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Research Paper / Araştırma Makalesi

Effect of Ultrasound Pretreatment on the Retention of Loquat Phenolics and Antioxidants during Tray and Freeze-Drying

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ABSTRACT

In this study, the effect of ultrasound (US) pretreatment (600W for 30 min) prior to tray drying (TD) (46±1°C at 1.10 m/s) and freeze drying (FD) (-58°C at 0.001 mbar) on the quality characteristics of loquat cubes was investigated. US pretreatment in TD demonstrated a significant decrease in drying time (19.67%) and an increase in drying rates (22.23%, $p<0.05$). With US pretreatment, the effective moisture diffusivity increased from 7.83×10^{-9} to 8.92×10^{-9} m^2/s in TD. The highest rehydration capacity (4.47±0.23 g/g) was found in FD samples with US. In comparison to TD and control samples, FD samples had higher phenolic contents. US pretreatment significantly reduced phenolic contents from 1551.19±142.12 to 1197.03±80.51 mg GAE/100g and from 770.12±125.93 to 374.78±71.95 mg GAE/100g for FD and TD, respectively ($p<0.05$). The antioxidant capacity of loquat cubes significantly decreased with TD compared to FD ($p<0.05$). Results indicated that FD is a viable option for drying loquat cubes with a low moisture content, high rehydration capacity, and enhanced phenolic content and antioxidant activity.

Keywords: *Eriobotrya japonica*, Drying rate, Quality, Freeze drying, Rehydration capacity

Ultrases Önişleminin Malta Eriğinin Tepsili ve Dondurarak Kurutulması Sırasında Fenolik ve Antioksidanların Muhafazasına Etkisi

ÖZ

Tepsili kurutma (TK) (46±1°C, 1.10 m/s) ve dondurarak kurutma (DK) (-58°C, 0.001mbar) yöntemleri öncesi uygulanan ultrases (US) (600W, 30 min) tekniğinin kurutulmuş yenidünya (Malta eriği) küplerinin kalitesine etkisi araştırılmıştır. Ultrases önişlem uygulaması ile T kurutma zamanında anlamlı düşme (19.67%) ve kurutma hızında anlamlı yükselme (22.23%, $p<0.05$) belirlenmiştir. Ultrases uygulaması ile TK yönteminde etkin difüzyon katsayısı 7.83×10^{-9}'dan 8.92×10^{-9} m^2/s'a yükselmiştir. En yüksek rehidrasyon kapasitesi 4.47±0.23 g/g ile US uygulaması sonrası DK yöntemiyle kurutulan örneklerde gözlemlenmiştir. DK ile kurutulan örneklerdeki fenolik madde miktarı TK ile kurutulan örneklere oranla yüksek olduğu belirlenmiştir. Ultrases önişlemi fenolik madde miktarını DK ile kurutulan örneklerde 1551.19±142.12'den 1197.03±80.51'e mg GAE/100g, TK ile kurutulan örneklerde de 770.12±125.93'ten 374.78±71.95'e mg GAE/100g azaltmıştır ($p<0.05$). Dondurarak kurutma işlemi ile karşılaştırıldığında, TK işlemi uygulanan yenidünya küplerinin antioksidan kapasiteside anlamlı düşüş gözlemlenmiştir ($p<0.05$). Yapılan çalışmanın sonucuna göre düşük nem oranı, yüksek rehidrasyon kapasitesi, yüksek fenolik madde miktarı ve antioksidan kapasitesi ile yenidünya küplerine DK tekniğinin uygulanabilir bir opsiyon olduğu belirlenmiştir.

Anahtar Kelimeler: *Eriobotrya japonica*, Kurutma hızı, Kalite, Dondurarak kurutma, Rehidrasyon kapasitesi

INTRODUCTION

The loquat fruit (*Eriobotrya japonica* L.) has taken place in people's lives since ancient times with its nutritional and medicinal value [1]. Besides the antibacterial property, this fruit has significant effects on health by fighting against the inflammation, diabetes, and cancer as well as a remedy of pain, allergy. Loquat fruit from the *Rosaceae* family is mostly grown in oval shape, and yellow/orange color [2]. Although the loquat fruit is generally grown in regions with temperate climates in the world, it is mostly grown in China, Japan, India, Pakistan, Cyprus, Egypt, Greece, Tunisia, and Turkey [3]. Loquats grow in subtropical and tropical climates, particularly in regions where citrus is cultivated. With unique taste and smell, loquat is a special tropical fruit containing plenty amounts of carotenoids, phenolic compounds, antioxidants, minerals, and vitamins [4]. Individual phenolic compounds in loquat fruit were identified as 5-caffeoylquinic acid (chlorogenic acid), neochlorogenic acid, hydroxybenzoic acid, 5-*p*-feruloylquinic acid, protocatechuic acid, 4-caffeoylquinic acid, epicatechin, *o*-coumaric acid, ferulic acid, and *p*-coumaric acid [5]. Yet, the fresh loquat fruit with an 90% moisture content can remain intact only 10 days at a temperature range of 20 to 25°C after harvesting due to its unique texture structure [2].

Drying is the most common technique to preserve the perishable produces long time, which is economical in terms of initial investment cost and energy consumption. Tray drying (TD) is widely used method in which the food product is placed on trays and exposed to the drying medium of circulated hot air by the fan [6]. Whereas, conventional drying processes can reduce the valuable bioactive components including vitamins, carotenoids, antioxidants, phenolics, and aroma compounds in fruits and vegetables, as well as their sensory characteristics. As a novel technology, freeze drying (FD) is an alternative technique especially beneficial for fruits and vegetables drying. During the FD, the food product is exposed to low pressure in the frozen state at the cryogenic temperature and then allowed water to pass from the solid to the gas phase. Therefore, the free water in the product is directly sublimated and removed from the food in gaseous form [7]. According to the researcher study, total phenolic content (TPC) and total flavonoid content (TFC) of freeze dried hawthorn fruit extracts were higher than fresh and oven-dried fruit extracts [8]. Besides, Mishra et al. [9] investigated TD and FD techniques to evaluate changes in chemical composition and sensorial quality of loquat slices. After TD, total phenolics, flavonoids, and antioxidant content of loquat slices significantly decreased when compared to freeze dried and fresh samples. With the porous structure, freeze dried loquats had the highest sensory ratings. Conversely, other researchers [10] found higher amounts of hydroxycinnamic acids in dried loquats using convective drying than FD. They also reported increasing temperature from 50 to 70°C in convective drying increased antioxidant activity (ABTS and FRAP assays) of dried loquat samples.

Numerous studies have shown that the various dehydration techniques were successfully used for inactivating the enzymes while maintaining the nutritional content of the food. It is necessary to select appropriate drying technique and processing parameter to minimize these undesirable changes in the sample. It is common practice to use ultrasound (US) in conjunction with other drying technologies. Being that the water transfer is primarily enhanced by alternating the expansion and compression cycles, the mechanical energy produced by the application of power ultrasonic helps to reduce both the internal and external resistances to the mass transfer [11]. Some research studies suggested using US as a pretreatment in drying processes since sonicated samples retained better nutrients than untreated ones [12, 13]. In contrast, other researchers have reported US pretreatment resulted in null effect or severe degradation of bioactive compounds in dried products [14, 15]. The purpose of this study was to assess the effect of US treatment prior to TD or FD methods regarding the quality characteristics of loquat cubes such as phenolic content, antioxidant properties, drying behavior, diffusivity, moisture content, and rehydration capacity.

MATERIALS and METHODS

Sample Preparation

The loquat (*Eriobotrya japonica* L.) cultivar of ‘Ayva Gobek’ was supplied from a retail market in Alanya (Antalya, Turkey). The loquat fruits were stored at 4±1°C and 95% relative humidity. Before drying processes, the loquats were washed, stems and seeds were removed, and then cut into cube-shaped pieces (0.9×0.9×0.9 cm). Fresh loquat was used as a control sample.

Ultrasound Pretreatment

An ultrasonic bath (VWR USC900TH ultrasonic cleaner, VWR Int. Radnor, PA, USA, 100% power: 600 W, 45 kHz) was used. The loquat fruit-to-water ratio was kept constant at 1:4 (w/w, weight basis), and US was used for 30 min. After US treatment, the samples were blotted with adsorbent paper to remove excess solution and then used for tray and freeze-drying processes.

Tray Drying

The loquat samples were dried in a laboratory-scale tray dryer (Armfield UOP8-G, England) operating at a uniform air velocity of 1.10 m/s. Loquat cubes were evenly distributed on trays and drying was conducted at 46±1°C until sample weight remained constant.

Determination of Drying Rates during Tray Drying

Drying rate (DR) was evaluated by using Equations 1, 2 and 3.

$$Xt = \frac{w-dw}{dw}, \quad (1)$$
$$X = Xt - Xe, \quad (2)$$
$$DR = -\left(\frac{dw}{A}\right) * \left(\frac{dx}{dt}\right). \quad (3)$$

where w is weight of wet sample and dw is weight of dry sample; X, Xe, and Xt are moisture content free, equilibrium, at any time (g H_2O/g dm), respectively; A is the surface area in m^2.

The effective diffusivity of samples (D_{eff}) was evaluated using the Fick's second law Equations 4, 5 and 6.

$$MR = \frac{Mt-Me}{Mi-Me}, \quad (4)$$
$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)^2}exp\left[-(2n-1)^2\pi^2\frac{Deff}{4L^2}t\right], \quad (5)$$
$$In(MR) = In\left(\frac{8}{\pi^2}\right) - (\pi^2\frac{Deff}{4L^2}t);\ Slope = \pi^2\frac{Deff}{4L^2}. \quad (6)$$

where MR, and Mi, Me, Mt are moisture ratio and moisture content of initial, equilibrium, at any time, respectively; t, L, and D_{eff} are time (s), thickness of samples (m), and effective diffusivity (m^2/s), respectively. Assumptions: initial moisture distribution is uniform throughout the sample; diffusivity is constant; sample is considered to be equivalent to thin slabs of constant density, size and shape.

Freeze Drying

The loquat samples were dried in a laboratory scale freeze dryer (ALPHA 1-2 LD plus, Osterode am Harz, Germany). The loquat samples were remained in the freezer (-18°C) for 24 h, then freeze dried at -58°C and 0.001 mbar for 24 h.

Moisture Content

The moisture content of the samples was measured by using an infrared moisture device (Shimadzu MOC-63U, Japan) Calculations were done based on dry basis [16].

Determination of Rehydration Capacity

The rehydration capacity (RC) of samples was determined by placing 2 g dry sample in a beaker filled with 50 mL distilled water and left for 24h in water bath (Memmert WNB 22, Schwabach, Germany) at ambient temperature. Then samples were filtered to remove excess water and weighed. The rehydration capacity was calculated using the Eq. 7.

$$Rehydration\ capacity\ (RC) = \frac{Wr}{Wd} \quad (7)$$

where W_r is weight after rehydration in g and W_d is weight of dry sample in g.

Spectrophotometric Analyses

UV-visible spectrophotometer (BioTek Instruments, Winooski, USA) was used for the spectrophotometric analysis. Total phenolic content (TPC) assay was carried out according to the method of Singleton and Rossi [17] by using the Folin-Ciocalteu reagent. The absorbance value was then measured at 760 nm. The calibration curve (y=2.8066x-0.0296) was plotted using gallic acid in the range of 0.01–0.4 mg/mL. The TPC was expressed in milligrams of gallic acid equivalents (GAE) per 100 g of dry weight (dw). The antioxidant capacities were determined by using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay [18] and cupric ion-reducing antioxidant capacity (CUPRAC) assay [19]. Measurements were performed at 450 and 517 nm, respectively. Trolox was used as a standard in the range of 0.01-0.4 mg/mL while calibration curves (y=4.0161x+0.0792 for DPPH, y=0.8475x-0.0139 for CUPRAC) were plotted in the assay. The result was reported as Trolox equivalents (TE) per 100 g of dw.

Statistical Analysis

All assays were carried out in triplicates and results were expressed as mean ± standard deviation. Statistical analysis was performed using JMP software (JMP version 13.0; Cary, NC, USA). Processing conditions were evaluated using one-way analysis of variance (ANOVA) followed by a Tukey's multiple comparison test at the 95% level was performed to evaluate differences between treatments. Coefficient of determination values (R^2) of the spectrophotometric analyses were calculated using Excel (Microsoft, USA) software.

RESULTS and DISCUSSION

Effect of US Pretreatment on Drying Rates during Tray Drying

During the drying process in the tray dryer, weight reduction of loquat cubes at certain time intervals was given in Figure 1. Regardless of US pretreatment, the weight of the loquat samples decreased exponentially with time. Whereas, application of US creates cavitation and channels obtained by the disruption of cells after cavitation is beneficial for transferring water [20]. In the current study, usage of US as a pretreatment increased the water loss of loquat samples and thereby decreased drying time in TD process. The drying time was found to be 270 min for the samples with TD only, whereas, after the US pretreatment it was 210 min, indicating application of US prior to TD reduced the drying time significantly by 22.23%.

The moisture content of loquat samples with respect to time are shown in Figure 2. Mostly studies reported that drying times were significantly reduced after US pretreatment compared to untreated samples [21, 22]. Because of the lower pressure above the sample, US pretreatment accelerates the drying rate and encourages water loss. The influence of ultrasound on drying time is caused by asymmetric implosions of cavitation bubbles adjacent to a solid surface, which form microjets in the direction of the surface and can affect mass transfer [23]. Rani and Tripathy [24] indicated that the samples lost moisture throughout the pretreatment process, as a result moisture content at the beginning of the drying process was different for the pretreated and untreated samples. In addition, samples prepared with US pretreatment had faster drying rates, and thereby reduced drying time, compared to untreated ones. Furthermore, US pretreatment was found to be effective on diffusion coefficients, which is calculated for TD and US+TD samples as $7.83x10^{-9}$ and $8.92x10^{-9}$ m^2/s, respectively, with $R^2>0.95$. In consistent with our study, US pretreatment raised effective water diffusivity value from $2.13x10^{-10}$ m^2/s to $2.88x10^{-10}$ m^2/s for the hot air drying of yellow cassava [25], from 1.739 $x10^{-10}$ m^2/s to 2.052 $x10^{-10}$ m^2/s for microwave-convective drying of basil [15], and from 6.50 $x10^{-10}$ m^2/s to 2.11 $x10^{-9}$ m^2/s for hot air drying of nectarine slices [26]. It is noteworthy that effective water diffusivity value may vary due to the products processed under different conditions including drying temperature, moisture content, and physical or chemical pretreatments as well as sample type, composition, and shape.

Changes in drying rate of loquat cubes based on moisture content is presented in Figure 3. According to these findings, drying occurred in the falling rate period rather than the constant rate period. Since water mobility was impacted by the energy that ultrasound waves transferred into the solid. The average drying rate of the TD samples was found to be 0.0356 g $H_2O/min.m^2$ while it was 0.0426 g $H_2O/min.m^2$ for the US+TD samples. Therefore, the drying rate increased by 19.67% in samples treated by US prior to TD. Similarly, other researchers found pretreatment of persimmon with high-intensity US increased drying rate in hot air drying at 50°C [27]. Further study suggested that US accelerates the drying rate of heat-sensitive fruits like strawberry [28].

Moisture Contents and Rehydration Capacities

As presented in Table 1, the moisture content of the fresh cube-shaped loquat pieces reduced from 87.7±0.1% to 30.43±0.18% for TD and 30.15±0.03 for US+TD processes based on dry basis, indicating US pretreatment had no significant effect on moisture content of TD samples ($p>0.05$). These results were consistent with a research study by [29] who found no significant difference between the moisture content of dried persimmons after application of US prior to hot air drying ($p>0.05$). When compared to TD, the FD was more effective process on loquat pieces with a significant decrease in moisture content to 15.77±0.12% ($p<0.05$). A similar trend was found in evaluation of loquat drying methods and FD was the most effective method compared to convective and vacuum-microwave drying [10]. In addition, throughout the treatments, the lowest moisture content (13.17±0.09%) was determined in loquat samples which were treated with the US prior to FD ($p<0.05$).

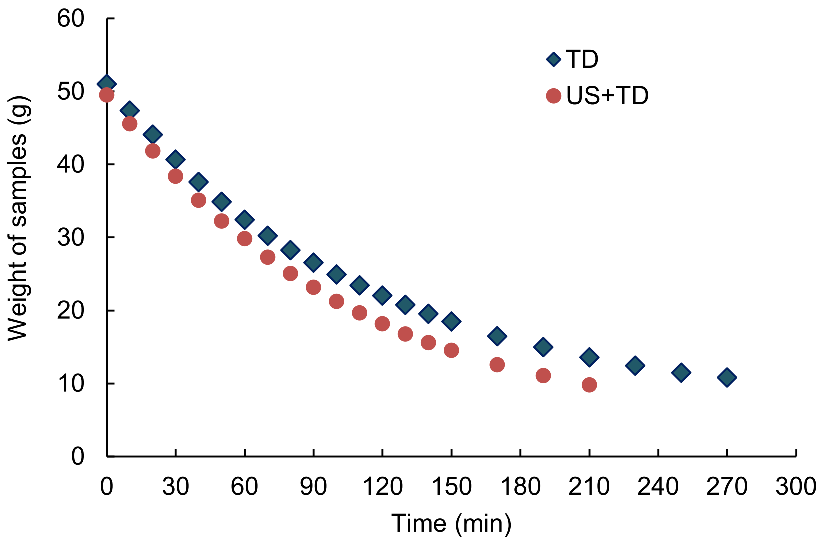

Figure 1. Effect of ultrasound (US) pretreatment on the weight loss of loquat cubes during tray drying (TD)

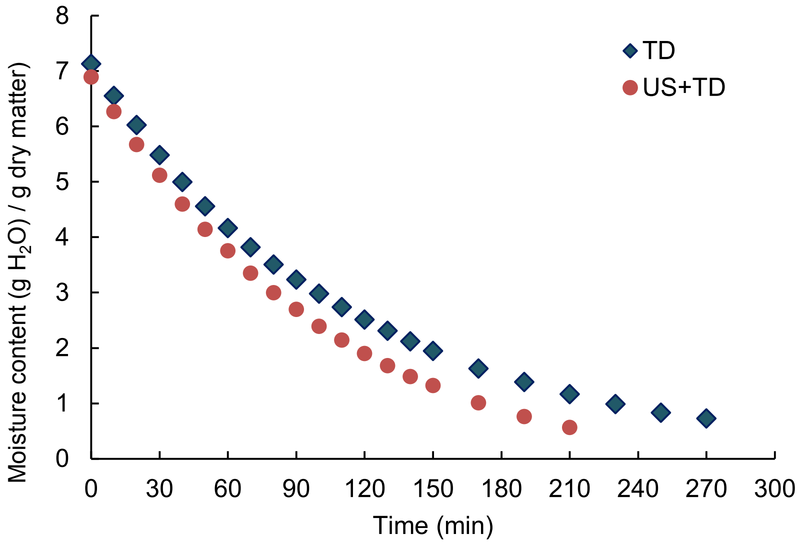

Figure 2. Effect of ultrasound (US) pretreatment on the moisture contents of loquat cubes during tray drying (TD)

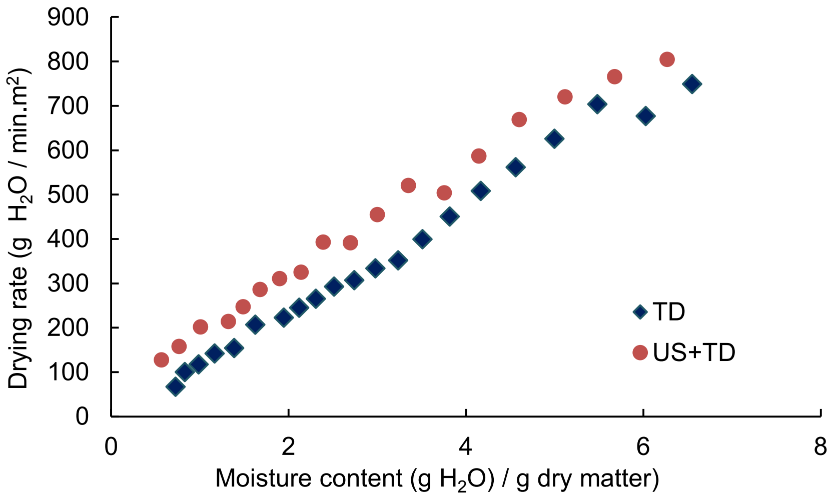

Figure 3. Effect of ultrasound (US) pretreatment on the drying rates of loquat cubes during tray drying (TD)

With regard the value of moisture content, a correlation was found with rehydration capacity of dried loquat cubes. With the lowest moisture content, the highest rehydration capacity ($p<0.05$) value was found to be 4.47±0.23 in US+FD sample (Table 2). Application of US prior to drying processes affected the rehydration capacity of loquat cubes, which was slightly increased in the TD samples ($p>0.05$); however, significant increment from 3.13±0.26 to 4.30±0.23 g/g was observed in FD ones ($p<0.05$). According to the previous studies, button mushrooms had been treated with US prior to TD had higher rehydration capabilities than the untreated samples, as well as for the cauliflower and brussels sprout samples [21]. In other research study, they found applying osmotic dehydration and ultrasonic pretreatment in a sucrose solution before drying dramatically increased the rehydration capacities of persimmon compared to the untreated group [29]. Overall, application of US as a pretreatment enhanced the rehydration capacities of fruits and vegetables.

Table 1. Moisture content and rehydration capacity of loquat samples

Treatments[1]	Moisture Content (%)	Rehydration Capacity (g/g)
C	87.70±0.10 [a]	
TD	30.43±0.18 [b]	2.46±0.19 [c]
US+TD	30.15±0.03 [b]	2.65±0.18 [bc]
FD	15.77±0.12 [c]	3.13±0.26 [b]
US+FD	13.17±0.09 [d]	4.30±0.23 [a]

[1] Treatments are indicated as C: Control (Fresh fruit), TD: Tray drying, US+TD: Ultrasound treatment prior to tray drying, FD: Freeze drying, and US+FD: Ultrasound treatment prior to freeze drying. [2] Data in the same column followed by same letter are significantly different ($p<0.05$).

Table 2. Phenolic contents and antioxidant capacities of loquat extracts

Treatments[1]	Total Phenolic Content (mg GAE/100 g dw)	Antioxidant Capacity (mg TE/100 g dw)	
		CUPRAC	DPPH
C	878.75±97.40 [c]	3094.35±229.05 [c]	807.97±40.33 [a]
TD	770.12±125.93 [c]	1716.74±128.92 [d]	410.84±40.28 [d]
FD	1551.19±142.12 [a]	4749.42±545.78 [a]	640.46±30.11 [b]
US+TD	374.78±71.95 [d]	1080.32±204.24 [d]	296.02±44.31 [e]
US+FD	1197.03±80.51 [b]	3753.50±474.46 [b]	579.88±18.71 [c]

[1] Treatments are indicated as Control (C): Fresh fruit, TD: Tray drying, US+TD: Ultrasound treatment prior to tray drying, FD: Freeze drying, and US+FD: Ultrasound treatment prior to freeze drying. [2] Data in the same column followed by same letter are significantly different (p<0.05).

Phenolic Contents and Antioxidant Capacities

The effect of US pretreatment on phenolic compounds of TD and FD loquat cubes is presented in Table 2. The highest phenolics was found to be 1551.2±142.1 mg GAE/100 g in FD loquat samples through other treatments (p<0.05). Ultrasound pretreatment significantly reduced total phenolics (p<0.05) from 15519.2±142.12 mg GAE/100 g to 1197.03±80.51 mg GAE/100 g and from 770.12±125.93 mg GAE/100 g to 374.78±71.95 mg GAE/100 g for FD and TD, respectively, which might be due to loss of nutrients during draining of water out of fruits. According to a recent study [30], significant decrease was observed in total phenolics of apples, which were treated with US prior to convective drying; however, no significant difference was determined in FD samples when pretreated with US. Furthermore, Bozkir et al. [29] found significant loss in phenolics of US pretreated persimmon slices after convective drying. When compared to control samples, there was no significant difference observed in phenolic content of TD loquat cubes; however, phenolics were much higher in FD samples (p<0.05). Moreover, phenolic content of FD loquat cubes was significantly higher than TD samples because of thermal degradation during the TD process. Thereby, the nutrient loss was induced by the water transfer that occurred during the application of this treatment [31]. Researchers have reported the decrease of total phenolic contents of mandarins, sour cherries, and blueberries with increasing temperature throughout the drying process [6, 32]. Additionally, Amami et al. [33] indicated that pretreatment with osmo-sonication also resulted in significant losses in total phenolic content of hot air-dried strawberries compared to the untreated samples. Besides the TD and FD conditions, acoustic application duration might affect the losses of phenolics from the loquat cubes. It is important to point out that long sonication periods ended up with significant reduction of phenolics in quince slices [34].

The effect of US treatment prior to TD and FD on antioxidant capacity of loquat cubes are shown in Table 2. Both DPPH and CUPRAC assays revealed that TD decreased the antioxidant capacity of loquat cubes significantly compared to FD and fresh samples. The reason for this situation can be interpreted as prolonged drying exposure of samples during TD may degrade certain phenolic compounds and thereby resulting in a decrease in antioxidant activity. In consistent to our study, López-Lluch et al. [10] have found higher antioxidant activity in dried loquats using FD than convective drying. According to the results of CUPRAC assay, the highest antioxidant capacity was observed in FD loquat samples when compared to other treatments and untreated sample (p<0.05). In addition, significant reduction was determined in FD loquat cubes when pretreated with US; however, their antioxidant capacity was higher than untreated ones (p<0.05). Similar result was obtained in application of US prior to TD, which might be due to loss of antioxidants in water bath. Other researchers have also reported decrease in antioxidants of blackberry after application of US in a water bath [35]. Conversely, Šic Žlabur et al. [36] found positive impacts of US pretreatment on vacuum dried honeyberry fruits. In fact, US frequency, power, and pretreatment time, as well as product structure, are effective parameters on bioactive compounds.

CONCLUSION

Application of US treatment prior to TD enhanced the drying rate and reduced drying time since implosions of cavitation bubbles adjacent to a solid surface forms microjets and then affects the mass transfer. Rehydration capacity of loquat cubes increased with US pretreatment for both TD and FD. Higher phenolic content was determined in FD and US+FD loquat cubes than TD, US+TD and fresh samples. Based in CUPRAC and DPPH assays, antioxidant activity of loquat cubes were higher in FD and US+FD when compared to TD and US+TD samples. According to the results of this study, the important point is that applying US before drying processes may increase the demand on dried fruits by accelerating drying rate thereby shortening the processing time and enhancing the quality. These methods can be evaluated on regionally available subtropical crops to produce value added products.

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