor, the isoamyl acetate is associated with apple, banana, glue, pear flavors, and phenethyl acetate is associated with flower, honey, and rose flavors [53]. These ingredients together were considered to give the JJ sample a slightly sour taste. A good vinegar, and even a good wine, can be produced from raw material with a high content of esters, and ethyl acetate. Therefore, this fruit was considered to be a suitable raw material for vinegar fermentation in terms of the aroma related volatile components in the final product.

Other highly-effective compounds were phenylacetaldehyde with 1.66%, ethyl caprylate with 1.47%, and hexyl acetate with 1.30%. The statistical differences between these compounds were not significant whereas significant differences were found when compared to other groups. Also, among the terpene group compounds, α-pinene had a higher ratio than other terpene compounds. Regarding the JJ sample, phenylacetaldehyde is associated with berry, geranium, honey, nut, and pungent flavors, ethyl caprylate is associated with apricot, fat, floral, and pineapple, and hexyl acetate is associated with apple, banana, grass, herb, and pear flavors [53].

As for the JW sample, the total alcohol ratio was the highest with 52.59%, followed by the ester group with 33.42%, and the aldehyde group with 11.17%. No terpene compounds were found in the samples. The total alcohol content of the wine obtained from jujube fruit was found to be the highest, followed by the ester group. On the other hand, in the JW sample, the ethanol, with 42.24%, had the highest percentage ($p < 0.05$). This was followed by phenylacetaldehyde with 10.92% and ethyl acetate with 10.40% ($p < 0.05$). In particular, the ethyl acetate compounds with aromatic, brandy, and grape flavor is the desired compound in a wine [54]. However, it was thought that the close presence of the phenylacetaldehyde with the ethyl acetate might suppress the effect of the ethyl acetate. Because the phenylacetaldehyde gives wine a honey-like effect [55]. This gave the JW sample a softer flavor profile.

High levels of alcohol group content are expected in wine. However, in the present study, the ester ratio, in particular, was an interesting result. This ester content was associated with the rich ester content in the juice of the fruit used. The other highly-effective compounds following these were ethyl caprylate with 6.51%, isoamyl alcohol with 5.95%, ethyl caproate with 5.14%, and ethyl decanoate with 4.94%. The statistical differences between these compounds were not significant, whereas significant differences were found when compared to other groups ($p < 0.05$). Ethyl caprylate is associated with apricot, fatty, floral, and pineapple flavors, ethyl capronate is associated with apple peel, brandy, fruit gum, overripe fruit, pineapple flavors, and isoamyl alcohol is associated with burnt, cocoa, malt flavors, and are not desirable compounds for a wine [53,56]. In one study, a total of 182 volatile compounds, comprising 30 alcohols, 16 acids, 62 esters, 19 ketones, 18 aldehydes, 10 hydrocarbons, 7 phenols, and 20 other compounds were detected for jujube wine [57].

In the JV sample, the total acid ratio was the highest at 60.28%, and the acetic acid compound is associated with acidic, fruity, pungent, sour vinegar flavors [53] ($p < 0.05$). This showed that the vinegar fermentation was properly realized. The acid group was followed by the ester group with 33.70%, and the alcohol group with 1.59% ($p < 0.05$). As for the other compound groups, the aldehyde group and ketone groups were determined to be 0.48% and 0.47%. Also, the terpene group was determined to be 0.13%.

For the JV vinegar, the main compound was acetic acid. The acetic acid content in the vinegar samples suggested that jujube can be efficiently used in vinegar production. The other prominent compounds were ethyl acetate, associated with aromatic, brandy, and grape flavors with 11.32%, isoamyl acetate, associated with apple, banana, glue, and pear flavors with 9.50%, and phenethyl acetate, associated with flower, honey, and rose flavors, with 8.37%. The statistical differences between these compounds were not significant, whereas significant differences were found when compared to other groups ($p < 0.05$). Also, these compounds positively affected the JV sample [33,54]. The ratio of 2-methyl butyl acetate compound, associated with apple, banana, and pear flavors, was determined to be 3.07%. As was the case in the present study, high levels of acid group content are expected in vinegar and ester compounds have positive effects on the flavor formation in vinegar [22]. It has been reported that the volatile aroma compounds of jujube vinegar were determined to be acids, esters, alcohols, aldehydes, and ketones. The main components of the vinegar content were determined to be acids and esters [50].

According to the volatile compound results, the compounds with a high ratio in the aroma profile of the JJ sample were ethyl acetate, ethanol, isoamyl acetate, and phenethyl acetate. The compounds with a high ratio in the aroma profile of the JW sample were ethanol, phenylacetaldehyde, and ethyl acetate. The other highly-effective compounds in the JW sample were undesirable the compounds; ethyl caprylate, isoamyl alcohol, ethyl capronate, and ethyl decanoate. The compounds with a high ratio in the aroma profile of the JV sample were acetic acid, ethyl acetate, isoamyl acetate, and phenethyl acetate. Therefore, these results showed that jujube fruit, in terms of the aroma-related volatile component, is suitable for vinegar production, but not for wine.

4. Conclusions

In this study, aroma profile-related volatile compound analysis, antioxidant capacity, organic acid, and phenolic compounds were determined in the juice, wine, and vinegar samples, which were produced from jujube fruits. It was determined that the total phenolic compound and the TEAC antioxidant activity content of jujube vinegar were higher than those of the jujube juice and jujube wine. Also, it has been determined that the samples had important parameters that had an effect on health in terms of organic acid and phenolic compound contents. The volatile aroma compound profiles of the samples comprised the ethyl acetate, ethanol, isoamyl acetate, and phenethyl acetate for the jujube juice; the ethanol, phenylacetaldehyde, and ethyl acetate, and also the undesirable ethyl caprylate, isoamyl alcohol, and ethyl capronate for the jujube wine. As for the volatile aroma compound profiles of jujube vinegar, the most abundant compounds were acetic acid, ethyl acetate, isoamyl acetate, and phenethyl acetate. I were concluded that the use of jujube fruit, which is a healthy, pleasant-flavored, and promising alternative in the production of fruit vinegar, is important in terms of contributing to the country's economy. This study will encourage investors who want to invest in vinegar production to increase the use of jujube vinegar with increased functional properties due to its bioactive compound properties by evaluating the jujube fruit.

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