

Fractionation of rosemary (*Rosmarinus officinalis* L.) essential oil using vacuum fractional distillation

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Rosmarinus officinalis L. is a perennial shrub from Mediterranean, of *Lamiaceae* botanical family. Chemical composition of its extracts and essential oil varies according to climatic factors during cultivation, genetic factors and extraction process. The essence of rosemary is a colorless to yellowish liquid, with camphorated scent. The composition of rosemary oil is from 95 to 98 wt. % of monoterpenes and derivatives and 2 to 5 wt. % of sesquiterpenes. Hydrocarbon terpenes are dominant, cineol is in appreciable amounts and ketones in small content. Literature presents many works regarding the antimicrobial and antioxidant activity of rosemary essential oil. The use of raw oil in biological control of agricultural pests (mainly fungi) is widely known, despite the problems of the scale-up process (1). Fractional distillation is a widely known and used method for separating liquid mixtures into their constituents. For the separation of essential oils, one of the possible processes is vacuum fractional distillation (2). The oil used in the fractionation process was commercial rosemary oil, obtained by steam distillation of the leaves. The column used for separation consisted of 2 stages and a 250 ml flask, which was the reboiler. The packing was glass Raschig rings with diameter of 8 mm. The maximum vacuum of the system was 10 kPa, measured by an analogic vacuum gauge. The electrical heating had a power yield of 150 W, being operated by PWM mode. The condensed sample from each stage was collected in vials connected to the column. GC/MS analysis of raw oil and fractions were performed on a HP 6890 model GC, coupled to a HP 6890/MSD5973 mass selective detector, equipped with a HP Chemstation software and a Wiley 275 spectral library. HP-5MS fused silica capillary column (30 m x 250 µm) with 0.50 µm film thickness (HP, Palo Alto, USA) was used. Column temperature was 60°C for 8 min, to 180°C at 3°C/min; to 230°C at 20°C/min. Injector temperature of 220°C, interface temperature of 250°C. Split ratio 1:100, helium at 56 kPa as carrier gas, flow rate of 1.0 mL/min, ionization energy of 70 eV. The results presented a separation pattern of hydrocarbon terpenes being distilled and collected at the top of the column in the four temperatures tested (70, 90, 115 and 125°C). Small amounts of other chemical functions (alcohols, ketones) were also distilled, the amount collected in the top increased with increase of the temperature. Compounds such as verbenone (5.5 wt. % in raw oil), linalool (2.4 wt. % in raw oil) and geraniol (1.4 wt. % in raw oil) had their concentrations in the bottom fraction increased more than three times (24.4 wt. % for verbenone, 4.4 wt. % for linalool and 7.4 wt. % for geraniol at 125°C), indicating the potential use of vacuum fractional distillation to obtain minor compounds from rosemary essential oil, for uses in food, cosmetics and pharmaceutical industries.

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