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ORIGINAL ARTICLE

Convective drying of golden delicious apple enhancement: drying characteristics, artificial neural network modeling, chemical and ATR-FTIR analysis of quality parameters

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Abstract
In recent years, many innovative methods have been investigated to provide alternative approaches to the food drying industry, but currently the most widely used method is convective drying. There are difficulties in integrating innovative methods into the food industry due to cost, inapplicability to every food material, or product quality, etc. In addition, it is possible to improve the convective drying method by pre-treating of foods. Thanks to the convective drying method with increased efficiency, shorter drying processes can be achieved. This study investigates the effects of ethanol and citric acid pretreatments on the convective drying process of apple slices and the drying rate, diameter and thickness shrinkage, color properties, total phenolic content (TPC), antioxidant activity (AA), ATR-FTIR spectra, and principal component analysis (PCA) of the dried samples. The results indicate that both ethanol and citric acid pretreatments significantly enhance the drying rate and decrease drying time, with the most favorable outcomes observed for apple slices immersed in an ethanol solution for 20 min. The study employs thin-layer and artificial neural network (ANN) modeling, revealing that ANN modeling outperforms thin-layer models in predicting moisture ratio. Shrinkage ratios in diameter and thickness were observed, but no significant statistical differences are found among the sample groups. The color properties of dried apple slices are influenced by pretreatments. L^* values decreased in the ethanol-pretreated samples, whereas a^* and b^* values increased in all samples. On the other hand, drying process leads to a decrease in TPC and AA. Ethanol pretreatments caused higher losses; lower losses were observed in the citric acid–pretreated and untreated apples slices. ATR-FTIR analysis suggests distinct spectral changes in dried samples, particularly influenced by ethanol and citric acid pretreatments. The ATR-FTIR spectra highlighted shifts in water and carbohydrate levels, proteins, fibers, organic acids, and the occurrence of Maillard reactions throughout the drying process. PCA reveals that samples dried with ethanol and citric acid share a similar plane, while fresh samples and those dried at 60 °C exhibit different arrangements.

Keywords Golden apple · Drying · Neural network · ATR-FTIR · Ethanol · Pretreatment

1 Introduction

Apple (*Malus domestica*) is one of the most consumed fruits in the world and China, the USA, Turkey, Poland, India, the Russian Federation, and Iran are the largest apple-producer countries [1]. Apple production in Turkey was 4.602.517 tons in 2023; this rate has a large share with approximately 18% of other fruits grown in Turkey [2]. According to the Food and Agriculture Statistics (FAOSTAT), while China is the world's largest apple producer in 2021, followed by Turkey [3]. Apple is a health beneficial fruit due to content of several bioactive compounds (such as polyphenols and vitamin C) and having high nutritional value [4]. Fresh fruits and vegetables with moisture content > 80% are classified as highly perishable products [5]. Water content of apples is notified in the range of 80–88% [6]. Thus, apples can be accepted as perishable fruits that prone to post-harvest losses, which poses a major obstacle to providing this nutritious product to the consumer in the best possible way [7]. Convective drying is one of the most common techniques for the preserving foods. After convective drying process, reduction of water activity occurred, thus, less, or

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no microbial growing and enzymatic reactions, reduction of volume and weight, decrease transportation costs and extension of shelf life [8–11]. Nevertheless, long drying time and high drying temperatures negatively affect some food quality characteristics such as nutritional compounds and flavor and cause alterations in shape such as shrinkage [12–16].

Pretreatment can be applied before drying to reduce the quality degradation caused by convective drying and to save energy. Immersion in ethanol and citric acid solution are some of these pretreatments. Recently, the use of ethanol immersion pretreatment has increased due to its ability to dry products without leaving any residues [17]. In the ethanol pretreatment, when ethanol is introduced into the sample, it mixes with water. During drying, the rapid evaporation of the surface ethanol promotes mechanisms that accelerate the drying process. One such mechanism is the Marangoni effect [18]. The Marangoni effect of ethanol creates a surface tension gradient on the sample surface. It dissolves some cell wall components and increases cell wall permeability. Collectively, these effects facilitate the transfer of moisture in the product, leading to enhanced drying [19, 20]. Acid solution immersion pretreatment is frequently used to improve product quality by inactivating enzymes, increasing pigment stability, and changing the texture of agricultural products. When the pH of the medium is reduced to 3, the activity of polyphenol oxidase, which has an optimum working pH of 6–7, can be inhibited, thus reducing the rate of enzymatic browning. However, the stability of pigments such as betalains and anthocyanins increases under acidic conditions and the texture of the product can be preserved due to the chelating properties of acidic solutions [21]. Citric acid, an organic acid, can change tissue properties by altering the gelation, hydrolysis, and depolymerization of pectin, which increases the rate of moisture removal from the tissue [22].

In order to evaluate drying from an engineering point of view, mathematical modeling is needed as well as method development. Thin-layer modeling, which is one of the commonly used mathematical models, makes the drying process predictable and allows the process to be controlled more easily [9]. In addition to this, artificial neural network modeling has been used recently. The correlation between the interconnection of unpredictable input and output process parameters is modeled using the artificial neural network (ANN) computational approach. Artificial neural networks are capable of modeling nonlinear and complex systems that involve a large number of input and output data. The prediction of a neural network is wholly reliant on its structure, which comprises the activation function type, total number of layers, and quantity of hidden layer neurons [1]. Ghasemkani et al. [23] stated that when compared to thin layer models, artificial neural networks demonstrated superior performance in accurately modeling the curves. Onwude et al. [24] reported that ANN modeling method was greater to theoretical methods in describing the drying behavior of pumpkins dried by convective drying. Additionally, even when new experimental data is introduced and alterations are made to the experimental conditions and dataset, the ANN model consistently produces great results. Yıldız et al. [25] reported that the moisture content of banana slices dried by convective drying was predicted at a higher rate by ANN modeling compared to theoretical models.

Fruits and vegetables have a high potential to form waste after harvest if not consumed due to high perishable properties. Finally, both environmental and economic issues can emerge. Additionally, it is important to produce alternative foodstuffs that have high durability, out of season. Drying is one of the best options. Therefore, the aim of this study was to investigate the effect of citric acid and ethanol immersion pretreatments on the drying characteristics of apple slices. For this purpose, quality parameters such as shrinkage ratios, color values, total phenolic content, and antioxidant activity of samples pretreated at different concentrations and times were evaluated. In addition, mathematical and ANN modeling of drying, attenuated total reflection-Fourier transform infrared spectroscopy (ATR-FTIR), and principal component analysis were performed.

2 Material and methods

2.1 Sample preparation for drying experiments

In the current study, apple (*Malus domestica* var. Golden Delicious) were employed for drying experiments. Apple samples were purchased from a local market Şebinkarahisar, a district of Giresun province of Turkey. Purchased apples were selected as possible as in equal sizes. Following washing for foreign materials and cutting by using a stainless-steel slicer, the apple slices had a thickness 4 ± 0.1 mm and a diameter of 6.42 ± 0.13 cm. Thickness and diameter of the samples were measured by a digital caliper. The initial moisture content of the fresh samples was calculated as $85.8 \pm 0.17\%$ on a wet basis (w.b.). Apple samples used in the study are given in Fig. 1.

2.2 Pretreatments

For ethanol pretreatments, apple slices were immersed in solutions consisting of 50% and 100% ethanol (Isolab, 99.9%) for 10 and 20 min, according to Rojas et al. [18]. The immersion ratio was 1:4 (w/v). At the end of the immersion, the samples were immediately filtered and the excess liquid on the surface was removed by using a filter paper. The sample codes were specified as 60 °C + 50 ET 10, 60 °C + 50 ET 20, 60 °C + 100 ET 10, and 60 °C + 100 ET 20.

Fig. 1 The apple samples used in the study

Citric acid pretreatment was carried out according to Tepe [9]. The sliced apple samples were immersed in solutions containing 1% citric acid for 2 and 4 min. Following the filtration of the samples after immersion, the excess solution on the surface was removed with a filter paper. The codes of the samples were identified as 60 °C + CA 2 and 60 °C + CA 4.

2.3 Conditions of drying experiment

In the drying experiments, 100 g of apple slices was dried by using drying tray in the drying oven (Nüve, FN 400) at a temperature of 60 °C ± 1 °C. The weight change was periodically measured during the drying experiments via digital weight measure with a precision of 0.01 g. The experiments were completed until the moisture content of the apple slices achieved to 10% on a w.b. or 0.1 g water g dry matter^{-1} on a dry matter (d.m.). All drying experiments were carried out in triplicate. The same conditions were used for the drying experiments of the untreated and pretreated samples.

2.4 Drying characteristics of the apple slices

2.4.1 Moisture content

During the drying experiments, changes in moisture content were monitored at intervals and calculated by applying Eq. (1), as proposed by Demiray et al. [11].

$$M_t = \frac{m - DM}{DM} \tag{1}$$

where "M_t" denotes the moisture content of the sample at any given time (g water g^{-1} d.m.), "m" represents the weight (g) of the sample, and "DM" signifies the dry matter content (g) of the sample.

2.4.2 Moisture ratio

Moisture ratio (MR) of the apple slices was calculated by Eq. (2).

$$\mathrm{MR} = \frac{\mathrm{M}_t - M_e}{M_i - M_e} \tag{2}$$

where "M_e" signifies the equilibrium moisture content, "M_t" represents the moisture content at any time, and "M_i" denotes the initial moisture content. The value of "M_e" was disregarded in the calculations due to its insignificance when compared to both "M_t" and "M_i" [26].

2.4.3 Drying rate

Drying rate (DR) was computed by Eq. (3) [11].

$$DR = \frac{M_t - M_{t+\Delta \mathrm{t}}}{\Delta \mathrm{t}} \tag{3}$$

The time difference between two measurement points is denoted by "Δ_t," whereas the moisture content at the given time difference is represented by "$M_t + \Delta_t$."

2.4.4 Effective moisture diffusivity

Fick's second law, proposed by Crank [27], was employed to analyze an infinite slab object with a constant moisture diffusivity represented by Eq. (4).

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n+1)^2}\exp\left(-(2n+1)\pi^2\frac{D_{eff}t}{4L^2}\right) \tag{4}$$

Equation (5) was employed to compute the effective moisture diffusivity (D_eff), where the drying time (t) and the half-thickness of the fresh sample (L) are utilized as variables. In cases where the drying time is significantly long ($n = 1$), a simplified version of Eq. (6) can be employed, as recommended by Demiray et al. [11].

$$ln(MR) = \ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2}{4L^2}D_{eff}t\right) \tag{5}$$

A linear relationship between the natural logarithm of MR and drying time (Eq. (5)) yields a straight line, with the slope of this line representing Eq. (6) [11].

$$\mathrm{Slope} = -\frac{\pi^2}{4L^2}D_{eff} \tag{6}$$

2.5 Mathematical modeling

The calculations for root mean square error (RMSE), reduced chi-square (χ^2), and determination coefficient (R^2) were performed using Eqs. (7), (8), and (9) respectively, according to Tepe [9].

$$\mathrm{RMSE} = \left[\frac{1}{N}\sum_{i=0}^{N}\left(MR_{pre,i} - MR_{exp,i}\right)^2\right]^{\frac{1}{2}} \tag{7}$$

$$\chi 2 = \frac{\sum_{i=0}^{N}\left(MR_{pre,i} - MR_{exp,i}\right)^2}{N-n} \tag{8}$$

$$R^2 = \frac{\sum\left(MR_{pre} - \sum MR_{exp}\right)^2}{\sum\left(MR_{pre,av} - \sum MR_{exp}\right)^2} \tag{9}$$

The data were represented by predicted moisture ratios (MR_{pre}) and experimental moisture ratios (MR_{exp}). N and n denote the constants of the thin-layer drying models and the number of observation data points, respectively. MATLAB software (R2015a, version 8.5) and its non-linear curve fitting toolbox with the trust-region approach were used to calculate statistical parameters and fit curves. The model selection approach was based on the higher the R^2 values, the lower the χ^2 values, and the lower the RMSE values [11]. Table 1 shows the thin-layer models used in the study.

2.6 Artificial neural network modeling

The Levenberg–Marquardt back-propagation technique, which is a highly recommended by Omari et al. [33], was employed in the study by using the MATLAB software (R2015a, version 8.5) with a Neural Net Fitting Toolbox. An artificial neural network (ANN) model was set up for the current study using the following inputs, i.e., ethanol or citric acid concentration, pretreatment time, and drying time, and output, i.e., moisture ratio. In accordance with the recommendation of Omari et al. [33], the tansig function was selected as the transfer function for the hidden layer. Upon examining mean square error (MSE) and R^2 at varying neuron counts, an ANN design including three neurons in the hidden layer was determined. ANN architecture is illustrated in Fig. 2. As previously mentioned, three drying tests were carried out. For ANN training and validation, the drying experiments' experimental data were divided into 50% training data, 25% validation data, and 25% testing data. These data sets were found to have the highest MSE and R values among the ANN models run on different training, validation, and testing data. Therefore, the data sets of this study were applied as specified. Training was completed at termination conditions to avoid overfitting. Termination conditions were a maximum of 1000 epochs, 6 validation assays, and 1e-07 performance gradient [25]. To evaluate the ANN model's performance, RMSE and R^2 values were calculated to compare the model's estimated data with the experimental data.

Table 1 Thin-layer models selected in the study

Model name	Model	References
Lewis	$exp(-kt)$	Lewis [28]
Henderson and Pabis	$aexp(-kt)$	Henderson and Pabis [29]
Page	$exp(-kt^n)$	Page [30]
Parabolic	$a + bt + ct^2$	Doymaz [31]
Midilli and Kucuk	$aexp(-kt^n) + bt$	Tunckal and Doymaz [32]

2.7 Color evaluation

The color values of apple samples were measured before and after drying. Color measurements were made using 3NHN-R10QC (China). The color measurements were carried out at five different locations on each sample's surface. The difference between the fresh and dried color values of the product was calculated using the total color differences (ΔE) method. For this purpose, Eq. (10) was used. The ΔE value quantifies the magnitude of color change, with higher values indicating more significant color alterations during drying [9].

$$\Delta E = \sqrt{\left(L_0^* - L^*\right)^2 + \left(a_0^* - a^*\right)^2 + \left(b_0^* - b^*\right)^2} \tag{10}$$

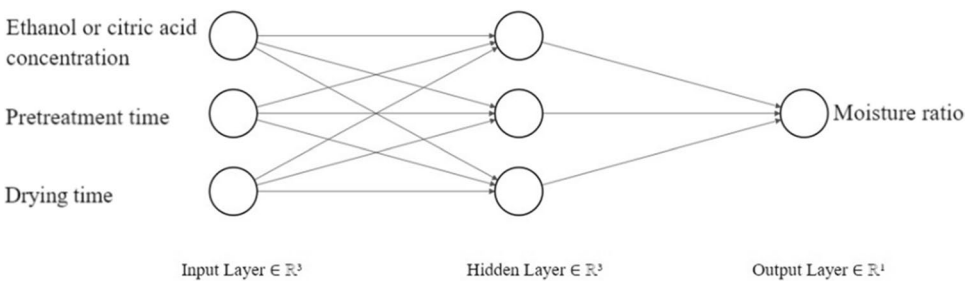

Fig. 2 ANN architecture

2.8 Shrinkage properties

In the current study, dimensional changes were evaluated during drying by measuring diameter shrinkage (DS) and thickness shrinkage (TS) according to Granella et al. [34]. DS indicates the percentage reduction in diameter, while TS represents the percentage reduction in thickness, comparing dried samples to fresh ones. The measurements were performed with the digital caliper. Equation (11) and Eq. (12) were used to calculate reduction rate of diameter and thickness of the samples, respectively. Ten apple slices for each treatment were analyzed to assess the extent of shrinkage.

$$DS = 100 - \left(\frac{D_f}{D_i}100\right) \tag{11}$$

$$TS = 100 - \left(\frac{T_f}{T_i}100\right) \tag{12}$$

D_i and T_i represent the initial size of the samples, whereas D_f and T_f are the final size of the samples after drying process.

2.9 Total phenolic content and antioxidant activity analysis

The investigation into total phenolic content (TPC) and antioxidant activity (AA) involved the preparation of a methanolic extraction, following the method suggested by Karadeniz et al. [35] with slight adjustments. Initially, a gram of apple samples was combined with a 10 ml methanol solution (70:30 v:v methanol:water) in a laboratory-type blender. The resulting mixture underwent thorough homogenization and subsequent centrifugation at 11,000 rpm for 1 min, a process repeated three times. The final centrifugation was carried out at 4000 rpm for 15 min. Post-centrifugation, the supernatant was meticulously separated and filtered to acquire the desired methanolic extract for subsequent TPC and AA analysis.

The assessment of TPC was executed using the methodology proposed by Karadeniz et al. [35] with a slight modification. 0.5 ml of methanolic solution of extracts, 7 ml of distilled water, and 0.5 ml of Folin Ciocalteu reagent (Merck) were mixed and kept in the dark for 3 min at room temperature. Following that, 2 ml of 20% sodium carbonate solution was added and mixed. The last mixture was kept in the dark for 2 h at room temperature. After 2 h, the absorbance of the samples was measured at 760 nm. To evaluate the antioxidant activity (AA) of the apple samples, the DPPH method by Thaipong et al. [36] was employed, with some minor modifications. A mixture was prepared by combining 150 µL of extracts with 2850 µL of DPPH methanolic solution, exhibiting an absorbance of 1.1 at 515 nm. This mixture was then incubated at room temperature in darkness for 60 min. Subsequently, the absorbance of the samples was measured at 515 nm after the 60-min incubation period. Both TPC and AA analyses were conducted in triplicate to ensure the precision and consistency of the results. TPC results were reported as gallic acid equivalent (mg GAE 100 g^{-1} d.m.), while AA results were expressed as mmol Trolox equivalent (mmol TE g^{-1} d.m.)

2.10 ATR-FTIR analysis

Fourier transform infrared spectroscopy (FTIR) was preferred to measure the spectra of apple samples. The absorption spectrum of the samples was obtained using FTIR spectroscopy (VERTEX 70 Series, Bruker, Germany) equipped with an ATR cell. For fresh apple samples, a 0.5-cm section was used, while for dried apple samples, ground apple samples were placed on a multiple-bounce ZnSe crystal, and scans were performed with a resolution of 2 cm^{-1} in the range of 400 to 4000 cm^{-1}. Spectra data obtained from the samples were baseline-corrected using "0" as reference and then normalized based on the highest absorbance value. The transformations were carried out using the "Speaktragryph© version 1.2.14" program [37].

2.11 Statistical analysis

The data were analyzed using one-way analysis of variance (ANOVA) with SPSS software (ver. 22 SPSS Inc., Chicago, IL, USA). Post hoc Tukey tests were conducted to compare means at a significance level of $p < 0.05$. Additionally, principal component analysis (PCA) was employed in Minitab® 18 (Minitab Inc., USA) to assess correlations among quality parameters.

3 Results and discussions

3.1 Drying of apple slices

Miraei Ashtiani et al. [38] stated that in convective drying, heat is transferred from the air to the product's surface through convection, creating a temperature gradient from the surface to the center. This gradient significantly hinders heat transfer and diminishes both mass migration and transfer within the product. This disadvantage can be minimized by pretreatment methods. Variation of MR (a) and DR (b) of apple slices are shown in Fig. 3. Besides, d_{eff} and drying times of the samples are given in Table 2. According to Fig. 1 and Table 2, pretreatments provided increment in the drying rate and decrement in drying time of the apple slices. The d_{eff} values of the samples varied between 1.46×10^{-10} and 3.74×10^{-10} m^2 s^{-1}. According to Zarein et al. [39],

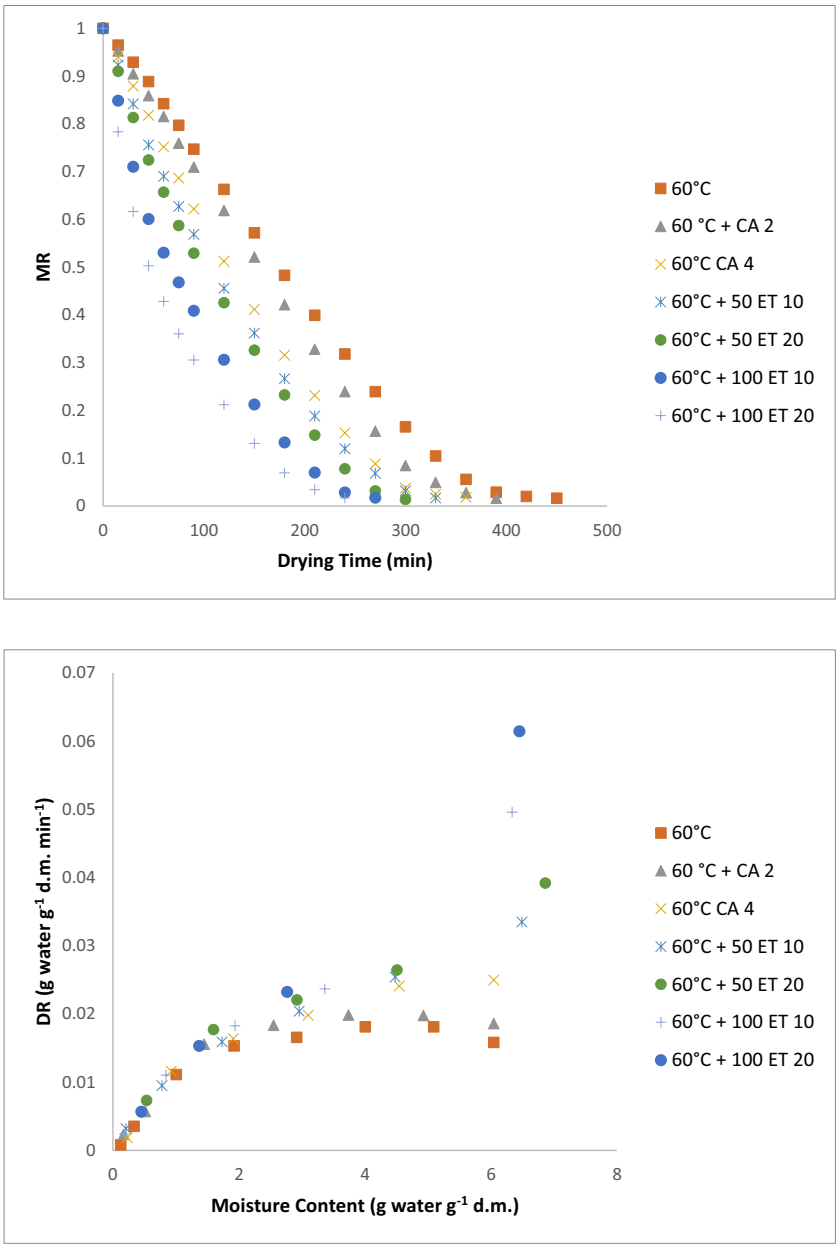

Fig. 3 Moisture ratio and drying rate of the apple slices

Table 2 The effective moisture diffusivity and drying time of the apple slices

Experiment	d_{eff} ($m^2\ s^{-1}$)	Drying time (min)
60 °C	1.46×10^{-10}	450
60 °C + CA 2	1.62×10^{-10}	390
60 °C + CA 4	1.92×10^{-10}	360
60 °C + 50 ET 10	2.09×10^{-10}	330
60 °C + 50 ET 20	2.26×10^{-10}	300
60 °C + 100 ET 10	2.91×10^{-10}	270
60 °C + 100 ET 20	3.74×10^{-10}	240

an increment in d_{eff} value indicates a decrement in drying time. In the current study, the highest d_{eff} value, the highest drying rate, and the lowest drying time were obtained from the apple slices immersed in ethanol solution for 20 min. Moreover, pretreatment time and ethanol concentration had a positive contribution to drying rate of the apple slices. The reduction of the drying time was found between 13.33 and 46.67%. As the pretreatment time and ethanol concentration increased, the drying rate and the d_{eff} value increased and the drying time decreased. The mechanism of the citric acid pretreatment is associated with the inactivating enzymes, textural modification, and pectin loosening [9, 21]. Especially, citric acid can change the texture properties by changing pectin gelation, hydrolysis, and depolymerization of pectin,

which increases the water removal rate and softens the material texture to reduce the hardness of dried products [22]. Thus, d_{eff} values of the samples pretreated with citric acid were found to be higher than control samples. Similarly, Tepe [9], Doymaz [31], and Doymaz [40] noted accelerating in drying rate of the citric acid–pretreated apple slices. This behavior was reported in different fruits and vegetables such as kiwifruits by Doymaz [41], quince slices by Doymaz et al. [42], pear slices by Öztekin and Sacılık [43], and peach slices by Doymaz and Bilici [44]. Moreover, ethanol pretreatment serves to eliminate intracellular air, disrupt, or thin cell walls, and extract specific compounds by permeating the sample. The higher evaporation rate of ethanol compared to water induces the Marangoni effect, causing a concentration disparity with a more ethanol-rich mixture on the surface than in the product's inner layers. This triggers swift ethanol evaporation, establishing a surface tension gradient that facilitates the transportation of water from the internal layers of the food during the drying process [9, 18, 45, 46] Likewise, Tepe [9], Rojas et al. [18], and Lin et al. [47] reported that ethanol pretreatment enhanced the drying rate of the apple slices. Tepe [9] and Lin et al. [47] noted an increment in concentration of ethanol solution for pretreatment provided upward trend in drying rates of the apple slices. In addition to this, Rojas et al. [18] and Tepe [9] found that a longer ethanol pretreatment time caused greater drying rate and shorter drying time in apple slices. The extended time of ethanol pretreatment is hypothesized to induce multiple effects on the food sample, potentially leading to heightened removal of air, increased thinning of cell walls, and enhanced permeability. Furthermore, the intensified Marangoni effect is assumed to be a consequence of the elevated ethanol concentration on the surface, a condition influenced by both a more rapid rate of immersion and the extended time of pretreatment. Positive effect of ethanol pretreatment to drying rate was also reported in other fruits and vegetables such as melon slices by da Cunha et al. [46], scallion by Wang et al. [48], eggplant slices by Zhao et al. [49], and pineapple slices by de Freitas et al. [50]. The results of the study showed a good agreement with these reports.

3.2 Modeling of drying curves of apple slices

In the current study, different modeling approaches were used: thin-layer and ANN modeling. Table 3 shows the statistical parameters of the modeling types. Among the thin-layer models, Midilli and Kucuk model provided the best prediction for experimental MR of the apple slices when evaluating the statistical parameters. χ^2, RMSE, and R^2 values of Midilli and Kucuk model were found to be in the range of 0.000142709–0.000397739, 0.01054–0.01744, and 0.9980–0.9993, respectively. Doymaz [40] noted that Verma et al. model gave the best prediction result for citric acid–pretreated apple slices, whereas untreated samples were described by Wang and Singh model. Parabolic model was reported the most appropriate model for untreated, citric acid–, and ethanol-pretreated apple slices by Tepe [9]. Researchers in the literature have presented diverse models, potentially influenced by factors like varying drying conditions, sample types, diameters, thicknesses, and the fitted models chosen in their respective studies. On the other hand, ANN modeling had greater performance than thin-layer models according to RMSE (0.0020379–0.0052434) and R^2 (0.9999 for all samples) values. Figures 4 and 5 present the best validation performance and regressions of the ANN modeling. During the training of ANN, it is essential to monitor for over-fitting. Undesirably, if the error curves for validation and test datasets exhibit opposing trends throughout training iterations, it suggests that the desired level of success in training the ANN has not been attained, as reported by Kurtulmuş et al. [51]. Conversely, a consistent trajectory in the validation and test error vectors, as depicted in Fig. 4, indicates an absence of over-fitting in the current study, according to Kurtulmuş et al. [51]. In the literature, there are studies that reported better ANN performance than thin-layer modeling for the MR prediction. Ghasemkani et al. [23] noted that the most suitable modeling of apple slices was ANN modeling according to the statistical parameters. Similar results were noted in onion by Jafari et al. [52] spearmint by Karakaplan et al. [53] and potato slices by Tepe [54].

3.3 Diameter and thickness shrinkage of apple slices

The high shrinkability of fruits and vegetables during drying arises from their porous and hygroscopic characteristics. As water is transported from cellular sites to the surrounding environment, it induces irregular volume changes in high-moisture foods. This reduction in volume is commonly referred to as material shrinkage in the context of drying [55]. The shrinkage of fruits and vegetables, often described as the deformation of material, represents a conspicuous physical phenomenon frequently during drying. The shrinkage observed in dried products has a multitude of unfavorable outcomes, impacting aspects from product quality to consumer satisfaction. Additionally, it gives rise to other adverse effects, such as surface cracking and a diminished capacity for rehydration [56].

Table 4 presents the diameter and thickness shrinkage ratio of untreated and pretreated apple slices after drying process. Dried apple slices are illustrated in Fig. 6. The shrinkage phenomenon was observed in the diameter and thickness of apple slices. As seen from Table 4, no mean statistical differences were found between the sample groups ($p > 0.05$). Several factors influence shrinkage,

Table 3 Statistical parameters and model constants of thin layer models and ANN

Model	Experiment	Model Constants				χ^2	RMSE	R^2
Page	60 °C	$k=0.0002127$	$n=1.583$			0.000775419	0.02634	0.9950
	60 °C + CA 2	$k=0.0003116$	$n=1.546$			0.00100577	0.02979	0.9934
	60 °C + CA 4	$k=0.0008677$	$n=1.400$			0.000613542	0.02317	0.9960
	60 °C + 50 ET 10	$k=0.002023$	$n=1.260$			0.000666278	0.02403	0.9953
	60 °C + 50 ET 20	$k=0.002719$	$n=1.222$			0.00101254	0.02946	0.9928
	60 °C + 100 ET 10	$k=0.009065$	$n=1.037$			0.000792163	0.02589	0.9940
	60 °C + 100 ET 20	$k=0.01898$	$n=0.933$			0.000438689	0.01912	0.9966
Henderson and Pabis	60 °C	$k=0.005399$	$a=1.108$			0.00601319	0.07335	0.9612
	60 °C + CA 2	$k=0.005984$	$a=1.097$			0.006095923	0.07334	0.9602
	60 °C + CA 4	$k=0.007115$	$a=1.082$			0.003789093	0.05758	0.9751
	60 °C + 50 ET 10	$k=0.007738$	$a=1.054$			0.002269526	0.04435	0.9840
	60 °C + 50 ET 20	$k=0.008371$	$a=1.044$			0.002315486	0.04455	0.9835
	60 °C + 100 ET 10	$k=0.01075$	$a=1.001$			0.000847563	0.02678	0.9936
	60 °C + 100 ET 20	$k=0.01381$	$a=0.9768$			0.00051817	0.02078	0.9959
Parabolic	60 °C	$a=1.041$	$b=-0.003725$		$c=0.000002996$	0.000750523	0.02514	0.9957
	60 °C + CA 2	$a=1.030$	$b=-0.004054$		$c=0.000003459$	0.000713487	0.02424	0.9959
	60 °C + CA 4	$a=1.021$	$b=-0.005022$		$c=0.000006036$	0.000217711	0.01330	0.9988
	60 °C + 50 ET 10	$a=0.9953$	$b=-0.005390$		$c=0.000007309$	5.36773E-05	0.00655	0.9997
	60 °C + 50 ET 20	$a=0.9835$	$b=-0.005731$		$c=0.000008287$	0.000204955	0.01269	0.9988
	60 °C + 100 ET 10	$a=0.9408$	$b=-0.007148$		$c=0.00001406$	0.001324552	0.03192	0.9917
	60 °C + 100 ET 20	$a=0.909$	$b=-0.008659$		$c=0.00002127$	0.00322752	0.04920	0.9795
Lewis	60 °C	$k=0.004807$				0.007739858	0.08563	0.9441
	60 °C + CA 2	$k=0.005372$				0.007353112	0.08319	0.9454
	60 °C + CA 4	$k=0.006494$				0.004636549	0.06593	0.9650
	60 °C + 50 ET 10	$k=0.007266$				0.002536924	0.04866	0.9793
	60 °C + 50 ET 20	$k=0.007952$				0.002352676	0.04674	0.9804
	60 °C + 100 ET 10	$k=0.01075$				0.000712194	0.02564	0.9936
	60 °C + 100 ET 20	$k=0.01420$				0.000516068	0.02175	0.9951
Midilli and Kucuk	60 °C	$k=0.0002617$	$a=0.9812$	$n=1.511$	$b=-0.0001792$	0.000314612	0.01576	0.9984
	60 °C + CA 2	$k=0.0003762$	$a=0.9768$	$n=1.468$	$b=-0.0002551$	0.000397739	0.01744	0.9980
	60 °C + CA 4	$k=0.001208$	$a=0.9881$	$n=1.303$	$b=-0.0002227$	0.000241562	0.01346	0.9988
	60 °C + 50 ET 10	$k=0.0036$	$a=0.9960$	$n=1.104$	$b=-0.0003496$	0.000142709	0.01023	0.9993
	60 °C + 50 ET 20	$k=0.005928$	$a=0.9993$	$n=1.008$	$b=-0.000542$	0.000203284	0.01205	0.9990
	60 °C + 100 ET 10	$k=0.01611$	$a=1.002$	$n=0.8761$	$b=-0.0004256$	0.000253973	0.01326	0.9987
	60 °C + 100 ET 20	$k=0.02736$	$a=1.002$	$n=0.8274$	$b=-0.0002955$	0.000166637	0.01054	0.9992
ANN	60 °C						**0.0030594**	**0.9999**
	60 °C + CA 2						**0.0039773**	**0.9999**
	60 °C + CA 4						**0.0052218**	**0.9999**
	60 °C + 50 ET 10						**0.0049404**	**0.9999**
	60 °C + 50 ET 20						**0.0052434**	**0.9999**
	60 °C + 100 ET 10						**0.0027774**	**0.9999**
	60 °C + 100 ET 20						**0.0020379**	**0.9999**

Bold indicates the most appropriate model for prediction of drying curves of the apple slices

encompassing material microstructure and properties, mechanical characteristics, and the specifics of the processing methods employed [34]. Likewise, Granella et al. [34] reported no statistically significant shrinkage in diameter and thickness of banana slices pretreated with ethanol in comparison to untreated samples. On the contrary, significant diameter shrinkage in potato slices pretreated with ethanol was noted by Tepe [54]. Additionally, no statistical difference in shrinkage ratio of citric acid–pretreated carrot slices after drying process when

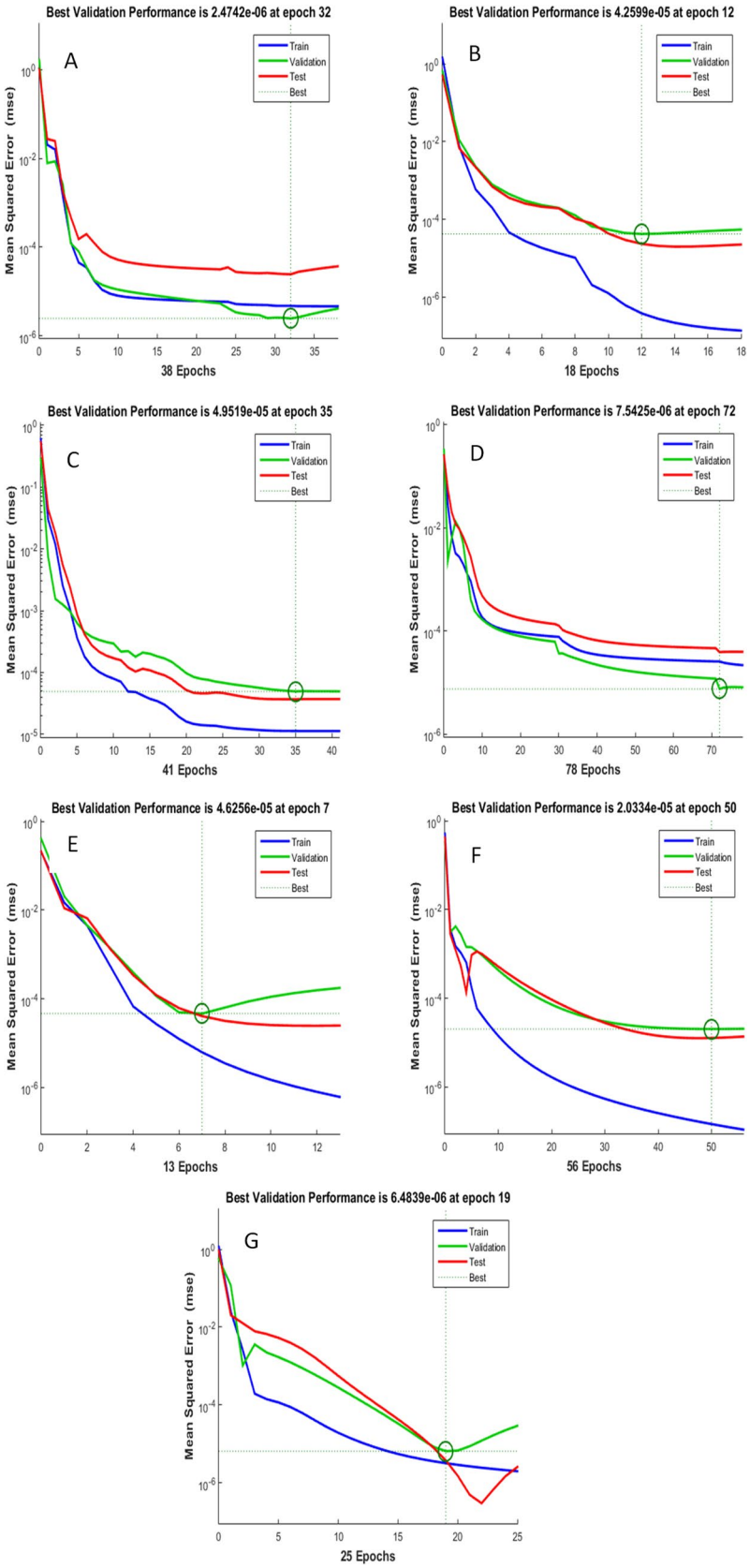

Fig. 4 Best validation performance of ANN modeling of the apple slices (**A** 60 °C; **B** 60 °C + CA 2, **C** 60 °C + CA 4, **D** 60 °C + 50 ET 10; **E** 60 °C + 50 ET 20; **F** 60 °C + 100 ET 10; **G** 60 °C + 100 ET 20)

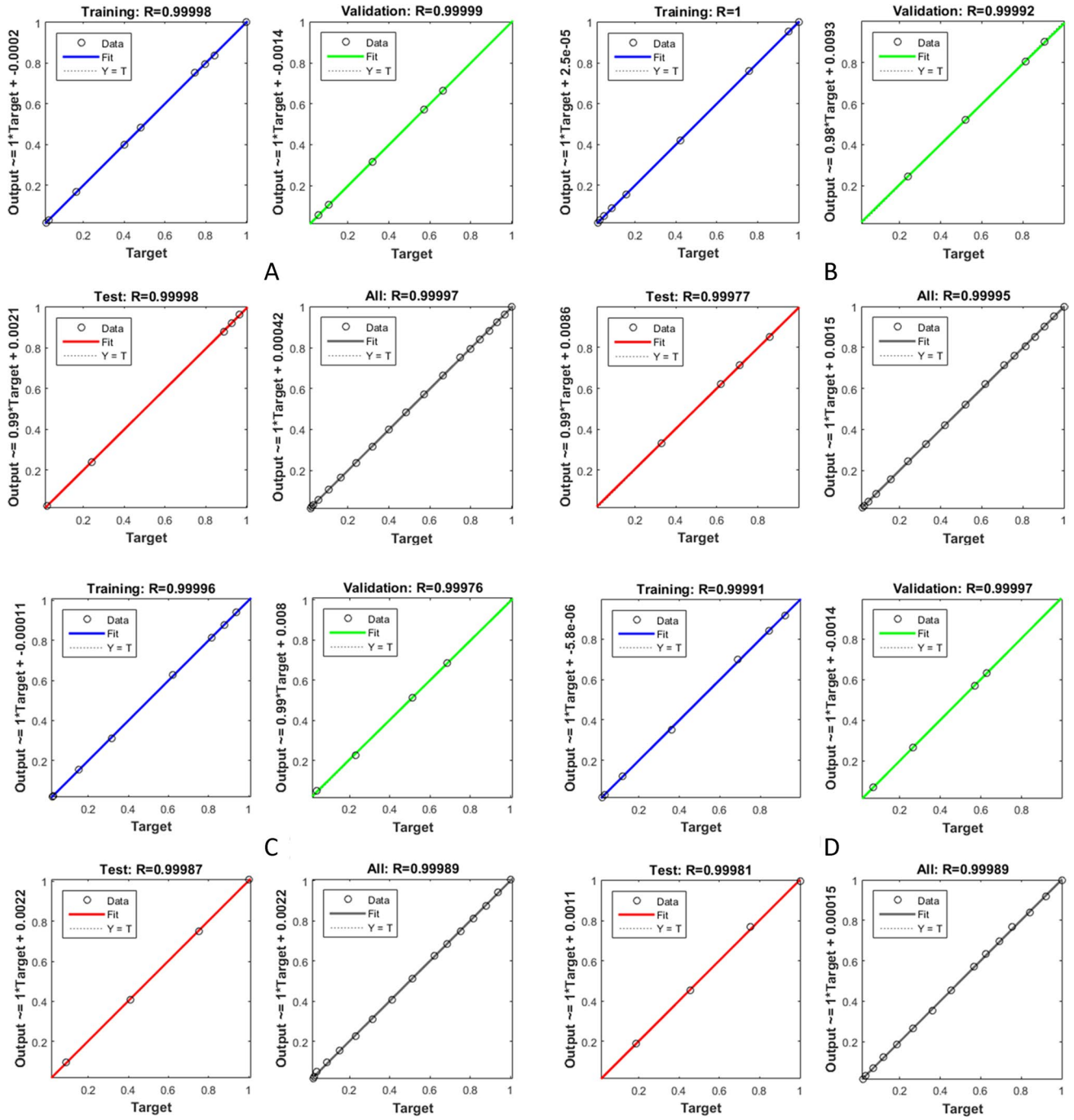

Fig. 5 Regressions of ANN modeling of the apple slices (**A** 60 °C; **B** 60 °C + CA 2, **C** 60 °C + CA 4, **D** 60 °C + 50 ET 10; **E** 60 °C + 50 ET 20; **F** 60 °C + 100 ET 10; **G** 60 °C + 100 ET 20)

compared to untreated samples was reported by Hiranvarachat et al. [57] The different results in the literature can be related several factors affecting shrinkage as mentioned above.

3.4 Color properties of apple slices

The sensory characteristics of food, particularly its visual presentation during the point of purchase, enable quick

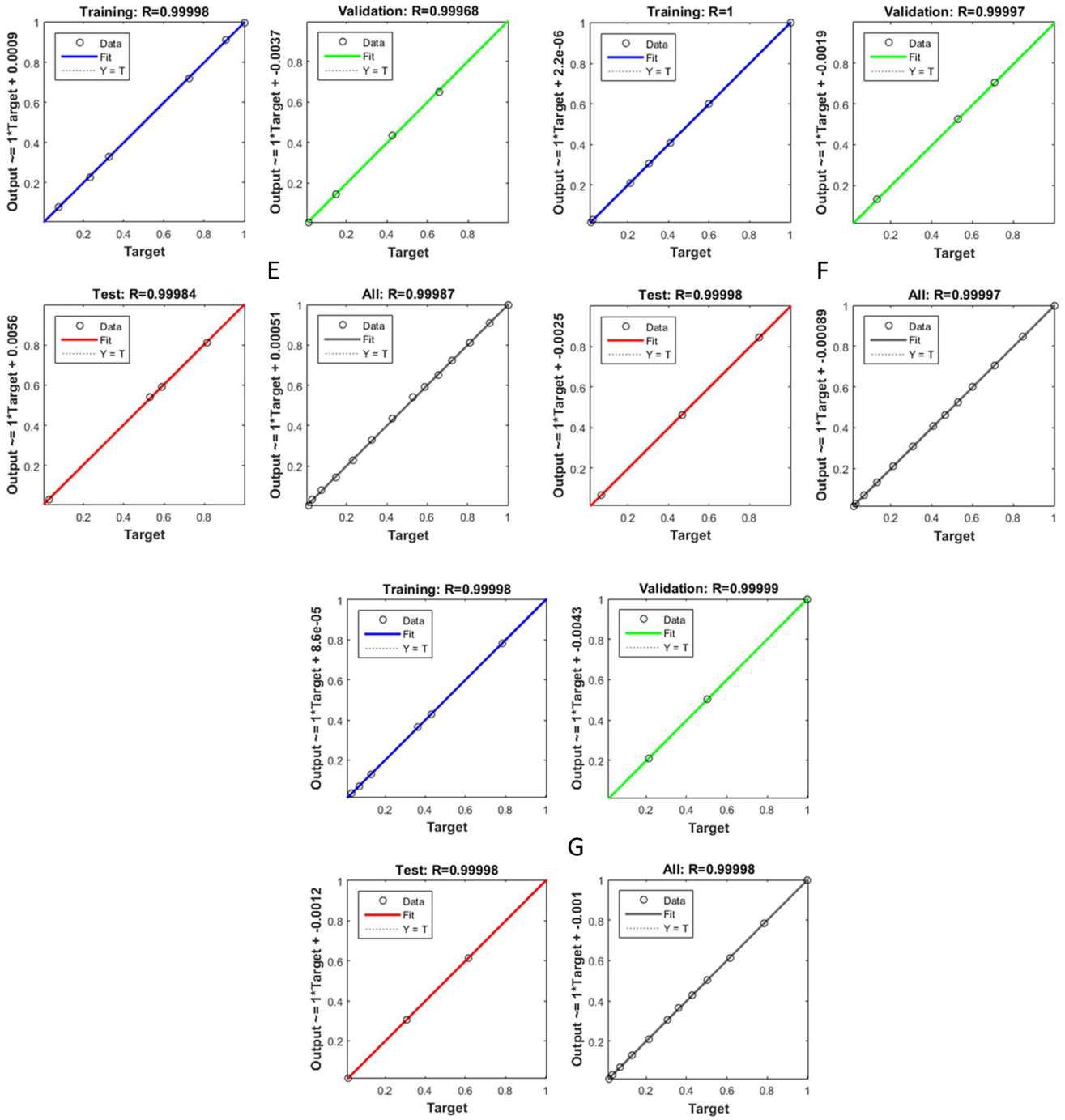

Fig. 5 (continued)

identification and significantly shape consumer acceptance. The acceptability of food is directly impacted by its color; therefore, it is essential to exercise caution in order to minimize pigment damage and prevent darkening reactions throughout the processing stages [46, 58, 59]. Table 5 presents the color results of the fresh and dried apple slices. Drying process caused the variation in the color values of the apple slices. No significant change in L^* values was observed in the untreated and citric acid–pretreated apple slices ($p > 0.05$), whereas L^* values showed downward trend in ethanol-pretreated apple slices ($p < 0.05$). The color properties of dried products are typically influenced by enzymatic browning, which arises from high levels of polyphenols, polyphenol oxidase, and peroxidase. Additionally, non-enzymatic browning processes, such as the Maillard reaction, caramelization, and chemical oxidation

Table 4 Diameter and thickness shrinkage ratio of the apple slices

Experiment	DS (%)	SD (±)	TS (%)	SD (±)
60 °C	16.88[a]	2.67	55.90[a]	4.12
60 °C + CA 2	21.13[a]	4.08	52.44[a]	3.69
60 °C + CA 4	22.06[a]	2.59	50.85[a]	5.88
60 °C + 50 ET 10	20.35[a]	4.41	54.94[a]	3.63
60 °C + 50 ET 20	21.03[a]	3.90	52.50[a]	2.16
60 °C + 100 ET 10	19.39[a]	2.67	49.92[a]	8.09
60 °C + 100 ET 20	19.30[a]	1.37	55.75[a]	4.89

Different letters in the same column indicate significant differences with a confidence of 95%

of polyphenols, as well as maderisation, further contribute to the alteration of the product's color [21]. In addition, chlorophyll degradation products such as pheophytin and pheophorbide may affect the browning of the dried product [60]. When cell wall and membrane disruption is considered in ethanol-pretreated samples, more phenolic content is released from the tissue. It means higher oxidable phenolic content. The decrement in L^* value can be related to these phenomena. Moreover, close value of L^* in the citric acid–pretreated samples to fresh samples can be clearly associated with preventing enzymatic browning reaction by copper chelating ability that enables to deactivating of the polyphenol oxidase [61]. Furthermore, it is important to note that the Maillard reaction, a significant member of non-enzymatic browning, is facilitated by higher pH values, as stated by Zhang et al. [62]. The addition of citric acid can mitigate non-enzymatic browning by lowering the pH value of apple slices. Also, higher L^* values of the untreated samples in comparison to ethanol-pretreated samples can be linked to lower cell damage which can cause lower phenolic content release from the tissue than ethanol-pretreated samples. On the other hand, a^* and b^* values of the apple slices increased after the drying process ($p < 0.05$). Chlorophylls, carotenoids, and anthocyanins play a crucial role in determining the color characteristics of fruits and vegetables, as noted by Cömert et al. [63]. McGhie and Ainge [64] and Nowacka et al. [65] reported the ability of chlorophylls to mask the colorization of carotenoids, primarily responsible for the yellow hue in foods. Zhu et al. [66] noted that the increase in yellowness of dried products correlates with the high concentration of yellowish phytochemicals post-water removal. This rise in the b^* value may signify an increase in yellowish phytochemical concentration and potential chlorophyll degradation, thus removing the masking effect on carotenoids responsible for the yellow color. Fijalkowska et al. [67] noted that the a^* value, indicating red color, is associated with enzymatic browning, explaining the potential rise in a^* value due to enzymatic browning. Furthermore, the minimal increase in a^* value in citric acid–pretreated samples supports this argument. Together, these statements explain the higher a^* and b^* values observed in apple slices at the conclusion of the drying process. Likewise, Tepe [9] noted a decrement in L^* values and an increment in a^* and b^* values of the ethanol-pretreated apple slices. Downward trend in L^* values and upward trend in a^* and b^* values of the ethanol-pretreated pineapple slices were reported by de Freitas et al. [50]. Moreover, the total color difference (ΔE) in apple slices reflects variations in color compared to the fresh samples. According to Abbaspour-Gilandeh et al. [68], a ΔE value exceeding 5 signifies a significant difference noticeable to non-trained observers, implying that a lower ΔE suggests improved color properties. In the current study, all dried samples exhibited ΔE values surpassing 5.

3.5 Total phenolic content and antioxidant activity of apple slices

TPC and antioxidant activity (AA) of the fresh and pretreated apple slices are presented in Table 6. TPC and AA of the fresh apple samples were measured as 1070.93 ± 18.46 mg GAE 100 g^{-1} d.m. and 0.52 ± 0.02 mmol TE g^{-1} d.m. As seen from Table 6, TPC and AA of the apple slices were negatively affected by drying process and pretreatments ($p < 0.05$) in comparison to fresh samples. Because of their unsaturated bonds, phenolics are known to have strong antioxidant qualities. Esparza et al. [69] stated that phenolics are very susceptible to heat, pH, light, enzyme activity, metal ions, and oxygen. Méndez-Lagunas et al. [70] notified that the drying process usually results in the expected loss of antioxidative compounds, including phenolics. Antioxidant activity and phenolics have a typically significant and favorable association, according to İzli et al. [71]. Wojdylo et al. [72] explained the degradation of TPC with irreversible oxidation and thermal degradation during drying process. The highest losses of TPC and AA were observed at the ethanol-pretreated samples, whereas the highest prevention in TPC was observed at citric acid–pretreated apple slices for 4 min. Additionally, increment in ethanol concentration and pretreatment time had a negative effect on the TPC and AA ($p < 0.05$). Likewise, loss of TPC and AA were reported in untreated, citric acid–, and ethanol-pretreated apple slices after drying by Tepe [9]. Besides, Tepe [9] emphasized that ethanol pretreatment caused a higher loss in TPC and AA of the apple slices and citric acid–pretreated apple samples had the higher TPC and AA. Similarly, Rojas et al. [18] observed greater losses in TPC and AA in apple samples that immersed in ethanol solution, in comparison to untreated samples. Similar findings were reported in other fruits and vegetables such as melon by da Cunha et al. [46], and uvaia by Gomes et al. [73]. Higher loss in TPC and AA can be attributed to some phenomena. Firstly, the pretreatment has the potential to increase the exposure of compounds to the

Fig. 6 The apple slices after drying process (**A** 60 °C; **B** 60 °C + CA 2, **C** 60 °C + CA 4, **D** 60 °C + 50 ET 10; **E** 60 °C + 50 ET 20; **F** 60 °C + 100 ET 10; **G** 60 °C + 100 ET 20)

oxidative effects of drying air. Specifically, the immersion in ethanol may eliminate air within the tissues and modify the cell walls of the porous structure, characterized by the high surface area and thin geometry of apple samples, thereby further exposing internal constituents [19]. Secondly, extraction of the phenolics and following that, migration to the ethanol solution during pretreatment can be responsible for the loss in TPC and AA [46, 73]. It can be proved with the using of ethanol and water for the phenol extraction [74]. Finally, considering the durability of free phenolics, Vuolo et al. [75] noted that bonded phenolics exhibit higher stability compared to their free counterparts. The convective drying process following ethanol pretreatment is anticipated to induce increased oxidation and thermal degradation, particularly in phenolics that transition from a bonded to a free state. On the other hand, citric acid prevention effect can be

Table 5 Color properties of the apple slices

Experiment	L^*	SD (±)	a^*	SD (±)	b^*	SD (±)	ΔE	SD (±)
Fresh	72.55^{a}	0.32	7.43^{d}	1.09	21.85^{d}	1.36	0^{d}	0
60 °C	72.86^{a}	2.11	11.66^{c}	1.39	35.47^{a}	2.70	14.36^{bc}	4.44
60 °C + CA 2	72.83^{a}	1.66	12.71^{c}	0.34	33.64^{ab}	1.23	13.00^{c}	1.78
60 °C + CA 4	73.31^{a}	2.29	12.78^{c}	1.56	36.51^{a}	2.98	15.81^{bc}	1.77
60 °C + 50 ET 10	58.77^{c}	2.95	17.05^{ab}	1.30	32.82^{abc}	1.73	20.33^{ab}	1.62
60 °C + 50 ET 20	50.40^{d}	1.59	17.87^{a}	0.29	29.82^{bc}	2.63	25.90^{a}	1.89
60 °C + 100 ET 10	61.67^{bc}	1.75	19.25^{a}	1.43	34.17^{ab}	1.32	20.45^{ab}	2.08
60 °C + 100 ET 20	66.09^{b}	1.31	14.04^{bc}	0.47	27.81^{c}	0.67	11.86^{c}	1.05

Different letters in the same column indicate significant differences with a confidence of 95%

Table 6 Total phenolic content and antioxidant activity of the apple slices

Experiment	TPC (mg GAE 100 g^{-1} d.m.)	SD (±)	AA (mmol TE g^{-1} d.m.)	SD (±)
Fresh	1070.93^{a}	18.48	0.52^{a}	0.023
60 °C	463.00^{c}	3.61	0.12^{b}	0.001
60 °C + CA 2	477.33^{c}	2.89	0.12^{b}	0.004
60 °C + CA 4	597.33^{b}	2.90	0.10^{bc}	0.003
60 °C + 50 ET 10	282.33^{d}	2.88	0.07^{d}	0.008
60 °C + 50 ET 20	269.00^{de}	5.00	0.08^{cd}	0.004
60 °C + 100 ET 10	257.33^{e}	2.95	0.09^{cd}	0.004
60 °C + 100 ET 20	255.67^{e}	2.84	0.10^{bc}	0.003

Different letters in the same column indicate significant differences with a confidence of 95%

easily explained by deactivating of the polyphenol oxidase that provides lower enzymatic oxidation than the other sample groups [61].

3.6 ATR-FTIR analysis of apple slices

ATR-FTIR spectra ranging from 4000 to 400 cm^{-1} obtained from fresh apple and apple samples dried with seven different treatments are illustrated in Fig. 7. The spectra depicted in the figure reflect the components of the apple, namely water and carbohydrates, followed by proteins, fibers, and organic acids. The broad absorption band in the range of 3700–2800 cm^{-1} is associated with the O–H stretching vibrations of carbohydrates and water. This band strongly overlaps with signals attributed to the symmetric and asymmetric stretching modes of C–H bonds in visible peaks at 2900 cm^{-1} and 2950 cm^{-1}. While fresh apple samples exhibit a very low peak in this band, all dried apple samples, except for those dried at 60 °C, show similar peaks.

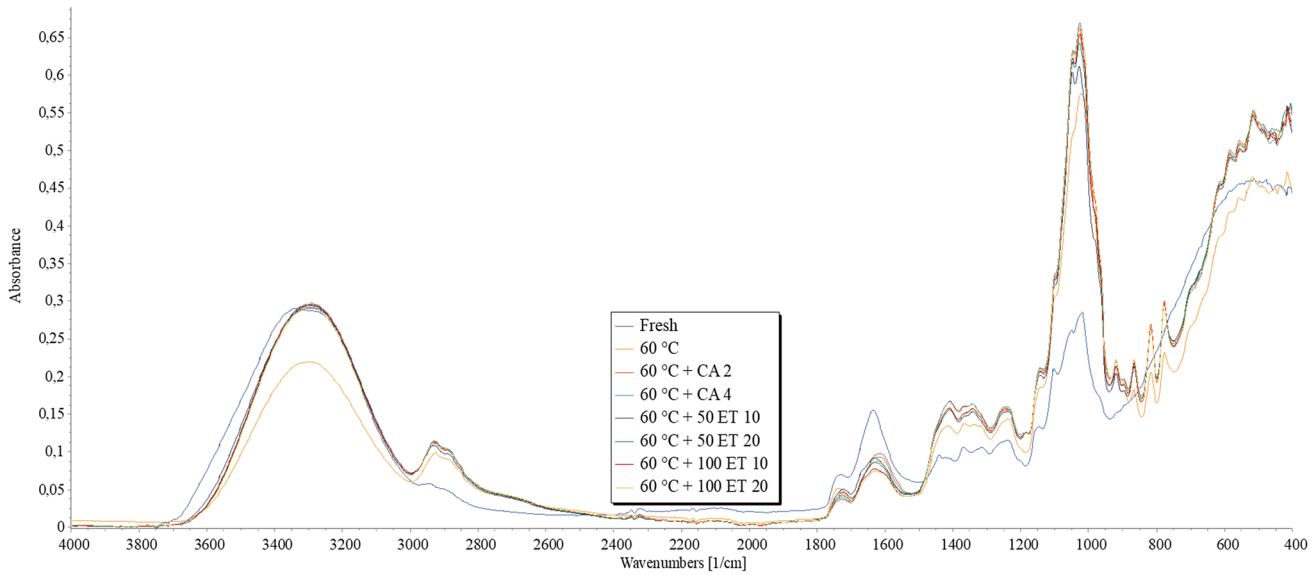

Fig. 7 ATR-FTIR spectra of the apple samples

The peaks in the range of 1800–1500 cm^{-1} are associated with the vibrations of absorbed water molecules of sugars present in the apple. The peak at approximately 1650 cm^{-1} is linked to the C = O stretching of lipids and organic acids. In this range, a noticeable decrease in peaks is observed in all dried samples compared to the fresh sample. Additionally, the amid I and amid II peaks of proteins fall within this spectral range. Peaks in the spectral range between 1500 and 1200 cm^{-1} mainly originate from the deformational modes of CH/CH_2 groups. Clear peaks emerge in all dried samples in this range, compared to the fresh sample, potentially attributed to Maillard reactions and the removal of water due to drying [76]. Previous studies have demonstrated a direct correlation between the intensity of absorption bands at 3750–2800 cm^{-1} and 1800–1500 cm^{-1} and the hydration degree of carbohydrates [76]. Carbohydrate absorption bands between 1200 and 800 cm^{-1} arise from the combination of C–O, C–C, and C–O–H stretching and starch C–O–H bending. The significantly lower peaks at 1500–1500 cm^{-1} in fresh apple samples compared to dried apple samples are believed to result from the applied drying processes. Except for the apple sample dried at 60 °C, all other dried apple samples show similar peaks. In Fig. 8 below, second derivatives of ATR-FTIR spectra in the ranges of 3200–2800 cm^{-1} and 1750–1200 cm^{-1} are presented. As observed in the second derivatives, it is more evident that, except for fresh apple

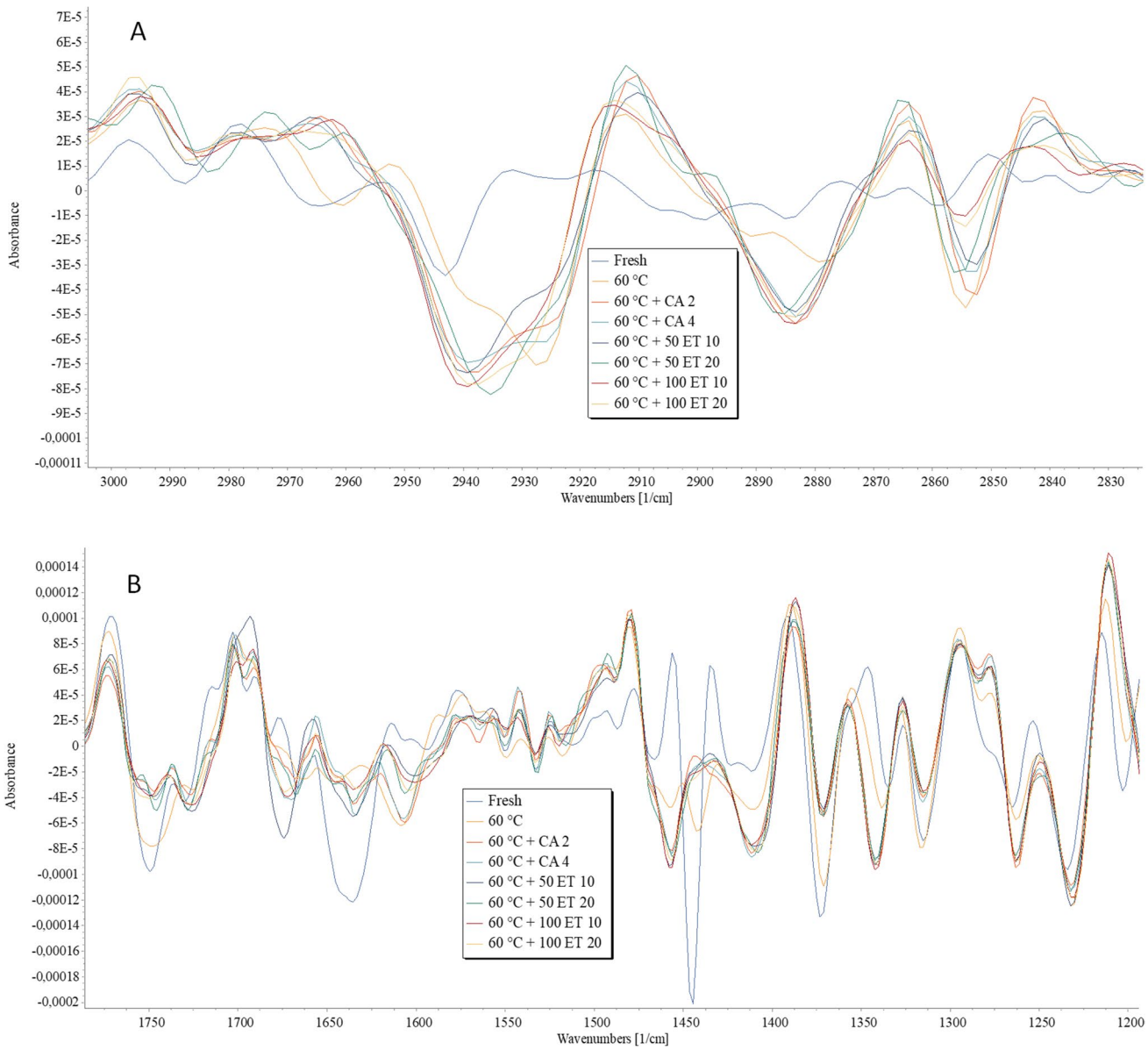

Fig. 8 The second derivatives of ATR-FTIR spectra of the apple samples in the range of 3200–2800 cm^{-1} (**A**) and 1800–1200 cm^{-1}.[1] (**B**)

samples and the apple sample dried at 60 °C, processed and dried apple samples exhibit distinct spectra. Ethanol and citric acid applications yield similar peaks in the dried apple samples, indicating that these pretreatments significantly affect the drying process.

3.7 Principal component analysis of apple slices

Figire 9A illustrates the TBA chart of apple samples, including TPC, AA, color parameters (L^*, a^*, and b^*), ΔE, diameter shrinkage, and thickness shrinkage values. On the other hand, Fig. 9B shows the PCA chart of total phenol and total antioxidant activity values. When evaluating Fig. 9A encompassing all results, it is observed that fresh samples occupy a separate plane from all other dried samples. Specifically, apple samples dried at 60 °C, 60 °C + CA 2, and 60 °C + CA 4 conditions exhibit a different arrangement compared to those dried with ethanol. Different ethanol-treated dried apple samples are positioned in the same plane. Examining PCA results in Fig. 9B, which only depict total phenol and total antioxidant activity, similar outcomes are observed. However, apple samples dried under 60 °C + CA 4 conditions show a different arrangement

According to PCA results in Fig. 9C–D, both wavelength ranges indicate that fresh apple samples and those dried at 60 °C are positioned significantly away from apple samples dried with ethanol and citric acid. As seen from Fig. 9C, the PCA results in the 3500–2800 cm^{-1} range indicate that all apple samples dried with ethanol and citric acid are aligned in the same plane. However, according to Fig. 9D, the PCA results in the 1800–1200 cm^{-1} range reveal that apple samples dried under 60 °C + 50 ET (10

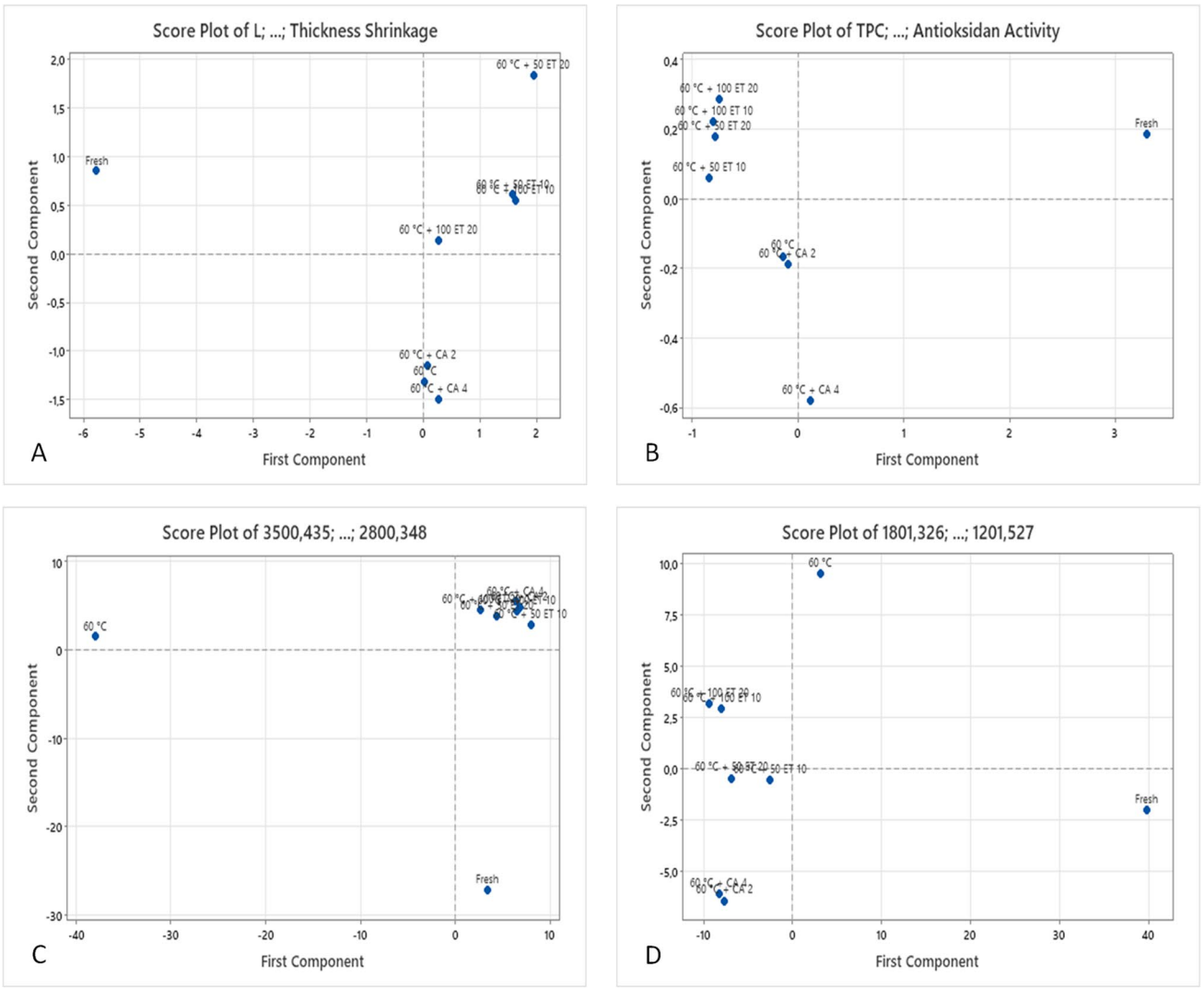

Fig. 9 PCA analysis of the quality parameters and ATR-FTIR spectra of the apple samples (**A**: TPC, AA, L*, a*, b*, ΔE, DS and TS, **B**: TPC and AA; **C**: ATR-FTIR spectra in the range of 3500 and 2800 cm^{-1}; **D**: ATR-FTIR spectra in the range of 1800 and 1200 cm^{-1})

and 20) and 60 °C + 100 ET (10 and 20) conditions are situated in a different plane. Apple samples dried under 60 °C + CA 2 and 60 °C + CA 4 conditions exhibit a similar arrangement.

4 Conclusion

The study investigated the drying process of apple slices with a focus on pretreatment methods (ethanol and, citric acid), modeling approaches (thin-layer and ANN), physical changes (diameter and thickness shrinkage), color properties (L^*, a^*, b^*, and ΔE), TPC, AA, spectral analysis using ATR-FTIR, and PCA. The key findings and conclusions are summarized as follows:

a. Ethanol and citric acid pretreatments significantly influenced the drying rate of apple slices. Drying rate showed upward trend as the ethanol or citric acid concentration and pretreatment time increased.
b. Thin-layer models, such as the Midilli and Kucuk model, were used to predict moisture ratio (MR) during drying. However, ANN modeling outperformed thin-layer models, providing better predictions based on lower RMSE and higher R^2 values.
c. Shrinkage in diameter and thickness of apple slices occurred during drying, a common phenomenon attributed to the porous and hygroscopic nature of fruits and vegetables.
d. Drying caused variations in color values, with ethanol-pretreated slices exhibiting a decrease in L^* values. Citric acid pretreatment preserved color properties. a^* and b^* values increased in all dried samples.
e. TPC and AA decreased after the drying process and pretreatments, with ethanol pretreatment causing the highest losses.
f. ATR-FTIR spectra revealed changes in water and carbohydrate content, proteins, fibers, organic acids, and the occurrence of Maillard reactions during drying. Ethanol and citric acid pretreatments showed distinctive spectra, indicating their significant impact on the drying process.
g. PCA results demonstrated clear distinctions between fresh and dried samples, with different drying conditions clustering together. Ethanol and citric acid pretreatments exhibited similar effects, influencing TPC, AA, color parameters, and physical changes in a comparable manner.

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Author contribution Tolga Kağan TEPE: conceptualization; investigation; data analysis and curation; writing—original draft; methodology; visualization; writing—review and editing.

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Data availability No availability of data.

Declarations

Ethical approval Not applicable.

Conflict of interest The author declares no competing interests.

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