# Fractioning of green mandarin essential oil by vacuum fractional distillation.

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The harvest of citrus fruits presents great importance worldwide, as its production, in 2013, was approximately 111.5 millions of tons. The main citrus fruits are orange, lemon, mandarin and lime, which have a high commercial value as sell 'in nature' or being processed in food industry (1). The citrus essential oil is a mixture of terpenes, many of them with important industrial applications in medicine, pharmacology, cosmetics and food industry. Though, to have a practical application for the minority compounds (which, in general, have more value), it is necessary to properly separate the mixture of substances in the raw oil (1,2). Aldehydes, alcohols and ketones that generally appear as minority compounds or as trace, are important in the composition of the oil flavor, being products of high value when compared to other terpenes present in the mixture. The green mandarin essential oil was obtained by pressing, being stored away from sunlight and at room temperature. The oil was distillated in a glass column, filled with Raschig rings with 8 mm of diameter. The column had four stages, including the bottoms. A vacuum pump was coupled to the condenser, to reduce the pressure inside the column. It was utilized 120 mL of raw essential oil in each batch. The vapor was collected for 10 minutes, from the time the vapor reached each of the stages. The distillation in the last stage was kept until the volume in the distillation flask (bottoms), and less than 10 mL. The collected samples and the raw oil were analyzed by an Agilent GC model 6890 Series, and using a HP Innowax column (30 m x 320 µm i.d.) with film thickness of 0.5 µm and by an Agilent mass spectrometer model MSD5973 with a fused silica HP-Innowax column (30 m x 250 µm) with film thickness of 0.5 µm. The column was set at 40 °C (8 min) to 180 °C at 3 °C/ min, 180 to 230 °C at 20 °C/ min and at 230 °C for 20 min. Injector temperature was 250 °C; split ratio of 1:50, flame ionization detector at 250 °C; $H_2$ as carrier gas at 1.0 mL/ min, at 34 kPa. Volume for injection was 1.0 µL. For GC-MS, the temperature program was the same as GC-FID, with interface at 280 °C; split ratio of 1:100, He as carrier gas at 56 kPa at 1.0 mL/ min; ionization energy of 70 eV; the injected volume was 1 µL, sample diluted in hexane (1:10). Volume of distillates were near to 30 mL, and the remaining bottoms were less than 5 mL. GC/MS analysis showed that only hydrocarbon terpenes were distillated. Although the raw oil was mainly composed by limonene (80.3 %) and γ-terpinene (19.1 %), the distillation patterns were different in each stage. The terpenes with other chemical functions (alcohols, aldehydes, amines) remained in the bottoms. For some compounds, the bottoms concentration was more than 20 times the concentration in the raw oil, as for dimethyl anthanilate and α-sinensal.

1. Castro, M.D.L.; Escobar, C.A.L. Tr. Food Sci. Technol., 2014, **39**, 63-75.
2. Simões, C. M. O., et al. Farmacognosia: da planta ao medicamento. 5a ed. Porto Alegre/ Florianópolis: Editora UFRGS/Editora da UFSC, 2004.

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