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The Pharma Innovation

ISSN (E): 2277- 7695
ISSN (P): 2349-8242
NAAS Rating: 5.03
TPI 2020; 9(7): 568-573

www.thepharmajournal.com
Received: 12-05-2020
Accepted: 16-06-2020

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Effect of different drying treatment on composition, nutritional and phytochemical content of mint leaves

Salve RV, Syed HM, More SG and Shinde EM

Abstract
The present work was proposed to development technology for drying of mint leaves *(Mentha spicata*) and carried out by using sun drying (30-35^{0}C for 8 Hr.), shade drying (27-32^{0}C for 15 days) and cabinet oven drying (60^{0}C for 2 hr.). The obtained result was evaluated of dried mint powder with respect to chemical composition, nutritional and phytochemical properties. The moisture was reduced from 82.60% to 4.20% whereas fat, protein, carbohydrate, crude fiber and ash increased from 0.60% to 2.95%, 4.75% to 13.90%, 8.95% to 53.88%, 5.70% to 17.60% and 2.10% to 9.70% respectively. Related to mineral content like calcium, potassium, iron, magnesium, zinc and phosphorus raised from 175.10 to 192.30 mg/100g, 481.63 to 503.30 mg/100g, 8.30 to 10.15 mg/100g, 88.54 to 96.20 mg/100g, 11.15 to 13.10 mg/100g and 56.72 to 69.09 mg/100g respectively. The variation in the concentration of such compounds as alkaloids, flavonoids, total phenol and β-Carotene and found more concentration in shade dried mint powder TM2 had values 12.28 g/100gm, 9.57 g/100gm, 0.79 µg/g and 5.74 g/100gm respectively. Finally energy consumption and time taken more in shade drying method but end-dried product is superior in all respect compared to other dried products.

Keywords: Drying methods, mint powder, composition, nutritional and phytochemical component

Introduction
Mint community (*Mentha spicata* L) is regarded as one of the most significant and valuable source of essential oil (Guenther 1995). Spearmint is cultivated in various parts of the world, such as in North America, England, Germany, the Netherlands and the Mediterranean region. Minor spearmint cultivation areas exist in China and India, where oils of high quality are produced (Court *et al*., 1993) [5]. The mint plants and their items are widely used as spice, flavouring and in traditional medicine. The oil is used in pharmaceutical, anti-septic, perfumery and food industries (Salim 1997) [19].

Mint is very famous in India and cultured mainly in southern parts of the himalayan region including Punjab, Himachal Pradesh, Haryana, Uttar Pradesh and Bihar. The essential mint oil was either extracted from freshly harvested mint leaves or from semi-trimmed or dried leaves through the process of distillation for industrial applications. The oils contain dozens to hundreds of compounds. Such essential oils are used as natural aromas in food and personal hygiene products products and due to their medicinal properties, many are used in traditional treatments and aromatherapy. Mainly monoterpene and sesquiterpene hydrocarbons and their oxygenated derivatives as well as phenylpropanoids are the compounds of essential oils (Adamiec & Kalemba, 2006) [3]. Various researchers have studied the chemical composition of the oils in mint. In all cases, carvone is the key component and the character-impact factor in mint accompanied by limonene.

A member of the mint family (Lamiaceae), *Mentha spicata*, is a herbaceous, perennial rhizomatous plant with smooth stems rising to 30–90 cm. The rhizomes are small, fleshy and have bare fibrous roots. The leaves are 4–9 cm long and 1.5–4 cm thick, dark green with reddish veins and strongly dented margins with an acute apex. Usually the leaves and stems are lightly hairy. The flowers are white, 6–8 mm long and about 5 mm in diameter, with a four-lobed corolla. They are generated around the stem in whorls which form thick, blunt spikes. Peppermint has a high concentration of menthol and is often used for ice cream, confectionery, chewing gum, tooth paste flavouring and tea. The oil also contains the menthone and esters of menthyl, particularly methyl acetate. One animal study suggested that peppermint can have radio-protective effects in cancer-treated patients (Baliga and Rao, 2008) [4]. The peppermint aroma has been discovered to enhance memory (Moss *et al*., 2008) [16].

Therefore, the work deals with the application of different drying methods, quantitatively analysis of proximate composition, mineral composition and phytochemicals content in dried mint leaves powder

Material and Methods

Materials: The raw material fresh mint leaves were obtained from the market area of Parbhani.

Chemicals and Glassware: Chemicals of analytical grade and sufficient glassware required were available in the laboratory, Department of Food Chemistry and Nutrition, College of Food Technology. V.N.M.K.V. Parbhani.

Analytical Equipment's: Electronic weighing balance, soxhlet extraction apparatus, steam distillation unit, hot air oven, incubator, spectrophotometer, Gas chromatography-mass spectrometry, Baking oven and muffle furnace available from the Department of Food Chemistry and Nutrition, College of Food Technology, VNMKV, Parbhani

Method of Drying and preparation of powder

Sun shade drying: Conventional sun drying techniques have been used. Separate the leaves from the stalks and wash them with running water. Washed leaves then spread over the floor and held those leaves at 30-35^{0}C temperature for 8 hours for sun drying. Once the dried leaves are collected, the dried leaves are grinded in grinder to make the fine powder, weighed and packed into a polyethylene container. Such powders were used for trials (Sathiya *et al*., 2015) [15].

Shade drying: Conventional shade-drying methods have been used. Separate the leaves from stalks and wash with running water underneath. Washed leaves then spread over the floor and kept those leaves at a temperature of 27-32^{0}C for 15 days for shade drying. Once the dried leaves are collected, the dried leaves are grinded in grinder to make the fine powder, weighed and packed into a polyethylene container. Such powders were used for trials (Sathiya *et al*., 2015) [15].

Cabinet oven drying: Segregate the leaves from stalks and wash with running water underneath. Washed leaves then spread over the tray and put the tray in the oven. Drying was performed two hrs at 60^{0}C. To achieve the required temperature, the dryer was switched on for 30 min before each run. Once the desired temperature was achieved, samples were placed into a layer onto the tray. The trays have been removed from the dryer, the dried leaves have been collected, finely grinded, weighed and packed into a polyethylene bag. These powders were used for trials (Sathiya *et al*., 2015) [15].

Different drying treatments given to mint leaves

Mint leaves given various drying treatment like sun, shade and cabinet oven drying and converted dried leaves into fine powder. The treatments and dried samples was mentioned below.

Table 1: Drying treatments given to mint leaves

Sr. No.	Treatment	Sample
1.	TM0	Fresh mint leaves
2.	TM1	Sun dried mint leaves powder
3.	TM2	Shade dried mint leaves powder
4.	TM3	Cabinet oven dried mint leaves powder

Proximate composition of dried mint leaves powder

Fresh mint leaves and its prepared powder were analyzed for proximate composition including moisture, fat, protein, total carbohydrate, crude fibre, ash and mineral composition was carried out as per the methods given by (AOAC, 2005).

Quantitative determination of phytochemicals in prepared powder

Total alkaloid: Five grams of the sample were weighed in a 250 ml beaker and 200 ml of 10 percent ethanol acetic acid was added and allowed to stand for 4 minutes, filtered and diluted to one quarter of the original volume in a water bath. Concentrated ammonium hydroxide added drop wise to the extract every time the precipitation was over. The entire solution was allowed to settle, and dilute ammonium hydroxide was used to absorb, wash, and filter the precipitate. Dried and weighed the residue, was alkaloid (Harbone, 1973) [9].

$$\text{Alkaloid (\%)} = \frac{W3 - W2}{W1} \text{ x } 100$$

Where,
W1 = Initial weight of sample, W2 = Weight of the extract and W3 = Final weight of the residue

Total flavonoid content: The total flavonoid content of cold and hot powder extracts was determined by a slightly changed method stated by Meda *et al.*, (2005) [14]. A 0.5 ml of sufficiently diluted sample was combined with 0.5 ml methanol, 50 µl 10% $AlCl_3$, 50 µl 1 mol L^{-1} potassium acetate and 1.4 ml water, and allowed to incubate at room temperature for 30 minutes. Afterwards the absorbance of the reaction mixture was measured at 415 nm. The total flavonoid was determined using quercetin formula as standard.

$$TFC = \frac{A \text{ x } DF}{A1\%\ 1cm \text{ x } (w - ld)}$$

Where, A = Absorbance, DF = Dilution Factor, A1% 1cm = Specific absorption by $AlCl_3$
w = Mass of plant material, ld = Loss on drying

Total phenolic content: The total phenolic content (TPC) of the powder extracts was measured using the process of Folin – Ciocalteu (Pinelo *et al.*, 2005) [17]. Next to 5 ml Folin - Ciocalteu reagent was added into 1 ml sample tube. Then introduced to the mixture of 4 ml of 7.5 per cent (w/v) of sodium carbonate. The absorbance was read at 765 nm against blank after 60 min of room temperature incubation (32±1^{0}C) The results were presented as an equivalent mg of gallic acid per gram of dry weight basis for the fresh sample (mg GAE / g dw base). The overall phenolic content of all the samples was estimated using the formula,
Total phenolic contents (TPC) from the powder extracts were quantified using

$$C = c\ V/m$$

Where
C = total phenolic content mg GAE/g dry extract, c = concentration of Gallic acid obtained from calibration curve in mg/mL, V = volume of extract in ml, m = mass of extract in gram.

β- Carotene: Carotene was extracted from vegetables using the Khalil and Varananis (1996) [12] method for "Reversed phased HPLC system." 10 g of the sample was homogenized to 30 ml of acetone, and 0.1% BHT solution in the acetone solution was used as an antioxidant. The resulting extract has been pumped through Buchner funnel. The residue was treated with acetone twice, until it was colourless. The excess was discarded and 20gm of anhydrous sodium sulphate was added to the filtrate. The anhydrous sodium sulfate was extracted by filtration, and rotatory evaporator reduced the amount of extract. The extract was quantitatively transferred to 100ml volumetric flask, and the amount was made with acetone and water up to the limit, so that the final extract contains 80 percent of acetone.

Standard Preparation of Beta Carotene Standard of beta carotene

1 g wrapped in a vial received from merck. Stock beta-carotene solution was prepared by taking 10 mg in 100ml n-hexane. The stock solution concentration was = 100 ppm. The stock solution was diluted to different known concentrations e.g. 20, 40 and 60 ppm, dilutions in 5 ml of each n-hexane solution were obtained. Each working standard solution was injected into the HPLC system and chromatographic condition was the Perkin Elmer HPLC program with the LC-1000 pump (Isocratic) column and the LC 250 UV / VIS detector was connected. The "CSW 32 program" for the HPLC method allowed peak detection and quantification. HPLC was measured at a rate of 2ml per minute by running the mobile process (acetonitrile, dichloromethane, and methanol 70:20:10. respectively. The wavelength was 452 nm. Column pressure was holding from 1800-2000 PSI. Each standard beta carotene solution (20µl) was injected at the time the injector was in load mode. The normal beta-carotene peak was reached at 4.7 minute retention time (Rt = 4.7). To get a straight line, the concentrations of the beta-carotene standards were plotted against the peak point.

Sample Assay

A sample of beta-carotene extract was used as usual in 80 percent acetone for HPLC assay; a sample of vegetables (20µl) was taken by micro liter syringe. The peak was immediately identified and quantified by comparing its sample retention time to the standard retention period.

Result and Discussion

In the present investigation efforts have been taken for qualitative and quantitative determination of chemical composition, mineral composition and phytochemical content of mint leaves powder given as following

Percent yield and drying parameters for mint leaves

Data pertaining to the various conditioned employed, time, temperature and drying yield were studied and results obtained are summarized in Table 2.

Table 2: Drying condition and yield of dried mint leaves

Sr No.	Drying methods	Conditioned employed	Time (hrs/days)	Drying Yield /100 gm
1	Sun drying	30-35^0 C	8 hrs	32.90
2	Shade drying	27-32^0 C	15 days	33.75
3	Cabinet oven drying	60^0 C	2 hrs	31.50

Results given in the table 2 indicated that the conditioned employed for sun drying was 30-35^0 C for 8 hrs., for shade drying 27-32^0 C for 15 days and for Cabinet oven drying 60^0 C for 2 hrs. It observed that cabinet oven drying was rapidly dried the leaves as compare to other methods. The data regarding drying yield was higher from shade drying method i.e. 33.75% as compare to lower yield from cabinet oven drying i.e. 31.50%.

In all these methods, the energy consumption and time taken for drying found to be more in shade drying method and produce quality of end-dried product is superior in all respect like colour, flavour taste as compared to other dried products. These results are comparable with those reported by (Rajkumar *et al.*, 2006 and Dadali *et al.*, 2007) [18, 6].

Proximate composition of mint leaves powder

Data pertaining in present finding of proximate composition of mint leaves powder with respect to moisture, fat, protein, carbohydrate, crude fibre and ash was conducted and obtained results were presented in Table 3.

Table 3: Effect of drying methods on proximate composition of mint leaves powder

Treatments	Values (g/100g)					
	Moisture	Fat	Protein	Carbohydrate	Crude fibre	Ash
TM0	82.60	0.60	4.75	8.95	5.70	2.10
TM1	4.80	2.95	13.25	52.02	17.60	9.38
TM2	4.95	2.81	13.90	51.98	17.10	9.25
TM3	4.20	2.40	13.62	53.88	16.20	9.70
SE+	0.4982	0.0322	0.2345	0.3969	0.3605	0.1329
CD at 5%	1.6315	0.1657	0.6012	1.7548	0.9928	0.5421
TM0	Fresh mint leaves					
TM1	Sun dried mint leaves powder					
TM2	Shade dried mint leaves powder					
TM3	Cabinet oven dried mint leaves powder					

*Each value is average of three determinations

The present data in table 3. Provide information on the effect of different drying methods (sun, shade and cabinet drying) on proximate composition of mint leaves powder and determine the best method for drying of mint leaves powder.

The moisture content of the dried samples was significant difference and it was drastically reduced as compared to fresh mint leaves samples TM0 (82.60). The samples (TM1, TM2 and TM3) subjected to drying till the final moisture content reaches to 4.80, 4.95 and 4.20 per cent respectively. The maximum moisture content in dried powder TM2 (4.95%) and minimum was in the TM3 (4.20%).

The fat content of mint leaves powder was increased after drying. The TM1 powder retained the highest fat content with a value of 2.95 per cent. The TM2 and TM3 sample had fat

content 2.81and 2.40 per cent and frsh mi. It shows that TM1 was significant with TM2 and found at par with TM2. It represents a good index of storability as it reduces the susceptibility of the powder to lipid oxidation.

The TM2 had highest protein content in all the drying treatments with a value of 13.90% which was significantly higher than other drying methods. The protein content of TM1 and TM3 powder was 13.25 and 13.62 per cent. The result shows that TM2 was significant with TM1 and TM3 with respect to protein content. The change in protein content could be attributed to mild heating effect associated with all the drying conditions which could result in the unzipping of hydrophobic forces leading to a partial distribution of the primary, secondary, tertiary and quaternary structure of the protein molecule (Ihekoronye and Ngoddy, 1985) [10].

The carbohydrate content in dried powder was increased as compared to fresh leaves. The carbohydrate content of dried powder was found in the range of 51.98 to 53.88 per cent. It was observed the TM3 drying was significant with TM1 and at par with TM2. The increase in these nutrients could be attributed to the application of heat.

The crude fibre of dried powder was found in the range of 16.20 to 17.60 per cent. It was observed the TM1 was significant with TM2 and at par with TM3. The ash content of mint leaves powder increased significantly as compare with fresh mint leaves i.e. TM0 (2.10). The ash content of mint leaves powder by TM1, TM2 and TM3 was found to be 9.38, 9.25 and 9.70 per cent, respectively. In case of ash content, TM3 was statistically significant with TM1 and TM2 drying. The TM3 had high ash content which might be due to less moisture as compared to TM1 and TM2 drying. The increase in ash content observed in this study could be due to the removal of moisture, which tends to increase the concentration of nutrients (Morris *et al.,* 2004). The protein, carbohydrate, fat, fibre and ash content of mint leaves powder were increased when subjected to sun, shade, cabinet oven drying (Satwase *et al.,* 2013) [21]. The similar trend of protein, fat, ash and carbohydrate was reported by Abioye *et al.,* (2014) [2].

Mineral composition of mint leaves powder

The observed values of minerals composition of the three different drying techniques (i.e. sun, shade and cabinet) drying on mint leaves powder with respect to calcium, potassium, iron, magnesium, zinc and phosphorous is dedicated in Table 4.

Table 4: Effect of drying methods on mineral composition of mint leaves powder

Treatments	Minerals content (mg/100g)					
	Calcium (Ca)	Potassium (K)	Iron (Fe)	Magnesium (Mg)	Zinc (Zn)	Phosphorus (P)
TM1	175.10	481.63	8.30	88.54	12.75	56.72
TM2	186.25	479.55	8.75	95.76	13.10	58.45
TM3	192.30	503.30	10.15	96.20	11.15	69.09
SE+	0.2105	0.0516	0.4236	0.3548	0.1644	0.2472
CD at 5%	0.7933	0.1515	1.2425	1.0409	0.3830	0.5123

TM1 Sun dried mint leaves powder
TM2 Shade dried mint leaves powder
TM3 Cabinet oven dried mint leaves powder

Table 4 shows that the TM3 was found significantly superior over TM1 and TM2 drying. The mineral contents were found to be increased upon drying. The calcium content was found in increased trends measured. The calcium content in TM1, TM2 and TM3 was 175.10, 186.25 and 192.30 mg/100g, respectively. According to Perez-Lopez *et al.,* (2002), the calcium content was affected by temperature, calcium chloride concentration and treatment time. The potassium content in TM1, TM2 and TM3 was 481.63, 479.55 and 503.50 mg/100g, respectively. This observation may be due to potassium being cationic element that does not polarizes easily in heating but forms oxides when exposed to light and air (Liman *et al.,* 2014) [13].

The iron content in TM1, TM2 and TM3 was 8.30, 8.75 and 10.15 mg/100g, respectively. The magnesium content of TM1, TM2 and TM3 powder was 88.54, 95.76 and 96.20 mg/100g respectively. The increase in magnesium content is probably be due to the heating effect of the drying minerals which do not escape/vaporize and as such higher values in magnesium were seen (Liman *et al.,* 2014) [13].

The TM2 mint leaves powder (13.10 mg/100g) recorded higher zinc content over TM1 (12.75 mg/100g) and TM3 powder (11.15 mg/100g). The TM3 mint leaves powder (69.09 mg/100g) recorded higher phosphorus content over TM1 (56.72 mg/100g) and TM3 powder (58.45 mg/100g).

However, the value of mineral contents increases in relation to the drying method. The increase or decrease of micronutrient of dried sample may be attributed to the removal of water molecule by drying. The similar findings were also reported by Joshi and Mehta (2010) [11], who studied the effect of dehydration on nutritive value of drumstick leaves and concluded that shade dried samples, had highest minerals retention followed by shade dried samples.

Phytochemical content of mint leaves powder

The phytochemical content of fresh mint leaves and dried powder not only showed the presence of these compounds but also revealed a variation in the concentration of such compounds as alkaloids, flavonoids, total phenol and β-Carotene using different drying methods and result tabulated in Table 5.

Table 5: Effect of drying methods on phytochemical content of mint leaves powder

Treatments	Phytochemical content (g/100gm)			
	Total Alkaloid	Total Flavonoid	Total Phenol (µg/g)	β-Carotene
TM0	11.50	8.10	0.65	4.80
TM1	12.02	9.20	0.77	5.32
TM2	12.28	9.57	0.79	5.74
TM3	10.30	8.15	0.59	4.77

TM0 Fresh mint leaves
TM1 Sun dried mint leaves powder
TM2 Shade dried mint leaves powder
TM3 Cabinet oven dried mint leaves powder

With reference to Table 5, the alkaloids, flavonoid, total phenol and β-Carotene recorded their highest value in the samples were TM2 i.e. 12.28, 9.57, 0.79 and 5.74 g/100g respectively compared to sample TM1 i.e. 12.02, 9.20, 0.77 and 5.32 g/100g respectively. There is not much critical difference observed in the phytochemical content in the treatments.

The alkaloids, flavonoid, total phenol and total flavonoid were affected by drying methods and results obtained were tabulated in Table 20. The maximum value of alkaloids content was found in TM2 (12.28 g/100g) than TM0, TM1 and TM3 i.e. 11.50, 12.02 and 10.30 g/100g respectively. Maximum reduction in flavonoid content found in TM2 (9.57

g/100g) aqueous extract to other samples i.e. TM0, TM1 and TM3 have 8.10, 9.20 and 8.15 g/100g respectively. The loss in alkaloids and flavonoids in sample TM1 and TM3 may due to breakdown or leakage by chemical reactions includes oxygen, enzymes and light (Davey *et al.*, 2000) [7]. Increasing preheating temperature decreased the enzyme activity to degrading enzyme such as polyphenoloxidase, which resulted in an increase in flavonoid content (Sukrasno *et al.*, 2011) [23].

The reduction in total phenol and β-Carotene content were also found by Mansour, (2016), who found that drying process significantly decreased the phenolic and β-Carotene contents i.e total phenol reduced from 0.79 g/100g (TM2) to 0.59 g/100g (TM3) and β-Carotene reduced from 5.74 g/100g (TM2) to 4.77 g/100g (TM3). These could be explained by the fact that the drying time was extended at higher temperatures, such that the samples had a longer duration of oxygen exposure resulting in increased redox activity and degradation of phenolic compounds (Hung and Duy 2012). Drying process led to loss of 30% of polyphenols in total phenol content and 25% loss of β-Carotene which reported in Felipe *et al.*, (2010) [8].

The thermal processing can affect the phytochemicals by thermal breakdown that affect the integrity of the cell structure which then results in the migration of components, leading to losses by leakage or breakdown by various chemical reactions involving enzymes, light and oxygen (Schieber *et al.*, 2001) [22]. The shade dried powder contained more alkaloids, flavonoid, total phenol and β-Carotene than sun drying and cabinet oven drying.

Conclusion

Energy consumption and time taken for drying found to be more in shade drying method and produce quality of end-dried product is superior in all respect like colour, flavour taste as compared to other dried products. This increment of proximate composition was seen in all dried powder prepared from different drying methods in all dried. The value of mineral contents increases in relation to the drying method. The increase or decrease of micronutrient of dried sample may be attributed to the removal of water molecule by drying. A variation in the concentration of such compounds as alkaloids, flavonoids, total phenol and β-Carotene and found more concentration in shade dried mint powder TM2 had values 12.28 g/100gm, 9.57 g/100gm, 0.79 µg/g and 5.74 g/100gm respectively.

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