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Parameter optimization in microwave-assisted distillation of frankincense essential oil

Mohammad Turk [a, *], Carole Mathe [b], Anne-Sylvie Fabiano Tixier [a], Diego Carnaroglio [c, d], Farid Chemat [a]

[a] UMR408 "Sécurité et qualité des produits d'origine végétale", Université d'Avignon et des pays de Vaucluse, 84000 Avignon, France
[b] UMR7263/IRD237, Avignon University/CNRS/IRD/AMU, Restoration Engineering of Natural and Cultural Heritage, 84000 Avignon, France
[c] Milestone, Via Fatebenefratelli, 1-5, 24010 Bergamo, Sorisole, Italy
[d] Dipartimento di Scienza e Tecnologia Del Farmaco, Centre for Nanostructured Interfaces and Surfaces, Università Degli Studi di Torino, Via Pietro Giuria 9, 10125 Torino, Italy

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ABSTRACT

The essential oil of *Boswellia* species is extracted by the conventional method of hydrodistillation (HD). In this study, we aimed to compare this reference to microwave-assisted distillation (MAD) at different power densities. The results showed that microwave extraction can result in a substantial reduction (48 min against 180 min for reference) of the batch time when the power density of 2 W/g is applied. Also, the energy consumed by the new process is 2.7 times less than HD. The essential oil produced by MAD has a chemical composition different from that of reference treatment. Although microwave treatment increased the proportions of the main components (α-thujene and α-pinene), the opposite tendency was observed for *o*-cymene, estragole, methyl eugenol, and δ-guaiene. A 26.5% decrease in oil extracted by MAD at 1 W/g is required to achieve 50% inhibition of the DPPH radical compared to HD. In conclusion, MAD is a promising technology for producing olibanum oil with new qualitative attributes.

1. Introduction

Frankincense is a resin derived from *Boswellia* genus. The gum is obtained by scraping the bark and/or making deep longitudinal incisions 4–8 cm long with a scalpel-like tool called a *mengaff*. The milky juice, left on the tree to dry, hardens on exposure to air into globular, pear-, or club-shaped pale yellow or pale amber gum tears [1]. The resin lumps are composed of essential oils (EOs; 5–9%), alcohol-soluble resins (65–85%), and the remaining water-soluble gums [2].

The ancient Egyptians used frankincense as a fumigant and in the embalming process [3]. The oils are currently used as a major ingredient in incense formulations, which are burned in Jewish, Roman Catholic, and Greek Orthodox religious rituals and ceremonies. The myriad of pharmacological properties attributed to frankincense includes its use in the treatment of asthma [4], rheumatoid arthritis [5], Crohn's disease [6], osteoarthritis [7]. Frankincense is used in perfumery and aromatherapy. It is also an ingredient sometimes used in skincare.

Boswellia products come in the form of resins, extracts, and EOs. The EO, which has a woody, spicy, and haunting smell, is usually obtained through steam distillation or hydrodistillation (HD) [8]. More than 300 volatiles in frankincense have been reported in the literature. A broad diversity has been found in the qualitative and quantitative composition of the volatiles with respect to different varieties of *Boswellia* [1,9]. Also, the abundance of EO is highly dependent on distillation process parameters (time and

* Corresponding author.
E-mail address: mohammad.turk@gmail.com (M. Turk).

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temperature) [9]. For instance, typical duration of distillation may vary between 6 h and up to 12 h [10]. Besides being an energy consumption technique, key labile constituents could be lost during this process at elevated temperatures for prolonged periods, whereas other compounds might be formed as artifacts.

The current environmental trends are to reduce energy and limit the carbon footprint. Novel techniques such as microwave (MW) HD are developed as an alternative to conventional processes for EO extraction [11]. This technique brings a number of advantages to EO extraction, thanks to their reduced equipment size, ability to control a process via mild increments in heating, all of which contribute to reducing qualitative degradation and an environmental impact. Many aromatic plants and spices such as *Xylopia aromatica* (Lamarck), *Lippia alba* (Mill.), *Zataria* multiflora Boiss., and so forth were submitted to this process for EO extraction [12–14]. For instance, Golmakani and Rezaei [15] found that microwave-assisted distillation (MAD) was superior in terms of saving energy and extraction time (3.2 times) than HD for the recovery of EOs from *Thymus vulgaris* L.

The literature analysis clearly shows a considerable effort to analyze the variability in the composition of Frankincense EOs related to the varietal factors. However, to the best of our knowledge, there is no study that has investigated the impact of the extraction process on the qualitative and quantitative aspects of EO extraction. The aim of this study was to compare and optimize the extraction kinetics, energy consumption, and profitability of the conventional and novel extraction techniques. Special attention will be given to the composition and antioxidant activity of Frankincense EO.

2. Material and methods

2.1. Raw material

Boswellia serrata oleo gum resin used in this study is presented in the form of amber to orange-brown lumps. They were purchased from Salus in Erbis (Rome, Italy) in 200 g bags and were stored at room temperature in a box protected from light.

2.1.1. Solvent for GC–MS analysis

Solvent and reagent for DPPH test: DPPH (1,1-diphényl-2-picrylhydrazil) was purchased from Sigma–Aldrich. Given the contradictory results about a possible antioxidant activity of *B. serrata* EO, natural lavender EO (Sigma–Aldrich), which has known antioxidant activity, has been used as a reference.

2.2. Extraction procedure

The experimental procedure is described in Fig. 1. Oleo gum resin lumps bags were assembled in one lot. The size of the lumps was reduced using an IKA MF10 basic microfine grinder (IKA, Germany) at 3000 rpm and a 4 mm sieve. The obtained powder was sieved using an RETSCH AS 200 sieve shaker set to a 2 min cycle. Only the fractions between 1 and 2 mm (94% of the sieved powder) were used for the essential extraction step.

B. serrata EO was recovered using two extraction processes: HD and MAD). HD device was equipped with a heater (Electrothermal, UK). The oleo gum resin powder (m_{RM} = 40 g) was introduced into a 1 L flask. Distilled water (solid/liquid ratio, 1:5 w/w) was added to the resin. The flask was connected to a Clevenger (Milestone, Italy) and a spiral double-jacketed condenser to ensure the condensation of the heated water/EO mixture. The same glassware was used for the MAD extraction process. Heating in the case of MAD extraction was provided by a Milestone ETHOS X oven (Milestone, Italy) equipped with two 950 W magnetrons and an infrared temperature sensor. The system additionally uses a rotating diffuser that evenly distributes the MWs throughout the cavity. Applied power, cycle time, and temperature limit were controlled via ETHOS X compact terminal.

The cooling system was a Smart H150-2100 chiller (LabTech, MA). The temperature and pressure of cooling

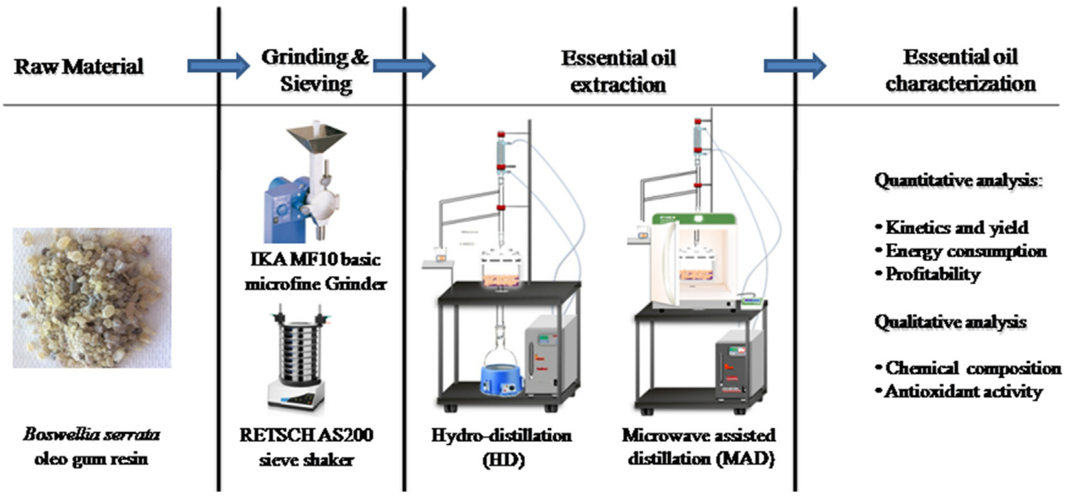

Fig. 1. Scheme of the experimental setup and the quantitative and qualitative analyses of the extraction of Frankincense EO.