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Effect of Freeze-Thaw and Ultrasonic Pretreatment on the Drying Rate and Quality of Lemon (*Citrus limon*) Slices

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ABSTRACT

The drying of lemon slices requires considerable time and often leads to unwanted alterations in their characteristics, such as discoloration and the degradation of bioactive compounds. Pretreatment has been widely applied to address these issues. This study aimed to investigate the effects of freeze-thaw and ultrasonic pretreatments on the drying rate and quality attributes of dried lemon slices. Lemon slices were subjected to ultrasonic pretreatment (40 kHz, 200 W) for 0, 10, and 20 min, either alone or combined with freeze-thaw (-20°C for 12 h), and subsequently dried using a convection oven at 60°C. The 10 min ultrasonic pretreatment without freeze-thawing was identified as the optimal approach based on process efficiency and quality retention. This treatment increased the drying rate ($p < 0.05$) and rehydration ratio ($p < 0.05$), reduced the browning index ($p < 0.05$) and total color difference (ΔE) ($p < 0.05$), maintained the total phenolic and total flavonoid contents of the dried lemons, and exhibited the lowest reduction in vitamin C content and antioxidant activity compared to the other pretreatments.

1. INTRODUCTION

Lemons (*Citrus limon*) are widely recognized for their health-promoting properties, as they are rich in vitamin C and bioactive compounds that exhibit antioxidant activity (Siddiq, 2012; Ding *et al*., 2017). Nevertheless, the high moisture content of lemons (approximately 89%) makes them highly perishable. Generally, lemons can be stored at room temperature for up to 14 days (Kayesh *et al*., 2018). Improper storage can result in microbial contamination and substantial postharvest losses (Malakar *et al*., 2023).

Drying lemons can extend their shelf life, reduce the risk of microbial contamination, ensure supply availability, and offer convenience by minimizing packaging requirements and reducing transport weight (Sun & Liang, 2020). Dried or powdered lemon is widely utilized in the food, beverage, and health industries. As these industries expand, the demand for dried lemon is also projected to increase. Convection drying is the most widely used technology as it can be easily adopted by small-scale enterprises. However, it is often limited by long processing durations, high energy consumption, and unfavorable alterations in quality such as degradation of thermolabile nutrients and discoloration (Roy *et al*., 2022). Color is one of the essential quality indicators during the drying of fruits, as it directly affects consumer acceptance and product marketability. Products with poor visual appearance are generally considered less attractive to buyers (Aksüt *et al*., 2023). Thus, alternative techniques are required to reduce drying time while simultaneously preserving the quality of lemon products dried in a convective oven.

To address this, various pretreatments have been applied to improve drying performance and maintain the quality of dried fruits, such as freeze-thaw and ultrasonic pretreatment (Deng *et al*., 2019). Freeze-thaw pretreatment, which involves freezing fruit at low temperatures followed by thawing, induces structural changes in the tissue that promote moisture removal and drying efficiency (Liu *et al*., 2021; Bassey *et al*., 2023). Ultrasonic pretreatment is a relatively new technology that utilizes mechanical waves with frequencies over 20 kHz to treat materials. Compared with conventional methods, it offers benefits such as easy operation, reduced energy use, and environmentally friendly processing (Zhao *et al*., 2024). Yildiz & Izli (2019) observed that ultrasonic pretreatment enhanced the drying of quince by producing samples with greater lightness ($L*$), higher rehydration ratio, and lower browning index than untreated samples. The synergistic use of freeze-thaw and ultrasound is particularly interesting to explore. For instance, Jiang *et al*. (2024) found that applying both treatments to strawberries improved drying rate, vitamin C, phenolic compounds, anthocyanin, and antioxidant activity. In contrast, Xu *et al*. (2021) reported that the same pretreatment decreased the antioxidant activity, total flavonoid and total phenolic content of dried okra. Such discrepancies highlight that the effects of these pretreatments are not yet consistent.

To date, limited studies have evaluated the combined effect of ultrasonic and freeze-thaw pretreatments on lemon drying. Therefore, this research was conducted to examine their influence on the drying rate, rehydration ratio, color, vitamin C, total phenolic content (TPC), total flavonoid content (TFC), and antioxidant activity.

2. MATERIALS AND METHODS

2.1. Sample Preparation

Lemons (*Citrus limon* cv. California) were procured from Pangalengan, Bandung, West Java, Indonesia (7.17° S and 107.57° E), harvested at 4 months of maturity, and had undergone a degreening treatment. Lemons were selected based on their color, shape, and weight (149±8.5 g), and the absence of physical defects to ensure uniformity. The lemons were then cut into slices of approximately 3 mm thickness using a slicer.

2.2. Design of Experiment

The experimental design used in this study was a factorial Completely Randomized Design, where the first factor was freeze-thaw pretreatment (without and with) and the second factor was ultrasonic pretreatment (0, 10, and 20 min), resulting in a total of six treatments. Ultrasonic pretreatment was performed by immersing the sliced lemon samples in an ultrasonic bath (J.P. Selecta; Barcelona, Spain) filled with distilled water, operated at 40 kHz and 200 W for 0 (without ultrasonication), 10, and 20 min. The ratio of sample to water was 1:4, and the process was conducted at room temperature (25 °C). After treatment, the slices were removed and blotted with filter paper to eliminate excess surface water. This procedure was adopted from the methods described by Wang *et al*. (2019) and Yildiz & Izli (2019).

Freeze-thaw pretreatment was performed by freezing the samples in a freezer (RSA; Royal Sutan Agung; Jakarta, Indonesia) at -20 °C for 12 h, followed by thawing the frozen samples at room temperature until they completely thawed (approximately 3 h). In the combined pretreatment, the freeze-thawed slices were subsequently exposed to ultrasonic treatment under the same conditions (the reverse sequence was not performed). The freeze-thaw method and its combination with ultrasonication were adapted from the methods of Bassey *et al*. (2023) and Xu *et al*. (2021).

The samples were placed on trays and dried using a convection oven (DNE601; Yamato Scientific; Tokyo, Japan) at 60 °C (air velocity of 0.7 m/s and RH 10%), until their moisture level decreased to about 10% (gravimetric method).

2.3. Moisture Content

Moisture content was determined using the gravimetric method by drying the samples in an oven at 105 °C until a constant weight was reached (approximately 24 h). All measurements were performed in duplicate. The moisture content of the samples was calculated using Equation (1).

$$\text{Moisture content (\%)} = \frac{\mathrm{m_0} - m}{\mathrm{m_0}} \times 100 \quad (1)$$

where m_0 represents the initial weight of the samples (g), and m represents the final weight of the samples (g).

2.4. Drying Rate

The drying rate was calculated according to Zhang *et al*. (2020) using Equation 2.

$$\text{Drying rate (g/g}\cdot\text{h)} = \frac{M_0 - M_t}{t} \tag{2}$$

where M_0 and M_t is respectively initial and final moisture content on dry basis (g/g), and *t* is drying time (h).

2.5. Rehydration Ratio

Rehydration ratio was assessed using the procedure of Kesbi *et al.* (2016) with minor modifications. About five grams of samples were immersed in 250 mL of distilled water at 50 °C for 60 min. After soaking, samples were drained, surface water was blotted with filter paper and then weighed. All measurements were performed in duplicate. The rehydration ratio was determined using Equation 3.

$$\text{Rehydration ratio (\%)} = \frac{w_1 - w_0}{w_0} \times 100 \tag{3}$$

where w_0 is weight of the samples before immersion (g), w_1 is weight of the samples after immersion (g).

2.6. Color

Color properties were determined using a Chromameter (CR-410; Konica Minolta; Tokyo, Japan) following Yildiz & Izli (2019). All measurements were performed in triplicate. The total color difference (*ΔE*) between fresh and dried samples was calculated with Equation 4, while the browning index (*BI*) was calculated using Equations 5 and 6.

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \tag{4}$$

$$\text{BI} = \frac{X - 0.31}{0.17} \times 100 \tag{5}$$

$$X = \frac{a^* + 1.75L^*}{5.645L^* + a^* - 3.012b^*} \tag{6}$$

where L_0*, a_0*, b_0* are the color parameters of fresh lemon, and L^*, a^*, b^* are the color parameters of dried lemon.

2.7. Vitamin C

The measurement of vitamin C content was performed using a spectrophotometric method adapted from Kholifah *et al.* (2023) with minor modifications. Five grams of lemon powder (20 mesh) was dissolved in 25 mL of ethanol (96%) and macerated for 3 days. The lemon extract was concentrated using a rotary evaporator at 40 °C. Subsequently, 100 mg of the lemon extract was dissolved in 100 mL of distilled water, then diluted to 100 µg/mL, and its absorbance was recorded at 266 nm using a UV-Vis spectrophotometer (Jenway 736501 Genova Plus; Keison Products; Essex, UK). Standard solutions of ascorbic acid in the range of 10–20 ppm were prepared for the calibration curve. All measurements were performed in duplicate.

2.8. Extraction of Phenolic Compounds

A total of 160 mg of dried lemon powder (40 mesh) was homogenized with 1.6 mL of 80% methanol and subjected to ultrasonic extraction for 30 min, followed by centrifugation at 10,000 rpm for 15 min at 4 °C. The resulting supernatant was stored at -20 °C prior to analysis (Jiang *et al*., 2024).

2.9. Total Phenolic Content (TPC)

The total phenolic content was measured using the Folin–Ciocalteu method (Phonphoem *et al*., 2022). Briefly, 200 µL of supernatant was diluted with 800 µL absolute methanol, then 20 µL of this solution was mixed with 0.1 mL of 10% Folin–Ciocalteu reagent. Afterward, 80 µL of Na_2CO_3 (1 M) was introduced, and the mixture was incubated in the dark for 20 min. The absorbance values were determined at 760 nm using Microplate Reader (BioTek Synergy HTX; Agilent; Santa Clara, CA, USA). Gallic acid was used as a standard, and the results were expressed as milligrams of gallic acid equivalents per gram of sample (mg GAE/g). All measurements were performed in triplicate.

2.10. Total Flavonoid Content (TFC)

The total flavonoid content was determined using the aluminum chloride colorimetric assay (Essiedu *et al*., 2023). In this procedure, 200 µL of supernatant was combined with 800 µL of absolute methanol. A 50 µL aliquot of this solution was then mixed with 50 µL methanol, 20 µL of 2.5% (w/v) $AlCl_3$, and 20 µL of CH_3COONa (0.3 M). After adding 0.1 mL of distilled water, the mixture was kept for 30 min in the dark. The absorbance values were recorded at 425 nm using Microplate Reader (BioTek Synergy HTX; Agilent; Santa Clara, CA, USA). Quercetin was used as a standard, and the results were expressed as milligrams of quercetin equivalents per gram of sample (mg QE/g). All measurements were performed in triplicate.

2.11. Antioxidant Activity

The antioxidant activity was evaluated through the DPPH (2,2-diphenyl-1-picrylhydrazyl) assay (Phonphoem *et al*., 2022). In this test, 200 µL of supernatant was mixed with 800 µL absolute methanol, and from this solution, 25 µL was added to 200 µL of DPPH reagent (150 µM). The reaction mixture was incubated in the dark at ambient temperature for 30 min, after which the absorbance was measured at 517 nm using Microplate Reader (BioTek Synergy HTX; Agilent; Santa Clara, CA, USA). Methanol served as the negative control, whereas 0.1% (w/v) ascorbic acid prepared in methanol (0–100 µg/mL) served as the positive control. All measurements were performed in triplicate. The radical scavenging activity was presented as the percentage inhibition of DPPH radicals, calculated using Equation 7.

$$\text{Scavenging effect (\%)} = \frac{A_0 - A_S}{A_0} \times 100 \tag{7}$$

where A_0 is absorbance of the negative control, and A_S is absorbance of the samples.

2.12. Data Analysis

All experiments were conducted in triplicate, and results are expressed as mean ± SD. Statistical evaluation was performed using two-way analysis of variance (ANOVA) with a 95% confidence interval ($p < 0.05$). The first factor was the freeze-thaw pretreatment (without and with), and the second factor was the ultrasonic pretreatment (0, 10, and 20 min). Significant differences between treatment means were further analyzed using Duncan's Multiple Range Test (DMRT). Data processing was executed using RStudio software (version 2024.12.1, Build 563; Boston, MA, USA).

3. RESULTS AND DISCUSSION

3.1. Drying Rate

Figure 1 presents the drying rates obtained for different pretreatments. Two-way ANOVA indicated that freeze-thaw, ultrasonic, and their interaction significantly enhanced the drying rate ($p < 0.05$). The findings demonstrate that the combined freeze-thaw and 20 min ultrasonic pretreatment yielded the highest drying rate of 1.17±0.03 g/g·h, which was 121% higher than that of the control (0.53±0.01 g/g·h). These results are in line with earlier studies, which demonstrated that applying both pretreatments improved drying performance in okra (Xu *et al*., 2021), apricot (Li *et al*., 2024), and strawberry (Jiang *et al*., 2024).

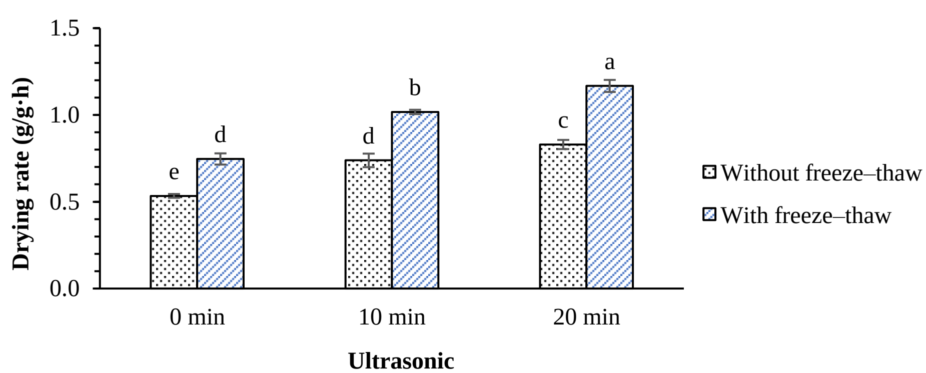

Figure 1. Effect of ultrasonic treatment on the drying rate. [Error bars indicate standard deviation from three (n = 3) measurement, and different letters above columns indicate significant difference ($p < 0.05$)]

Sonication generates cavitation and mechanical forces that cause cyclic expansion and contraction in the tissue, producing micro-channels that facilitate water movement and removal during drying (Xu *et al*., 2022). Similarly, freeze-thaw treatment damages the cell matrix, modifies membrane permeability, and accelerates water diffusion (Liu *et al*., 2021; Li *et al*., 2024). When both treatments are applied together, the process achieves the greatest dehydration efficiency, confirming the synergistic contribution of the two pretreatments in shortening drying duration (Jiang *et al*., 2024; Li *et al*., 2024).

3.2. Rehydration Ratio

Drying causes irreversible changes; however, the product is expected to regain an appearance like its fresh state through hydration before consumption (Xu *et al*., 2022). Rehydration ratio is one of the essential quality attributes for dried products (Wang *et al*., 2018; Li *et al*., 2024). The rehydration ratio of the dried lemon samples for each pretreatment is shown in Figure 2. Two-way ANOVA showed that freeze-thaw and ultrasonic pretreatments significantly improved the rehydration ratio of the samples ($p < 0.05$). However, the interaction between the two pretreatments had no significant effect. The highest rehydration ratio was observed in samples subjected to both freeze-thaw and 20 min ultrasonic pretreatments (368.19±9.66%), which was 49.6% higher than that of the control (246.17±5.01%). As shown in Figure 2, a positive correlation was observed between the rehydration ratio and the duration of ultrasonication.

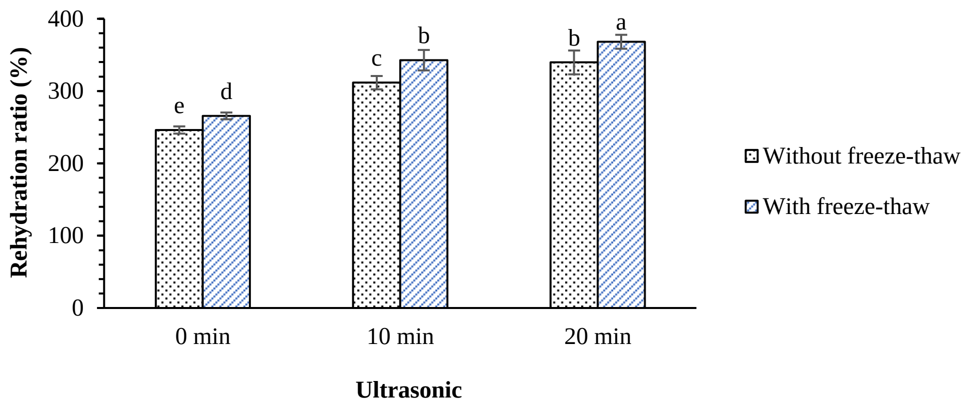

Figure 2. Rehydration ratio of dried lemons from each pretreatment, where error bars indicate mean ± SD ($n = 3$) and different letters above columns indicate significant difference ($p < 0.05$)

The rehydration ratio is strongly influenced by the porosity of the dried tissue, with higher porosity leading to better water uptake. Ultrasonic pretreatment facilitates moisture penetration into the sample by generating micro-channels through cavitation, thereby improving rehydration performance (Li *et al*., 2024). According to Wang *et al*. (2019), extended ultrasonic treatment enlarges micro-channels and pores, further enhancing water absorption. Similarly, ice crystal formation during freeze-thaw disrupts internal cellular structures, producing porous tissue that promotes rehydration via capillary action (Li *et al*., 2024). According to Jiang *et al*. (2024), freeze-thaw treatment enlarges the cell wall gaps of the material and forms numerous irregular channels, thus increasing its rehydration capacity.

3.3. Color Analysis

The effect of pretreatments on the color of dried lemon is summarized in Table 1. The L^* value reflects the lightness level of the sample and is inversely related to browning intensity; a lower L^* value indicates a greater browning reaction (Wang *et al*., 2018).

Two-way ANOVA revealed that freeze-thaw and ultrasonic pretreatments significantly increased the L^* value ($p < 0.05$), but their interaction had no significant effect. The highest L^* value was achieved in samples subjected to both freeze-thaw and 20 min ultrasonic pretreatment (71.24±1.17), which was 11% higher than the control (64.16±0.66).

The results showed that ultrasonic pretreatment alone was able to increase the $L*$ value compared to the control, with the $L*$ value increasing as the ultrasonic duration increased. Furthermore, freeze-thaw pretreatment also increased the $L*$ value compared to the control. In general, a decrease in the $L*$ parameter is undesirable as it means the product becomes darker, which is unacceptable for dried lemon (Salehi & Kashaninejad, 2018).

These findings are consistent with previous reports. For instance, Yildiz & Izli (2019) observed that ultrasonic pretreatment in quince drying significantly improved $L*$ values compared to untreated controls. Similarly, Nzimande *et al*. (2024) found comparable results in tomato drying, and Xu *et al*. (2021) confirmed that both ultrasonic and freeze-thaw pretreatments could increase $L*$ values during okra drying.

The browning index (*BI*) serves as an indicator to assess the extent of brown pigmentation resulting from both enzymatic and non-enzymatic reactions during drying (Wang *et al*., 2018). The lowest browning index was found in samples subjected to both freeze-thaw and 20 min ultrasonic pretreatment (see Table 1), which was 42% lower than the control. Two-way ANOVA revealed that only ultrasonic pretreatment was proven to significantly decrease the browning index ($p < 0.05$), whereas freeze-thaw pretreatment and its interaction with ultrasonic pretreatment had no significant effect. The results show that the longer the ultrasonic duration, the lower the browning index value. Previous research also reported similar findings, where ultrasonic pretreatment was able to reduce the browning index in the drying of kiwifruit (Wang *et al*., 2019) and quince (Yildiz & Izli, 2019). The decrease in the browning index with ultrasonic pretreatment may be related to the inactivation of the polyphenol oxidase (PPO) enzyme, which catalyzes the formation of undesirable brown pigments. Previous research has reported that ultrasonic treatment significantly reduces PPO activity in apple, pear, and strawberry purees (Sulaiman *et al*., 2015).

Table 1. Color parameters of dried lemon samples from each pretreatment

Pretreatment		$L*$	$a*$	$b*$	ΔE	BI
Ultrasonic	Freeze-thaw					
0 min	Without Freeze-thaw	64.16±0.66^{d}	6.35±0.17^{a}	27.85±1.07^{a}	62.83±2.93^{a}	14.77±0.70^{a}
	With Freeze-thaw	66.15±0.19^{c}	4.03±0.26^{c}	28.59±0.51^{a}	59.52±1.11^{a}	12.63±0.29^{b}
10 min	Without Freeze-thaw	68.23±0.89^{b}	5.07±0.62^{b}	27.04±0.43^{a}	54.78±2.30^{b}	11.22±0.98^{bc}
	With Freeze-thaw	68.35±0.38^{b}	3.53±0.08^{c}	27.90±1.33^{a}	54.92±3.12^{b}	10.76±0.91^{cd}
20 min	Without Freeze-thaw	70.51±0.17^{a}	4.11±0.46^{c}	26.89±1.26^{a}	51.22±3.35^{bc}	9.56±1.15^{de}
	With Freeze-thaw	71.24±1.17^{a}	2.21±0.30^{d}	27.40±0.60^{a}	49.59±1.94^{c}	8.56±0.57^{e}

Note: ΔE = Total color difference (compared to the fresh samples); *BI* = Browning index. Values are expressed as mean ± standard deviation (n = 3). Means in the same column followed by different letters are significantly different ($p < 0.05$).

The total color difference (ΔE) is commonly applied to evaluate the extent of deviation between fresh and dried samples. Smaller ΔE values suggest closer similarity to the fresh product (Wang *et al*., 2018). Two-way ANOVA showed that freeze-thaw and ultrasonic pretreatments could independently and significantly decrease the ΔE value ($p < 0.05$), but the two treatments did not interact. The lowest ΔE was recorded in samples subjected to both freeze-thaw and 20 min ultrasonic pretreatment (see Table 1), which was 21% lower than the control. This outcome is in line with Li *et al*. (2024), who reported that apricot drying with these pretreatments also led to lower ΔE values compared to untreated samples. Wang *et al*. (2019) similarly noted that ultrasonic pretreatment decreased ΔE and browning index values in kiwifruit drying. Pretreatments generally shorten drying time and reduce pigment degradation by limiting direct exposure to hot air (Gu *et al*., 2024).

3.4. Vitamin C

Vitamin C is a key nutritional indicator in fruit drying. Being a water-soluble compound, it is highly unstable and easily degraded by exposure to temperature, light, pH, and oxygen (Wang *et al*., 2019). The vitamin C levels of samples under different pretreatments are shown in Table 2. Two-way ANOVA revealed that both freeze-thaw and ultrasonic pretreatments significantly reduced vitamin C content ($p < 0.05$), with a significant interaction between the two treatments ($p < 0.05$). The control samples retained the highest amount of vitamin C (65.23±0.99 mg/g), indicating that the pretreatments accelerated nutrient loss.

Table 2. Bioactive compounds and antioxidant activity of dried lemon samples from each pretreatment

Pretreatment		Vitamin C (mg/g)	TPC (mg GAE/g)	TFC (mg QE/g)	Antioxidant Activity (%)
Ultrasonic	Freeze-thaw				
0 min	Without Freeze-thaw	65.23±0.99^{a}	6.08±0.22^{a}	0.44±0.00^{a}	43.75±1.61^{a}
	With Freeze-thaw	47.07±4.63^{cd}	5.43±0.22^{bc}	0.33±0.01^{c}	38.43±1.84^{b}
10 min	Without Freeze-thaw	54.60±3.58^{b}	5.78±0.20^{ab}	0.43±0.01^{a}	40.01±1.21^{b}
	With Freeze-thaw	40.68±3.09^{e}	4.96±0.44^{c}	0.34±0.01^{c}	33.80±1.65^{c}
20 min	Without Freeze-thaw	50.92±4.00^{bc}	5.53±0.08^{b}	0.40±0.01^{b}	39.73±0.59^{b}
	With Freeze-thaw	42.87±2.59^{de}	4.99±0.32^{c}	0.35±0.02^{c}	33.91±1.42^{c}

Note: data are presented on a wet basis (w.b); TPC = Total Phenolic Content; GAE = Gallic acid equivalent; TFC = Total Flavonoid Content; QE = Quercetin equivalents. Values are expressed as mean ± standard deviation (n = 3). Means in the same column followed by different letters are significantly different ($p < 0.05$).

These findings are consistent with Li *et al*. (2024), where dried apricot samples pretreated with ultrasonication, freeze-thaw, and their combination had lower vitamin C content than the control. Similar results were observed by Nian *et al*. (2024) during chili pepper drying. The reduction in vitamin C during freeze-thaw is associated with the formation of ice crystals, which damage the cellular structure, leading to drip loss and the release of vitamin C from disrupted cells during thawing (Li *et al*., 2024). Moreover, the long duration of the thawing process also contributes to the degradation of vitamin C due to oxidation (Nian *et al*., 2024). Combining this with ultrasonication intensified the degradation, as evidenced by the combined freeze-thaw and 10 min ultrasonic pretreatments yielding the lowest vitamin C content (40.68±3.09 mg/g). The cavitation effect during sonication damages cells and causes ascorbic acid to be released into the surrounding water medium, owing to its soluble properties (Wang *et al*., 2019).

3.5. Total Phenolic Content (TPC)

Table 2 presents the total phenolic content of dried lemons under different pretreatments. Both freeze-thaw and ultrasonic treatments independently led to a significant reduction in TPC ($p < 0.05$), while no interaction effect was detected. The highest total phenolic content was found in the control samples (6.08±0.22 mg GAE/g), while the lowest was recorded in those subjected to both freeze-thaw and ultrasonic pretreatments, whether for 10 min (4.96±0.44 mg GAE/g) or 20 min (4.99±0.32 mg GAE/g). The total phenolic content in samples from the 10 min ultrasonic pretreatment without freeze-thawing (5.78±0.20 mg GAE/g) showed a non-significant decrease, indicating that this treatment had better retention than the other treatments, preserving 95.07% of the TPC compared to the control.

Based on the results, prolonged ultrasonic treatment caused a greater loss of phenolic compounds due to their dissolution into the water used as the ultrasonic medium. Furthermore, the findings indicated that the freeze-thaw pretreatment also reduced the phenolic content of the samples. This finding aligns with a report by Xu *et al*. (2021) on okra drying, which found that ultrasonic, freeze-thaw, and their combined pretreatments produced samples with lower total phenols than the control, with ultrasonic pretreatment showing better retention than freeze-thaw.

3.6. Total Flavonoid Content (TFC)

Two-way ANOVA showed that freeze-thaw pretreatment was the main factor in significantly decreasing the total flavonoid content ($p < 0.05$). Ultrasonic treatment on its own showed no notable effect; however, when combined with freeze-thaw, the interaction significantly influenced the decline in flavonoid levels of the dried lemons ($p < 0.05$). The total flavonoid content of samples subjected to the 10 min ultrasonic pretreatment without freeze-thawing (0.43±0.01 mg QE/g) was not significantly different from the control samples (0.44±0.00 mg QE/g), indicating that this pretreatment was better at retaining the flavonoid content of dried lemon compared to the other pretreatments (see Table 2), preserving 97.73% of the TFC compared to the control.

The freeze-thaw pretreatment significantly reduced the total flavonoid content of dried lemon samples compared with the control, demonstrating its negative impact on flavonoid retention. Zhang *et al*. (2022) reported that freeze-thaw pretreatment caused a decrease in total flavonoids in the drying of lotus roots compared to untreated samples, with the decrease being related to the drip loss that occurs due to cell damage during the freeze-thaw pretreatment. From the results, the combined freeze-thaw and ultrasonic pretreatments, whether for 10 min (0.34±0.01 mg QE/g) or 20 min (0.35±0.02 mg QE/g), had the lowest total flavonoid content. This shows that the combination of the two pretreatments increased the total loss of flavonoid content.

3.7. Antioxidant Activity

Table 2 summarizes the antioxidant activity of the dried lemons across all pretreatment conditions. Two-way ANOVA showed that freeze-thaw and ultrasonic pretreatments significantly reduced antioxidant activity ($p < 0.05$), although their interaction was not significant. The highest antioxidant activity was observed in the control samples (43.75 ± 1.61%), while the lowest was recorded in those subjected to both freeze-thaw and ultrasonic pretreatments, whether for 10 min (33.80±1.65%) or 20 min (33.91±1.42%). These results are consistent with those reported by Xu *et al.* (2021), where freeze-thaw and ultrasonic pretreatments reduced the antioxidant activity of dried okra.

In this study, the antioxidant trend corresponded with the reductions observed in vitamin C, total phenolics, and flavonoids. The Pearson correlation test results showed a strong, positive, and significant correlation between antioxidant activity and vitamin C ($r = 0.87$), TPC ($r = 0.89$), and TFC ($r = 0.72$). Therefore, the decrease in antioxidant activity in the samples is likely attributed to the reduction of these components caused by the applied pretreatments.

This study presents a novel contribution by systematically evaluating the individual and combined effects of ultrasonic and freeze-thaw pretreatments on lemon drying, addressing a gap in the literature where previous findings on other fruits were inconsistent. As a practical contribution to the food industry, this research identifies the 10 min ultrasonic pretreatment without freeze-thawing as the optimal strategy. This specific approach effectively accelerates drying time and improves physical quality (rehydration and color), while maximizing the retention of essential bioactive compounds such as vitamin C, phenolics, and flavonoids.

4. CONCLUSION

The freeze-thaw pretreatment significantly affected the drying rate, rehydration ratio, total color difference (ΔE), vitamin C content, total phenolic content, total flavonoid content, and antioxidant activity of the dried lemons. The ultrasonic pretreatment significantly influenced the drying rate, rehydration ratio, browning index, total color difference (ΔE), vitamin C content, total phenolic content, and antioxidant activity. Meanwhile, the interaction between the freeze-thaw and ultrasonic pretreatments only had a significant effect on the drying rate and total flavonoid content of the dried lemons. The 10 min ultrasonic pretreatment without freeze-thawing was identified as the optimal approach based on process efficiency and quality retention. This treatment increased the drying rate and rehydration ratio, reduced the browning index and total color difference (ΔE), maintained the total phenolic and total flavonoid contents of the dried lemons, and exhibited the lowest reduction in vitamin C content and antioxidant activity compared to the other pretreatments. Overall, while this pretreatment successfully enhanced the physical quality attributes, it led to a reduction in the nutritional aspects.

AUTHOR CONTRIBUTION

Author	C	M	So	Va	Fo	I	R	D	O	E	Vi	Su	P	Fu
MNA	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓
RH	✓	✓		✓	✓					✓		✓		
LON	✓	✓		✓	✓					✓		✓		

C: Conceptualization	Fo: Formal Analysis	O: Writing - Original Draft	Fu: Funding Acquisition
M: Methodology	I: Investigation	E: Writing - Review & Editing	P: Project Administration
So: Software	D: Data Curation	Vi: Visualization	
Va: Validation	R: Resources	Su: Supervision	

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DECLARATION OF GENERATIVE AI AND AI-ASSISTED TECHNOLOGIES IN THE MANUSCRIPT PREPARATION

Not applicable. The authors did not use any generative AI tools or services in the preparation of this manuscript. All content is entirely the intellectual work of the authors.

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