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The application of ultrasound and microwave to increase oil extraction from *Moringa oleifera* seeds

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ABSTRACT

Microwave-assisted extraction (MAE) and ultrasound-assisted extraction (UAE) are novel methods, which can reduce the extraction time. However, little information is known regarding the effect of UAE and MAE on the oil extraction from *Moringa oleifera* seeds as well as oil quality. This study aimed to evaluate the influence of MAE and UAE on oxidative stability and physicochemical properties of oil from *Moringa oleifera* seeds. The UAE and MAE showed higher oil recoveries ranged 91%–94% in short extraction duration as compared with conventional extraction (CE) which gave 90%. There were no significant differences in fatty acid composition, acylglycerol profile and thermal properties of oils among the extraction techniques (UAE, MAE, and CE). Furthermore, the UAE and MAE did not greatly lower the oxidative stability of the oils during the whole 70 days of storage time at 25 °C. Therefore, the UAE and MAE could reduce extraction time without greatly affecting oil quality. This study provides important information about developing UAE and MAE techniques for producing high yield, short extraction duration, desirable physicochemical properties and acceptable stability of Moringa oil.

1. Introduction

Moringa oleifera is an Indian tree, now widely cultivated in many countries of Asia, South America, and Africa (Dalei et al., 2016). As a traditionally important food commodity, *Moringa oleifera* has received attention as "natural nutrition of the tropics". Almost every part (i.e., flowers, seeds, leaves and fruits) of this plant is traditionally used for dietary purpose or various ailments (Anwar et al., 2007a). Besides, the kernels of *Moringa oleifera* seeds contain 35%–40% oil wherein oleic acid is the main fatty acid (up to 78%) (Nadeem and Imran, 2016). The Moringa oil produced from *Moringa oleifera* seeds is similar to olive oil in terms of oleic acid. Oleic acid-rich olive oil has received great attention because of its high oxidative stability (Gómez-Rico et al., 2007) and reduction in the risk of cardiovascular diseases (Schwingshackl and Hoffmann, 2014). Similarly, Moringa oil is potentially a new source for producing oleic acid-type vegetable oil (Ruttarattanamongkol et al., 2014). Interestingly, Moringa oil has a lower content of linoleic acid (< 4.2%) than common vegetable oils (such as soybean, palm and canola oils) (Nadeem and Imran, 2016). Recent studies showed that Moringa oil exhibited better frying performance over some conventional frying oils (such as canola, palm and soybean oils) due to its good oxidative stability (Abdulkarim et al., 2007; Anwar et al., 2007b). Moringa oil becomes popular and is suitable for both edible and non-edible applications (such as cooking oil, biodiesel, and cosmetics), due to its healthful properties and excellent oxidative stability.

Oil extraction from *Moringa oleifera* seeds is performed using various techniques such as solvent extraction, enzyme-assisted aqueous extraction, and supercritical carbon dioxide extraction (SC-CO_2) (Bhutada et al., 2016; Latif et al., 2011; Ruttarattanamongkol et al., 2014). Among the methods, the solvent extraction technique is a conventional technique used for vegetable oils extraction. The Soxhlet technique, a conventional solvent extraction method, is widely used to extract oil from *Moringa oleifera* seeds for characterizing the physicochemical prosperities of it (Anwar and Rashid, 2007). However, the application of Soxhlet technique for the large-scale production of Moringa oil is limited due to its long extraction time (> 7 h), possible degradations of labile compounds and limited solvent choice (Arias et al., 2009; Bhutada et al., 2016; Mani et al., 2007). Up to now, little information is available on producing Moringa oil using other solvent extraction techniques such as mechanical stirring-assisted solvent extraction (also named conventional solvent extraction, CE), ultrasound-assisted solvent extraction (UAE) and microwave-assisted solvent extraction (MAE). In this context, it is a challenge for researchers to explore and develop more effective approaches for Moringa oil extraction.

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Meanwhile, microwave and ultrasound technologies have shown to be efficient means for accelerating solvent extraction from plant matrices and requiring less solvent (Mason et al., 2011). However, the oxidative stability and physicochemical properties of oils from various plant matrices are impacted by microwave and ultrasound effect (Samaram et al., 2015; Wroniak et al., 2016). It is unclear how these technologies influence the oxidative stability and physicochemical properties of oil from *Moringa oleifera* seeds.

Therefore, the objective of the present investigation was to evaluate the suitability of UAE and MAE as compared with CE technique for oil recovery from *Moringa oleifera* seeds. Firstly, the effects of various parameters of UAE, MAE and CE were investigated on oil yield. Secondly, the effects of various oil extraction techniques were evaluated on the oil yield, fatty acid composition, oxidative stability and other physicochemical properties of Moringa oil. Finally, morphological changes of *Moringa oleifera* seed sample before and after extraction were observed in order to clarify the extraction mechanism.

2. Materials and methods

2.1. Sample preparation and reagents

Moringa oleifera seeds (moisture content 5.10 ± 0.04%) imported from India were supplied from local market. All the samples were dehulled, winnowed and the obtained meat was crushed into powder with particle sizes between 0.18 to 2.00 mm. The resulted powder was used for oil extraction. Petroleum ether was supplied from Chengdu Kelong Chemical Reagent Co., Ltd. (Sichuan, China). All other chemicals were of analytical or chromatographic grade.

2.2. Oil extraction methods

2.2.1. Soxhlet extraction

The total oil content of *Moringa oleifera* seeds was determined by Soxhlet extraction, according to the standard method GB 5009.6 (2016c). Briefly, petroleum ether (boiling point 30–60 °C) and 4 g of the *Moringa oleifera* seeds powder (< 0.43 mm) were heated to reflux for 8 h.

2.2.2. Conventional solvent extraction (CE)

To optimize the process conditions for maximum yield of extraction, 10 g of the powder sample was mixed with petroleum ether (boiling point 60–90 °C) in a conical flask. The mixture was stirred on a DF-101S mechanical stirrer (Gongyi Yuhua Instrument Co., Ltd., China) at 325 r/min at different temperatures for different periods. The supernatant was separated from the residue by a vacuum pump. The obtained liquid was evaporated at 40 °C under vacuum. The obtained oil sample was then placed in an oven at 100 °C until constant weight was achieved. The effects of main factors (sample size varying from 0.18 to 2.00 mm, liquid/solid ratio varying from 4:1 to 10:1, extraction temperature varying from 30 to 50 °C, and extraction time varying from 10 to 70 min) on oil yield were investigated. The oil yield and oil recovery were calculated using Eqs. (1) and (2), respectively.

$$\text{Oil yield } (\%) = \frac{m_1}{m_2} \times 100 \tag{1}$$

$$\text{Oil recovery } (\%) = \frac{m_1}{m_3} \times 100 \tag{2}$$

where m_1 is the weight (g) of oil extracted from the seed powder processed, m_2 is the weight (g) of the seed powder processed, m_3 is the weight (g) of oil extracted by the Soxhlet extraction.

Moringa oil was extracted by CE under optimized conditions (liquid/solid ratio of 8:1, stirring speed of 325 r/min, extraction temperature of 30 °C, extraction time of 50 min), in order to compare the effects of various extraction techniques on the physicochemical properties and oxidative stability of the extracted oil. After evaporation, the oil was purged with nitrogen to further remove the residual solvent and was then stored under nitrogen at −20 °C until use.

2.2.3. Ultrasound-assisted solvent extraction (UAE)

For each extraction, 10 g of *Moringa oleifera* seeds powder and 80 mL of petroleum ether (boiling point 60–90 °C) were added to a flask. The mixture was treated for varying ultrasound time (5, 10, and 15 min) in an ultrasonic bath (KQ 500DV, Kunshan Ultrasonic instruments Co., Ltd., Kunshan, China) with a power of 200 W and frequency of 40 kHz. The temperature of the bath was set at 30 ± 1 °C and was monitored by a thermometer. No increase in temperature of the bath was observed during ultrasound. Then, the mixture was stirred on a DF-101S mechanical stirrer at 325 r/min at 30 °C for 15 min. After the extraction period, the supernatant was separated from the residue by a vacuum pump. The obtained liquid was evaporated at 40 °C under vacuum. The obtained oil sample was then placed in an oven at 100 °C until constant weight was achieved.

Moringa oil was extracted by UAE under optimized conditions (ultrasound time of 5 min, ultrasound power of 200 W, and ultrasound temperature of 30 °C, followed by mechanical stirring of 15 min at 30 °C), in order to compare the effects of various extraction techniques on the physicochemical properties and oxidative stability of Moringa oil. After evaporation, the oil was purged with nitrogen to further remove residual solvent and was then stored under nitrogen at −20 °C until use.

2.2.4. Microwave-assisted solvent extraction (MAE)

A conical flask containing petroleum ether (boiling point 60–90 °C) and *Moringa oleifera* seed powder (10 g) was placed in a MAS-II microwave oven (Sineo Microwave Equipment Co., Ltd., Shanghai, China). After extraction, the mixture was then filtered by a vacuum pump. The obtained liquid was evaporated at 40 °C under vacuum to obtain the oil phase. The obtained oil sample was then placed in an oven at 100 °C until constant weight was achieved. In determining the effect of MAE on oil yield, experiments were accomplished using microwave power ranging from 300 to 900 W, liquid/solid ratio ranging from 4:1 to 10:1, particle size of the seed powder ranging from 0.18–2.00 mm and microwave time ranging from 5 to 13 min. For each microwave time, it was not carried out by continuous microwaving but with 5 times intervals and there is a resting time of 30 s between intervals.

Moringa oil was extracted by MAE under optimized conditions (liquid/solid ratio of 8:1, microwave power of 300 W for 9 min), to compare the effects of various extraction techniques on the physicochemical properties and oxidative stability of Moringa oil. After evaporation, the oil was purged with nitrogen to further remove residual solvent and was then stored under nitrogen at −20 °C until use.

2.3. Measurement of oil color

The color of Moringa oil samples obtained from any of the extraction techniques was determined using a spectrocolorimeter (UltraScan PRO, Hunter Associates Laboratory, USA). Each oil sample was poured into a 1-cm quartz cuvette. Under the tristimulus color coordinate system, the L^* value indicates the lightness of a color from 0 (black) to 100 (white), a^* value varies from negative to positive (green to red), and b^* value varies from negative to positive (blue to yellow).

2.4. Fatty acid composition analysis

Oil samples obtained from various extraction techniques were converted to corresponding fatty acid methyl ester by reacting with KOH-methanol solution. The KOH-catalyzed methylation was carried out according to the method of Liu (1994) with some modifications. Briefly, about 10 μL of oil sample was dissolved in 2 mL of hexane.