

Drying characteristics and quality attributes of apple slices dried by a non-thermal ultrasonic contact drying method

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ABSTRACT

Drying is one of the most prevalent methods to reduce water activity and preserve foods. However, it is also the most energy-intensive food processing unit operation. Although a number of drying methods have been proposed and tested for the purpose of achieving a time- and energy-efficient drying process, almost all current drying methods still rely on thermal energy to remove moisture from the product. In this study, a novel use of power ultrasound was explored for drying of apple slices without the application of heat. The non-thermal ultrasonic contact drying (US-CD) was performed in the presence of an air stream (26–40 °C) flowing over product surface to remove mist or vapor produced by the ultrasound treatment. The effects of the non-thermal US-CD, hot-air drying (HAD), and freeze drying (FD) on the changes in rehydration ratio, pH, titratable acidity, water activity, color, glass transition temperature, texture, antioxidant capacity, total phenols, and microstructures of the samples were evaluated. The moisture content of the apple slices reached below 5% (w.b.) after 75–80 min of US-CD, which was about 45% less than that of the HAD method. The antioxidant capacity and total phenol contents of the US-CD samples were significantly higher than that of the AD samples. The non-thermal ultrasonic contact drying is a promising method which has the potential to significantly reduce drying time and improve product quality.

1. Introduction

Drying, as a liquid–solid separation process, is one of the most widely used methods for reducing the moisture content of porous materials (often biopolymers) so as to reduce the costs of packaging, transportation, storage, and preservation [1]. It commonly involves removal of moisture via a simultaneous mass and heat transfer process [2]. Drying is also considered to be the most energy-intensive unit operation due to the need of supplying significant amount of thermal energy to remove water via a phase change (evaporation and/or sublimation) [3]. Drying consumes about 12% of the total energy used in the manufacturing industries in the U.S., and a major part of this energy is used for drying of food, agriculture, and forest materials [4]. Since thermal energy is required in a drying process, plus the fact that water removal in the falling rate period is difficult, drying is featured by a long exposure of the product to elevated temperatures. Therefore, drying is also a process known to degrade product quality, especially nutritional quality for foods.

Due to relatively low cost and simple applicability to various

products, drying with hot air has become the most commonly utilized drying method [5]. However, this drying method has many disadvantages, such as significant quality degradation, long drying time, and low energy efficiency [6]. On the other hand, freeze drying (FD) is considered to be a superior drying method for food and pharmaceutical products, due to its ability to prevent shrinkage, retain nutrients and bioavailability, taste, aroma and flavor of the final product. However, FD is an expensive and slow dehydration method that limits its applications. The demand for high quality food has led to the development of a number of relatively new drying methods, such as vacuum-microwave drying [7], Refractance Window drying [8], and Infidri™ drying (or Radiant-Zone drying) [9], each has its own unique advantages and limitations. These new methods produce products with quality comparable to FD, but at a lower cost than FD. Nevertheless, in all of the current drying methods, thermal energy has to be applied to remove the moisture, which lowers the activation energy of quality-degrading reactions. Therefore, there is a need to develop innovative drying methods or novel energy forms for drying food and other heat sensitive products.

In recent years, power ultrasound (20–100 kHz) has recently been

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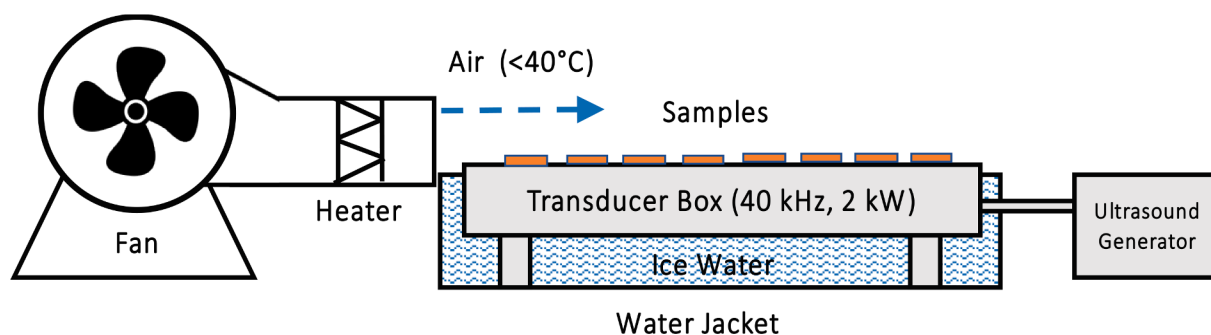


Fig. 1. Non-thermal ultrasonic contact drying setup.

employed for enhancing drying processes [10–12]. The majority of the published works has focused on using ultrasound as a pre-treatment for another drying method (HAD or FD), using airborne ultrasound to enhance HAD that is mostly a surface treatment, or applying sonication in an osmotic dehydration process to reduce resistance to mass transfer [13]. For pretreatment applications, acoustic cavitation produced by ultrasound is reported to increase the permeability of plant and animal cell membranes, leading to enhanced transport.

In this study, a novel non-thermal ultrasound contact drying (US-CD) method was proposed and tested for drying of selected plant foods, e.g. Gala apples. An ultrasonic transducer box (400 × 400 × 90 mm) containing 10 piezoelectric transducers (40 kHz) with a rating power of 1 kW was used to dry Gala apple slices. Physical and chemical quality attributes, including rehydration ratio, pH, titratable acidity, a_w , color, glass transition temperature, texture, antioxidant capacity, total phenols, and microstructures of the samples dried with US-CD were evaluated, in comparison with those dried with HAD and FD methods.

2. Materials and methods

2.1. Sample preparation

Fresh Gala (*Malus × domestica*, Borkh) apples were purchased from a local market in Urbana, IL, USA. The apples were stored at 4 ± 2 °C to keep them fresh until they were used in the experiments. The fresh apples had an initial moisture content in the range of 83.5–86.5% (w.b.), as determined by a vacuum oven method [14]. For each experiment, apples were washed with distilled water, peeled by a stainless-steel knife, and sliced using an automatic food slicer (FS-9001A, Mlter Co., Shenzhen, China) to 2 mm thickness. The average diameter of the apple slices was 37 mm with an average weight of 1.8 g/slice. The drying experiments were performed until the final moisture content of the samples reached about 5% (w.b.).

2.2. Drying experiments

Drying experiments were conducted according to an American Society of Agricultural and Biological Engineers standard (ASABE S448.1). Specifically, the drying was performed after the drying system has reached constant conditions, and the samples were clean and damage free. The apple slices were dried with a non-thermal ultrasonic dryer (US-CD), a single-channel hot air dryer (HAD), and a freeze dryer (FD). The performance of the US-CD was compared with that of the HAD and FD methods.

The non-thermal US-CD of apple slices was performed with a custom-designed system as shown in Fig. 1. The apple slices were placed on the top surface of an ultrasonic transducer box (1 kW, 400 (L) mm × 400 (W) mm × 90 (H) mm) that vibrated at 40 kHz to facilitate vibrational moisture removal. Ambient air was heated by a heater (Intertek Inc., Model 1DKX3, 8900, China) and allowed to flow (4.1–4.9 m/s) parallel to the top surfaces of the apple slices on the transducer box by a

centrifugal air blower (Ebmpapst, Farmington, Connecticut, USA) to carry away mist or moisture coming out of the apple slices. The ambient air with a relative humidity of 18.5% was heated to 26–40 °C to maintain non-thermal conditions. The output section of the blower was aligned with the transducer box to provide a uniform air velocity profile over the transducer box surface. The transducer box was submerged in a custom-designed water jacket with water level about 0.5 in. to its top surface. The water jacket was filled with cold water with ice cubes (~4–5 °C) to lower the surface temperature of the transducer box during US-CD drying.

For hot air drying, a custom designed single-channel hot air dryer unit was used. The hot-air dryer consisted of six sections to maximize the efficiency of the drying process. These six sections included a dehumidifier (Frigidaire, Charlotte, North Carolina, USA) to lower the dryer inlet air humidity for low humidity drying; a centrifugal air blower (Ebmpapst, Farmington, Connecticut, USA) to draw air into the dryer system; an ultrasonic humidifier chamber, which included a 20-mm ultrasonic transducer (HM 2412, Honda Inc., Japan) installed in a custom-made pan to increase air humidity for high humidity drying; two Omegaflux heaters (Omega engineering, Stamford, Connecticut, USA) to provide thermal energy; a drying section including a square or round sample holder (36" × 5" × 5") and a temperature-humidity sensor (Omega engineering, Stamford, Connecticut, USA) to monitor the humidity and temperature of intake air, and a load cell to monitor sample weigh changes. To control the air velocity, a Fantech IR series iris damper (System air, Sweden) was mounted to the fan, and the air velocity was estimated by the following equation: $q = k^* \sqrt{\Delta P}$, where $q = \frac{f^3}{min}$ or CFM.

Hot-air drying of apple slices (in total around 5–6 g) with similar sizes was performed at 60 ± 1 °C, 18.5% relative humidity, and 4.6–5 m/s air velocity. For freeze drying, sliced samples (30 g) were treated with liquid nitrogen to obtain frozen apples. Then frozen samples were stored at –40 °C for 16–18 h before placing in a freeze dryer (Harvest Right Inc., Utah, USA). The apple slices were freeze-dried at a heating plate temperature of 46 °C for 24 h. At the end of the drying, the freeze-dried samples were sealed in Ziploc bags and stored in a desiccator until they were used for analyses.

2.3. Moisture content and drying rate (DR)

Moisture content (wet basis) was determined using the vacuum oven method at 70 °C for 24 h [14]. The average moisture content of three replicates were calculated and reported.

The drying rate at time t can be calculated using Eq. (1).

$$DR = \frac{M_{t+dt} - M_t}{dt} \quad (1)$$

where M_t = moisture content at time t (g water/g dry solids), M_{t+dt} = moisture content at $t + dt$ (g water/g dry solids), and t = drying time (h).

2.4. Color measurement

Changes in color of the fresh and dried apple slices were monitored with a reflectance colorimeter (LabScan XE, Hunter Associates Laboratories, Inc., Reston, VA, USA) based on the CIE L^* , a^* , and b^* color coordinates. The L^* indicates brightness/darkness index (0–100/black to white), a^* indicates redness/greenness ('+' values for red, '-' values for green), and b^* indicates yellowness/blueness ('+' values for yellow, '-' values for blue). Before measurement, the colorimeter was calibrated using a white and a black ceramic plate. A slice of sample was placed in a 35-mm plastic dish (Corning, NY, USA), and three-color readings (L^* , a^* , and b^* values) per slice were taken at room temperature. Color of four individual apple slices were measured for each treatment and the average L^* , a^* , and b^* values were reported. The L^* , a^* and b^* values were used to calculate the browning index (BI), a parameter to measure the degree of brown color formation in the apple slices. It is also an important parameter indicating if enzymatic and/or non-enzymatic browning reactions have taken place [15]. The BI was calculated by Eqs. (2) and (3) [16]

$$BI = \frac{100(x - 0.31)}{0.17} \quad (2)$$

$$x = \frac{a^* + 1.75L}{5.645L + a^* - 3.012b^*} \quad (3)$$

where L^* = brightness/darkness; a^* = redness/greenness; and b^* = yellowness/blueness. Total color change (ΔE) represents the level of total color alteration between initial and final samples. Eq. (4) was used to calculate the total color change (ΔE) of the apple slices [16–19]

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (4)$$

where ΔL^* , Δa^* and Δb^* were the difference of individual L^* , a^* , b^* color readings from control sample.

The hue angle used to specify the color in food product was calculated by Eq. (5) [20].

$$H^\circ = \tan^{-1} \left(\frac{b^*}{a^*} \right) \quad (5)$$

Chroma is an index indicating the intensity or purity of the hue and a degree of color change from gray to pure chromatic colors [21]. The chroma values of the apple slices were calculated by Eq. (6) [20].

$$C = \sqrt{a^{*2} + b^{*2}} \quad (6)$$

2.5. Rehydration ratio

The rehydration ratio of the dried apples was estimated according to the method of Jambrak et al. [22] with some modifications. The dried slices (5–5.2 g) were weighted and placed in a beaker; then 100 mL distilled water was added and covered by a lid allowing to stand for 10 min at room temperature. After that, the sample was poured into a strainer, allowed to drain for 60 s, and weighted by an electric balance with an accuracy of ± 0.0001 g (Ohaus Corp., AP110S, Switzerland). Three replications were used for each experiment. The rehydration ratio of the samples was then calculated by

$$\text{Rehydration ratio} = \frac{D_2}{D_1} \quad (7)$$

where D_2 = weight of rehydrated product and D_1 = weight of dehydrated product.

2.6. Titratable acidity and pH

The samples were powdered, and 0.5 g of the powder was mixed with

20 mL distilled water. The mixtures were equilibrated at 20 °C for an hour. For titratable acidity measurement, the prepared mixtures were titrated with 0.1 M NaOH solution to pH 8.1 using an Accumet Research AR15 pH meter (Fisher Scientific, USA) and the results were calculated as grams of malic acid per 100 g of dry sample [14]. The pH of samples also was determined using the Accumet pH meter and all experiments were performed in triplicates.

2.7. Water activity (a_w) and bulk density

Water activity of the apple samples was measured in triplicates using an Aqualab 4TE (Decagon Devices Inc., Pullman, WA, USA) chilled dew point mirror water activity meter with precision of 0.003 a_w . During measurement, the apple samples were put in a small sample cup filling it half full and placed into the sample chamber (26.7 cm $\times$ 17.8 cm $\times$ 12.7 cm), then the readings were obtained in about 5 min. The bulk density of samples was defined as the weight of dry samples (g) over total volume (mL) of the dried apple slices. The total volume of the dried samples was measured in triplicates using the glass bead displacement method [23].

2.8. Glass transition temperature (T_g)

TA Instruments Q2000 Differential Scanning Calorimetry (DSC) was used to measure the glass transition temperature of the apple samples. Dried samples were powdered and around 10–12 mg powders were placed in T_{zero} hermetically sealed aluminum pans (TA Instruments, T180628, Switzerland). The hermetically sealed aluminum pans were placed in a DSC chamber and cooled to -50 °C at 10 °C/min. The samples were equilibrated at -50 °C for 1 min and scanned from -50 to 80 °C at a rate of 10 °C/min to determine the thermal behavior of the dried apples. An empty hermetically sealed aluminum pan was used as reference (air). Nitrogen gas (50 mL/min) was used as purge gas. The obtained data were analyzed by using TA Universal Analysis software (TA Instruments, New Castle, DE, USA) to determine the glass transition temperatures (onset, midpoint and offset) and changes in specific heat capacity at T_g (ΔC_p). The glass transition temperature (T_g) was reported as the middle temperature in heat flow versus temperature the curves [24].

2.9. Total phenolic content and DPPH free radical scavenging activity

Sample extraction preparation: To prepare the sample extracts, 1 g of grounded dried and fresh apple slices were weighted, placed in a test tube, and mixed with a total of 20 mL of 80% aqueous methanol (v/v) (acidified with 0.1% HCl) (Sigma Chemical, St. Louis, Missouri, USA) for 2 min using a magnetic stirrer. Afterwards, the extracts were shaken at 150 rpm with a benchtop incubator shaker for 2 h at room temperature (New Brunswick Scientific I-24, Eppendorf, Germany), and centrifuged at 3000g for 15 min. The supernatants were used for the analysis of total phenolic content and antioxidant capacity following the method of [25].

Total phenol content was determined in triplicates from the reduction of Folin-Ciocalteu reagent (Sigma Chemical, St. Louis, Missouri, USA) by phenolic compounds, with a formation of a blue colored complex. The extract (0.5 mL) was mixed with 1 mL Folin-Ciocalteu reagent and 7.5 mL distilled water and vortexed. After 3 min of incubation at room temperature, 1 mL of aqueous sodium carbonate solution was added, tubes were vortexed, and the total phenolic content was determined after 90 min of incubation at room temperature. The absorbance of the blue colored mixtures was measured at 765 nm using a spectrophotometer (Lambda 1050 UV/VIS/NIR Spectrometer, PerkinElmer, Waltham, MA, USA) using acidified methanol as a blank. The measurements were quantified with respect to the standard curve of gallic acid (Sigma Chemical, St. Louis, Missouri, USA). The results were recorded as gallic acid equivalents (GAE), mg/100 g of dry mass. All experiments were performed in triplicates.

The 1,1-diphenyl-2-picrylhydrazyl radical (DPPH•) scavenging

Table 1

Moisture contents, water activities, texture values, rehydration ratios and bulk densities of fresh apple and samples dried with different methods.

Drying Methods	Moisture Content (% w.b)	Water Activity (a_w)	Texture		Rehydration Ratio	Bulk Density (kg/m^3)
			Hardness (Force (N))	Number of Peaks		
Fresh	85.90	0.988 ^a	NP	NP	NP	NP
Freeze Drying	3.71	0.316 ^b	8.65 ^c	18.00 ^a	3.27 ^a	350 ^b
US-CD Drying	5.09	0.386 ^c	16.86 ^b	3.10 ^b	2.39 ^b	420 ^b
Hot Air Drying	4.54	0.345 ^b	20.44 ^a	1.70 ^b	1.59 ^c	610 ^a

^{a-c} a_w , texture values, rehydration ratios and bulk densities means between treatments with the same letter in each sample are not significantly different ($p < 0.05$). NP, not performed.

capacity was performed in triplicate based on the method of Ajitha et al. [26] with slight modifications to measure the antioxidant capacity. Dried and fresh apple sample extract solution (0.1 mL) and freshly prepared 3.9 mL of 0.06 mM 2,2-Diphenyl-1-picrylhydrazyl (DPPH) radical solution (Sigma-Aldrich, MO, USA) in methanol were mixed and vortexed for 15 s. Afterwards, the mixtures were incubated in the dark for 30 min. After incubation, the absorbance of the solution was measured at 517 nm with the Lambda spectrophotometer. The absorbance of DPPH radical without sample was used as the control. The DPPH free radical scavenging rate of apple samples was calculated by the following equation:

$$\text{DPPH free radical scavenging rate; (\%)} = \frac{A_c - A_t}{A_c} \times 100 \quad (8)$$

where A_c is the absorbance of control and A_t is the absorbance of dried samples.

2.10. Microstructural characteristics of fresh and dried apples

2.10.1. Environmental scanning electron microscopy (ESEM)

The surface micro-images of the fresh and dried apple slice samples (2 mm thickness) were taken using an environmental scanning electron microscope (FEI Quanta FEG 450 ESEM, Hillsboro, OR, USA) operating at low vacuum mode (0.1–1.5 torr). The samples were mounted onto an ESEM aluminum stub using a carbon adhesive tape and micro-photographed at different magnification ranges in 20 kV acceleration voltage [27]. Multiple images were taken from each sample. Collected ESME images were also used to analyze the pore size distribution over

the surface of fresh and dried apple samples. The darker spaces in the collected images present the size of pores where the fluid could flow through, while the lighter regions signify the solid cells and boundaries. MATLAB® is used to perform the image analysis to distinguish the pixels with dark and light colors for providing the area of pores and cells. Since more shrinkage will take place on the surface layer of the apple tissue during drying, the image analysis using surface SEM images will provide a worst-case estimation of the microstructural changes of the apple tissue.

2.10.2. Micro CT imaging

The apple slices were wrapped in a polymer paper and placed inside a plastic tube to prevent the dehydration, which may cause undesired movement of the sample in the CT scan chamber. The samples were scanned using an Xradia Micro-CT (MicroXCT-200, Rockville, CA) system at the voltage of 40 kV. The apple samples were rotated from -180° to 180° to obtain images over 360° angle with a scanning time of 120 min [28]. After scanning, the microstructure of apple slices was quantified by applying a range of 2D and 3D algorithms which resulted in morphometric and geometric 3D visuals of the microstructures. Image processing of the 2D images was performed with a XMReconstructor software (Xradia Software, Xradia Inc., Pleasanton, CA, USA). The 3D images and videos of the sample were taken using a TXM3DViewer software (Xradia Software, Xradia Inc., Pleasanton, CA, USA).

2.10.3. Analysis of 3D optical surface profile

3D surface profiles of the dried apple slices were captured by a high-resolution 3D optical profiler (VK-X1000, Keyence, Japan) at voltage of

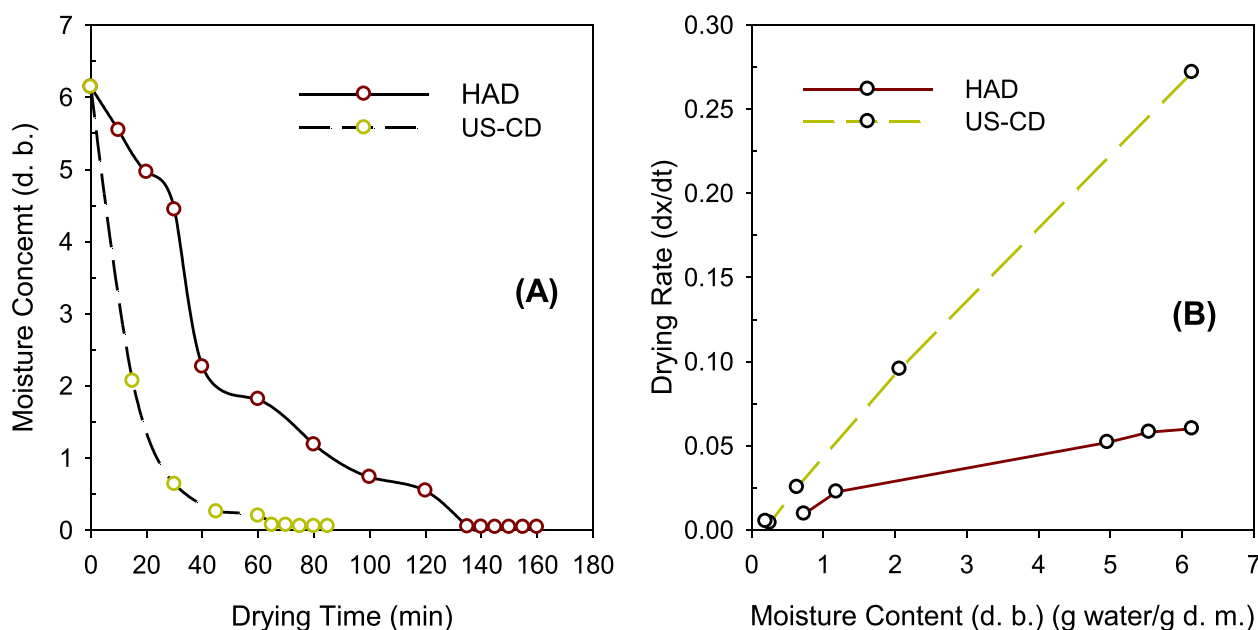


Fig. 2. Drying curves (A) and drying rate curves (B) of HAD (60 °C, 18.5% RH, 4.6–5 m/s air velocity) and (B) US-CD (<40 °C, 18.5 RH, 40 kHz, 4.1–4.9 m/s air velocity) samples.

Table 2

Color readings of the fresh and the dried apple slices.

Drying Methods	Color			Total Color Change (ΔE)	Browning Index (BI)	Hue (H°)	Chroma (C)
	L*	a*	b*				
Fresh	62.79 ^a	1.59 ^c	21.41 ^c	NA	NA	85.78 ^a	21.47 ^c
Freeze Drying	62.76 ^a	2.24 ^c	23.65 ^b	2.94 ^c	48.71 ^c	84.59 ^a	23.76 ^b
US-CD Drying	62.56 ^a	4.55 ^b	28.56 ^a	7.83 ^b	64.56 ^b	80.95 ^b	28.92 ^a
Hot Air Drying	51.86 ^b	8.55 ^a	29.21 ^a	15.15 ^a	91.540	73.68 ^c	30.44 ^a

^{a-c}L*, a*, b*, ΔE , BI, H° , and C means between treatments with the same letter in each sample are not significantly different ($p < 0.05$).

0.5 V. During measurement, the laser scanning microscope scanned the sample surfaces and collected optical images and surface profile data with nanometer-level resolution. The images were obtained by analyzing the intensity of the returned laser light relative to the z-position of the laser due to the combination of conventional white light with a laser light source. Using the lowest magnification (5 $\times$), the sample was placed at center of the stage manually. The focus of microscope was adjusted using a coarse then the measurement was performed and analyzed using a MultiFileAnalyzer software (VK-H2X, Keyence, Japan).

2.11. Texture analysis

Textural properties of the dried apple samples were determined by a compression test using a TA-HDPlus Texture Analyser (Stable Micro Systems Ltd., Godalming, UK) equipped with a 30-kg load cell. A 4-mm diameter probe (TA-54, Stable Micro Systems Ltd., Godalming, UK) and a Texture Exponent 32 software (Stable Micro Systems Ltd., Godalming, UK) were used for the analysis. Pre-test, test, post-test speeds and strain rate were adjusted to 3, 1, 10 mm/s and 90%, respectively [29]. Ten replications were performed for each treatment and the average of the ten readings were reported.

2.12. Statistical analysis

The results were analyzed by analysis of variance using the General Linear Models (PROC GLM) procedure in SAS (version 9.1, SAS Institute, Inc., Cary, North Carolina, USA). Differences among the mean values were obtained by Fisher's Least Significant Difference (LSD) test at $\alpha = 0.05$.

3. Results and discussion

3.1. Moisture content, water activity and drying rate

Moisture content (wet basis) and water activity (a_w) of the apple slices dried with freeze drying (FD), hot air drying (HAD), and ultrasound contact drying (US-CD) are shown in Table 1. The moisture content and a_w of the fresh apple slices were 85.9% (w.b.) and 0.988, respectively. The HAD and US-CD were performed until changes in the weight of the samples became constant. During drying, the moisture content and a_w decreased rapidly. The US-CD samples lost ~87.2% of their initial weight. The final moisture content (w.b.) for the FD, HAD, US-CD was 3.71%, 4.54%, and 5.09%, respectively. The lowest a_w (0.316) was found in the FD samples. Similar results were observed in the study of Schulze et al. [23] for their freeze dried and hot-air dried apple slices.

Fig. 2(A) shows drying curves of the apple slices dried with HAD and US-CD. While the US-CD drying at 26–40 °C obtained a final moisture content of 5.09% (w.b) in 75 min, it took 135 min for the HAD at 60 °C samples to reach a final moisture content to 4.54% (w.b.). The drying time for the US-CD was about 45% less than that of the HAD.

The linear vibrational forces applied by ultrasound to the apple tissues might be the reason behind improved drying performance. Hypotheses include those in the previous studies on airborne ultrasound or

pretreatment applications [30,31], which postulated that cavitation activities lead to changes on the surface of the samples with formation of micro-channels, which enhanced the mass transfer due to an increase in the number of pores and their sizes. Through these channels, the sample lost water to the gap between the sample and transducer surface. The water released from the sample was then atomized on the transducer surface immediately [32]. In the ultrasonic contact drying performed in this study, the apple tissue was in direct contact with the vibrating transducer surface. Misting of water droplets was observed in the very beginning of the drying. Afterwards, the drying enhancement should still be linked to the vibrational forces applied on the tissue, which may introduce the sponge effect as reported by Miano et al. [33] and Fan et al. [34], as well as lowering of activation energy for water molecules to escape from the biopolymer matrix of apple tissues. The exact mechanism for enhance drying by ultrasound at near room temperature remains to be discovered.

The drying rate curves of the apple slices dried with US-CD and HAD are shown in Fig. 2(B). No constant rate period can be observed on the drying curves of both drying methods, a phenomenon often seen in the drying of fruits and vegetables. During the falling rate period, the moisture movement in the sample is controlled by molecular diffusion [6]. The drying rate of the US-CD samples was much higher than that of the HAD samples, thus the US-CD was able to speed up drying in the falling rate period. This is an advantage especially for heat sensitive products since prolonged drying in the falling rate period, where the moisture is low and thus evaporative cooling is diminished, could damage the heat sensitive component, such as nutrients in the product.

3.2. Color

Color is one of the most important quality parameters of dried fruits and is a critical factor affecting consumer acceptance of the product. Mean color values including hue angle, Chroma, browning index, and total color change of the apple samples from different drying methods are presented in Table 2.

The L*, a* and b* of the fresh apples were 62.79, 1.58, and 21.41, respectively. The L* value (62.56) of the ultrasound-dried samples was very close to the fresh apples (62.70) and the FD samples (62.76), whereas a significant reduction in L* value (51.86) was observed in the HAD samples. These oxidative reactions occurring during HAD process may be responsible for the significantly lowered L* value in the HAD samples [8]. A significant increase in a* and b* values were observed with all drying methods. The increase for a* and b* values were in the order of HAD > US-CD > FD. The HAD apple slices had a darker appearance compared to the FD and US-CD samples, as shown by significantly lower L* value, which might be caused by the long-time exposure to heat during drying. On the other hand, an increase in a* and b* values indicated that the yellowness and redness of samples increased due to exposure to elevated temperature in HAD. Similar trends in color changes were obtained in previous studies in which the effects of freeze drying and hot air drying on the colors of different fruits were compared [30,8,17].

Significant differences were observed for the ΔE and BI values among all the drying methods. The FD samples had the least changes in ΔE and BI values, followed by the US-CD samples, while the HAD caused the

Table 3

pH, titratable acidity (TA), glass transition temperature (T_g) and change in specific heat (ΔC_p) values values of fresh apple and samples dried with different methods.

Drying Methods	pH	Titratable Acidity (TA)	Tg (°C)	ΔC_p (J/g °C)
Fresh	4.23 ^a	5.10 ^a	NP	NP
Freeze Drying	3.81 ^b	3.57 ^b	16.32 ^a	1.22 ^a
US-CD Drying	3.69 ^{bc}	2.98 ^c	14.07 ^a	1.05 ^b
Hot Air Drying	3.60 ^c	2.35 ^d	12.60 ^b	0.97 ^c

^{a-d}pH, TA, Tg and ΔC_p means between treatments with the same letter in each sample are not significantly different ($p < 0.05$). NP, not performed.

largest increase in ΔE and BI. The ΔE of the HAD samples is over 4-fold higher than that of the FD apples. Similarly, the HAD samples had the highest BI (91.54), 88% higher than that of the FD samples. BI is used to indicate the overall changes in browning color and is one of the most common indicators of browning in food products containing sugar [35]. The higher the ΔE and BI values, the bigger the color difference between the fresh samples and the dried samples, and the more formation of browning pigments during drying. In the study of Djekic et al. [16], it was also observed that the air-dried apple slices had the largest BI and ΔE values compared to that of freeze-dried samples.

Hue angle and Chroma values are also utilized to express the color intensity of food products. The hue angle values of the FD, US-CD and HAD samples were significantly different, with the highest change observed in the HAD apple slices. On the other hand, in all drying methods, the Chroma values increased with respect to fresh sample.

Color changes during drying might be due to various factors, such as Maillard reaction, pigment degradation, enzymatic browning, and ascorbic acid oxidation, among others [36,37]. The combination of these factors acting over a longer drying time can cause a decrease in lightness (L^*), an increase in redness (a^*) and yellowness (b^*), and a higher browning index and total color change in the food materials. As reported by other researchers, prolonged drying contributes to increase browning reactions that cause a change in color parameters [38,39]. Therefore, the better color retention in the US-CD samples compared to the HAD samples could be attributed to shorter drying time and reduced temperature.

3.3. Texture, rehydration ratio and bulk density

Texture is a principal quality parameter that determines the acceptability of dried food products by consumers. Two important textural parameters, hardness and number of peaks, of the FD, US-CD and HAD samples are tabulated in the Table 1. Hardness is one of the highly considered texture attributes of dried apples by consumers [40,30]. Hardness refers to the maximum force value on the force deformation curve of a sample. This value indicates the correlation between the amounts of force required to compress the sample. Therefore, the higher the value, the relatively firmer the texture of the samples. Based on the obtained data, the hardness was significantly ($P < 0.05$) affected by drying method (Table 1). The FD apple slices demonstrated a significantly ($P < 0.05$) lower hardness (8.65 N) than that dried by other methods. On the other hand, the hardness value of HAD samples (20.44 N) were significantly higher than that of the US-CD samples (16.86 N). Similar results were reported by Hu et al. [41] that the air-dried apples had a higher chewiness value, while lower hardness and chewiness values were observed in the freeze-dried samples thus having a softer texture. The number of peaks on the force deformation curve during the fracture was associated with the crispness which is another critical quality attribute for the dried food materials [42]. Our results showed that while the number of peaks observed in FD samples was significantly higher than the others, there was no significant difference between the US-CD and HAD samples (Table 3).

The nature of cellular matrix and the soluble solids inside the food

matrix, both having various interactions with water molecules, have a decisive effect on the textural properties of the dried food samples [30]. In HAD, surface moisture migrates to sample surfaces, causing the formation of a relatively firm structure and a hard-external layer. This is known as the case-hardening phenomenon [43], which can explain the reason behind the highest hardness values and number of peaks of the HAD samples. On the other hand, during freeze drying, plant cells and intercellular spaces remain intact as a result of sublimation of ice crystals. This provides high porosity and less elasticity to the structure. Moreover, due to the low temperature of freeze drying, the chemical reactions, such as pectin solubilization, are relatively slow compared with the HAD [30]. However, the FD samples had a more fragile structure and a lower hardness than those of the HAD and US-CD samples.

Rehydration ratio is a comprehensive quality parameter of dried foods, which is affected by processing conditions and composition of the product [17]. For the dried products, high rehydration ratio has a strong correlation with the higher water reabsorption and that indicates a less damage of the medium structure [42]. It is a measure of the severity of the physical and structural changes taking place during drying. The internal structures of the dried food material and the level of damage on the water-holding components, such as protein and starch molecules, caused by drying determine its ability to reconstitute [8]. The experimental data for rehydration ratio of the apple slices dried by different methods are presented in Table 1. The samples dried by different methods showed significantly different rehydration ratios ($P < 0.05$). The FD samples had the highest rehydration ratio while the HAD samples had the lowest. Similar results were obtained in Lewicki and Pawlak's [44] study, in which considerable differences in total porosity and size of pores between freeze drying and hot-air cabinet drying were observed. As can be seen in Table 1, the US-CD samples exhibited significantly higher rehydration ratios than the HAD samples. This is in agreement with the report of Mothibe et al. [45] who found that the samples treated with ultrasound displayed a better rehydration capability than that dried with other methods.

The bulk density measurements are also given in Table 1. No significant difference was observed between the bulk density values of the FD and US-CD samples, having a value of 350 and 420 kg/m³, respectively. The HAD samples exhibited the highest bulk density (610 kg/m³). Schulze et al. [23] also reported that the apple chips made by freeze drying had a more porous structure than the air or microwave vacuum dried samples, and thus had a relatively low bulk density. The lower bulk density of the US-CD samples than the HAD indicates that the former has a more porous structure than the latter, which should be responsible for the high rehydration ratio of the US-CD samples.

3.4. Glass transition temperature (T_g) and specific heat change (ΔC_p)

The glass transition temperatures (T_g) of the FD, US-CD and HAD apple samples are listed in Table 3. T_g is a thermodynamic second-order phase transition which leads to a change in the heat capacity of an amorphous material and causes an alteration in its structural state from glassy to rubbery. It occurs over a temperature range which is a characteristic for each material and may vary from 10 to 20 °C for amorphous sugars. The T_g is usually designated as the midpoint temperature of such a range [46].

T_g is a good indicator for stability of biopolymers, including foods, which is a function of the food composition, water content, and molecular weight of the solute components in the material. A high T_g value could imply a better storage ability [30]. In this study, the FD and US-CD apple slices showed a significantly higher T_g than the HAD samples, thus could be more stable at room temperature. The longer drying time and higher temperature, which cause more structural damage to the food product leading to the collapsing of its tissue, might be a reason for HAD samples to have a lower glass transition temperature [47]. On the other side, the higher T_g in the US-CD samples might be due to low

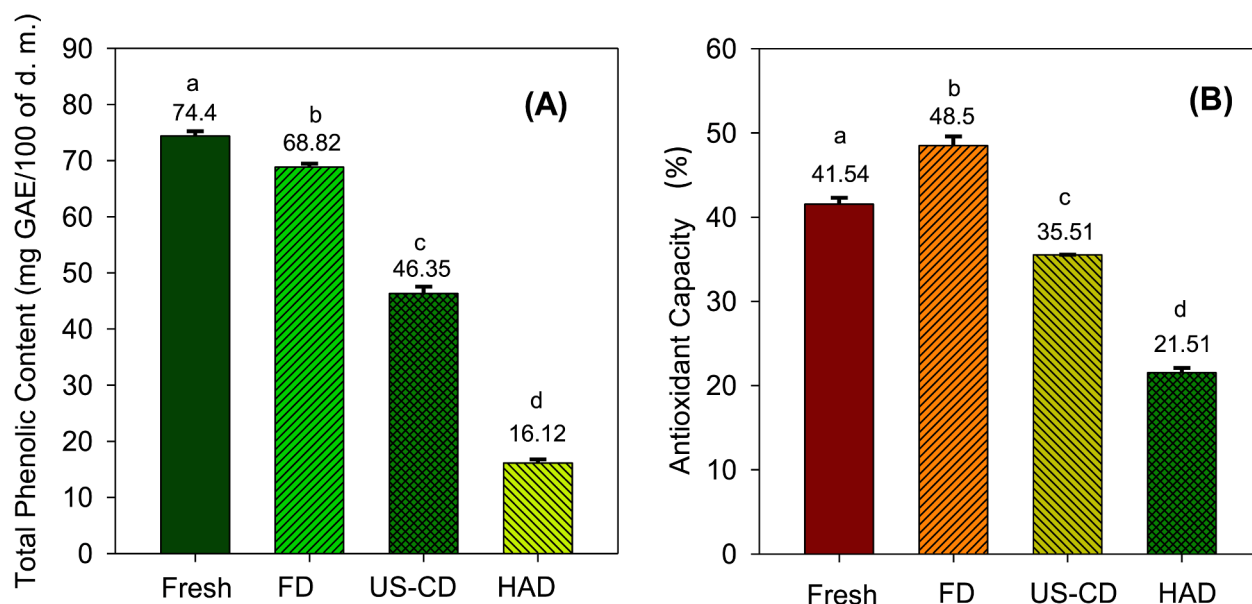


Fig. 3. Total phenolic content (TPC) (A) and antioxidant capacity (AC) (B) of fresh apple slices and samples dried with different methods. ^{a-d}TPC and AC means between treatments with the same letter in each sample are not significantly different ($p < 0.05$).

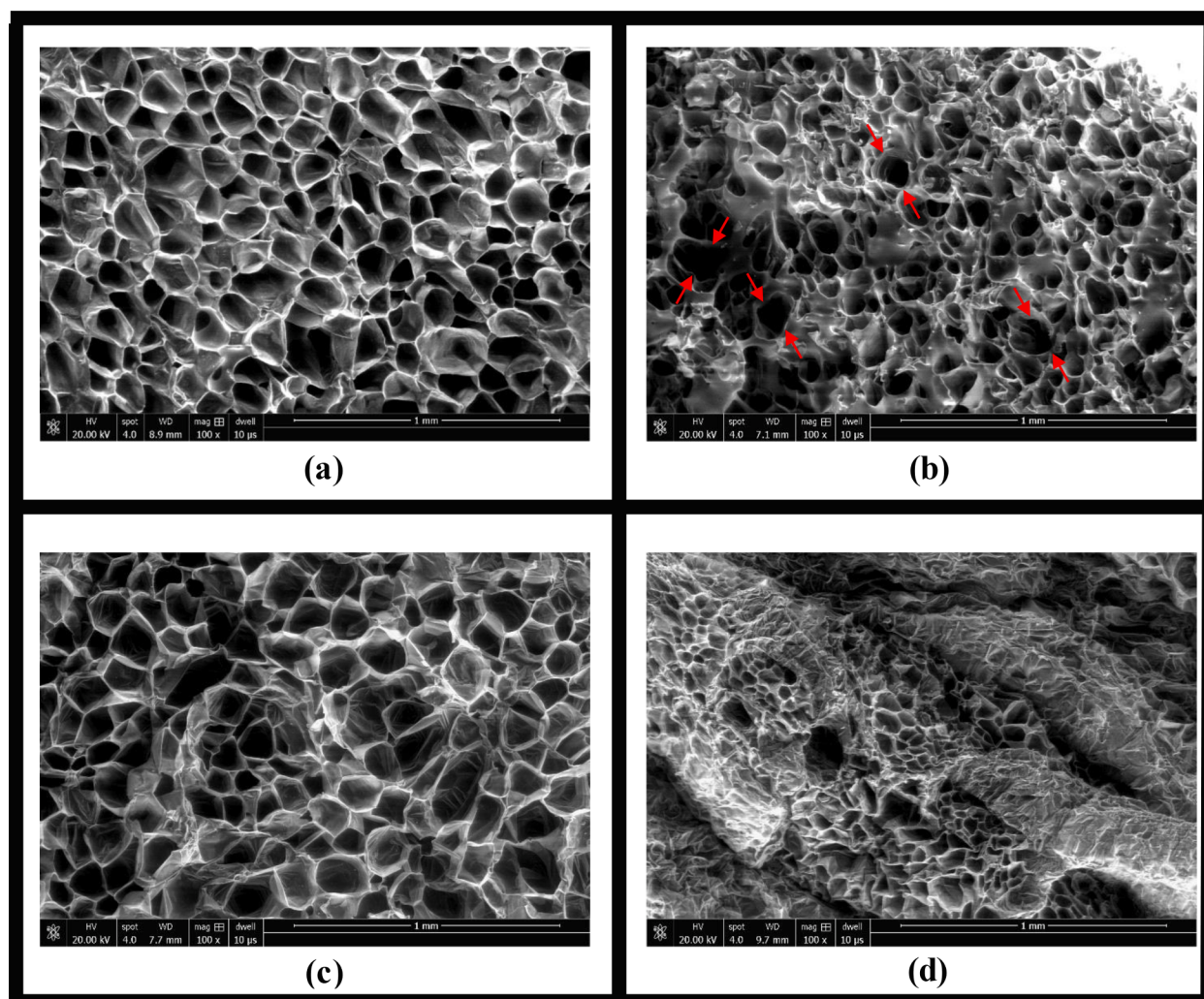


Fig. 4. ESEM micro-images of fresh (a), FD (b), US-CD (c) and HAD (d) apple slices.

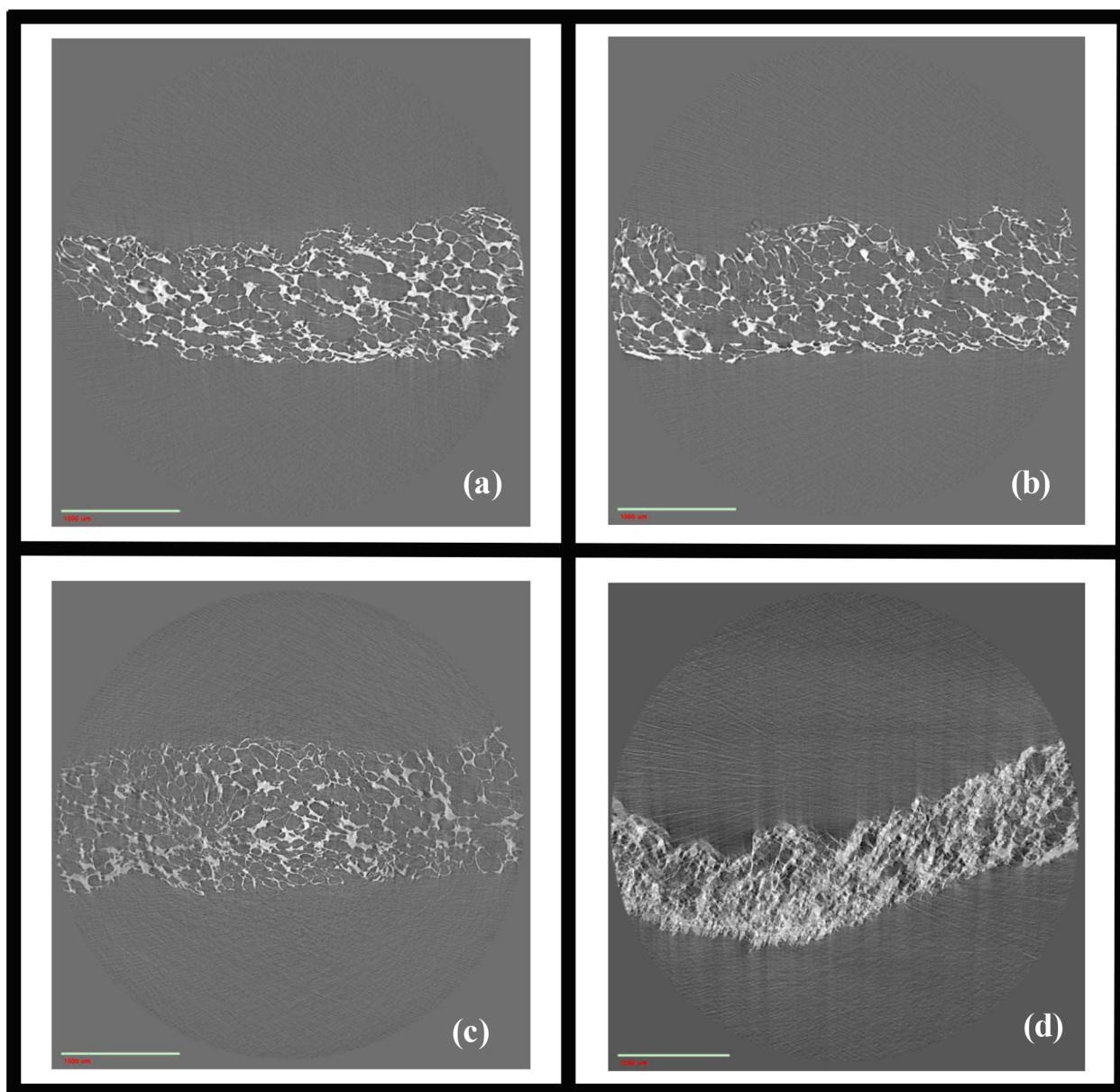


Fig. 5. 2D Micro-CT images of fresh (a), FD (b), US-CD (c) and HAD (d) apple slices.

temperature and resulting increase in the free volume for the movement of the molecules than the HAD [48].

The specific heat (ΔC_p) at the glass transition of the FD, US-CD and HAD samples are also shown in Table 3. It can be seen that the lower the T_g values of the samples, the lower the ΔC_p value. While all the drying methods showed a significantly lower ΔC_p at their glass transition temperatures, the US-CD samples had a ΔC_p value of 1.05 J/g °C significantly higher than the HAD samples (0.967 J/g °C). As mentioned by Mayhew et al. [49], this might be due to the existence of a higher solid composition in the amorphous state of the material than its crystalline state.

3.5. pH and titratable acidity

The pH readings of the fresh, FD, US-CD and HAD samples are listed in Table 3. While all the samples had a pH higher than 3.5, they were all significantly lower than the pH of the fresh apple slices (pH 4.23). The pH of the FD and US-CD samples were not significantly different, but the pH of the HAD samples was significant lower (3.60). As shown in

Table 3, the titratable acidity (TA) values of the fresh, FD, US-CD and HAD samples were 5.10, 3.57, 2.97 and 2.35% malic acid d.m., respectively. The results were all significantly different from each other ($P < 0.05$) with the highest TA observed in the HAD samples. The decreases in the pH and titratable acidity values of the HAD samples might be attributed to an increase in dissociation of the organic acids at elevated temperatures [50].

3.6. Total phenol contents and DPPH free radical scavenging activity

The TPC of the fresh apple was 74.40 mg GAE/100 g of dry mass by the Folin-Ciocalteu method (Fig. 3(A)). A maximum retention of TPC (68.82 mg GAE/100 g of dry mass) was observed in the freeze-dried slices, due to its low processing temperature. In US-CD, the mechanical stress occurred from ultrasonic wave propagation might cause the reduction in polyphenol content by assisting the leakage of intracellular compound and oxidative enzymes from the food matrix into the solvent [51]. However, as it is shown in Fig. 3(A), the US-CD samples exhibited a significantly higher TPC (46.35 mg GAE/100 g of dry mass) than the

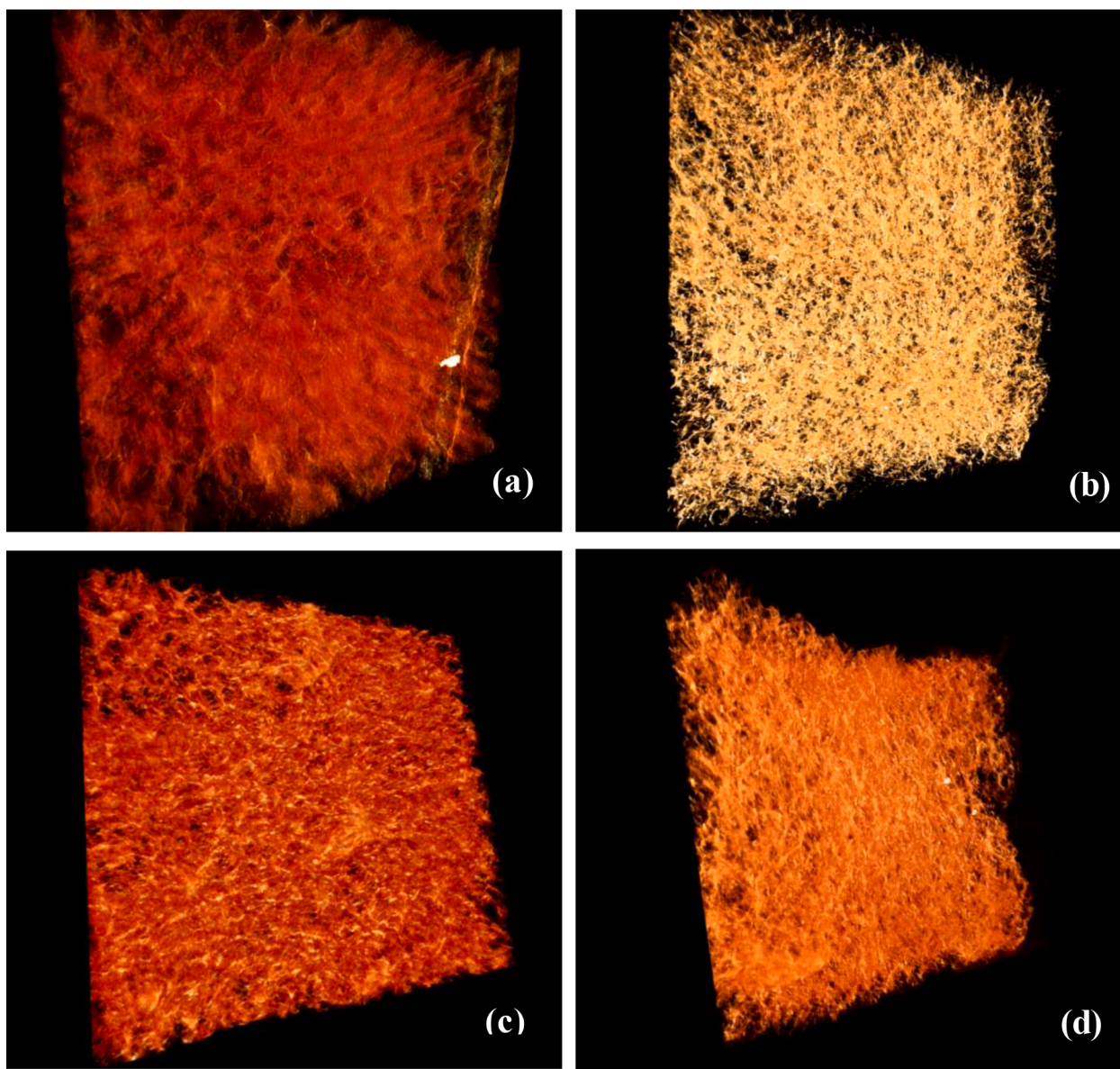


Fig. 6. 3D Micro-CT images of fresh (a), FD (b), US-CD (c) and HAD (d) apple slices.

HAD samples. Similar results were obtained in the study of Shewale and Hebbar [17], in which the effect of freeze and hot-air drying processes on quality of apple slices was investigated. Heat treatment, especially prolonged heating processes, will reduce enzymatic activities and promote degradation of phenolic compounds [52,17]. As a result, a higher reduction in TPC of HAD samples was recorded compared to other drying methods.

As one of bioactive compounds, the total polyphenol content is directly related to antioxidant activity. The initial DPPH radical scavenging activity for the fresh apple was 41.54% (Fig. 3(B)). The FD and US-CD samples had DPPH activities of 48.5 and 35.51%, respectively, which were significantly higher ($P < 0.05$) than the HAD samples (21.51%). In previous studies, the reduction in antioxidant capacity (AC) of ultrasound-assisted drying was attributed to the cell damage caused by the mechanical stress created by the acoustic waves [51]. However, due to the mild drying conditions in US-CD, the antioxidant capacity was preserved better than in the HAD apples. These results are in accordance with the observations of Tiwari and Cummins [52] and Rodríguez et al. [51] who reported a lower degree of antioxidant degradation when apple was freeze-dried. Additionally, similar to the

findings of Shewale and Hebbar [17], an increase in antioxidant capacity with freeze drying was observed in this research. Maillard reaction products formed during drying, such as melanoidins, might be responsible for improved antioxidant capacity of freeze-dried apple slices [38].

3.7. Microstructure

The microstructure of the fresh, FD, HAD and US-CD apple slices was observed by ESEM. Images of the fresh apple and apples dried are shown in Fig. 4.

During thermal drying, plant tissues are exposed to simultaneous thermal and moisture gradient stresses which cause significant changes in their structure [53–55]. These changes are mainly microstructural deformations and shrinkage formed due to the removal of moisture from the cells and reduction in turgor pressure. According to Lewicki and Pawlak [44], the reaction of the cell wall to the turgor loss occurs in two steps. In the first step, the cell wall loses its elasticity as the turgor pressure decreases with drying. Although this causes a reduction in the cell size, due to the positive turgor pressure inside the cell, it can still

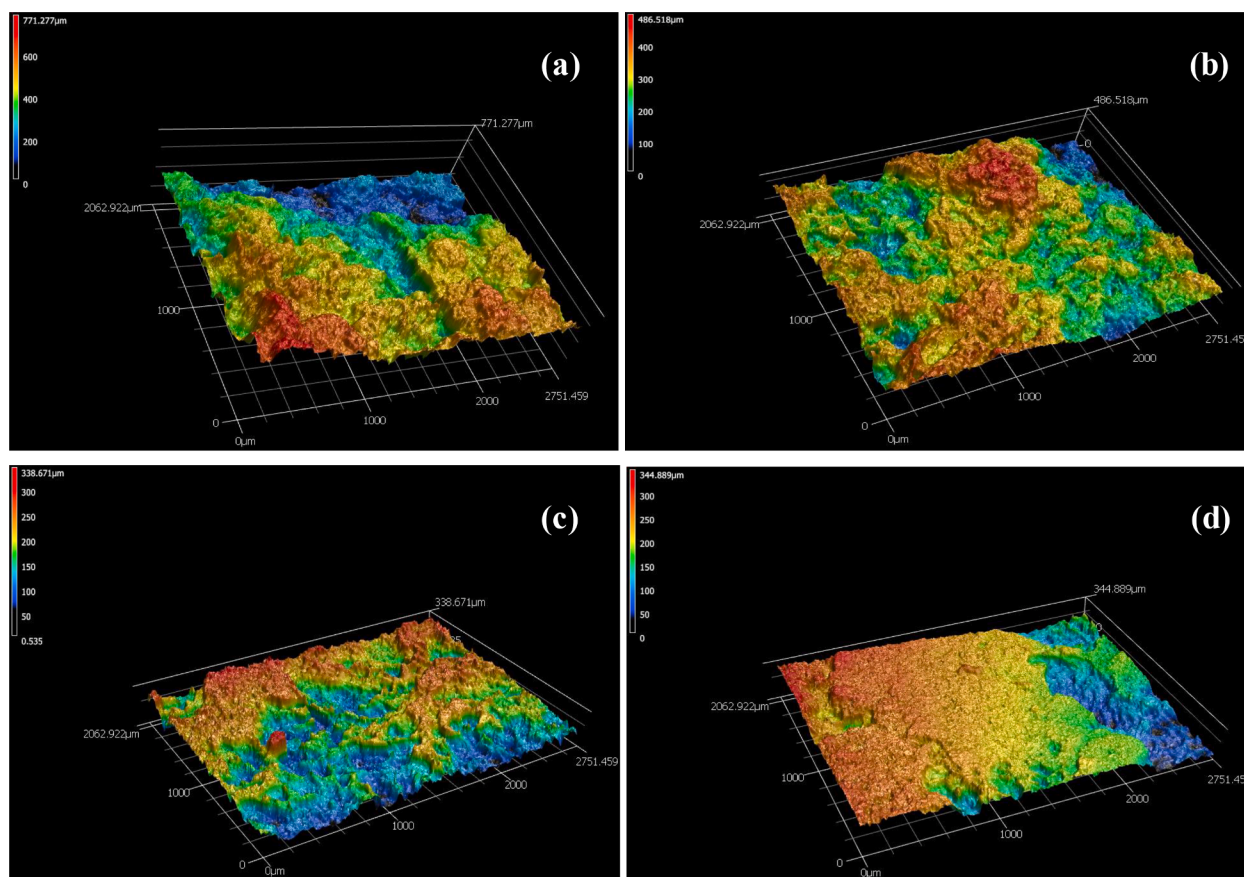


Fig. 7. 3D surface profiles of fresh (a), FD (b), US-CD (c) and HAD (d) apple slices.

retain its original circular shape during this step. Meanwhile, reductions in cellular dimensions of individual cells might be seen because of the contractions of cell wall occurred by drying. Further drying can lead to removal of more cell fluid which causes the turgor pressure to reach a minimum level. In this stage, more deformation occurs in the cell wall because of the decreased tension to adapt itself to lower cell fluid volumes. Significant volume reduction during drying was reported in many studies supporting the mechanism proposed by Lewicki and Pawlak [44,56–58].

As shown in Fig. 4(a), the majority of the cells in fresh apple tissues is intact and uniform in size. Karunasena et al. [59] also observed that the cells of fresh apple were mostly 2D circular cells, may be due to the tension created on the cell wall as a result of the turgor pressure applied by the cell fluid. Some structural deformations can be clearly observed in the freeze-dried apple slices (Fig. 4(b)). Compared with the fresh apple sample, the tissue of the FD sample lost its organized structure due to the collapsed cells, texture breakages, membrane breakdowns and formation of larger intercellular spaces as shown by the red arrows in Fig. 4(b). In the studies of Chassagne-Berces et al. [60] and Laurienzo et al. [27], they also observed some large pores in the FD samples, which might be attributed to the formation of large ice crystals during freezing. Moreover, starch accumulation in some localized areas was detected in the FD samples [27]. Significant shrinkage of cell volume, and partial collapse of the tissue can be observed on the HAD apple samples (Fig. 4(d)). It may be induced by the high drying temperature and long drying time in HAD, and the resulting cell deformation and formation of intercellular spaces due to the high amounts of collapsing cells. These observations are in agreement with the studies of Askari et al. [61] and Karunasena et al. [59], in which the conventional dried samples were not able to protect their original microstructures. Among the drying methods, the US-CD samples showed the best microstructure retention (Fig. 4(c)).

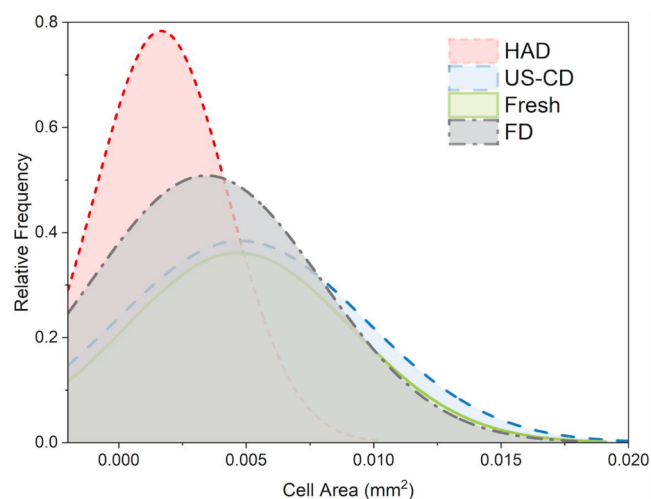


Fig. 8. Distribution of the pore size area (mm^2) of fresh and dried apple samples calculated from ESEM images.

Moreover, the distribution of the pore size areas in fresh and dried apple samples was presented in Fig. 8. Drying processes could cause significant changes in the size of the pores and their distribution on the surface area. In this study, the maximum values for the area of pore sizes occur when it is equal to around 0.005, 0.005, 0.0035, and 0.002 mm^2 for fresh, US-CD, FD, and HAD samples, respectively. The pores in US-CD and FD treated samples have a similar cross-section area as fresh sample, and larger area comparing to the HAD samples. It can be noticed that the microstructure of US-CD apple samples looks quite similar to the fresh

apple samples except some small deformations in the cell shapes and sizes. The enhanced mass transfer due to ultrasound [31] and the near room temperature drying might contribute to preserving the microstructures of the apple tissues.

The microstructures of the apple samples were also investigated with Micro-CT in 2D and 3D modes (Figs. 5 and 6). During Micro-CT analysis, the sample was first scanned by the X-ray from different angles by rotating it and transformed into a grayscale 2D image by the detector, which was then turned into a 3D model via a reconstruction algorithm [28]. The cross-section images and the 3D models of the fresh and dried apple slices revealed that the FD and US-CD better protected the microstructures than the HAD (Figs. 6 and 7). While losses in structural integrity, deformation in cell shape, and cell wall breakdown can be observed in the HAD samples, the FD and US-CD samples retained the porous structure seen in the fresh samples. Mendoza et al. [62] also examined the 3D structure of apple tissue by using a similar method and detected similar porous characteristics of fresh apple samples.

The morphological changes in the samples after drying were also analyzed with a 3D optical profiler. The images belonged to the fresh, FD, US-CD and HAD samples are presented in Fig. 7. The 3D optical surface profile analysis provided detailed visuals of the microstructures by utilizing a color scale to illustrate their porosity levels. The amount and distribution of indentations shown by different colors represent the degree of porosity that the structure has. From the images one can see that while there are many indentations on the surface of the FD and US-CD samples as in the fresh samples, the structure of the HAD samples display a smoother surface profile. Therefore, the 3D optical surface profile analysis also confirms that the FD and US-CD are more successful in protecting the sample's native structure.

4. Conclusion

In this research, drying characteristics, quality attributes, and microstructures of apple slices dried with FD, HAD, and US-CD were examined. Overall, the US-CD showed a drying performance close to that of the FD with regards to quality retention and microstructural changes. The drying time with US-CD was significantly shorter than the FD and HAD. The rehydration ratio, color and textural properties of the US-CD apple slices were greatly improved when evaluated against the HAD. The total phenolic content and antioxidant capacity of the US-CD samples were 2.25 and 1.65 times higher than that of the HAD counterparts, respectively. The FD-dried and US-CD-dried apple slices had a significantly higher Tg than the HAD samples, indicating a better stability at room temperature. The microstructural changes in the apple slices shown by ESEM, Micro-CT, and 3D surface profile analysis confirmed that the HAD was detrimental to the microstructure of the food products, while the US-CD exhibited a better protection of the porous structure and less change in cell size and shape. The findings showed that the US-CD might be a good alternative to HAD due to its superior quality retention ability, improved structural behavior, and good drying characteristics along with significantly decreased drying time and temperature.

CRedit authorship contribution statement

Ozan Kahraman: Conceptualization, Methodology, Writing - original draft. **Hao Feng:** Conceptualization, Methodology, Supervision, Writing - review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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