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Research article

Effect of convective and vacuum drying on some physicochemical and phytochemical characteristics of peppermint leaves

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Abstract: This study examines the effects of convective air and vacuum drying at 40, 50, and 60 °C on the drying behavior, color, pigments, phenolic content, and antioxidant capacity of peppermint leaves. The drying data were modeled using eight drying models, with the Midilli model being the best fit for both drying methods with the highest R^2 (>0.99) and lowest values of χ^2 (<0.003) and root mean square error (RMSE) (<0.035). Results showed that convective drying at 60 °C had the highest drying rate (0.62 d.b./h) compared to vacuum drying (0.25 d.b./h) at the same drying temperature. Effective moisture diffusivity increased with the increase in drying temperature and ranged from 1.00×10^{-13} to 5.16×10^{-13} ($m^2\ s^{-1}$). Activation energy ranged from 39.72 to 41.46 (kJ mol^{-1}). Furthermore, vacuum drying resulted in higher lightness and lower redness (a*) values than convective drying at higher temperatures. Both methods increased chlorophyll a and b contents, while β-carotene and phenolic contents significantly decreased, particularly at higher temperatures. This study highlights that both convective and vacuum drying methods affect the drying behavior and quality of peppermint leaves, with lower temperatures being more effective in preserving color and antioxidant properties. Future studies should focus on optimizing drying conditions to further enhance the retention of key bioactive compounds and explore the potential of other drying techniques for improved peppermint preservation.

Keywords: antioxidants capacity; drying behavior; peppermint leaves; temperature effect; thin-layer models

1. Introduction

Peppermint (*Mentha piperita* L.) is regarded as one of most valuable aromatic plants and has over 30 known substances in its essential oil, including neomenthol, menthone, menthol, and iso-menthone. It also has unique flavor characteristics [1]. In 2022, the world production of peppermint was 51,081 tons, led by Morocco with 84% of the total and Argentina with 14%, followed by Mexico, Bulgaria, and Spain [2]. Peppermint leaves are rich in many bioactive compounds, such as flavonoids, phenols, beta-carotenes, chlorophylls and antioxidants, which give the plant its special importance among medicinal plants. Flavonoids and phenols are useful in medicine and cosmetics; hence, they are present in several cosmetic and skincare products, as well as formulation enhancers [3]. Because of their possible health benefits, phenols and flavonoids are also frequently employed as components in dietary supplements [4]. β-carotene can reduce the risk of age-related macular degeneration, cataract, osteoclast genesis, and coronary heart diseases [5]. Medicinal plants (e.g., peppermint) that content chlorophyll are employed to address a wide array of illnesses [6]. Antioxidants play an important role in the promotion of human health by protecting against the harmful free radicals which are implicated in the pathogenesis of several chronic diseases [7]. Peppermint is the second most important essential oil plant after the Citrus species, approximately 2000 tons of world essential oil is obtained from the Mentha species [8]. Essential mint oil is extracted either from freshly harvested or dried leaves through distillation process for industrial applications. Moreover, dehydrated peppermint is widely consumed in the food industry as a seasoning and in herbal teas.

Drying of food products is aimed at longer storage periods, lower packaging requirements and shipping weights [9]. Reducing water activity by drying primarily aims to stop microbiological growth, inhibit enzyme activity, minimize oxidation, avoid non-enzymatic reactions, and reduce loss of nutrients. This prolongs the product's shelf life while causing the least amount of quality losses [10–12]. Bioactive compounds, which determine the utility value of products, are affected in the process and their losses occur especially when drying is conducted at too high temperatures or when the drying process lasts too long [13]. Herbs, such as peppermint, are particularly susceptible to the drying process, which can harm them or lessen the appeal of the finished product by altering their quality features in addition to protecting its vital components [14]. Therefore, it is necessary to develop efficient preservation techniques to ensure the quality and safety of herbs and medicinal plants.

Vacuum drying is recommended for sensitive and valuable raw materials such as peppermint. This will ensure process efficiency and allow for the potential to lower drying temperature [15]. At low pressure, the boiling point of water can be decreased makes it feasible to dry materials more quickly and at a lower temperature than with traditional drying, producing products of greater quality [16]. Freeze-drying, microwave-vacuum drying, and combination techniques are other techniques, have been used with the vacuum chamber [17]. Because they require less thermal energy, unconventional drying techniques can also be economically feasible [18]. When implementing the commercial drying of medicinal plants, extra caution should be exercised when selecting the drying method and temperature. Simulation models can be used to optimize drying conditions and save energy [19]. Thin-layer drying models are most known and popular because they are simple and easy to apply, unlike more complex theoretical models that require many parameters to be evaluated [9]. Thin-layer drying equations describe drying phenomena in a unified manner regardless of controlling mechanism. On the other hand, theoretical models that describe coupled heat and mass transfer during drying process are very complex and practically need to solve a set of partial differential equations with boundary conditions and material properties as a function of temperature and moisture content.

Due to its importance, several studies have carried out on peppermint with different purposes. Some studies focused only on drying kinetics in a cabinet dryer [20], microwave and hot air drying [21], convective drying [9], infrared [22], solar dryer [23,24], and freeze drying [25]. Other studies focused on the effect of some drying methods on some compounds of peppermint, such as effect of sun and microwave drying on minerals, color and phenolics content [26]; freeze drying on essential oil content [27]; infrared, vacuum, and microwave drying on essential oil content and color [28]; infrared and solar drying on color changes [29]; some with especial purposes, such as desorption and sorption isotherms [30]; and energy analysis of solar drying [31].

The majority of the above studies focused on either drying kinetics or functional properties only. Studies based on drying kinetics can only recommend drying conditions from an energy and time consumption point of view without considering the effect on bioactive compounds. While studies based on quality attributes can only recommend drying conditions that maintain the compounds without considering the energy and time consumption. The balance between these parameters is essential for drying process optimization. This study aims to address this gap in literature and emphasize the importance of determining the drying kinetics with bioactive compounds effect of different drying methods and temperatures.

It is also clear that information on drying kinetics (especially moisture diffusivity) and changes in bioactive compounds after vacuum drying of peppermint is very limited. However, there is limited research available on using thin-layer drying models for peppermint leaves under vacuum drying. Analyzing the effect of drying on these bioactive compounds (flavonoids, phenols, beta-carotenes, chlorophylls, and antioxidants) in peppermint leaves is crucial as these compounds are key indicators of the plant’s nutritional and medicinal value.

Accordingly, and due to the importance of high-quality dried peppermint for the market, this intensive work was conducted to investigate the drying kinetics, physicochemical and phytochemical characteristics of peppermint leaves under convective and vacuum drying process at different drying temperatures. In addition, the drying process of peppermint was modeled with eight mathematical models to appropriately fit the drying curves of peppermint leaves.

2. Materials and methods

2.1. Preparation of peppermint samples

The needed amount of fresh peppermint plant was harvested from the farm and moved immediately to the lab. To prepare the samples for drying, foreign materials, such as spoiled parts, weeds and dirt, were entirely removed from the peppermint plants. The peppermint leaves were gently separated from the plant stem. The thickness of the fresh peppermint leaves used in the drying experiments was estimated by a digital caliper (Mitutoyo, Japan; ±0.01 mm) and found to be 0.16 mm. In order to ascertain the peppermint's initial moisture content (Mi), Samples were dried at 70 °C in a hot air oven until they attained a constant weight [32]. M_i of fresh peppermint was found to be 82% on wet basis (10.25 dry basis). This procedure was repeated before each drying experiment.

2.2. Drying methods

Peppermint samples were dried using two drying methods (convective dryer and vacuum dryer) as presented in Figure 1. The convective dryer (FCO-125D, USA; ±1 °C) consists of a fan at the bottom to uniformly distribute the heated air with an average velocity of 0.6 ± 0.1 m s^{-1}. The vacuum oven (VO-52D, USA; ±1 °C) was operated at a pressure of 0.05 MPa. To investigate the impact of drying temperature, the peppermint leaves were dried at drying temperatures of 40, 50, and 60 °C in both dryers. In total, six drying experiments were carried out by different drying methods and three temperature levels for each drying method.

Peppermint samples were distributed uniformly as a thin layer inside the rectangular boxes made from stainless steel wire mesh with dimensions of 15 × 10 cm and 5 cm height at a rate of about 10 g of fresh peppermint per box (Figure 1). These boxes were used to simply obtain the weight reduction of samples during drying. Five boxes were used for each drying experiment and assigned as replicates for data analysis. Before drying, the weight of the boxes with the fresh peppermint samples were recorded using accurate balance (BS-Series, China, capacity, 200g and accuracy of 0.001 g), and during drying, the sample boxes were taken out of the oven each 30 or 60 min (based on the drying temperature and method) to record the wight reduction in each sample. In each drying experiment, the peppermint samples were dried continuously until a desired moisture content of 12 ± 2% (d.b.) was achieved and the drying process ended at this time. Dried samples were collected for further physicochemical and phytochemical characteristics measurements.

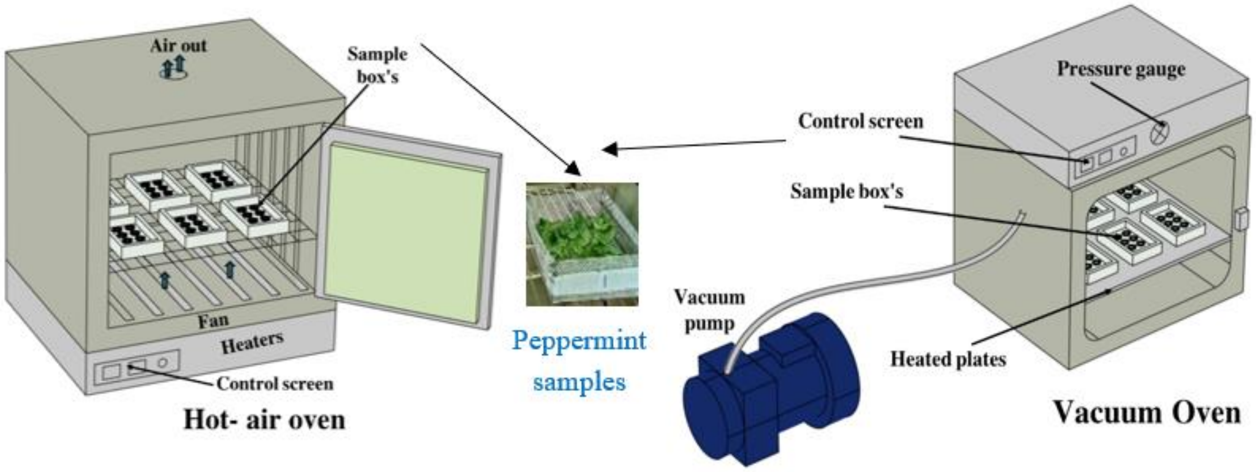

Figure 1. Drying setup and samples preparation.

2.3. Drying characteristics

Drying kinetics of peppermint leaves were determined based on the experimental results in terms of moisture content, drying rate, moisture ratio, effective moisture diffusivity, and activation energy as follows:

The drying rate (*Dr*) was calculated as:

$$Dr = \frac{M_t - M_{t+1}}{\Delta t} \tag{1}$$

and the moisture ratio (*MR*) was defined as:

$$MR = \frac{M_t - M_e}{M_i - M_e} \tag{2a}$$

where *DR* is the drying rate (d.b./ h), *M* is the MC of the samples (d.b) at time *t* and *t*+1 (min), and the subscripts *i* and *e* refer to the initial moisture and equilibrium MCs, respectively. Because the value of M_e is much lower than M_0 and M_t values, the Equation (2a) can be expressed as follows [33]:

$$MR = \frac{M_t}{M_i} \tag{2b}$$

The diffusivity coefficients (D_{eff}) of peppermint leaves can be determined based on Fick's second law of diffusion that describes drying process of agro-food materials [10].

$$\frac{\partial MR}{\partial t} = D_{eff} \nabla^2 MR \tag{3}$$

Assuming that temperature and diffusion coefficients are constant during drying, moisture migration is only caused by diffusion and shrinking is negligible; thus, the solution of Fick's law for an infinite slab (peppermint leave) can be given as follows:

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} exp\left[-\frac{(2n+1)^2 \pi^2 D_{eff} t}{4L^2}\right] \tag{4}$$

where *L* is the half thickness of peppermint leave (m) and *n* is the number of terms in the diffusion cycles. Utilizing the first term of the series when the drying times are considerable, this equation can be reduced to:

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

Therefore, the slope of the linear relationship between the dependent variable $(f(t) = ln\, MR)$ and the drying time (t) was then used to estimate the effective moisture diffusivity (D_{eff}) according to Eq. (6):

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

The activation energy (*Ea*) was calculated using Arrhenius type equations [23]:

$$D_{eff} = D_0 e^{(-\mathrm{Ea}/\mathrm{R}\theta)} \tag{7}$$

From Eq. (7), the plot of ln(D) versus 1/θ gives a straight slope of B:

$$B = Ea/R \tag{8}$$

where D_0 is the pre-exponential factor of the Arrhenius equation ($m^2 \cdot s^{-1}$) and R is the universal gas constant ($8.3144\ J \cdot k^{-1} \cdot mol^{-1}$).

2.4. Drying curve modeling

Mathematical modeling is helpful for understanding the drying process of materials. The peppermint moisture ratio determined during drying experiments was fitted to the selected eight drying models as listed in Table (1). Data fitting was done using a quasi-Newton numerical approach to solve a nonlinear least squares regression. The statistical parameters, such as R^2, RMSE, and χ^2, were calculated and used to select suitable model fitting the experimental data. The selected parameters were calculated from the following equations:

$$R^2 = \frac{\sum_{i=1}^{N} \left(MR_{\exp,i} - MR_{pre,i}\right)^2}{N-n} \tag{9}$$

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^{N} \left(MR_{exp,i} - MR_{pre,i}\right)^2} \tag{10}$$

$$\chi^2 = \frac{\sum_{i=1}^{N} \left(MR_{exp,i} - MR_{pre,i}\right)^2}{N-n} \tag{11}$$

where R^2 is the determination coefficient, RMSE is the root mean square error, χ^2 is the reduced chi-square. The subscripts *exp* and *pre* refer to experimental and predicted moisture ratio, respectively. N is the number of measurements and *n* is the number of model constants [34]. The appropriate model to describe the peppermint drying behavior was selected based on the higher value of R^2 and the lowest values of χ^2 and RMSE.

Table 1. Models of thin layer utilized to describe the drying of peppermint.

S.N.	Model	Model equation	References
1	Newton	$MR = e^{(-kt)}$	Erbay and Icier [35]
2	Page	$MR = e^{(-ktn)}$	Gürlek, Özbalta [36]
3	Henderson and Pabis	$MR = a \times e^{(-kt)}$	Henderson [37]
4	Two-term EXP	$MR = a \times e^{(-kt)} + (1 - a) \times e^{(-kat)}$	Kishk, ElGamal [34]
5	Wang and Singh	$MR = 1 + at + bt^2$	Wang and Singh [38]
6	Logarithmic	$MR = a \times e^{(-kt)} + c$	Hacihafizoglu, Cihan [39]
7	Midilli	$MR = a \times e^{(-ktn)} + bt$	Midilli, Kucuk [40]
8	Approximation of diffusion	$MR = a \times e^{(-kt)} + (1-a)e^{(-kbt)}$	Ertekin and Yaldiz [41]

2.5. Color measurement

Mitigation of the color change in dried peppermint would allow producers to achieve better-looking herbs that will ultimately command a higher price. As the color is the primary quality feature measured by consumers for product acceptance, it is used for assessing the effect of drying process on peppermint quality. Color of fresh and dried peppermint leaves was measured using a Minolta CR–400 Chroma Meter (Ltd., Japan). Color was expressed in the CIE system as L*, a*, and b* to represent darkness–lightness, redness–greenness, and yellowness–blueness, respectively. Finally, the total color difference (ΔE) was estimated as follows:

$$\Delta E^*_{ab} = \sqrt{(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2} \tag{12}$$

where 1 and 2 subscripts refer to the fresh and dried samples, respectively.

2.6. Pigments determination

The concentration of β-carotene and chlorophyll a, b was estimated using the procedure of Barros, Cabrita [42]. Peppermint leaves powder aggressively mixed with the solvent mixture (4 acetone: 6 hexane) for 5 min, the mixture was filtered, and the extract was made up to 10 mL. The samples were measured at a wavelength of 662 nm for chlorophyll a (*Chlo. a*), 664 nm for Chlorophyll b (*Chlo. b*) and 440 nm for β-carotene using spectrophotometer (Jenway LTD, Felsted, Dunmow, UK, 6505 UV/VIS). The formulas presented by Meften, Abdel-Razik [43], were utilized to determine the amounts of the pigments, which were then expressed in mg per 100 g dry weight:

$$\beta - carotene_{\left(\frac{mg}{ml}\right)} = (4.695 \times A_{440}) - 0.268(\mathrm{Chlo.a} + \mathrm{Chlo.b}) \tag{13}$$

$$Chlorophyll\ a_{\left(\frac{mg}{ml}\right)} = (9.784 \times A_{662}) - (0.99 \times A_{664}) \tag{14}$$

$$Chlorophyll\ b_{\left(\frac{mg}{ml}\right)} = (21.426 \times A_{664}) - (4.65 \times A_{662}) \tag{15}$$

Note A is the optical density at the wavelength indicated.

Specifications of the reagents used for this analysis are, acetone (ACS reagent, ≥99.5%, Sigma-Aldrich, St. Louis, USA) and hexane **(**HPLC grade, ≥95%, Merck)

2.7. Preparation of bioactive components and antioxidants extracts

Methyl alcohol (HPLC grade ≥99.9% purity, Fisher Scientific) was utilized as a solvent for the preparation of bioactive components and antioxidants extract using the procedure of Barros, Cabrita [42]. One g of chopped fresh peppermint leaves or finely dried powder that had been combined with 25 mL of methyl alcohol on an orbital shaker (LAB-LINE Instruments, Inc., USA) at 100 rpm for 60 min at ambient temperature (35 ± 1 °C). 25 mL of methanol was used to extract the residue once again. After mixing, the methanol extracts were kept at 4°C until additional examinations.

2.8. Determination of total phenolics content

Total phenolics content was also estimated according to the procedure outlined by Barros, Cabrita [42] using the methanolic extract. One mL aliquot of the extract (diluted 1:5 with water) was mixed with 5 mL of Folin–Ciocalteu phenol reagent (diluted with water 1:10 v/v) and 4 mL of sodium carbonate (75 g/L). For color development, the tubes were vortexed for 30 s and then let remain at ambient temperature (30 ± 1 °C) for 60 min. Using a spectrophotometer (see section 2.6), the absorbance was read at 765 nm. Gallic acid (0–0.10 mg/mL) calibration curve ($R^2 = 0.99$) was created and subjected to comparable treatments.

Specifications of the reagents used for this analysis are, Folin-Ciocalteu reagent (Sigma-Aldrich, St. Louis, USA), gallic acid (≥98% purity, Sigma-Aldrich, St. Louis, USA),

2.9. Determination of antioxidant activity

The DPPH scavenging activity was measured by the procedure of Ravichandran, Saw [44]. Briefly, DPPH solution (2,2-Diphenyl-1-picrylhydrazyl, Sigma-Aldrich, St. Louis, MO, USA) 6×10^{-5} M was mixed with 5-fold dilution of the methanol extracts for 30 s. The reaction was left for 30 min, and the samples were read at 515 nm. As a blank, DPPH solution without extract was examined. The antioxidant activity was computed as:

$$DPPH\ antioxidant\ activity\ (\%) = \left[\frac{(A_{blank} - A_{sample})}{A_{blank}}\right] \times 100 \tag{16}$$

where A is the measured absorbance at 515 nm.

The ABTS•+ radical scavenging activity was measured using the procedure of Maria do Socorro, Alves [45] using ABTS reagent (2,2'-azino-bis [3-ethylbenzothiazoline-6-sulfonic acid]) (Sigma-Aldrich, St. Louis, USA).

2.10. Statistical analysis

Statistical analysis of the experimental data was performed using the SPSS software (version 17.0, SPSS Inc, Chicago, USA). Data analyzed by one–way analysis of variance (ANOVA) and significant differences between the means were determined by the least significant difference (LSD) test at a level of significance of $P < 0.05$.

3. Results and discussion

3.1. Drying kinetics

The change in the MR of peppermint leaves at sequential drying times during drying convective and vacuum drying are presented in Figure 2. As expected, at lower temperatures, longer times were required to accomplish the drying process especially under vacuum drying (Figure 2). This behavior could be attributed to the high correlation between the moisture diffusivity of material and the drying temperature. A greater heat flux results from increasing the temperature differential between the medium and product, which raises the effective moisture diffusivity [46]. For instance, convective drying at 40 °C took 8 h to reach the desired moisture content compared to about 3 h when dried at a higher temperature of 50°C. It can be seen also in Figure 2 that the peppermint leaves dry very fast at the beginning of drying due to the big surface area and small thickness of the leaves which fasten the water evaporation from the leaves. Therefore, peppermint leaves take a short time for drying compared to other products at the same drying conditions. The results also showed that MR reduced as the drying time passes. The changes in MR gained in this study are similar with those reported by [47] in drying studies carried out on commercial pumpkin drying at different temperatures.

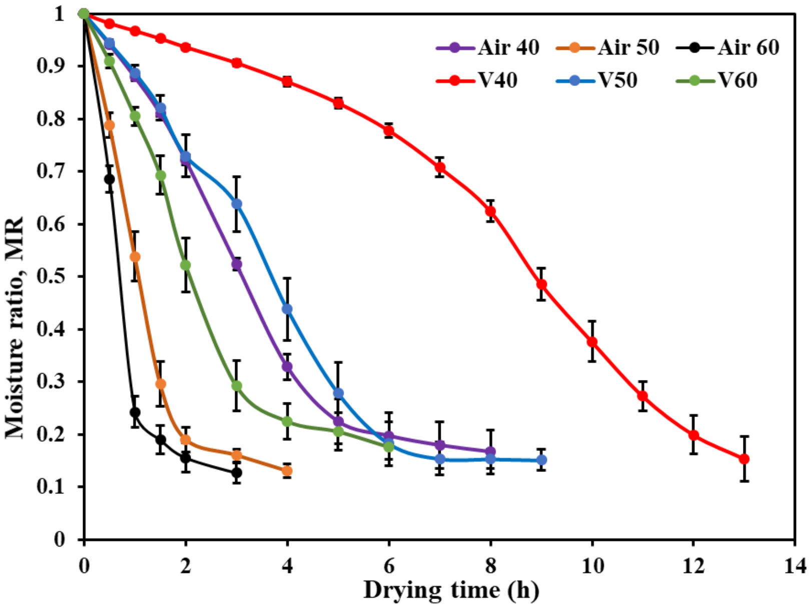

Figure 2. Measured moisture ratio of peppermint leaves during convective (Air) and vacuum (V) drying at temperatures of 40, 50, and 60 °C.

The drying rate of peppermint under convective and vacuum drying with different drying temperatures presented in Figure 3. In general, peppermint leaves followed only a falling rate period (phase 2) and no constant rate period (phase 1) was observed. Initially, the drying rates were found to be faster. This effect was related to the high amount of free moisture availability, which was easily removed in the initial stage of drying. Drying during phase 2, the drying is governed by the water diffusion in the material. This phenomenon was observed for other products [9,14]. The drying rate increased with the beginning of drying until reached a peak point at a specific MR value (critical moisture content) then decreased to a minimum value with the end of the drying process. In general, peppermint samples in convective drying showed higher drying rates than vacuum drying, and this could be due to the air velocity in the convective dryer which accelerate the moisture removal from the samples [48]. Convective drying at 60 °C recorded the highest drying rate of 0.62 (d.b./h) at MR of 0.24 (Figure 3). As the convective drying temperature decreased the peak of drying rate decreased and recorded at higher moisture ratios of peppermint samples. The drying rate peak recorded at 50 and 40 °C were 0.40 (d.b./h) at MR of 0.31 and 0.16 (d.b./h) at MR of 0.52, respectively. For vacuum drying, the drying rate peak was recorded at higher MR than convective drying at tested drying temperatures. Higher drying temperature of vacuum drying (60 °C) also recorded the higher drying rate of 0.25 (d.b./h) at MR of 0.48 compared with 0.13 (d.b./h) at MR of 0.59 and 0.11 (d.b./h) at MR of 0.48 at vacuum drying temperatures of 50 and 40 °C, respectively (Figure 3).

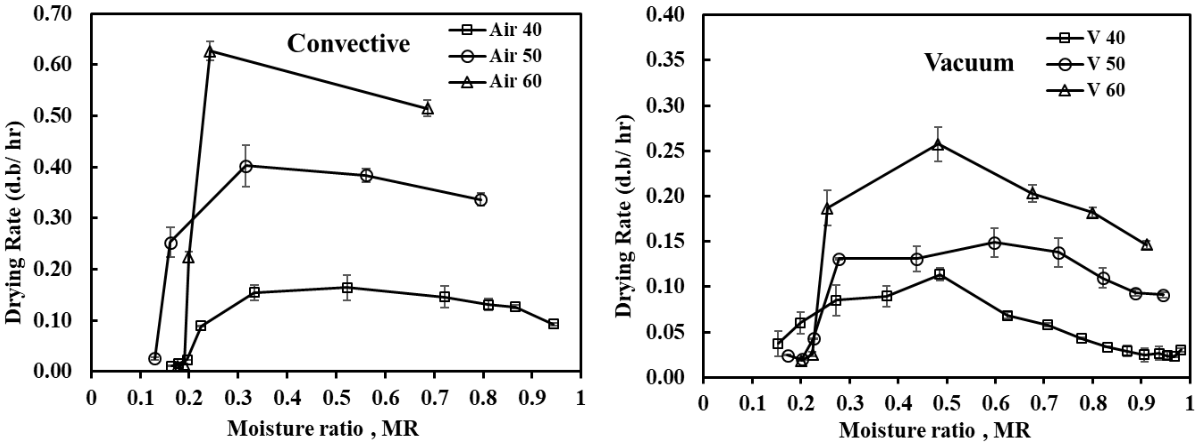

Figure 3. Drying rate of peppermint under different drying temperatures for convective (Air) and vacuum (V) drying.

Moisture diffusivity (D_{eff}) of peppermint leaves increased with the increase in drying temperature in both drying methods as presented in Table 2. Moreover, the peppermint leaves dried in convective dryer showed higher moisture diffusivity than samples dried in vacuum dryer. D_{eff} values varied from 1.88×10^{-13} to 5.16×10^{-13} $m^2·s^{-1}$ for convictive drying under temperature range from 40 to 60 °C. The corresponding values of D_{eff} for vacuum drying varied from 1.00×10^{-13} to 2.34×10^{-13} $m^2·s^{-1}$ under the same range of drying temperature. These values are within the general range 10-12 to 10–13 $m^2·s^{-1}$for drying of mint leaves [30]. Table 2 also shows R^2 values of the linear relationship between ln(MR) and time in determining D_{eff} as shown in Figure 4 for different drying conditions.

Activation energy (*Ea*) of peppermint leaves under both drying methods also presented in Table 2. convictive drying showed higher activation energy (41.46 kJ·mol^{-1}) than the vacuum drying (39.72 kJ·mol^{-1}) in the same range of drying temperature (40–60 °C).

Table 2. Moisture diffusivity (D_{eff}) and activation energy (*Ea*) of peppermint leaves under studied drying conditions.

Drying method	Convictive drying			Vacuum drying		
Temperature (°C)	40	50	60	40	50	60
$D_{eff} \times 10^{-13}$ ($m^2·s^{-1}$)	1.88	4.89	5.16	1.00	1.81	2.34
R^2	0.963	0.936	0.840	0.938	0.967	0.953
Ea (kJ·mol^{-1})	41.46			39.72		
R^2	0.948			0.820		

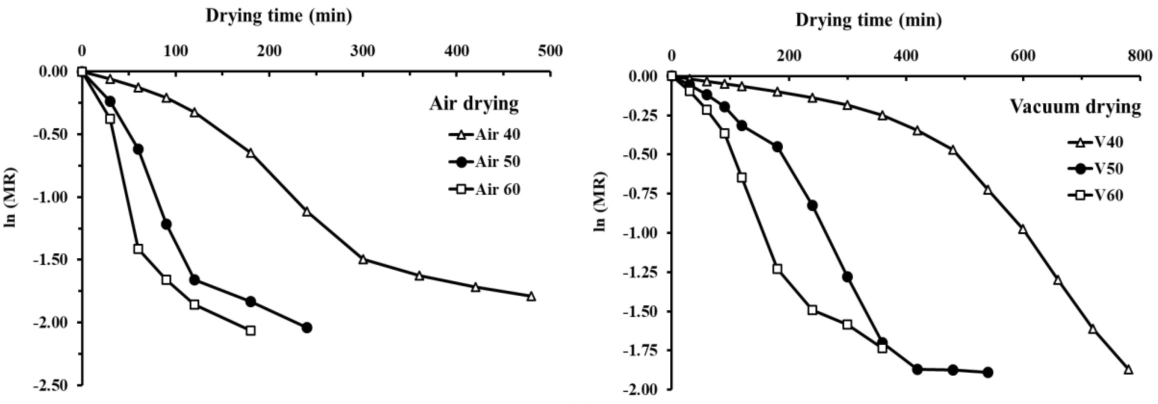

Figure 4. Effective moisture diffusivity of peppermint leaves under tested drying conditions.

3.2. Mathematical models

The curve fitting of moisture ratio of peppermint leaves were conducted on eight different drying models (Table 1). The statistical parameters (R^2, χ^2, RMSE) calculated to assess the goodness of each drying model are presented in Table 3. The models' constants and coefficients resulted from the nonlinear least squares regression are tabulated in Table 4. As illustrated in Table 3, most of the tested models showed a good fitting of the experimental moisture ratio with R^2 higher than 0.90. The data of vacuum drying at lower temperature of 40 °C showed R^2 less than 0.89 with some models.

Table 3. The statistical parameters used to evaluate the goodness of drying models for convective and vacuum drying of peppermint.

Drying method	Model	T = 40 °C RMSE			T =50 °C			T = 60 °C		
		R^2	χ^2	RMSE	R^2	χ^2	RMSE	R^2	χ^2	RMSE
Air drying	Newton	0.976	0.001	0.006	0.973	0.004	0.059	0.936	0.0080	0.083
	Page	0.934	0.010	0.082	0.989	0.002	0.035	0.934	0.0100	0.082
	Henderson and Pabis	0.974	0.007	0.052	0.972	0.005	0.055	0.937	0.0100	0.083
	Logarithmic	0.976	0.009	0.049	0.975	0.005	0.051	0.954	0.0090	0.068
	Two term EXP	0.989	0.003	0.034	0.973	0.005	0.059	0.939	0.0090	0.079
	Wang and Singh	0.983	0.006	0.048	0.986	0.003	0.041	0.956	0.0070	0.066
	Midilli	0.998	0.001	0.015	0.997	0.001	0.017	0.988	0.0040	0.035
	Approximation of diffusion	0.990	0.004	0.032	0.991	0.002	0.035	0.954	0.0090	0.071
Vacuum drying	Newton	0.885	0.015	0.119	0.982	0.003	0.056	0.967	0.004	0.062
	Page	0.993	0.001	0.029	0.983	0.001	0.025	0.977	0.003	0.047
	Henderson and Pabis	0.871	0.013	0.106	0.979	0.002	0.045	0.966	0.004	0.055
	Logarithmic	0.951	0.005	0.064	0.985	0.002	0.037	0.968	0.005	0.054
	Two term EXP	0.949	0.005	0.047	0.994	0.001	0.023	0.979	0.003	0.044
	Wang and Singh	0.991	0.001	0.028	0.988	0.002	0.038	0.976	0.004	0.052
	Midilli	0.994	0.001	0.032	0.996	0.002	0.020	0.996	0.001	0.019
	Approximation of diffusion	0.955	0.005	0.063	0.995	0.002	0.0023	0.982	0.002	0.041

Table 4. Models' coefficients and constants for convective and vacuum drying of peppermint.

Drying method	Model	Models' coefficients and constants		
		T = 40 °C	T = 50 °C	T = 60 °C
Convective drying	Newton	k = 0.2314	k = 0.6940	k = 0.9906
	Page	k = 0.1472; n = 1.3396	k = 0.6197; n = 1.3769	k = 0.9946; n = 0.8857
	Henderson and Pabis	k = 0.2548; a = 1.0776	k = 0.7293; a = 1.0458	k = 0.9970; a = 1.0058
	Two term EXP	k = 0.3735; a = 1.9327	k = 1.1142; a = 1.9618	k = 1.7170; a = 0.4080
	Wang and Singh	a = −0.1943; b = 0.0107	a = −0.5791; b = 0.0937	a = −0.8146; b = 0.1832
	Logarithmic	k = 0. 2058; a = 1.1815; c = −0.1209	k = 0.6002; a = 1.1440; c = −0.1091	k = 1.4067; a = 0.9046; c = 0.1239
	Midilli	k = 0.1117; n = 1.6793; a = 0.9776; b = 0.0196	k = 0.6797; n = 1.6739; a = 0.9910; b = 0.0354	k = 1.5831; n = 1.7803 a = 1.0046; b = 0.0730
	Approximation of diffusion	k = 3197; a = 1.3674; b = 3.6754	k = 0.9922; a = 1.4864; b = 3.1731	k = −0.1215; b = 0.0005 b = 2653360
Vacuum drying	Newton	k = 0.0783	k = 0.2051	k = 0.3395
	Page	k = 0.0028; n = 2.5279	k = 0.1252; n = 1.3478	k = 0.2662; n = 1.2809
	Henderson and Pabis	k = 0.0924; a = 1.1087	k = 1.0716; a = 0.2252	k = 1.0618; a = 0.3685
	Two term EXP	k = 0.1590; a = 2.1734	k = 0.3287; a = 1.9105	k = 0.5481; a = 1.8995
	Wang and Singh	a = −0.0180; b = −0.0039	a = −0.1651; b = 0.0071	a = −0.2972; b = 0.0262
	Logarithmic	k = 0.0002; a = 3200; c = −3200	k = 0.1486; a = 1.3212; c = −0.2764	k = 0.3205; a = 1.1323 c = −0.0802
	Midilli	k = −0.0001; n = 1.2865; a = 1.0819; b = −0.0396	k = 0.0837; n = 1.7855 a = 0.9491; b = 0.0179	k = 0.2454; a = 1.8312 a = 0.9821; b = 0.0396
	Approximation of diffusion	k = 0.2245; a = 4031; b = 1.0003	k = 0.2964; a = 1.4811; b = 2.8210	k = 0.4471; a = 1.2551; b = 5.4772

From Table 3, the model's goodness was related to the temperature and drying method. For the convective drying, the higher drying temperature of 60 °C showed lower R^2 values, higher χ^2 values and RMSE for all tested models. On the contrary, for vacuum drying the lower drying temperature of 40 showed lower values of R^2 and higher values for χ^2 and RMSE for all tested models (except Wang and Singh model). To be able to select the suitable model for each drying method, the average values of the statistical parameters (R^2, χ^2, RMSE) were calculated for each model at the different three drying temperatures. In general, it can be concluded that for both convective and vacuum drying, the model of Midilli gave the highest average R^2 value (>0.99) and lowest χ^2 average values (<0.003) and RMSE (<0.035) at the different drying temperatures compared with the other examined models. It should be noted here also that the Page model shows a good fit for vacuum drying data with R^2 (>0.97). Therefore, these models could effectively explain the drying process of peppermint leaves by convective and vacuum drying. These models were reported by many researchers as suitable models for different products. The Midilli model was selected to illustrate the drying behavior of peppermint leaves [1]. Page model was also stated as suitable model for coffee beans [49].

The drying process of peppermint leaves under various drying conditions is not well explained by any generic model that has been documented in the literature. Some models have been identified as the most effective models for peppermint leaves drying under particular conditions, such as two-term model for drying in tunnel dryer [9], the logarithmic model for convective drying [20], and Wang and Singh model for the solar and open sun drying [23]. Modified H.&P. model was found to be the more appropriate for the solar drying of whole peppermint plant [50].

Midilli model for the convective and vacuum drying of peppermint can be expressed according to the projected coefficients as a function in drying temperature as follows:
Midilli model form:

$$MR = a\exp(-kt^n) + bt \tag{17}$$

For convective drying:

$$MR = (0.0014T + 0.9236) \times \exp\left[-(0.0736T - 2.887) \times t^{(0.0006T\hat{}2 - 0.0509T + 2.8189)}\right] + (0.0027T - 0.0908) \times t \tag{18}$$

For vacuum drying:

$$MR = (0.0041T + 0.7385) \times \exp\left[-(0.0122T - 0.5026) \times t^{(0.0087T\hat{}2 - 0.9477T + 27.527)}\right] + (0.0015T - 0.0515) \times t \tag{19}$$

note MR is the moisture ratio, t is the time (h) and T is the drying temperature (°C).

Figure 5 demonstrates the link between the experimental measured values and predicted MR values for drying peppermint leaves using two distinct drying techniques, as predicted by the Midilli model. Strictly speaking, this model provides an excellent match between the expected and experimental MR of peppermint leaves dried in both convective and vacuum dryers.

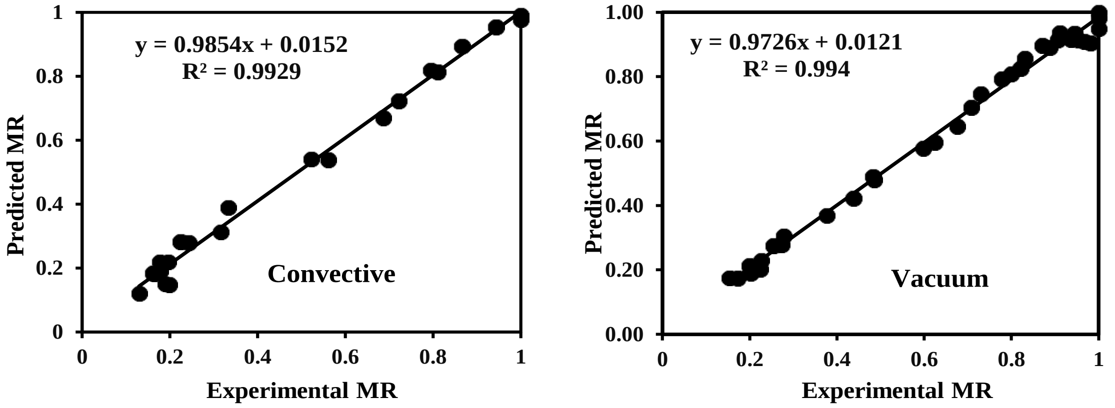

Figure 5. The experiment measured MR and the predicted MR values by Midilli model for peppermint.

3.3. Color change

The mean color values (L*, a*, and b*) and the total color difference (ΔE) of peppermint leaves are shown in Table 5. The color values of fresh leaves were L* = 41.46 ± 0.19, a*= −3.60 ± 0.29, and b* = 26.38 ± 0.22. The results showed that all color values significantly changed ($p < 0.05$) after drying under different drying methods and drying temperatures as presented in Table 3. This is due to the changes in cellular structure during drying which significantly impact color by altering pigment distribution and concentration. The cell walls and membranes break down under water stress and high temperatures, exposing the chlorophylls and carotenoids more to oxygen and light, which can lead to pigment degradation and discoloration [51]. These structural breakdowns not only reduce pigment content but also affect the light-reflecting properties of the leaf surface, contributing to visible color changes.

As shown in Table 5, both drying methods slightly decreased the color lightness (L^*), this indicates that dried samples had a bright color close to the fresh samples. It's possible that drying temperature and drying time are to blame for the decline in L* values observed at all drying temperatures [52]. However, leaves dried at all temperatures showed an increase in a* value. Vacuum-dried leaves revealed significantly ($p < 0.05$) higher lightness and lower a^* values than that of convective dried leaves at high temperature (50 and 60 °C). On the other hand, there was a decrease in b* values for dried peppermint leaves. The vacuum dried peppermint leaves were characterized by a higher yellowish and luminosity color than in convective dried peppermint leaves. The mixture of chlorophyll and magnesium ions is responsible for the natural green color of leaves. During drying, the chlorophyll molecules could be changed to pheophytin and pyro-pheophytin [53]. In general, the lower a*/b* and higher L* values are preferable in dried food products. According to Dwivedy, Rayaguru [54], drying temperature greatly increased the a* values in convective dried leaves, causing them to rise from −0.41 (at 50 °C) to −0.04 (at 70 °C). The reason behind this could be the non-enzymatic browning response, which caused the samples to become less green as the temperature increased [55]. The total color difference ΔE increased significantly with increasing the convective drying temperature. Meanwhile in vacuum drying, the increases in drying temperature showed no signific effect on ΔE (Table 5). Some studies revealed that the color of peppermint and bay leaves did not significantly change with the drying temperature [9,56,57].

Table 5. Chromatic color values recorded peppermint leaves samples.

Drying methods	Lightness L*	Redness/Greenness a *	Yellowness/Blueness b *	Total color difference ΔE
(Fresh sample)	41.46 ± 0.19^a	-3.60 ± 0.29^c	26.38 ± 0.22^a	-
Convective (40 °C)	38.34 ± 0.98^b	-0.27 ± 0.03^a	15.15 ± 0.18^b	12.12 ± 0.21^a
Convective (50 °C)	33.87 ± 0.43^c	-0.54 ± 0.10^b	12.93 ± 0.21^c	15.74 ± 0.19^b
Convective (60 °C)	32.03 ± 0.56^d	-0.11 ± 0.27^a	12.81 ± 0.27^c	16.89 ± 0.23^c
(Fresh sample)	41.46 ± 0.19^a	-3.60 ± 0.29^c	26.38 ± 0.22^a	-
Vacuum (40 °C)	38.04 ± 0.92^b	-0.14 ± 0.01^a	14.22 ± 0.08^b	13.10 ± 0.22^a
Vacuum (50 °C)	35.68 ± 0.46^b	-0.62 ± 0.31^b	14.76 ± 0.29^b	13.32 ± 0.25^a
Vacuum (60 °C)	35.80 ± 0.39^b	-0.17 ± 0.04^a	14.19 ± 0.22^b	13.87 ± 0.18^a

Means within a column showed with different superscript letters are significantly different at ($p < 0.05$).

3.4. Pigments

The concentrations of chlorophyll *a*, b, and β-carotene in fresh peppermint leaves before being dried were 131.87 ± 0.82, 99.24 ± 1.98, and 56.82 ± 0.52 mg/100 g (dry weight, DW), respectively. In general, both drying approaches produced a significant ($p < 0.05$) increase in chlorophyll *a* and b contents and a significant ($p < 0.05$) decrease in β-carotene content of peppermint leaves as presented in Table 6. Moreover, vacuum drying and lower temperatures showed less effect than convictive drying and higher temperatures. This is due to the thermal and oxidative factors affected by drying temperatures and techniques. Higher temperatures appear to stabilize chlorophyll by concentrating it as other cellular components break down, giving an impression of increased chlorophyll on a dry weight basis. Chlorophylls are also relatively more resistant to oxidation, especially in lower-oxygen environments like vacuum drying, helping retain their structure better during the drying process [58]. This findings in agreement with the results reported by Rocha, Marty-Audouin [59] who found that the concentration of total chlorophyll increased by 27% when the drying temperature increased by 20 degrees (from 40 to 60 °C). Conversely, carotenoids, which are more sensitive to heat and oxygen, degrade quickly under elevated temperatures and oxidative conditions, particularly during convective drying where oxygen is more readily available [60]. According to [61], the carotenoids in the leafy vegetables was significantly reduced by drying techniques including air, oven, and microwave drying.

Water stress during drying also affects chlorophyll and carotenoid content by influencing pigment stability and degradation. Rapid moisture loss under high temperatures alters cellular structure and increases solute concentration, which can accelerate oxidative reactions. This stress can lead to chlorophyll stability on a dry weight basis, as water loss effectively concentrates chlorophyll molecules. In contrast, carotenoids, which are more heat-sensitive, degrade faster under water stress, especially when exposed to oxygen, leading to their reduced content [62]. Studies on drying techniques indicate that carotenoid loss is inevitable at high temperatures, with convective drying leading to a more significant decline compared to vacuum drying, where oxygen levels are controlled [63]. Consequently, drying temperature and method critically affect pigment retention, with chlorophylls being more stable and carotenoids being highly susceptible to degradation.

Table 6. Main pigment's contents (mean ± SD) of fresh and dried peppermint leaves.

Sample	Chlorophyll a (mg/100 g DW)	Chlorophyll b (mg/100 g DW)	Carotenoids (mg/100 g DW)
Fresh	131.87 ± 0.82^{c}	99.24 ± 1.98^{d}	56.82 ± 0.52^{a}
Convective (40 °C)	119.20 ± 1.89^{d}	136.12 ± 0.88^{c}	49.31 ± 0.44^{b}
Convective (50 °C)	188.02 ± 1.25^{a}	173.95 ± 1.60^{b}	22.83 ± 0.80^{c}
Convective (60 °C)	154.97 ± 0.70^{b}	266.27 ± 1.69^{a}	11.37 ± 0.91^{d}
Fresh	131.87 ± 0.82^{c}	99.24 ± 1.98^{d}	56.82 ± 0.52^{a}
Vacuum (40 °C)	127.08 ± 1.11^{d}	169.85 ± 1.40^{c}	45.37 ± 0.72^{b}
Vacuum (50 °C)	146.11 ± 1.30^{b}	180.26 ± 1.74^{b}	43.80 ± 1.97^{c}
Vacuum (60 °C)	198.82 ± 2.10^{a}	198.95 ± 0.80^{a}	11.16 ± 0.37^{d}

Means within a column showed with different superscript letters are significantly different at ($p < 0.05$)

From Table 6, convective drying at temperature of 40 °C caused significant decrease in chlorophyll *a* content in dried samples compared to fresh samples (from 131.87 to 119.20 mg/100g DW). Drying at 50 °C showed higher value for chlorophyll *a* than other drying temperatures. On the other hand, dried leaves by convective drying at temperatures of 40 °C revealed significant ($p < 0.05$) increase in the values of the chlorophyll b content compered to fresh samples (from 99.24 to 136.12 mg/100g DW). Increase the drying temperature to 50 and 60 °C resulted in a significant ($p < 0.05$) increase chlorophyll b content in dried samples. For vacuum drying, the increase in drying temperature increased the contents of chlorophyll *a*, b significantly ($p < 0.05$).

The carotenoids content is reduced by both drying methods when the drying temperature increased. Similar to chlorophyll *a*, convective drying at temperature of 40 °C caused a nonsignificant decrease in carotenoids content in dried leaves compared to fresh leaves (from 56.82 to 49.31) as shown in Table 6. While increase the drying temperature to 50 and 60 °C decreased carotenoids content significantly ($p < 0.05$) to 22.83 and 11.37, respectively (Table 4). The vacuum drying at 40 °C resulted in a significant reduction in carotenoids content in dried peppermint leaves compared to fresh leaves (from 56.82 to 45.37). While increasing the temperature in vacuum drying to 50 °C shows a nonsignificant effect on carotenoids content compared with vacuum drying at 40 °C. Further increase in the vacuum drying temperature to 60 °C decreased carotenoids content significantly ($p < 0.05$). Therefore, in order to maintain carotenoids content in dried peppermint leaves temperature of 40 °C should be used in convective drying and this temperature can be increased to 50 °C in vacuum drying.

3.5. Polyphenols and antioxidants activities

Total phenolics contents of fresh peppermint leaves were found to be 71.93 mg GAE per 100 g (DW) sample. The results in Table 7 proven that total phenolics content were significantly ($p < 0.05$) differed based on drying methods. It is clear that, dried samples extract always showed higher phenolic concentration than this from fresh samples. The improved extraction of the insoluble phenols like condensed tannins or phenolic acids, from the dried samples may be associated with the increased total phenolic content in those samples [64] related to the cell wall polysaccharides or, more specifically, proteins [65]. Additionally, the moderate heat treatment might have caused the phenolic sugar glycosidic linkages to break and phenolic aglycons to form. These aglycons react more favorably with the Folin-Ciocalteu substance, which results in higher total phenolic content values. These findings in agreement with Martínez-Las Heras, Heredia [66] who found that the drying process persimmon leaves had a significant effect on the extraction of phenols. Furthermore, they reported that the extracts from persimmon leaves dried by convective drying at air temperature of 100 °C had the highest concentration of total phenolics than lyophilized leaves, air-dried leaves at 180 °C, and shade-dried leaves. Al-Farsi, Alasalvar [67] and Chang, Lin [68] attributed the increment in phenolic compounds in dried tomato and apple, by 56.9 and 4.9 fold, respectively, to the release of bound phenolics and the hydrolysis of complex phenolic compounds, leading to the low MW compounds production.

It is also clear from Table 7 that the lower convective drying temperature of 40 °C showed high total phenolics content of 141.35 (mg GAE per 100 g) and this value decreased nonsignificant to 139.85 (mg GAE per 100 g) when drying temperature increased to 50 °C (Table 7). Meanwhile, further increase in drying temperature to 60 °C decreased the total phenolics content significantly ($p < 0.05$) to 130.44 (mg GAE per 100 g). In other words, a drying temperature of 50 ºC can be used instead of 40 ºC to increase drying rate without significant effect on total phenolics content in dried peppermint leaves. On the other hand, different findings showed in vacuum drying, where drying at 50 ºC showed higher values for total phenolics content and the increase or decrease of drying temperature to 40 °C or 60 °C decreased the total phenolics content significantly ($p < 0.05$) as showed in Table 7. The reduction in total phenolics content at high temperatures might be due to the long exposure time for temperature which 13 h (Figure 2) to end the drying process compared with 6 h and 9 h at drying on 60 °C and 50 °C, respectively.

Table 7. Total phenols contents and antioxidant capacity (mean ± SD) of fresh and dried peppermint leaves.

Sample	Total phenols (mg GAE per 100 g)	DPPH (%)	ABTS (μmol trolox per 100 g)
Fresh	71.93 ± 1.01^{c}	$46.34^{c} \pm 0.46^{c}$	481.61 ± 1.98^{d}
Convective (40 °C)	141.35 ± 0.97^{a}	49.21 ± 0.31^{a}	1335.63 ± 2.97^{a}
Convective (50 °C)	139.85 ± 0.44^{a}	48.10 ± 0.33^{b}	1317.47 ± 3.95^{b}
Convective (60 °C)	130.44 ± 0.67^{b}	42.90 ± 0.47^{d}	1275.09 ± 2.92^{c}
Fresh	71.93 ± 1.01^{d}	46.34 ± 0.46^{c}	481.61 ± 1.98^{d}
Vacuum (40 °C)	134.29 ± 2.03^{b}	51.10 ± 0.15^{a}	1335.03 ± 2.47^{a}
Vacuum (50 °C)	142.79 ± 2.75^{a}	48.12 ± 0.27^{b}	1306.57 ± 2.97^{b}
Vacuum (60 °C)	117.79 ± 1.96^{c}	46.63 ± 0.24^{c}	1168.53 ± 2.99^{c}

Means within a column showed with different superscript letters are significantly different at ($p < 0.05$).

The intricacy of plant composition and potential interactions with their structure make it impossible to assess the antioxidant activity using a single technique [69]. Therefore, in this work, the antioxidant activity was determined by DPPH and ABTS methods. Fresh peppermint leaves showed values of 46.34% and 481.61 μmol trolox per 100 g DW for DPPH and ABTS, respectively. Antioxidant activity values were higher in dried leaves as compared to fresh leaves (except for samples dried at 60ºC in convective oven drying showed significantly lower value of 42.90% as measured by DPPH scavenging activity method), which is consistent with the total phenolic content data (Table 7). The amount of phenolic compounds is closely related with the antioxidant capacity [70]. These results are in agreement with results obtained by Benjamin, Ng [71] who found that bamboo leaves dried by different drying methods showed higher antioxidants capacity compared to control samples. The higher antioxidant activity in dried leaves could be due to the thermal inactivation of antioxidant oxidative enzymes like polyphenol oxidase, which suppresses oxidation [65]. In addition, Capecka, Mareczek [72] reported that drying method might be enabled the release of cell wall compound, and as a result, their measured antioxidant content increased as well as the disintegration of polymeric substances caused an increase in monomer forms that are determined by the assay [73].

From Table 7, it is clears that both drying methods had significant ($p < 0.05$) increment effects on DPPH and ABTS scavenging activity of peppermint leaves. When comparing dried samples, the antioxidant capacity determined by ABTS method was slightly higher in samples dehydrated by convective drying than vacuum dried samples. Drying temperature showed significant ($p < 0.05$) effect on antioxidant capacity in both drying methods (Table 7). When the temperature increased the antioxidant capacity in dried peppermint samples was significantly ($p < 0.05$) decreased in both drying methods (Table 7). Generally, during food dehydration, substances with differing levels of antioxidant activity might accumulate and generate compounds that may have antagonistic or synergistic effects with other sample contents or with one another. Research is still ongoing on these intricate chemical interactions that affect the food's functional qualities during drying [74,75].

4. Conclusions

The Midilli model presented the thin layer drying behavior of peppermint leaves in hot air and vacuum dryers with the highest R^2 and lowest values of χ^2 and RMSE. A longer time was required to accomplish the drying at the lower temperature of 40 °C, especially under vacuum drying. Effective moisture diffusivity increased with the increase in drying temperature and ranged from1.00×10^{-13} to 5.16×10^{-13} ($m^2 s^{-1}$). Activation energy ranged from 39.72 to 41.46 (kJ mol^{-1}).

All tested drying conditions slightly decreased the lightness (L^*) of dried peppermint leaves and the dried samples showed a bright color similar to the fresh samples. The two drying methods resulted in a significant increase in the chlorophyll a and b contents of peppermint leaves while causing a reduction in β-carotene content.

To maintain carotenoid content in dried peppermint leaves, a temperature of 40 °C should be used in convective drying and this temperature can be increased to 50 °C in vacuum drying. The total phenolics content significantly ($p < 0.05$) changed according to the drying method and temperature used. Drying temperature showed a significant ($p < 0.05$) effect on antioxidant capacity in both drying methods where the antioxidant capacity in dried peppermint was decreased with the increase of temperature in both drying methods. The results of this study are helpful for understanding the influence of drying method and temperature on the quality attributes of peppermint and optimizing the drying process to maintain the quality of the dried products.

Use of AI tools declaration

The authors declare they have not used Artificial Intelligence (AI) tools in the creation of this article.

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Conflict of interest

All authors declare no conflicts of interest in this paper.

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