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Textural Properties Changes During Facile Drying of Sour Cherry Using Ultrasound Waves: Investigation of Sonication Parameters Using Response Surface Methodology

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ABSTRACT: There are a number of fruit drying methods and hot air flow drying has been considered the most common one. However, it is not an economical way due to the huge amount of energy consumption. Using ultrasound waves as a pre-treatment in drying may rise drying efficiency as well as alleviating the cost level. In this research, drying of sour cherry has been accomplished, applying ultrasound waves as a pre-treatment. In this case, the effects of ultrasound parameters i.e., sonication temperature, power intensity, and sonication time are discussed. The preliminary results illustrate the role of ultrasound waves as a pre-treatment, in which the drying time is already reduced from 7 hours to 3 hours; on the other hand, the maximum water loss has been achieved with sonication temperature of 30° C, power intensity of 147 w/cm^2 and sonication time of 60 min. Moreover, the amount of moisture loss is surged through increasing the ultrasound exposing time, as compared to a conventional sample. It is worth mentioning that among these mentioned parameters, the temperature plays the most significant role on the water loss. Consequently, using ultrasound waves is a promising way for reducing the temperature, time and energy in hot air flow drying process.

Keywords: *Sour Cherry, Sonication Parameters, Textural Properties, Ultrasound-Assisted Drying.*

Introduction

Sour cherry is mainly produced in Germany, Hungary, Turkey and Azerbaijan. Sour cherry is used in order to produce a wide array of products such as beverages, sauces, jams. Sour cherries are rich in antioxidants such as phenolic compounds, especially anthocyanins. Furthermore, their stability depends on temperature, action light, oxygen, etc. Drying is one of the efficient solutions in order to increase the shelf-life of sour cherry (Doymaz, 2004). Water is one of the most determinative compounds in food products, affecting the physical and chemical properties of the foodstuff (Lerici *et al*., 1988; Lewicki, 2004). Since water content is a crucial parameter for the micro-organism growth, it should be removed from food products in order to increase their shelf-life. Numerous techniques including canning, pasteurization, bottling, irradiation,

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preservatives have been widely applied to increase the shelf-life of food products (Kouassi & Roos, 2001). Yet, one of the most widely used ways of keeping food products is drying them. Drying of food products can be carried out traditionally or by combination of modern methods, based on their application. It has been widely accepted that, one of the most well-known ways for drying the foodstuff is employing hot air; in which the usage of hot air is not considered as an economical means of drying, from the point of energy as well as cost Additionally, due to the reduction of the food thermal conductivity through the hot air drying process, the rate of heat transfer into the inner parts of the food would be reduced.

A pre-treatment method can not only facilitate the mass transfer operation during drying of the foodstuff also remove a part of their moisture content (Vahideh *et al*., 2008). A variety of methods are available as pre-treatment methods, including osmotic dehydration, ultrasound waves, microwave, etc. One of the contemporary means of drying pre-treatment is the usage of ultrasound waves, in which these waves have been applied before drying using hot air (Gallego-Juarez *et al*., 2007). The usage of ultrasonic waves may increase the moisture penetration coefficient and dropping the drying time during the drying step through the use of hot air. The acoustic cavitation produced during the irradiation can keep the porosity in the food sample, which facilities the flow of water out of the solid structure of the food sample via forming the microscopic channels. As a result, the mass transfer would increase through drying using hot air (Fernandes *et al*., 2006; Isadora *et al*., 2006). The usage of ultrasound waves is an efficient method to reduce the temperature, time and energy consumption in hot air drying (Adu & Otten, 1996). Siucinska *et al*. (2015) analyzed the effects of ultrasound on the quality and nutritional aspects of dried sour cherries throughout shelf-life. They concluded that the application of ultrasound resulted in the mass transfer enhancement after osmotic dehydration (higher dry matter content and lower water activity with higher ultrasound power). Storage caused the deterioration of anthocyanin content and after eight weeks of storage significant differences between samples became visible. Samples, furthermore, treated by ultrasound method have had a lower antiradical capacity compared to those are not treated. It can be anticipated that ultrasound application is going to make a negative impact on anthocyanin content and may speed up the loss of antioxidant potential. In 2017, Correa et al examined influence of ultrasound application on both the osmotic pretreatment and subsequent convective drying of pineapple. It was demonstrated that a diffusion model permitted to quantify the influence of the factors studied (time of pretreatment, ultrasound application during pretreatment, drying temperature and ultrasound application during drying) in drying kinetics. The increase in drying temperature and the application of ultrasound during drying significantly accelerated the drying process by reducing both the internal and the external mass transport resistance. On the contrary, the osmotic pretreatments reduced the drying rate by increasing the external resistance. Comparing to the other researches in this case , the current study is focused on using a non-thermal pre-treatment i.e., ultrasound waves before the hot air drying step as well as, study of the effects of sonochemical parameters i.e., ultrasonic power intensity, sonication time and temperature on textural properties of dried sour cherries in order to facilitate mass transfer, accelerate the drying time, reduce the amount of wrinkles, maintain tissue integrity and increase rehydration compared to the conventional method of drying. Additionally, in order to investigate the interaction of different sonochemical variables and statistical analysis, response surface methodology (RSM) method is used.

Materials and Methods

- *Raw Material*

Sour cherries were purchased from the local market located in Karaj (Iran); after harvesting the fruits were washed, frozen, and finally stored until processing.

- *Pre-treatment with Ultrasonic Waves*

Ultrasonic transducer (UP200Ht model, manufactured by Hielscher, Germany) is employed (25 KHz, 200 W). Sour cherry samples (fruit to syrup ratio of 1:4) were irradiated by ultrasound waves at various sonication times, power intensity and sonication temperature. The sonication temperatures were maintained at 10, 20 and 30°C, while the sonication times lasted 30, 45, 60 min and the power intensity was 27, 75, 147 W/cm^2, respectively (Table1). The experiments were carried out in triplicate order. (Siucinska, Mieszczakowska-prac, potubok & Konopacka, 2014b).

- *Drying*

After sonication, the sour cherries were transferred to hot air dryer and dried at 80^{0}C.Moisture loss of the samples is calculated according to equation (1). (Jazini & Hatamipour, 2010 & Ioannou et al., 2011 & Menges & Ertekin 2006).

$$WL^{1}\ (\%) = \frac{Wi - Wf}{Wi} \times 100 \quad (1)$$

Where, Wi is the primary weight of the sample before drying (g) and W_f is the final weight of the sample after drying (g).

- *Control Sample*

Control sample (i.e., conventional sample) was only dried with conventional hot air dryer at 80 $^\circ$C with rate of 80 m/s.

[1] Water Loss

- *Physicochemical Analyses*

Physical and chemical analyses were carried out after drying of all samples. pH, soluble solids content, total phenolic compounds, ascorbic acid, antioxidant activity, the total amount of anthocyanin, color, shrinkage, sensory test, and BET of samples were measured.

The soluble solids content is analyzed using the refractometer method (Siucinska & Konopacka, 2014). The total phenolic content was determined according to Folin-Ciocialteu method (Chaovanalikit & Wrolstad, 2004). The ascorbic acid content was determined with the use of 2, 6-Dichloroindophenol as a titrant. The total amount of anthocyanin in sour cherry was determined according to the differential pH method (Ersus & Yurdagel, 2007), based on the dominant anthocyanin of sour cherry i.e., cyanidin-3-glucoside (Ferretti *et al*., 2010). The antioxidant activity was determined according to Shimada method (Shimada, 1992) using free radicals, such as DPPH. The color assessment was measured at the beginning of storage and immediately after drying with the colorimeter (CR-2600d Konica Minolta Sensing, INC (Japan)). The colors of the sample were indicated by CIElab color scales L, a, b. Total color change (ΔE) was calculated according to equation (2). (Dadali *et al*., 2007 & Falade *et al*., 2007).

$$\Delta E= [(L^*-L)^2+ (a^*-a)^2+ (b^*-b)^2]^{0.5} \quad (2)$$

Where, L* is lightness (+) or darkness (−), a* is red (+) or green (−) and b* is yellow (+) or blue (−).

The shrinkage percentage was determined according to (Yan et al., 2008) by using a solvent (Toluene) displacement method (equation3).

$$V=\frac{M}{\rho}=\frac{m-m'-m''}{\rho} \quad (3)$$

Where,
V= Volume of toluene shifted (cm^3)
m= Pycnometer mass contains toluene and sour cherry sample (g)
m´=Sour cherry mass (g)
m̋= Empty pycnometer mass (g)
ρ= Toluene density (g/cm^3)

The values of the coefficient of shrinkage and the volume ratio of cherry samples were measured according to the equations 4 and 5, (Dissa et al., 2008 & Seiiedlou et al., 2010).

$$Sh=[1-\frac{Vt}{V0}] \quad (4)$$

Where,
Sh= Percentage of wrinkles
Vt= The volume of dried samples in the desired moisture content (cm^3)
V0= Initial volume of samples before drying (cm^3)

$$DR=[\frac{Vt}{V}] \quad (5)$$

Where, D_R is Volumetric ratio.

The BET surface area of the optimum ultrasonic sample and control sample measured by the nitrogen adsorption. Sensory evaluation was investigated according to (Serdaroglu & Ozsumer, 2003) by five persons which were expert in the Hedonic test scale and five-point method.

- ***Statistical Analysis***

Response surface methodology is commonly used to design the experiments and it minimizes the numbers of experiments for a specific number of factors and their levels, possessing manifold advantages over the Taguchi method of design. Experiments are conducted as per the experimental design and the responses such as the moisture content are recorded. Analysis of variance has been accomplished so as to identify the factors significantly affecting the responses. Regression equations are developed in order to predict the response, while the process parameters are optimized for obtaining a specific objective function (Chelladurai, 2020).

Accordingly, 27 treatments tested by Design-Expert software (version 10.0.4.0) are presented in table 1. 95% confidence interval is used to evaluate the effect of each parameter. Each experiment, however, was repeated three times. Moreover, for the remaining qualitative factors, the one-way ANOVA was applied for data gathered, checking the influence of the ultrasound waves. As a consequence, the statistical significance was determined using a Duncan multiple range test at $P<0.05$.

Results and Discussion

As can be observed in Figure 1, through the surge of time, the moisture content of the sour cherries samples is reduced. One the hand, in conventional drying method, sour cherry samples are required up to seven hours to reach a constant moisture content, eventually, at the end of the drying period, due to the hardening of the upper crust of the samples, they hardly lose their moisture. Therefore, samples dried with the conventional method require more time to be dried in comparison with sonication samples. On contrary, the optimal ultrasonic sample is required only up to three hours to be dried. This may illustrate a positive effect of employing ultrasound waves through the drying process. It can be attributed to the acoustic cavitation phenomenon resulting in hot spots with high temperature and pressure of 5000K and 1000 atm, respectively (Askari & Halladj, 2012).

- ***Effects of Ultrasound on Physicochemical Characteristics of Dried Samples***
- ***pH***

As can be observed in Table 2, applying ultrasound waves do not have a significant effect on the pH of dried sour cherry. Then, pH value of the control sample is closed to that of the optimum ultrasound sample (S_{10}).

Table 1. Impact of ultrasonic pretreatment on the moisture loss before conventional drying step

Sample	Power intensity (w/cm^2)	Sonication time (min)	Sonication temperature (°C)	Moisture loss (%)
S1	75	60	30	2.9
S2	27	45	20	1.6
S3	75	45	30	2.5
S4	147	60	10	2.35
S5	27	30	30	1.75
S6	75	60	10	1.9
S7	75	45	10	1.75
S8	75	30	20	1.85
S9	27	60	30	2.75
S10	147	60	30	4
S11	147	45	10	2.25
S12	27	30	10	0.9
S13	75	60	20	2.1
S14	147	60	20	2.75
S15	75	30	10	1.5
S16	27	60	20	1.9
S17	147	45	20	2.5
S18	147	30	20	2.15
S19	147	45	30	3.25
S20	75	30	30	2.25
S21	75	45	20	2
S22	27	45	10	1.35
S23	27	30	20	1.4
S24	27	45	30	2.25
S25	147	30	10	2
S26	147	30	30	2.5
S27	27	60	10	1.5

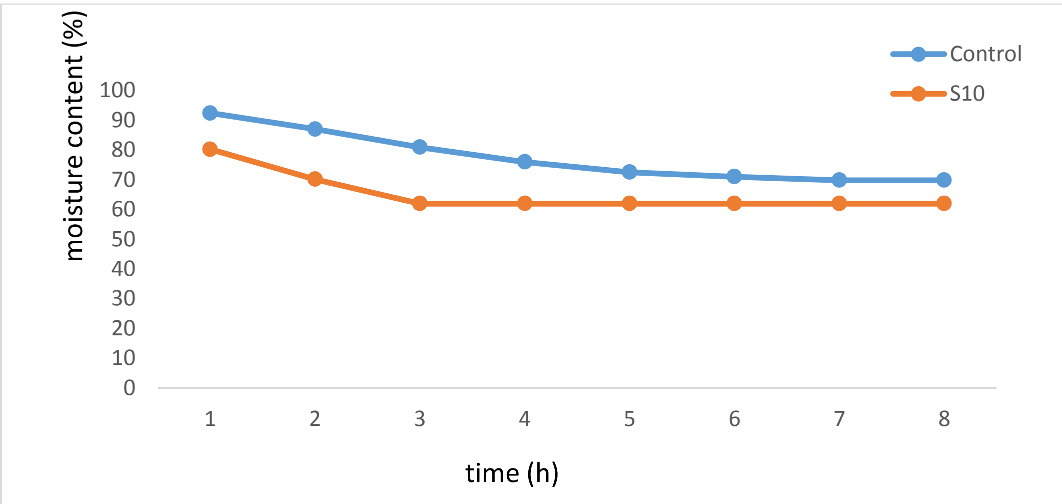


Fig. 1. Comparison of moisture loss of optimum ultrasonic sample (S_{10}) with control sample.

Table 2. Physicochemical properties of the fresh, optimal ultrasonic and control samples

Sample	pH	Brix (%)	Total phenolic (mg/ml gallic acid)	Ascorbic acid (%)	Anthocyanin (μg/100g dw)	Antioxidant properties (%DPPH scavenge ring)	L*	a*	b*	ΔE
Fresh sample	3.873±0.005^{b}	21.643±0.045^{c}	210.846±0.041^{a}	10.770±0.052^{a}	27.080±0.030^{a}	8.323±0.025^{a}	24.366±1.106^{a}	5.920±0.262^{a}	2.96±0.23^{b}	-
Optimal ultrasonic sample (S_{10})	3.756±0.005^{c}	24.083±0.076^{b}	195.850±0.050^{c}	6.580±0.080^{b}	22.210±0.030^{c}	6.840±0.040^{c}	20.593±0.710^{b}	4.040±0.250^{b}	2.51±0.36^{b}	12.72±0.18^{b}
Conventional sample	3.746±0.005^{c}	26.636±0.060^{a}	196.800±0.040^{b}	2.423±0.075^{c}	24.233±0.035^{b}	7.760±0.040^{b}	9.733±0.115^{c}	1.670±0.253^{c}	1.34±0.20^{c}	13.65±0.05^{a}

Means in columns marked with the different letter do differ significantly according to Duncan´s multiple range test, $P<0.05$.

- ***Soluble Solids***

The results show that the amount of brix of the ultrasound sample is less than that of the control sample, owing to a fact that a part of the soluble solids enters into the water when applying ultrasound waves. When the high power intensity of ultrasonic waves is propagated in the environment, microbubbles with oscillating pressure are formed, in which bursting increases the temperature and ambient pressure, therefore providing a situation for leaving soluble solids from the sour cherry sample (Table 2).

- ***Total Phenolic Compounds***

The results show that the amount of total phenolic of the ultrasonic sample is less than the control sample. The compelling reason is that much more soluble solids enter into the water by applying ultrasound waves due to high temperature and pressure of local hot spots. Indeed, a higher driving force for mass transfer is produced (Table 2).

- ***Ascorbic Acid***

The amount of ascorbic acid in the samples is reported in Table 2. It can be observed that the amount of ascorbic acid of the ultrasonic sample is much higher than the control sample. The ascorbic acid is so sensitive to heat; so, the more heat provided, the more decomposition of ascorbic acid of the samples is expected. Since the conventional sample is much more affected by the heat for a longer time, the ascorbic acid amount of the control sample is sharply reduced compared to the optimum ultrasound sample (S_{10}).

- ***Total Anthocyanin Compounds***

Making a comparison between the amount of anthocyanin compounds of the ultrasonic sample and that of the conventional sample (Table 2) shows that these compounds are reduced by applying ultrasound waves. Consequently, it can be explained that applying ultrasound waves as a pre-treatment before drying leads to occurring more mass transfer operation of the anthocyanin compounds from sour cherry into the water.

- ***Antioxidant Activity***

The difference in the antioxidant activities is shown in Table 2. The ultrasonic process reduces the antioxidant activity of dried cherries probably due to the degradation of antioxidant compounds during ultrasound treatment which has been attributed mainly to free radical production. i.e., hydroxyl radical by ultrasonic irradiation in the aqueous solution demonstrated by equation (6) (Talebi *et al*., 2010).

$$H_2O \xrightarrow{\text{Sonication}} {}^{\bullet}OH + {}^{\bullet}H \qquad (6)$$

- ***Colorimetric Test***

The changes in LAB color parameters of the stored sour cherry samples are presented in Table 2. The results show that the ultrasonic sample has a higher L* value, as a result, a higher luminosity. While it also possesses a higher amount of a* value or more redness as well as higher b* compared to those of the conventional sample. Also, the total color change during the conventional hot air drying is more which can be caused by thermal treatment. It may be strongly related to pigment degradation, and the formation of brown pigments via non-enzymatic (Maillard reaction) and enzymatic reactions (Dueik, Marzullo, & Bouchon, 2013).

- ***The Sensory Evaluation***

According to Table 3, the application of ultrasound has a significant effect on the sensory properties of dried samples. Pretreated samples with ultrasound significantly possess more acceptability of color, taste, texture, shrinkage, and overall acceptance which can be attributed to less destruction of samples using ultrasound irradiation as a reversible driving force for mass transfer operation with the formation of micro-channels. Contrary to the ultrasonic sample, the control sample is exposed to the high temperature for a much higher time which has an obviously undesirable effect on its sensory properties.

Table 3. The sensory evaluation and textural properties of dried control and optimum ultrasound sample (S10).

Sample	Sensory evaluation			Shrinkage (%)	Textural Properties		
	Color	Flavor	Texture		BET (m^2/g)	Total pore volume (cm^3/g)	Average pore diameter (nm)
Optimal ultrasonic (S_{10})	4.563 ± 0.202^a	4.290 ± 0.259^a	4.643 ± 0.272^a	0.380 ± 0.03^b	2.12×10^{-3}	3.19×10^{-3}	1.64
Conventional sample	3.013 ± 0.041^b	3.910 ± 0.101^b	2.326 ± 0.130^b	0.593 ± 0.02^a	0.346×10^{-3}	0.455×10^{-3}	1.64

Means in columns marked with the different letter do differ significantly according to Duncan´s multiple range test, P<0.05

- ***Shrinkage***

Table 3 also presents the shrinkage of control and ultrasonic samples. It is obvious that the ultrasonic sample has less shrinkage compared to the conventional one. Ultrasound irradiation disrupts the cellular structure and results in pores which are larger than those in fresh samples. This fact can improve the drying rate by making an easier water pathway, this in turn, leads to a higher moisture diffusivity, lower drying time, and finally lower shrinkage (Rodríguez *et al*., 2014).

- ***BET Analysis***

The textural properties of the optimum ultrasonic sample and the control sample measured by the nitrogen adsorption desorption experiment are listed in Table 3. In the case of sample S10 dried using ultrasonic pre-treatment, the surface area is significantly upper than that of dried by the conventional method. It can be explained by the less destruction of sour cherry samples treated by the ultrasound during the drying. Applying ultrasound before hot air drying results in formation of micro-channels as the extra paths in sour cherry structure which later may help easy diffusion of water from solid to hot air. The diffusion limitation for water molecules trapped in the center of fruit solid, therefore, is removed and there is no need for exposure the sample to the heat for a long time which can cause deep destruction of fruit cells in order to diffuse out the water. Additionally, total pore volume of ultrasonic sample is 3.19×10^{-3} cm^3/g, which is almost six times greater than that of conventional sample. This result is in consistent with the BET surface area result as well as the percent of shrinkage, showing the positive effect of ultrasonic pretreatment in keeping the structure of fruits throughout the drying process. Figure 2 shows mesopore size distribution (BJH) of the optimum ultrasonic sample and control sample. Both samples presented a broad range of pore size distributions, with pore size peaking at 1.6 nm.

- ***Effect of Sonication Parameters on Moisture Content***

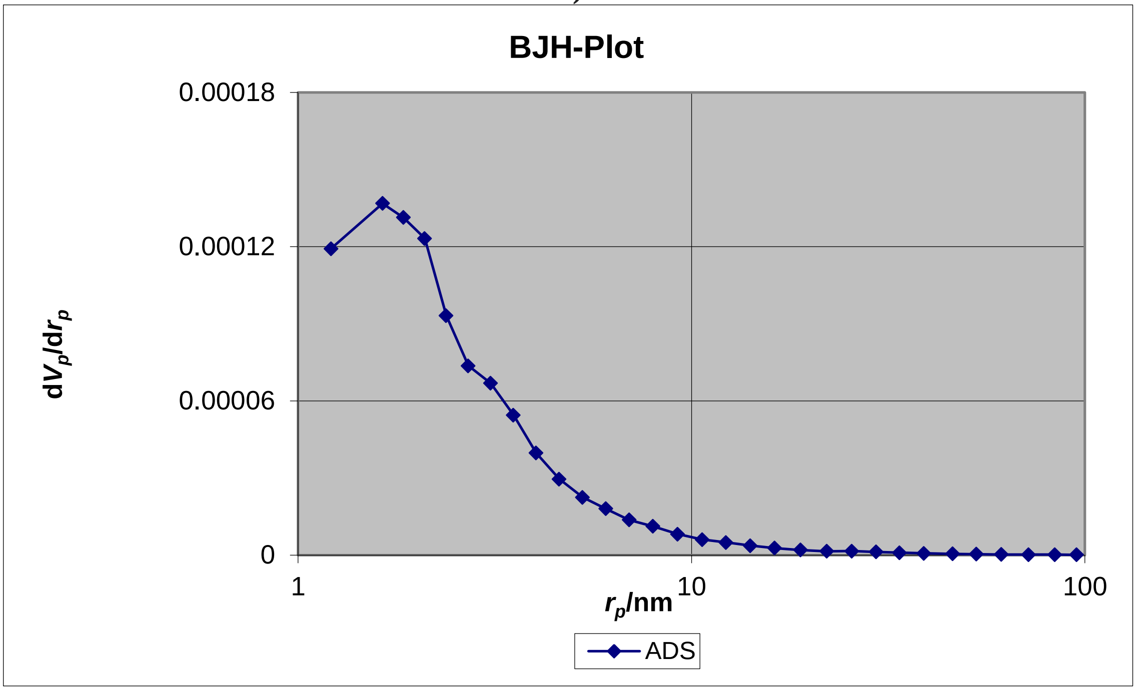
Figure 3 shows the effect of the sonication temperature on the moisture content. It can be seen that increasing the sonication temperature leads to higher moisture loss. In fact, the high operating temperature increases the maximum temperature and pressure during the bubble collapse, while it ultimately increases the moisture loss of the product. The effect of the ultrasonic power intensity on moisture content is shown in Fig .4. With increasing the power intensity, the moisture content decreases. This can be explained by increasing the mass transfer following the phenomenon of acoustic cavitation with the formation of local hot spots (Askari & Halladj, 2012).

To verify the role of ultrasonic irradiation time on the ultrasonic samples, samples S10, S19, and S26 were synthesized. Ultrasonic irradiation time was adjusted from 30 to 60 min while maintaining the same ultrasonic intensity level at 147 W/m2. The changes in moisture content of the samples with different irradiation times are shown in Fig 5. As can be seen, increasing the sonication time results in higher moisture loss. In fact, the maximum applied time, which is 60 min, leads to highest moisture loss.

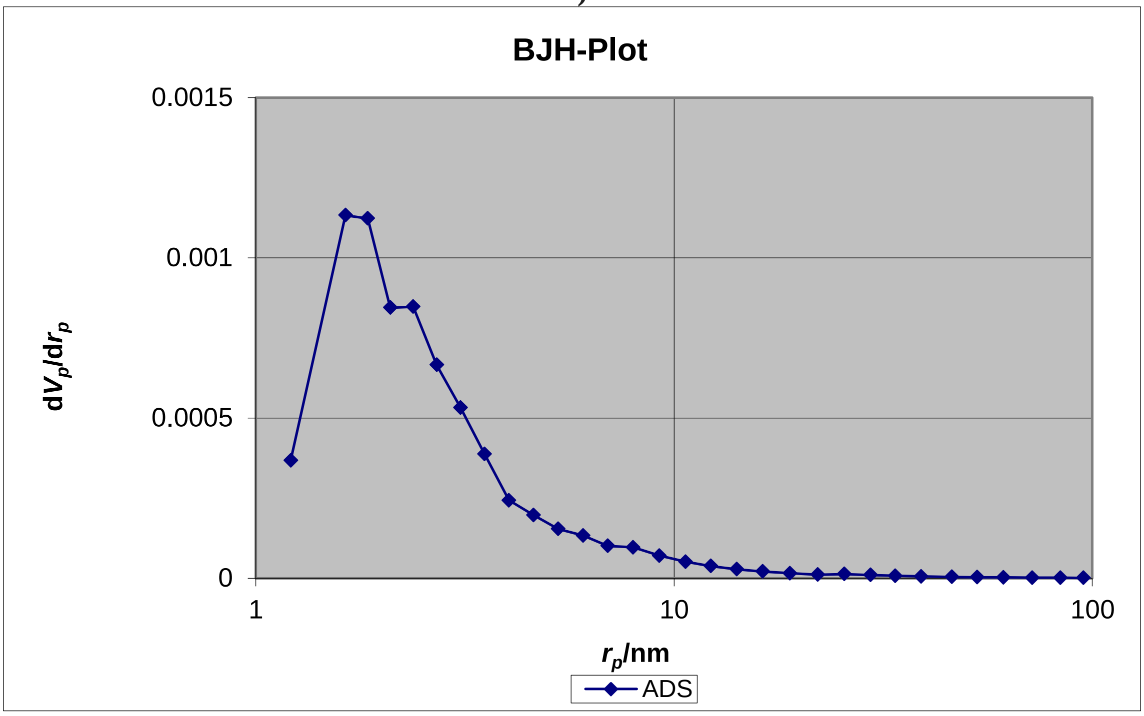
- ***Analysis of Variance (ANOVA)***

According to the ANOVA analysis for the response (moisture content) in Table 4, the fitting model possesses a low amount of P-value (0.0001). This shows that the probability of being incorrect for the model is less than 0.01%, hence the fitting model is significant. Accordingly, sonication temperature, sonication time, and power intensity are considered as the main effective parameters on the responses (Roohollahi *et al*., 2018).

a)



b)



(rp: pore radius)

Fig. 2. Mesopore size distribution of a) conventional sample b) optimum ultrasonic sample.

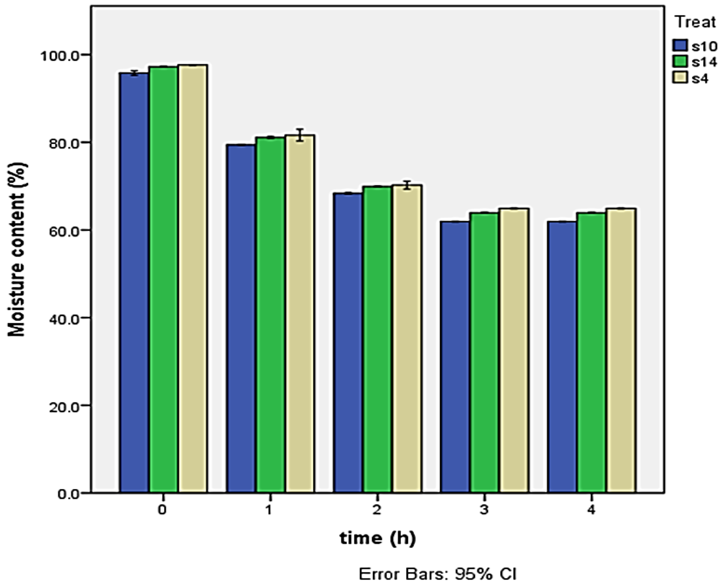


Fig. 3. Effect of sonication temperature on moisture content.

Table 4. Variance analysis table (ANOVA) for moisture content (%)

Parameters	Sum of Square	df	Mean Square	F-value	p-value Prob>F	Std.Dev
Model	0.41	4	0.10	86.97	<0.0001 significant	0.034
A-Amplitude	0.15	1	0.15	132.09	<0.0001	
B-Time	0.076	1	0.076	64.83	<0.0001	
C-Temperature	0.17	1	0.17	141.75	<0.0001	
BC	0.011	1	0.011	9.21	0.0061	
Residual	0.026	22	1.173E-003			
Cor Total	0.43	26				

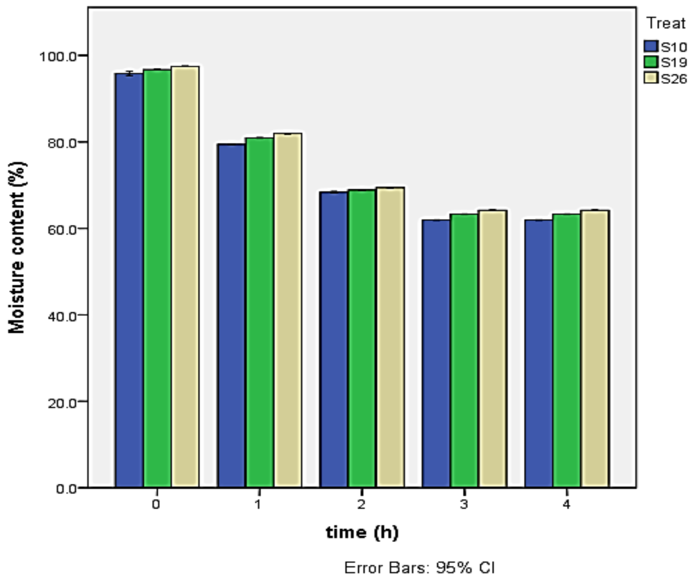


Fig. 5. Effect of sonication time on moisture content.

- ***Normal Plot of Residuals***

Figure 6 shows a normal plot of residuals according to the statistical analysis. It can be observed that it is approximately linear supporting the condition that the error terms are normally distributed. Therefore, it can be concluded that the chosen model is a satisfied one.

- ***Interaction of Effective Parameters on the Moisture Content***

Figure 7 shows the interaction of temperature and time on the moisture content of the ultrasonic sample and the weight loss of the sample. It can be seen that at a typical temperature, increasing the irradiation time results in decreasing moisture content of the samples. Furthermore, this effect can be more noticeable at a higher temperature.

The interaction between the sonication temperature and ultrasonic power intensity (amplitude) on the response is shown in Figure 8 Both parameters have a significant effect on the moisture loss of samples. Increasing the power intensity of ultrasonic pretreatment leads to the formation of more acoustic cavities which produce more hot spots after their collapsing. On the other hand, increasing the sonication temperature can increase the maximum temperature and pressure of hot spots. Former and later both increase the driving force for mass transfer of water. Figure 9 shows the interactive effect of sonication time and power intensity on moisture content. Through increasing sonication time and power intensity, the amount of water loss would be risen

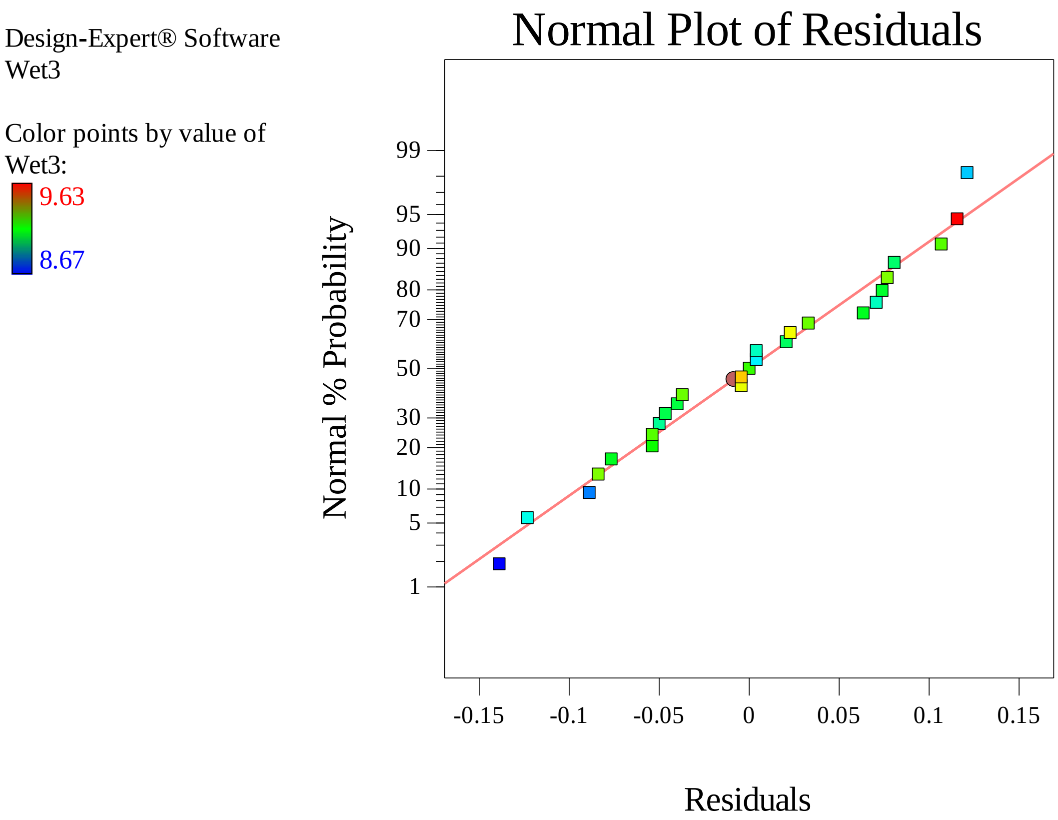


Fig. 6. Normal plot of residuals.

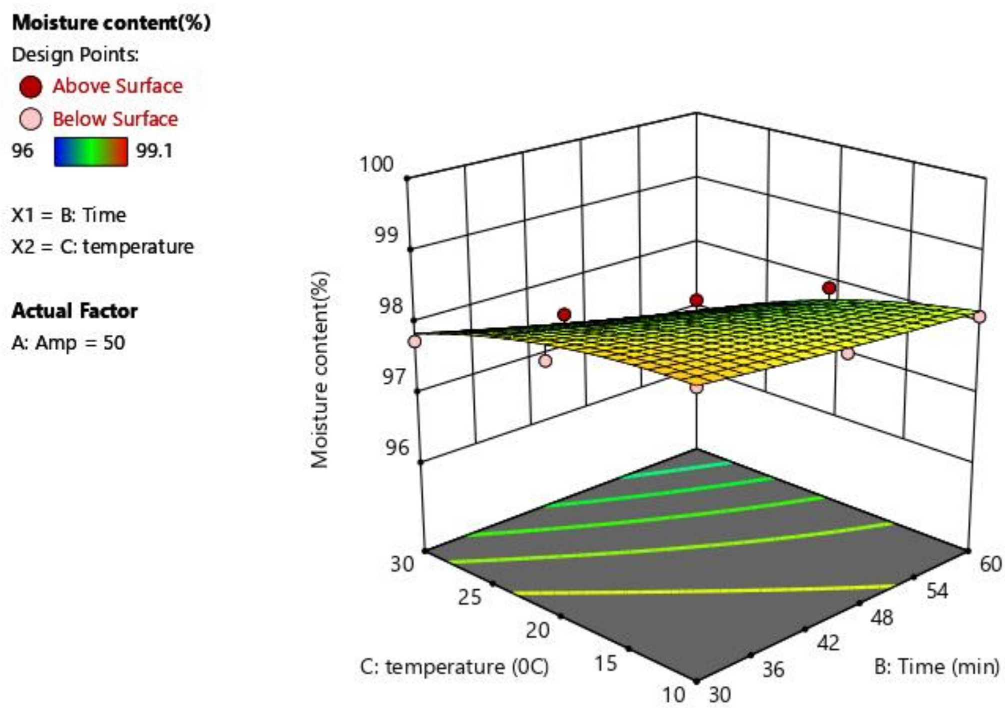


Fig. 7. Three-dimensional graph of the interaction of temperature and sonication time on moisture content (%).

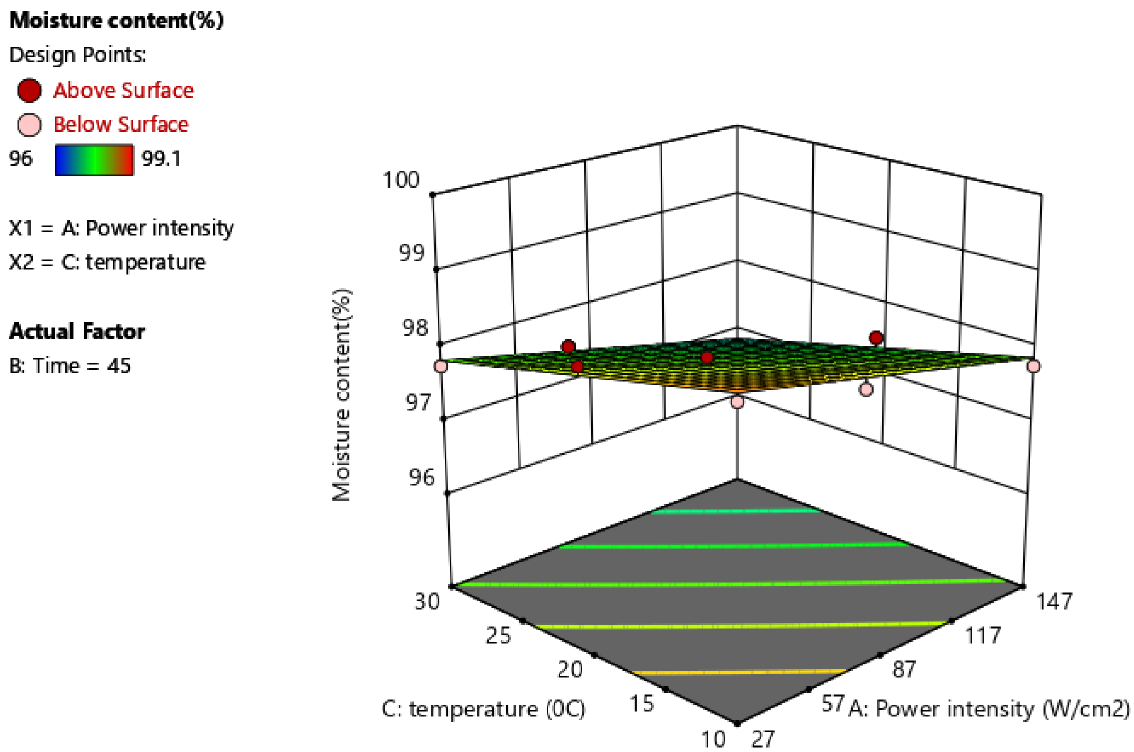


Fig. 8. Three-dimensional diagram of interaction of sonication temperature and power intensity on moisture content (%).

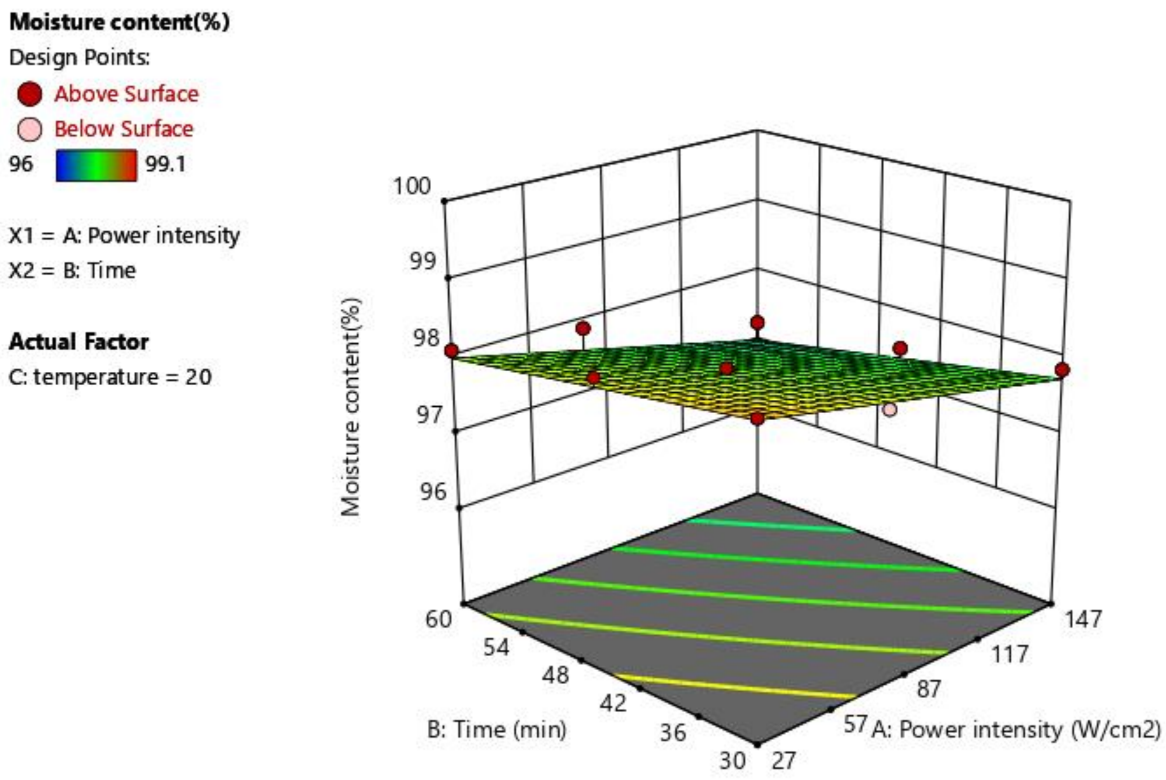


Fig. 9. Three-dimensional graph of interaction between sonication time and power intensity on moisture content (%).

Conclusion

The application of ultrasound in drying of sour cherries results in enhancement of mass transfer before extra drying with oven. Additionally, this study shows the positive effect of ultrasonic irradiation on color and sensory properties as well as improvement of physicochemical properties of samples during the drying process. In fact, the usage of ultrasonic pre-treatment, due to the creation of micro-channels, facilitates the mass transfer operation and accelerates the drying process. Therefore, the application of this non-thermal new technology can overcome some barriers to the drying of heat-sensitive foodstuffs. As a result, with an increase in the time of applying ultrasound waves, the moisture content is reduced faster than the conventional drying. The results of the experiments show that the highest amount of moisture loss belongs to the sample irradiated at 30 °C under ultrasonic pre-treatment, power intensity of 147 w/m^2, over a period of 60 minutes. Using ultrasound waves as a pre-treatment in drying, increases drying efficiency as well as reducing the cost. Using ultrasound waves is a promising way of reducing the temperature, time, and energy in the hot air flow drying method.

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