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A COMPARATIVE STUDY BETWEEN FRESH AND DRIED WALNUT BASED ON INDUSTRIAL PROCESSING

Onur Taskin[1,2]*, Zhongli Pan[2]
[1]Department of Biosystems Engineering, Faculty of Agriculture, Bursa Uludag University, Bursa, TÜRKİYE
[2]Department of Biological and Agricultural Engineering, University of California, Davis/CA, USA

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ABSTRACT
Post-harvest processes of walnuts in the industry are preliminary before consumption. In this study, the dimensions, nut density, terminal velocity, drag coefficient, color, oil quality, protein content, water permeation pathway, and microstructure variation of the fresh and dried walnut samples were researched. The results indicated that every physical and aerodynamic property of fresh samples (except drag coefficient) was a higher value than dried walnuts. In terms of total color difference, the difference was calculated as 6.3. However, no significant ($p<0.05$) difference was determined between samples in AV, PV, and protein content. By comparison of scanning electron microscopy images, both fresh and dried walnut kernels of the surface had a smooth surface and no apparent porous structure. In conclusion, it was determined that the industrial drying method did not affect the internal structure while affecting the external structure.
Keywords: Walnut drying, dimensions, color, oil, microstructure, quality

ENDÜSTRİYEL İŞLEME ESASLI TAZE VE KURU CEVİZİN KARŞILAŞTIRILMASI

ÖZ
Endüstride cevizin hasat sonrası işlemleri tüketim öncesinde ön hazırlık niteliğindedir. Bu çalışmada taze ve kurutulmuş ceviz örneklerinin boyutları, tane yoğunluğu, kritik hızı, sürtünme katsayısı, rengi, yağ kalitesi, protein içeriği, su geçirgenlik yolu ve mikroyapı değişimleri araştırılmıştır. Sonuçlar taze ceviz örneklerinin tüm fiziksel ve aerodinamik özelliklerinin (sürtünme katsayısı hariç) kuru cevizlere göre daha yüksek değerde olduğunu göstermiştir. Toplam renk farkı 6.3 olarak hesaplanmıştır. Ancak yağ kalitesi ve protein içeriği açısından örnekler arasında anlamlı bir fark saptanmamıştır ($p<0.05$). Taramalı elektron mikroskopu görüntüleri karşılaştırıldığında, hem taze hem de kurutulmuş ceviz yüzeyinin pürüzsüz bir yapıya sahip olduğu ve belirgin gözenekli bir yapıya sahip olmadığı görülmüştür. Sonuç olarak, endüstriyel kurutma yönteminin dış yapıyı etkilerken içyapıyı etkilemediği tespit edilmiştir.
Anahtar kelimeler: Ceviz kurutma, boyut, renk, yağ, mikro yapı, kalite

* *Corresponding author* / Yazışmalardan sorumlu yazar
E-mail: onurtaskins@gmail.com Tel: (+90) 224 294 16 02, Fax: (+90) 224 294 14 02

Onur Taskin; ORCID no: 0000-0002-5741-8841
Zhongli Pan; ORCID no: 0000-0002-0914-4006

INTRODUCTION

Walnuts hold the distinction of being the most prevalent variety of tree nuts on a global scale. They are extensively grown for commercial purposes across various regions, including the United States, eastern Asia, western South America, southern Europe and northern Africa (Martinez et al., 2010). In 2021, approximately 3,500,172 tons of walnuts (in shell) were produced globally. The top producers are China, with an annual production of 1,100,000 tons, followed by the United States (657,710 tons) and Iran (386,976 tons). Türkiye with 325,000 tons production increased the walnut cultivation area from 60,496 ha in 2001 to 153,520 ha in 2021 whereas the walnut cultivation area in Iran decreased from 61,795 ha in 2001 to 53,504 ha in 2021 (Mao and Wang, 2021; FAO, 2023). These data show that Türkiye is going to lead its region in the near future.

Walnuts play a significant role in the food industry due to their considerable economic value as a crop. In order to obtain the edible part of the walnut, which is the kernel or meat, several essential steps are involved, including harvesting, hulling, drying, and shelling (Khir and Pan, 2019). As part of a standard post-harvest procedure in the industry, mechanically dehulled walnuts are typically immersed in water for varying durations to remove surface dirt. Subsequently, they are moved to continuous system dryers (Chen et al., 2020a). On the other hand, the increasing production of walnuts is driving a growing demand for large-scale drying technologies within the industry (Taj et al., 2023).

The primary commercial form of walnuts is in the dried state, although consumers also appreciate the distinctive taste of fresh walnuts. However, the drying process introduces certain alterations to the flavor and bioactive constituents of walnuts, thereby impacting their nutritional value and antioxidant activity. Moreover, the quality of walnut kernels varies significantly due to diverse factors such as variety, climate, and processing techniques (Wang et al., 2022).

The purpose of this research was to examine the industrial processing of walnuts and conduct a quality analysis to determine the alterations in fresh and dried walnut kernels. The specific aims were as follows: (1) Investigate the dimensions, nut density, terminal velocity, and drag coefficient of walnuts; (2) Determine the color, oil quality, and protein content changes of walnuts; (3) Research the soaking/washing process on water permeation pathway of walnuts; (4) Present the microstructure images of dried walnuts.

MATERIAL AND METHODS

Walnuts (Chandler cultivar) were harvested in 2021 from the fields of Glenn, California, USA. The orchard was planted in distance between rows and distance between trees in a row of 7.6 and 4.5 m, respectively. The soil profile of the orchard is called the Zamora series, which consists of very deep, well-drained soils that formed in alluvium from mixed rocks. The average daily results of the whole production season for the ambient temperature, relative humidity, precipitation, solar radiation, and max wind speed were 17.38 °C, 52.36 %, 0.30 mm, 237.11 W/m^{2,} and 3.42 m/s, respectively.

Fresh samples were kept in plastic bags under refrigeration conditions (4 ± 0.5°C) until quality experiments were done. The fresh walnut samples were initially found to have a moisture content of 11.33 (w.b.). A stadium-type dryer at 44 °C was used to drop the moisture content of the walnut samples to 8.51 (w.b.).

Dimensions

Using a digital caliper with a precision of 0.001 mm, the major diameter or length (L), intermediate diameter or width (D_1), and minor diameter or thickness (D_2) of the chosen walnuts were measured to determine the dimensions of the principal axes. Equations 1 and 2 were employed to calculate the geometric mean diameter (D_g) in millimeters and sphericity (φ).

$$D_g = (L \times D_1 \times D_2)^{0.333} \quad (1)$$

$$\varphi = \frac{(L \times D_1 \times D_2)^{0.333}}{L} \quad (2)$$

$$A_p = \frac{\pi}{4} D_g^2 \quad (3)$$

A_p represents the projected area measured in mm², while D_g is the geometric mean diameter in mm. The major (L), intermediate (D_1), and minor (D_2) diameters are denoted in mm (Khir et al., 2014).

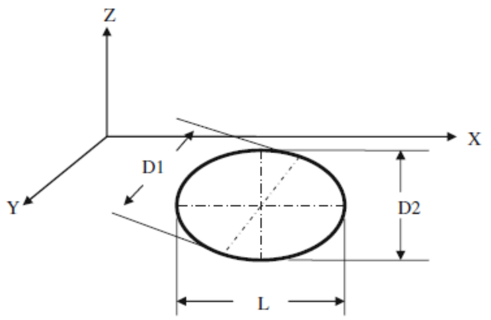


Figure 1. Three primary axial dimensions of walnuts (Khir et al., 2013)

Nut density

To determine the individual nut density (V), the weight and volume of each nut were determined. Each walnut's weight was measured using a precise (accuracy of 0.01g) electronic balance (Denver Instrument, Arvada Co., USA). The volume of each walnut was calculated using Equation (4) (Khir et al., 2014).

$$V = \frac{\pi}{6}(\mathrm{L} \times D_1 \times D_2) \quad (4)$$

Terminal velocity

A cylindrical air column made of transparent plastic with a 10 cm diameter and 100 cm height was used to measure the terminal velocity of individual walnuts. The vertical airflow in the column was produced by a centrifugal fan (Dayton electric blower MFG Co. Chicago, USA). Three layers of mesh screens were used to distribute airflow homogeneously. A motor speed regulator (Dayton, MFG, Co. Niles, IL, USA) was operated for the airspeed and a digital anemometer (Anemo-thermometer, Friendswood, TX, USA) having a 0.1 m/s sensitivity was used to define the air velocity. Each walnut was set at 50 cm height and once the walnut lifted off the screen, the velocity at which the nut was barely lifted off the screen was regarded as the terminal velocity (Khir et al., 2014).

Drag Coefficient

In order to characterize the aerodynamic characteristics of walnuts in an air stream, the drag coefficient (Cd) was calculated using the following Equation.

$$C_d = \left(\frac{2mG}{A_p \rho V_t^2}\right) \quad (5)$$

In equation (5), the drag coefficient (Cd) is a dimensionless value, *m* is the mass of nut (kg), *G* is the acceleration due to gravity (m/s²), V_t is the terminal velocity (m/s), *Ap* is the projected area (m²), and ϱ is nut density (kg/m³) (Khir et al., 2014).

Color

The color of walnuts was measured using a colorimeter. The color measurement device used is a tristimulus colorimeter, designed to assess color indices specified by the CIE (International Commission on Illumination). The L* index represents the lightness component, ranging from 0 to 100, while the a* and b* parameters represent the two chromatic components, varying from green to red and from blue to yellow, respectively, with a range from -120 to 120. Additionally, the total color difference (*ΔE*) was computed as a single value, considering the disparities between the L*, a*, and b* values of the dried sample and the fresh. This calculation is based on Equation (6), where L*, a*, and b* are the measured values, and L, a, and b represent the fresh walnuts (Taj et al., 2023). To describe the visual color appearance more effectively, two parameters, the hue angle (*h**) and chroma (*C*), were calculated using Equations (7) and (8), respectively.

$$\Delta \mathrm{E} = [(L^* - L)^2 + (a^* - a)^2 + (b^* - b)^2]^{\frac{1}{2}} \quad (6)$$

$$h^* = \left(tan^{-1}\frac{b}{a}\right) \quad (7)$$

$$C = (a^2 + b^2)^{\frac{1}{2}} \quad (8)$$

Quality analyses of walnut oils

Walnut oil extraction was carried out using hexane in a MaxQ 4000 shaker (Thermo

Scientific, Waltham, MA, USA) operating at 3.33 Hz and 4 °C for a duration of 1 hour. Subsequently, the oil and solvent were separated from the solids by employing vacuum filtration and a rotary evaporator (Büchi R-3, Switzerland). The PV (Peroxide Value) and AV (Acid Value) of the oil samples were determined following the AOCS standard methods Cd 8–53 and Ca 5a-40, respectively, using a Metrohm titrino plus 848 potentiometric titrator (Chen et al., 2020b).

The peroxide value is expressed as the amount of sodium thiosulfate:

$$PV = \frac{(\mathrm{S}-\mathrm{B}) x\ \mathrm{N} x\ 1000}{mass\ of\ test\ portion\ (g)} \quad (9)$$

Where S is the sodium thiosulfate amount used for samples (mL), B is the sodium thiosulfate amount used for blanks (mL) and N is the sodium thiosulfate solution concentration (0.025 N).
The acid value is expressed as mg KOH per g of fatty acids:

$$AV = \frac{(\mathrm{S}-\mathrm{B}) x\ \mathrm{N}}{mass\ of\ test\ portion\ (g)} \quad (10)$$

Where S is the Potassium hydroxide amount used for samples (mL), B is the Potassium hydroxide amount used for blanks (mL), and M=56.1 in this case.

Protein content
Kjeldahl (Nx6.25) method was used for protein analysis of the samples (Çelik et al., 2011). Samples burned in the combustion set (K-437, Buchi, Switzerland) with sulfuric acid and catalyst (K_2SO_4+$CuSO_4$+Se) and were distilled in an alkaline environment (K- 350, Buchi, Switzerland). The amount of ammonia (NH_4) produced was titrated with sulfuric acid and the amount of nitrogen was measured. The results obtained will be multiplied by a factor of 6.25 to calculate the protein content of the samples (Bremmer, 1965).

Water permeation pathway
Randomly selected walnut samples from the same batch were divided into two sets: the experimental group and the control group. In the experimental group, the stem pore of the walnuts was sealed using water-resistant epoxy glue to simulate walnuts without the natural stem pore. On the other hand, the control group walnuts were left untreated, resembling walnuts with their original stem pore intact.

After the sealing process, there was a minimal change in walnut weight, less than 1%, and the moisture content (MC) of the glue was disregarded. Subsequently, both groups were left at room temperature for 30 minutes to allow the glue to solidify.

To create the fluorescence tracking solution, a light yellow food-grade dye (GloMania, USA) was dissolved in water. The walnuts were then immersed in this fluorescent solution and continuously stirred to simulate the industrial washing process. Following the soaking period, five samples were taken out from the solution, carefully wiped clean with a paper tissue, and subsequently placed inside a cool desiccator for a duration of 2 hours. Next, the samples were cut through using a band saw in various directions (cross-section, alongside the suture, and perpendicular to the suture), allowing the kernel to be meticulously separated into halves. To activate the fluorescence dye in the walnuts, a USPAR F15T8 UV light was used. For comparison purposes, the identical samples were also positioned under an incandescent lamp for imaging. Photographs of the water permeation pathways within each individual walnut were captured and observed using a digital camera (Chen et al., 2020a).

Microstructure
To study the microstructures of walnuts, the walnuts were cracked open, and the kernels were carefully separated and broken into smaller pieces. Scanning electron microscopy (SEM - Nova™ NanoSEM 430, FEI, USA) was conducted with an accelerating voltage of 10 kV and a magnification of 200×. Before conducting the SEM observations, the samples were affixed to an aluminum stub using double-sided adhesive tape and then coated with a layer of gold using the EMS150R ES sputter coater (Quorum

Technologies Ltd., Newhaven, UK) for a duration of 5 minutes (Chen et al., 2020c).

Statistics

MS-Excel program was used to process the data to be obtained in triplicate. Besides, Statistical analyses were performed using JMP software (Version 7.0, SAS Institute Inc., Cary, NC, USA) with the assistance of a randomized plot factorial design. The least significant difference test (LSD) was applied, considering a significance level of 5%.

RESULTS AND DISCUSSION

Dimensions, Nut density, Terminal velocity and Drag Coefficient

The mean values and standard deviations for the dimensions, nut densities, terminal velocity and drag coefficient of walnuts (fresh and dried) are presented in Table 1. The results of dimensions indicated the axial dimensions of L, D_1 and D_2 for fresh walnuts were greater than dried walnuts. Additionally, the D_g values of fresh walnuts are higher than dried walnuts. The shape of the walnut is very close to spherical. Due to the loss of water due to drying, the weight of the dried walnuts was found to be lower. Also, the terminal velocity of fresh walnuts was greater than that of dried walnuts. The drag coefficient increased after drying and the airstream velocity required to suspend individual walnut was decreased. Altuntas and Ozkan (2008) studied three Turkish walnut varieties (Bilecik, Kaman, and Sebin). They reported that the length, width, thickness, and geometric mean diameters were 38.58, 33.41, 31.16, and 34.11 mm, respectively, for the Bilecik cultivar; 44.66, 36.68, 34.42, and 38.20 mm for Kaman cultivar, and 36.57, 29.17, 30.75, and 31.89 mm for Sebin cultivar. The unit mass (g) and sphericity (%) of walnuts were 11.40, 88.48; 16.54, 85.58; and 9.75, 87.28 for Bilecik, Kaman and Sebin cultivars, respectively. The bulk density (kg/m3) and volume (cm3) of Bilecik, Kaman and Sebin walnut cultivars were 235.5, 10.68; 225.10, 12.57, and 240.8, 8.75, respectively. Also, the results by Ercisli et al. (2011) presented the two walnut cultivars widely grown in Turkey, namely 'Maraş-18' and 'Yalova-1'. The average length (mm), width (mm), thickness (mm), geometric mean diameter (mm), mass (g), sphericity (%), and surface area (cm^2) determined 41.57, 34.18, 33.74, 36.33, 12.44, 87.41, and 41.48 for cv. 'Maraş-18'. The corresponding values were 45.48, 34.06, 32.29, 36.83, 12.70, 81.08, and 42.66 for cv. 'Yalova-1'. These findings show a slight difference between the Chandler cultivar presented in this research.

Table 1. Properties related to the physical and aerodynamic characteristics of walnut samples

	Fresh	Dried
L (mm)	39.79 ± 4.22	37.60 ± 2.14
D1 (mm)	34.12 ± 1.67	32.23 ± 3.20
D2 (mm)	32.84 ± 1.53	31.57 ± 1.39
D_g (mm)	35.23 ± 2.38	33.49 ± 1.22
φ	0.92 ± 0.33	0.89 ± 0.05
Ap (mm^2)	978.92 ± 113.99	884.39 ± 100.37
W (g)	13.00 ± 1.91	10.56 ± 2.30
V (mm^3)	23421.19 ± 3705.13	20103.63 ± 3086.73
TV (m/s)	18.75 ± 1.24	17.47 ± 1.64
C_d	134.23 ± 22.06	149.05 ± 33.80

Color

As seen in Figure 2, the color parameters of the walnuts were affected by the drying application. In comparison to the fresh walnut samples (62.67), the dried walnut samples exhibited a darker value (68.82). Furthermore, the fresh walnut sample contained lower a* values (6.54) than the dried walnut samples (7.30). There were statistically significant differences determined for L* and a* values of the fresh and dried samples (p<0.05). The b* value was fresh and dried samples were 27.07 and 26.63 without any significant changes, respectively (p<0.05). The hue angle of the fresh walnut samples (76.47) was closely followed by dried values (74.73). Considering the hue angle of fresh and dried samples presented a significant difference (p<0.05). The C values (27.87 and 27.62, respectively) were almost similar for fresh and dried walnut samples and there were significant changes between the samples (p<0.05). Compared to the fresh walnut sample, the total color difference was determined as 6.3. Altuntas and Erkol (2009) reported the L*, a*, b*, hue angle, and chroma values (color properties) of Yalova-1, Yalova-3 and Bilecik shelled walnut varieties in different moisture content. Contrary to our study, the 'L' (lightness) color values increased from 50.85 (15.76 %, d.b.) to 55.09 (10.00 %, d.b.) for the Bilecik cultivar; from 39. 85 (23.16 %, d.b.) to 56.49 (11.46 %, d.b.) for the Yalova-1cultivar; from 46.84 (19.47 %, d.b.) to 53.21 (11.25 %, d.b.) for the Yalova-3 cultivar. Furthermore, Hue angle and chroma values of Yalova-1, Yalova-3, and Bilecik cultivars at all moisture levels were slightly lower than Chandler cultivars.

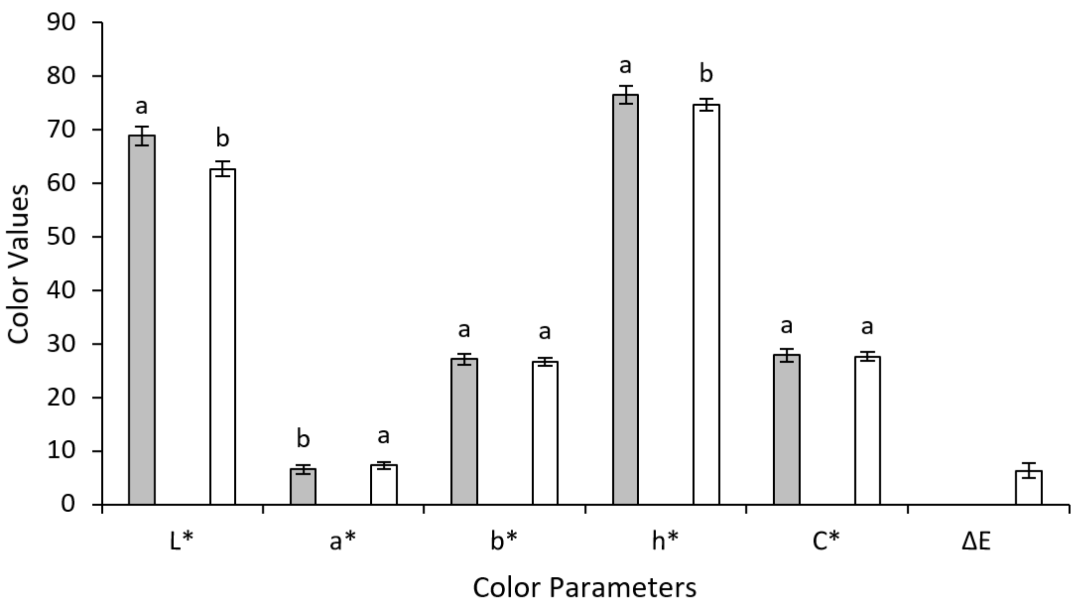
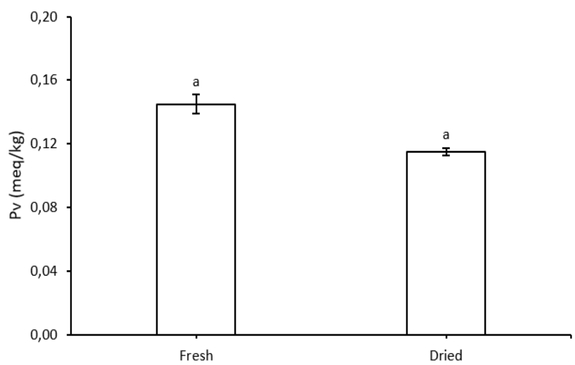


Figure 2. Color values of fresh (grey bars) and dried (white bars) walnuts

Oil quality

The peroxide value and acid value of the fresh and dried walnut oils were researched as shown in Figure 3. For the measurement of peroxide value, the oil sample (1 g) is firstly weighed in a beaker (50 ml), the PV solution (25 ml) is then poured into the beaker and mixed by an electrical agitator. Then the saturated KI solution (1 ml) is added to the solution by 1ml titrator. The solution will be stirred for 1 min and then 10 ml water will be added to the solution immediately. The adding of water is to stop the reaction between KI and oil sample, since the water and acetic are intersoluble, but not with iso-ocatane. Octane dissolves the oil, so the adding of water will separate KI with oil peroxides and achieve the purpose of stopping the reaction. As a result of Equation 9, the PV of fresh walnuts were 0.15, which was slightly higher than the dried walnuts (0.12). However, the drying treatment did not differ significantly (P>0.05). At the same time with PV measurements, the AV were also determined. In a typical procedure of the oil sample (1 g) is firstly weighed in a beaker (50 ml), the AV solution (35 ml) is then poured into the beaker and stirred by an electrical agitator. Then 2 drops of Phenolphthalein solution (colorless) is then added to the solution. After mixing, the KOH solution is added gradually by the titration machine (Titrino plus, model 848, Metrohm Ltd., Switzerland) until the color of the solution turns light purple (which corresponds to approximately pH=10). When the hydrogen ion (H+) is completely neutralized by the hydroxide ion (OH-), the electrical potent of the solution will change suddenly and the value of KOH added is then automatically recorded. As a result of Equation 10, the AV of fresh and dried walnuts were found 0.064 and 0.060, respectively. Compared with the fresh sample, no significant ($p<0.05$) decrease was determined in the dried samples. Pakrah et al., (2022) reported the fresh walnut kernels from Hartley, Damavand, Ronde de Montignac, and Pedro cultivars and dried kernels (sun, 20 °C and 30 °C and) which were grown in Iran. The extracted oil from fresh walnut kernels showed a peroxide value ranging from 0.37 meq/kg to 0.22 meq/kg of oil., with the dried kernels exhibiting a higher oil content when compared to the fresh kernels. Zhou et al., (2018) studied developing an effective drying method for in-shell walnuts (*Juglans regia* L.) with three drying methods. The AV (mg/g) and PV (meq/kg) values of hot air, vacuum, and hot air-assisted radio frequency drying were 0.69 and 0.57, 0.42 and 0.33, 0.44 and 0.38, respectively. The disparity in oil content could be attributed to the country of origin.



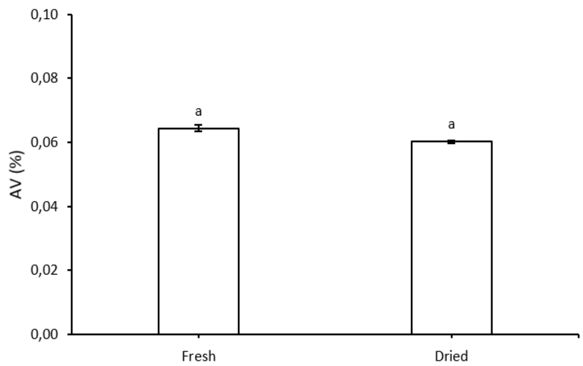


Figure 3. PV and AV of walnuts

Protein content

Figure 4 demonstrates the protein changes in fresh and dried walnuts using industrial drying application. The presented results indicate that the reduced water content during drying led to slightly higher protein levels in the dried samples compared to the fresh ones. The protein contents of fresh and dried samples were determined 13.3 and 13.7%, respectively. There were significant differences between industrial application drying in terms of the contents of protein compared with the fresh samples ($p<0.05$). Pakrah et al., (2021) tested three drying methods (20 °C, 30 °C, sun drying) on the protein content of Chandler variety walnuts. The sun-drying method resulted in a lower protein content (7.99%), while walnut kernels dried at 20°C exhibited a higher protein content (9.81%). Drying reduced protein content compared to fresh walnut (10.79 %). Han et al., (2019) investigated the protein contents of walnut after heat drying (60 ℃, 105 ℃ and 140 ℃) and freeze-drying (–55 ℃) process. As the temperature rose from -55°C to 105°C, the protein values increased by 1.6%. However, with an increase in temperature up to 140°C, a decline in performance was observed. Therefore, the protein content in walnuts reached its highest value at 105°C, reaching 19.6%. The differences in protein content after drying could be explained by drying temperature sensitivity.

Water permeation pathway

During the industrial process, walnut washing time is less than 5 minutes. Therefore, the soaking time was limited to 5 minutes to allow the water to transfer into the walnut samples. Figure 5 presents the alongside cutted non-sealed and sealed walnuts under visible Light and blacklight. The fluorescence dye was run by the ultraviolet and the fluorescence pattern was determined.

Later on soaking times (5 mins) in either wet or dry, no obvious fluorescence pattern was observed within the non-sealed. However, there was a slight water entrance in sealed walnuts. When the initial and final weights of these samples were compared, a weight increase of 0.48% in fresh samples and 4.45% in dry samples was determined. It has been observed that the short-term (5 min) soaking process for cleaning the walnuts in the industry can be considered negligible considering the effect on the fresh product weight increase.

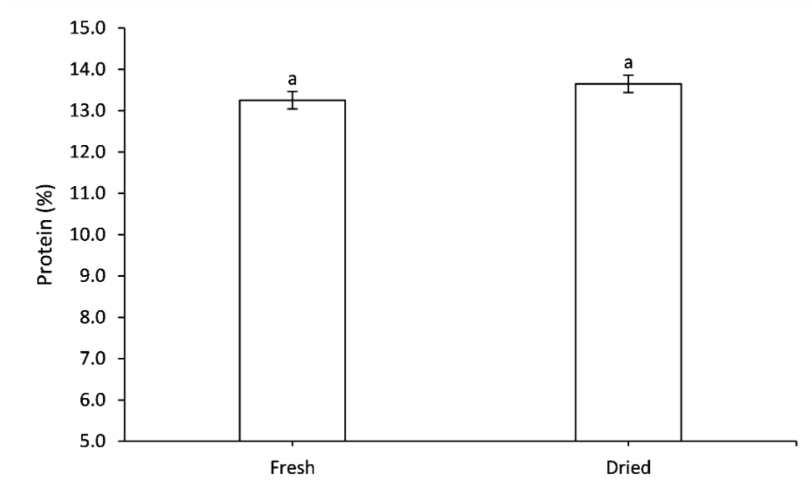


Figure 4. Protein values of walnuts

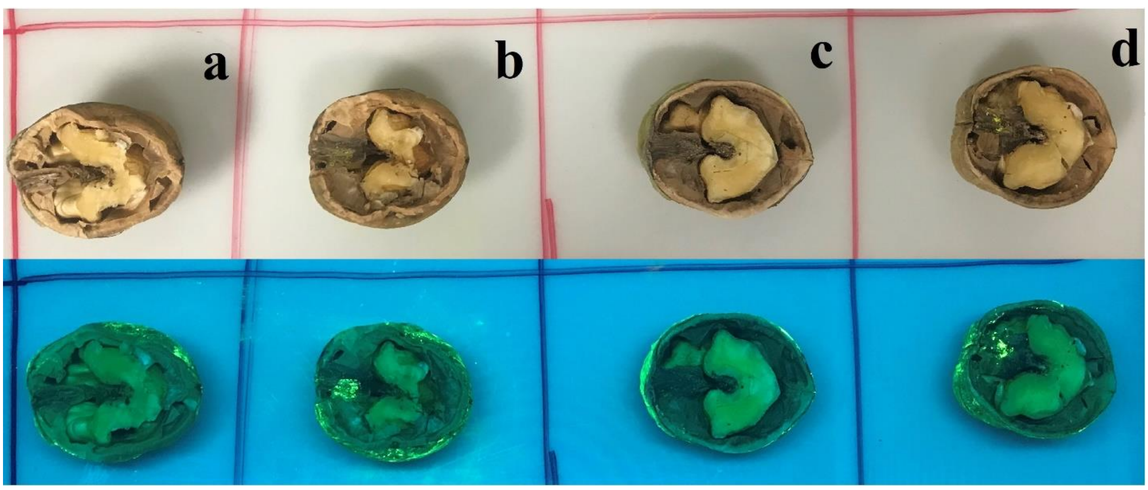


Figure 5. Water permeation of walnuts (fresh before soak (a), fresh after soak (b), dried before soak (c), and dried after soak (d))

Microstructure

The scanning electron microscope (SEM) utilizes a focused electron beam to scan the surface of a sample and generate a high-resolution image. The interaction of electrons in the beam with the sample produces diverse signals, which can be utilized to gather valuable information. SEM images offer essential topography and composition data to companies spanning from microelectronics to food processing, aiding in product quality assurance, failure analysis, and guiding product development efforts. Figure 6 shows the scanning electron microscopy images for the microstructures of the fresh and dried walnut kernel. It was evaluated as a surface and cross-sectional. Both fresh and dried walnut kernels of the surface had a smooth surface and no apparent porous structure. However, the cross-sectional images showed that dried walnut had higher porosity and larger pore size than the fresh ones. On the other hand, a slight deterioration in the cell structure of the dried samples was observed regarding the cross-section image. As noted in the prior report, McArthur and Mattes (2020) observed the internal structure of cotyledons, such as lipid-bearing tissue with transmission electron microscopy (TEM). Although the sizes of walnut, almond, and pistachio cells were comparable (33 μm, 31 μm, and 35 μm, respectively), the raw walnuts exhibited thinner cell walls.

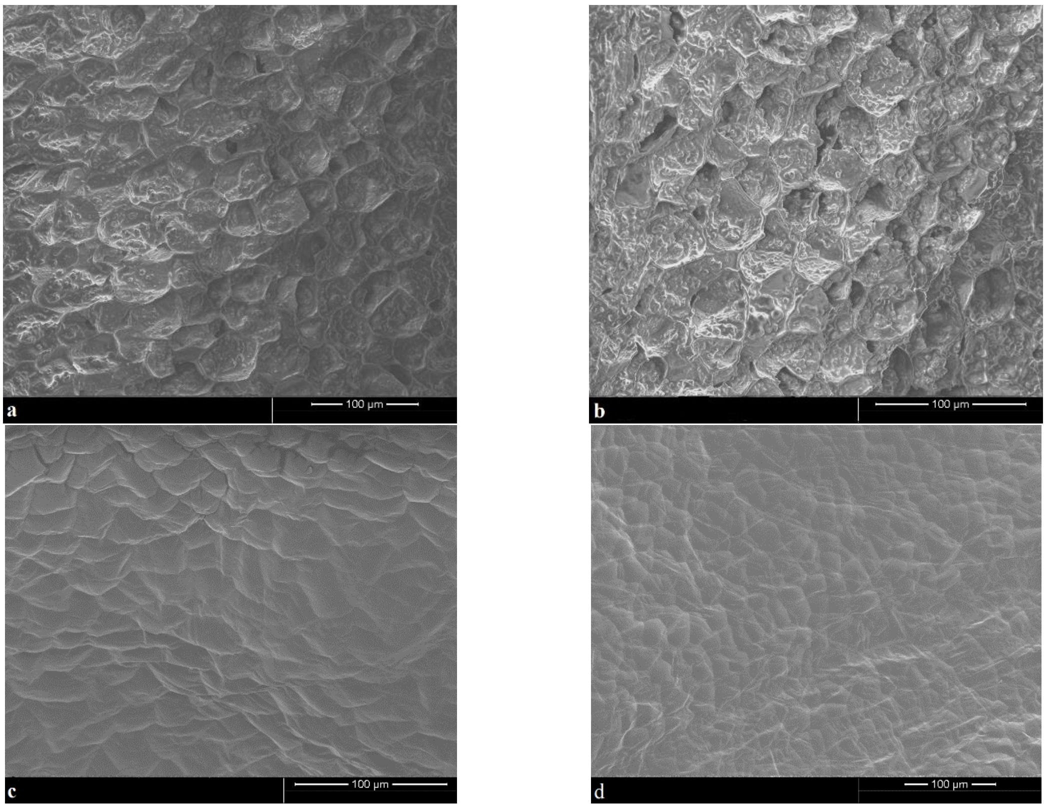


Figure 6. SEM images of walnuts of fresh cross-section (a), dried cross-section (b), fresh surface (c) and dried surface (d)

CONCLUSION

In conclusion, the industrial processing of walnuts was investigated and the quality assessment of fresh and dried walnut kernels was compared. The terminal velocity of fresh walnuts was found greater and with a lighter color value than that of dried walnuts. No significant differences were determined in oil and protein contents between fresh and dried samples ($p<0.05$). The water permeation showed that the weight of fresh samples increased by 0.48%. A slight deterioration in the cell structure of the dried samples was observed regarding the cross-section image. Consequently, the overview of the walnuts with the current drying method used in the industry has been revealed.

CONFLICT OF INTEREST

The authors have declared no conflict of interest.

AUTHORS' CONTRIBUTIONS

Onur Taskin: Literature review, Methodology, Data Curation, Analysis, Writing-original draft. Zhongli Pan: Writing-review and editing.

All authors read and approved the final manuscript.

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