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Sonication processing of mallow vinegar: effects on the bioactive compounds, amino acids, organic acid, sugar, mineral and microstructure

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Abstract

This research was aimed with the objective of investigation the effects of sonication treatment on quality characteristics of mallow vinegar such as organic acids, sugar, amino acids, minerals, bioactive compounds, and microstructure. For the enhancement of bioactive components in mallow vinegar (*Malva saylvestris L.*), the response surface methodology (RSM) was employed using the central composite design to determine the combined effects of sonication treatment on the maximization of contents in vinegar. The maximum optimization results for the bioactive components were obtained at 8 minutes and 50.9 amplitude. As a result of sonication treatment, increases were detected in bioactive components compared to the control mallow vinegar sample, while decreases were detected in the mallow vinegar samples treated with thermal pasteurization. A statistically significant increase was observed in phenolics (protocatechuic acid, catechin), amino acids (threonine, proline, lysine, glutamic acid, alanine, arginine, aspartic acid), minerals (Na, Zn) were found in mallow vinegar sonicated compared to control. It is the first study concerning the impact of sonication and thermal pasteurization on the minerals, sugars, organic acids, and amino acids of mallow vinegar, so further experimental work is required to understand the precise phenomena.

Keywords: non thermal technology; sonication; mallow vinegar; response surface methodology; bioactive compounds.

Practical Application: This study shows that sonication technology can increase the phenolic compound, mineral, amino acid content, and antioxidant activity of mallow vinegar.

1 Introduction

Malva sylvestris L. is a perennial biennial herbaceous plant with a height of 40-120 cm (Yücel et al., 2012). *M. sylvestris L.* has high pharmacological properties because of the presence of flavonoids, amino acids, sterols, coumarins, terpenoids, enzymes, mucilage, and phenol derivatives. Mallow has anti-inflammatory, antifungal, and antibacterial activities (Mravčáková et al., 2020).

Novel food processing technologies focus on retaining sensory attributes and bioactive compounds while offering microbiologically safe products. For this purpose, interest in nonthermal technologies, especially sonication, has emerged for the protection of food products (Glover et al., 2022; Lino et al., 2022; Pokhrel et al., 2017). The word "sonication" refers to sound. It was reported that sonication technology improves the quality of milk and dairy products (Guimarães et al., 2021; Scudino et al., 2020). Portela et al. (2022), determined that product which was treated ultrasound technology, evoked docile, adventurous, joyful, and curious emotions on consumers. Response surface methodology (RSM) is successfully applied for indicating the significance level of ultrasound treatment factors through statistical analysis in food products (Aboulghazi et al., 2022; Silva et al., 2022; Zhang et al., 2021).

In spite of the health potential of mallow vinegar, there is no scientific literature purposed at investigation the effect of sonication on microstructure, bioactive compounds, organic acids, sugar, amino acids, and minerals in mallow vinegar. Therefore, the main target of this study was to assess the impact of sonication treatment on the quality characteristics of mallow vinegar including antioxidant activity capacity, organic acid and sugar composition, phenolic, mineral, amino acid content, and microstructural characteristics.

2 Materials and methods

2.1 Preparation of vinegar

Fresh mallow flowers were supplied from Tekirdag, Turkey. The flowers were sorted and cleaned. They were then washed with water. In this process, deionized water (1:1, w:w) and 15% pine honey were added. *Saccharomyces cerevisiae* (3%) was inoculated into mallow juice. Periodic acidity measurements were performed, and samples were stored at −20 ± 1 °C at the end of fermentation.

2.2 Thermal pasteurization treatment

Mallow vinegar samples were pasteurized at 85 ± 1 °C in a water bath (Wisd-Model, WUC-D06H, Daihan, Korea) for 2 minutes and name as pasteurized mallow vinegar (P-MV).

2.3 Sonication treatments

Mallow vinegar was processed using a 200 W ultrasonic processor at a frequency of 26 kHz (Hielscher Ultrasonics Model UP200St, Berlin, Germany). Sonication treatment was

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with different amplitudes (40%, 50%, 60%, 70%, and 80%) and times (2, 4, 6, 8, and 10 min.).

2.4 Experimental design

The Response Surface Method (RSM) was used to understand the effect of sonication treatment on the bioactive components in mallow vinegar. Then results were analyzed by using Minitab Statistical Analysis Software (Minitab 18.1.1 version, USA). Five-level, two-factor experiment design was created. Independent variables were determined as duration within the range of X_1 (time) and X_2 (amplitude). The following quadratic polynomial formula was used to create the equation models (Equation 1):

$$y = b_o + b_1 x_1 + b_2 x_2 + b_{12} x_1 x_2 + b_{11} x_1^2 + b_{22} x_2^2 \quad (1)$$

The coefficients of the polynomial were represented by b_0 (constant term), b_1 and b_2 (linear effects), b_{11} and b_{22} (quadratic effects), and b_{12} (interaction effects).

2.5 Total Phenolic Contents (TPC) and Total Flavonoids Contents (TFC)

The total phenolic contents (TPC) was determined by the Folin-Ciocalteu method using SP-UV/VIS-300SRB spectrophotometer (Spectrum Instruments, Australia) (Singleton & Rossi, 1965). Firstly, 50 µL of the mallow vinegar samples were mixed with distilled water (450 µL) and 0.2 N Folin–Ciocalteu reagent (2.5 mL). After 5 min, saturated sodium carbonate (2 mL) were added. The absorbance was measured at 765 nm and the total phenolic content was expressed as milligrams of gallic acid equivalents (mg GAE/L).

The total flavonoids content (TFC) was determined through colorimetric technique as previously described Zhishen et al. (1999). Briefly, distilled water and 0.3 mL $NaNO_2$ were added. After 5 min, solution was mixed with $AlCl_3$ (3 ml). NaOH (2 ml) was added and made up to 10 mL with distilled water. The absorbance was measured against a blank at 510.

2.6 Determination of total antioxidant capacity by CUPRAC

The CUPRAC method (Cu (II) ion reducing antioxidant capacity) was used to determine the antioxidant capacity as described by Apak et al. (2006). Firstly, 10^{-2} M $CuCl_2$ solution (1 mL), 7.5×10^{-3} M neocuproine alcoholic solution (1 mL), and NH_4Ac buffer solution were added. Solution was mixed with water. The absorbance measurements were carried out a UV-VIS spectrophotometer (SP-UV/VIS-300SRB, Spectrum Instruments, Australia) at 450 nm.

The formula of CUPRAC values is written as follows (Equation 2):

$$CUPRAC\ (\%\ inhibition) = (A_0 - A_1)/A_0) * 100 \quad (2)$$

A_0 = absorbance of control, A_1 = absorbance of sample

2.7 Determination of antioxidant activity by DPPH

Antioxidant activity was assessed using DPPH scavenging activity method, previously described by Grajeda-Iglesias et al. (2016) with some modifications. The prepared sample (100 µL) was added into 2 mL of a methanol solution of 0.1 mM DPPH (Sigma-Aldrich, USA) and incubated in dark for 30 min. Then the mixed solution was measured at 517 nm absorbance. The calculation of DPPH was done using Equation 3:

$$DPPH\ (\%\ inhibition) = (A_0 - A_1)/A_0) * 100 \quad (3)$$

A_0 = absorbance value of control, A_1 = absorbance value of examined sample

2.8 Organic acid and sugar composition

For the measurement of organic acid and sugar content, high-performance liquid chromatography (HPLC system, model 1260 Infinity LC, Agilent Technologies, Santa Clara, CA, USA) as described by Coelho et al. (2018) was used with some modifications. Mallow vinegar samples were filtered through a 0.45 µm syringe filter and a volume of 20 µL was injected into the device. Agilent Hi-Plex H (300 x 7.7 mm) was used as the ion exchange column. The temperature of the column was 65 °C while the RID flow cell was maintained at 35 °C. The flow rate applied was 0.6 mL min−1 with a run time of 20 min. The phase was 10.0 mML−1 H_2SO_4 in ultrapure water. Standard solutions were injected to obtain the retention time for each compound. For the determination of lactic, propionic acetic, and piruvic acid were conducted in the DAD at 210 nm. Fructose, glucose, sucrose, turanose, arabinose, and ksilose sugar detection was carried out using a refractive index detector (RID). Results are given as g/L.

2.9 Phenolic compounds

For detection of phenolic compounds was completed as described by (Portu et al., 2017) using a C-18 Age Generix column (250 × 4.6 mm; 5 µm packing; Agilent)

2.10 Amino acid content

The amino acid content was determined by a method as described by Bilgin et al. (2019) with some modifications. LC was performed using an Agilent 6460 (Agilent Technologies, Waldbronn, Germany) LC system. MS/MS analyses were conducted on an Agilent 6460 triple quadruple LC-MS equipped with an electrospray ionization interface. The mallow vinegar samples were injected into the LC-MSMS system after filtering without acidic hydrolysis and dilution. The results are given in mg/100 mL.

2.11 Mineral content

In mallow vinegar, Ca, Fe, Mg, Mn, Na, Cr, Pb and Zn quantity analyses were performed with a simultaneous inductively coupled plasma atomic emission spectrometer (ICPOES) instrument (Thermo Scientific iCap 6000 Dual view, Cambridge, UK). The amounts of K and Cd were analyzed with an atomic absorption spectrometer (AAS) (Thermo Scientific iCE 3000). Series, Cambridge, England). The defrosting process was used a microwave burning system (Berghof Instruments, Speedwave, Germany). Analysis was performed in the ICP-OES and AAS devices with settings: Ag; 328.0 nm, Ca; 317.9 nm,

Cu; 324.7 nm, Fe; 259.9 nm, Mg; 279.5 nm, Mn; 257.6, Na; 588.9 nm, Zn; 213.8 nm, Cd; 228.8 nm, and K; 766.5 nm wave. To plot the calibration curves at the specified wavelengths, the multielement standard solution (Merck, Item No: 111355, Germany) was studied in the ICP-OES K standard solution (Chem-Lab, Belgium) and Cd standard solution (Chem-Lab, Belgium) were used for K and Cd studied in the AAS (Sezer et al., 2019).

2.12 Microstructure

The microstructure of mallow vinegar was observed with a reflected fluorescence system in an Olympus CX41 light microscope (Olympus, Tokyo, Japan). Mallow vinegars (Control, pasteurized, and sonication microwave treatment) were dropped (~40 µL) on a microscope slides and the samples were allowed to dry at room temperature. After they were crosswise covered with a coverslip, pictures were obtained under 200X magnification with a digital camera (Kameram 2.1, Argenit, Istanbul, Turkey).

2.13 Statistical analysis

Each test was repeated three times. Statistical analyses were conducted using Statistical Package for the SPSS 22.0 software (SPSS Inc., Chicago, IL). Sigmaplot 12.0 Statistical Analysis Software (Systat Software, Inc., San Jose, USA) was used for three-dimensional RSM plots. One-way analysis of variance (ANOVA) was used to analyze the data, and Tukey's test was used to carry out multiple comparisons between means. The significance level was defined as $p < 0.05$.

3 Results and discussion

3.1 Optimization of bioactive compounds

Sonication is an alternative nonthermal technology used for the enrichment of bioactive compounds of foods and food safety (Yıkmış, 2020). Experimental and predictive results for the TPC, TFC, CUPRAC, and DPPH values of mallow vinegar samples at different levels of time and amplitude are given in Table 1. The results of the RSM optimization for TPC, TFC, CUPRAC, and DPPH responses are given in Equations 4-7.

$$\mathbf{TPC\,(mg\ GAE\,/\,100\ mL)} = 27,01 + 5,478X_1 + 0,7183X_2 + 0,01047X_1^2 - 0,001953X_2^2 - 0,09239X_1X_2 \quad (4)$$

$$\mathbf{TFC\,(mg\ CE\,/\,L)} = -9,48 + 1,156X_1 + 0,7183X_2 - 0,4862\,X_1^2 - 0,004049X_2^2 - 0,00325\,X_1X_2 \quad (5)$$

$$\mathbf{CUPRAC\,(\%\ Inhibition)} = 7,50 + 4,265X_1 + 1,1684X_2 - 0,18865X_1^2 - 0,008389X_2^2 - 0,03023X_1X_2 \quad (6)$$

$$\mathbf{DPPH\,(\%\ Inhibition)} = 12,61 + 3,923X_1 + 0,8270X_2 - 0,09976X_1^2 - 0,004929X_2^2 - 0,04329\,X_1X_2 \quad (7)$$

Table 2 shows the analysis of variance for TPC, TFC, CUPRAC, and DPPH. Linear effects of X_1 ($p < 0.05$) and X_2 ($p < 0.001$) applied to mallow vinegar samples on TPC and TFC values were found to be statistically significant. Cross interactions of factor X_2 with mallow vinegar were significant for TPC, TFC, CUPRAC, and DPPH ($p < 0.001$). Two-way interactions were found to be statistically significant ($p < 0.001$). R^2 of the models used in the study for TPC, TFC, CUPRAC, and DPPH were found to fit at 99.71, 98.97, 99.47, and 99.79 levels, respectively. The interactions of the variables were graphically showed in Figure 1. (A-D). When TPC, TFC, DPPH, and CUPRAC models were examined, X_1 and X_2 factors caused a linear increase in bioactive components. At the end of RSM, TPC, TFC, DPPH, and CUPRAC values were determined as 65.36 mg GAE/100 mL, 7.78 mg CE/100 mL, 49.30%, and 54.99% for 8 minutes and

Table 1. Measured responses used in the experimental design for RSM and the results of C-MV and P-MV.

Run no.	Independent variables		Dependent variables							
			TPC (mg GAE/100 mL)		TFC (mg CE/100 mL)		CUPRAC (% inhibition)		DPPH (% inhibition)	
	Time (X_1)	Amplitude (X_2)	Experimental data	RSM predicted	Experimental data	RSM predicted	Experimental data	RSM predicted	Experimental data	RSM predicted
1	8 (+1)	50(-1)	65.65	65.58	7.65	7.73	54.83	54.90	49.39	49.32
2	8 (+1)	70 (+1)	60.35	60.48	7.15	7.21	53.34	53.30	47.11	47.10
3	6 (0)	60 (0)	63.07	63.06	8.15	8.11	55.35	55.32	48.64	48.85
4	2 (-1.41)	60 (0)	63.00	62.99	6.80	6.73	51.63	51.55	46.70	46.74
5	6 (0)	60 (0)	63.07	63.06	8.15	8.11	55.35	55.32	48.87	48.85
6	6 (0)	60 (0)	63.07	63.06	8.15	8.11	55.35	55.32	48.87	48.85
7	6 (0)	60 (0)	63.07	63.06	8.15	8.11	55.35	55.32	48.87	48.85
8	10 (+1.41)	60 (0)	63.48	63.47	7.05	7.02	53.05	53.05	47.75	47.77
9	4 (-1)	70 (+1)	63.81	63.93	7.09	7.20	53.71	53.76	48.34	48.32
10	6 (0)	80 (+1.41)	60.98	60.87	6.15	6.11	51.55	51.57	46.40	46.39
11	4 (-1)	50 (-1)	61.72	61.64	7.33	7.45	52.78	52.94	47.16	47.08
12	6 (0)	60 (0)	63.07	63.06	8.15	8.11	55.35	55.32	48.87	48.85
13	6 (0)	40 (-1.41)	63.60	63.69	6.93	6.87	52.45	52.36	47.30	47.36
	C-MV		60.75		6.85		50.48		46.85	
	P-MV		58.45		6.10		49.2		44.2	

TPC: Total phenolic content; TFC: Total flavonoid content; CUPRAC: Cupric reducing antioxidant capacity; DDPH: Radical scavenging activity; GAE: Gallic acid equivalent; CE: Catechin equivalent; C-MV: Mallow vinegar; P-MV: Thermal pasteurized mallow vinegar.

Table 2. Corresponding p-values of linear, interaction and quadratic terms of regression coefficients obtained by RSM of responses for TPC, TFC, CUPRAC, and DPPH experiments.

Source	DF	F-Value	P-Value	F-Value	P-Value	F-Value	P-Value	F-Value	P-Value
		TPC (mg GAE/100 mL)		TFC (mg CE/100 mL)		CUPRAC(% inhibition)		DPPH (% inhibition)	
Model	**5**	473.84	0.0000	133.90	0.0000	665.67	0.0000	262.82	0.0000
Linear	**2**	346.94	0.0000	32.54	0.0000	136.10	0.0000	84.02	0.0000
X_1	**1**	19.82	0.0030	8.44	0.0230	214.15	0.0000	88.78	0.0000
X_2	**1**	674.06	0.0000	56.65	0.0000	58.04	0.0000	79.26	0.0000
Square	**2**	62.97	0.0000	301.11	0.0000	1435.89	0.0000	405.24	0.0000
X_1^2	**1**	4.56	0.0700	279.59	0.0000	1645.57	0.0000	408.44	0.0000
X_2^2	**1**	99.15	0.0000	482.70	0.0000	2033.53	0.0000	623.21	0.0000
2-Way Interaction	**1**	1549.36	0.0000	2.17	0.1840	184.35	0.0000	335.59	0.0000
X_1*X_2	**1**	1549.36	0.0000	2.17	0.1840	184.35	0.0000	335.59	0.0000
Error	**7**								
Lack-of-Fit	**3**	*	*	*	*	*	*	0.6400	0.6300
Pure Error	**4**								
Total	**12**								
R^2		99.71%		98.97%		99.79%		99.47%	
Adj R^2		99.49%		98.23%		99.64%		99.09%	
Pred. R^2		97.05%		92.76%		98.17%		98.11%	

X1: Time; X2: Amplitude; DF: Degree of freedom; TPC: Total phenolic content; TFC: Total flavonoid content; CUPRAC: Cupric reducing antioxidant capacity; DDPH: Radical scavenging activity; GAE: Gallic acid equivalent; CE: Catechin equivalent.

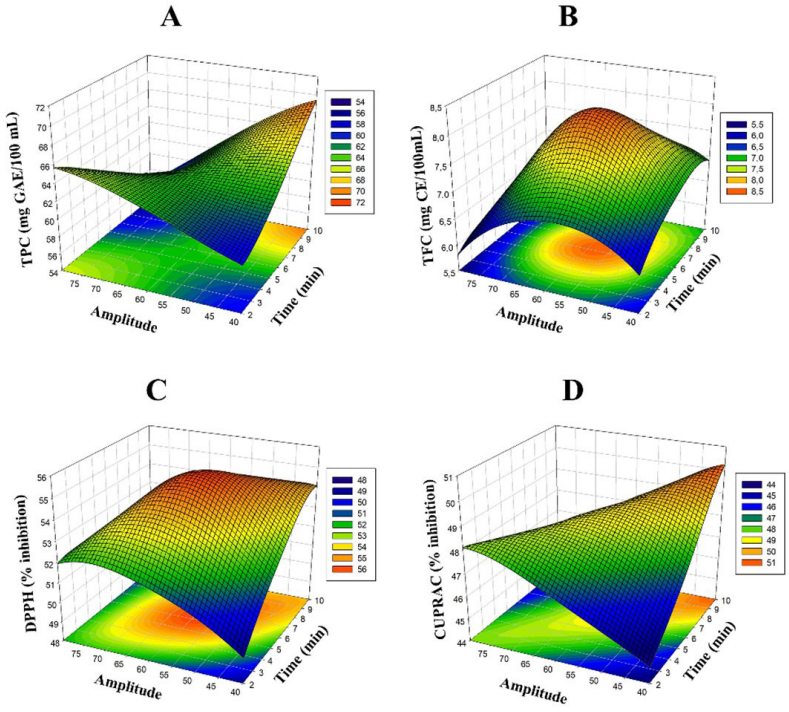

Figure 1. Response surface plots of TPC (A), TFC (B), DPPH (C), and CUPRAC (D) analysis.

50.9 amplitude, respectively. After sonication treatment, increases were detected in bioactive components compared to the C-MV sample, while decreases were detected in the C-MV sample after thermal pasteurization. While phenols alone have not any antioxidant activity, sonicated phenol solutions showed a significant antioxidant property (Ashokkumar et al., 2008). The amount of bioactive components increased after sonication treatment applied to samples of apple vinegar, gilaburu vinegar, verjuice vinegar, tomato vinegar, chokanan, mango juice, uruset, mulberry juice fermented with lactic acid, and grape marc

(Bermúdez-Aguirre et al., 2011; Erdal et al., 2022; Kwaw et al., 2018; Santhirasegaram et al., 2013; Yıkmış et al., 2019, 2021b). The increase in the amount of TPC and TFC with the sonication process can be attributed to the breaking of cell walls with the effect of cavitation pressure, and thus the release of forms bound to the bioactive ingredients (Aadil et al., 2013). The reason for the increase in total antioxidants is that the general increase in the amount of polyphenols caused by cavitation during sonication treatments applied to verjuice vinegar, purple basil sirkencubin syrup and tomato vinegar may be due to increases in the amount of antioxidants (Doguer et al., 2021; Yıkmış et al., 2020, 2021a).

3.2 Organic acid and sugar composition

Organic acids are considered the most important compounds that comprehensively affect the general appeal, flavor, and taste of vinegar (Wang et al., 2017). The results regarding the effects of treatment using sonication and thermal pasteurization on organic acid contents are shown in Table 3. There was an insignificant ($p > 0.05$) decrease in lactic acid, acetic acid, and propionic acid content in sonication treated mallow vinegar. There was a significant ($p < 0.05$) increase in lactic acid and acetic acid content and was significant ($p<0.05$) decrease in propionic acid content in pasteurized mallow vinegar. Gomes et al. (2017) similar to our study; they reported that sonication followed by high pressure processing has not shown significant differences in the concentration of organic acids in prebiotic cranberry juice. Bruna-Maynou et al. (2020) found that the acetic acid values of control and sonication treatment, 1.594 ± 0.540 and 1.433 ± 0.349 respectively in flavored sherry vinegar. Contrary to our results, Wang et al. (2017) found that higher content of lactic acid in the ultrasonic treatment vinegars. However, Siddeeg et al. (2019) also found an increase in the amount of acetic acid in sonication treatment palm vinegar. There was a significant ($p < 0.05$) increase in lactic acid and acetic acid content and was significant ($p < 0.05$) decrease in propionic acid content in pasteurized mallow vinegar.

While the contents of arabinose, turanose, ksilose, and glicose in the ST-MV were found to be lower compared to C-MV ($p > 0.05$), the content of sucrose was found to be higher compared to C-MV ($p > 0.05$). Jabbar et al. (2014) determined significant increase in glucose and fructose sonicated carrot juice samples compared to controls, on the contrary in our study. Numerous studies have been reported that a significant increased in sugar contents in sonication treated grape mash juice, grapefruit, melon, and apple juice samples as compared to control (Aadil et al., 2015; Abid et al., 2014; Fonteles et al., 2012; Lieu & Le, 2010). Acetic

Table 3. Organic acids, sugar, and phenolic component analysis results of C-MV, P-MV and ST-MV.

Analyzes		Samples		
		C-MV	P-MV	ST-MV
Organic acid content (g/L)	**Pyruvic acid**	0.06 ± 0.01^{a}	0.05 ± 0.07^{a}	0.06 ± 0.01^{a}
	Lactic acid	2.74 ± 0.25^{b}	6.11 ± 0.25^{a}	2.57 ± 0.06^{b}
	Acetic acid	103.4 ± 0.6^{b}	113.65 ± 2.54^{a}	98.58 ± 0.47^{b}
	Propionic acid	6.75 ± 0.18^{a}	4.71 ± 6.65^{a}	6.3 ± 0.01^{a}
Sugar content (g/L)	**Glicose**	0.18 ± 0.01^{a}	0.18 ± 0.03^{a}	0.14 ± 0.01^{a}
	Fructose	0.18 ± 0.01^{a}	0.27 ± 0.03^{a}	0.18 ± 0.02^{a}
	Turanose	0.48 ± 0.03^{a}	0.47 ± 0.04^{a}	0.36 ± 0.04^{a}
	Sucrose	0.35 ± 0.03^{a}	0.3 ± 0.06^{a}	0.49 ± 0.14^{a}
	Ksilose	0.35 ± 0.02^{b}	0.44 ± 0.03^{a}	0.3 ± 0.00^{b}
	Arabinose	3.91 ± 0.09^{b}	5.39 ± 0.02^{a}	3.73 ± 0.02^{b}
Phenolic compounds (mg/L)	**Gallic Acid**	94.88 ± 4.79^{a}	91.71 ± 7.50^{a}	115.57 ± 10.51^{a}
	Vanillic acid	16.23 ± 19.31^{a}	14.26 ± 16.77^{a}	22.35 ± 26.54^{a}
	Protocatechuic acid	17.34 ± 0.32^{a}	16.31 ± 0.29^{a}	23.77 ± 0.75^{b}
	Catechin	20.34 ± 1.02^{a}	18.74 ± 1.22^{a}	27.06 ± 1.46^{b}
	Hydroxybenzoic acid	2.16 ± 0.32^{ab}	1.76 ± 0.04^{a}	3.19 ± 0.29^{b}
	Gentisic acid	1.96 ± 2.76^{a}	1.80 ± 2.54^{a}	13.72 ± 13.8^{a}
	p-coumaric acid	1.55 ± 0.13^{a}	1.36 ± 0.12^{a}	2.27 ± 0.10^{b}
	Rutin	0.69 ± 0.16^{a}	0.35 ± 0.49^{a}	1.00 ± 0.17^{a}
	Ascorbic acid	3.17 ± 0.01^{a}	2.44 ± 0.13^{a}	1.84 ± 2.60^{a}
	Ferulic acid	4.06 ± 0.30^{a}	3.74 ± 0.27^{a}	9.81 ± 5.59^{a}
	Naringin	0.62 ± 0.25^{a}	0.43 ± 0.01^{a}	0.93 ± 0.56^{a}
	o-coumaric acid	1.34 ± 0.44^{a}	0.76 ± 0.22^{a}	2.48 ± 0.62^{a}
	Neohesperidin	0.40 ± 0.01^{a}	1.93 ± 2.14^{a}	2.49 ± 2.79^{a}
	Coumarin	0.12 ± 0.04^{a}	0.13 ± 0.00^{a}	0.15 ± 0.00^{a}
	Quercetin	0.09 ± 0.04^{a}	0.10 ± 0.14^{a}	0.21 ± 0.04^{a}
	trans-cinnamic acid	0.02 ± 0.02^{a}	0.01 ± 0.01^{a}	0.05 ± 0.07^{a}

Values with different letters within the line are significantly different ($p < 0.05$). Results are presented as mean ± standard deviation ($n = 3$). C-MV: Mallow vinegar; P-MV: Pasteurized mallow vinegar; ST-MV: Sonication-treated mallow vinegar.

acid, as a major organic acid in mallow vinegar, is suppressed by carbohydrates hydrolyzing enzymes. Therefore acetic acid was suggested as a key factor in decreasing disaccharidase activity, sucrase, lactase, and maltase activities (Ousaaid et al., 2022).

3.3 Phenolic compounds

The aim was to investigate the change in phenolic compounds after the processing of mallow vinegar with sonication and thermal pasteurization. In the present study, sonication treatment was having more effective results were observed compared to thermal pasteurization. As shown in Table 3, sonication treatment enhanced the protocatechuic acid, catechin, and p-coumaric acid content in mallow vinegar ($p < 0.05$).

In this research, a decrease in the ascorbic acid content was observed in mallow vinegar by sonication and heat treatment when compared to control ($p > 0.05$). Santhirasegaram et al. (2013) and Wang et al. (2019a) parallel effects were seen; they reported that after sonication treatment there was a decrease ascorbic acid in chokanan mango juice and kiwi juice respectively. Zenker et al. (2003) found a higher decrease of ascorbic acid in thermal-treated orange juice compared sonication-treated juice. There are also studies that reported an increased amount of ascorbic acid after the sonication procedure in apple and grapefruit juices (Aadil et al., 2013; Abid et al., 2013). The gallic acid, hydroxybenzoic acid, vanillic acid, gentisic acid, rutin, ferulic acid, naringin, o-coumaric acid, neohesperidin, coumarin, quercetin, trans-cinnamic acid were all higher than the fresh vinegar. Similar increases in the amount of gallic acid were also reported with sonication treatments of strawberry juice (Wang et al., 2019b). Vanillic acid, present in vinegar, can inhibit α-glucosidase and α-amylase activities through a specific binding between the methoxy group of the active sites and aromatic ring of these enzymes (Ousaaid et al., 2022). Except for the neohesperidin, coumarin, and quercetin compounds, a decrease was detected after treatment with thermal pasteurization. Some researchers reported that similar increases in the amount of rutin with sonication treatments of juice (Margean et al., 2020; Olawuyi et al., 2021). Ultrasonic processing of mallow vinegar was found to be superior to thermal pasteurization in enriching the phenolic content.

3.4 Amino acid content

Hydroxyl radicals generated by sonication cavitation bubbles can form new covalent bonds between protein polymer chains by modifying amino acids with sulfhydryl and phenolic residues (Bermúdez-Aguirre et al., 2011). As shown in Table 4, amino acids in vinegar were rich in type and content. While the content of arginine, alanine, aspartic acid, glutamic acid, lysine, proline, and threonine in ST-MV was found to be higher compared to C-MV ($p < 0.05$), while the content of ornitine was found to be lower in C-MV ($p < 0.05$).

The highest content of threonine, alanine, and proline were obtained with pasteurization treatment ($p < 0.05$). Alves et al. (2020) similar to our study; they determined that an increase in threonine, methionine, and phenylalanine levels was obtained after 3 minutes of sonication exposure in dry fermented sausages. Ahmed et al. (2019) reported that the significant ($p < 0.05$) increased in the free amino acid contents of thermo sonication treated at 30 ◦C for 20 min wheat plantlets juice. Siddeeg et al. (2019) found to increase glutamic acid in date vinegar, which was in agreement with our results ($p > 0.05$). Ding et al. (2019) also reported that there was a significant enhancement in the levels of alanine, glutamic acid, γ-aminobutyric acid, phenolic, and free sugar compounds in sonication pretreatment of oats (*Avena sativa* L.).

3.5 Mineral

Minerals are important for human health by playing a role in physical and biochemical processes. Type of food, duration of treatment, presence of water, and stability of the element; are the factors affecting the mineral content in food processing (Ahmed et al., 2019). The mineral components in vinegar in the; control, pasteurized, and sonication treatment samples are shown in Table 4. The results showed that 7 minerals were detected in the C-MV, P-MV, and ST-MV samples. Among these minerals, K content (5.056 ± 0.002 ppm) was the highest, followed by Na (0.554 ± 0.008 ppm) and Mg (0.495 ± 0.004 ppm) in ST-MV samples.

In the study where sonication was applied, it was determined that Ca content increases and Zn content decreased wheat plantlet juice showed parallelism with our study (Ahmed et al., 2019). In contrast, it was reported that significant increases in the amount of Zn after thermo-sonication treatments in grapefruit juice (Aadil et al., 2015). The increase in concentrations of Na are in accordance with the investigations of Sert et al. (2011) who reported the same increasing in the yolk of sonication treated eggs, but our results regarding K showed opposite trend to this study. Pb was not detected with vinegar samples and this result was in agreement with Codex Alimentarius Commission (CODEX), which definite the maximum content of Pb at 0.2 mg/L (Ousaaid et al., 2022). It was reported that significant increases in the amount of Fe were detected in thermo-sonication treatments applied to mango juice, but no significant effect was observed in our study (Wang et al., 2020). This is the first study concerning the effect of sonication treatment and thermal pasteurization on minerals of mallow vinegar, so more research work is required to understand the exact phenoms.

3.6 Microstructure

Microscopic images of the microstructure of mallow vinegar samples are shown in Figure 2. When the images are examined, it is seen that the cell walls of the C-MV samples are intact. However, in the P-MV sample, reductions in color pigments are seen at the end of thermal heat. It is seen that cell wall damage and color pigments increase in mallow vinegar with sonication treatment. In the study of Yıkmış et al. (2021a) it was emphasized that cell rupture occurred through cavitation caused by sonication treatment, and the surface area of suspended particles may have increased. At the same time, the cavitation and shear force created by the effect of sonication treatment can increase the effectiveness (Wu et al., 2008). As seen in Figure 2A, the color pigments are localized within the cell. Cell rupture is evident as seen in sonication treatments and

Table 4. Results of amino acid, mineral element compounds of C-MV, P-MV and ST-MV samples.

Analyzes		Samples		
		C-MV	**P-MV**	**ST-MV**
Amino acid content (mg/100 g DW)	**Alanine**	1.50 ± 0.04^{a}	1.78 ± 0.00^{c}	1.64 ± 0.00^{b}
	Arginine	0.1 ± 0.00^{a}	0.11 ± 0.00^{ab}	0.11 ± 0.00^{b}
	Aspartic Acid	2.82 ± 0.01^{a}	2.44 ± 0.00^{b}	2.95 ± 0.00^{c}
	Cystine	n.d	n.d	n.d
	Glutamic Acid	1.21 ± 0.00^{a}	1.36 ± 0.00^{b}	1.34 ± 0.01^{b}
	Glycine	0.57 ± 0.05^{a}	0.73 ± 0.01^{b}	0.67 ± 0.01^{ab}
	Histidine	n.d	n.d	n.d
	Isoleucine	0.85 ± 0.01^{a}	0.91 ± 0.01^{b}	0.89 ± 0.02^{ab}
	Leucine	1.57 ± 0.03^{a}	1.75 ± 0.03^{b}	1.61 ± 0.03^{a}
	Lysine	1.41 ± 0.00^{a}	1.66 ± 0.01^{b}	1.65 ± 0.01^{b}
	Methionine	0.25 ± 0.01^{a}	0.27 ± 0.00^{a}	0.28 ± 0.02^{a}
	Ornitine	0.92 ± 0.00^{b}	0.92 ± 0.00^{b}	0.91 ± 0.00^{a}
	Phenylalanine	1.27 ± 0.01^{a}	1.38 ± 0.06^{a}	1.38 ± 0.00^{a}
	Proline	4.82 ± 0.00^{a}	5.44 ± 0.00^{c}	5.19 ± 0.00^{b}
	Serine	1.89 ± 0.05^{a}	2.15 ± 0.02^{b}	1.88 ± 0.05^{a}
	Threonine	1.24 ± 0.02^{a}	1.55 ± 0.01^{c}	1.44 ± 0.00^{b}
	Tyrosine	0.96 ± 0.10^{a}	1.08 ± 0.06^{a}	1.02 ± 0.04^{a}
	Valine	1.42 ± 0.00^{a}	1.60 ± 0.05^{b}	1.53 ± 0.01^{ab}
	Taurine	n.d	n.d	n.d
Minerals (mg/L)	**Mg**	0.513 ± 0.001^{a}	0.483 ± 0.009^{b}	0.495 ± 0.004^{ab}
	Na	0.521 ± 0.004^{c}	0.652 ± 0.003^{a}	0.554 ± 0.008^{b}
	Fe	0.023 ± 0.001^{a}	0.03 ± 0.003^{a}	0.03 ± 0.004^{a}
	K	5.454 ± 0.013^{b}	5.79 ± 0.005^{a}	5.056 ± 0.002^{c}
	Mn	0.001 ± 0.001^{a}	0.001 ± 0.001^{a}	0.001 ± 0.001^{a}
	Zn	0.023 ± 0.002^{c}	0.042 ± 0.00^{a}	0.033 ± 0.001^{b}
	Ca	0.506 ± 0.006^{a}	0.568 ± 0.003^{a}	0.254 ± 0.359^{a}
	Cd	n.d	n.d	n.d
	Cr	n.d	n.d	n.d
	Pb	n.d	n.d	n.d

Values with different letters within the line are significantly different ($p < 0.05$). Results are presented as mean ± standard deviation (n = 3). C-MV: Mallow vinegar; P-MV: Pasteurized mallow vinegar; ST-MV: Sonication-treated mallow vinegar; n.d: Not detected.

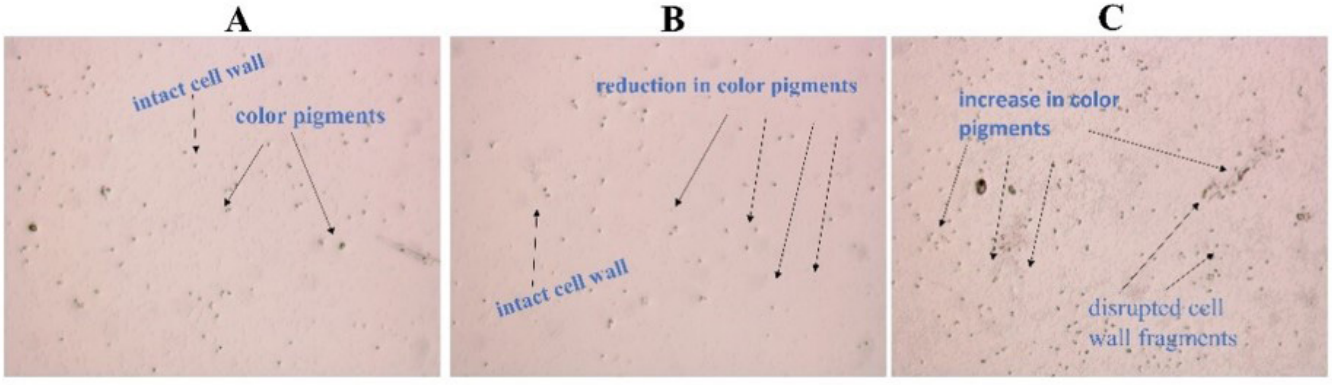

Figure 2. Microstructure of (A) C-MV: Mallow vinegar, (B) P-MV: Pasteurized mallow vinegar, (C) ST-MV: Sonication-treated mallow vinegar.

has been found to release color pigments and other compounds into the serum. Similarly, it has been found that sonication treatments cause microstructural changes in liquid foods such as mango nectar (Huang et al., 2018), tomato (Bot et al., 2017) and guava (Campoli et al., 2018) and peach juices (Rojas et al., 2016). The increase in microstructure degradation can also be used to explain the reasons for the increase in TPC and TFC numbers (Table 1).

4 Conclusion

In this study, we evaluated the effect of sonication treatment on microstructure attributes, bioactive compounds, amino acid, organic acid, sugar, mineral compounds which are important content for mallow vinegar. There was no significant difference in organic acid, sugar content, and most minerals in sonication treated mallow vinegar samples compared to untreated samples, whereas a significant increase in phenolic compounds except ascorbic acid after sonication treatment. Thermal treatment it was found to have detrimental effects on most of the phenolic compounds of mallow vinegar. Thus, ultrasonic treatment is a good alternative to thermal treatment. In this study, sonication treatments were applied to mallow vinegar, and as a result of RSM optimization, vinegar was enriched in terms of total flavonoid content and total antioxidant (DPPH and CUPRAC) amounts. The results showed that sonication processing technologies were a good alternative comparing to pasteurization treatment to the improvement quality and functionality of mallow vinegar.

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