



Response surface Optimizing of the mint leaves weight loss, phenolic compounds and antioxidant properties variation during the infrared drying process

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ARTICLE INFO

Article History:

Received 2022/ 12/ 13
Accepted 2023/ 01/ 28

Keywords:

Infrared drying,
Mint leaves,
Phenolic compounds,
Antioxidant properties,
Response surface method.

DOI: 10.22034/FSCT.19.133.141
DOR: 20.1001.1.20088787.1401.19.133.12.7

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ABSTRACT

In this study, the effect of infrared drying process condition (drying temperature and time) on weight loss, total phenolic compounds and antioxidant properties of mint leaf was investigated and optimized by response surface methodology. The results showed that with increasing drying temperature, the weight loss of mint samples decreased, but with increasing drying time up to 176 minutes, the weight loss of the samples increased and then decreased. Drying time had a greater effect on weight loss changes in samples compared to drying temperature. The trend of changes in total phenolic compounds of samples with drying time was similar to the weight loss of samples, but with increasing drying temperature, the amount of total phenolic compounds of samples increased. The DPPH radical scavenging ability of the samples decreased with increasing drying time (up to 238 minutes) and then increased with increasing drying time. The results showed that applying drying temperatures up to 55°C increased DPPH radical scavenging ability of samples but applying higher drying temperatures led to a decrease in DPPH radical inhibition ability of the samples. Increasing the drying temperature increased the total antioxidant capacity and reduced the ferric reducing activity of the samples. Also, increasing the infrared drying time led to a decrease in the total antioxidant capacity of the samples. The best conditions for infrared drying of mint leaves are using drying temperature of 56.58 °C for 52 minutes and by applying the optimal conditions, the amount of weight loss (moisture loss), total phenolic compounds, and DPPH inhibition percentage of produced dry mint were 76.62%, 8.722 mg/gram of dry weight, 91.97% respectively and the desirability of this optimal condition was 0.727.

1. Introduction

Fruits and vegetables are highly perishable products due to high humidity and enzyme activity, and for their long-term storage, various methods such as heating, using cold, using preservatives and reducing humidity are used [1]. Drying, as one of the oldest methods of food preservation, is a very complex process in which mass and energy transfer occur simultaneously. During the drying process, food products undergo a series of physicochemical changes and the quality characteristics of the product, such as shape, color, texture, taste, etc., the cost of transportation, packaging and warehousing of the product [3]. Due to their long duration, drying processes may have severe adverse effects on the nutrient composition, qualitative and chemical characteristics of the product [4]. The main adverse changes in food during drying include color and nutritional value changes due to browning reactions, odor, decomposition and reduction of fat-soluble vitamins due to oxidation of fats, color loss due to decomposition of carotenoid pigments, wrinkling and lack of proper water reabsorption. The product and its solubility, adverse texture changes (hardening and rubbery product shell) and finally the loss of volatile organic compounds are responsible for creating flavor in food [5-7]. Also, the bioactive compounds in the food such as phenolic compounds, antioxidants and vitamins in it are affected. Many factors such as drying time and temperature, size and thickness of the food, distance from the heat source, moisture level, surface of the food and pre-treatments before drying, etc., are effective on the drying speed of the food [8]. Today, manufacturers seek to increase production with lower costs and shorter process time, while maintaining and improving product quality, so a lot of research has been done in the field of fast food drying methods, such as microwave drying, deep-frying drying, and combined dryers. has taken so that they can increase the drying speed and reduce the adverse effects of prolonged drying time on the food. Infrared rays increase the drying speed and have a higher efficiency by creating quick and direct heat on the product. The infrared rays are absorbed by the surface molecules of the food, which causes the product to heat up quickly and reduce the thermal stress

in the product, thus maintaining its quality better [9].

Many researches on the effect of deep-fried drying on the quality and efficiency of drying eggplant [10], tomato slices [9], pomegranate seeds [11], parboiled rice [12], edible mushrooms [13] and apricots [14]. There have been. The results of these researches all confirmed the significant effect of infrared drying in increasing the drying speed and removing moisture from the food texture and reducing the drying time. Reducing the process time during drying leads to the production of a product with minimal loss of nutrients and undesirable color changes. Also, the results showed that by increasing the power of the irradiation lamp and decreasing the distance of the sample from the lamp, the effective diffusion coefficient of moisture increases and the rate of weight loss (drying) of the product increases. In the field of the effect of drying on the bioactive compounds and antioxidant properties of fruits and vegetables, not much research has been done in the country, so in this research, the effect of the parameters of deep-fried drying temperature and time on the weight loss, the amount of phenolic compounds and the changes in the antioxidant properties of mint is investigated and The possibility of producing optimal dried mint was investigated using the infrared drying method and optimized by the response surface method.

2- Materials and methods

2-1- Deep fry drying

In this study, mint was purchased from the local market in Hamedan and dried after washing at temperatures (40-60°C) and times (50-300 minutes) in an infrared dryer (Kern/Sartorius equipped with a scale with an accuracy of 0.00001). became The temperature range and time used for drying were selected through trial and error and reaching equilibrium moisture. The amount of weight loss of mint leaves during drying was expressed as moisture loss and was calculated from equation (1):

$$WR = \frac{M_0 - M_t}{M_0} \times 100$$

In the above relation WR, the weight loss percentage of mint leaves, M_0 The initial mass of mint leaves (gr), M_t The mass of mint leaves after drying at time t is (gr) [15-16].

2-2- Measuring the amount of total phenolic compounds

The amount of total phenolic compounds was investigated using Folin Ciocalto's colorimetric method [17]. In this method, the total amount of phenolic compounds was calculated in terms of mg of gallic acid per gram of dry sample. The procedure was that 20 microliters of the prepared extract was mixed with 1.16 milliliters of distilled water and 100 microliters of Folin reagent was added to the above solution. After 5 minutes, 300 microliters of 20% sodium carbonate solution was added to the solution and the samples were kept in a Bain-Marie at 40°C for 30 minutes after stirring with a tube stirrer. Then the absorbance of the samples was read with a spectrophotometer at a wavelength of 760 nm. Gallic acid was used as a standard to draw the calibration curve and the results were calculated in terms of milligrams of gallic acid per gram of dry weight of the sample [17].

2-3- Measurement of antioxidant activity by reducing power method

The ability of extracts to reduce trivalent iron was determined by the method of Yildirim et al. (2001) [18]. In this method, 1 ml of different extracts were used and mixed with 2.5 ml of phosphate buffer (pH=6.6 and M=0.2) and 2.5 ml of potassium ferricyanide (10 g/l) and left for half an hour at room temperature. It was kept at 50 degrees Celsius. Then, 2.5 ml of trichloroacetic acid (100 g/l) was added to the above mixture and mixed completely. Then the samples were centrifuged (g1700) for 10 minutes. After that, 2.5 ml of the supernatant solution was mixed with 2.5 ml of distilled water and 0.5 ml of iron (III) chloride (1 g/l) and the absorbance was read at a wavelength of 700 nm. The amount of absorption shows the regenerative power of the extracts [19-20].

2-4- Measurement of antioxidant activity by DPPH radical scavenging method

2 and 2-diphenyl-1-picrylhydrazyl (DPPH) is a lipophilic radical that has a maximum absorption at 517 nm. To measure the percentage of DPPH absorption of extracted extracts, it was done as follows.

First, 1 ml of a 1 mM DPPH methanolic solution was mixed with 3 ml of the solution of the produced extracts and mixed vigorously. The resulting mixture was kept for 30 minutes at room temperature in the dark and finally their absorbance was read at 517 nm wavelength. The activity was calculated in terms of the

relative percentage of DPPH according to equation 2 [21-22].

$$= \text{DPPH active radical inhibition percentage} \\ 100 \times \frac{\text{Absorption percentage of the control sample} - \text{Absorption percentage of the experimental sample}}{\text{The absorption percentage of the control sample}}$$

5-2- Measurement of antioxidant activity by total antioxidant capacity method

This method is based on the reduction of molybdenum (VI) to molybdenum (V) by the sample and the formation of a green molybdenum phosphate complex in an acidic medium. In this method, 0.1 ml of each of the produced extracts was poured into an Eppendorf tube and 1 ml of the reagent solution (a mixture of 0.6 M sulfuric acid, 28 M sodium phosphate and 4 M Ammonium molybdate) was added to it. After sealing, it was kept in a Bain-Marie (95°C) for 90 minutes, and after cooling to room temperature, the absorbance was read at 695 nm wavelength against a control. The control solution contained 1 ml of the reagent solution and 0.1 ml of the used solvent, which was incubated under the same conditions as the rest of the samples [23-24].

6-2- Statistical analysis and optimization

Optimizing the drying conditions of mint leaves under the effect of drying time and temperature using the response surface method (Design Expert 11.1.2.0 software)¹(and the central composite plan)²CCD) was performed with 3 levels and 5 repetitions at the central point (+1, 0, -1) (Table 1). A range of independent temperature variables (₁X) and drying time (₂X) was deduced from the preliminary tests. The experimental plan with the actual levels of the independent variables of infrared drying time and temperature and the results related to changes in weight, antioxidant properties and phenolic compounds of mint leaves under the influence of independent variables using the response surface method are presented in Table (2).

Table 1 Independent variables and their applied levels for optimizing infrea red drying of mint leaves

Independent variables	Variables level		
	-1	0	+1
Drying temperature (°C)	40	50	60
Drying time (minute)	52	176	300

¹ . Design Expert, 11.1.2.0 Trial, Stat-Ease Inc.

² . Central Composite Design

2-4-1- Validation of optimal conditions for drying mint leaves

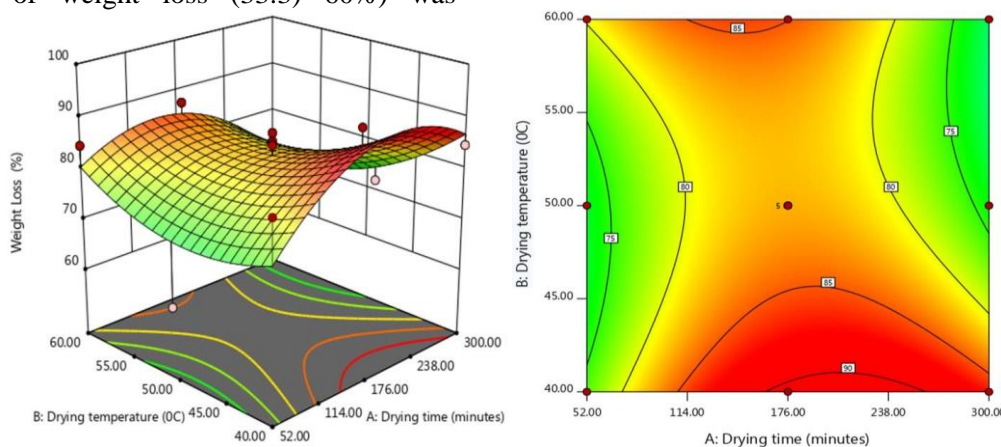
In order to evaluate the efficiency and accuracy of the models and relationships resulting from the optimization, as well as to validate the optimal conditions resulting from the response surface method, mint leaf samples in the optimal conditions of temperature and time, dry and qualitative characteristics such as weight loss, the amount of total phenolic compounds and properties. The antioxidant capacity of the production samples was investigated and evaluated.

3. Results and Discussion

3-1- Moisture changes (weight loss) of mint leaves during infrared drying

As in Fig(1) It can be seen that with the increase of drying time up to 176 minutes, the rate of weight loss increases, while in longer drying times (above 176 minutes), the rate of weight loss of the samples decreases. Also, with increasing drying temperature, the weight loss (moisture loss) of mint leaves has a downward trend, and this trend increases slowly at higher drying temperatures. As can be seen, with increasing drying time, the weight loss of mint leaves has a steeper slope than increasing the drying temperature, which shows the greater effect of time. Drying depends on the weight loss of mint leaves compared to the drying temperature. The three-dimensional and two-dimensional curves of changes in weight loss (moisture loss) show that the highest amount of weight loss (87.30%) corresponds to the temperature range of 60 degrees Celsius and drying time of 176 minutes, while the lowest amount of weight loss (33.3) 60%) was

observed at a drying temperature of 50 °C and drying times less than 52 minutes. As can be seen, drying time and temperature have an intensifying effect on the weight loss of mint leaves, but at the end of the drying time, the weight loss curve begins to decline, which can be due to reaching the decline period during drying. During drying, due to the closing of the pores and capillary tubes inside the food tissue, due to the phenomenon of surface hardening.³ The amount of moisture leaving the food is reduced and the drying speed is fixed and reduced. The results of this research were consistent with the results of Mazandarani et al. (2017), Salehi et al. (2017) and Aghajani et al. (2021). These researchers stated that the drying time and temperature had a significant effect on the weight loss of pomegranate seeds and eggplant slices, and with increasing drying time and temperature, the weight loss of these products increases [4, 10-11]. Aghajani et al. (2021) stated that With increasing drying time and temperature, the weight loss (moisture loss) of Pomegranate seeds had an upward trend, and with increasing drying temperature, the weight loss of Pomegranate seeds had a steeper slope compared to increasing drying time, which indicates the greater effect of temperature. Drying was on the weight loss of pomegranate compared to drying time. The results of these researchers showed that the highest amount of weight loss (78.85%) was related to the temperature range of 71-75 degrees Celsius and the drying time was 370-530 minutes, while the lowest amount of weight loss (48.42%) was in the temperature range of Drying less than 58°C and drying times less than 290 minutes were observed.



³. Case hardening

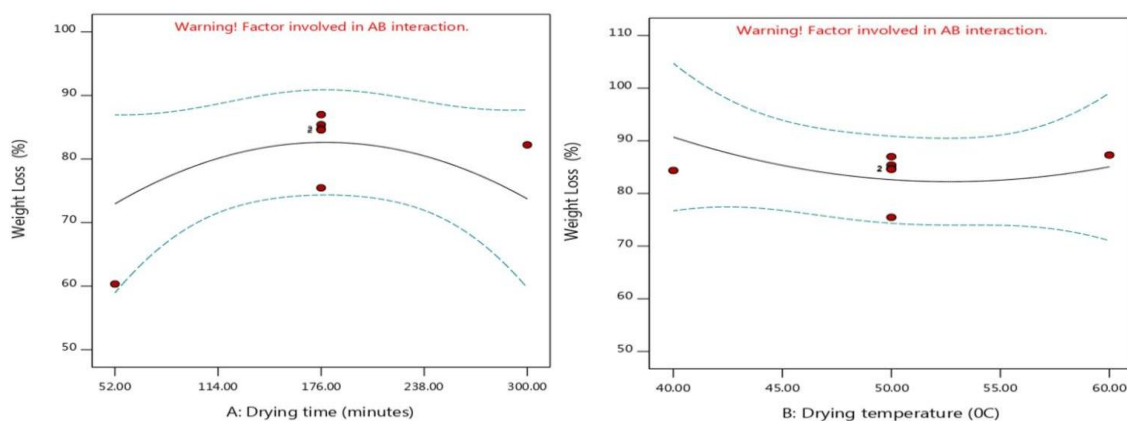


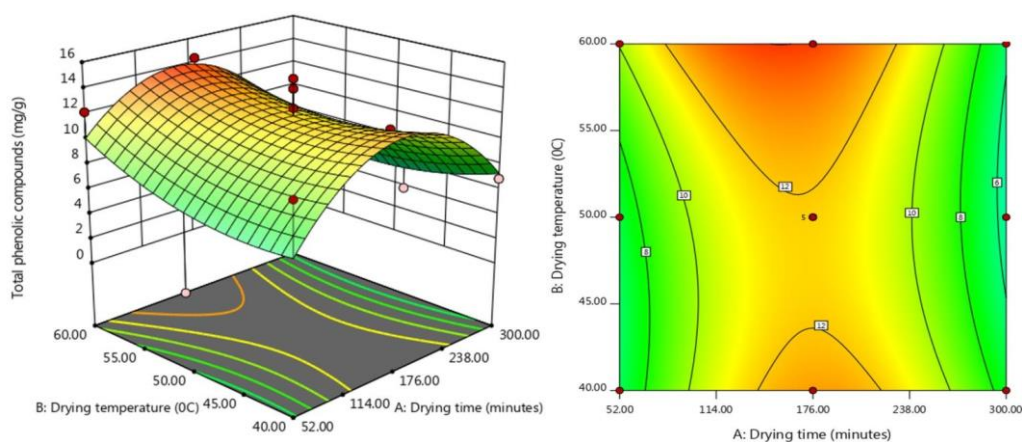
Fig 1 3D surface, contour plot and one factor effect of the simultaneous effect of different drying temperature and drying time on water loss (moisture content) of infrared dried mint leaves

2-3- Changes in the amount of phenolic compounds of whole mint leaves during infrared drying

The trend of changes in vitamin content of phenolic compounds of whole mint leaves under the influence of infrared drying time and temperature is shown in Figure (2). As can be seen, by increasing the drying time up to 176 minutes, the amount of total phenolic compounds increases. There is an upward trend, while at longer drying times (above 176 minutes) the amount of total phenolic compounds decreases. Samples are in a downward trend. Also by increasing the drying temperature, total phenolic compounds of mint leaves have an upward trend with a constant and slow slope.

As can be seen, drying time has a more severe destructive effect than drying temperature on the amount of total phenolic compounds. So

that with the increase of drying temperature, the amount of total phenolic compounds shows a slow and slight upward trend, but with the increase of drying time, the amount of total phenolic compounds shows a more drastic change. Total phenolic compounds are heat-sensitive compounds that are quickly oxidized so that they begin to decompose and decrease rapidly with increasing drying time and temperature. In order to maintain the nutritional value and prevent the degradation of phenolic compounds, as well as to achieve the highest drying efficiency, the use of high-temperature-short-time processes (HTST) is recommended, therefore, by using temperatures higher than 70 °C and drying times less than 176 minutes, the loss of total phenolic compounds is lower, and at the same time, the amount of moisture is also reduced to an optimal level [21].



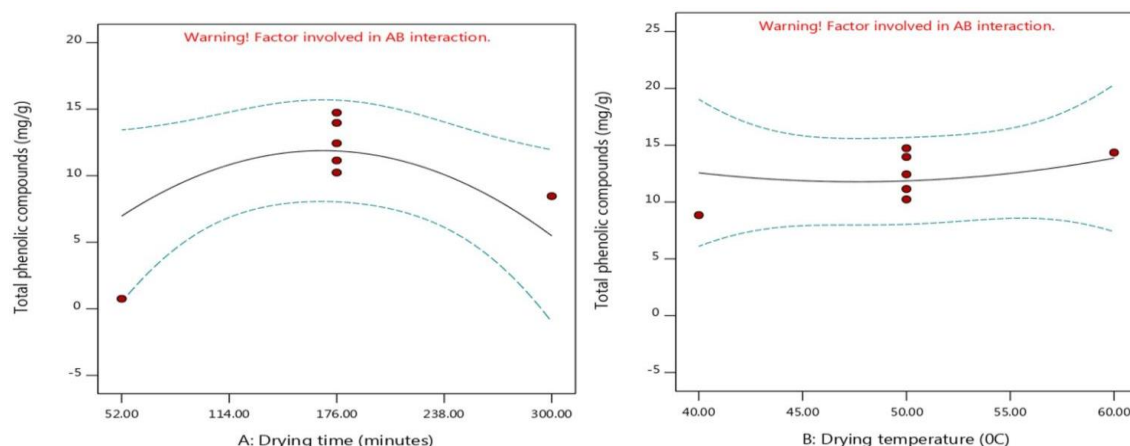


Fig 2 3D surface, contour plot and one factor effect of the simultaneous effect of different drying temperature and drying time on total phenolic compounds of infrared dried mint leaves

3-3- Changes in the antioxidant properties of mint leaves during infrared drying

Figure 3-5 shows the changes in the antioxidant properties of mint leaves during infrared drying. As can be seen in Figure 3, the amount of DPPH radical inhibition decreases with increasing drying time up to 238 minutes and then increases with increasing drying time and shows an increasing trend with increasing drying temperature up to 55°C and application of dry temperatures. A higher concentration leads to a decrease in the DPPH radical scavenging ability of the samples. The DPPH free radical scavenging ability of the samples in the range of drying time between 114-238°C decreases with increasing infrared drying temperature, while it slightly decreases in the temperature range of less than 50 and above 55°C with increasing drying time (Fig. 3).

As can be seen in Figure 4, with increasing drying temperature, the antioxidant capacity of dried samples has a slight increasing trend, while it decreases with increasing infrared drying time (especially at high drying temperatures). The results of Fig 5 It shows that the characteristic of regeneration power of dried samples decreases with increasing drying temperature, while with increasing drying time, it first decreases and then increases after 176 minutes.

During drying, various physicochemical changes occur in the food material, which changes the quality characteristics of the dried final product compared to the initial fresh product [25]. Color is an important quality characteristic of dried products and it changes during drying and long-term storage due to some chemical and biochemical reactions. Different colorants including anthocyanins, chlorophyll and phenolic compounds are the main factors that form the color, appearance

and sensory characteristics of the product and have a significant impact on its marketability. These characteristics are affected by the type of process and play a significant role in determining the final price of the product. 26].

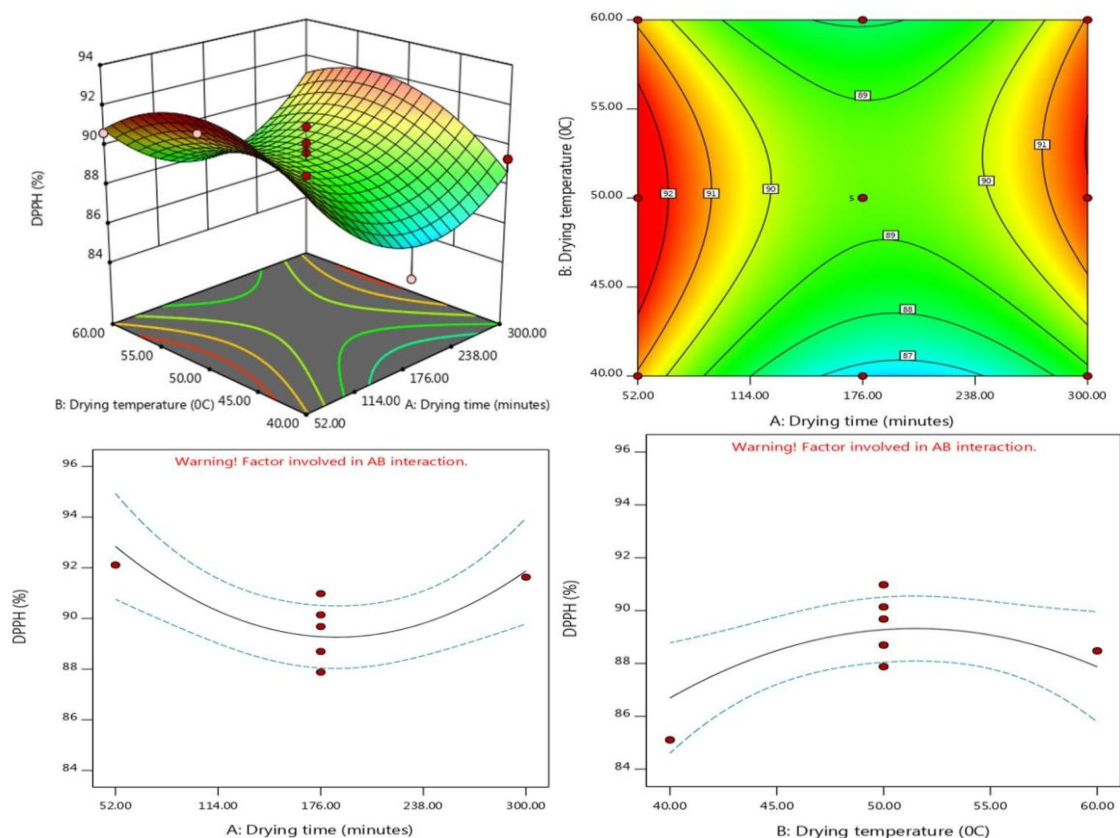


Fig 3 3D surface, contour plot and one factor effect of the simultaneous effect of different drying temperature and drying time on DPPH radical scavenging activity of infrared dried mint leaves

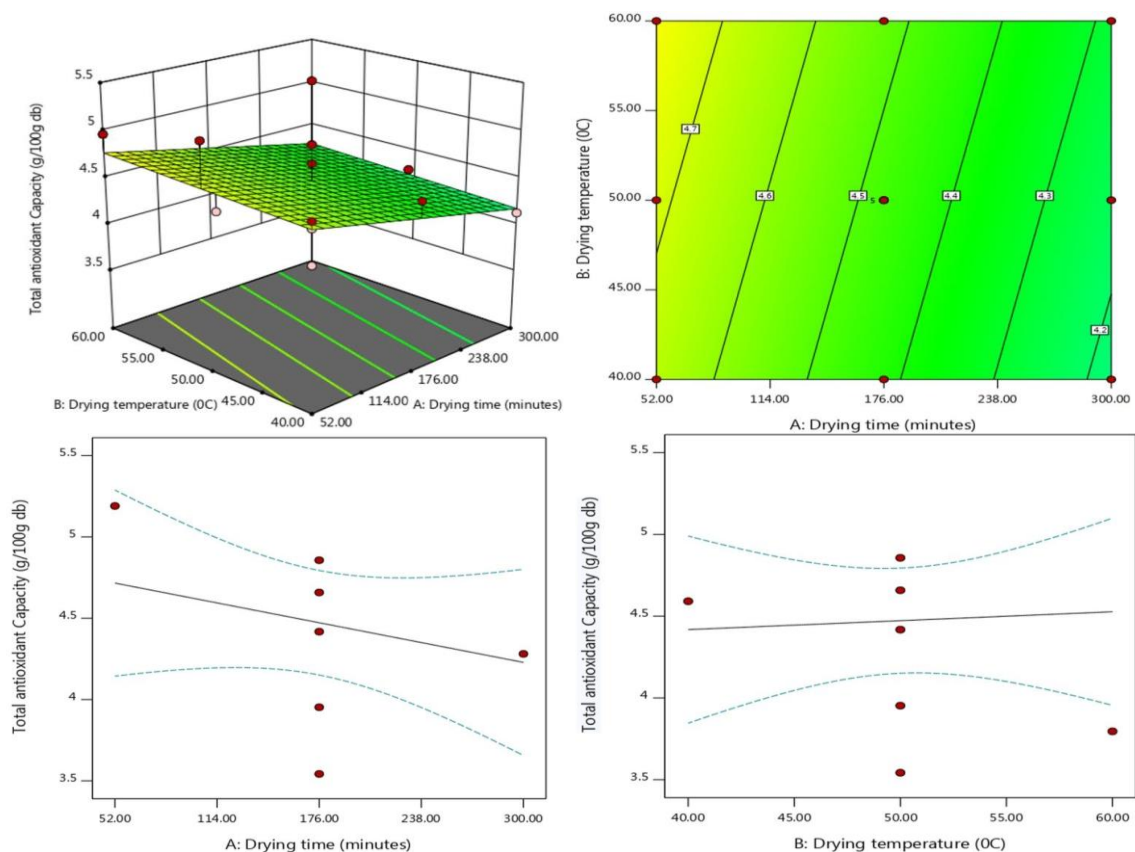


Fig 4 3D surface, contour plot and one factor effect of the simultaneous effect of different drying temperature and drying time on total antioxidant capacity of infrared dried mint leaves

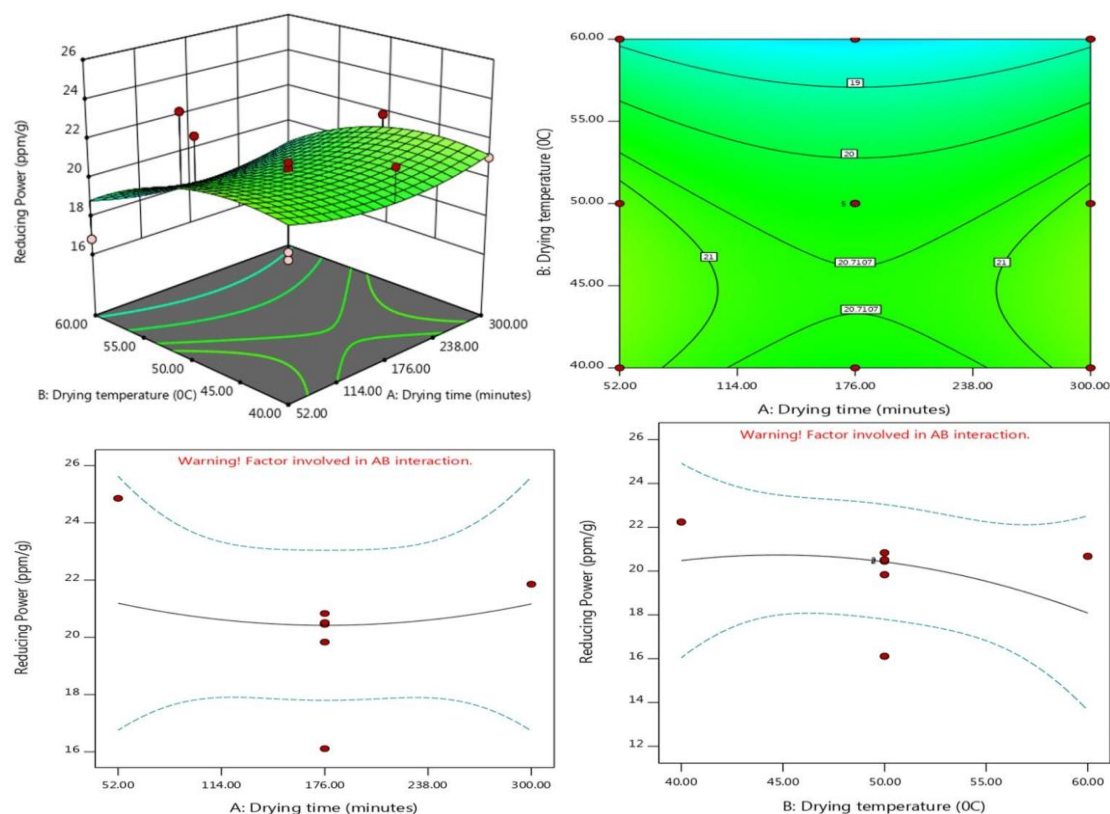


Fig 5 3D surface, contour plot and one factor effect of the simultaneous effect of different drying temperature and drying time on Ferric reducing ability of plasma (FRAP) of infrared dried mint leaves

Antioxidant properties of fruits and vegetables due to phenolic acids (gallic acid, caffeic acid, caffeic acid, ferulic acid, chlorogenic acid and p-coumaric acid) and flavanols (epicatechin, epicatechin gallate, epigallocatechin gallate and catechin) and compounds such as vitamin C. It is related in the composition of fruits and vegetables. The decrease in antioxidant activity is largely related to the decrease in the concentration of polyphenols, as well as the phenomena of oxidative degradation and polymerization-condensation of some compounds [27-28]. In addition, the results of the present study are consistent with the studies of other researchers, which show that the amount of phenolic compounds and antioxidant activity are affected by processes such as cooking with steam, boiling or drying [29-31]. Many reactions that occur during drying lead to the change of compounds responsible for color and affect the phenolic and antioxidant compounds of the product. Among these reactions, we can mention the degradation of pigments, oxidation of ascorbic acid, enzymatic browning, non-enzymatic browning (Millard) and polymerization of phenols. Other factors such as acidity, variety or type of product, temperature and time of the thermal process are

also effective on the existing color and phenolic compounds that are responsible for the antioxidant properties in the product [32].

During the drying process, the product usually becomes darker, and this color change is due to the degradation and decomposition of the pigment and enzymatic and non-enzymatic browning reactions [33]. By using fast drying methods such as infrared drying, due to the short drying time, the destructive effect of heat on food grains is less and the color and antioxidant properties of the product are preserved, and the results of this research confirm the positive effect of infrared drying. Red was based on the antioxidant properties of the product.

3-4- Optimizing the drying conditions of mint leaves

Table (2) shows the experimental plan with the actual levels of the independent variables of infrared drying time and temperature and the results related to the changes in the quality characteristics of mint leaves under the influence of the independent variables using the response surface method. Figure (6) shows the conditions determined for the independent variables (to optimize the effect of temperature and time of the infrared drying process on the

weight loss and antioxidant properties of mint leaves) and the optimized conditions. Considering the destructive effect of long times on the destruction of nutritious and bioactive compounds in food, to determine the range of independent variables of infrared drying temperature and time, the idea of the HTST process was used for this purpose, the maximum drying temperature and the minimum drying time in It was considered, while the amount of weight loss (moisture loss) was considered within the scope of the experiments and the antioxidant properties of total phenolic compounds were considered as the goal of the maximum process [21]. In the optimization process, all independent parameters were given the same weight and importance. According to the desired conditions, the predicted solutions based on the highest desirability are presented in Figure (6) and the closer the desirability is to 1, the most appropriate and best conditions will be, and the first solution is considered as the best conditions to achieve the optimal conditions. was taken and by applying the process conditions obtained in the optimization, a product with the appropriate amount of moisture and the maximum amount of phenolic compounds and antioxidant properties will be

obtained. As can be seen in Figure (6), the best conditions for drying mint leaves include the use of a temperature of 58.56 degrees Celsius and a drying time of 52 minutes, and by applying the optimal conditions, the amount of weight loss (moisture loss), total phenolic compounds, and the antioxidant characteristics include the percentage of DPPH inhibition, total antioxidant capacity and FRAP of the produced dried mint equal to 76.62%, 8.722 mg/g dry weight, 91.97%, 4.75 and 19.91%, respectively, which shows the desirability This optimal condition is 0.727.

In order to validate the optimal conditions resulting from the response surface method, the mint leaf samples were dried under optimized infrared drying temperature and time conditions, and the quality characteristics of the produced product such as weight loss, the amount of total phenolic compounds and antioxidant characteristics including inhibition percentage DPPH, total antioxidant capacity and FRAP of the produced samples were equal to 76.18, 8.545 mg/g dry weight, 92.10%, 8.55 and 20.04% respectively, which are very similar to the results related to the optimal conditions. It is similar to the response surface method.

Table 2 Central composite design, Actual levels of independent variables and different quality attributes (response) of infrared dried mint leaves

Independent variables			Actual dependent variable (Response)				
Treatment number	Drying Temperature (X ₁) (°C)	Drying Time (X ₂) (minute)	water loss (%)	Total phenolic compounds (mg/g)	DPPH (%)	Total antioxidant Capacity (g/100g)	FRAP (ppm/g)
1	50	300	82.23	8.46	91.63	4.28	21.86
2	50	52	60.33	0.76	92.11	5.19	24.86
3	50	176	84.58	14.74	90.98	3.95	20.83
4	60	176	87.30	14.35	88.48	3.80	20.67
5	60	300	64.84	3.82	90.65	5.02	18.28
6	50	176	85.40	12.44	89.68	4.42	20.45
7	40	176	84.36	8.85	85.11	4.59	22.24
8	60	52	84.49	12.15	90.65	4.97	16.81
9	50	176	84.76	11.14	87.88	4.66	19.83
10	40	52	84.54	10.71	91.78	4.74	19.63
11	40	300	84.66	6.88	89.35	4.13	21.09
12	50	176	86.99	13.97	88.70	3.54	20.51
13	50	176	75.47	10.24	90.14	4.86	16.11

4- General conclusion

The results of this research showed that with increasing drying time and temperature, the amount of weight loss (moisture loss) and phenolic compounds of the whole mint leaf

increased and decreased, respectively, and the time **dry**

to do More influence on. The changes of these two parameters were compared to the drying temperature.

The trend of changes in total phenolic compounds of the samples with drying time

was similar to the weight loss of the samples, but with increasing drying temperature, the amount of total phenolic compounds of the samples increased. The DPPH radical scavenging ability of the samples decreased with increasing drying time (up to 238 minutes) and then increased with increasing drying time. The results showed that applying drying temperatures up to 55°C increased DPPH

inhibition, and applying higher drying temperatures led to a decrease in DPPH radical inhibition ability of the samples. Increasing the drying temperature increased the antioxidant capacity and reduced the reductive activity of the samples. Also, increasing the infrared drying time led to a decrease in the antioxidant capacity of the samples.

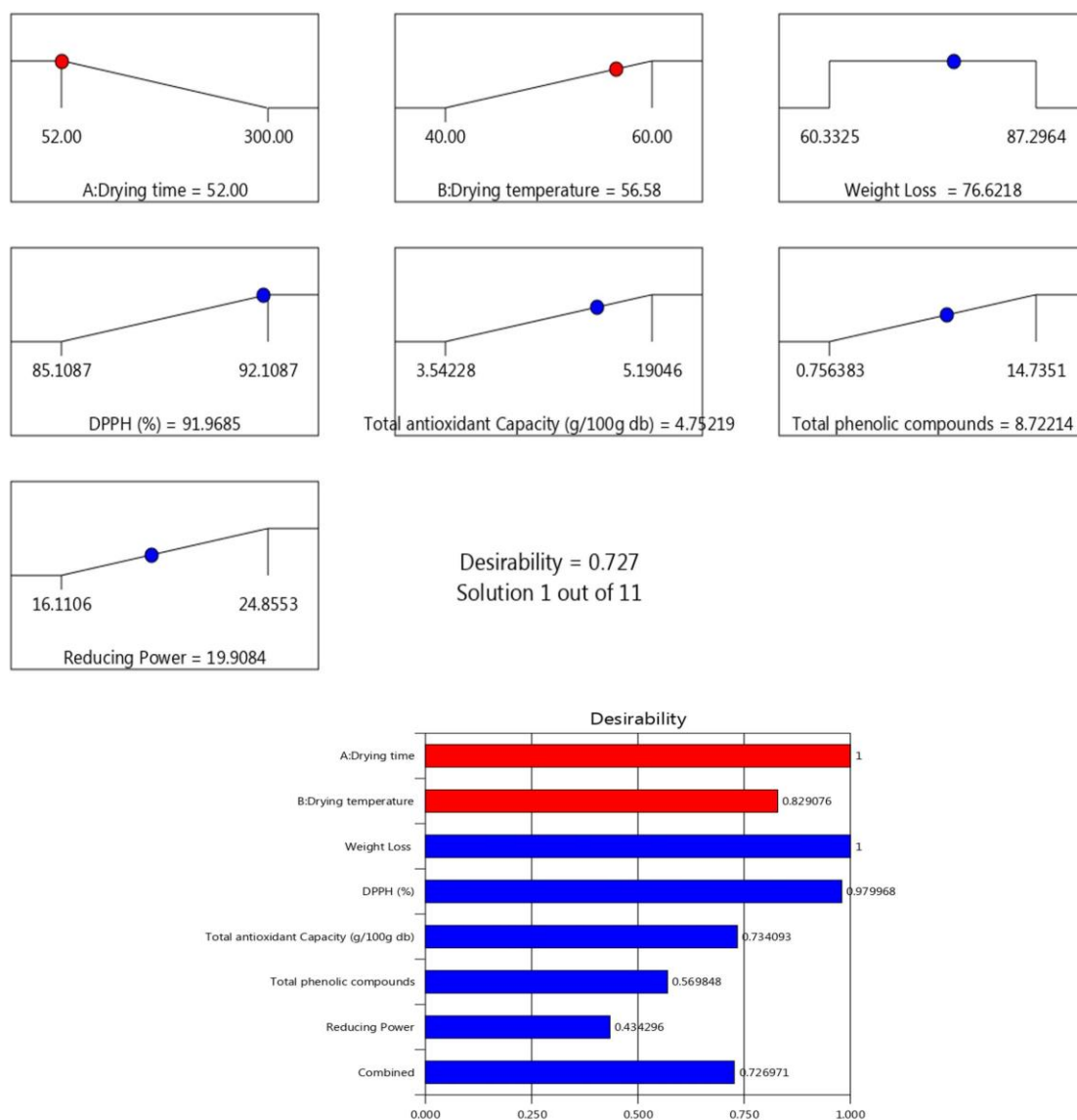


Fig 6 The optimum conditions and desirability graph for infrared drying process of mint leaves

The best conditions for drying mint leaves include the use of a temperature of 58.56 degrees Celsius and a drying time of 52 minutes, and by applying the optimal conditions, the amount of weight loss (moisture loss), total phenolic compounds, and DPPH inhibition percentage of dry mint produced to The order is equal to 76.62%, 8.722 mg/gram of dry weight, 91.97%, and the desirability of these optimal conditions is 0.727. The results of

the validation of the optimal conditions showed that by applying the conditions obtained from the optimization, the quality characteristics of the produced product such as weight loss, the amount of total phenolic compounds and antioxidant characteristics including the percentage of DPPH inhibition, total antioxidant capacity and FRAP of the produced samples were equal to 18 respectively. 76.8, 545.8 mg/g of dry weight, 92.10%, 8.55 and

20.04 were obtained, which are very similar to the results related to the optimal conditions predicted by the response surface method.

5- Resources

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بهینه سازی تغییرات افت وزن، ترکیبات فنولی و ویژگی های آنتی اکسیدانی برگ نعنای طی فرآیند

خشک کردن فرو سرخ با روش سطح پاسخ

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چکیده

اطلاعات مقاله

تاریخ های مقاله :

تاریخ دریافت: ۱۴۰۱/۰۹/۲۲

تاریخ پذیرش: ۱۴۰۱/۱۱/۰۸

کلمات کلیدی:

خشک کردن فرو سرخ،

برگ نعنای،

ترکیبات فنولی،

ویژگی های آنتی اکسیدانی،

روش سطح پاسخ.

در این تحقیق تأثیر دما و زمان خشک کردن فرو سرخ بر میزان افت وزن، ترکیبات فنولی کل و ویژگی های آنتی اکسیدانی برگ نعنای بررسی و با روش سطح پاسخ بهینه سازی شد. نتایج نشان داد که با افزایش دمای خشک کردن میزان افت وزن نمونه های نعنای کاهش یافت اما با افزایش زمان خشک کردن تا ۱۷۶ دقیقه میزان افت وزن نمونه ها افزایش و سپس کاهش یافت. زمان خشک کردن تأثیر بیشتری بر تغییرات افت وزن نمونه ها در مقایسه با دمای خشک کردن داشت. روند تغییرات ترکیبات فنولی کل نمونه ها با زمان خشک کردن مشابه افت وزن نمونه ها بود اما با افزایش دمای خشک کردن میزان ترکیبات فنولی کل نمونه ها افزایش یافت. قابلیت مهار رادیکال فعال DPPH نمونه ها با افزایش زمان خشک کردن (تا ۲۳۸ دقیقه) کاهش و سپس با افزایش زمان خشک کردن افزایش یافت. نتایج نشان داد که اعمال دماهای خشک کردن تا ۵۵ درجه سانتی گراد باعث افزایش قابلیت مهار رادیکال DPPH نمونه ها شد ولی اعمال دماهای خشک کردن بالاتر منجر به کاهش قابلیت مهار رادیکال DPPH نمونه ها شد. افزایش دمای خشک کردن باعث افزایش ظرفیت آنتی اکسیدانی کل و کاهش فعالیت احیاء کنندگی آهن نمونه ها شد. همچنین افزایش زمان خشک کردن فرو سرخ منجر به کاهش ظرفیت آنتی اکسیدانی کل نمونه ها شد. بهترین شرایط برای خشک کردن فرو سرخ برگ های نعنای شامل استفاده از دمای ۵۷/۵۸ درجه سانتی گراد و زمان خشک کردن ۵۲ دقیقه می باشد و با اعمال شرایط بهینه میزان افت وزن (اتلاف رطوبت)، ترکیبات فنولی کل، و درصد مهار DPPH نعنای خشک تولیدی به ترتیب برابر ۷۶/۶۲٪، ۸/۷۲۲ میلی گرم بر گرم وزن خشک، ۹۱/۹۷٪ می باشد که مطلوبیت این شرایط بهینه ۰/۷۲۷ بود.

DOI: 10.22034/FSCT.19.133.141

DOR: 20.1001.1.20088787.1401.19.133.12.7

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