Convective, vacuum and microwave drying kinetics of mallow leaves and comparison of color and ascorbic acid values of three drying methods

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Abstract
Mallow leaves (Malva sylvestris L.) with initial moisture of 5.02±0.003 on dry basis (82.5% on wet basis) were dried using three different drying methods, microwave, convective and vacuum. The leaves that weigh 75 g each were dried until their moisture fell down to 0.10±0.005 on dry basis (approximately 9% on wet basis). The following drying levels were used in each of the drying processes: 6.67, 8.67, 10, 11.33 W g⁻¹ microwave power density; 50, 75, 100 and 125 °C for convective drying; and 3, 7 kPa at 50 and 75 °C for vacuum drying. Drying periods ranged from 6-10, 26-150 and 38-130 min. for microwave, convective and vacuum drying, respectively. Effective moisture diffusivities ranged from 2.04403×10⁻¹⁰ to 1.85599×10⁻¹¹ m²·s⁻¹ for microwave drying, respectively. According to ascorbic acid content and color parameters, the best microwave power density was found 10 W g⁻¹ with a drying period of 6.5 min.

Keywords: ascorbic acid; convective drying; color; mallow; microwave drying; vacuum drying.

1 Introduction
Mallow (Malva sylvestris L.) is a species of the Mallow genus Malva in the family of Malvaceae (Zohra et al., 2013). It is commonly used as vegetable. Edible uses are concerned with folk gastronomy. Young leaves are eaten raw in salads, leaves and shoots are consumed in soups and as boiled vegetables (Samavati & Manoochehrizade, 2013). Mallow leaves have been used in folk medicine of Brazil and several parts of world for the treatments of colitis and stomatitis, in case of chronic bronchitis, against furuncle and abscess, contusions and hemorrhoids as well as other dolorous and inflammatory processes (Zohra et al., 2013).

Drying can extend the consumption period of mallow leaves while maintaining the nutrition content by evaporating the moisture in the product up to certain threshold value (Alibas, 2007). The removal of moisture prevents the growth and reproduction of microorganisms causing decay and minimizes many of the moisture-mediated deterioration reactions (Balbay et al., 2012). Convective drying methods can cause adverse changes in the taste, color and nutrient content of the dry product as a result of the long drying period, required high temperature, and reduction in density (Drouzas et al., 1999; Maskan, 2000). Microwave drying of products has become common because microwave drying prevents a decline in the quality of the product and ensures the rapid and efficient distribution of heat within the material (Li et al., 2009; Dong et al., 2011; Silva et al., 2007). Moreover, microwave drying reduces the drying time and saves energy during drying in producing high quality dry products (Balbay et al., 2011; Li et al., 2010). Vacuum drying includes lower process temperatures, less energy usage and hence greater energy efficiency, improved drying rates, and in some cases, less shrinkage of the product (Alibas, 2007, 2009).

The aims of this study were i) to evaluate the efficacy of microwave, convective and vacuum drying techniques for mallow leaves; ii) to examine the changes in color and ascorbic acid content of the product after drying; iii) to determine the effective moisture diffusivities for drying methods; and iv) to determine the optimum drying method for drying mallow leaves considering the color, ascorbic acid and drying period.

2 Material and methods
2.1 Biological material
Fresh mallow leaves (Malva sylvestries L.) which were selected from healthy and uniform plants used for the drying experiments were collected from plants in the Uludag University Campus area of Bursa, Turkey in 2013.

2.2 Drying equipment and drying method
Microwave and convective drying treatment was performed in a domestic digital combine oven (Arcelik MD 592, Turkey) with technical features of 230 V~ 50 Hz and 2900 W. The microwave oven has the capability of operating at eight different microwave stages among 90 and 1000 W. The convective oven has the capability of operating at nine different temperature stages among 50 and 250 °C at 1 m·s⁻¹ air velocity. The area on which microwave and convective drying is carried out was 327 × 280 mm diameter at the base of the oven. Time adjustment is

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done with the aid of a digital clock located on the oven. Vacuum drying treatment was performed in a laboratory type vacuum oven (Nuve EV 0180, Turkey) with technical features of 220 V, 50 Hz, 3.5 A and 800 W. The temperature of vacuum oven has a sensitivity of 1 °C, and temperature being 250 °C. The area on which vacuum drying is carried out was 300 mm × 200 mm × 250 mm in size. A laboratory-type vacuum pump (Carpanelli MMDE80B4, Bologna, Italy) was used in the vacuum drying trials under operating conditions of 220/240V, 50/60 Hz, and 5.1/4.8 A. The vacuum pump is reached to the lowest vacuum value (0.1 kPa) within 20 s. An analogue vacuum-meter which indicates the vacuum value in terms of kPa exists on the vacuum oven. Time adjustment is done with the aid of a programmable clock located on the oven.

Drying experiments were conducted using three different drying methods, namely, microwave, convective and vacuum drying. Three different experimental designs were formed for each one of the methods. Microwave drying (MD) trial was carried out at four different microwave generation powers being 850, 750, 650 and 500 W for wight of 75 g. Convective drying (CD) trial was carried out at four different temperatures being 50, 75, 100 and 125 °C. Four different vacuum-temperature combinations were obtained in vacuum trials by combining two different vacuum levels i.e. 3 and 7 kPa and two different temperature regimes at 50 and 75 °C, and the trials were realised under the combinations of 50 °C – 3 kPa, 50 °C – 7 kPa, 75 °C – 3 kPa and 75 °C – 7 kPa. The mallow leaves to be dried were 75±0.09 g in weight and selected from the uniform, and healthy plants. Three different drying trials were conducted at each drying technique and the values obtained from these trials were averaged and the drying parameters were determined. Dried mallow leaves which were being dried were removed from the oven periodically (every 30 s for microwave drying and every five min for vacuum and convective drying) during the drying period, and the moisture loss was determined by weighing the plate using digital balance (Sartorious EX 2000A, Germany) with 0.01 g precision. All weighing processes were completed in 10 s during the drying process. Drying process continued until the moisture content of mallow fell down to 0.1±0.005 on dry basis (Alibas, 2009). Energy consumption of microwave, air and vacuum oven (with vacuum pump) was determined using a digital electric counter (Kaan, Type 101, Turkey) with 0.01 kWh precision.

The following common semi-empirical Page’s equation (Equation 1) was used to describe the thin layer drying kinetics of mallow leaves (Alibas, 2007). Where: is the moisture ratio; is the moisture content db; is the equilibrium moisture content db; is the drying constant in min⁻¹; and is the dimensionless exponent. The equilibrium moisture content was assumed to be zero for laboratory type drying methods (Alibas, 2007).

\[ MR = \frac{X - X_e}{X_0 - X_e} = \exp(-kt^n) \]  

**2.3 Data analysis**

The research was conducted using randomized plots factorial experimental design. Determination of the investigated components was carried out in three replicates. Mean differences were tested for significance by using an LSD (MSTATC) test at 1% level of significance. Non-linear regression analysis was performed using SPSS 13.0 to estimate the parameters k and n of semi empirical Page’s equation (Equation 1). Regression results include the coefficients for the equation and coefficient of determination .

**2.4 Color parameters and ascorbic acid**

Leaf color was determined by two readings on each of the different symmetrical faces of the leaf using a Minolta CR 400 colorimeter (Konica-Minolta, Osaka, Japan), calibrated with an a white standard tile. The color brightness coordinate L measures the whiteness value of a color and ranges from black at 0 to white at 100. The chromaticity coordinate a measures red when positive and green when negative, and the chromaticity coordinate b measures yellow when positive and blue when negative (Soysal, 2004).

Ascorbic acid was determined by extracting the samples with oxalic acid (0.4%) and then reading and calculating the absorbency values at 520 nm with a spectrophotometer (Shimadzu UV-120-01, Shimadzu Co., Duisburg, Germany) (Alibas, 2009).

**2.5 Effective moisture diffusivity and activation energy**

Experimental results can be interpreted by using Fick’s diffusion equation. Fick’s second law of unsteady state diffusion given in Equation 2 (Sarimeseli, 2011; Evin, 2012; Dadali et al., 2008; Demirhan & Ozbek, 2011).

\[ MR \ = \frac{M - M_0}{M_0 - M_e} = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n+1)^2} \exp\left(-\frac{(2n+1)^2 D_{eff} \pi^2}{4L_s^2} t\right) \]  

where: is the moisture ratio; is the moisture content at a specific time [kg (moisture)/kg (dry matter)]; is the initial moisture content [kg (moisture)/kg (dry matter)]; is the equilibrium moisture content [kg (moisture)/kg (dry matter)]; is the effective moisture diffusivity (m² min⁻¹); is the half thickness (drying from both sides) of mallow leaves (m) (L_s = 0.34 ± 0.010mm), and is drying time (min). For long drying times, n=1, Equation 3 can be written as:

\[ MR \ = \frac{M - M_0}{M_0 - M_e} = \frac{8}{\pi^2} \exp\left(-\frac{D_{eff} \pi^2}{4L_s^2} t\right) \]  

\[ \ln(MR) = \ln\left(\frac{8}{\pi^2}\right) - \frac{D_{eff} \pi^2}{4L_s^2} t \]  

\[ S = \frac{D_{eff} \pi^2}{4L_s^2} \]  

Diffusivities are typically determined by plotting experimental drying data in terms of lnMR versus drying time.
Drying of mallow leaves

3 Results and discussion

3.1 Drying curves

Microwave drying trials were conducted at the microwave power densities of 6.67, 8.67, 10 and 11.33 W g\(^{-1}\). Convective drying trials were conducted at air temperatures of 50, 75, 100 and 125 °C. Four different combination levels were tested by combining temperature levels of 50 and 75 °C with vacuum of 3 and 7 kPa. Moisture-time diagrams of mallow leaves along the microwave, convective and vacuum drying period on a dry basis are presented in Figure 1 which shows a reduction in drying time occurred with the increase in microwave power level. The drying periods of mallow leaves from an initial moisture content of 5.02±0.003 db (92.5% wb) to a moisture content of 0.1±0.005 db (9% wb) basis were 6, 6.5, 8 and 10 min in microwave power densities of 11.33, 10, 8.67 and 6.67 W g\(^{-1}\), respectively. A marked decline in the drying period of the leaves was noted with increasing microwave power levels (Alibas, 2009; Soysal, 2004; Sarimeseli, 2011; Evin, 2012; Dadali et al., 2008; Demirhan & Ozbek, 2011; Al-Harahsheh et al., 2009). The drying time at the 6.67 W g\(^{-1}\) microwave power density was 1.67 times longer than that of 11.33 W g\(^{-1}\). The drying periods of the mallow leaves were 26, 40, 62 and 150 min at the temperature levels of 125, 100, 75 and 50 °C, respectively, applied during the convective drying procedure. The drying period shortened with an increase in temperature during the convective drying (Togrul & Pehlivan, 2003; Doymaz, 2008). The drying period at 125 °C was shortened by 5.77, 2.38 and 1.53 times compared with the drying processes conducted at 50, 75 and 100 °C, respectively. The drying time was reduced by 25, 10.33, 6.67, and 4.33 times when the leaves were dried at 50, 75, 100 and 125 °C temperatures compared with the drying treatment at 11.33 W g\(^{-1}\) of microwave power density. A marked decline was observed in the vacuum drying period of mallow leaves with the increasing temperature level and decreasing vacuum level. Drying time at 50 °C temperature was found as 115 and 130 min for 3 and 7 kPa, respectively, and at 75 °C, it was found as 38 and 53 min for 3 and 7 kPa vacuum values, respectively. Increase in temperature level in vacuum drying had an important effect on the reduction of drying time (Alibas, 2007, 2009). The extent of drying realised at 50 °C temperature and 7 kPa vacuum with the longest drying period was 3.42 times higher compared with the drying processes realised at 75 °C and 3 kPa, with the shortest drying period. When the drying process realised at 50 °C temperature without vacuum effect was compared with the drying processes at 50 °C temperature and with 3 and 7 kPa vacuum values, the drying period was shortened by 1.30 and 1.15 times, respectively, compared with the drying without vacuum effect. Similarly, when the drying applications realized at 75 °C with 3 and 7 kPa vacuum values were compared with drying process without vacuum at 75 °C, the drying period was reduced by 1.63 and 1.17 times, respectively under vacuum.

The drying rates (DR) [kg water. (kg dry matter\(^{-1}\).min\(^{-1}\)] obtained in unit time of microwave, convective and vacuum drying are shown in Figure 2. The average microwave drying rates of the mallow leaves at the microwave power densities of 11.33, 10, 8.67 and 6.67 W g\(^{-1}\) were 0.14, 0.12, 0.10 and 0.08 kg water. (kg dry matter\(^{-1}\).min\(^{-1}\)), respectively. The moisture content of the material was very high during the initial phase of the drying, which resulted in a higher absorption of microwave power and higher drying rates because of the higher moisture diffusion. As the drying progressed, the loss of moisture in the product caused a decrease in the absorption of microwave power and resulted in a fall of the drying rate (Alibas, 2009; Soysal, 2004; Sharma & Prasad, 2001). The average convective drying rates during the drying of mallow leaves at 50, 75, 100 and 125 °C were 0.009, 0.015, 0.029 and 0.044 kg water. (kg dry matter\(^{-1}\).min\(^{-1}\)), respectively. Average drying rates also increased with an increase in temperature because of the increase in the quantity of water lost from the material (Doymaz, 2008; Johnson et al., 1998). Depending on the drying conditions, average vacuum drying rates of mallow leaves ranged from 0.010 to 0.027 kg water. (kg dry matter\(^{-1}\).min\(^{-1}\)) between 50 °C temperature and 7 kPa vacuum values and 75 °C temperature and 3 kPa vacuum values, respectively. Increase in temperature for the same vacuum levels led to increment in the drying rate. Moreover, vacuum value had a notable effect on drying rate, as well. The drying rate increased with increasing vacuum rate for a given temperature (Alibas, 2007, 2009).

![Figure 1](image-url) The MD, CD and VD curve of mallow leaves on dry basis, respectively.
3.2 Modeling drying data

The parameters k and n of semi-empirical Page’s thin layer drying equation (Equation 1) for a given microwave, convective and vacuum drying condition were estimated using non-linear regression technique in Table 1 and the fitness is illustrated in Figure 3.

The model gave an excellent fit for all the experimental data points with values for the coefficient of determination of greater than 0.9985 in microwave drying at 6.67 W g⁻¹. It is determined that the value of the drying constants “k” increased with the increase in microwave power and the value of the drying constants “n” decreased with the increase in microwave power. This data points out that following the increase in microwave output power, drying curve becomes steeper, indicating faster drying of the product. As a result, measured moisture ratio values and predicted moisture ratio values were found similar to each other (Alibas, 2007). In convective drying, the model gave an excellent fit for all the experimental data points with values for the coefficient of determination of greater than 0.9993 (50 °C – 7 kPa).

Table 1. Non-linear regression analysis results of Page’s equation (Equation 1) for microwave, convective and vacuum drying of mallow leaves.

<table>
<thead>
<tr>
<th>Drying method</th>
<th>k, min⁻¹**</th>
<th>n**</th>
<th>SEE(±)</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>MD</td>
<td>11.33 W g⁻¹</td>
<td>0.1217</td>
<td>1.8482</td>
<td>0.02141</td>
</tr>
<tr>
<td></td>
<td>10 W g⁻¹</td>
<td>0.0809</td>
<td>1.9778</td>
<td>0.01668</td>
</tr>
<tr>
<td></td>
<td>8.67 W g⁻¹</td>
<td>0.0550</td>
<td>1.9894</td>
<td>0.01511</td>
</tr>
<tr>
<td></td>
<td>6.67 W g⁻¹</td>
<td>0.0328</td>
<td>2.0295</td>
<td>0.01456</td>
</tr>
<tr>
<td>CD</td>
<td>125 °C</td>
<td>0.0416</td>
<td>1.4206</td>
<td>0.01677</td>
</tr>
<tr>
<td></td>
<td>100 °C</td>
<td>0.0197</td>
<td>1.4334</td>
<td>0.00796</td>
</tr>
<tr>
<td></td>
<td>75 °C</td>
<td>0.0045</td>
<td>1.5883</td>
<td>0.01417</td>
</tr>
<tr>
<td></td>
<td>50 °C</td>
<td>0.0036</td>
<td>1.4125</td>
<td>0.00785</td>
</tr>
<tr>
<td>VD</td>
<td>75 °C-3 kPa</td>
<td>0.0244</td>
<td>1.3163</td>
<td>0.02898</td>
</tr>
<tr>
<td></td>
<td>75 °C-7 kPa</td>
<td>0.0149</td>
<td>1.3941</td>
<td>0.01354</td>
</tr>
<tr>
<td></td>
<td>50 °C-3 kPa</td>
<td>0.0142</td>
<td>1.1593</td>
<td>0.01192</td>
</tr>
<tr>
<td></td>
<td>50 °C-7 kPa</td>
<td>0.0068</td>
<td>1.2838</td>
<td>0.00796</td>
</tr>
</tbody>
</table>

**Means with same letter do not show significance at P<0.01; SEE (±), standard error of estimate; R², coefficients of determination; k, drying rate constant, min⁻¹; n, drying exponent.

3.3 Color parameters and ascorbic acid content

The color parameters (L, a, b) formed in microwave, convective and vacuum drying methods of mallow leaves are compared in Table 2. Among all the drying trials, the color criteria (L, a, b) most similar to those of fresh leaves occurred at the 10 Wg⁻¹ in microwave drying (Li et al., 2010; Soysal, 2004; Piotrowski et al., 2004), while the color criteria (L, a, b) least similar to those of fresh leaves was noted at the 125 °C (Alibas, 2007) temperature level of the convective drying method. Similar findings were found by several researchers (Alibas, 2007, 2009). Santo et al. (2013) said that color is an important attribute in food acceptance.

The ascorbic acid levels in mallow leaves dried by microwave, convective and vacuum were compared with the levels in a fresh sample; the results, which are presented in Table 2. When the three drying methods were compared with respect to energy consumption values, it was noted that the lowest energy consumption occurred in microwave drying method and this was followed by convective- and vacuum-drying methods. The best result with regard to ascorbic acid was obtained from 10 W g⁻¹ microwave power density among all drying methods. The worst value in all drying methods regarding ascorbic acid was noted in convective drying at 125 °C.

3.4 Energy consumption

The energy consumption values obtained during microwave, convective and vacuum drying of mallow leaves are given Table 2. When the three drying methods were compared with respect to energy consumption values, it was noted that the lowest energy consumption occurred in microwave drying method and this was followed by convective- and vacuum-drying methods. The best result with regard to energy...
consumption was obtained from 11.33 W g⁻¹ microwave power density among all drying methods. Energy consumption at this level was 0.10 kWh. The highest value in all drying methods regarding energy consumption was noted in vacuum drying process consisting of 50 °C temperature and 7 kPa vacuuming rate, with 1.44 kWh. There was a 14.4-fold difference between the highest (50 °C-7 kPa) and the lowest (11.33 W g⁻¹) energy consumption values. Similar findings were found by several researchers (Alibas, 2007, 2009).

### 3.5 Estimation of effective moisture diffusivity and activation energy

The slope and effective moisture diffusivity of mallow leaves was described using the moisture ratios. Non-linear regression technique was used to estimate slope (S) and effective moisture diffusivity (Dₑ) of Fick's diffusion equation Equation 6 and 5, respectively. Depending on the microwave drying conditions, effective moisture diffusivities of mallow leaves ranged from 2.04403 × 10⁻¹⁰ to 3.63996 × 10⁻¹² m² s⁻¹ for the microwave power densities between 6.67 and 11.33 W g⁻¹, respectively. According to convective drying data, effective moisture diffusivities of mallow leaves ranged from 1.70182 × 10⁻¹⁰ to 5.94559 × 10⁻¹² m² s⁻¹ for drying temperatures between 50 and 125 °C, respectively. The effective moisture diffusivities of vacuum dried mallow leaves ranged from 1.85599 × 10⁻¹¹ to 3.63996 × 10⁻¹² m² s⁻¹ for vacuum and temperature effects between 50 °C-7 kPa and 75 °C-3 kPa, respectively. The effective moisture diffusivities and slopes for microwave, convective and vacuum drying conditions were presented in Table 3. Effective moisture diffusivities reduced with increase of drying time; therefore, the biggest effective moisture diffusivities were found in microwave drying which is the much shorter drying method than convective and vacuum drying. Similar findings were found by several researchers (Sarimeseli, 2011; Evin, 2012; Dadali et al., 2008; Demirhan & Ozbek, 2011).


