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Effects of Hybrid Drying on Kinetics, Energy Analysis and Bioactive Properties of Sour Black Mulberry (*Morus nigra* L.)

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HIGHLIGHTS

- Sour black mulberries were evaluated in terms of drying method.
- Logistic model showed the best fitting performance.
- Specific energy consumption increased with increase in microwave power.
- Increase in the temperature caused a significant decrease in anthocyanin content.

Abstract: Due to the short harvest season and their sensitivity to storage, the preservation of fresh mulberry fruits is a very important process. Drying is a method used to preserve mulberry fruits in the long term. In this study, response surface methodology (RSM) was applied for the optimization of hybrid drying conditions of two different sour black mulberries. The linear and interaction effects of independent parameters such as temperature (50, 60 and 70°C) and microwave power (100, 200 and 300 W) variables were determined on mulberries. Bioactive properties and energy aspects were monitored as influenced by drying conditions. According to the results increase in microwave power provided a significant decrement in the specific energy consumption (SEC) and the total anthocyanin content (TAC), while increase in the energy efficiency (η_{en}) and total phenolic content (TPC) for both genotypes. In all cases, statistical values showed that all drying curves of black mulberry were best described by the Logistic model. Multiple response optimization was carried out for studied parameters and it was concluded that maximum antiradical activity (ARA), TPC, TAC, η_{en} and minimum drying time (DT) and SEC values would be at 300 W-50 ºC (desirability=0.842) and 300 W-66.5 ºC (desirability=0.744), for *Morus nigra* 1 (MN1) and *Morus nigra* 2 (MN2), respectively. According to the finding, the greatest TPC, ARA, TAC, DT, SEC and η_{en} were determined as 20.10 mg GAE/g, 86.00%, 456 mg/kg, 330 min, 18.59 kWh/kg and 9.04% for MN1, and 18.08 mg GAE/g, 83.92%, 835.81 mg/kg, 330 min, 16.16 kWh/kg and 10.40 % for MN2, respectively.

Keywords: *Morus nigra* L.; energy; modeling; phenolics; anthocyanin.

INTRODUCTION

Morus nigra L. often called black mulberry in the literature is known as sour black mulberry in Turkey and is an important fruit belonging to the Morus genus in the Moraceae family [1]. This species occupies a unique position, exhibiting decosaploidy (22x) with chromosome 2n = 308, the highest number recorded for any known species [2]. Sour black mulberry trees can live for many years and also, they are durable [3]. In the 1930s, it was determined that the oldest tree grown in England was planted in 1548. Mulberry trees are widely distributed in India, China, Japan, North Africa, Arabia, South Europe etc. *Morus nigra* is considered native to the Near East, particularly Iran and Turkey [4, 5].

Most mulberry species predominantly possess either white or black fruits, while *Morus alba* fruits have colors ranging from black to white. Studies on black mulberry are not highly reliable since the other mulberry species with black fruits *(Morus alba, Morus rubra, Morus macruara, Morus pendula* etc.) caused other species to be confused with black mulberry (*Morus nigra*) too much [6]. Additionally, the blackberry is called as *Morus nigra* in Brazil increasing information pollution on this species. Because of this confusion, papers coming from Brazil identified black mulberry as invasive species [5]. The dried fruits of *Morus nigra* have a sour taste and added to dishes and salads to give a sour taste like lemon and sumac in Turkey. For this reason, it is called mainly sour black mulberry in Turkey since old times.

Sour black mulberry is highly appreciated and considered food for dukes and sultans in West and East, respectively and its fruits are regarded as the healthiest in mulberries [7]. Sour black mulberry has fruits with high antioxidant capacity and is rich in anthocyanins. It prevents cardiovascular diseases and provides protection against cancer. It plays an important role in protecting the brain. It is rich in vitamins C, K, E, and iron. Its fruits have been used as a traditional medicine in the treatment of many diseases, especially mouth sores, for hundreds of years. It has been reported that its health benefits are due to compounds such as phenolic acid and anthocyanin [8-11]. Sour black mulberries are generally consumed locally as they have a short shelf life and are not an attractive fruit for the market. Sour black mulberries collected in some regions are sold by squeezing their fresh juice. However, as with other mulberry species, it is used by turning into processed products such as molasses, syrup, jam and marmalade. It is sold to companies that process ice cream, jam, fruit juice, and fruit yogurt by being frozen in freezing facilities under 0°C under commercial conditions [12].

The fruits are easily damaged after harvesting due to their high moisture content. For this reason, it is necessary to subject the fruits to either drying processes or cold storage processes in order to protect them from long-term deterioration. The costs and energy consumption values of cold storage are higher than drying processes, so cold storage cannot be used by every grower. The primary goal of the drying process is to reduce water activity by largely removing product moisture. In addition, drying significantly reduces fruit weight and volume, facilitating loading-unloading and transportation processes [13]. Estimation of drying rates for thin layer drying and effective moisture diffusion factors of fruits and vegetables are important topics for drying simulation with models and are the basis of moisture transfer analysis [14]. One of the most common techniques is microwave-convective combined (hybrid) drying. Hybrid drying has a higher drying rate than conventional methods. In addition, a successful drying process is carried out due to the interaction with dipolar water molecules in the food [15]. Response surface methodology (RSM) is applied to predict drying behavior of products, optimize the drying parameters, and control and simulate the drying process [16]. RSM represents a collection of statistical and mathematical techniques and is often used for the development, improvement, and optimization of processes in which certain responses are affected by various variables [17].

Sour black mulberry is a mysterious species both in Turkey and in the world. Post-harvest preservation methods are mostly focused on other mulberry species. Determining the best drying method for sour black mulberry will make significant contributions to the literature and food industry. The innovative aspect of this study is the drying of sour black mulberries, which were not dried before and are difficult to store, by a new technique such as hybrid drying. In the literature, there are several studies on drying mulberry [13, 19, 29, 31]. However, there was no published paper regarding the optimization study to determine the best drying conditions, especially both the energy aspect and bioactive properties of the sour black mulberry. The main purposes of the present study were to determine drying kinetics, energy aspects, effective moisture diffusivity, and bioactive properties of sour black mulberry which dried in a hybrid dryer. Besides, drying temperature and microwave power were selected, and the response surface methodology approach was used to determine the effective factors and optimized drying conditions.

MATERIAL AND METHODS

Fresh fruit

Two cultivated sour black mulberry (*Morus nigra* L.) genotypes (*Morus nigra* 1: MN1, *Morus nigra* 2: MN2) were used as the material. Sour black mulberry samples were cultivated in an orchard in Kayseri province (MN1: 38°49'6.00"N,35°26'50.44"E; MN2: 38°40'54.71"N, 35°32'5.13"E) of Turkey in August 2021. Undamaged ones with similar lengths and diameters were selected and preserved at -18°C relative humidity for one week. Before drying processes, samples were washed through tap water to clean out the dust and exposed to drying with different techniques. Fruits of the sour black mulberry during harvest are given in Figure 1.

Figure 1. Fruits of sour black mulberry (*Morus nigra* L.)

Experimental setup

In the study, a hybrid dryer (Arçelik KMF 833 I, Turkey) with air convective and microwave drying processes were used for drying sour black mulberry (Figure 2). Air-convective and microwave characteristics could be used simultaneously. Microwaves are transmitted from the upper section of the oven. The dryer has 900 W output power at 2.465 MHz frequency. The temperature could be adjusted from 40 to 280°C. The dryer includes an air circulation fan and perforated polyamide platforms and trays to hold the samples. In hybrid drying, air velocity was selected as 0.5 m s^{-1} in all tests. For thin layer drying of the samples, 120±2 g, sour black mulberries at about 78.59 and 88.36% (w.b.) initial moisture content for MN1 and MN2, respectively. To determine initial moisture contents, sour black mulberries were dried in an oven (Memmert UN55, Germany) at 105 ºC for 24 h and final weights were measured. Sour black mulberries were placed into the dryer and the drying process was continued until the samples reached an equilibrium moisture. The initial average thickness values of the products were measured as about 13.50 mm and 17.00 mm for MN1 and MN2, respectively.

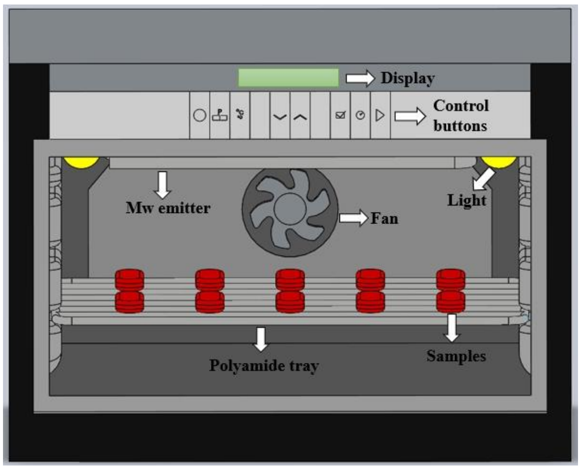

Figure 2. Schematic representation of hybrid drying system

Drying kinetics and modeling of thin layer drying

The moisture ratio (MR) of sour black mulberries during the thin-layer drying was calculated using Eq. (1) [18]:

$$MR = \frac{M_t - M_e}{M_0 - M_e} \quad \text{Eq.1}$$

Where, M_t is the moisture content on dry basis (d.b.) at drying time of t, kg kg^{-1}; M_0 is the initial moisture content (d.b.), kg kg^{-1}; M_e is the equilibrium moisture content (d.b.), kg kg^{-1} of the sample.

Experimental drying curves of the sour black mulberries were fitted to Page [18], Henderson & Pabis [19], Wang & Singh [20], Logistic [21], Newton [22] and Two-term [14] models. The mathematical modeling of drying curves was performed with SigmaPlot software (SigmaPlot for Windows version 14.0, Erkrath, Germany). The terms used to evaluate the fitting performance of the models were the determination coefficient (R^2), reduced chi-square (χ^2; Eq. (2)) and root mean square error (RMSE, Eq. (3)). The highest R^2 and the lowest χ^2, and RMSE values indicate the best model [18].

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{\exp,i} - MR_{pre,i})^2}{N - z} \quad \text{Eq.2}$$

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}(MR_{pre,i} - MR_{\exp,i})^2}{N}} \quad \text{Eq.3}$$

Where, $MR_{exp,i}$ is the experimental moisture ratio obtained from drying experiments, $MR_{pre,i}$ is the predicted moisture ratio, N is the number of experimental data and z is the number of parameters in the model. Experiments were carried out with 3 replications.

Energy aspects

In a hybrid dryer, the energy consumption (E_c) values were recorded using a power meter (Tt Technic PM-001, Turkey) in kWh. Specific energy consumption (SEC) is the energy required to evaporate the unit mass of water in the sample, energy efficiency (η_{en}) is the ratio of the heat energy used to evaporate the water in the sample to the heat supplied by the dryer and is calculated by using Eq. (4-5). Additionally, latent heat and specific heat capacity of the material were calculated using Eq. (6) [23].

$$SEC = \frac{E_c}{m_w} \quad \text{Eq.4}$$

$$\eta_{en} = \frac{m_w \lambda_{wp}}{E_c} \quad \text{Eq.5}$$

$$\frac{\lambda_{wp}}{\lambda_w} = 1 + 23\exp(-0.4X) \quad \text{Eq.6}$$

Where, m_w is the mass of evaporated water (kg), X is moisture content of samples (kg water kg dry matter^{-1}), λ_w is the latent heat of free water (J kg^{-1}) and λ_{wp} is the latent heat of product (J kg^{-1}). Experiments were carried out with 3 replications.

Biochemical properties

Extraction of the dried mulberry samples

Before bioactivity analyses, dried sour black mulberries were ground using a grinder (Bosch TSM6A014R, Germany). For the extraction of the samples, 1 g of powder sour black mulberry was weighed and 30 ml of 70% methanol and 1% of phosphoric acid were incorporated. The mixture was exposed to extraction for 30 min in a shaker and finally the samples were centrifuged at 7500 g for 5 min at +4 ºC (Sigma 4-16KS, Germany). The supernatant was filtered by 0.42 µm syringe filter and used for further analysis.

Determination of total phenolic content

The total phenolic content of the samples was determined using the modified method suggested by Singleton and Rossi [24]. For this aim, 200 µl of the extract was mixed with 1800 µl of distilled water. Then 1 mL of diluted (1/10) Folin Ciocalteau reagent (Merck, Germany) and then 2 mL of sodium carbonate (2% w/v) were incorporated. After that, the samples were incubated for 2 h at room temperature and dark conditions. After the incubation, the absorbance values of the samples were measured at 765 nm using a UV-Vis spectrophotometer (UV1800 Shimadzu, Japan). The total phenolic content of the samples was calculated as mg GAE/g sample using a calibration curve. Experiments were carried out with 3 replications.

Determination of DPPH radical scavenging activity

DPPH radical scavenging activity of the samples was determined as described by He and coauthors [25]. A 100 µL of extract sample was mixed with 3900 µL of DPPH radical solution (2,2-diphenyl-1-picrylhydrazyl, Merck, Germany) in methanol (2mM) and mixed well using vortex. After the incubation of the samples at room conditions in a dark place for 30 min, the absorbance values were recorded at 517 nm by a UV-Vis spectrophotometer (UV1800 Shimadzu, Japan). Experiments were carried out with 3 replications. DPPH radical scavenging capacity was calculated as % inhibition using the following equation):

$$\% \text{ Inh.}_{(Remaining)} = 100-[(Abs_{control}-Abs_{sample})/Abs_{control}]x100 \qquad \text{Eq.7}$$

Determination of total anthocyanin content

Total anthocyanin content was determined by using pH differential methods as suggested by Karasu and coauthors [26] and Giusti and Wrolstad [27]. One mL extract was mixed with 4 mL of buffer at pH 1 and 4.5 and incubated for 30 min at room conditions in a dark place. Finally, the absorbances of the samples for both pH 1 and pH 4.5 were recorded at 510 and 700 nm using a UV-Vis spectrophotometer (UV-1800 Shimadzu, Japan). The total anthocyanin content of the samples was calculated as mg of cyanidin-3-glucoside per kg of dried samples. Experiments were carried out with 3 replications.

$$\text{Total anthocyanin content (mg/L)} = (\Delta A/\varepsilon{*}L)10^3 * MW * DF \qquad \text{Eq.8}$$

Where, ΔA is the absorbance ($A= ((A_{510nm}- A_{700nm})\ pH_{1,0}) – ((A_{510nm}- A_{700nm})\ pH_{4,5})$), Ɛ: molar absorbance (28800), L: the cuvette length (=1), MW: molecular weight, DF: dilution factor

Data analysis, modeling and optimization

In the present study, a 3-level factorial experimental design Box-Behnken experimental design [40] with five replicates at the center point as 200 W microwave power and 60 °C air temperature was used to develop predictive models based on a second-order polynomial equation in Eq. (9) for studied parameters [41]. The two factors were used as microwave power and temperature. The regression coefficients of linear, quadratic and interaction terms were determined by using Design Expert package software for each output parameter. The computational work including designation of experimental points, randomization, analysis of variance, fitting of the second-order polynomial models and graphical representations as well as optimization was performed using a statistical package, Design-Expert® Software Version 7.0 (Stat-Ease Inc., Minneapolis, USA).

$$Y = \beta_0 + \sum_{i=1}^{N} \beta_i x_i + \sum_{i=1}^{N} \beta_{ii} x_i^2 + \sum_{\substack{i=1 \\ i<j}} \sum_{j=i+1} \beta_{ij} x_i x_j + \varepsilon \qquad \text{Eq.9}$$

where, Y is the corresponding predicted response value, b_0 is the intercept term, b_i is the linear term, b_{ii} is the quadratic term, b_{ij} is the interaction term, x_i and x_j are the coded levels of the independent variables, N is the number of input variables and ε is the standard random error.

Uncertainty analysis

Uncertainty analysis is considered a sensitive method for error analysis. It is important to test the accuracy of the measured values for the data obtained in experimental studies. The errors that may occur during the experiments are the most important factors affecting the accuracy. In this study, the total error in the measurement of a parameter was calculated with the use of Eq. (10) by taking fixed, random and process errors into consideration [42].

$$W_R = \left[\left(\frac{\partial R}{\partial x_1} w_1 \right)^2 + \left(\frac{\partial R}{\partial x_2} w_2 \right)^2 + \left(\frac{\partial R}{\partial x_3} w_3 \right)^2 + \dots\dots\dots + \left(\frac{\partial R}{\partial x_n} w_n \right)^2 \right]^{1/2} \qquad \text{Eq.10}$$

Where, R is the magnitude to be measured; x_1, x_2, x_3 and …x_n are n number of independent variables influencing this magnitude; w_1, w_2, w_3, …w_n error rates for each independent variable and W_R is total uncertainty of magnitude R.

RESULTS and DISCUSSION

Fitting of the drying curves

The moisture ratio to drying time curves for hybrid drying of sour black mulberry as affected by various air temperatures (50, 60 and 70°C) and microwave power (100, 200 and 300 W) are shown in Figure 3. Results revealed that the moisture content of sour black mulberry decreased exponentially with elapsed drying time. After the drying process, the average moisture contents for MN1 and MN2 were determined as 3.70% and 5.14%, respectively. The results showed that drying time decrease extremely when drying temperature increase. Experimental data of drying of sour black mulberry were converted into moisture ratio and fitted to six semi-empirical models. Nonlinear regression was applied to determine the parameters of the models. The R^2, RMSE and χ^2 values were calculated from the curve fitting computations for the moisture ratio with the drying time and hybrid drying conditions given in Table 1. The best model to describe the drying behavior of mulberry was selected based on the highest R^2, and the lowest χ^2, and RMSE values. In all cases, the statistical values show that all drying curves of sour black mulberry were best described by Logistic model. The R^2 values of the Logistic model varied from 0.9902 to 0.9989 for MN1 and from 0.9753 to 0.9985 for MN2, respectively. Predicted values of moisture ratio by the Logistic model were plotted against experimental data (Figure 3). If the model fits well with the drying kinetics, it can be used to describe the overall drying behavior. In addition, the model gives an idea of the drying rate and the start time of the last part of the falling rate period [28]. It was observed that the microwave power showed a significant effect for both mulberry genotypes and the changes in the drying time of the samples were demonstrated in Figure 4 ($p<0.05$, Table 3). As is seen clearly, an increase in the microwave power increased the drying time. The quadratic model explained the variation in the drying time of the samples by showing the quite high determination of coefficient for MN1 (R^2:0.983) and MN2 (R^2:0.983). Comply with the results of the present study, Rad and coauthors [29] reported that the Logistic model gave successful statistical results (R^2: 0.9969, RMSE: 0.09949) with the experimental data of infrared-convective (500, 1000 and 1500 W, 40, 55 and 70°C) dried mulberries. Amiri Chayjan and coauthors [30] reported that combined heat (40, 55 and 70°C) and microwave power (270, 450 and 630 W) assisted drying for hawthorn fruit gave the best fit (R^2:0.9992) of the Logistic model to the experimental data. Input different microwave power plays an important role in drying kinetics of sour black mulberry. The drying time decreased with the rise in microwave power from 100 to 300 W at the same levels of air temperature. Rises in microwave power, cause a rise in mass transfer and the drying process can be carried out faster. Thus, the slope of the drying curve rises with rising in microwave power. Higher drying air temperatures and microwave power result in a rise in the drying rate because these parameters cause a higher reduction of the moisture content.

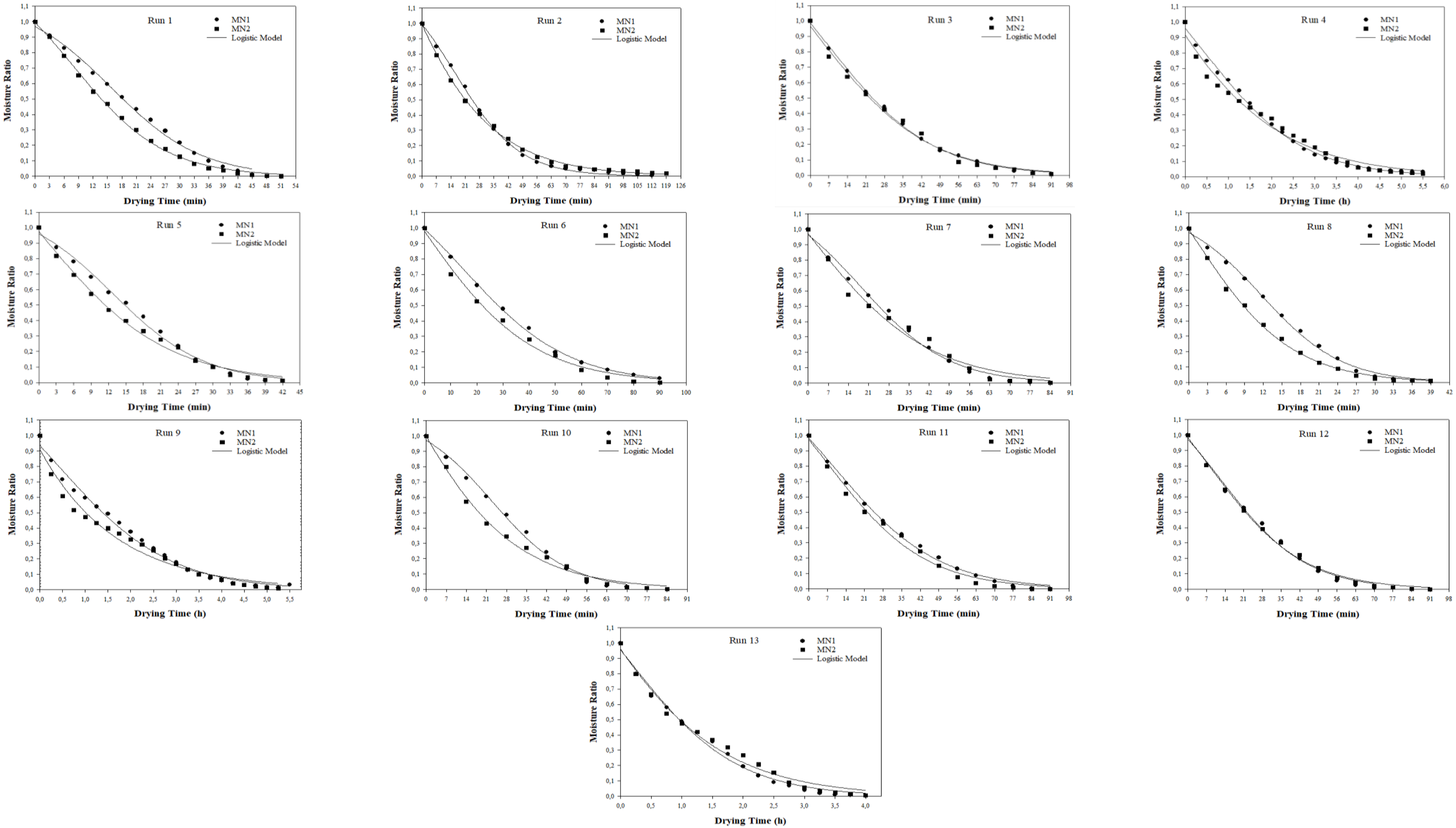

Figure 3. Drying curves of black mulberry genotypes under hybrid drying process

0 **Table 1.** Estimated values of statistical analyses obtained from various thin layer drying models for drying of black mulberry samples

No	Models	Page		Henderson & Pabis		Wang & Singh		Logistic		Newton		Two-term	
	Cultivar	MN1	MN2	MN1	MN2	MN1	MN2	MN1	MN2	MN1	MN2	MN1	MN2
	R^2	0.9910	0.9978	0.9556	0.9808	0.9977	0.9992	0.9951	0.9983	0.9438	0.9735	0.9556	0.9808
1	RMSE	0.0303	0.0130	0.0757	0.0523	0.0633	0.0402	0.0207	0.0130	0.0757	0.0523	0.0633	0.0402
	χ^2	1.05x10^{-3}	1.91x10^{-4}	6.55x10^{-3}	3.08x10^{-3}	4.58x10^{-3}	1.82x10^{-3}	5.28x10^{-4}	2.04x10^{-4}	6.12x10^{-3}	2.90x10^{-3}	5.34x10^{-3}	2.08x10^{-3}
	R^2	0.9990	0.9991	0.9823	0.9976	0.9889	0.9728	0.9989	0.9985	0.9768	0.9974	0.9823	0.9976
2	RMSE	0.0139	0.0112	0.0481	0.0147	0.0380	0.0140	0.0105	0.0112	0.0481	0.0147	0.0380	0.0140
	χ^2	2.20x10^{-4}	1.41x10^{-4}	2.62x10^{-3}	2.42x10^{-4}	1.64x10^{-3}	2.20x10^{-4}	1.34x10^{-4}	1.50x10^{-4}	2.46x10^{-3}	2.27x10^{-4}	1.89x10^{-3}	2.51x10^{-4}
	R^2	0.9975	0.9896	0.9884	0.9851	0.9981	0.9916	0.9985	0.9921	0.9860	0.9847	0.9884	0.9851
3	RMSE	0.0158	0.0254	0.0371	0.0377	0.0316	0.0370	0.0116	0.0254	0.0371	0.0377	0.0316	0.0370
	χ^2	2.91x10^{-4}	7.53x10^{-4}	1.60x10^{-3}	1.66x10^{-3}	1.16x10^{-3}	1.60x10^{-3}	1.70x10^{-4}	8.22x10^{-4}	1.48x10^{-3}	1.53x10^{-3}	1.40x10^{-3}	1.92x10^{-3}
	R^2	0.9869	0.9709	0.9797	0.9733	0.9894	0.9494	0.9902	0.9746	0.9784	0.9697	0.9797	0.9732
4	RMSE	0.0235	0.0300	0.0297	0.0389	0.0286	0.0311	0.0152	0.0300	0.0297	0.0389	0.0286	0.0311
	χ^2	6.04x10^{-4}	9.85x10^{-4}	9.65x10^{-4}	1.66x10^{-3}	8.96x10^{-4}	1.06x10^{-3}	2.64x10^{-4}	1.03x10^{-3}	9.21x10^{-4}	1.58x10^{-3}	9.90x10^{-4}	1.17x10^{-3}
	R^2	0.9894	0.9914	0.9590	0.9866	0.9955	0.9930	0.9940	0.9931	0.9509	0.9859	0.9590	0.9866
5	RMSE	0.0336	0.0242	0.0723	0.0359	0.0628	0.0344	0.0228	0.0242	0.0723	0.0359	0.0628	0.0344
	χ^2	1.30x10^{-3}	6.78x10^{-4}	6.03x10^{-3}	1.49x10^{-3}	4.55x10^{-3}	1.36x10^{-3}	6.53x10^{-4}	7.35x10^{-4}	5.60x10^{-3}	1.38x10^{-3}	5.38x10^{-3}	1.61x10^{-3}
	R^2	0.9976	0.9909	0.9820	0.9870	0.9983	0.9920	0.9986	0.9923	0.9779	0.9868	0.9820	0.9870
6	RMSE	0.0158	0.0271	0.0483	0.0366	0.0402	0.0360	0.0121	0.0271	0.0483	0.0366	0.0402	0.0360
	χ^2	3.14x10^{-4}	9.20x10^{-4}	2.91x10^{-3}	1.68x10^{-3}	2.02x10^{-3}	1.62x10^{-3}	2.08x10^{-4}	1.05x10^{-3}	2.59x10^{-3}	1.49x10^{-3}	2.70x10^{-3}	2.16x10^{-3}
	R^2	0.9900	0.9787	0.9698	0.9745	0.9973	0.9835	0.9936	0.9804	0.9658	0.9740	0.9934	0.9894
7	RMSE	0.0328	0.0427	0.0605	0.0500	0.0266	0.0319	0.0241	0.0427	0.0605	0.0500	0.0266	0.0319
	χ^2	1.27x10^{-3}	2.16x10^{-3}	4.33x10^{-3}	2.96x10^{-3}	8.34x10^{-4}	1.21x10^{-3}	7.52x10^{-4}	2.37x10^{-3}	3.97x10^{-3}	2.71x10^{-3}	1.02x10^{-3}	1.47x10^{-3}
	R^2	0.9941	0.9973	0.9571	0.9910	0.9931	0.9950	0.9971	0.9978	0.9465	0.9894	0.9936	0.9981
8	RMSE	0.0259	0.0144	0.0781	0.0322	0.0266	0.0136	0.0165	0.0144	0.0781	0.0322	0.0266	0.0136
	χ^2	7.80x10^{-4}	2.42x10^{-4}	7.12x10^{-3}	1.21x10^{-3}	8.26x10^{-4}	2.17x10^{-4}	3.48x10^{-4}	2.64x10^{-4}	6.57x10^{-3}	1.11x10^{-3}	9.92x10^{-4}	2.60x10^{-4}
	R^2	0.9852	0.9770	0.9818	0.9753	0.9882	0.9106	0.9902	0.9753	0.9818	0.9639	0.9930	0.9952
9	RMSE	0.0352	0.0358	0.0391	0.0491	0.0241	0.0179	0.0251	0.0358	0.0391	0.0491	0.0241	0.0179
	χ^2	1.36x10^{-3}	1.41x10^{-3}	1.67x10^{-3}	2.65x10^{-3}	6.34x10^{-4}	3.53x10^{-4}	7.24x10^{-4}	1.49x10^{-3}	1.60x10^{-3}	2.53x10^{-3}	7.01x10^{-4}	3.92x10^{-4}
	R^2	0.9899	0.9939	0.9538	0.9913	0.9942	0.9860	0.9935	0.9939	0.9449	0.9906	0.9943	0.9967
10	RMSE	0.0336	0.0241	0.0786	0.0300	0.0251	0.0177	0.0253	0.0241	0.0786	0.0300	0.0251	0.0177
	χ^2	1.38x10^{-3}	6.87x10^{-4}	7.55x10^{-3}	1.07x10^{-3}	7.71x10^{-4}	3.72x10^{-4}	8.82x10^{-4}	7.56x10^{-4}	6.80x10^{-3}	9.78x10^{-4}	9.92x10^{-4}	4.54x10^{-4}
	R^2	0.9951	0.9884	0.9843	0.9804	0.9988	0.9939	0.9964	0.9903	0.9816	0.9791	0.9990	0.9939
11	RMSE	0.0210	0.0298	0.0405	0.0444	0.0082	0.0229	0.0173	0.0298	0.0405	0.0444	0.0082	0.0229
	χ^2	5.15x10^{-4}	1.04x10^{-3}	1.92x10^{-3}	2.30x10^{-3}	7.75x10^{-5}	6.14x10^{-4}	3.82x10^{-4}	1.13x10^{-3}	1.77x10^{-3}	2.12x10^{-3}	9.30x10^{-5}	7.37x10^{-4}
	R^2	0.9897	0.9958	0.9758	0.9861	0.9977	0.9977	0.9924	0.9969	0.9731	0.9839	0.9971	0.9977
12	RMSE	0.0325	0.0171	0.0526	0.0403	0.0173	0.0150	0.0266	0.0171	0.0526	0.0403	0.0173	0.0150
	χ^2	1.29x10^{-3}	3.43x10^{-4}	3.38x10^{-3}	1.89x10^{-3}	3.66x10^{-4}	2.62x10^{-4}	9.75x10^{-4}	3.74x10^{-4}	3.04x10^{-3}	1.75x10^{-3}	4.70x10^{-4}	3.15x10^{-4}
	R^2	0.9878	0.9840	0.9835	0.9838	0.9899	0.9759	0.9910	0.9858	0.9833	0.9837	0.9953	0.9959
13	RMSE	0.0332	0.0330	0.0389	0.0369	0.0205	0.0184	0.0264	0.0330	0.0389	0.0369	0.0205	0.0184
	χ^2	1.25x10^{-3}	1.24x10^{-3}	1.71x10^{-3}	1.54x10^{-3}	4.76x10^{-4}	3.85x10^{-4}	8.46x10^{-4}	1.33x10^{-3}	1.61x10^{-3}	1.45x10^{-3}	5.49x10^{-4}	4.45x10^{-4}

1

Energy aspects

The variation of drying time, specific energy consumption, and energy efficiency are given in Table 2. In this study, while the energy consumption of the drying condition decreased with decreasing drying times, increasing microwave power caused an increase in E_c. Energy consumption ranged from 0.54 kWh to 1.72 for both varieties. The highest SEC values occurred as 18.59 kWh kg^{-1} and 16.16 kWh kg^{-1} for MN1 and MN2 at the 4^{th} run, respectively. The most effective conditions in terms of energy efficiency were 9.04% for MN1 and 10.40% for MN1 in the 8^{th} run, respectively. As seen in Table 3, microwave power showed a significant effect on SEC and η_{en} ($p<0.05$). The change of SEC of the mulberry samples for both genotypes showed a good fitting ability to the quadratic model as R^2:0.898 for MN1 and R^2:0.928 for MN2 and the change in SEC of the mulberry samples is shown in Figure 5. As is illustrated clearly from the figures, an increase was observed in the SEC with the increase in microwave power, and it was found that the linear effects of microwave power were found as significant ($p<0.05$, Table 3). According to these results increase in microwave power provided a significant decrement in the SEC. The variation of energy efficiency of hybrid dryers depending on the processing variables for both genotypes was shown in Figure 5 and the effects of processing variables were described well by quadratic model as R^2:0.892 for MN1 and R^2:0.937 for MN2. An increase in microwave power caused a significant increment in the energy efficiency for both genotypes.

As a result of drying time raised, energy consumption raised. With a rise in microwave and air temperature the consumed heat raised strongly, and therefore, SEC raised. Some literature results were given for similar conditions to the present study. Adabi and coauthors [31] indicated that the SEC values in the hybrid (IR-air convective) dryer for mulberry samples in the microwave pretreatment group were changed between 8.07 kWh kg^{-1} and 27.43 kWh kg^{-1}. In addition, the authors obtained similar E_c values. Contrary to the present findings, Rad and coauthors [29], for hybrid dried white mulberry samples, the highest SEC value is 916.89 kWh kg^{-1} (air velocity of 1.6 m s^{-1}, air temperature of 70°C and infrared power 500 W), and the lowest value is 113.92 kWh kg^{-1} (air velocity of 0.4 m s^{-1}, air temperature of 40°C and radiation infrared 1500 W). This is thought to be due to intense infrared powers and high air velocities. Horuz and coauthors [28] reported similar findings for the energy efficiency values in the sour cherries at 50 and 60 ºC air convective drying as 1.12% and 3.05%, respectively.

Bioactive characteristics

Bioactive properties of two different genotypes of sour black mulberry samples exposed to a hybrid drying process composed of different microwave power and temperature values were characterized and the results were tabulated in Table 2. As is seen in Table 2, the total phenolic content of the samples was in the range of 10.25-20.10 mg GAE/g and 8.77-18.08 mg GAE/g for the genotypes of MN1 and MN2, respectively. The lowest total phenolic content of the MN1 genotype (10.25 mg GAE/g) was determined for the sample dried at 100 W and 50 ºC and MN2 genotypes also showed the lowest total phenolic content under the same conditions (Table 2). As is seen from the Table 3, both microwave power and temperature showed a significant effect on the total phenolic content of the MN1 and MN2 mulberry genotypes ($p<0.05$). The change of total phenolic content of the mulberry samples for both genotypes showed a good fitting ability to the quadratic model (R^2=0.761 and 0.826, $p<0.05$) and the change in total phenolic content of the mulberry samples is seen in Figure 2 for MN1 and MN2 genotypes As is monitored clearly from the figures, an increase was observed in the total phenolic content with the increase of both microwave power and temperature and it was found that the linear effects of these two processing variables were found as significant ($p<0.05$, Table 3). According to these results increase in microwave powers provided a significant increment in the total phenolic content of the samples. Atalay and Erge [32] reported that the bioactive properties of the mushroom samples exposed to hot air and microwave drying process and they revealed that the increase in microwave power resulted in a significant increase in the total phenolic content of the final samples. They stated the total phenolic content of the mushroom sample as 1.08 and 2.83 mg GAE/g for the samples dried at 90 and 600 W, respectively. The same increase effect of microwave power on the total phenolic content (TPC) was reported by Hamrouni-Sellami [34] on the sage plant. Authors indicated that the highest TPC was found in plants dried by MW (800 W/30 g of fresh plant) and was 4.2 times higher than those of fresh plants. In another study, Wojdyło [34] revealed that the negative effect of temperature on the content of phenolic compounds was also observed during the drying of sour cherry samples by vacuum–microwave drying. The gentle final drying at 120 W instead of 240 W resulted in lower temperatures of the material (61.3 °C vs. 64.4 °C), which yielded higher contents of polyphenols in the dried product (from 7,320 to 7,587 mg/kg dm). Similar effects were also reported by Inchuen [35] on dried red curry powder.

Table 2. Mean values for the bioactive and drying parameters of black mulberry samples

Runs	MWP (Watt)	T (ºC)	MN1						MN2					
			TPC (mg GAE/g)	ARA (%)	TAC (mg/kg)	DT (min)	SEC (kWh/kg)	η_{en} (%)	TPC (mg GAE/g)	ARA (%)	TAC (mg/kg)	DT (min)	SEC (kWh/kg)	η_{en} (%)
1	300	60	18.94	82.71	252.14	45	6.49	8.04	10.07	69.83	772.63	51	6.47	8.37
2	200	60	13.63	73.00	281.42	112	10.88	4.79	11.52	65.75	528.41	119	10.34	5.24
3	200	60	16.27	70.58	157.28	91	8.89	5.87	11.69	64.67	522.71	91	7.92	6.83
4	100	60	12.78	62.58	440.65	330	18.59	2.80	9.27	62.25	787.94	330	16.16	3.35
5	300	50	19.08	83.88	258.16	42	5.96	8.83	10.45	69.50	609.58	42	5.35	10.23
6	200	50	19.07	79.13	185.18	90	8.56	6.15	8.79	55.83	651.12	90	7.39	7.40
7	200	60	15.98	76.04	155.59	84	8.10	6.44	11.30	64.33	576.99	84	7.33	7.38
8	300	70	16.16	86.00	239.40	39	5.71	9.04	18.08	83.92	163.49	39	5.15	10.40
9	100	50	10.25	63.71	456.26	315	12.07	4.36	8.77	57.71	835.81	315	10.70	5.11
10	200	70	20.10	79.29	130.93	70	7.18	7.18	14.69	76.88	346.21	84	7.54	7.11
11	200	60	18.74	75.42	76.53	91	8.75	5.96	13.58	70.50	558.05	91	7.97	6.79
12	200	60	16.61	81.54	150.61	70	6.82	7.64	12.69	70.42	586.14	91	7.87	6.88
13	100	70	16.09	73.96	271.72	240	16.18	3.19	9.64	57.58	460.93	240	14.55	3.68

MWP: Microwave power, T: Temperature, TPC: Total phenolic content, ARA: Antiradical activity, TAC: Total anthocyanin content, DT: Drying time, SEC: Specific energy consumption, ηen: Energy efficiency

Table 3. ANOVA showing the processing effects on the bioactive and drying parameters of black mulberry samples

Source	df	MN1						MN2					
		TPC	ARA	TAC	DT	SEC	η_{en}	TPC	ARA	TAC	DT	SEC	η_{en}
Model	5	4.31[a]	9.061[a]	7.18[a]	82.68[a]	12.26[a]	11.55[a]	6.66[a]	5.73[a]	8.44[a]	79.12[a]	17.20[a]	20.91[a]
A-MWP (watt)	1	10.92[a]	37.763[a]	8.17[a]	326.91[a]	49.80[a]	54.89[a]	9.37[a]	16.38[a]	5.97[a]	319.54[a]	70.16[a]	96.24[a]
B-Temperature (ºC)	1	0.76[a]	2.169	3.088	5.45	0.37	0.0011	16.27[a]	9.79[a]	26.06[a]	3.98	1.70	0.81
AB	1	5.54	1.365	1.920	4.41	1.73	0.65	5.37	2.49	0.16	4.38	2.89	1.30
A^2	1	3.94	1.431	21.21[a]	74.11[a]	9.10[a]	1.02	2.16	0.00	5.40	65.98[a]	10.10[a]	0.68
B^2	1	1.80	3.766	0.392	3.30	2.76	1.96	0.80	0.00	8.26[a]	3.53	4.81	6.16[a]
Residual	7												
Lack of Fit	3	1.10	0.35	0.20	1.64	1.62	0.27	4.10	3.89	22.23	2.37	1.06	0.42
Pure Error	4												
Cor Total	12												
R^2		0.755	0.866	0.837	0.983	0.898	0.892	0.826	0.804	0.858	0.983	0.925	0.937

MWP: Microwave power, T: Temperature, TPC: Total phenolic content, ARA: Antiradical activity, TAC: Total anthocyanin content, DT: Drying time, SEC: Specific energy consumption, ηen: Energy efficiency $p<0.01$

The main mechanism of the increased phenolic content for the samples exposed to higher microwave power is explained by the release of the phenolic compounds with the help of the microwave process. As is known microwave produce intense heat in a very short time and so, the plant cell wall polymer is deteriorated with the intense heat and caused a high vapor pressure and temperature in the tissue. After this reaction, the bounded phenolics are released and more phenolics could be extracted [33, 35]. Horuz and coauthors [36] compared the bioactive properties of hybrid and convective air-dried apricots and they concluded that the hybrid drying provided well preservation for the bioactive compounds and so showed higher total phenolic content compared to conventional drying. They reported that the increase in microwave power and temperature significantly increased the total phenolic content of the final apricot samples. The main mechanism for the increase in total phenolic content of the samples is attributed to the fast inactivation of oxidative enzymes, reduction of contact time with oxygen and light and so the phenolics are better protected (Samoticha and coauthors, 2016) [37].

Antiradical activity values of both sour black mulberry genotypes were also characterized by using DPPH radical and the results depending on the processing variables were given in Table 2. As is seen, radical scavenging capacity values of the dried mulberry samples were in the range of 62.58-86% and 55.83-83.92% for the MN1 and MN2, respectively. The lowest antiradical activity values for MN1 and MN2 mulberry samples were recorded for the samples dried at 100 W and 60 ºC and 200 W and 50 ºC, respectively. It was observed that the microwave power showed a significant effect for both mulberry genotypes and the changes in the radical scavenging activity of the samples were illustrated in Figure 4 ($p<0.05$, Table 3). As is seen clearly, an increase in the temperature and microwave power increased the radical scavenging activity of the final samples ($p<0.05$). The quadratic model explained the variation in the radical scavenging activity of the samples by showing a quite high determination of coefficient ($R^2>0.804$). It was also monitored that the increase in the total phenolic content provided an increment in the radical scavenging activity of the sample due to a positive correlation between the total phenolic and antiradical activity for both genotypes (R=0.760 and 0.909 for MN1 and MN2, respectively). Similar findings were also reported by Atalay and Erge [32] for the mushroom dried by a hybrid drying process. They revealed that the antioxidant performance of the samples significantly increased with the increase of microwave power from 90 to 600 W and they attributed this increment to the release of phenolics from the tissues by the deterioration of the cell wall structure due to the intense heat by produced the microwave. Nindo [38] indicated that the power level of 2 W/g and 60 °C heated air resulted in the greatest retention of total antioxidant activity (TAA) in the combined microwave and spouted bed drying. In all cases, the tip portion of asparagus retained more TAA after drying than either the middle or basal parts. The results of the increased antioxidant activity by the microwave power were also reported by Wojdyło [34].

The anthocyanin content of the mulberry samples was also determined and tabulated in Table 2. It was determined that the anthocyanin levels of the samples ranged between 76.53-456.26 mg/g and 163.49-835.81 mg/g for the genotypes MN1 and MN2, respectively. As is seen from the table, the highest total anthocyanin levels were observed for the samples dried at 100 W and 50 ºC for both genotypes. It was determined that both microwave power and temperature showed a significant effect on the total anthocyanin content ($p<0.05$, Table 3). The change of total anthocyanin levels of the mulberry samples depending on the processing variables for both genotypes was shown in Figure 4 and the effects of processing variables were described well by the quadratic model ($p<0.05$, $R^2>0.837$). An increase in the microwave power and temperature caused a significant decrease in the anthocyanin content for both genotypes. Wojdylo and coauthors [34] investigated the effect of convective and vacuum–microwave drying on the bioactive properties of sour cherry, and they reported that the anthocyanin levels decreased with the increase in microwave power and temperature. In this study, cyanidin-3-*O*-glucoside which is a popular anthocyanin compound were determined as 5.77 and 5.03 mg/kg for the fresh and freeze-dried cherry sample. After the heat treatment during drying, namely the convective or vacuum microwave process, its level decreased significantly and was determined as 4.63, 4.23 and 3.56 mg/kg for the sample dried at 50, 60 and 70 ºC, respectively. Similarly, the cyanidin-3-*O*-glucoside level was determined as 4.10 and 3.59 mg/kg for the sample dried at 240 and 360 W, respectively. Sun and coauthors [39] studied the effect of microwave-assisted foam-mat drying on the anthocyanin content and stability of blue honeysuckle and they reported that the increase in microwave power and temperature resulted in a decrement in the anthocyanin levels because of the degradation. They also reported that the anthocyanin degradation level increased with the increase in drying time and temperature although the moisture levels of the samples provided a decrease in the degradation ratio.

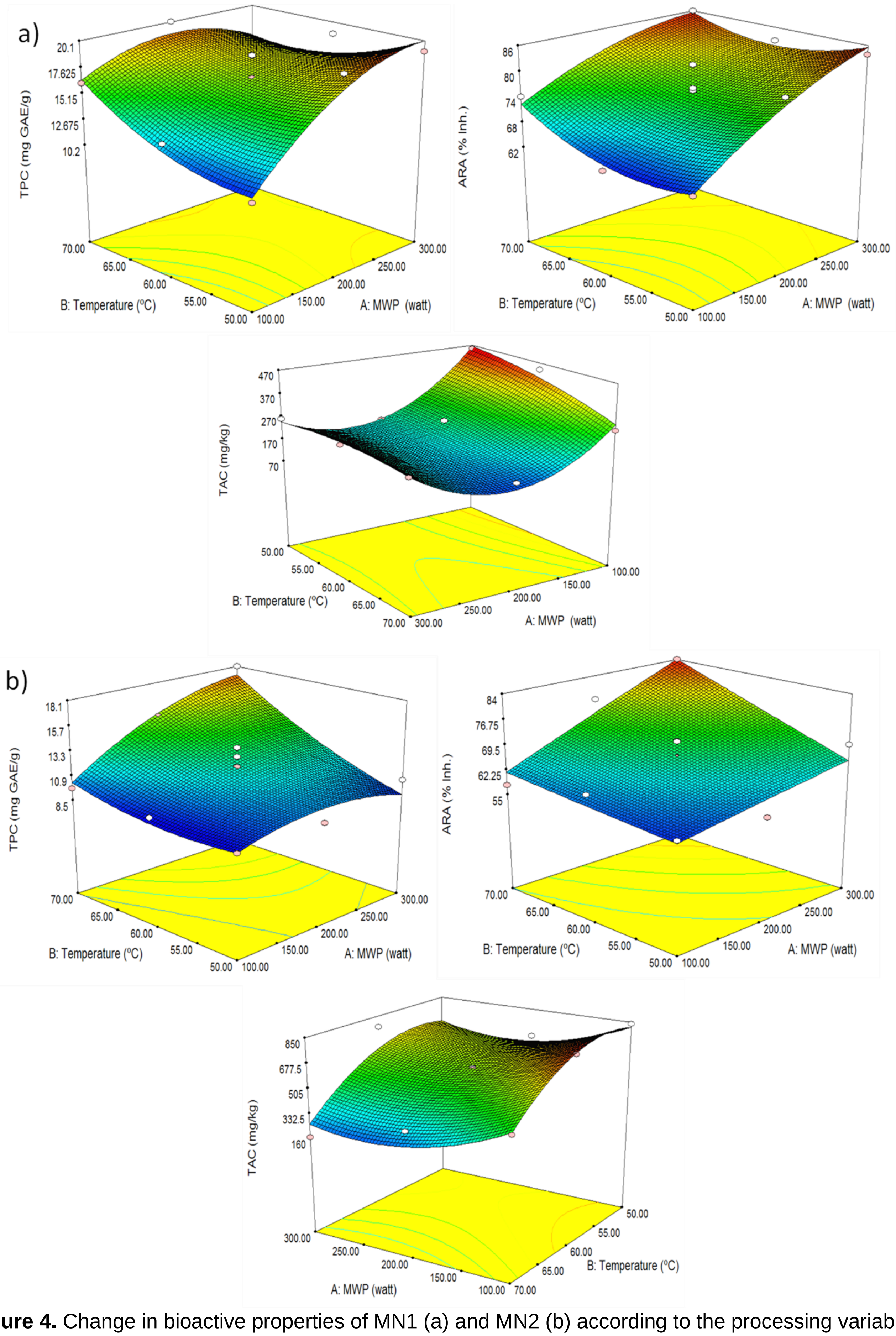

Figure 4. Change in bioactive properties of MN1 (a) and MN2 (b) according to the processing variables

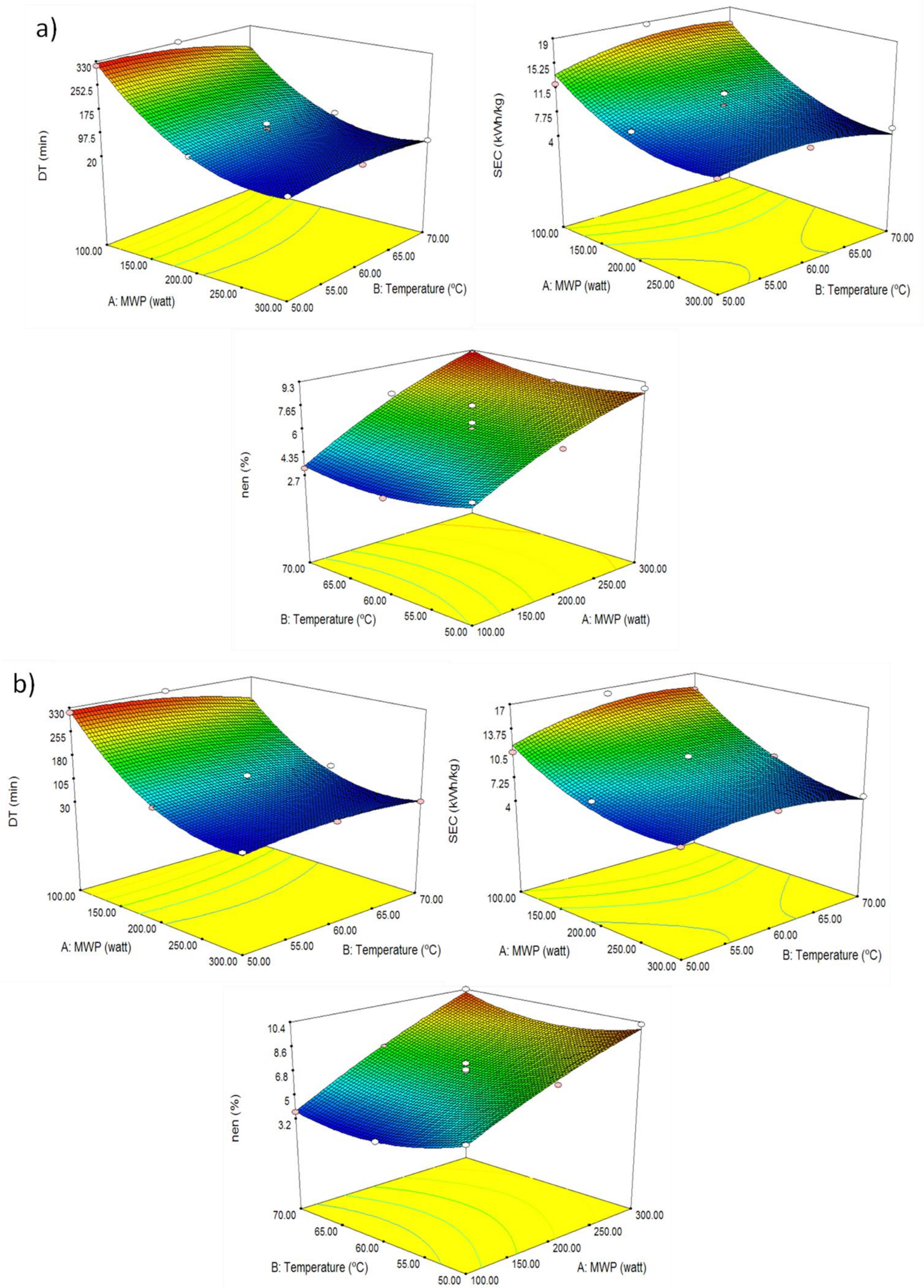

Figure 5. Change in drying parameters of MN1 (a) and MN2 (b) according to the processing variables

Optimization of the bioactive and drying parameters

Optimization studies showed that the maximum total phenolic content would be at 300 W and 50.5ºC as 20.10 mg GAE/g for MN1 and 300 W and 70 ºC as 17.2 for MN2. For the minimum total phenolic content, the microwave power would be at 100 W and the temperatures at 50 and 55 ºC for MN1 and MN2 genotypes, respectively. Similarly, the maximum radical scavenging activity of MN1 and MN2 would be at 300 W and 70 ºC as 85.61% and 80.37%, respectively. The minimum radical scavenging performance for MN1 would be at 100 W and 55ºC as 62.99% and for MN2, 103W and 50 ºC as 53.69%. For the maximum total anthocyanin content for MN1 would be at 100 watt and 50 ºC as 457.8 mg/kg and for MN2, 102W and 54.5 ºC as 836.4 mg/kg. For the optimization of drying parameters, the minimization process revealed that the minimum drying time for MN1 (31.9 min) and MN2 (34.7 min) would be at 262.3W and 67.9 ºC and 288.2W and 50.76 ºC, respectively. In addition to that, it was calculated that the minimum SEC values for MN1 (5.58 kWh/kg) and MN2 (5.02 kWh/kg) for 279.22 and 68.1 ºC and 291.4W and 69.4 ºC while the maximum η_{en} values for MN1 (9.1%) and MN2 (10.2%) for 295.8 W and 69.4 ºC and 300 W and 70 ºC. Additionally, multiple response optimization was carried out for the studied parameters for both mulberry genotypes and it was concluded that the maximum TPC, ARA, TAC, η_{en} and minimum DT and SEC values would be at 300 W and 50 ºC (desirability=0.842) and 300 W and 66.5 ºC (desirability=0.744), for MN1 and MN2, respectively. Optimization results revealed that the hybrid drying combined with conventional, and microwave resulted in a good performance for the drying mulberry samples.

Uncertainty analysis of measurements

Uncertainty of measured parameters and total uncertainties in E_c, SEC, η_{en}, TPC, ARA and TAC calculations are provided in Table 4. Present uncertainty values identified for drying analysis of sour black mulberries were quite below the acceptable limit of 5% [43].

Table 4. Uncertainties of the experimental measurements

Parameter	Unit	Value
Temperature measurement	°C	±0.1
Drying time measurement	h	±0.1
Weight of samples measurement	g	±0.01
Mass measurement	g	±0.01
Moisture content	%	±1.21%
Total uncertainty for E_c	kWh	±3.14%
Total uncertainty for SEC	kWh kg^{-1}	±2.08%
Total uncertainty for η_{en}	%	±2.27%
Total uncertainty for TPC	mg GAE g^{-1}	±2.68%
Total uncertainty for ARA	%	±2.39%
Total uncertainty for TAC	g kg^{-1}	±2.19%

CONCLUSION

In the present study, MN1 and MN2 sour black mulberry varieties were dried using a hybrid drying method and different drying parameters effects were evaluated to determine the best drying conditions showing high bioactive samples and low drying time. Experiments were carried out according to the designs created by RSM using the Box-Behnken design and the desirability function process was applied for the optimization. The optimization results showed that hybrid drying is applicable for the mulberry samples at 300 W and 50-60 ºC. According to the optimization results, the maximum TPC, ARA, TAC, η_{en} and minimum DT and SEC values would be at 300 W and 50 ºC (desirability=0.842) and 300 W and 66.5 ºC (desirability=0.744), for MN1 and MN2, respectively. The Logistic model was determined as the best mathematical model because it fits the experimental data very well. The findings of the research provided the optimum drying conditions for the sour black mulberry for the food drying industry based on air temperature and microwave power.

In recent years, the development of drying techniques and methods has not only enabled the preparation of dried products in a wide range, but also significantly improved the quality, stability and functional requirements of the products and made them economically advantageous. As a result of this study, it was aimed to optimize the drying process in order to make the parameters and the process efficient, to determine the appropriate drying method especially for fruits with a short shelf life, and to contribute to the widespread use of drying by increasing its applicability in the industry.

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