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Investigating the effects of grinding time and grinding load on content of terpenes in extract from fennel obtained by supercritical fluid extraction

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ABSTRACT

This paper investigated effects of grinding time (GT) and mass of raw material in mill (m_s) on both global SFE yield from Fennel and its main volatile oil content namely Anethol and Fenchone. For this purpose, extractor of SFE equipment was filled with milled Fennel obtained at various values of m_s, from 15 g to 35 g, and GT, from 15 s to 20 min. Extractor was subjected to pressure of 200 bar, temperature of 313 K, and supercritical CO_2 flow rate of 1.67×10^{-4} kg/s for 10 min. Then, extract composition was evaluated by Gas Chromatography (GC) analysis. Moreover, for better understanding how GT and m_s affect SFE yield and extract composition, their effects were initially investigated on temperature raising and diameter of Fennel seeds during grinding process. It was found that m_s and GT have considerable effects on Anethole and Fenchone content of Fennel extract.

1. Introduction

Fennel (Foeniculum vulgare) is a plant belonging to Apiaceae family, which is cultivated in several countries like Brazil, England, Germany, China and so on (Brender et al., 1997). Fennel extracts can be employed as antispasmodic, anti-inflammatory, expectorant, diuretic, and laxative (Jahromi et al., 2003). It also has applications in treatment of nervous disturbances, carminative, analgesic, and stimulant of gastrointestinal mobility (Jahromi et al., 2003). Anethole and Fenchone are two of the main ingredients in Fennel volatile oil that compose, respectively, 40–70% and 1–20% of it (Bernath et al., 1996; Coşge et al., 2008; Raghavan, 2006). Extraction of volatile oil from Fennel is generally carried out by several techniques such as SFE, liquid CO_2 extraction, Soxhlet extraction, accelerated solvent extraction, and steam distillation (Baby and Ranganathan, 2016; Rodríguez-Solana et al., 2014a,b; Bodsgard et al., 2016; Johner and Meireles, 2016). From them, SFE is usually a preferable technique due to its advantages such as high extraction performance, simple separation of solvent from extract, and its selectivity capability (Durante et al., 2014; Ding et al., 2017). SFE from Fennel have been reported in various research works. Moura et al. (2005) studied effect of harvesting season, degree of maturation, bed geometry, operating temperature and pressure on SFE yield from fennel. They also studied extraction of Fennel by hydrodistillation and low-pressure solvent extraction, and compared their results with SFE. They found that not only SFE process produced larger relative proportion of anethole and fenchone, but also its overall efficiency was 5.2 larger than that of hydrodistillation (Moura et al., 2005). In another relevant paper, Pereira and Meireles (2007) investigated economic analysis for obtaining extracts and essential oils from Rosemary, Fennel, and Anise using SFE and steam distillation. They concluded that in spite of high cost of SFE equipment, it was more economical than steam distillation due to higher quality of its product, higher extraction yield, and lower energy consumption (Pereira and Meireles, 2007). Another paper in this topic was written by Hammouda et al. (2014), whom studied extraction yield and extract quality from Fennel using SFE, microwave-assisted extraction (MAE), and hydrodistillation. Their study showed that MAE gave higher overall yield and higher percentage of Fenchone than others. SFE, on the other side, gave the maximum percentage of Anethol (Hammouda et al., 2014). In another interesting paper, Johner and Meireles (2016) constructed a laboratory SFE unit that contained one extractor and two separators. They validated the equipment using SFE from Annatto and Fennel. Fennel extract was obtained by employing 200 bar pressure and 313 K temperature in extractor, 80 bar and 312 K in first separator, 20 bar and 279 K in second separator, mass flow rate of 12 g/min, and solvent per feed ratio of 10. Their results indicated an overall SFE yield of 2.8 g extract/100 g of ground seeds. Moreover, they observed that 97.5% of the whole extract was collected in the first separator (Johner and Meireles, 2016). Reverchon et al. (1999) carried out successfully extraction of fennel seeds in two steps including SFE at 90 bar and 50 °C, and SFE at 200 bar and 40 °C using three CO_2 mass flow rates of 0.5, 1.0, and 1.5 kg/h. They also modeled the process based on mass

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conservation law and demonstrated the reliability of the model by comparing it with experimental data. Piras et al. (2014) employed SFE and hydrodistillation (HD) on Sardinian wild fennel and measured composition of volatile extracts using gas chromatography −mass spectrometry. They found that percentage of the main components in SFE extract were 7.1% (fenchone), 34.9% (estragole), and 24.6% ((E)-anethole), while corresponding number for HD extract were 8.8%, 42.6%, and 43.4%, respectively. Rodríguez-Solana et al. (2014b) performed Oleoresin extraction from fennel seeds using SFE and optimized the process for the aim of attaining maximum estragole per kg of dry plant. They found the optimal operating condition as the pressure of 24 MPa, temperature of 333.15 K, extraction duration of 3.41 h, and methanol percentage of 3%. In this condition, they obtained 1320 ± 260 mg of estragole per kg dry plant. Ivanovic et al. (2014) increased the yield of SFE (increase by up to 1350%) for several plant materials by employing different mechanical pretreatment such as flaking, impact plus shearing, and cutting plus grinding. They found Flaking as the most convenient method for increasing the yield of SFE from fennel seeds.

According to the above literature review, two of the attractive research areas in the field of SFE from Fennel are either maximizing overall yield or maximizing Anethole and Fenchone content of the volatile oil. The first necessary step for these goals is, however, grinding the Fennel seeds to increase their specific area. Although the effect of GT was already investigated on the SFE yield of other raw materials, they limited themselves to small GT. For example, Wilkinson et al. (2014) investigated the effect of GT on the SFE yield from soybeans at 48.3 MPa and 80 °C. They placed 60 g of soybeans in a mechanical grinder for different GT from 10 to 60 s. They concluded that increasing the GT enhances the extraction yield due to the reduction of particle sizes. The SFE yield was increased from around 6% to 22% by decreasing the particle diameters from 0.20 mm to 0.07 mm. In another study by Yahya et al. (2010), SFE yield from Pandan leaf increased up to 50% after a pretreatment grinding for 30 s. Grosso et al. (2008) evaluated the effects of mean particle size (0.4, 0.6 and 0.8 mm) together with the effects of pressure, temperature, and CO_2 flow rate on the yield and composition of SFE extract from Italian coriander seeds. They found that a decrease in particle size didn't have a considerable effect on the SFE volatiles composition, but it increased the SFE yield as more ducts were destroyed with longer milling time. Taking into account the yield and composition of the extract, they found the best operating conditions to be the pressure of 90 bar, temperature of 40 °C, CO_2 flow rate of 1.10 kg/h, and mean particle size of 0.6 mm. Shrigod et al. (2017) carried out SFE of mint leaves, and they investigated the effects of temperature (35–55 8C), pressure (100–300 bar), extraction time (20–90 min), and particle size (0.2–1.0 mm) on both SFE yield and carvone content in volatile oil. They found that SFE yield was mainly influenced by particle size followed by pressure, temperature, and extraction time, however, carvone content in extract was mostly affected by pressure followed by particle size and extraction time. The effect of particle size in SFE process was also studied by Sodeifian et al. (2016) and Chougle et al. (2016). Nevertheless, as the effect of milling process on yield and quality of SFE from Fennel were not discussed in literature, the current study aimed to focus on this area for a board range of GT and various m_s. Using higher GT not only affects the particle sizes, but it also heats up the raw materials and probably affects volatile components.

2. Material and methods

This section is organized as follows. First in subsection 2.1, the raw material and its pretreatment process are presented. In order to conceptual understanding how m_s and GT affects the SFE yield and extract composition, their effects should be firstly investigated on the temperature raising and diameter of Fennel seeds during grinding process. The method for doing this investigation is fully explained in subsection 2.2. Then, in subsection 2.3, the SFE equipment as well as the experiments procedure is discussed. Finally, a short description about GC analysis is given in subsection 2.4.

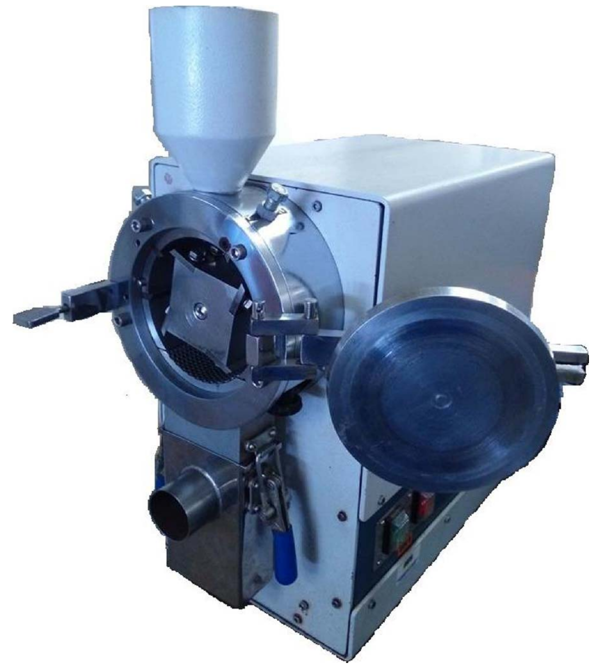

Fig. 1. Schematic diagram of the Mill.

2.1. Sample preparation

Dried Fennel seeds were supplied from a municipal market, called "Temperos Brasil", in Campinas, São Paulo, Brazil, and they were kept in a domestic freezer at 255 K. Prior to each grinding experiment, the raw material were taken out from the freezer to stabilize at the laboratory temperature, 297 K.

2.2. Grinding raw material

The Fennel seeds were ground in a mill (Marconi, model: MA 340, São Paulo, Brazil), which is shown in Fig. 1. Its main parts are a rotor, a crushing chamber, a collector container, and stainless steel knives (cutting edge). It has weight of 58 kg, dimensions of 27 × 48 × 50 cm, electrical power of 1600 W, and a fixed speed rotor of 1750 rpm.

An initial study was performed by using the Mill to determine the influence of m_s and GT on physical properties of Fennel. For this purpose, five levels of GT and three levels of m_s were considered. These levels are 15 s, 2 min, 6 min, 12 min, and 20 min for GT, and 15 g, 25 g, and 35 g for m_s. Each grinding process was performed two times to ensure that the obtained results are reliable. At the end of each run, Fennel temperatures were measured from the top of the crushing chamber by using an infrared thermometer with an uncertainty of 0.1 K. The idea of measuring the Fennel temperature in the central parts of the crushing chamber was not applicable as opening this chamber, for this purpose, took several seconds and, during this period, temperature decreased quickly. In the next step of the experiment, the milled Fennel was subjected to a vibratory agitator and sieves (Bertel, model MAGNETICO, Sao Paulo, Brazil) to determine the particle size distribution. In this step, six sieves with mesh sizes of 18, 25, 35, 50, 80, and 100 were employed, and they were vibrated for 15 min to ensure that the material in each sieve didn't change with time. After that, materials on each sieve and the bottom pan were accurately weighed and recorded. For calculating the mean particle diameter, the Standard ASAE method was used (Standard, 2003).