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# *Short Communication*
# Preparation of Menthol Crystals from Mint (*Mentha arvensis*)

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## ABSTRACT

Essential oil of partially dried leaves of *Mentha arvensis* was extracted by steam distillation method. From this essential oil, menthol crystals were prepared by chilling at different temperatures and yield of both essential oil and menthol crystals was 2.4 and 52.3%, respectively. Various physical constants of menthol crystals like refractive index, melting point, specific gravity and optical rotation were also determined. The crystals of menthol were colorless hexagonal needle like with pleasant minty odour and slightly pungent taste and possessed strong cooling effect.

**Key Words:** Menthol; Mint

## INTRODUCTION

Mentha (mint) was used as medicinal herb in ancient times, but menthol crystals are still used in different pharmaceutical products and cosmetics as antiseptic, stimulant and inhibitor. It gives minty flavor to various food products. It is also used in oral products e.g. tooth paste and mouth fresheners due to its physiological cooling effect. Pakistan spends billion of rupees in importing menthol crystals for industrial uses.

Mint is an erect, hairy and fragrant herb of Labiatae family. The essential oil obtained from different mint species often contains menthol as major constituent (40-85%). The mint species that have higher menthol contents are *Mentha piperita* known as American peppermint (50-55%) and *Mentha arvensis* termed as Japanese mint (80-85%). These species represent the most economical source for extraction and crystallization of menthol (Guenther, 1964).

Therefore, present work was aimed at extraction and crystallization of menthol from mint (*Mentha arvensis*).

## MATERIALS AND METHODS

The essential oil was extracted from partially dried leaves of *Mentha arvensis* by steam distillation method. The moisture/water was removed from oil by adding anhydrous $Na_2SO_4$ and oil was filtered (Furnis *et al*., 1978). Yield of oil was determined and menthol was crystallized by chilling the oil at +14, +10 and –5°C for 8 hours each, respectively in well sealed plastic containers which were placed in simple freezers. Menthol crystals were separated from mint oil. As dementholized mint oil still contained menthol, racemic and isomenthols and menthone; therefore, for complete recovery of menthol crystals, it was treated with 8 g boric acid in distillation flask for 3 h to distill off menthone. Distillation residues containing borates of menthol were saponified by steam distillation over 70 g of 15% NaOH solution and thereby separating the menthol crystals from liquid mixture containing racemic and isomenthol. Crystals were dried at 26°C and yield was determined (Guenther, 1964). Some physical constants determined were refractive index by Abbe's Refractometer in 70% alcohol solution (Kirk & Othmer, 1984a), optical rotation in 50% alcohol solution by Lippich polarimeter (Shoemaker *et al.,* 1989), specific gravity by volumanometer method (Kirk & Othmer, 1984b) and melting point by capillary tube method (Furnis *et al*., 1978).

## RESULTS AND DISCUSSION

Calculated yield (%) of essential mint oil on the basis of quantity of partially dried mint herb was 2.4% as shown in Table I. The yield of freshly cut leaves of *M. arvensis* of SanBi and Ninomiya was 1.6 and 1.7%, respectively as reported by Guenther (1964). The higher yield (2.4%) of the oil in this study might be due to the use of partially dried herb instead of fresh cut leaves. Percent yield of menthol crystallized from mint oil was 52.3% as shown in Table I, which was quite closer to that reported value (53.92%) in Hokkaido but lesser than Yamagata yield which was 76.09% (Guenther, 1964). Yield of menthol crystals depends upon many factors including menthol contents of mint oil, absence of impurities in oil, properly air tight container and sufficient cooling time.

**Table I. Yield of essential oil of *Mentha arvensis* and Menthol crystals**

| **Total leaves used** | **11 kg** |
|---|---|
| Oil yield | 260 g and 2.4% oil on partially dried leaves wt. basis |
| Total oil used for crystallization | 250 g |
| Yield of menthol crystals on basis of wt. of mint oil | |
| 1. By chilling the mentholized mint oil | 110.8 g and 44.3% |
| 2. From remaining dementholized mint oil | 20.1 g and 20.1% |
| Total yield of menthol crystals (%) | 130.9 g and 52.3% |

The physical characteristics of menthol crystals have been presented in Table II. Optical rotation of menthol crystals was –49.25 in 50% alcohol solution. The findings were in accordance with Guenther (1952), -49 in 50% alcohol solution and was –49 to –51 in 50% alcohol solution as reported by Gildmeistar and Hoffman (1937). This property in general depends upon temperature, solution concentration, path length, oxidation and polymerization of oil. Refractive index (RI) was 1.44 at 20°C in 70% alcohol solution. The range of RI reported by Guenther (1952) was 1.440 to 1.460.

**Table II. Physical constants of menthol crystals**

| **Optical rotation** | **-49.25 in 50% alcohol solution at 20°C** |
|---|---|
| Refractive index | 1.44 at 20°C |
| Specific gravity | 0.878 at 25°C |
| Melting point | 43.5°C |

Gildemeister and Hoffman (1937) showed 1.457 RI at 25°C. Refractive index generally depends upon climate, crop condition, polymerization, oxidation of oil and temperature. Specific gravity determined was i.e. 0.878 at 25°C, which was quite in accordance with Gildemeister and Hoffman (1937) who reported it as 0.896 at 20°C, and Huggett (1942) reported as 0.891 at 20°C. The result was also within the range showed by Huckel and Reimer (1937) 0.862-0.888. This variation was due to temperature, experimental error and density of menthol crystals. Melting point of menthol crystals determined was 43.5°C, which was close to the range showed by Gildemeister and Hoffman (1937) i.e. 42 to 44°C. Tewari *et al.* (1998) reported 45°C melting point of the menthol crystals. It was observed that the traces of adhering oil, also lowered the melting point of menthol crystals.

## CONCLUSION

As physical constants of prepared menthol crystals were very closed to the standard physical constants, therefore, these crystals are quite suitable for industrial use.

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