



The role of debittering process with normal water, NaOH and lye on chemical properties of brine, antioxidant activity, phenolic compounds and sensory characteristics of fermented caper (*Capparis ovata* Desf. var. *Ovata*) buds

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Abstract

In this study, the effect of the application of salt water (brine), lye and caustic (NaOH) on salt content, acidity, pH in caper brine during fermentation was investigated. The total carotenoid quantity of the caper buds were assessed to be between 0.46 (lye) and 1.57 µg/g (hot water) on the 5th day, while the total carotenoid quantities of the buds on the 30th are found between 0.22 (normal water) and 0.94 µg/g (brine). The total carotenoid values of the caper buds on the 45th day were changed to be between 0.05 (hot water) and 0.75 µg/g (brine). The total phenol quantities of the buds on the 30th day were detected between 111.23 (caustic) and 194.48 mg GAE/100 g (hot water), while the total phenol quantities of the caper buds on the 45th day were assigned between 66.87 (lye) and 159.33 mgGAE/100 g (brine). The total flavonoid quantities of the caper buds were assigned between 289.29 (lye) and 458.81 mg catechin equivalent (CE)/100 g (normal water) on the 45th day. The highest decrease in phenolic components during fermentation was detected in catechin, rutin and resveratrol. Fermented caper buds, whose bitterness was removed with normal water on the 45th day, received the most appreciation. At the end of fermentation, the buds whose bitterness was removed with soda water, normal and hot water, normal water and caustic were most appreciated in terms of color, smell, taste and hardness.

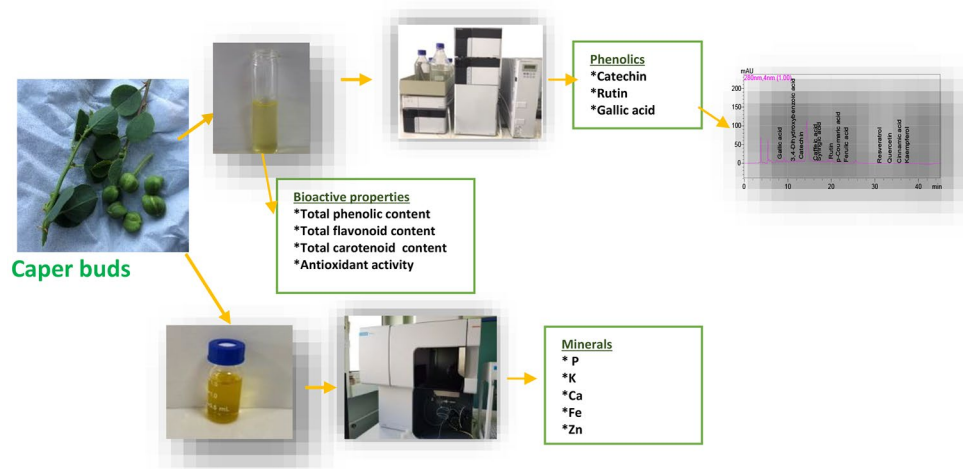
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Graphical abstract



In this study, the effect of the application of salt water (brine), oak wood ash and caustic (NaOH) on salt content, acidity, pH in caper brine during fermentation and to determine the moisture, bioactive properties (antioxidant activity, total phenol carotenoid, flavonoid), phenolic constituents and sensory characteristics of caper buds using hot water as an alternative to the bittering process with normal water in the production of fermented caper buds was investigated.

Keywords Caper bud · Fermentation · Debittering · Lye · Bioactive properties · Phenolic compounds · Sensory analysis

Introduction

Caper, a long-lived shrub plant belonging to the Cappara-ceae family, is a plant with medicinal and aromatic properties. It grows naturally in countless species (> 350) on many different continents of the World and everywhere (Inocencio et al., 2006). Caper, a tropical/subtropical plant, is known as bubu, gebre, gabar, gevil, caper, kedi tırnağı, keper, kebere, pickle grass, sebellah in different parts of Turkey [1–3]. Over the last two decades, much attention has been paid to the pharmacological effects of different parts of *Capparis* spp. due to its high number of bioactive and polyphenolic constituents [4, 5]. Pickled caper production has been used by people since ancient times and is mentioned in many works. It is a product that has been known and used for a very long time, especially in Mediterranean countries, and it attracts attention as it has an important place in nutritional habits in Turkey. Caper buds are mostly produced and consumed in pickled form due to their long shelf life. The most evaluated part of the caper is the flower bud [6]. Caper buds mostly contribute to the flavor of foods by entering into the structure of bakery products, salads, meat products, other foods or products. The flower buds of the caper plant can not be used directly as food due to their pungent, pungent taste and aroma. Hundreds of wild plants grow in nature, but after a while they dry out and their above-ground parts disappear. Sometimes the buds, fruits, leaves and shoots

of these plants, and sometimes their roots, bulbs and root barks are evaluated [7]. Recently, interest in fermented capers, one of these plants, has been increasing. However, there are no detailed studies on bitterness removal before fermentation and no standardization studies to create consumer appreciation for the consumption of caper buds. In this study, taking these issues into consideration, obtaining a fermented product that will create consumer appreciation and bringing wild plant parts into the economy is considered to be one of the other elements that constitute the originality of the project [8–10]. The most common method used worldwide to remove the bitterness of caper buds is soaking in normal drinkable water [6]. However, different processing methods are used in table olive production to remove its natural bitterness. Phenolic compounds of olive fruit, especially oleuropein, are the main phenolic component responsible for bitterness in olives [11]. The main purpose of the method of removing bitterness with water, which is one of the systems of sweetening the olive, is to ensure that the bitter substance in the olive passes into the water and to make the olive sweeten in a short time by doing this process several times at regular intervals [12]. The aim of present work was to investigate the effect of the debittering application of salt water (brine), oak wood ash and caustic (NaOH) on salt content, acidity, pH in caper brine during fermentation and to determine the moisture, bioactive properties (antioxidant activity, total phenol carotenoid, flavonoid),

phenolic constituents and sensory characteristics of control and debittered caper buds.

Materials and methods

Material

Fresh caper buds to be used in this investigation were collected from caper plants growing wild in Konya in late April or early May. Caper buds were stored in a cold environment until brought to the laboratory. After cleaning the medium-sized caper flower buds, chemical analysis and pickling process of the raw buds were carried out. Initial chemical properties (moisture, total phenol, flavonoid, carotenoid, antioxidant activity, phenolic constituents) of caper flower buds were determined.

Methods

Processing of raw caper buds in brine

Before the buds are left to fermentation, certain amounts of caper buds (200 g) are placed in clean jars (1:2/ w/v), and 1% oak lye water, normal drinkable water, hot water heated to 50 °C, 0.5% NaOH solution and 10% salt. The buds were kept in brine for 24 h to remove the bitterness. After the debittering process, the caper buds subjected to each debittering process, along with the control group, were fermented separately in 10% brine at normal room temperature for 45 days. In addition to acidity, pH and salt analysis in the brines on the 5th, 30th and 45th days of fermentation, moisture, total phenol, carotenoids, flavonoids, antioxidant activity and phenolic components were determined in the buds. Also, the sensory characteristics of the fermented buds in each fermentation period were subjected to sensory testing by 10 experienced panelists and consumer taste was determined.

Moisture contents

Moisture quantities (%) of caper buds were measured in the oven (Nüve FN055 Ankara, Turkey) at 105 °C.

Extraction processing of caper buds

For the extraction of caper buds, after 20 mL of methanol water (80:20 v/v) were added to 2 g of sample, the solution was shaken for 3 h. After 20 ml of hexane were added to the extract obtained after filtration, the mixture was subjected to phase separation in the separation funnel. Then,

the remaining methanol phase was carefully transferred into tubes for analysis [13].

Total phenolic amounts of caper buds

Total phenolic quantities of the caper buds were recorded using Folin Ciocalteu chemical according to the study explained by Yoo et al. [14]. After pretreatments, the absorbance were read at 725 nm. The findings were explained as mg gallic acid equivalent (GAE)/100 g.

Total flavonoid amounts of caper buds

Total flavonoid quantities of caper buds were established according to the report explained by Özcan et al. [7]. At the end of these treatments, the absorbance of solution was read at 510 nm. The findings are stated as mg catechin equivalent (CE)/g (dw).

Total carotenoid amounts of caper buds

Total carotenoids quantities in caper buds were recorded according to the report explained by da Rocha et al. [15]. After pre- procedures, absorption was measured with a spectrophotometer at 450 nm. The findings of total carotenoid quantities of fermented buds were depicted as µg/g.

Antioxidant activity of caper buds

Antioxidant capacity values of caper buds were evaluated using 2,2-diphenyl-1-picrylhydrazyl (DPPH) based on the study reported by Özcan et al. [7]. The absorbance values of the samples were measured at 517 nm after being kept in the dark and at room temperature for 30 min. The results are assigned as mmol trolox (TE)/kg.

Determination of phenolic constituents of caper buds

Polyphenol compounds of caper buds were identified via HPLC (Shimadzu) montaged with PDA detector and Inertsil ODS-3 (5 µm; 4.6×250 mm) column. The flow rate of the mobile phase and the injection volume were 1 ml/min at 30 °C and 20 µl, respectively [7].

Brine analysis

Determination of salt in brine during fermentation

The salt values (%) of the brines were evaluated by titrating with 0.1 N AgNO₃ [16].

Determination of titratable acidity in brine during fermentation

The titratable acidity (%) of the brine samples was recorded by titration against 0.1 N NaOH solution in the presence of phenolphthalein indicator solution [16].

Determination pH in brine during fermentation

The pH values of the brines during fermentation were assessed according to the report explained by AOAC method [16].

Sensory evaluation of caper buds during fermentation

Sensory analysis was applied to fermented caper buds at regular intervals from the beginning of fermentation. The ranking test was applied in sensory analysis. Sensory evaluation was conducted by 10 panelists. Panelists evaluated the samples by giving scores between 1 and 5 for sensory parameters [17].

Statistical analysis

Analysis of variance (ANOVA) was carried out by using JMP version 9.0. Differences between groups in the research findings determined by analysis of variance were determined by the Duncan Multiple Comparison Test.

Results and discussion

Bioactive properties of raw and fermented caper flower buds

The chemical and bioactive characteristics of caper buds debittered by the normal water (tap water), hot water (50 °C), brine, lye and caustic (NaOH) and fermented for 45 days are depicted in Table 1. Raw caper flower buds (control) contained 76.01% water, 3.06 µg/g total carotenoid, 436.41 mg GAE/ 100 g total phenol and 848.21 mg CE/100 g total flavonoid. Also, untreated caper bud has 16.86 mmol TE/kg antioxidant activity value. While the moisture amounts of caper buds subjected to fermentation are determined between 78.69 (brine) and 82.44% (hot water) on the 5th day, the moisture quantities of caper buds were changed to be between 78.89 (lye) and 82.77% (hot water) on the 30th day of fermentation. Additionally, the moisture contents of the fermented caper buds on the 45th day of fermentation were reported to be between 82.02% (lye) and 83.36% (caustic). In general, the moisture amount of the buds was higher during fermentation compared to the control. This increase may probably be due to the brine liquid diffusing into the bud during fermentation. The highest moisture result was reported on the 45th day. The moisture result of caper buds debittered by the lye was established to be partially lower than those of the buds debittered by the other debittering agents on the 30th and 45th days of fermentation.

Carotenoid contents of caper buds changed during fermentation depending on the bittering process (Table 1). The total carotenoid contents on the 5th day of caper

Table 1 Chemical and bioactive properties of fermented caper buds

	Processes	Moisture (%)	Total carotenoid contents (µg/g)	Total phenolic contents (mg GAE/100 g)	Total flavonoid contents (mg CE/100 g)	Antioxidant activity (mmol TE/kg)
5. day	Control	76.01 ± 0.73d*	3.06 ± 0.02a	436.41 ± 6.04a	848.21 ± 17.74a	16.86 ± 0.65a
	Normal water	80.61 ± 0.01b	0.55 ± 0.02e	233.49 ± 3.24c	546.43 ± 15.83d	7.74 ± 0.01b
	Hot water	82.44 ± 0.97a	1.57 ± 0.02b	180.91 ± 3.15f	480.71 ± 14.04f	7.10 ± 0.01e
	Brine (salt)	78.69 ± 1.67c	0.94 ± 0.07d	277.94 ± 3.59b	707.86 ± 18.52b	7.64 ± 0.01c
	Lye	80.93 ± 0.58b	0.46 ± 0.02f	216.31 ± 3.27e	596.90 ± 17.82c	7.67 ± 0.01bc
30. day	NaOH (caustic)	80.89 ± 0.80b	1.16 ± 0.00c	223.37 ± 2.68d	540.71 ± 17.18e	7.38 ± 0.11d
	Normal water	80.98 ± 2.18b	0.22 ± 0.02e	145.67 ± 2.91d	412.14 ± 16.06e	5.05 ± 0.28d
	Hot water	82.77 ± 0.53a	0.24 ± 0.05e	194.48 ± 3.12b	530.71 ± 17.28b	7.63 ± 0.07b
	Brine (salt)	80.13 ± 0.08b	0.94 ± 0.02b	175.44 ± 4.51c	476.90 ± 27.11d	6.91 ± 0.17c
	Lye	78.89 ± 1.55c	0.43 ± 0.05c	131.15 ± 2.27e	507.38 ± 48.74c	4.07 ± 0.15e
45. day	NaOH (caustic)	82.62 ± 0.35a	0.36 ± 0.02d	111.23 ± 3.48f	90.71 ± 11.43f	2.79 ± 0.07f
	Normal water	83.15 ± 0.05a	0.39 ± 0.05c	140.91 ± 1.76c	458.81 ± 29.79b	5.33 ± 0.12bc
	Hot water	83.33 ± 0.58a	0.05 ± 0.00f	110.20 ± 3.29e	384.05 ± 12.36d	4.21 ± 0.16c
	Brine (salt)	82.97 ± 1.52b	0.75 ± 0.07b	159.33 ± 1.25b	426.90 ± 16.73c	5.66 ± 0.22b
	Lye	82.02 ± 0.25c	0.22 ± 0.02d	66.87 ± 3.10f	289.29 ± 24.10f	0.99 ± 0.05e
	NaOH (caustic)	83.36 ± 0.65a	0.17 ± 0.02de	112.18 ± 0.49d	326.43 ± 11.43e	3.08 ± 0.06d

*values indicated by different letters in each column are significantly different at $p < 0.05$

buds debittered by different methods were recorded to be between 0.46 (lye) and 1.57 $\mu\text{g/g}$ (hot water), while the total carotenoid quantities of the buds on the 30th day are determined to be between 0.22 (normal water) and 0.94 $\mu\text{g/g}$ (brine). However, on the 45th day of fermentation, the total carotenoid quantities of caper buds were assigned to be between 0.05 (hot water) and 0.75 $\mu\text{g/g}$ (brine). Generally, the greatest decrease was determined in buds on the 45th day of fermentation (excluding bittering in normal water). While total carotenoid amounts are highest in the first stage of fermentation, a partial decrease in the total carotenoid contents of the buds was monitored towards the end of fermentation. While the total carotenoid amount of caper buds debittered with hot water is at the highest level (1.57 $\mu\text{g/g}$) at the beginning of fermentation (day 5th), it was decreased significantly at the end of fermentation (0.05 $\mu\text{g/g}$). This decrease may be likely due to the breakdown of the structures of carotenoids by the debittering treatments and the transfer of carotenoids to the brine by the biochemical reactions during fermentation.

Total phenolic amounts of the buds debittered by different methods and fermented for 45 days are displayed in Table 1. As a result of the analysis performed on the 5th day of fermentation, the total phenolic quantities of caper buds were established to be between 180.91 (hot water) and 277.94 mg GAE/100 g (brine). Additionally, the total phenolic amounts of the buds on the 30th day of fermentation were between 111.23 (caustic) and 194.48 mg GAE/100 g (hot water), while the total phenol results of the caper buds on the 45th day fermentation were between 66.87 (lye) and 159.33 mg GAE/100 g (brine). The total phenolic amounts of fermented caper buds decreased significantly by the fermentation time compared to the control. While this decrease is approximately 50% in the first analysis period of fermentation, it was determined to be approximately 75% at the end of fermentation. Although initially, the debittering process caused a decrease of approximately 40–50% in the total phenolic result of caper buds, the decrease in total phenolic content continued partially until the end of fermentation. The decrease in the total phenol amounts of the buds from the beginning to the end of fermentation may be due to the enzymes, microorganisms and biochemical reactions of the phenolic components in the bud, together with the destarvation processes applied.

Total flavonoid contents of caper buds fermented for 45 days after debittering with different debittering methods are depicted in Table 1. The results showed some fluctuations based on the debittering types and fermentation times. The total flavonoid quantities of caper buds on the 5th day of fermentation depending on the debittering types varied between 480.71 (hot water) and 707.86 mg CE/100 g (brine), while the total flavonoid amounts of the buds on the 30th day are

found between 390.71 (caustic) and 530.71 mg CE/100 g (hot water). However, the total flavonoid amounts of caper flower buds were recorded between 289.29 mg (lye) and 458.81 mg CE/100 g (normal water) on the 45th day. In general, the total flavonoid results of caper flower buds debittered with caustic (NaOH) were found to be partially lower than the other debittered buds on the 30th and 45th days. The highest decrease in the total flavonoid amounts of caper flower buds were monitored in caper buds debittered with lye on the 45th day. The total flavonoid amounts of caper buds debittered by various methods decreased significantly during fermentation compared to the control (untreated). This decrease may be due to the deterioration of the structures of some flavonoids during the bittering processes and metabolic activities that may occur during the fermentation process.

Antioxidant capacity results of caper flower buds fermented for 45 days after being subjected to bitter removal processes with different methods (normal water, hot water brine, soda water and caustic) are presented in Table 1. The antioxidant capacity results of caper flower buds on the 5th day were between 7.10 (hot water) and 7.74 mmol TE/kg (normal water), while the antioxidant activity results of the buds on the 30th day of fermentation were found between 2.79 (caustic water) to 7.63 mmol TE/kg (hot water). Additionally, the antioxidant capacities of caper flower buds were measured to be between 0.99 (lye) and 5.66 mmol TE/kg (brine) at the end of fermentation (day 45). When the antioxidant capacity results of caper flower buds were compared to the control, a decrease of approximately 50% on the 5th day was observed. The greatest decrease in antioxidant capacity values was detected in caper flower buds subjected to bitterness removal with lye and caustic on the 30th and 45th days. This decrease in the antioxidant activity values of the buds may be due to the deterioration of the structure of the phenolic compounds with antioxidant activity in the structure of the buds during bitterness removal and fermentation.

Total phenolic, antioxidant and flavonoid compounds were affected by fermentation debittering methods and storage [18]. Total phenolic, flavonoid and antioxidant capacity values of caper fruits were recorded to be between 6.5 and 11.1 mgGAE/g and 2.42–4.6 mgQE/g and 0.98–1.48 gTE/100 g, respectively [19]. Antioxidant capacity results of caper fruits according to their size were found to be 5.2–6.0 (small), 12.5–15.5 (medium) and 13.3–16.1 mM Trolox (large) [20]. Total phenolic and total flavonoid amounts of caper fruits on different sizes were assigned as 61.5–86.2 mg GAE/100 g and 39.5–51.2 mg RE/100 g (small), 41.2–81.6 mg GAE/100 g and 67.8–101.3 mg RE/100 g (moderate) and 66.8–119.2 mg GAE/100 g and 47.6–66.2 mg RE/100 g (large), respectively [20]. The findings showed

significant differences when compared to the results of last reports [9, 20]. The total phenol, flavonoid quantities and antioxidant activity values of our findings were higher than the total phenolic, flavonoid and antioxidant capacity values of caper buds in the study conducted by Jimenez-Lopez et al. [19]. These changes may arise from variety, location, climatic factors, harvest time, processed and unprocessed state of the bud and analytical procedures.

Physico-chemical properties of brines during fermentation

Changes in pH, titratable acidity (%) and salt values of the brines during the fermentation of the caper flower buds are depicted in Table 2. The pH values of the brine depending on the fermentation time and the bitter removal process applied were measured to be between 4.90 (hot water) and 6.52 (ash water) on the 5th day of fermentation, while the pH value of the brine is established between 4.49 (normal water) and 5.21 (lye) on the 30th day. In addition, on the 45th day, the pH results of the brine were determined to be between 4.51 (normal water) and 4.93 (caustic). In general, it was observed that the pH values of the brines during the fermentation of capers debittered with lye and caustic were higher than the pH values of the brines in which other debittered buds were fermented. If the properties of the brine in the environment are suitable for the development of lactic acid bacteria, the pH of the brine will decrease during fermentation due to the increases in lactic, acetic and propionic acids in this environment [21]. The increase in pH values in the brine of fermented capers may be due to the use of alkaline lye and caustic.

The titratable acidity values of the brines on the 5th day of fermentation were determined to be between 0.08% (caustic) and 0.16% (hot water), while the titratable acidity

of the brines on the 30th day is determined between 0.15 (lye and caustic) and 0.24% (brine). The titratable acidity of the brines was partially affected by the fermentation time and the debittering processes. In addition, the titratable acidity results of the brines on the 45th day of fermentation were recorded to be between 0.17 (lye) and 0.29% (normal water and brine). The titratable acidity results of the brines in which caper buds debittered with lye and caustic were fermented were found to be lower compared to the acidity results of other processed buds.

The salt quantity of the fermented brine of caper buds, whose bitterness was removed by different methods, showed differences (Table 2). The salt quantities of the brine on the 5th day were assessed to be between 6.17 (hot water) and 8.04% (normal water), while the salt quantities of the brine on the 30th day is determined between 6.78 (hot water) and 9.32% (lye). Additionally, the salt quantities of the brine on the 45th day of fermentation varied to be between 6.52 (hot water) and 9.26% (lye). In general, the salt amount of caper fermented brines, whose bitterness was removed with soda water, was higher than that of other brines. The possible reason for this may be due to the excess Na in the lye used and some of it passing into the bud and therefore diffusing into the brine. Acidity, pH and salt results of caper bud brine fermented for 42 days were determined to be between 0.350 and 0.401%, 4.94–4.59 and 5.595–5.702% [22]. pH values measured in brine depending on the fermentation time are similar to the results in the study conducted by Argun [22]. Although the results exhibited some similarities with the literature values, the values measured during fermentation showed partial differences. The main factors causing these changes may probably be due to the composition of the raw material, fermentation conditions, substances added to brine and their chemical properties and analytical conditions.

Table 2 Physico-chemical properties of brine samples in which caper buds were fermented

	Processes	pH	Titratable acidity (%)	Salt (%)
5. day	Normal water	5.00 ± 0.00b*	0.13 ± 0.00b	8.04 ± 0.03a
	Hot water	4.90 ± 0.00 cd	0.16 ± 0.04a	6.17 ± 0.03d
	Brine (salt)	4.98 ± 0.01c	0.09 ± 0.02c	7.80 ± 0.15bc
	Lye	6.52 ± 0.03a	0.09 ± 0.00c	7.89 ± 0.00b
	NaOH (caustic)	6.29 ± 0.04ab	0.08 ± 0.01 cd	7.39 ± 0.03c
30. day	Normal water	4.49 ± 0.00d	0.20 ± 0.00c	7.54 ± 0.00c
	Hot water	4.51 ± 0.00b	0.22 ± 0.00b	6.78 ± 0.00e
	Brine (salt)	4.53 ± 0.00b	0.24 ± 0.01a	7.80 ± 0.03b
	Lye	5.21 ± 0.00a	0.15 ± 0.01d	9.32 ± 0.03a
	NaOH (caustic)	4.97 ± 0.00c	0.15 ± 0.01d	7.07 ± 0.00d
45. day	Normal water	4.51 ± 0.00de	0.29 ± 0.02a	7.31 ± 0.06bc
	Hot water	4.58 ± 0.01d	0.25 ± 0.00b	6.52 ± 0.03d
	Brine (salt)	4.62 ± 0.00c	0.29 ± 0.00a	6.98 ± 0.03c
	Lye	4.79 ± 0.00b	0.17 ± 0.01 cd	9.26 ± 0.03a
	NaOH (caustic)	4.93 ± 0.00a	0.19 ± 0.01c	7.36 ± 0.00b

*values indicated by different letters in each column are significantly different at $p < 0.05$

Phenolic compounds of raw and fermented caper flower buds

The phenolic constituents of the caper buds fermented in 10% brine for 45 days after different bitter removal processes are depicted in Table 3. The gallic acid amounts of the buds on the 5th day of fermentation varied to be between 8.32 (brine) and 15.59 mg/100 g (hot water), while the gallic acid quantities of the buds on the 30th day vary between 9.40 (brine) and 10.69 mg/100 g (lye). Additionally, the gallic acid quantities of the caper buds on the 45th

day were identified to be between 8.27 (normal water) and 14.71 mg/100 g (hot water). The 3,4-dihydroxybenzoic acid quantities of fermented caper buds on the 5th and 30th days were 2.01 (lye) and 46.06 mg/100 g (brine) to 17.09 (caustic) and 38.79 mg/100 (hot water), respectively. However, the 3,4-dihydroxybenzoic acid values of caper buds on the 45th day were recorded to be between 9.29 mg/100 g (brine) and 42.65 mg/100 g (hot water). While the catechin amounts of fermented caper flower buds vary between 15.40 (brine) and 111.63 mg/100 g (caustic) on the 5th day, the catechin quantities of the buds were assessed to be

Table 3 Phenolic components of fermented caper buds (mg/100 g)

Phenolic compounds	Control	Normal water	Hot water	Brine	Lye	NaOH
5. day						
Gallic acid	27.50 ± 0.63a*	14.60 ± 2.77c	15.59 ± 3.04b	8.32 ± 0.35f	10.22 ± 1.24d	9.23 ± 0.30e
3,4-Dihydroxybenzoic acid	31.93 ± 4.04e	32.61 ± 8.89d	33.91 ± 7.16c	46.06 ± 8.19b	2.01 ± 0.11f	57.24 ± 6.99a
Catechin	130.24 ± 7.02a	60.21 ± 14.96c	16.71 ± 2.99e	15.40 ± 2.21f	37.10 ± 5.07d	111.63 ± 38.19b
Caffeic acid	38.31 ± 4.62b	32.52 ± 6.41c	30.93 ± 0.31d	26.03 ± 9.06e	41.50 ± 3.39a	11.25 ± 0.54f
Syringic acid	42.00 ± 4.53b	24.98 ± 5.75d	18.10 ± 8.29e	48.91 ± 11.45a	1.26 ± 0.35f	26.54 ± 2.86c
Rutin	23.27 ± 1.89c	47.17 ± 20.25b	1.35 ± 0.35e	10.54 ± 1.07d	0.83 ± 0.11ef	134.03 ± 62.97a
<i>p</i> -Coumaric acid	3.93 ± 0.29a	0.48 ± 0.06d	0.25 ± 0.03e	2.19 ± 0.30c	0.22 ± 0.03f	2.79 ± 0.60b
Ferulic acid	1.99 ± 0.15c	0.72 ± 0.17de	0.74 ± 0.08d	2.80 ± 0.56b	0.28 ± 0.05f	4.39 ± 0.81a
Resveratrol	9.54 ± 1.61a	5.57 ± 1.83b	0.40 ± 0.08e	3.34 ± 0.81c	2.90 ± 1.10d	0.30 ± 0.05ef
Quercetin	155.84 ± 5.13a	85.47 ± 12.33b	59.61 ± 1.59c	22.29 ± 8.72e	57.18 ± 3.70d	11.75 ± 2.00f
Cinnamic acid	2.81 ± 0.29b	1.20 ± 0.27d	3.42 ± 0.27a	1.50 ± 0.32c	1.55 ± 0.40c	0.22 ± 0.06e
Kaempferol	7.27 ± 0.42c	4.48 ± 0.72d	11.78 ± 1.51a	2.06 ± 0.39e	8.02 ± 0.12b	4.48 ± 0.74d
30. day						
Gallic acid	27.50 ± 0.63a	10.62 ± 0.39bc	10.17 ± 0.13d	9.40 ± 0.10ef	10.69 ± 0.51b	9.52 ± 0.05e
3,4-Dihydroxybenzoic acid	31.93 ± 4.04c	20.70 ± 7.57d	38.79 ± 14.20a	38.77 ± 8.38a	34.39 ± 6.77b	17.09 ± 6.74e
Catechin	130.24 ± 7.02a	12.80 ± 2.21f	39.02 ± 7.69d	71.09 ± 30.69c	78.41 ± 11.32b	18.90 ± 6.90e
Caffeic acid	38.31 ± 4.62a	4.62 ± 1.31e	5.40 ± 1.16d	10.06 ± 3.62b	9.37 ± 1.78c	1.40 ± 0.17f
Syringic acid	42.00 ± 4.53b	18.06 ± 1.86c	53.41 ± 3.74a	11.11 ± 5.05d	5.17 ± 1.98e	3.62 ± 0.55f
Rutin	23.27 ± 1.89c	29.41 ± 13.26b	83.18 ± 36.80a	2.02 ± 0.11d	0.52 ± 0.08e	22.23 ± 10.37c
<i>p</i> -Coumaric acid	3.93 ± 0.29a	2.04 ± 0.84c	2.76 ± 0.90b	0.31 ± 0.01d	0.30 ± 0.04d	0.25 ± 0.04de
Ferulic acid	1.99 ± 0.15c	2.23 ± 0.91b	2.43 ± 1.07a	0.50 ± 0.09de	0.55 ± 0.10d	0.43 ± 0.02ef
Resveratrol	9.54 ± 1.61a	1.87 ± 0.23d	3.89 ± 1.61b	0.84 ± 0.27e	0.67 ± 0.12ef	2.83 ± 1.17c
Quercetin	155.84 ± 5.13a	10.52 ± 1.41f	26.10 ± 2.25d	14.31 ± 1.28e	29.04 ± 3.91c	33.82 ± 9.17b
Cinnamic acid	2.81 ± 0.29a	0.84 ± 0.03de	1.91 ± 0.48c	0.89 ± 0.17d	2.07 ± 0.42b	2.06 ± 0.48b
Kaempferol	7.27 ± 0.42b	3.85 ± 0.64f	5.26 ± 1.35d	6.43 ± 1.34c	4.40 ± 1.13e	10.58 ± 2.82a
45. day						
Gallic acid	27.50 ± 0.63a*	8.27 ± 0.60f	14.71 ± 0.96b	9.37 ± 1.47de	9.78 ± 0.65c	9.55 ± 0.12 cd
3,4-Dihydroxybenzoic acid	31.93 ± 4.04c	29.71 ± 9.34d	42.65 ± 4.09a	9.29 ± 1.27f	17.29 ± 7.00e	35.03 ± 7.15b
Catechin	130.24 ± 7.02a	117.07 ± 32.89b	26.16 ± 7.63f	28.76 ± 8.52e	40.83 ± 15.23d	48.60 ± 14.47c
Caffeic acid	38.31 ± 4.62a	4.10 ± 0.69b	1.95 ± 0.36d	2.18 ± 0.81c	0.34 ± 0.08f	1.38 ± 0.35e
Syringic acid	42.00 ± 4.53a	3.79 ± 0.50b	0.46 ± 0.03d	1.18 ± 0.44c	0.47 ± 0.05d	0.25 ± 0.05de
Rutin	23.27 ± 1.89b	33.46 ± 13.94a	3.84 ± 0.72e	6.41 ± 1.25 cd	0.52 ± 0.09f	6.81 ± 2.00c
<i>p</i> -Coumaric acid	3.93 ± 0.29a	0.24 ± 0.08de	0.39 ± 0.08c	0.26 ± 0.01d	0.23 ± 0.03ef	0.74 ± 0.28b
Ferulic acid	1.99 ± 0.15c	0.90 ± 0.26d	4.86 ± 2.22b	0.08 ± 0.00f	0.42 ± 0.09e	12.99 ± 1.61a
Resveratrol	9.54 ± 1.61a	6.43 ± 1.70b	0.36 ± 0.08ef	3.17 ± 0.91d	0.96 ± 0.35e	4.97 ± 1.06c
Quercetin	155.84 ± 5.13a	59.95 ± 11.53d	75.87 ± 10.15b	43.29 ± 2.18e	32.48 ± 0.63f	66.69 ± 4.59c
Cinnamic acid	2.81 ± 0.29e	7.47 ± 0.50d	13.36 ± 0.90a	9.40 ± 0.96c	10.54 ± 0.58b	1.64 ± 0.16f
Kaempferol	7.27 ± 0.42f	18.64 ± 3.60b	17.66 ± 0.82c	19.98 ± 0.52a	17.19 ± 0.70 cd	12.81 ± 1.50e

*values indicated by different letters in each row are significantly different at $p < 0.05$

between 12.80 (normal water) and 78.41 mg/100 g (lye) on the 30th day. Additionally, the catechin quantities of caper samples on the 45th day were assessed to be between 26.16 (hot water) and 117.07 mg/100 g (normal water). Caffeic acid quantities of caper buds has been identified to be between 11.25 (caustic) and 41.50 mg/100 g (lye) to 1.40 (caustic) and 10.06 mg/100 g (brine) on the 5th and 30th days of fermentation, respectively. The caffeic acid values of fermented caper flower buds on the 45th day were established to be between 0.34 (lye) and 4.10 mg/100 g (normal water). The syringic acid quantities of fermented caper buds on the 5th day changed to be between 1.26 (lye) and 48.91 mg/100 g (brine), while the syringic acid amounts of caper buds on the 30th day were recorded to be between 3.62 (NaOH) and 53.41 mg/100 g (hot water). However, the syringic acid amounts of fermented caper buds on the 45th day varied to be between 0.25 (caustic) and 3.79 mg/100 g (normal water). The rutin quantities of fermented caper buds on the 5th and 30th days were identified to be between 0.83 (lye) and 134.03 mg/100 g (NaOH) to 0.52 (lye) to 83.18 mg/100 g (hot water), respectively. Rutin values of caper buds on the 45th day were assigned to be between 0.52 (lye) and 33.46 mg/100 g (normal water). Resveratrol quantities of caper buds were recorded to be between 0.67 (lye) and 3.89 mg/100 g (hot water). The resveratrol quantities fermented caper buds on the 45th day of fermentation were assessed to be between 0.36 (hot water) and 6.43 mg/100 g (normal water). Moreover, the quercetin quantities of fermented caper buds on the 5th and 30th days changed to be between 11.75 (NaOH) to 85.47 mg/100 g (normal water) and 10.52 (normal water) to 33.82 mg/100 g (normal water), while the quercetin quantities of fermented caper buds on the 45th day of fermentation vary between 32.48 (lye) and 75.87 mg/100 g (hot water), respectively. The kaempferol quantities of fermented caper buds were recorded to be between 2.06 (brine) and 11.78 mg/100 g (hot water) on the 5th day, while the kaempferol amounts of the buds are recorded to be between 3.85 (normal water) and 10.58 mg/100 g (NaOH) on the 30th day. Apart from this, kaempferol quantities of caper buds were assessed to be between 12.81 (caustic) and 19.98 mg/100 g (brine) on the 45th day.

The applied debittering processes and fermentation times had a significant effect on the phenolic constituents of caper flower buds. In general, the phenolic constituents of fermented caper flower buds were significantly decreased compared to the control. The highest decrease in phenolic constituents was observed in catechin, rutin, resveratrol and quercetin. The 3,4-dihydroxybenzoic acid contents on the 5th day of caper buds debittered with salt water and caustic partially increased compared to the control. In addition, the kaempferol quantities of caper buds debittered with hot

water and lye on the 5th day were partially higher compared to the control. The phenolic compound quantities of fermented caper buds subjected to debittering were significantly reduced compared to the control on the 30th day. However, the quantities of some debittered phenolic compounds were found to be higher compared to the control. The kaempferol values of fermented caper buds debittered with caustic increased compared to the control. The phenolic constituents of fermented caper flower buds on the 45th day decreased significantly compared to the control. The highest decrease was monitored in catechin, caffeic acid, syringic acid, rutin and quercetin. In general, the phenolic constituents of caper buds decreased during fermentation. The possible reason for the decrease in the quantities of phenolic components of fermented caper buds compared to unprocessed caper flower buds (control) may be due to the fact that factors such as lye, caustic and hot water used to remove bitterness and biochemical activities during fermentation cause the structures of phenolic components to deteriorate. Here, it has generally been found that the most effective debittering process is lye and caustic solution.

Maldini et al. [23] determined the flavone glycoside rutin in *Capparis spinosa*. In a study, it was reported that the fermentation process decreased the epicatechin concentration from the beginning to the end of the fermentation [24]. The phenolic compound found in the highest amount among the five polyphenols detected in caper fruits, was rutin [10]. Conforti et al. [25] reported that quercetin and rutin were also determined in caper fruits. Rutin can also be directly hydrolyzed by hesperidinase to produce quercetin [26]. According to results from this study, fermented caper buds are explained by a different polyphenolic profile compared to the unprocessed fruits. Phenolic constituents of fresh (raw) and fermented caper buds have been monitored in previous caper bud and fruits studies to be related to the concept that phenolic constituents decrease with fermentation. Results exhibited some changes compared with results the last reports [10, 23, 24]. These changes may probably be due to the caper species and variety, the degree of development of the buds, harvest period and growing factors.

Sensory evaluation of fermented caper flower buds

The results of sensory analysis performed at certain periods during the fermentation of caper buds debittered by various methods are assigned in Table 4. It has been determined that the sensory parameters (color, smell, taste and hardness) of caper buds vary depending on the applied bittering process and fermentation time. The color results of fermented caper buds on the 5th and 30th days were evaluated between 4.33 (hot water and brine) and 5.00 (lye) to 4.33 (lye and caustic) and 5.00 (normal water and hot water), respectively. The

Table 4 Sensory properties of fermented caper buds

	Processes	Color	Odor	Flavour	Hardness	General appreciation
5. day	Normal water	4.67 ± 0.47b*	4.67 ± 0.47b	4.67 ± 0.47a	4.33 ± 0.47a	4.67 ± 0.47a
	Hot water	4.33 ± 0.47c	4.67 ± 0.47b	3.67 ± 0.94c	3.67 ± 0.47c	4.00 ± 0.82b
	Brine (salt)	4.33 ± 0.47c	4.67 ± 0.47b	3.67 ± 0.47c	4.00 ± 0.00b	4.00 ± 0.00b
	Lye	5.00 ± 0.00a	5.00 ± 0.00a	4.00 ± 0.00b	4.33 ± 0.47a	4.67 ± 0.47a
	NaOH (caustic)	4.67 ± 0.47b	4.33 ± 0.94c	3.67 ± 0.47c	4.00 ± 0.00b	3.67 ± 0.47c
30. day	Normal water	5.00 ± 0.00a	4.67 ± 0.47b	5.00 ± 0.00a	5.00 ± 0.00a	5.00 ± 0.00a
	Hot water	5.00 ± 0.00a	4.33 ± 0.94c	4.67 ± 0.47b	5.00 ± 0.00a	5.00 ± 0.00a
	Brine (salt)	4.67 ± 0.47b	4.67 ± 0.47b	4.00 ± 0.82d	5.00 ± 0.00a	4.00 ± 0.00c
	Lye	4.33 ± 0.47c	5.00 ± 0.00a	4.33 ± 0.94c	4.67 ± 0.47b	4.50 ± 0.50b
	NaOH (caustic)	4.33 ± 0.47c	4.67 ± 0.47b	4.00 ± 0.82d	4.67 ± 0.47b	4.50 ± 0.50b
45. day	Normal water	4.00 ± 0.00c	5.00 ± 0.00a	5.00 ± 0.00a	5.00 ± 0.00a	5.00 ± 0.00a
	Hot water	4.00 ± 0.00c	5.00 ± 0.00a	4.33 ± 0.47c	5.00 ± 0.00a	4.33 ± 0.47c
	Brine (salt)	3.00 ± 0.00d	4.00 ± 0.00c	4.00 ± 0.00d	5.00 ± 0.00a	4.00 ± 0.00d
	Lye	5.00 ± 0.00a	4.67 ± 0.47b	4.67 ± 0.47b	5.00 ± 0.00a	4.67 ± 0.47b
	NaOH (caustic)	4.67 ± 0.47b	4.00 ± 0.82c	5.00 ± 0.00a	5.00 ± 0.00a	4.67 ± 0.47b

*values indicated by different letters in each column are significantly different at $p < 0.05$

color values of the buds on the 45th day were determined to be between 3.00 (brine) and 4.67 (caustic). While the odor values of caper buds are evaluated between 4.33 (caustic) and 5.00 (lye) on the 5th day of fermentation, the odor values of the buds changed between 4.33 (hot water) and 5.00 (lye) on the 30th day of fermentation. The odor scores of the buds on the 45th day were established between 4.00 (brine and caustic) and 5.00 (normal water and hot water). The taste values of caper buds on the 5th, 30th and 45th days were evaluated between 3.67 (hot water, brine and lye) and 4.67 (normal water), 4.00 (brine and lye) and 5.00 (normal water) and 4.00 (brine) and 5.00 (normal water and caustic), respectively. However, the hardness values of fermented caper buds on the 5th day varied between 3.67 (hot water) and 4.33 (normal water and lye), while the hardness scores of caper flower buds on the 30th day are evaluated between 4.67 (lye and caustic) and 5.00 (normal water, hot water and brine). Sensory parameters of all samples on the 45th day received the highest score (5.00). In terms of general appreciation, fermented caper buds that were debittered with normal water and hot water on the 30th day of fermentation and normal water on the 45th day received the most appreciation. The hardness values of caper buds debittered with lye increased towards the end of fermentation. This increase may be due to the lye that has diffused into the bud and passed into the brine during fermentation, causing the product texture to harden. Additionally, the taste of caper buds generally increases during fermentation. The reason for this increase in taste may be due to the decomposition of the bitter phenolic compounds in the caper bud and the decrease in bitterness due to biochemical reactions that occur during both bitterness removal and fermentation. Özcan [6] found that samples containing 6% salt and 0.5% lactic acid were the most appreciated in lactic acid-added caper pickles, and

the sensory characteristics of pickles with fermented caper fruits varied depending on the additive added to the brine.

Conclusion

The moisture results of caper buds increased partially during fermentation. The highest moisture result was reported on the 45th day of fermentation. The water content of caper buds debittered with lye on the 30th and 45th days of fermentation was found to be partially lower than that of other debittered buds. The greatest reduction was determined in buds on the 45th day of fermentation (excluding bittering in normal water). While total phenol amounts are established to be highest in the first period of fermentation, a partial decrease was observed in the total carotenoid quantities of the buds towards the end of fermentation. The total phenolic quantities of fermented caper flower buds decreased significantly with the fermentation time compared to the control. The highest decrease in the total flavonoid quantity of the caper flower buds was observed in caper flower buds debittered with lye on the 45th day. When the antioxidant capacity results of the caper flower buds detected during fermentation were compared to the control, a decrease of approximately 50% on the 5th day and 60–70% on the other days of fermentation was observed. The greatest decrease in antioxidant activity values was detected in caper flower buds subjected to bitterness removal with lye and caustic on the 30th and 45th days. In general, the pH results of the brines fermented with capers debittered with lye and caustic during fermentation were found to be higher than the others. During fermentation, the titratable acidity results of the brines in which caper buds debittered with lye and caustic were fermented were low compared to the acidity values of

other processed buds. In general, the salt result of the brines fermented with capers debittered with lye was higher than the others.

As a result, the phenolic component amounts of fermented caper flower buds were significantly reduced compared to the control. The highest decrease in phenolic components was observed in catechin, rutin, resveratrol and quercetin. The 3,4-dihydroxybenzoic acid contents of caper buds debittered with salt water (brine) and caustic on the 5th day partially increased compared to the control. In addition, on the 5th day, the rutin result of caper flower buds debittered with normal water and caustic and subjected to fermentation was assigned to be higher compared to the control. In addition, the kaempferol amounts of caper buds debittered with hot water and lye on the 5th day were partially higher compared to the control. The phenolic constituents amounts of fermented caper buds subjected to debittering on the 30th day decreased significantly compared to the control. However, the amounts of some debittered phenolic compounds were found to be higher compared to the control. Additionally, the rutin quantity of fermented caper buds debittered with normal water increased compared to the control. It was observed that the phenolic constituents of caper buds decreased during fermentation.

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Data availability Data supporting the results of this study are available from the corresponding author upon reasonable request.

Declarations

Conflict of interest No conflict of interest.

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