Pre-distillation drying and its impact on aroma profile of Rosemary elite genotype (cv. ‘CIM-Hariyali’)

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Rosemary (Rosmarinus officinalis Linn.) leaves are well known flavouring agent for different type of dishes. The volatile oil from the leaves is used in perfumery and medicine. In order to find best drying method for rosemary leaves, different approaches, viz. shade, sun, microwave, hot air and oven drying were compared. The essential oil content was found to vary from 0.18 to 1.1% under different methods of drying. The essential obtained from hydrodistillation of dried leaves was analysed by GC and GC-MS. The major components of the oils were 1,8-cineole, camphor, α-pinene and verbenone. The quantitative composition of the oils was significantly affected by mode of drying. The monoterpene hydrocarbons were dominated in the fresh, shade and sun dried leaves (24.7 - 25%) whereas oxygenated monoterpenes were found to be higher in microwave and oven dried leaves (83.6 and 82.4%, respectively).

Keywords: Aroma profile, Drying method, Essential oil, Leaves, Rosemary, Rosemary cv. ‘CIM-Hariyali’, Rosmarinus officinalis.

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Introduction
Rosemary (Rosmarinus officinalis Linn.) is a very common aromatic plant which has been cultivated in every Mediterranean country for centuries. Nowadays, it is widely used because of its numerous biological effects. The leaves of rosemary are used in foodstuffs, especially for the control of microbial infections. It has also stomachic, antidiarrhoeic and anti-rheumatic properties. It showed antioxidant, antibacterial and even insecticidal activities. Scientific investigation of the chemical composition of the rosemary oil has been made by several workers in India and other countries.

Most of the herbs and spices are marketed dried, because, due to high water content in the fresh state they undergo severe deterioration after microbial growth and biochemical changes. Water removal by dehydration of herbs and spices reduces microbial growth. However, it has been reported that drying plant materials before distillation could affect the yield and composition of the oil considerably. The essential oil components are however, evaporated or some oxidation products can also appears during drying, therefore, the method of drying is very important. In this paper, we report the effect of drying methods on yield and chemical composition of the essential oil of R. officinalis cv. ‘CIM-Hariyali’ grown in Kumaon region of western Himalaya.

Materials and Methods

Plant material
The fresh leaves of R. officinalis cv. ‘CIM-Hariyali’ (Plate 1) were collected from the experimental farm of Central Institute of Medicinal and Aromatic Plants Research Centre, Purura, Uttarakhand, India in the first week of May. A total 18 random samples, weighing 100 g each (fresh weight basis) were divided in to 6 batches of three samples per batch. The first batch was distilled in fresh condition; second batch was left to dry in the shade (under normal air at room temperature 30±1°C); third batch was dried under the sun (32±1°C); fourth batch was dried in microwave oven (900 W); fifth batch was dried under hot air, while sixth batch was dried in the oven at 50°C.

Isolation of essential oil
The essential oil was extracted by hydro-distillation for 3 h using Clevenger type apparatus. The oil content (v/w %) was estimated on the fresh weight basis. The oil samples obtained were dehydrated over
Gas Chromatography (GC)

The GC analyses of the oil samples was carried out on a Nucon gas chromatograph model 5765 and PerkinElmer Auto XL GC equipped with FID and two different stationary phases, BP-20 (30 m × 0.25 mm × 0.25 µm film thickness) and PE-5 (60 m × 0.32 mm; 0.25 µm film coating) fused silica capillary columns, respectively. Hydrogen was used as carrier gas at 1.0 ml/min. Temperature programming was done from 70-230°C at 4°C/min with initial and final hold time of 2 min (for BP-20) and from 70-250°C at 3°C/min (for PE-5). Split ratio was 1:30. The injector and detector temperatures were 200 and 230°C on BP-20 and 220 and 300°C on PE-5 column, respectively.

Gas Chromatography-Mass Spectrometry (GC-MS)

GC-MS recorded on a PerkinElmer Auto System XL GC and Turbo Mass Spectrometer fitted with fused silica capillary column, PE-5 (50 m × 0.32 mm, film thickness 0.25 µm). The column temperature was programmed 100-280°C at 3°C/min, using helium as carrier gas at constant pressure of 10 psi. MS conditions were: EI mode 70 eV, ion source temperature 250°C.

Identification of compounds

Identification of components were done by comparing the retention times, retention indices and mass fragmentation pattern to literature values and by peak enrichment on co-injection with authentic samples wherever possible to corroborate identities. The peak area percentage was computed from the peak areas without applying FID response factor correction.

Results and Discussion

The essential oil yield in fresh, shade dried, sun dried, microwave dried, hot air dried and oven dried leaves was 1.1, 1.05, 1.0, 0.18, 1.0 and 0.9%, respectively on fresh weight basis (Table 1). The loss in essential oil content was found to be higher in microwave dried samples (83.6%) followed by oven dried samples (18.2%) and sun dried (9.1%) or hot air dried samples (9.1%). Variation in essential oil yield due mode of drying has also been reported in other aromatic plants.

The analysis of these oil samples showed that drying method had significant effect on the chemical composition of rosemary leaves (Table 1). The major components in oil of fresh leaves were 1,8-cineole (31.7%), camphor (27.4%), α-pinene (12.7%), verbenone (6.5%), camphene (5.2%), β-pinene (2.9%), bornyl acetate (2.9%), β-myrcene (2.5%) and α-terpineol+borneol (4.2%). The concentration of α-pinene was slightly higher in shade and sun dried leaves (13.4 and 13.9%, respectively) when compared to fresh leaves while its concentration was only 0.1% in microwave dried leaves. The percentage of 1,8 cineole was found to be lowest in microwave dried leaves (4.3%) whereas in other methods of drying it was higher (31.8-33.9%) than fresh leaves. However, the amount of camphor was observed to be higher in microwave dried leaves (32.3%) followed by oven dried leaves (31.7%), while in shade, sun and hot air dried leaves it was 26.6, 26.9 and 27.1%, respectively. Interestingly, the concentration of verbenone was found to increase on drying (6.5-18.4%). Its amount was observed to be maximum in microwave dried leaves (18.4%) followed by oven dried leaves (9.4%). α-Terpineol+borneol was also recorded higher in microwave and oven dried leaves (17.4 and 4.5%, respectively) compared to other samples. Further, the microwave dried leaves were also found to be rich in bornyl acetate (9.5%), β-caryophyllene (6.1%), α-humulene (4.7%) and terpinen-4-ol (1.3%) whereas in rest of the samples these components ranged between 1.9-2.9%, 0.4-0.5 %, 0.2-0.3% and 0.4-0.7%, respectively. The results also shown that the monoterpenes hydrocarbons which dominated in the fresh, shade dried and sun dried leaves (24.7-25%) was reduced to 23.4, 16.4 and 0.2 % in the hot air dried, oven dried and microwave dried leaves, respectively. On the other hand microwave dried and oven dried leaves were richer in oxygenated monoterpenes (83.6 and 82.4%, respectively) while in...
rest samples it ranged from 73.8 to 75.4%. Further, the amount of sesquiterpenes also recorded higher in microwave dried leaves.

The changes in the regimes of volatile compounds during drying have been reported to depend on several factors such as drying method and change to species or family\textsuperscript{22,23}. The components of the essential oils that lost in the dried leaves were those stored on or near the leaf surfaces\textsuperscript{24}. Therefore, the results of present study reinforced the fact that there were quantitative and qualitative differences in the essential oil of fresh and dried plant materials.

**Conclusion**

In general, the shade and sun drying did not cause major variation in the essential oil yield and chemical composition whereas hot air and oven drying methods moderately changed the composition of essential oil. However, microwave drying significantly reduced the oil yield, monoterpenes and 1,8-cineole concentration. Hence, on the basis of essential oil yield and composition, shade drying method was found to be most suitable followed by sun and hot air drying for rosemary leaves.

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