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A parametric investigation of castor oil (*Ricinus comminis* L) extraction using supercritical carbon dioxide *via* response surface optimization

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ABSTRACT

The optimal conditions of oil yield from castor (*Ricinus communis L*) seed using supercritical CO_2 as extracting solvent were studied. Response surface methodology (RSM) was employed to show explicitly the influence of the process parameters such as temperature, pressure and the CO_2 flow rate on the oil yield using Box–Behnken design. The linear terms of pressure, CO_2 flow rate and temperature and the quadratic terms of temperature, pressure and CO_2 flow rate, had a significant effect on the oil yield. The maximum oil yield obtained from the mathematical model was predicted to be 9.29% under the conditions of temperature 63.72 °C and pressure 29.90 MPa with CO_2 flow rate of 4.15 mL/min. The fatty acid constituents of the seed oil extracted using supercritical CO_2 were determined by gas chromatography–mass spectrometry (GC–MS) and Fourier transform infrared spectrometer (FTIR). Palmitic, stearic, oleic, linoleic, linolenic and ricinoleic acids were identified by GC–MS analysis after the formation of fatty acid methyl ester (FAME).

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1. Introduction

Castor (*Ricinus communis L*) is grown mainly in the Tropics and the Mediterranean region [1]. The resemblance of its leaves to the palm of the hand led to the plant being named *Kiki* by the Romans. Castor is the oldest cultivated crop and presently it represents about 0.15% of the vegetable oil produced globally. The oil is of great importance to the chemical industry due to its commercial source of hydroxylated fatty acid. Castor oil is unique with an exceptional variety of applications industrially, its ability to grow in drought and saline conditions [2].

Industrial seed oils are generally obtained through both mechanical and chemical separation processes [3–5]. Mechanical separation process is often associated with low yields as against chemical separation process which has (>99 wt%) of oil yield [6,7]. Although chemical separation process such as extraction methods in most cases employ solvents such as *n*-hexane. However, its volatility is high and toxic in nature at relatively low concentration. The vapors need to be monitored as uncontrolled amount could lead to explosion during industrial oil extraction. Moreover, conventional extraction methods are time consuming, laborious, has no selectivity and has low extraction yields, has no fractionation capabilities, leaves solvent residue in the product, allows manipulations of limited variables, damage the heat-sensitive components of the materials and the requirement of post-extraction process for solvent removal [8–12]. Supercritical fluid extraction (SFE) is a technique that can overcome these drawbacks of the conventional solvent extraction process [13]. The negligible environmental impact of this process represents a prospect for changing the relative concentration of the various lipid moieties [14]. SFE is fast becoming a powerful means of extraction of solid samples especially seeds oil. It can be considered a technological revolution in the extraction industry [15].

The most widely used supercritical fluid is CO_2, which is non-toxic, relatively inert, and non-flammable; it is considered as a GRAS (generally recognized as safe) solvent and available as a by-product of the chemical industry. SC-CO_2 extraction allows a constant modification in dissolution power as a result of changes in the solvent density [16]. Moreover, CO_2 can be used to extract thermolabile compounds due to its low critical temperature (304.21 K). Finally, another interesting property is that CO_2 is gaseous at ambient conditions of temperature and pressure thus allowing a spontaneous and complete separation. At industrial scale, CO_2 is recycled, hence enabling a clean and compact operation [17].

Although few studies have been conducted on the use of SC-CO_2 extraction of castor oil [18], however, the versatility of the oil is a

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major factor for researchers to carry out detailed studies because the extraction conditions have not yet been optimized fully. In general, many factors, such as temperature, pressure and the CO_2 flow rate can affect the efficiency of SC-CO_2 extraction. Moreover, where multiple variables may influence the oil yield, the response surface methodology (RSM) is an effective statistical tool for optimizing the process [19,20]. Thus, the present study is to evaluate the effects of the extraction conditions, *i.e.* temperature, pressure and the CO_2 flow rate on the oil yield and to optimize these conditions using RSM. The fatty acid compositions and functional groups of the castor oil were also analyzed by gas chromatography–mass spectrometry (GC–MS) and Fourier transform infrared spectroscopy (FTIR) respectively.

2. Material and methods

2.1. Materials

The castor bean seeds were purchased from Ancient Greenfield Pvt. Ltd, India. CO_2 (purity 99.99%), contained in a cylinder, was supplied by the MOX Co. (Petaling Jaya, Malaysia). All chemicals and solvents used were of either analytical grade or GC grade and were purchased from Fisher Scientific Chemical (Loughborough, UK) and Merck (Darmstadt, Germany).

2.2. Preparation of the sample

The encased seeds were air dried for 24 h until the casing split and the seeds were shed. The shells were then separated from the nib by lateral airflow. The cleaned seeds were further dried for 5 h until a constant weight was achieved. The moisture content was then calculated using Eq. (1).

$$\%\,\text{Moisture} = \frac{W_1 - W_2}{W_2} \times (100) \tag{1}$$

where W_1 and W_2 are the original and final weights of samples before and after drying respectively. The dried materials were crushed and the crushed seeds were separated into a nominal, *i.e.* particle sizes 1 mm using a vibrator sieve. The particles were stored in a refrigerator at −4 °C before extraction.

2.3. Extraction of castor seed oil using supercritical carbon dioxide extraction

The SFE analyses were carried out using a CO_2 HPLC pumps (Lab Alliance SFT-24). The CO_2 was delivered from the supply tank to a cooling jacket fitted to the CO_2 HPLC pump. A mixture of 1:1 (v/v) ground (5 g) of castor seeds and (1 mm diameter) glass beads was placed in a 50 mL stainless steel extraction vessel (Model EV-3, Jasco Corporation, Japan) in each experiment and the vessel was placed in an oven at selected temperature. Carbon dioxide flowed through the preheating 4 m length coil and it was ensured that the desired temperature was attained before contacting the sample in the extraction vessel. A back pressure regulator (BPR) (Model BP-2080, Jasco Corporation, Japan) was used to control the extraction pressure. The CO_2 flow rate was maintained at 3, 4 and 5 mL/min and the extraction time was measured using a stop watch. Operational conditions such as temperature, pressure, and flow rate were chosen according to the preliminary study. The extraction was then conducted in accordance with the Box–Behnken design of the RSM. The dynamic extractions were initiated when the system reached a pre-determined pressure and temperature. The dynamic extraction was conducted for 90 min. A 5 mL vial was utilized as a collection vessel for the extracts. The oil was then refrigerated at −4 °C after extraction. The schematic diagram of the experimental setup for the supercritical carbon dioxide is shown in Fig. 1.

2.4. Yield calculation

Extraction yields were measured gravimetrically by collecting the oils precipitated at the collection vial. The oil yield was determined according to the following equation [21]:

$$\text{Oil yield}\,(\%) = \frac{\text{Weight of extracted oil}}{\text{Weight of castor seed}} \times 100 \tag{2}$$

2.5. Fatty acid analysis

The fatty acid compositions were estimated by preparing fatty acid methyl esters in methanol by GC–MS. To analyze the FAME a GC, Agilent 6890 N (Agilent Technologies, Wilmington, USA) was used. A helium carrier gas (1.0 mL/min) was used. The injector and detector temperature were programmed at 260 °C. The column temperature was also programmed at 140 °C for 5 min. The oven temperature was at first maintained at 140 °C and raised to 240 °C at 4 °C/min and finally maintained at 240 °C for 14 min. The column was then injected with 1 μL of the FAME, and the identified fatty acids were compared with the retention times of the national institute of standards and technology (NIST) library.

2.6. FTIR spectroscopy

Surface functional groups were determined using FTIR spectrometer (Perkin Elmer C72956/US) equipped with a beam splitter and

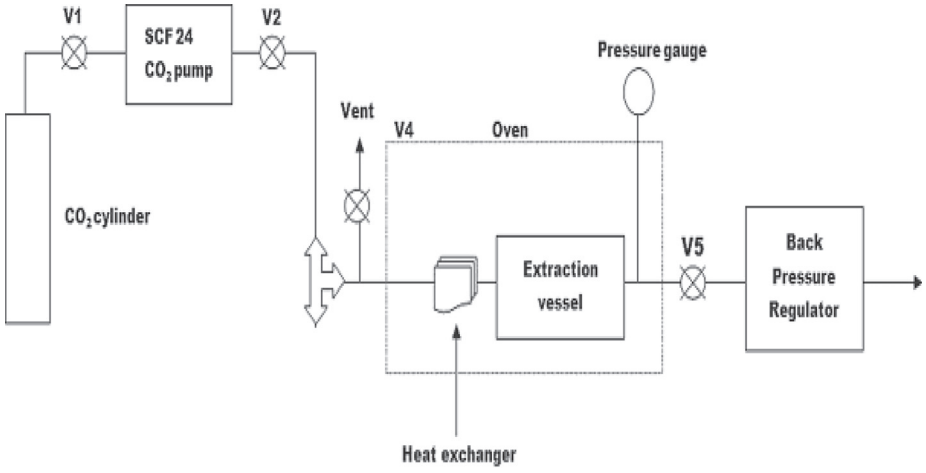

Fig. 1. Schematic diagram of supercritical carbon dioxide extraction.

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