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Review

Comparison of extraction of patchouli (*Pogostemon cablin*) essential oil with supercritical CO_2 and by steam distillation

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ABSTRACT

Patchouli essential oil is an important raw material for the perfume and cosmetics industries, besides being used as a natural additive for food flavoring. Patchoulol and α-patchoulene are important compounds of patchouli essential oil, and their concentrations are directly proportional to the quality of the oil. Nowadays, the usual method employed to obtain patchouli essential oil is steam distillation; however, this causes thermal degradation of some oil compounds. In this study patchouli essential oil was extracted with supercritical carbon dioxide ($scCO_2$) under different conditions of pressure (8.5 and 14 MPa) and temperature (40 and 50 °C) and also by steam distillation to compare the extraction methods. It was demonstrated that the extraction with supercritical carbon dioxide provided a higher yield and a better quality of patchouli essential oil.

Contents

1. Introduction

Patchouli oil is obtained from the leaves of *Pogostemon cablin* (patchouli), a plant of the Lamiaceae family, originating from Malaysia and India [1]. It is an important essential oil in the perfume industry, used to give a base and lasting character to a fragrance [2,3,4]. The essential oil is very appreciated for its characteristic pleasant and long lasting woody, earthy, and camphoraceous odor, as well as for its fixative properties, being suitable for use in soaps and cosmetic products [5,6]. It is also on the FDA's (Food and Drug Administration) list of substances approved for human consumption, in section 172.510, as a natural additive for food flavoring [7]. Moreover, the plant (*P. cablin*) is widely used in traditional Chinese medicine as it offers various types of pharmacological activity according to the composition of the oil [1,8].

The composition of patchouli oil is unique and complex because it consists of over 24 different sesquiterpenes, rather than a blend of different mono-, sesqui- and di-terpene compounds [5]. The sesquiterpene patchoulol is the major constituent and is the primary component responsible for the typical patchouli aroma. This essential oil is also characterized by a large number of other sesquiterpene hydrocarbons such as α-/β-/γ- patchoulenes, α-guaiene, seychellene, and α-himachalene. Although α-patchoulene is found in small amounts, it is an important constituent of

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patchouli oil because, together with patchoulol, it also determines the aroma of the oil. Also, it is believed that the antifungal activity of the essential oil is closely related with these two compounds [2,5,8]. Thus, the greater the concentration of these compounds in the essential oil, the better the quality and the higher the commercial value [9].

Therefore, as the commercial value of patchouli essential oil is directly correlated with its qualitative and quantitative composition, which varies according to the cultivation region and extraction technique [1,2], an improved process for its extraction would be of industrial interest. It should be noted that patchouli plants are the only commercial source of patchoulol and that cost-effective synthetic routes for enantiomerically pure patchoulol have yet to be developed [5].

Nowadays, patchouli essential oil is traditionally obtained by steam distillation [1,2,5]. This procedure, performed at a high temperature, can cause the degradation of thermally labile compounds resulting in the formation of undesirable compounds [10]. In this regard, extraction of essential oils using supercritical carbon dioxide ($scCO_2$) has been the subject of considerable interest, mainly for the extraction of natural products. Carbon dioxide has several unique characteristics and physico-chemical properties, since it is non-toxic and inert and has low critical pressure (7.38 MPa) and temperature (31.1 °C). Compared with conventional extraction methods, extraction with $scCO_2$ has many advantages including more selective extracts without thermal degradation and which are solvent-free, thus providing an oil of superior quality [11,12]. The selectivity of carbon dioxide in relation to the essential oil can be adjusted by changing the temperature and pressure conditions, leading to oils with different compositions. Carlson et al. [12] observed that the best condition for the extraction of lemongrass essential oil was 12 MPa and 40 °C.

The objective of this study was to compare the variations in the yield and chemical composition of patchouli essential oil obtained under different conditions (pressure and temperature) of supercritical extraction with CO_2 and by steam distillation. No information could be found in the literature regarding the use of $scCO_2$ for patchouli essential oil extraction.

2. Experimental

2.1. Material

Patchouli plants [*P. cablin* (Blanco) Benth] were collected in November 2002 from “Colônia Penal Agrícola” (Palhoça, SC, Brazil). For the extraction, the leaves were collected manually from the plants and all of them were from the same lot.

Patchouli leaves were dried in an oven with air circulation (Model TE – 394/2, TECNAL, Brazil) for 1440 min at 30 °C and 180 min at 35 °C. These temperatures were selected because they have previously been used in the drying of patchouli leaves for essential oil extraction [13]. The dried leaves were ground with a knife grinder (Model MA – 580, Marconi, Brazil) and, in the case of the supercritical extractions, were then sieved (mesh 30) in order to standardize the size of the particles.

2.2. Supercritical equipment

The extractions with $scCO_2$ were performed in a pilot unit schematically represented in Fig. 1.

A gas booster (3) (Model DLE 15-1, MAXPRO Technologies, Germany) received liquid CO_2 (99% purity, White Martins, Brazil) from a cylinder (1) and pressurized a jacketed surge tank (6) (Labsolda, UFSC, Brazil, 4.6×10^{-3} m^3 volume) which in turn provided gas to a jacketed extraction vessel (9) (Labsolda, UFSC, Brazil, 1×10^{-3} m^3 volume and 0.55 m height). The jacketed surge tank was placed between the gas booster and the extraction vessel in order to avoid potential pressure overshoots allowing a better pressure control. The temperatures of the surge tank and extraction vessel were controlled by a thermostatic water bath (18) (Model MQBTC 99-20, Microquímica, Brazil).

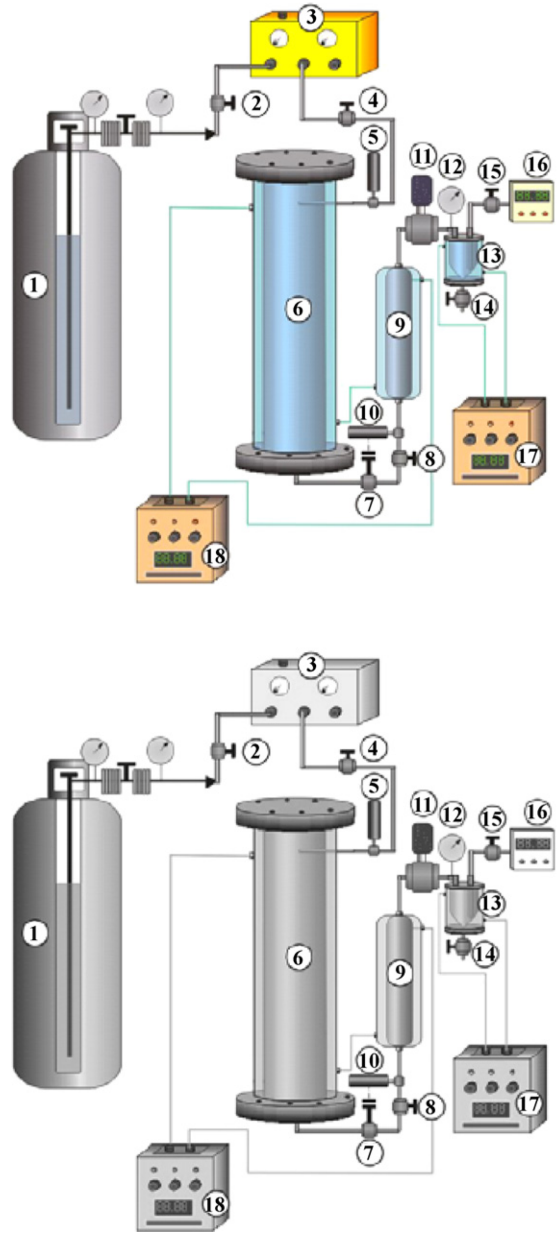

Fig. 1. Experimental unit of supercritical fluid process: (1) CO_2 cylinder; (2, 4, 8, 14) flow control valves; (3) gas booster; (5 and 10) pressure transducers; (6) jacketed surge tank; (7) pneumatic control valve; (9) jacketed extraction vessel; (11) forward pressure regulator; (12) manometer; (13) separation vessel; (15) micrometer valve; (16) flow meter; (17 and 18) thermostatic water baths.

The extraction pressure was maintained by the gas booster, monitored by a pressure transducer (10) (Model RTP12/BE53R, AEP, Italy) and controlled by a pneumatic control valve (7) (Model: 807, Badger Meter, USA). The samples were collected at different time intervals in a separation vessel (13) at a pressure of 2.4 MPa and temperature of 34 °C, allowing the separation of the oil by changing the CO_2 phase. The temperature was maintained by a thermostatic water bath (17) and the pressure by a forward pressure regulator