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VIBRO-FLUIDIZED BED HEAT PUMP DRYING OF MINT LEAVES WITH RESPECT TO PHENOLIC CONTENT, ANTIOXIDANT ACTIVITY AND COLOR INDICES

Article Highlights

- We study vibro-fluidized bed drying assisted heat pump of mint leaves
- Temperature caused a remarkable loss of green color due to chlorophyll degradation
- The values of energy activation were within the reported range for food materials

Abstract

Due to high porosity and stickiness, good fluidization of mint leaves can be difficult to achieve. In this study, a vibro-fluidized bed dryer assisted heat pump system was designed and fabricated to overcome this problem. The drying experiments were carried out at temperatures of 40, 50 and 60 °C. The quality of the dehydrated samples was assessed based on color indices, antioxidant activity, and total phenolic content. Drying process primarily occurred in falling rate period. The effective coefficient of moisture transfer of the samples was increased with air temperature and varied from 4.26656×10^{-11} *to* 2.95872×10^{-10} $m^2\ s^{-1}$ *for heat pump drying (HPD) method, and* 3.71918×10^{-11} *to* 1.29196×10^{-10} $m^2\ s^{-1}$ *for none-heat pump drying (NHPD) method. The color indices for temperatures of 40 and 50 °C were very close to each other, whereas by increasing temperature to 60 °C, a remarkable loss of green color was observed. The highest phenolic content was found in methanolic extract for HPD at 60 °C, and NHPD at 50 °C contained the lowest amount of phenolic compounds. NHPD treatments showed lower antioxidant activity compared to HPD treatments at the same temperature due to the longer drying times.*

Keywords: drying kinetics, fluidized bed drying, moisture diffusivity, pharmaceutical plants.

Mint (*Mentha spicata*) is one of the most important spices throughout the world and a perennial plant belonging to Lamiaceae family. Its leaves are used for flavoring, tea infusions and spicing. The use of mint leaves in a variety of dishes such as vegetable curries, chutney, fruit salads, vegetable salads, salad dressings, soups, desserts, juices, and sherbets has been reported [1,2].

Processing method influences the volatile oils of medicinal plants considerably. Drying, as one of the oldest methods of food preservation, represents a very important aspect of food processing. The main purpose of drying is to extend product shelf life, minimize packaging requirements and reduce shipping weights [3]. Phenolic content, antioxidant activity, and color indices are the main characteristics which must be taken into consideration in terms of drying medicinal and spice plants. Hossain *et al.* [4] assessed the effect of different drying methods on phenolic compounds and antioxidant capacity of six Lamiaceae herbs. Among the drying methods tested, air-drying was found to be the best for all samples. Siriamornpun *et al.* [5] evaluated the effect of some drying treatments on color, antioxidant activities and carotenoids of marigold flower. However, there is no report regarding the effect of drying on total phenolic and antioxidant activity of mint leaves.

Among drying methods, fluidized bed drying (FBD) has the advantage of high drying rate due to

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high rates of heat and mass transfer, and consequently high thermal efficiency with uniform and closely controllable temperature in the bed [6]. In the case of FBD, batch mode is preferred for small-scale production and heat sensitive materials. Therefore, fluidized bed technology is widely employed by pharmaceuticals, foods, fertilizers, and many other chemical industries, where either wet granulation or drying solid materials is a fundamental stage of these industries [7].

In order to reduce energy consumption per unit of product moisture, it is important to investigate different methods for enhancing the energy efficiency of the drying operation. Equipping convective hot air dryers with heat pumps has been recognized as an ideal approach for this purpose. A heat pump dryer (HPD) is an economic, environmentally friendly, hygienic drying device used to dry food materials [8]. The HPDs available in the market can allow the energy demand to be reduced by 50% [9]. A HPD can be operated over a wide range of temperatures, providing good conditions for drying heat sensitive materials. The technology requires less energy, as the system can recover the latent heat in a closed loop, and it can be independent of ambient weather conditions. Strommen *et al.* [10] found that HPDs consumed 60-80% less energy compared with conventional dryers operating at the same temperature.

In spite of the vast applications of FBD, it has not been used for drying pharmaceutical plants, especially leaves products such as mint. This could be due to high porosity of these products, and stickiness of the leaves to each other during fluidization. Moreno *et al.* [11] reported vibration as a method for improving the quality of fluidization and avoiding problems such as channeling and defluidization. The aims of this study were investigating drying kinetics of mint leaves using a vibro-fluidized bed dryer (VFBD) assisted heat pump system in batch mode, determining moisture diffusivity under different drying conditions, and analyzing qualitative parameters including color indices, total phenolic content and antioxidant activity of the dehydrated product.

MATERIALS AND METHODS

Sample preparation

Fresh mint leaves were purchased from a local market in Isfahan (central Iran), and stored in a refrigerator at 5 °C before the drying experiments. The samples without any pretreatment were used for conducting tests. The initial moisture content of the mint leaves was determined using AOAC [12] standard method (vacuum drying at 70 °C for 24 h). Drying of the leaves was finished until the moisture content did not change and the weight of samples became constant.

Vibro-fluidized bed heat pump drying

In preliminary tests [13], due to high porosity and stickiness, the mint leaves could not be fluidized and stacked to each other during fluidization. Therefore, a laboratory vibro-fluidized bed dryer (VFBD) assisted heat pump system (Figure 1) was designed and fabricated at Isfahan University of Technology (IUT) to conduct the experimental part of this study. A 1.5 kW blower (Motogen 90L2A; Motogen Co. Ltd., Tabriz, Iran), 2830 rpm, was coupled to an inverter (Teco 7300 CV, with ±0.01 Hz accuracy; TECO Electric & Machinery Co. Ltd., Taipei, Taiwan) and used for supplying and controlling the airflow rate. The drying air was heated with a 10 kW electric heater. The exit temperature of air from the heater was maintained constant within ±0.5 °C using a PI control system.

The air velocity was measured using an air velocity transmitter (AVT; HK instruments Co. Ltd., Muurame, Finland) placed downstream from the heater. The distributor plate was constructed from a circular Plexiglas plate with the thickness of 5 mm, along with 1630 holes of 2 mm diameter.

The cylindrical bed column was made of Plexiglas with a wall thickness of 5 mm, an internal diameter of 140 mm, and the length of 1000 mm. Three sensors (SHT75; Sensirion AG, Staefa, Switzerland) were used to measure the relative humidity and temperature at the inlet of the bed as well as locations at 10 and 300 mm above the distributor plate (Figure 1). Three other sensors were located before and after the evaporator, and after the condenser of the heat pump system. A digital balance (Kern 572-57, with ±0.1 g accuracy; Kern & Sohn GmbH, Balingen, Germany) was used to measure the weight of samples during the experimental runs. All the signals from the SHT75 sensors, air velocity sensor, digital balance, heater and energy module were acquired simultaneously every 15 s using the data acquisition board and the LabView software and stored in a desktop computer for subsequent analysis. To evaluate the influence of air temperature on drying curves, the experiments were carried out at 40, 50 and 60 °C. The mint samples were dried under heat pump drying (HPD), and non-heat pump drying (NHPD) treatments.

In order to apply vibration to the drying chamber, a single-phase motor (Motogen CRS 90L2A; Motogen Co. Ltd., Tabriz, Iran), and a 3 mm eccentric mechanism with 80 Hz frequency of vibration were used.

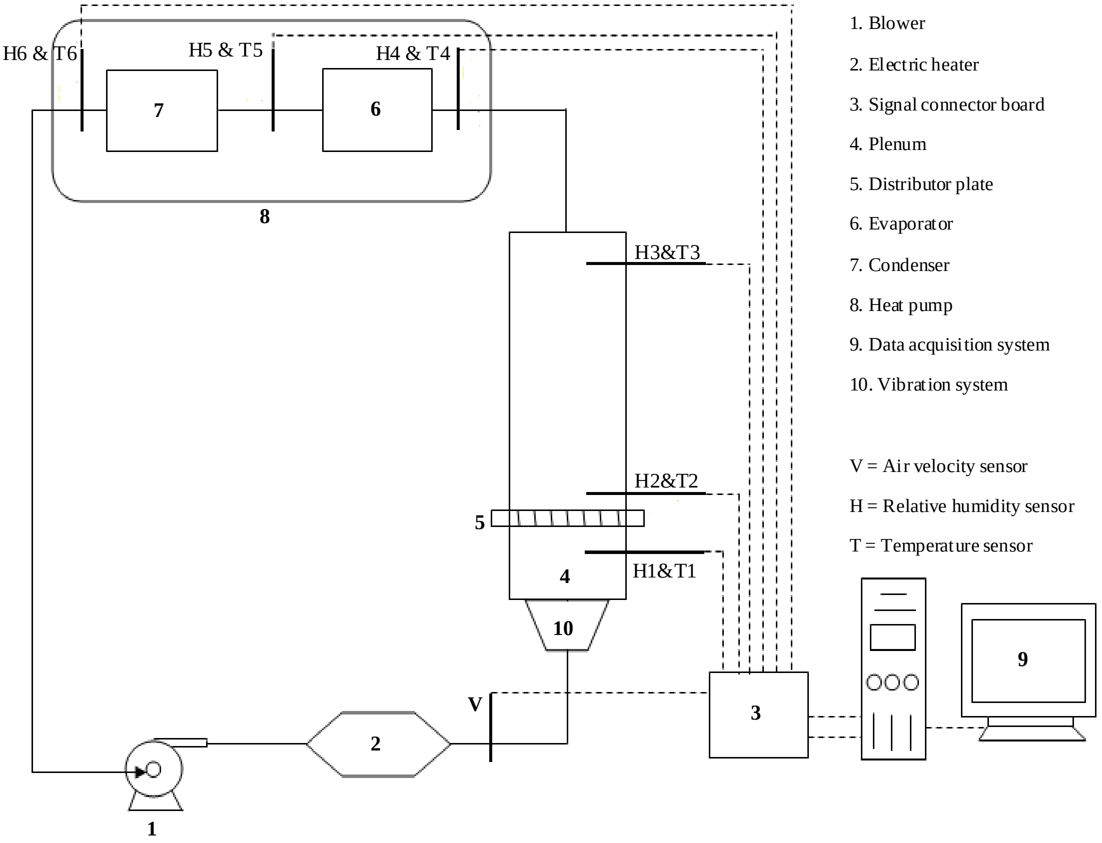

Figure 1. Schematic diagram of vibro-fluidized bed heat pump drying system.

Table 1 presents the information regarding the conditions of the experiments.

Table 1. Some physical characteristics of mint leaves and the conditions of the experiments

Parameter	Value
Frequency of vibration, Hz	80
Amplitude of vibration, mm	3
Initial solid (leaves) temperature, °C	22
Inlet air humidity, %	9
Air velocity, m s^{-1}	2
Bed height, mm	150
Bed porosity, %	92
Thickness of leaves, mm	0.187±0.01
Leaves size, mm	20×40±3
Particle density, kg m^{-3}	903

Before each experiment, the unit was run without any sample for about 30 min to reach the thermal steady state.

Drying kinetics and moisture diffusivity

The moisture ratio (*MR*) and drying rate (*DR*) of mint leaves during drying experiments were calculated using Eqs. (1) and (2), respectively [14,15]:

$$MR = \frac{M - M_e}{M_0 - M_e} \tag{1}$$

$$DR = \frac{1}{A}\frac{\mathrm{d}M}{\mathrm{d}t} = \frac{1}{A}\frac{M_{t+\mathrm{d}t} - M_t}{\mathrm{d}t} \tag{2}$$

where M, M_e, M_0, M_t and $M_{t+\mathrm{d}t}$ are the moisture content at any time, the equilibrium moisture content, the initial moisture content, the moisture content at t and the moisture content at $t+\mathrm{d}t$, respectively, t is the drying time (min), and A is the drying area (m^2).

Fick's second diffusion equation was used to determine the effective coefficient of moisture transfer of the samples. The mint leaves were considered the slab geometry [16]. The equation is expressed as [17]:

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)^2}\exp\left(-\frac{(2n-1)^2\pi^2 D_{eff}t}{4l_0^2}\right) \tag{3}$$

where D_{eff} is the effective coefficient of moisture transfer (m^2 s^{-1}), l_0 is the half thickness of the slab (m) and n is the positive integer. For long drying times, only the first term of Eq. (3) can be used [18]:

$$MR = \frac{8}{\pi^2}\exp\left(-\frac{\pi^2 D_{eff}t}{4l_0^2}\right) \tag{4}$$

$$\ln(MR) = \ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2 D_{eff} t}{4 l_0^2}\right) \tag{5}$$

The slope (K_0) is calculated by plotting ln (MR) versus t according to Eq. (5) to calculate the effective coefficient of moisture transfer for different temperatures:

$$K_0 = \frac{\pi^2 D_{eff}}{4 l_0^2} \tag{6}$$

Activation energy

Temperature and effective coefficient of moisture transfer can be related using Arrhenius equation [18] as follows:

$$D_{eff} = D_0 \exp\left(-\frac{E_a}{R(T+273.15)}\right) \tag{7}$$

where D_0 is the constant in Arrhenius equation (m^2 s^{-1}), E_a is the activation energy (kJ mol^{-1}), T is the temperature of air (°C), and R is the universal gas constant (8.3145 J mol^{-1} K^{-1}). Equation (7) can be written in the form of:

$$\ln D_{eff} = \ln D_0 - \left(\frac{E_a}{R(T+273.15)}\right) \tag{8}$$

The slope (K_E) is calculated by plotting ln D_{eff} *versus* T^1 according to Eq. (8) to determine the activation energy as follows:

$$K_E = \frac{E_a}{R} \tag{9}$$

Color measurements

The color of the mint samples was evaluated before and after drying according to CIE (Commission International de l'Eclairage) [19]. The color values were measured using a spectrophotometer Text Flash (Datacolor Corp., Switzerland). The lightness (L^*), redness (a^*), and yellowness (b^*) were captured for each treatment. The total color difference (ΔE) was calculated using Eq. (10). This index is a single value that takes into account the differences between the L^*, a^* and b^* of the sample and standard. Chroma or strength of color (C^*), and hue angle (h^*), which are related to a^* and b^*, were also calculated by Eqs. (11) and (12) [19]:

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

$$C^* = \sqrt{\left(a^*\right)^2 + \left(b^*\right)^2} \tag{11}$$

$$h^* = \tan^{-1}\left(\frac{b^*}{a^*}\right) \tag{12}$$

where L_0*, a_0* and b_0* are the values for fresh mint leaves.

Total phenolic extraction

For phenolic extraction, 6 g of powders was extracted with 200 ml of 80% methanol. The extraction was carried out using an orbital shaker (150 rpm) at 25 °C for 24 h. The extracts were filtered through four layers of cheesecloth to remove the solid debris. The extraction was done four times. Total phenolics were determined colorimetrically using Folin-Ciocalteu reagent as described by Pinelo *et al.* [20]. In this regard, ten-fold diluted reagent (2.5 ml), 7.5% sodium carbonate (2 ml) and methanolic extract (0.5 ml) were mixed. Then, after heating at 45 °C for 15 min, the absorbance was measured at 765 nm against a blank. The phenolic content was expressed as tannic acid equivalent per gram dry weight of sample.

Antioxidant activity measurements

Free radical scavenging activity of the mint leaves extracts and standard antioxidant was assessed using the 1,1-diphenyl-2-picrylhydrazyl (DPPH) method [21]. Different dilutions of the mint leaves extracts (equivalent to 50, 100, 300 and 500 ppm) were prepared in methanol. Butylated hydroxytoluene (BHT) was used as standard antioxidant in 1-100 μg ml^{-1} solution. Five milliliters of a 0.1 mM methanolic solution of DPPH was mixed with 0.1 ml of sample and standard solutions separately. Radical scavenging of the extracts was calculated by employing Eq. (13) and using methanol (80%) and DPPH solution (0.1 mM, 5 ml) as a blank and control sample, respectively:

$$\%\ \text{Radial scavenging activity} = \left(OD\ \text{control} - \frac{OD\ \text{sample}}{OD\ \text{control}}\right) \times 100 \tag{13}$$

RESULTS AND DISCUSSION

Drying kinetics

The results of variation in moisture ratio versus drying time, and drying rate versus moisture content, as obtained for HPD and NHPD treatments carried out at 40, 50 and 60 °C, are shown in Figures 2 and 3, respectively. As expected, with increasing drying temperature, the drying time was decreased. The constant rate drying period was not detected in drying curves. In other words, drying of mint leaves occurred primarily in falling rate period, indicating that initial mass transfer occurred by diffusion. The same results have been reported for drying mint leaves [16,22].

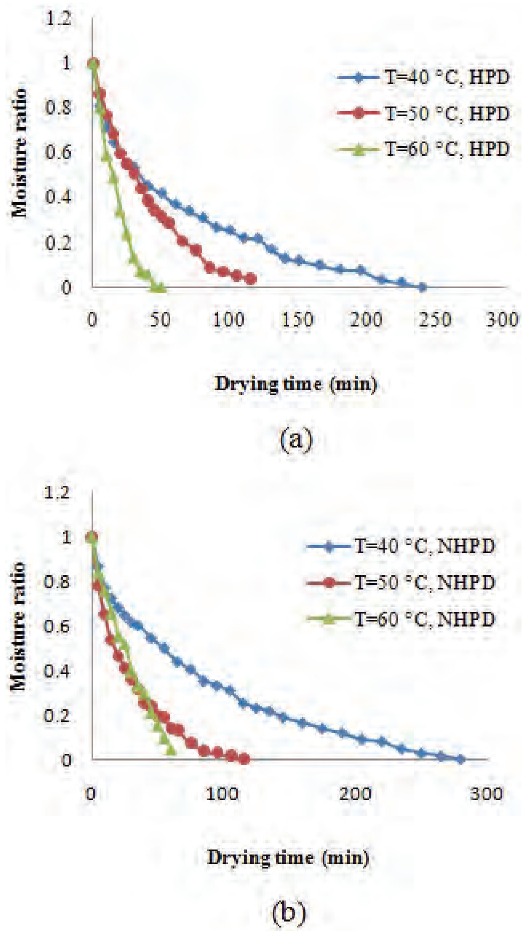

Figure 2. Drying curve for mint leaves dehydrated at different air-drying temperatures: a) heat pump drying (HPD) method and b) non-heat pump drying (NHPD) method.

Moisture diffusivity and activation energy

Values of effective coefficient of moisture transfer (D_{eff}) for mint leaves dehydrated under HPD and NHPD treatments at various temperatures are shown in Table 2. The values of D_{eff} were increased with increasing air-drying temperature. Effective coefficient of moisture transfer values were varied from 4.25656×10^{-11} to 2.95872×10^{-10} m^2 s^{-1} for HPD, and from 3.71918×10^{-11} to 1.29196×10^{-10} m^2 s^{-1} for NHPD at temperatures ranging from 45 to 60 °C. It is observed that the difference between the values for HPD and NHPD methods was not remarkable. This is due to the fact that the effective coefficient of moisture transfer is an internal parameter. Similar variations were also observed during drying of mint [22]. Generally, effective coefficient of moisture transfer values for food materials are in the range of 10^{-9} to 10^{-11} m^2 s^{-1} [23].

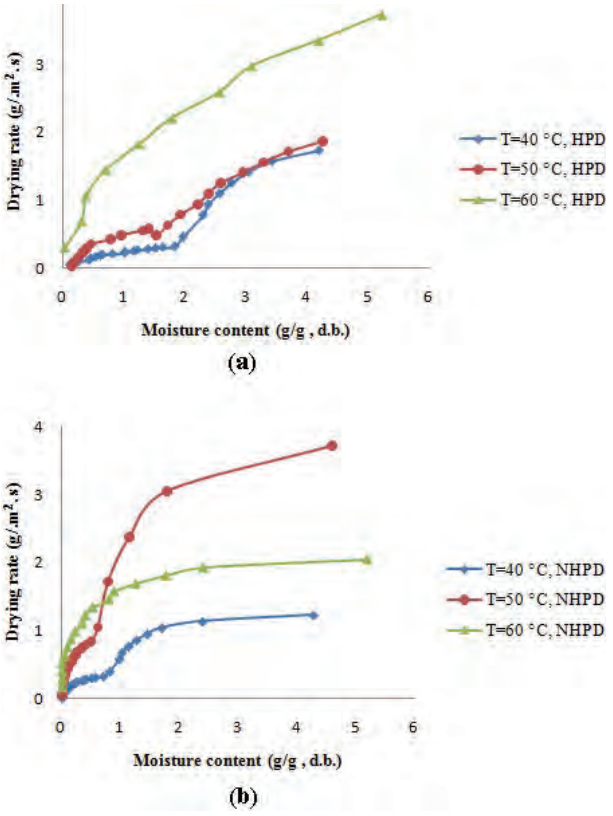

Figure 3. Drying rate curve for mint leaves dehydrated at different air-drying temperatures: a) heat pump drying (HPD) method and b) non-heat pump drying (NHPD) method.

Table 2. Values of effective coefficient of moisture transfer (m^2 s^{-1}) of mint leaves dehydrated at different air-drying temperatures and two drying methods

Temperature, °C	Drying method	
	HPD	NHPD
40	4.25656×10^{-11}	3.71918×10^{-11}
50	1.04859×10^{-10}	1.02623×10^{-10}
60	2.95872×10^{-10}	1.29196×10^{-10}

Values of ln D_{eff} *versus* T^{-1} for experiments information are plotted in Figure 4. The activation energy was determined to be 84 kJ mol^{-1} for HPD and 54.34 kJ mol^{-1} for NHPD. Park *et al.* [1] and Doymaz [16] reported the values of 82.93 and 62.96 kJ mol^{-1} for drying mint leaves, respectively.

Color, total phenolic content and antioxidant activity

The L^*, a^*, b^*, c^* and h^* values of the samples dried using HPD and NHPD methods at different temperatures are presented in Table 3. Low negative a^* value (-7.48) confirms greenness and consequently, a high (116.34° > 90°) hue angle, thereby indicating more greenness for fresh mint leaves. L^* value was also approximately high (47.71) for fresh sample. L^* value is a measure of the color in the light-

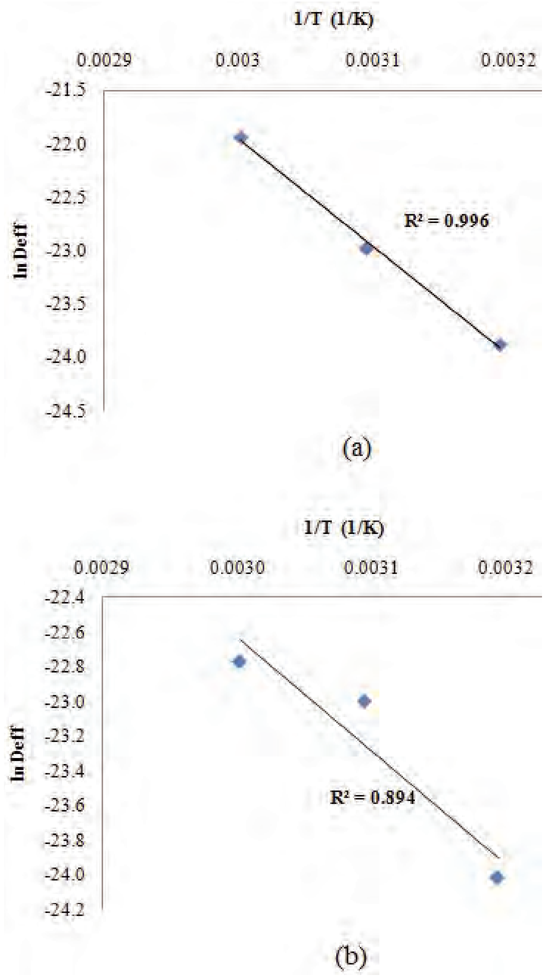

Figure 4. The relationship between air temperature and effective coefficient of moisture transfer for mint leaves dehydrated: a) heat pump drying (HPD) method and b) non-heat pump drying (NHPD) method.

-dark axis. Reduction in this parameter indicates that the samples were turning darker. As observed, there is an improvement in color indices with increasing drying time (using the temperature of 40 °C compared with 60 °C, Table 3). However, the color indices for temperatures of 40 and 50 °C were very close to each other, whereas by increasing temperature to 60 °C, a remarkable loss of green color was observed. This might be caused by chlorophyll degradation in leaf cells [24]. Previous reports have shown that degradation of chlorophyll occurred at temperatures exceeding 50 °C in thyme and 60 °C in broccoli juice [25].

The total color difference (Δ*E*), which is a combination of Hunter *L** *a** *b** values, is a colorimetric parameter extensively used to characterize the variation of colors during processing of foods [23]. Less values of Δ*E* indicate minimum differences between *L**, *a** and *b** of the samples and fresh ones. The samples dehydrated at 40 °C had minimum Δ*E*, and the samples at 60 °C presented the maximum value. The chroma value indicates the degree of saturation of color and is proportional to the strength of the color. Large changes were found in chroma between fresh and dried mint leaves at 60 °C (Table 3). This reveals lack of stability of green color in mint leaves for this treatment.

For all color indices, a remarkable difference was not observed between HPD and NHPD methods at a constant temperature. Drying of agricultural and food products depends on the temperature and humidity of the drying air. One of the functions of the HPD method is to isolate the humid environment from the drying process, while, drying by NHPD (open--type) method may prolong the drying duration because of humid air entering into the drying chamber in high humid areas. The present study was conducted under low humidity conditions. Therefore, in such arid and semi-arid areas, the use of heat pump system could not establish a meaningful difference in drying times. This is the reason for having the same color parameters under different methods.

Total phenolic content was varied from 30.46 to 67.32 mg tannic acid per 1 g dry weight of the samples. The highest phenolic content was found in methanolic extract of 60 °C for HPD, whereas air temperature of 50 °C for NHPD contained the lowest amount of phenolic compounds (Figure 5). There are similar reports regarding the effect of different drying methods on total phenolic content of plants. Asami *et al.* [26] assessed the effect of three postharvest treat-

Table 3. Values of L, a*, b*, c*, h* and ΔE for mint leaves dehydrated at different air-drying temperatures and two drying methods*

Treatment	*L**	*a**	*b**	*c**	*h**	Δ*E*
HPD, T=40 °C	38.05	-3.37	15.68	16.04	102.14	10.51
NHPD, T=40 °C	39.34	-2.76	16.38	16.61	99.56	9.69
HPD, T=50 °C	39.15	-2.87	16.72	16.96	99.73	9.85
NHPD, T=50 °C	38.66	-3.12	16.50	16.80	100.72	10.14
HPD, T=60 °C	35.79	0.43	13.71	13.71	88.22	14.37
NHPD, T=60 °C	34.83	-0.06	14.20	14.20	90.23	14.89
Fresh mint	47.71	-7.48	15.11	16.86	116.34	0.00

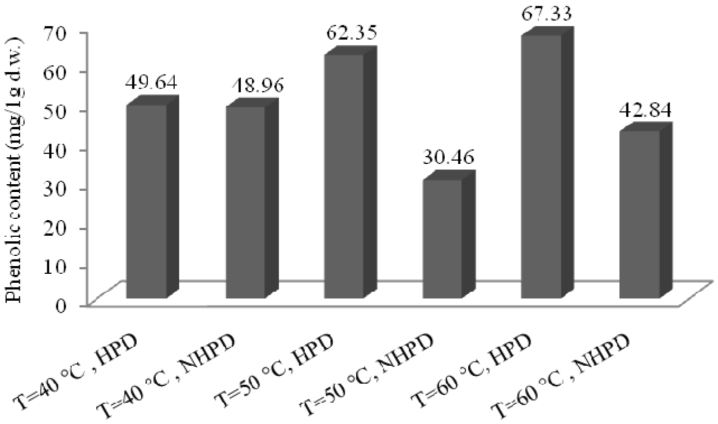

Figure 5. The total phenolic content values for mint leaves dehydrated at different air-drying temperatures and two drying methods.

ments (freezing, freeze-drying, and air-drying) on total phenolic content of some fruits.

Sejali and Anuar [27] evaluated the effect of three drying treatments (shade, oven-dried at 45 and 70 °C) on total phenolic contents of Neem (*Azadirachta indica*) leaf powder. Similar to the present study, lower temperatures and fine particle size led to higher phenolic content. It was also observed that higher phenolic content in dried oregano could be reached by the reduction of temperature [28].

The antioxidant activity values of samples were also measured at different treatments (Figure 6). High values of IC50 indicate less antioxidant activity. As depicted, antioxidant activity was increased in higher temperatures. All the NHPD treatments showed lower antioxidant activity compared with HPD treatments at the same temperature due to the longer drying time. Two factors can affect the antioxidant activity and major components of plant species: drying temperature and drying time [24]. As increasing the temperature can lead to degrading the antioxidants, the probable reason for increasing antioxidant activity is the shorter drying time when applying higher temperatures in the methods used in the present study. Madrau *et al.* [29] reported similar results in apricot drying. In their study, higher antioxidant activity was observed when temperature was increased from 50 to 70 °C. As the drying time is reduced with increasing temperature, it is concluded that the antioxidant activity of the samples was affected by drying time rather than drying temperature.

CONCLUSIONS

Drying of mint leaves was performed using a laboratory vibro-fluidized bed assisted heat pump system. Values of effective coefficient of moisture transfer were increased as air-drying temperature was increased. But the values between heat pump drying (HPD) and non-heat pump drying (NHPD) methods did not show any remarkable difference. The values of energy activation were within the reported range for food materials. As a result of chlorophyll degradation in leaf cells, increasing temperature to 60

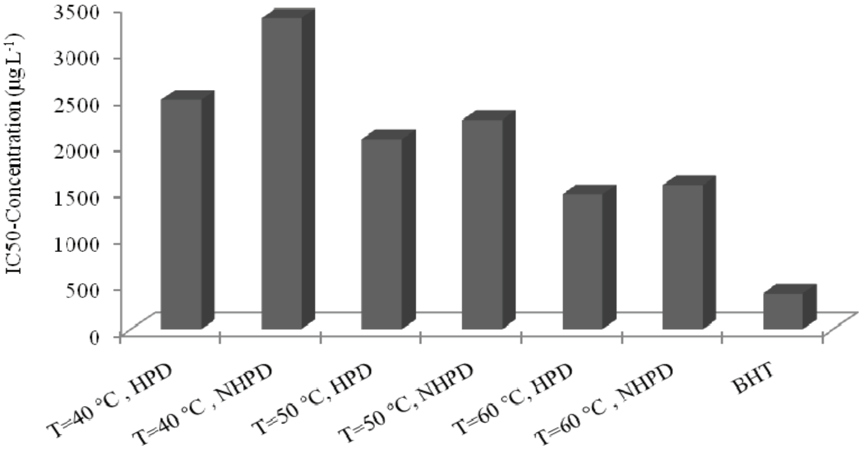

Figure 6. The antioxidant activity values for mint leaves dehydrated at different air-drying temperatures and two drying methods.

°C caused a remarkable loss of green color. However, the values between HPD and NHPD treatments did not show a meaningful difference. It was revealed that the effect of drying time on antioxidant activity of the samples was more than that of temperature. Overall, it was concluded that in arid and semi-arid areas, the use of heat pump system could not establish a considerable difference in drying process.

Acknowledgements

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Nomenclature

a^*	redness
M	moisture content at any time (g water/g dry matter)
b^*	yellowness
M_e	equilibrium moisture content (g water/g dry matter)
C^*	chroma
M_o	initial moisture content (g water/g dry matter)
D_{eff}	effective coefficient of moisture transfer ($m^2 s^{-1}$)
M_t	moisture content at t time (g water/g dry matter)
D_0	constant in Arrhenius equation ($m^2 s^{-1}$)
M_{t+dt}	moisture content at t+dt time (g water/g dry matter)
E_a	activation energy (kJ mol^{-1})
MR	moisture ratio (dimensionless)
HPD	heat pump drying
$NHPD$	non-heat pump drying
h^*	hue angle
n	exponent and positive integer
k_0, k_E	slope
R	gas constant (J mol^{-1} K^{-1})
l_0	slab thickness
T	temperature (°C)
L^*_0, a^*_0, b^*_0	values for fresh mint
t	drying time (min)
DR	drying rate (g m^{-2} s^{-1})
L^*	lightness
A	drying area (m^2)
ΔE	total color difference

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NAUČNI RAD

UTICAJ SUŠENJA MENTE U VIBRO-FLUIDIZOVANOM SLOJU UZ KORIŠĆENJE TOPLOTNE PUMPE NA SADRŽAJ FENOLA, ANTIOKSIDATIVNU AKTIVNOST I INDEKSE BOJE

Zbog visoke poroznost i krutosti, lišće mente nisu mogli biti dobro fluidizovani tokom fluidizacije. U ovom radu je projektovana i izrađena sušara sa vibro-fluidizovanim slojem da bi bio prevaziđen ovaj problem. Eksperimenti sušenja su izvedeni na temperaturama od 40, 50 i 60 °C. Kvalitet dehidrisanih uzoraka je ocenjen na osnovu indeksa boje, antioksidativne aktivnosti i sadržaja ukupnih fenola. Proces sušenja se prvenstveno dešava u period opadajuće brzine. Efektivna koeficijent prenosa vlage iz uzoraka se povećava sa temperaturom vazduha i varira od $4,27\times10^{-11}$ *do* $2,96\times10^{-10}$ m^2 s^{-1} *za sušenje pomoću toplotne pumpe (HPD), a* $3,72\times10^{-11}$ *do* $1,29\times10^{-10}$ m^2 s^{-1} *za sušenje bez toplotne pumpe (NHPD). Indeksi boja za temperature od 40 i 50 °C su veoma blizu jedan drugom, a povećanjem temperature do 60 °C, uočen je izuzetan gubitak zelene boje. Najviši sadržaj fenola nađen u metanolnom ekstraktu za HPD na 60 °C, a ekstrakt NHPD na 50 °C je sadržao najniži sadržaj fenolnih jedinjenja. NHPD tretmani su pokazali manju antioksidativnu aktivnost u odnosu na HPD tretmane na istoj temperaturi usled dužeg vremena sušenja.*

Ključne reči: kinetika sušenja, sušenje u fluidizovanom sloju, difuzivnost vlage, medicinsko bilje.