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Ultrasound-assisted extraction of oil from flaxseed

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Abstract

Flaxseed oil is the main component of the flaxseed and has many beneficial functions to human health. In this paper, the ultrasound-assisted extraction of oil from flaxseed is reported. The effects of some operating parameters such as ultrasonic power, extraction time, extraction temperature and solvent to solid ratio on the yield of flaxseed oil have been investigated and some of the results have been compared with that of conventional method. It has been found that ultrasound-assisted extraction requires a shorter extraction time and a reduced solvent consumption. The yield of flaxseed oil has been found to increase with the increase of the ultrasonic power and to decrease as the temperature is increased. Scanning electronic microscopy analysis was carried out on the flaxseed powder after the extraction. The images are powerful evidences to show the effect of ultrasound. The fatty acid compositions of the oils extracted by the ultrasound-assisted method and the conventional method have been analyzed using gas chromatography. It has been shown that the compositions of the flaxseed oils were not affected significantly by the application of ultrasound ($p > 0.05$). The ultrasound-assisted extraction may be an effective method for lipid production.

Keywords: Extraction; Ultrasound-assisted; Oil; Flaxseed

1. Introduction

Flaxseed, which is also called linseed, is an important oilseed in the world. It is mainly grown in Canada, Argentina, America, China and India [1]. Usually, flaxseed contains about 40% oil, 30% dietary fiber, 20% protein, 4% ash, and 6% moisture [2]. In the past, flaxseed was mainly used to provide flaxseed oil for industrial purposes such as the production of paints, linoleum, varnishes, inks and cosmetics [3,4]. However, flaxseed is playing an important role in functional foods for its nutritional and pharmaceutical values. The nutritional components of flaxseed are oil, protein, lignans, soluble fiber, minerals and vitamin, etc. [5]. Flaxseed is known as the richest source of the *n*-3 fatty acid, alpha linolenic acid (ALA), which comprises approximately 55% of the total fatty acids and this percentage is 5.5 times higher than that in the next-highest sources [6]. ALA can be metabolized to eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA) in human intestine due to the action of some kinds of enzyme [7]. It is well established, in human bodies, that an increase in the ingestion of long-chain poly-unsaturated fatty acids (LC-PUFA), especially EPA and DHA, in diet can reduce the risk of diseases [8]. Previous studies have proven that flaxseed oil has positive effect on the minimization of many diseases such as hyperlipidemia [9], colon tumor [10], mammary cancer [11–13] and atherosclerosis [14,15].

At present, flaxseed oil is mainly extracted from flaxseed by press extraction and solvent extraction. Press extraction is often associated with lower yield and more energy consumption, while solvent extraction often involves longer extraction time. The use of large quantities of organic solvents is not desirable and can be harmful to human and environment. Moreover, flaxseed oil is thermally unstable and may degrade at higher temperatures. For these reasons, an improved or better extraction technique is desirable. Thus, supercritical fluid extraction, aqueous enzymatic extraction and ultrasound-assisted extraction (UAE) techniques can be further developed.

Recent studies have shown that the ultrasound-assisted extraction can enhance the extraction efficiency through acoustic cavitation and some mechanical effects [16–18]. Acoustic cavitation can disrupt cell walls facilitating solvent to penetrate into the plant material and allowing the intracellular product

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release. Another mechanical effect caused by ultrasound may also be the agitation of the solvent used for extraction, thus increasing the contact surface area between the solvent and targeted compounds by permitting greater penetration of solvent into the sample matrix. Therefore the main advantages of ultrasound-assisted extraction include the reduced extraction time and reduced solvent consumption [19]. In addition, ultrasound-assisted extraction can be carried out at a lower temperature which can avoid thermal damage to the extracts and minimize the loss of bioactive compounds [20].

So far, there seem to be no report about ultrasound-assisted extraction oil from flaxseed. The aim of this study was to determine the effects of some factors on the yield of flaxseed oil with or without ultrasound treatment. The results from UAE were compared with that obtained from the conventional solvent extraction, i.e. maceration extraction (ME). After the extraction of flaxseed oil, microscopic images of flaxseed powder were obtained by scanning electron microscope (SEM) to prove the existence of ultrasonic effects. The fatty acid compositions of flaxseed oils extracted by UAE and ME were measured using gas chromatography (GC) to investigate the effect of ultrasound on the quality of flaxseed oil extracted.

2. Materials and methods

2.1. Samples and reagents

The flaxseed with a moisture content of 6.2 wt% was purchased from the market of Hebei Province of China. The flaxseed was cleaned by hand carefully to remove the foreign materials such as other seeds, stones and small stalks. The cleaned flaxseed was dried for 12 h at 105 °C in an oven, and then crushed into powder in a grinder (B-400, Büchi Labortechnik AG, Switzerland) with a size range of 0.45–1.2 mm. The resulted powder was kept in a vacuum dryer until use. *n*-Hexane used in the extraction was analytical grade and was purchased from Beijing Beihua Chemical Company.

2.2. Conventional extraction

The crushed flaxseed powder (10 g) was mixed with *n*-hexane in a flask. The flask was put into the water bath with a controlled temperature. After each extraction experiment, the extracts were filtered through the Whatman No. 1 filter paper under vacuum, and then the solution was collected and concentrated with a rotary evaporator (RE-2S, Beijing Jing Zhi Jie Laboratory Apparatus Co. Ltd., China) to acquire the flaxseed oil. The acquired flaxseed oil was further dried in a vacuum dryer to remove the residual *n*-hexane.

2.3. Ultrasound-assisted extraction

For the UAE experiments, a 250 W, 20 kHz ultrasonic emulsifier (88-1, Institute of Acoustics, Chinese Academy of Sciences, China) with a 2.00 cm flat tip probe was used (Fig. 1). The ultrasonic output power could be set to a desired level ranging from 0 to 100% of the nominal power by the amplitude controller. Ultrasonic output powers were determined calorimetrically and ranged from 10 to 50 W according to the method described by Li et al. [16]. The ultrasound-assisted extraction used in this study was similar to that described by Zhao et al. [21], with a little modification. The flaxseed powder was mixed in 100 mL *n*-hexane contained in a 200 mL plastic beaker. The ultrasonic probe was inserted into the mixture directly. The samples were extracted under continuous ultrasonic waves at 20 kHz at different levels of power output. During extraction, the temperature was controlled at a desired level within ±1 °C. The post-treatment of the extracts was the same as that mentioned in the conventional extraction. In this study, all the experiments were performed in triplicates, and the results reported here are the means of the three trials.

2.4. Yield determination

The yield of flaxseed oil was calculated using the following formula:

$$\text{yield}\ (\%) = \frac{W_e}{W_t} \times 100$$

where W_e is the mass of flaxseed oil extracted from the sample (g) and W_t is the mass of total oil in the sample (g). W_t was obtained by Soxhlet standard extraction mode of the Buchi extraction system (B-811, Büchi Labortechnik AG, Switzerland) after hydrolyzing for 30 min in 4 M HCl in a hydrolysis Unit (B-411, Büchi Labortechnik AG, Switzerland).

2.5. Scanning electron microscopy (SEM) analysis

SEM can be used to reveal the microstructure of the material [22]. In order to investigate the influence of ultrasound during extraction on the structure of the materials and to understand the extraction mechanism, the solid remained was collected and dried in air after the extraction of flaxseed oil for the SEM analysis. Sample particles were fixed on the silicon wafer and sputtered with gold to a thickness of about 100 nm. The shape and the surface characters of the samples were observed and recorded on the scanning electron microscope (KYKY-2800, KYKY Technology Development Ltd., China).

2.6. Gas chromatographic analysis

Fatty acid compositions of the oils extracted by UAE and ME were determined using GC after derivatization to fatty acid methyl esters (FAME). The preparation of FAME was performed via saponification in 0.5 M NaOH–MeOH solution and methylation with 14% BF_3–MeOH (Sigma, USA), according to the 5509 ISO method [23]. FAME separation and identification were carried out on the gas chromatograph (6890N, Agilent Technologies, USA) equipped with a flame ionization detector and capillary column HP-Innowax (30 m × 0.32 mm × 0.25 μm). The amount of each sample injected was 1.0 μL. Nitrogen, at a constant flow 1.0 mL/min, was used as the carrier gas and a spilt/spiltless injector was used with a split ratio of 50:1. The injector temperature was 250 °C and the detector temperature