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Araştırma Makalesi (Research Article)

**Investigation of Effect of Ultrasound Pretreatment on Drying and Rehydration Characteristics and Microstructure of Apple Slices**

Senem TÜFEKÇİ<sup>1</sup>, Sami Gökhan ÖZKAL<sup>2\*</sup>

<sup>1</sup>Department of Food Processing, Vocational School of Acıpayam, Pamukkale University, 20800, Denizli, Turkey

<sup>2</sup>Department of Food Engineering, Faculty of Engineering, Pamukkale University, 20160, Denizli, Turkey

<sup>1</sup><https://orcid.org/0000-0002-4613-2418> <sup>2</sup><https://orcid.org/0000-0001-5040-9814>

\*Corresponding author e-mail: [sgozkal@pau.edu.tr](mailto:sgozkal@pau.edu.tr)

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**Abstract:** In this study, the effects of ultrasound pretreatment on hot air drying and rehydration characteristics and microstructure changes of apple slices were examined. Slices of apple cv. Granny Smith having 5 mm thickness were used as the material. Pretreatments with ultrasound application were done by an ultrasonic generator with 20 kHz frequency. Effect of pretreatment time (10, 20, 30 min) and amplitude (55 and 100 %) were evaluated. Drying experiments were performed in a cabinet drier at 60°C with 0.3 m.s<sup>-1</sup> constant air velocity. Application of ultrasound pretreatment decreased the drying time of apple slices in some conditions. Amongst the five thin layer drying models tested, the Page and Modified Page models found to be the best fitting models for the expression of the drying of apple slices. Microstructural changes caused by ultrasound pretreatment enhanced the rehydration ability of dried apples. As the pretreatment time and ultrasound amplitude increased, the rehydration ratio had an increasing trend. Peleg model represented the rehydration kinetics of the samples successfully. Changes in the microstructure of the apple samples caused by the ultrasound pretreatment were clearly observed with scanning electron microscopy images. Structural changes, caused by ultrasound pretreatment in apple tissues, resulted in low shrinkage values.

**Ultrases Ön İşleminin Elma Dilimlerinin Kurutma ve Rehidrasyon Karakteristikleri ile Mikroyapısına Etkisinin Araştırılması**

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**Anahtar kelimeler**

Elma,  
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**Öz:** Bu çalışmada, ultrasonik ön işlem uygulamasının elma dilimlerinin sıcak hava ile kurutulması, rehidrasyon özellikleri ve mikro yapısında meydana gelen değişiklikler üzerine etkisi araştırılmıştır. Materyal olarak 5 mm kalınlığında Granny Smith elma çeşidinin dilimleri kullanılmıştır. Ultrases ön işlemi 20 kHz frekanslı ultrases üreticisi ile yapılmıştır. Ultrases ön işleminin etkisi seçilen süre (10, 20, 30 dakika) ve genlik (% 55 ve % 100) değerlerinde incelenmiştir. Kurutma deneyleri kabin kurutucuda 60 °C'de ve 0.3 m.s<sup>-1</sup> sabit hava hızında gerçekleştirilmiştir. Ultrases ön işlemi, bazı koşullarda elma dilimlerinin kuruma süresini kısaltmıştır. Denenen beş ince tabaka kurutma modeli arasından Page ve Modifiye Page modelleri elma dilimlerinin kurutulmasını ifade eden en uygun modeller olarak bulunmuştur. Ultrases ön işleminin neden olduğu mikroyapısal değişiklikler, kurutulmuş elmaların rehidrasyon kabiliyetini arttırmıştır. Ön işlem süresi ve ultrases genliği arttıkça rehidrasyon oranı artış eğiliminde olmuştur. Peleg modeli

örneklerin rehidrasyon kinetiğini başarılı biçimde temsil etmiştir. Ultrases ön işlemi ile elma örneklerinin mikroyapısında meydana gelen değişimler taramalı elektron mikroskobu görüntüleri ile açık bir şekilde gözlemlenmiştir. Ultrases ön işlemi ile elma dokusunda meydana gelen yapısal değişiklikler düşük büzüşme oranı değerleri olarak sonuçlanmıştır.

## 1. Introduction

Apple is used as a raw material in many food formulations and cultivated in almost every part of the World because of its flavor and rich nutrients (Andres et al., 2004; Aras and Eşitken, 2019). Consumption of apple can be in fresh or processed forms such as juices, jams, marmalades, purees and dried products (Sacilik and Elicin, 2006). In recent years demand for the dried snack fruit products increasing due to their ease of use and healthy effects (Turgut and Topuz, 2020). Developing new apple preservation techniques is important.

Hot air drying is widely used for preservation of food and agricultural products. However, this method has several disadvantages like undesirable flavor and color, nutrient loss due to long drying time (Contreras et al., 2008). The use of ultrasound as a new technology may defeat some of the disadvantages of hot air drying (Garcia-Perez et al., 2007). Utilization of ultrasound technology has attracted large interest due to its promising effects in food processing (Knorr et al., 2004). Pre or simultaneous application of ultrasound energy in convective drying has increased the drying rate without a notable loss of quality in the product. Ultrasound has also increased the diffusion of water by reducing the internal resistance caused by the inner boundary layer (Rastogi, 2011).

In literature, the impact of ultrasound energy on drying behavior and characteristics of apples has been investigated widely with different drying methods such as low temperature air drying (Sabarez et al., 2018), convective air drying (Rojas et al., 2020; Banaszak and Pawłowski, 2018), atmospheric freeze drying (Moreno et al., 2017), vacuum freeze drying (Zhang et al., 2020; Ren and Bai, 2018), osmotic dehydration (Fernandes et al., 2019; Mierzwa and Kowalski, 2016) and microwave drying (Wiktor and Witrowa-Rajchert, 2020; Lv et al., 2019).

The objective of this study was to determine the influence of ultrasonic pretreatment on hot air drying and rehydration characteristics, shrinkage, rehydration ratio, water holding capacity and microstructural changes of Granny Smith apple slices. This study differs from the previous studies reported in literature with the application method of ultrasound energy and modeling the experimental drying data by using thin layer mathematical models.

## 2. Materials and Methods

Fresh apples (*cv.* Granny Smith) were purchased from a bazaar in Denizli. Samples were stored in a refrigerator at +4°C in polyethylene freezer bags until being dried. Moisture content was determined by gravimetric method in a hot air oven at 105 °C.

### 2.1. Ultrasound pretreatment

Apples were washed, peeled and cut into cylindrical slices of constant thickness (5 mm) by an electronic fresh vegetable cutter (Moulinex Fresh Express, France). Then the apple slices were dipped in distilled water at 25°C for sonication. Solid/liquid ratio used was 1:4 (w/w). The sonication processes were done by using an ultrasonic generator (Bandelin Sonopuls, Model HD 2200) working at 200 W power and 20 kHz frequency having a 13 mm diameter titanium probe (TT 13). Time and amplitude values of ultrasound pretreatment were chosen as 10, 20 and 30 min, and 55 and 100 %, respectively (Tüfekçi and Özkal, 2017).

Variations in temperature during ultrasonic pretreatment depending on conditions applied were determined by measuring the temperature of the distilled water before and after the pretreatment. Mass changes of the samples were determined by weighing the samples after removing superficial water at the end of pretreatments.

## 2.2. Drying experiments

Drying of apple samples were performed in a tray dryer (Yucebas Machine, İzmir, Turkey) (Demiray and Tülek, 2014). To achieve steady state conditions, the dryer was powered on 1 h before each drying experiment. The apple slices were weighed and placed uniformly as a single layer on the trays. The trays were inserted into the drier which were set to a constant air temperature (60 °C) and velocity (0.3 m.s<sup>-1</sup>).

The samples were weighed at every 5 min during the first 30 min, then every 30 min until constant weight conditions were observed. All drying experiments were performed in duplicate and average values of each condition were reported.

## 2.3. Shrinkage

For the determination of shrinkage values, bulk volume of samples was measured by a gas pycnometer (Micromeritics, Model AccuPyc II 1340) and the shrinkage values were calculated from the volume reduction of apple slices as percent shrinkage (*S*) (Equation 1) (Deng and Zhao, 2008);

$$S = [(V_0 - V_s) / V_0] \times 100 \quad (1)$$

Where  $V_0$  and  $V_s$  are volumes of the initial (fresh) and dried sample, respectively. For each measurement, 5 apple slices were selected and the measurements were performed in duplicate and the average value of percent shrinkage of the samples at each condition were reported.

## 2.4. Rehydration experiments

The rehydration experiments of dried apple samples were done at a constant temperature of 60°C. Dried apple samples were immersed into distilled water at a solid/liquid ratio of 1:50 (w/w). Samples were weighed after each 5 min of immersion during the first 30 min then every 30 min until a constant weight was attained. The superficial water was removed by draining samples over a mesh for 30 s prior to each measurement.

Rehydration ratio (*RR*) was estimated from the Equation 2;

$$RR = w_{rt} / w_{r0} \quad (2)$$

Where  $w_{rt}$  is mass of the rehydrated sample and  $w_{r0}$  is mass of the dry sample.

To obtain the water holding capacity (*WHC*), rehydrated samples were centrifuged at 4000 rpm for 10 min at 5°C in tubes having a mesh in the center and water removed from samples were weighed. The *WHC* values were estimated from the measured masses of the samples by using the Equation 3 (Vega-Gálvez et al., 2009);

$$WHC = [(Mr \cdot Xr - Ms) / (Mr \cdot Xr)] \cdot 100 \quad (3)$$

Where *Mr* is the mass of the sample after rehydration, *Xr* is the moisture content wet basis and *Ms* is the mass of the removed liquid from rehydrated sample after centrifugation.

## 2.5. Drying kinetics

The equations given below (Equation 4 and 5) were used for estimation of the moisture ratio (*MR*) and drying rate (*DR*) of the apple samples:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad (4)$$

$$DR = \frac{M_{t+dt} - M_t}{dt} \quad (5)$$

Where,  $M_0$  is the initial moisture content,  $M_e$  is the equilibrium moisture content,  $M_t$  is the moisture content at any time  $t$ , and  $M_{t+dt}$  is the moisture content at  $t + dt$  (g water.g dry matter<sup>-1</sup>) and  $t$  is the drying time (min) (Sacilik and Elicin, 2006).

Mathematical modeling is necessary for food technology to understand the phenomena of drying and rehydration process (Cruz et al., 2015). Five different thin layer models given in Table 1 were used for estimation of drying kinetics of experimental data of the apple samples.

Table 1. Thin layer models used (Demiray and Tülek, 2014)

Model no	Model name	Model equation
1	Lewis	$MR = \exp(-kt)$
2	Henderson and Pabis	$MR = a \exp(-kt)$
3	Page	$MR = \exp(-kt^n)$
4	Modified Page	$MR = \exp(-(kt)^n)$
5	Logarithmic	$MR = a \exp(-kt) + c$

## 2.6. Rehydration kinetics

Peleg model (Equation 6) was used for modeling of the data obtained from the rehydration experiments;

$$M = M_0 + t/(k_1 + k_2t) \quad (6)$$

In this equation  $M_0$  is the initial moisture content,  $M$  is the moisture content at time  $t$ ,  $k_1$  is the Peleg rate constant and  $k_2$  is the Peleg capacity constant (Ricca et al., 2016).

## 2.7. Statistical analysis

To describe the most suitable model representing the drying and rehydration curves of the apple samples, the  $R^2$ , reduced chi-square ( $\chi^2$ ) and root mean square error (RMSE) were used as the statistical parameters.

$$\chi^2 = \frac{\sum_{i=1}^N (MR_{exp,i} - MR_{pre,i})^2}{N - n} \quad (7)$$

$$RMSE = \left[ \frac{1}{N} \sum_{i=1}^N (MR_{pre,i} - MR_{exp,i})^2 \right]^{1/2} \quad (8)$$

The model which has the greatest  $R^2$ , and least  $\chi^2$  and RMSE was selected as the best representing model of the drying kinetics of the apples.

## 2.8. Microstructure

To investigate the impact of ultrasonic pretreatment on the microstructure of the apple slices, scanning electron microscopy (SEM) micrographs of the control (untreated samples) and the 20 min sonicated samples with 55 and 100 % amplitudes dried at 60 °C were obtained by using FEI Quanta 250 FEG instrument.

### 3. Results

#### 3.1. Temperature and mass change during ultrasound pretreatment

Temperature changes of the immersing water used in ultrasonic pretreatment and mass changes of the apple samples after ultrasound pretreatment were presented in Table 2.

Before ultrasound pretreatment, the temperature of the immersing water was 25 °C. It increased up to 56.50 °C during ultrasound pretreatment. Additionally, mass loss in the samples increased with increasing pretreatment time and ultrasound amplitude. As a result, mass of the apple slices decreased.

Table 2. Changes occurred during ultrasound pretreatments

Pretreatment conditions		Change in temperature (°C)*	Change in mass (%)*
Amplitude (%)	Time (min)		
100	10	29.75±0.50	-0.96±0.07
	20	50.00±0.83	-3.10±0.14
	30	56.50±1.29	-3.35±0.44
55	20	29.00±0.82	-0.80±0.05

\*Values are given as “mean ± standard deviation” n=4

#### 3.2. Drying characteristics

Sample codes indicating the drying temperature and pretreatment conditions for apple samples were listed in Table 3 and the same codes were used throughout the study. Total drying times of apple samples at 60 °C to the final moisture content of 0.1 g water.g dry matter<sup>-1</sup> were also reported in Table 3.

Table 3. Times needed to reach final moisture content for coded samples.

Drying temperature (°C)	Pretreatment conditions		Sample code	Time needed to reach final moisture content (min)
	Amplitude (%)	Time (min)		
60	-	-	60 °C control	143
	100	10	60° C - 100 % - 10 min	210
		20	60 °C - 100 % - 20 min	117
		30	60 °C - 100 % - 30 min	113
		55	60 °C - 55 % - 20 min	183

Influence of ultrasound pretreatment on drying kinetics of apple slices are given in Figure 1 and 2. Figure 1 shows drying rate versus moisture content curves and Figure 2 shows the changes in *MR* values with the drying time.

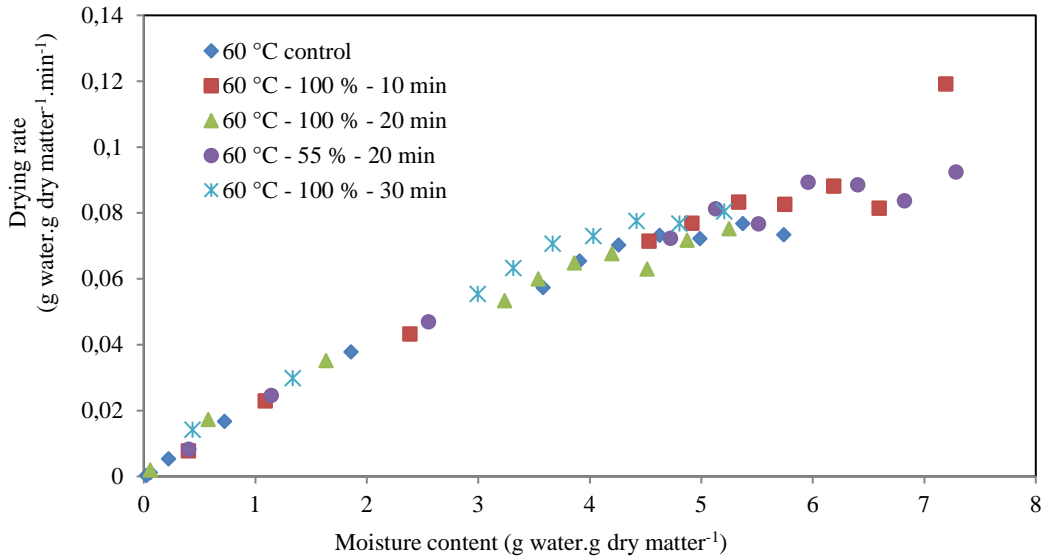


Figure 1. Drying curves of apple slices.

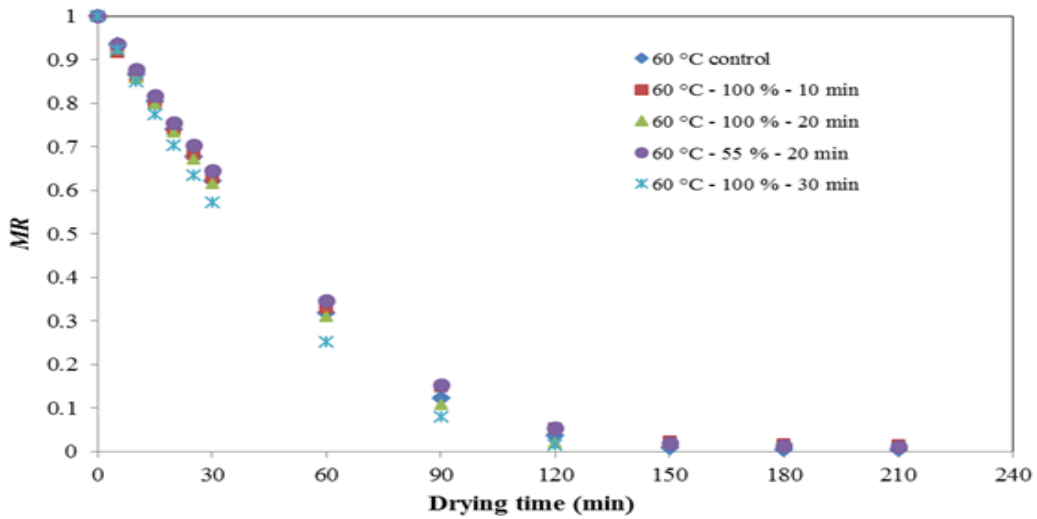


Figure 2. Drying kinetics of apple slices.

### 3.3. Modeling of drying kinetics

The *MR* values of the apple slices were fitted to five different mathematical drying models and model constants and statistical comparison parameters for apple drying are given in Table 4. Page and Modified Page models were found to be the best fitted models for representing the drying kinetics of apple by providing higher  $R^2$  and lower  $\chi^2$  and RMSE values for all conditions.

Table 4. Model coefficients and statistical comparison parameters for apple drying

Sample Code	Model No	Model Coefficients			R <sup>2</sup>	RMSE	χ <sup>2</sup>
60 °C	1	k = 0.0268			0.9780	0.0278	0.0108
	2	a = 1.2418	k = 0.0284		0.9838	0.0252	0.0097
	3	k = 0.0079	n = 1.2384		0.9958	0.0046	0.0003
	4	k = 0.0200	n = 1.2384		0.9958	0.0046	0.0003
	5	a = 1.5938	k = 0.0373	c = 0.0049	0.9570	0.0604	0.0617
60 °C - 100 % - 10 min	1	k = 0.0229			0.9857	0.0190	0.0051
	2	a = 1.1515	k = 0.0241		0.9906	0.0169	0.0044
	3	k = 0.0115	n = 1.1237		0.9926	0.0053	0.0004
	4	k = 0.0188	n = 1.1237		0.9926	0.0053	0.0004
	5	a = 1.4965	k = 0.0351	c = 0.0167	0.9580	0.0532	0.0477
60 °C - 100 % - 20 min	1	k = 0.0272			0.9570	0.0322	0.0125
	2	a = 1.2572	k = 0.0296		0.9696	0.0319	0.0136
	3	k = 0.0114	n = 1.2297		0.9908	0.0243	0.0080
	4	k = 0.0207	n = 1.2297		0.9908	0.0243	0.0080
	5	a = 1.1360	k = 0.0258	c = 0.0175	0.9708	0.0201	0.0061
60 °C - 55 % - 20 min	1	k = 0.0236			0.9808	0.0242	0.0083
	2	a = 1.2176	k = 0.0253		0.9890	0.0228	0.0080
	3	k = 0.0081	n = 1.2024		0.9955	0.0043	0.0003
	4	k = 0.0183	n = 1.2024		0.9955	0.0043	0.0003
	5	a = 1.1470	k = 0.0331	c = 0.0116	0.9649	0.0491	0.0407
60 °C - 100 % - 30 min	1	k = 0.0300			0.9664	0.0348	0.0134
	2	a = 1,3010	k = 0.0335		0.9674	0.0389	0.0189
	3	k = 0.0092	n = 1.2397		0.9928	0.0061	0.0005
	4	k = 0.0228	n = 1.2397		0.9928	0.0061	0.0005
	5	a = 1.1677	k = 0.0296	c = 0.1516	0.9739	0.0530	0.0402

### 3.4. Shrinkage

The shrinkage ratio of the ultrasound pretreated samples reduced gradually as the pretreatment time and amplitude increased (Table 5).

Table 5. Shrinkage ratios of apple slices

Sample code	Shrinkage Ratio*(%)
60 °C control	88.00±0.44
60 °C - 100 % - 10 min	88.85±1.90
60 °C - 100 % - 20 min	85.33±0.24
60 °C - 55 % - 20 min	87.76±0.22
60 °C - 100 % - 30 min	85.80±1.77

\* Values are given as ± standard deviation n=4

### 3.5. Rehydration kinetics

Rehydration curves of dried apple slices, model coefficients and statistical parameters of Peleg model are given in Figure 3 and Table 6, respectively. The Peleg model had high R<sup>2</sup> and low χ<sup>2</sup> and RMSE values which were represented empirical data successfully.

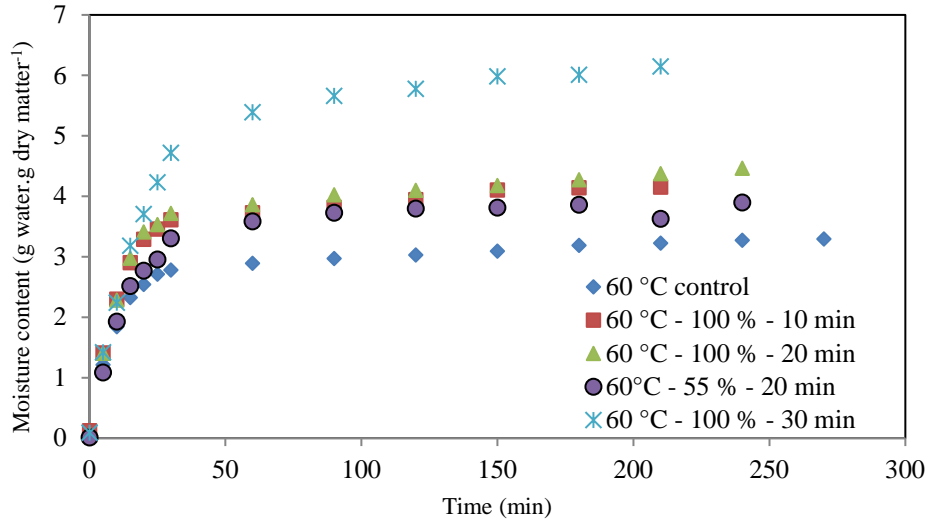


Figure 3. Rehydration curves of dried apple slices.

Table 6. Peleg model coefficients and statistical parameters for rehydration curves of dried apple slices

Sample code	$k_1$	$k_2$	$R^2$	RMSE	$\chi^2$
60 °C control	0.0421	0.2992	0.9992	0.0289	0.0136
60 °C - 100 % - 10 min	0.0337	0.2392	0.9992	0.0423	0.0266
60 °C - 100 % - 20 min	0.0357	0.2229	0.9989	0.0452	0.0313
60 °C - 55 % - 20 min	0.0363	0.2520	0.9974	0.0428	0.0282
60 °C - 100 % - 30 min	0.0418	0.1535	0.9989	0.0525	0.0397

### 3.6. Rehydration ratio and water holding capacity

The rehydration ratio increased with the increase in ultrasound pretreatment time and amplitude (Figure 4).

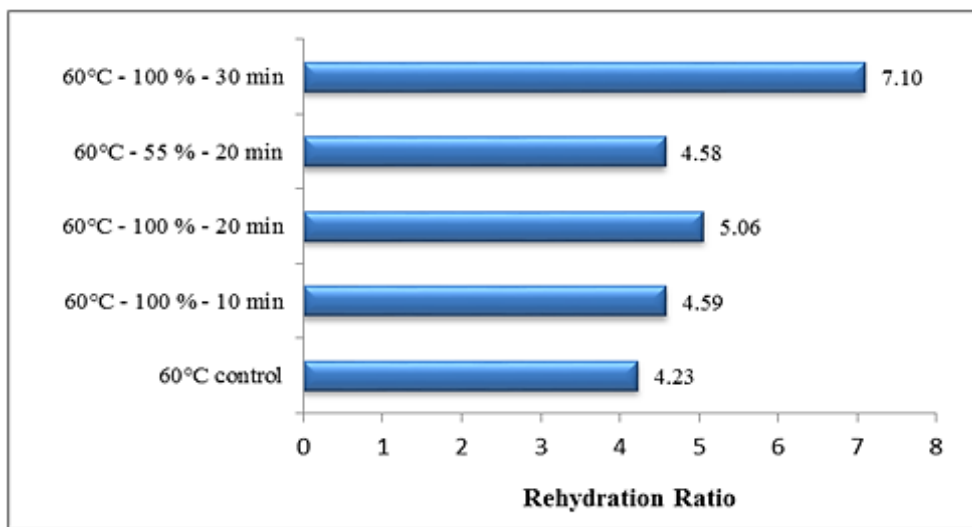


Figure 4. Rehydration ratio of apple slices.



As can be seen in Figure 5 water holding capacity values increased with ultrasound pretreatment. With 10 min pretreatment at 100 % amplitude ultrasound highest water holding capacity value was seen which is 3.83 times of the control sample.

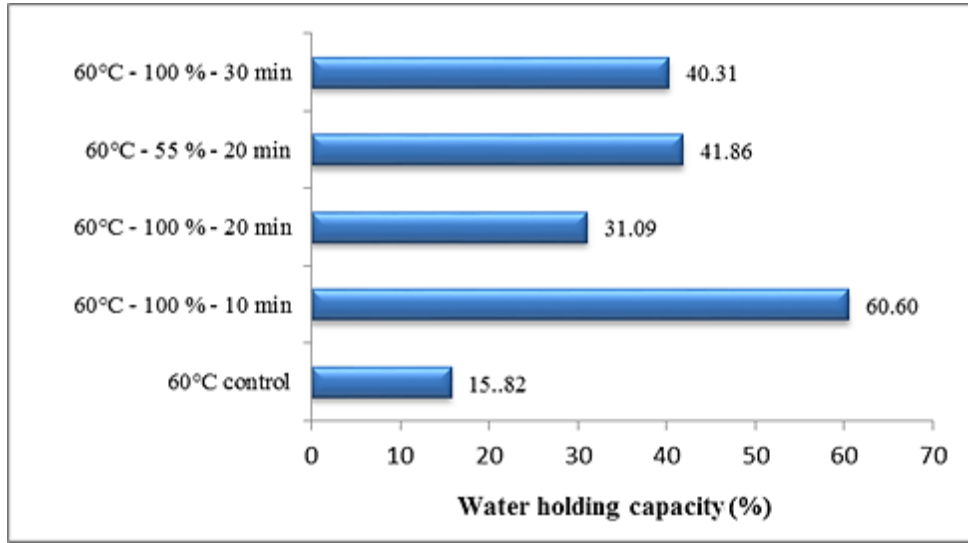


Figure 5. Water holding capacity of apple slices.

### 3.7. Microstructural changes

SEM micrographs of the untreated sample and the ultrasonically pretreated samples at 55 and 100 % amplitudes for 20 min which were dried at 60 °C are given in Figure 6. Ultrasound pretreatment applied at different conditions caused differences in microstructure of the dried apple slices. The apple slices dried without pretreatment have more organized cell structures in comparison with pretreated samples. Disruption of cell walls and cell deformation occurred in apple tissues in consequence of acoustic cavitation which can be seen clearly in Figure 6b-c.

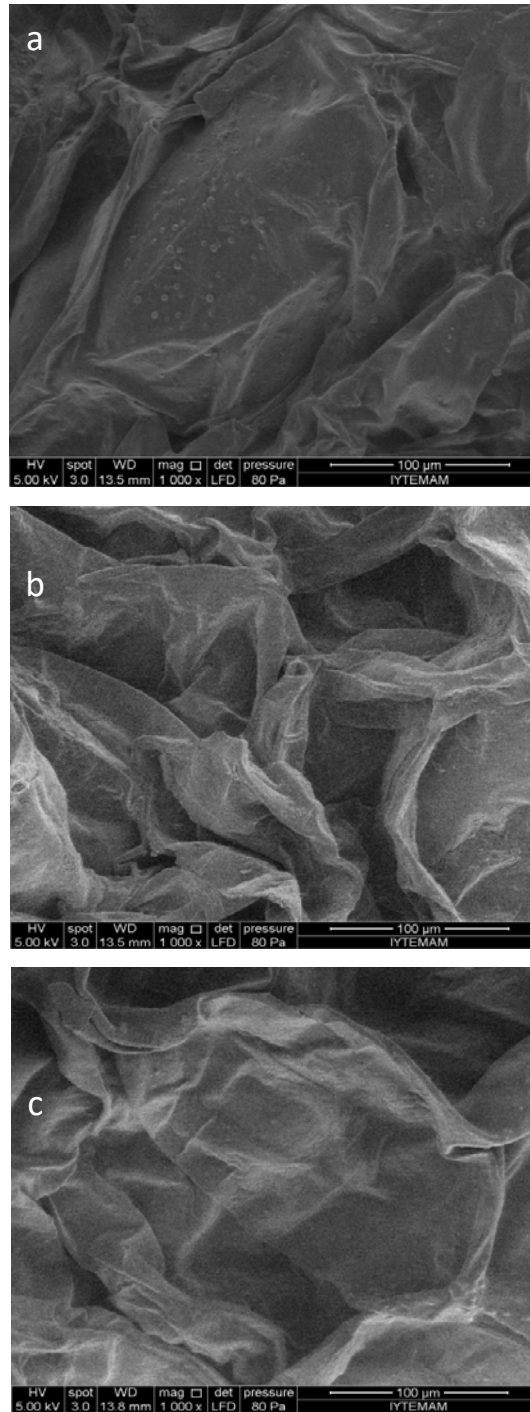


Figure 6. SEM micrographs of apple slices dried at 60°C: (a) Control (b) 20 min sonicated with 55 % amplitude (c) 20 min sonicated with 100 % amplitude.

#### 4. Discussion and Conclusion

Rise in temperature of surrounding water during ultrasound pretreatment (Table 2) is due to the fact that the magnitude of acoustic energy transferred into the medium (surrounding water and apple slices) increases with increasing pretreatment time and ultrasound amplitude. Similar temperature rises based upon ultrasound energy were reported in the literature for mushroom, Brussel sprout and cauliflower (Jambrak et al., 2007).

Associated with ultrasound pretreatment, mass loss in the apple slices occurred as a result of soluble solid loss from the samples due to concentration gradient caused by the sugar content of the samples (Table 2). Ultrasonically pretreated pineapple (Fernandes et al., 2009) soluble solid loss also thought to be based on sugar content of fruit.

As seen in the drying curves of ultrasonically pretreated apples, only the falling rate drying period was observed in all of the samples (Figure 1). Therefore, diffusion is the major physical mechanism of mass transfer in the apple samples. The same result was reported in hot air drying of Granny Smith apple (Andres et al, 2004) and ultrasound assisted convective drying of orange peel (Mello et al., 2020). Contreras et al. (2008) indicated that the Page and Modified Page models were represented the drying kinetics of apple products better than the other models like our study. Also, Page model successfully described the drying kinetics of ultrasonically pretreated pumpkin (Rojas et al., 2020), raspberry fruit (Szadzińska et al., 2019) and banana (Azoubel et al., 2010).

Shrinkage of food materials was an undesirable result of drying for consumer because of the changes in shape and loss of volume (Senadeera et al., 2020). For this study ultrasound pretreatment reducing effect on shrinkage ratio can be considered as a positive effect on dried product quality. Similar results were reported for hot air drying of Mirabelle plum (Dehghannya et al., 2016) and freeze drying of quince slices (Yildiz and İzli, 2019). The falling tendency in shrinkage values with ultrasound application might be due to microstructure damages which improve water diffusivity in apple tissue (Rodriguez et al., 2014). The relation between shrinkage and microstructural changes depending on ultrasound applications were also discussed for orange peel (Garcia-Perez et al., 2012).

Rehydration is a quality characteristic of dried foods and agricultural products that aims to recover the properties of raw materials which give information about the damage caused on the material due to drying and pretreatment methods (Cunningham et al., 2008). As a result of the structural deformation occurred in ultrasound pretreatment, increasing the pretreatment time and ultrasound amplitude accelerated the water gain (Figure 3). Wang et al. (2019) also reported that rehydration kinetics of kiwifruit was closely associated with microstructural changes that occurred after ultrasound pretreatment.

Rehydration kinetics of ultrasonically pretreated dried apples (cv. Idared) (Nowacka et al., 2012) and carrots (Ricce et al., 2016) were also well described by Peleg model with  $R^2$  values above 0.99. Peleg model constant  $k_1$  and  $k_2$  which related to mass transfer rate and final equilibrium moisture content, affected by pretreatment and drying conditions. The lower value of  $k_1$  means faster moisture gain. In comparison to other rehydrated apple samples, 10 min sonicated sample with 100 % amplitude ultrasound had the lowest  $k_1$  value which indicates the highest rehydration rate.  $k_2$  changed oppositely with the equilibrium moisture content. So, the 30 min sonicated sample with 100 % amplitude ultrasound had the highest equilibrium moisture content. The value of  $k_2$  was influenced by process temperature and structural changes in rehydrated material (Deng and Zhao, 2008). In this rehydration process, differences in the values of  $k_2$  can only be due to changes in chemical and physical structures of the apple slices during the ultrasound pretreatment just because of the constant rehydration temperature (60°C).

Higher rehydration ratio with increasing ultrasound pretreatment time and amplitude can be explained by the increases in pore formation which was caused by cavitation (Figure 4). Rehydration capacity of apple (Mothibe et al., 2014) and sea cucumber (Duan et al., 2008) were improved significantly by ultrasound pretreatment. Similarly, during the ageing of beef; sonicated samples reached higher water holding capacity values (Stadnik et al., 2008).

Researchers reported that the cavitation generated by the ultrasonic energy creates microscopic channels and facilitates the diffusion of water for carrot (Chen et al., 2016) and purple sweet potato (Liu et al., 2017). This situation improves the mass transfer by generating alternative diffusion pathways which resulted in lower drying time and shrinkage (Dehghannya et al., 2016). Increasing the ultrasound amplitude flattened the cells and collapsed the apple tissues. Similar tissue changes based on ultrasound applications were also found in previous studies for Fuji apples (Deng and Zhao, 2008) and pineapple (Fernandes et al., 2009).

In conclusion, ultrasound application affects water transport in food tissue so the drying rate increases, the drying time and shrinkage decreases. However, there is a need for further studies for the optimization of ultrasound application conditions in order to produce different products at higher quality and nutrient values at shorter drying times.

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