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Research Article

Effect of Various Drying Methods on Quality and Flavor Characteristics of Mint Leaves (*Mentha spicata* L.)

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ABSTRACT

Mint leaves were dehydrated by hot air, shade as well as microwave drying and the respective drying time found, to lower the moisture content from (88%) to around (5%). The qualities of the dried products were assessed by determining the moisture, chlorophyll, carotenoid, polyphenols, color and volatile oil. In microwave drying, despite the less drying time, there were appreciable losses of volatile oil, chlorophyll and other components when compared to the fresh mint leaves. Results showed that Hot Air Drying (HAD) at 45°C followed by Micro Wave Drying (MWD) at 900 Watts possessed better quality parameters in the dried products suggesting that Hot Air Drying (HAD) was most suitable although it took more time compared to Micro Wave Drying (MWD).

Keywords: drying, dehydration, mint leaves

1. Introduction

Mint (*Mentha spicata* L.) is a common name for members of the Labiatae (Laminaceae Family). It is a large family of annual or perennial herbs and widely grown all over the world to reap its special herbal characteristics. The essential oils of mints are widely used as flavourings in food, cosmetic and pharmaceutical industries. Green leaves of the plant are used for flavouring culinary preparations, vinegar, jellies and iced drinks. A soothing tea is also brewed from the leaves (Wealth of India, 1962). Mint leaves are known for refreshing, antiseptic, antiasthmatic, stimulative, diaphoretic, stomachic, and antispasmodic features. They are used in both fresh and dried forms in different cuisines. Various authors (Park et al., 2002; Columbia Electronic Encyclopedia, 2005; Thompson, 2003) have indicated the use of mint leaves in variety of dishes such as vegetable curries, chutney, fruit salads, vegetable salads, salad dressings, soups, desserts, juices, sherbets etc. An acceptable instant mint chutney powder was prepared by using shade dried leaves (Satyanarayana et al., 2001). Mint is very popular in India and mainly cultivated in southern parts of Himalayan range including Punjab, Himachal Pradesh, Haryana, Uttar Pradesh and Bihar.Essential mint oil is extracted either from freshly harvested mint leaves or from semidried or dried leaves through distillation process for industrial applications. The oils contain dozens to hundreds of compounds. Such essential oils are used as natural aromas in food and toiletry products and due to their medicinal properties many are used in conventional medicines and aromatherapy. The compounds of essential oils are largely monoterpene and sesquiterpene hydrocarbons and their oxygenated derivatives as well as phenylpropanoids (Adamiec & Kalemba, 2006). The chemical composition of the oils in mint has been studied by different researchers. Carvone is the major component in all cases and is the character impact component in mint followed by limonene.

Dry herbs have a great importance, not only for the culinary purposes, but also for medicinal uses (Hedrick, 1972). The aim of drying is to reduce the moisture content of the product from actively growing in the field to a level that prevents deterioration of the product and allows storage in a stable condition. The drying of mint is an effective method that increases the shelf life of the final product. However, drying cause's changes in the product mainly associated with fragrance and appearance (Consuelo et al., 2003). Drying of the plant material can be achieved by several processes

including hot-air and freeze drying. Although freeze drying can be used to avoid damage caused by heat, producing a product with superior physical and chemical qualities is considered a costly and time consuming process (Ratti, 2001).

Volatile aroma compounds are the most sensitive components in the process of drying. The effect of drying on the component of essential oil of various aromatic plants, fruits and vegetables has been the subject of numerous studies, which show that the changes in the concentration of the volatile compounds during drying depends on several factors, such as drying method and drying conditions (temperature, air, velocity, relative humidity) (Venskutonis, 1996; Yousif et al., 2000; Kaya & Aydin, 2009; De Torres et al., 2010). The effect of a particular drying method on the release or retention of volatile compounds is not predictable and depends on the compound and the spice concerned. Oven-drying and freeze-drying of dill lead to decrease in most of the volatile compounds compared with the levels in the fresh spice (Huopalahti et al., 1985; Raghavan et al., 1994). The same occurs in parsley (Consuelo et al., 2003). In contrast, the effect of oven drying at 30 ℃ and freeze-drying on the volatile compounds in thyme and sage has been minor, where as losses at 60°C were 43% in thyme and 31% in sage (Venskutonis, 1996). Recently, many studies have been conducted on the drying behavior of different aromatic plants.

The effect of air temperature on the retention of principal volatile compounds in lemon myrtle when subjected to fluidized bed drying was studied (Buchaillot et al., 2009).A similar study was done on betel leaves dried in hot air (Pin et al., 2009). Studies were done on the effect of freeze-drying time on the concentration levels of the two main components of fennel essential oil (Gardeli et al., 2010). Changes in the concentrations of the volatile compounds of mint during drying also depend on several factors, such as drying conditions (temperature, air velocity), moisture content, variety and age of plant, climate, soil, and harvesting method (Asekun et al., 2007; Tarhan et al., 2010; Braga et al., 2009; Rohloff et al., 2005).

The present study has examined the influence of different drying methods like Hot air drying, Microwave drying and Shade drying on the quality, volatile compounds and antioxidant activity in mint leaves.

2. Materials and Methods

2.1. Plant Material

Mint leaves was procured from local market and cleaned by removing undesired stems and waste materials. The leaves were washed and the excess water was removed with the help of blotting paper. The damaged and black leaves were separated manually before subjecting to various drying methods.

2.2. Chemical Reagents

Chemicals and solvents used in the study were of analytical and laboratory grade and were procured from SD Fine-Chem Ltd. (Mumbai, India).

2.3. Mineral Estimation

Fresh mint leaves (5 g) were weighed in a silicacrucible and ignited. The sample was further kept in a preheated muffle furnace set at 550 ℃ for 6 h. The obtained white ash was weighed and dissolved in 10% HCl and placed on water bath, and transferred through a filter paper into a 100 ml volumetric flask and the volume was made up using distilled water. The ash solution was used for the determination of Fe, Cu, Mn, Na, K, Mg, and Zn in triplicates using Atomic Absorption Spectrophotometer (AOAC, 1999) (Varian Model AA_{220} Australia). The concentration of minerals was expressed as mg/100g.

2.4. Drying Treatments

The destalked mint leaves were subjected to various drying processes, such as Microwave, Hot air drying and Shade drying.

2.4.1. Hot Air Drying (HAD)

Hot air drying was carried out for mint leaves in a cross flow tray drier at 45, 55 and 65 °C. The dryer was switched on for 30 min prior to each experimental run to attain required temperature. After attaining the desired temperature, samples were loaded onto the tray in a single layer. The trays were removed from the dryer and weighed regularly at intervals of 30 min until a constant weight was attained.

2.4.2. Microwave Drying (MWD)

Microwave drying was carried out in a domestic microwave oven (Samsung, India) at two different power levels (180W & 900W). The oven was fitted with a rotatable circular glass plate. The microwave oven had the capability of operating at five different microwave output power levels: 180, 300, 450, 720 and 900W. The fresh leaf material was uniformly spread on a microwave safe tray, for even absorption of microwave energy. Moisture loss was recorded at 30 sec intervals drying at the end of power-on time by removing the turn-table from the microwave, and periodically placing the leaf sample, on the digital balance (Soysal et al., 2006) and the data analyzed was an average of these results. All weighing processes were completed in less than 10 sec during the drying process. The microwave power was applied until the mass of the sample attained a constant weight.

2.4.3. Shade Drying

Fresh leaves were spread uniformly on a floor area inside a room for 72 h (30 ± 2 ℃).

2.5. Analysis of Fresh and Dehydrated Mint Leaves

The fresh mint leaf samples were analyzed for the contents of moisture, chlorophyll and color as per the standard methods (Ranganna, 2010).The mint leaves obtained after various drying processes were coarsely ground using a domestic mixer grinder. The dried powders were analyzed for quality parameters like moisture, color value, chlorophyll, carotenoids, DPPH activity, total polyphenols, and volatile oils. Volatile

oilcontent in fresh and dried leaves were extracted by Clevenger hydro distillation method.

2.5.1. Color Measurement

Mint powder was subjected to color measurement (Hunt, 1991).The change of color was measured and compared using Hunter Colorimeter (Hunter Associates Laboratory, USA). Of the three color coordinates, namely L*, a* and b*, "L*" represents the lightness index, "a*" represents red-green, while ''b*'' represents yellow-blue color components. The measurement of L*, a*& b* values of color was replicated three times and the average values were reported.

2.5.2. Chlorophyll Estimation

Estimation of chlorophyll in fresh and dehydrated mint leaves was carried out according to the procedure of (Ranganna, 2010).The sample (0.5-1.0 g) was macerated with acetone in a pestle and mortar. The supernatant layer was decanted and the extraction was repeated until the residue was colorless. The extracts were then pooled, filtered and made up to 100 ml in a volumetric flask. About 25-50 ml aliquot of the acetone extract was taken into a separating funnel and mixed with 50 ml diethyl ether and water was added untilthe water layer was apparently free of all the fat-soluble pigments. The water layer was drained off and the ether layer washed with 25 ml portions of distilled water until the layer was free of acetone. The ether layer was taken into a 50 ml volumetric flask. 3-4 g of anhydrous sodium sulfate was added to remove the moisture.The absorbance was taken at wavelengths of 660 and 642.5 nm.

Total chlorophyll = 7.12 x O.D at 660nm + 16.8 x O.D at 642.5nm; Chlorophyll "a"= 9.93 x O.D. at 660nm - 0.777 x O.D at 642.5nm; Chlorophyll ''b"= 17.60 x O.D. at 642.5 nm - 2.81 x O.D at 660 nm

The above equations provide chlorophyll content in mg/l in the solution used for recording absorbance. The chlorophyll content in the mint samples was calculated taking the dilution factor into consideration, and the results were expressed as mg/100 g on dry basis.

2.5.3. Extraction of Carotenoids

The estimation of total carotenoids was done after extraction of the sample (1 g) with acetone and further purification with petroleum ether and distilled water. The resulting solution was filtered with anhydrous sodium sulphate and read on a spectrophotometer at 452 nm against petroleum ether as a blank.

2.5.4. Determination of Total Phenols and Free Radical Scavenging Activity of Mint Leaf Extract

0.2 g of mint powder was soaked in 50 ml of 80% ethanol for 1 h and allowed for agitation using magnetic stirrer. The total phenol content in the dried mint powder was determined using Folin-Ciocalteu method (Sadasivam & Manickam, 1997). The ethanol extract (0.5 ml), Folin-Ciocalteu (0.5 ml) reagent and distilled water (8 ml) were added. The contents were vortexed for 2 min and allowed to stand at RT for 1 h. The intensity of the color developed was read at 675 nm and total phenolic content was calculated and expressed as gallic acid equivalents, g/100 g.

DPPH (2, 2-diphenyl-1-picrylhydrzyl) radical scavenging activity of extracts of mint leaf powder (1 g/100 ml) was measured by using aliquots of 0.1, 0.2, 0.3, 0.4 and 0.5 ml. Methanolic solution of DPPH (0.004%) (4ml) was added and vortexed (Remi, Mumbai, India) for 30 sec. The contents were incubated at room temperature (RT) 30 ± 2 ℃ for 30 min. The decrease in color intensity during incubation was measured in terms of optical density at 517 nm. A control sample was prepared as above without extract, and methanol was used for the baseline correction. All analyses were run in triplicate and the values averaged. Radical scavenging activity was expressed as the percentage inhibition and was calculated using the following formula:

Radical scavenging activity (%) = (Control OD –Sample OD/Control OD) x 100

2.5.5. Extraction of Essential Oil and Determination of Volatile Oil Yield in Mint Leaves

Around 10 g each of mint powderby different drying methods was homogenized with distilled water (500 ml). Theslurry was subjected to Clevenger hydrodistillationmethod. Distillation was carried out until the maximumpossible quantity of oil was obtained (4–5 h). The volatileoil yield was determined on a dry weight basis. The paleyellow oils collected were dried over anhydrous sodium sulphate and stored at 4 ℃ prior to analysis.

2.5.6. Gas Chromatography (GC) Analysis

The flavor components of the volatile oil were analyzed on a Varian CP-3800 model gas chromatograph with Galaxy software system equipped with flame ionization detector (FID) and an electronic integrator. Separation of the compounds was achieved employing a Varian CP-Sil 5CB capillary column (50 m X 0.25 mm ID; film thickness 0.25µm). The operating conditions of the instrument were as follows

Nitrogen was used as the carrier gas at a constant flow rate of 0.4 ml/min. The column temperature was programmed from 100℃ (held for 2 min.) to 240 ℃ (held for 8 min) at 8 ℃/min ramp rate. The injector and detector temperature were set at 250 ℃ and 300 ℃ respectively. Samples of 0.2 µl were injected with a 20:100:20 split ratio. Retention indices were generated with a standard solution of n-alkanes (C_6-C_{19}). The composition was reported as a relative percentage of the total peak area without FID response factor correction.

2.5.7. Chemical Compounds Identification

The identification of the essential oil constituents was based on a comparison of their retention indices relative to homologous series of n-alkanes (C_6-C_{19}; Poly Science; Niles, USA)

3. Results and Discussion

The data pertaining to Minerals estimation of mint leaves are presented in (Table 1). The data indicated that mint leaves are a rich source of Potassium and a fairly good source of Magnesium. Fresh mint leaves which were having a moisture content of about 88.5% were dried by Hot air drying (HAD), Microwave Drying (MWD) and Shade drying (SD). In HAD, drying time reduced with increase in temperature. MW drying was faster followed by HAD while SD took more time (Table 2). The wide variation in drying time could be explained by the individual drying principle lying behind each method. The drying time requirement of microwave drying was less in comparison to hot air dryingand it is because of the obvious reasons of high rate of mass transfer at the higher temperature generated due to electromagnetic field. Determination of chlorophyll, volatile oil content and volatile oil composition of mint leaves is not only important with respect to the product quality after drying but also for better preservation.

Table 1. Mineral content of fresh mint leaves

Elements	mg/100g
Iron	2.9012 ± 0.030
Zinc	0.4936 ± 0.067
Copper	0.1474 ± 0.033
Manganese	0.2330 ± 0.025
Sodium	5.888 ± 0.077
Potassium	242.32 ± 0.407
Magnesium	25.098 ± 0.020

#average of triplicate analysis

3.1. Color

The mean surface color values obtained L* (Lightness), a* (+redness, -greenness), b* (+yellowness, -blueness) of leaves dried at different temperature are results of color parameters obtained (Table 3). Results indicated significant reduction in L*, a*, b* values in the dried leaves in comparison to fresh ones. It is noticed that with increase in temperature, the color of mint leaves became darker implying more browning of the leaves. However, all three different drying methods yielded negative 'a*' values, indicating retention of green color to some extent. Green color was better retained in HAD at 45 ?C followed by MW at 900W.

3.2. Chlorophyll and ?-Carotene Content

The chlorophyll content of the fresh and dried mint leaf samples by spectrophotometric method are recorded (Table 3). The results reveal that chlorophyll content was better retained in HAD at 45 ?C in comparison with all drying methods (Rudra et al., 2008) reported that high temperature could lead to the replacement of magnesium in the chlorophyll by hydrogen, thereby converting chlorophylls to pheophytins. When the leaves were dried by MWD with lower energy input (180W), the loss was found to be higher in comparison to 900W. This might be due to the shorter time of exposure of leaves to the microwave energy in MWD at 900W. Similar results were obtained during drying of Betel leaf by MWD (Ramalakshmi et al., 2002). Also SD samples showed higher loss of pigments due to longer drying time. This might be due to the sensitivity of chlorophyll towards heat. Retention of chlorophyll could be improved by blanching as reported in rosemary and marjoram (Meenakshi Singh et al., 1996).

In the present study, decrease in carotenoid content was observed in all methods of drying (Table 3). It was seen that dehydration at higher temperatures of 55 °C and 65 °C led to greater destruction of carotenoid. Also the degradation of carotenoids with increasing the exposure time led to decrease in yellowness. Nevertheless, appreciable amount of carotenoid still remained in all the samples. Chlorophyll and carotenoids was better retained in HAD at 45 °C than other drying methods indicating that HAD been more suitable for drying of mint.

3.3. Total Polyphenols

Evaluation of total phenols in methanolic extracts of mint leaves dried by different methods as estimated by the method of Folin-Ciocalteu revealed that mint leaves exhibited high and variable contents (Table 3). The highest total phenolic content (TPC) was recorded in leaves dried by HAD at 45 ?C followed by shade drying. The microwave dried samples recorded less phenolic content. Similarly, the microwave drying of some green leafy vegetables recorded lower phenolic content (Sahar Kamel et al., 2013).

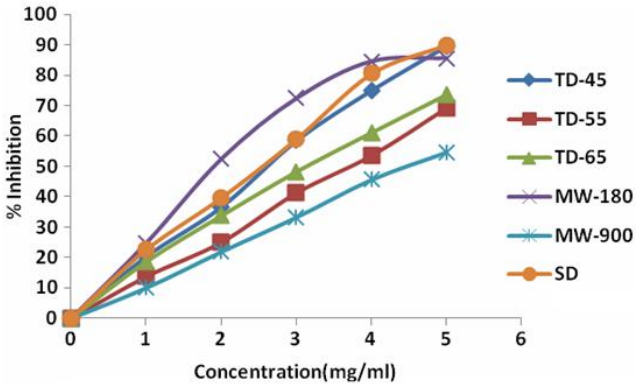

Fig. 1. Antioxidant activity of ethanolic extract of fresh and dried mint leaves

3.4. Radical Scavenging Activity (RSA)

The DPPH antioxidant assay is based on the ability of 1-1-diphenyl-2-picrylhydrazyl, a stable free radical to decolorize in the presence of antioxidants. All the extracts from different dryings of mint leaves showed DPPH radical scavenging activity (Fig 1). Among the extracts analyzed the highest DPPH scavenging activity corresponding to the lowest IC_{50} values was found in the extracts of microwave-dried mint leaves at 180W (1.9 mg/ml). Mint leaves dried by HAD at 45 ?C and SD ranked second in the order of DPPH scavenging activity with an IC_{50} of 2.6 mg/ml (Table 3).

Table 2. Volatile oil yield in fresh and dried mint leaves

Drying Method	Time Required	Volatile oil (%)	Moisture (%)
Fresh	-	3.13 ± 0.014	88.5 ± 1.003
HAD (45 °C)	3.5 h	3.16 ± 0.029	5.165 ± 0.046
HAD (55 °C)	2 h	2.10 ± 0.022	4.805 ± 0.015
HAD (65 °C)	1.5 h	2.10 ± 0.016	5.081 ± 0.019
MWD 180W	45 min	1.04 ± 0.022	4.60 ± 0.019
MWD 900W	9 min	0.95 ± 0.028	5.435 ± 0.028
SD	72 h	1.89 ± 0.022	4.955 ± 0.034

#average of triplicate analysis ± SD

Table 3. Chlorophyll, carotene, total phenols and RSA in fresh and dried mint leaves

Drying Method	Colour	Total Chlorophyll, mg/100g	Carotenoids, mg/100g	Total phenols, mg/100g	RSA IC_{50} value, mg/ml
Fresh	**L*:** 45.56 ± 0.304 **a*:** -13.79 ± 0.080 **b*:** +24.80 ± 0.008	8464 ± 2.944	2095.6 ± 0.374	4165 ± 2.160	-
HAD (45 °C)	**L*:** 41.71 ± 0.107 **a*:** -12.28 ± 0.083 **b*:** +22.79 ± 0.051	1908 ± 1.414	305.79 ± 0.225	7188 ± 2.160	2.6 ± 0.008
HAD (55 °C)	**L*:** 40.63 ± 0.258 **a*:** -8.12 ± 0.042 **b*:** +15.24 ± 0.028	430 ± 1.414	189.08 ± 0.045	5886 ± 2.944	3.6 ± 0.016
HAD (65 °C)	**L*:** 37.24 ± 0.122 **a*:** -5.32 ± 0.022 **b*:** +14.80 ± 0.008	550 ± 2.160	442.48 ±0.203	4066 ± 1.414	3.1 ± 0.008
MWD 180W	**L*:** 40.81 ± 0.367 **a*:** -5.12 ± 0.054 **b*:** +15.93 ± 0.029	67 ± 1.414	68.134 ± 0.005	1104 ±2.828	1.9 ± 0.014
MWD 900W	**L*:** 44.74 ± 0.231 **a*:** -13.12 ± 0.036 **b*:** +21.40 ± 0.014	163 ± 0.816	493.84 ± 0.064	4004 ± 2.944	4.4 ± 0.022
SD	**L*:** 40.37 ± 0.151 **a*:** -1.37 ± 0.059 **b*:** +18.71 ± 0.024	164 ± 2.828	85.22 ± 0.022	6657 ± 1.414	2.6 ± 0.045

#average of triplicate analysis ± SD

Table 4. Percentage of volatile oil components in fresh and dried mint leaves

S.No	Component	Retention time (min)	Fresh (db)	HAD (45°C)	HAD (55°C)	HAD (65°C)	MWD 180W	MWD 900W	SD
1.	Limonene	11.43	14.21	7.37	23.57	23.14	9.09	15.28	13.08
2.	Menthone	13.59	0.17	0.28	0.14	0.14	0.24	0.26	0.17
3.	Isomenthone	13.90	0.45	0.60	0.45	0.49	0.40	0.52	0.69
4.	Menthol	14.25	0.99	1.04	1.35	1.29	1.02	1.28	1.53
5.	Carvone	14.63	67.62	66.83	57.08	55.36	31.82	40.42	66.84
	Total		83.44	76.12	82.59	80.42	42.57	57.76	82.31

#average of duplicate analysis

Results showed that the less activity was found in leaves dried at MW 900W, which corresponds to the highest IC_{50} value (4.4mg/ml).

3.5. Volatile Compounds Identified By GC

The volatile oil yield extracted from fresh and dried mint leaf samples by Clevenger hydro distillation are presented (Table 2). It is observed that the recovery was higher in HAD at 45 ?C samples compared to other methods of drying. Carvone was the most important and major compound identified in mint leaves followed by limonene. The highest losses in volatiles occurred in microwave dried samples. It was observed that the extraction of volatile

oil was maximum in HAD samples followed by SD samples. However, SD took a longer time (72 h) and also the drying conditions are difficult to control. Microwave drying is a quicker method of dehydration of mint leaves. However, higher loss was observed in volatile oil content, but retention of green color was good. Similarly, Microwave drying produced greater losses in volatile compounds than oven drying in rosemary although it did preserve the spice's characteristic green color (Jaganmohan Rao et al., 1998). Leaves dried at HAD 45 ⍰C showed better qualities pertaining to volatile oil (3.16%) and retention of carvone content (66.80%) when compared to other drying methods (Table 4). Chromatogram obtained for fresh mint oil is shown in (Fig 2).

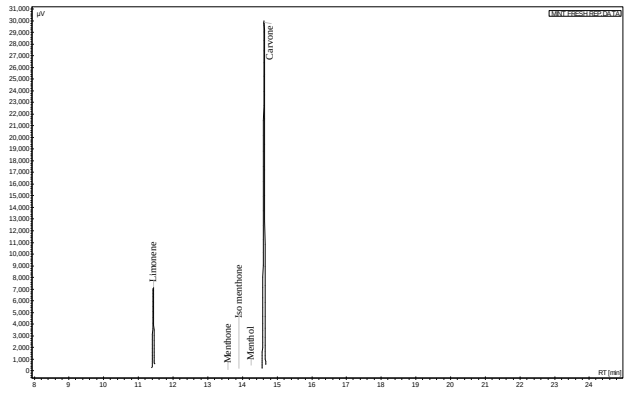

Fig. 2. Chromatogram of Mint fresh oil

4. Conclusions

Although there was loss in the quality characteristics during HAD, a considerable amount is still preserved in samples dried at 45 ⍰C. Therefore, 45 ⍰C may be the optimum temperature for hot air drying of mint leaves. Similarly, it was observed that oven drying of spearmint at 45 ⍰C and air drying at ambient temperature was the methods that produced the best results (Consuelo et al., 2003). There was a substantial reduction in drying time in MWD as compared to that of HAD and SD. But the leaves lost most of the components for which it is valued. Based on the quality characteristics, volatile oil, chlorophyll and carotenoid retention, HAD at 45 ⍰C appears to afford superior product compared to MWD and SD. This study is very much useful to prepare dehydrated mint powder which can be used in the preparation of spice powder to sprinkle on different type of snack foods.

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