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A short extraction time of high quality hydrodistilled cardamom (*Elettaria cardamomum* L. Maton) essential oil using ultrasound as a pretreatment

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ABSTRACT

The influence of ultrasonic assisted extraction (UAE) followed by hydrodistillation of *Elettaria cardamomum* L. seeds was investigated. The yield, volatile components and sensory characteristics of the extracted essential oils (EO) were evaluated. Power of ultrasonic and time of sonication were optimized. The chemical composition of the EO was identified by gas chromatography–mass spectrometry (GC–MS). Results revealed that the major components of cardamom essential oils varied between 26.59% and 39.34% for 1,8-cineole, and between 22.94% and 40.56% for α-terpinyl acetate, depending on the extraction conditions. The UAE technique facilitated short time extraction, improved extraction efficiency and produced good quality cardamom essential oil.

1. Introduction

Small cardamom, known as the "Queen of spices", which belongs to the family of Zingiberaceae, is a rich spice obtained from the seeds of a perennial plant, *Elettaria cardamomum* L. (Maton) (Chempakam and Sindhu, 2008). Its dried fruit is one of the highly priced spices in the world. The dried fruit is used either whole or in ground form as a flavoring agent and also in the medicinal preparations. The most functionally important constituent of cardamom is its volatile oil (Pruthi, 1976). The small cardamom is more in demand than large cardamom *(Amomum subulatum* Roxb.) as a commercial product, because of its fine aroma (Govindarajan et al., 1982). In Arab countries and India, it is a common flavoring ingredient for coffee (Raghavan, 2007). In Scandinavia, as well as in Germany and Russia, it is used to flavor cakes, pastries and sausages (Mahmud, 2008).

Cardamom is primarily cultivated in southern India, Sri Lanka, Tanzania and Guatemala (Ravindran and Madhusoodanan, 2002). The chemical composition of cardamom varies considerably with variety, region and age of the product. The content of volatile oil in the seeds is strongly dependant on storage conditions (Korikontimath et al., 1999). Cardamom fruits are gathered just before they are ripe in order to conserve the seeds inside the capsule, and then distilled to obtain the essential oil with an average yield from 2% to 5% (Lucchesi et al., 2007).

Different methods, viz; steam distillation, hydrodistillation, supercritical extraction, etc., are being used for essential oil recovery from spices. Hydrodistillation is the common and most frequently used method for extraction of essential oil (Chandran et al., 2012). This method is time consuming. On the other hand, the volatile and thermally sensitive components of essential oils may be lost in hydrodistillation conditions (Xie et al., 2013). The confines of the conventional techniques make obstinate to search for the new techniques that are equally competent and at the same time economically viable (Ashok kumar and Mason, 2007). In tissues where the desired components are located within cells, pre-ultrasound treatment by size reduction to maximize surface area is critical for achieving rapid and complete extraction (Vinatoru, 2001; Riera et al., 2004; Balachandran et al., 2006). Ultrasound assisted extraction technique (UAE) is an inexpensive, simple and efficient alternative to conventional extraction techniques (Wang and Weller, 2006). The technical advantages of UAE are mass transfer intensification, cell disruption, improved solvent penetration and capillary effect, high recovery yield and short extraction time confirmed that it is an acceptable extraction method (Yang and Zhang, 2008; Chemat and Zill-e-Huma Khan, 2011; Samarama et al., 2014).

The main objective of this research was to evaluate the suitability of ultrasound assisted extraction (UAE)–hydrodistillation combined technique compared to conventional hydrodistillation

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method for the recovery of high quality essential oil from cardamom seeds.

2. Materials and methods

2.1. Plant material and chemicals

Green colored, dry cardamom seeds (*E. cardamomum* L. Maton) of Guatemala origin were obtained from the local market in Saudi Arabia. Reference compounds 1,8-cineole and α-terpinyl acetate were purchased from Sigma–Aldrich (Germany).

2.2. Isolation of essential oil

2.2.1. Hydrodistillation (HD)

The cardamom seeds (40 g) were separated from the husk, ground into a fine powder by a hammer mill and sieved through a 0.5 mm screen, immersed in 1 L water, placed in a 2 L round bottom flask. Distillation was carried out using a lighter than water Clevenger type apparatus for collecting the oil according to the European Pharmacopoeia (Conseil de l' Europe, 1996), until no more essential oil was obtained (technique 1). From the distillate, the oily layer was separated, dried over anhydrous sodium sulphate and stored in a dark glass bottle at 4 °C till analysis. Hydrodistillations were performed in triplicates and the mean values of the extraction yields were reported.

2.2.2. Ultrasound assisted extraction (UAE)

Forty grams of the powdered sample was weighed and placed in a 1 L beaker. The ultrasound extraction was carried out using solid to water ratio of 1:12 (w/v). The mixture was subjected to ultrasonic radiation using ultrasound generator (Fisher Sonic Dismemberator, Model 300, 50 Hz, USA), equipped with a 19 mm diameter tip, ultrasonic was used as a preliminary extraction step in the extraction process of essential oil from cardamom seeds. The ultrasound probe was immersed into the mixture at a depth of approximately 5 mm. The ultrasound assissted extraction was carried out at 10% of the maximal output power (30 W) for 15 and 30 min (techniques 2 and 3, respectively) and at 20% of the maximal output power (60 W) for 10 and 15 min (techniques 4 and 5, respectively), at room temperature. Ultrasonic energy introduced in the system was 27, 54, 36, and 54 KJ for techniques 2, 3, 4, and 5, respectively. After ultrasonic step, 500 mL of distilled water was added and the resulting mixture was immediately submitted to hydrodistillation in a Clevenger type apparatus (until no more essential oil was obtained). Each extraction was performed at least three times and the mean values of the extraction yields were reported.

2.3. Gas chromatography–mass spectrometry (GC–MS)

GC analysis was performed on a Shimadzu GCMS-QP 2010 Ultra gas chromatograph fitted with flame ionization detector (FID) and RTX–5 column (30 m × 0.25 mm, film thickness 0.25 μm) with helium as carrier gas at 1.33 ml/min. The injection port was maintained at 210 °C, the detector temperature was 230 °C. The split ratio was 1:10 and ionization voltage maintained at 70 eV. One μL sample was injected. The oven was programmed as follows: at 40 °C for 2 min and then increased to 210 °C at 5 °C/min at which the column was maintained for 5 min. Identification of volatiles was based on retention indices calculated by using *n*-hydrocarbons (C_9—C_{22}) and mass spectra by computerised matching of essential oil compounds with NIST 2008 and Wiley libraries. 1,8-Cineole and α-terpinyl acetate were confirmed and quantified by co-injection of external standard compounds. In order to evaluate the amount of 1,8-cineole and α-terpinyl acetate in samples, standards at different concentration levels were selected according to their relative amounts in the sample, ranging from 400 to 800 mg/dL. Linear calibration curves were obtained with correlation coefficients R^2 of 0.999 and 1.000 for α-terpinyl acetate and 1,8-cineole, respectively.

2.4. Sensory evaluation

Ten trained panelists conducted the sensory evaluation of essential oils. Randomly coded samples were individually served to panelists. Five descriptive adjectives warm spicy, pungent, aromatic, cooling taste and burnt note were submitted to these panelists. Marks were given from 0 to 10 for each adjective; 0 indicated that essential oil was not in accordance with the adjective and 10 that the essential oil was in perfect accordance with the descriptive adjective.

2.5. Statistical analysis

Results of the physical characteristics of cardamom seeds and yields are reported as mean ± standard deviation (SD). Data of essential oils recovery were analyzed using one way analysis of variance (ANOVA). Duncan's multiple range test was used to compare the significance of differences at $^*P < 0.05$.

3. Results and discussion

3.1. Physical characteristics of Guatemalan cardamom

Physical characteristics such as weight of 100 capsules, number of capsules in 100 g, the seed to husk ratio and length of capsules are given in Table 1.

These results are in accordance with those obtained by Kizhakkayil et al. (2006) for Guatemalan cardamom.

3.2. Extraction time, yield and chemical composition of the essential oil

The effect of extraction method on the total extraction time, yield and chemical composition are summarized in Table 2 and illustrated in Figs. 1 and 2 .

Concerning the comparison of the five techniques in terms of total extraction time and yield, combined UAE–HD techniques (ultrasound assisted extraction followed by hydrodistillation) were clearly fast (≤1 h) while 6 h were required for full extraction of essential oil from cardamom seeds by traditional hydrodistillation. Time of hydrodistillation with or without ultrasound assisted extraction was set when no more essential oil was obtained. The combined technique 3 provided significantly higher extraction yield compared with that obtained by HD. On the other hand, the ultimate yield of cardamom essential oil obtained by other combined UAE–HD techniques were not significantly different from that obtained by means of HD. Results are in agreement with those reported by Assami et al. (2012) who found that ultrasound treatment engenders a rapid release of essential oil after only 30 min of extraction for treated *Carum carvi* L. seeds versus 90 min for untreated seeds. Pingret et al. (2014) reported that the use of ultrasound allowed the recuperation of the same amount of orange

Table 1
Physical characteristics of Guatemalan cardamom.

Parameter	Cardamom seeds
Weight of 100 g capsules (g)	10.49 ± 0.25
No. of capsules in 100 g	931 ± 1.15
Seed husk ratio	2.2:1
Length of capsules (cm)	1.7 ± 0.0125