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Optimization of microwave-assisted extraction of oil from tiger nut (*Cyperus esculentus* L.) and its quality evaluation

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ABSTRACT

In this study, Microwave-assisted extraction (MAE) was firstly applied in extraction of tiger nut oil (TNO). Parameters of MAE were optimized by response surface methodology. The optimum extraction conditions were a mixture of petroleum ether (boiling range 60–90 °C) and acetone (2:1, v/v), microwave power 420 W, temperature 75 °C, liquid to solid ratio 7.0 mL/g and time 55 min, which could obtain yield of 24.12%. The fatty acid composition of oil by MAE was similar to that of oil by Soxhlet extraction (SE), whereas oil by MAE exhibited superior physicochemical properties and oxidation stability. Moreover, oil by MAE had a higher content of total phenolic, α-tocopherol, β-carotene, phospholipids and phytosterols. Scanning electron microscopy demonstrated that structural disruption of tiger nut caused by microwave irradiation was the main reason for rapid extraction. So MAE could be a good alternative for extraction of TNO.

1. Introduction

Tiger nut (*Cyperus esculentus* L.), belonging to the Cyperaceae family, is a perennial herb from rhizomes with hard tubers at its endings. It is also known as yellow nut sedge, earth almond, zulu nuts and chufa. Its origin can be traced back to 4000 years ago, while today it is extensively cultivated in many countries and has also became daily diet in Spain and North Africa (Oderinde and Tairu, 1988; Adel et al., 2015). Tiger nut was introduced to China in 1950s and now mainly cultivated in the middle and northern regions of China. Tiger nut is easy to grow on sand dunes or acidic soils and its average output per mu has reached 900 kg, taking only 90–110 days to mature (Qu et al., 2007). Tiger nut is rich in carbohydrate and fat, particularly the fat constituting about 25–33% of the total dry weight. Tiger nut oil (TNO) contains a lot of unsaturated fatty acids, vitamin E and phenolic compounds (Sánchez-Zapata et al., 2012).

At present, the energy crisis will be a great challenge in the middle of this century. Strengthening the utilization of renewable energy is an effective measure to deal with the increasingly serious energy and environmental problems. Biodiesel is an important renewable energy (Zhang et al., 2016). In China, natural reserves of oil sources are limited, and each year more than thirty million tons of crude oil are imported in recent past. It is crucial to develop biodiesel to meet energy demand. Tiger nut possesses the characteristics of strong environmental adaptability, short growth cycle, high oil production and so on. The viscosity, specific gravity, energy content and iodine value of TNO were comparable with those of soybean oil, sunflower oil and rapeseed oil. (Zhang et al., 1996; Qu et al., 2008). So TNO has been used as an important raw material for biodiesel production in China (Lu et al., 2007).

Generally, the extraction of TNO is conducted by methods, such as expeller pressing extraction, organic solvent extraction, aqueous enzymatic extraction and supercritical carbon dioxide ($SC–CO_2$) extraction (Li and Jing, 2014; Yeboah et al., 2012; Lasekan and Abdulkarim, 2012). Among these methods, the yield of expeller pressing extraction is comparatively low, whereas organic solvent extraction and aqueous enzymatic extraction take long extraction time. Moreover, owning to complexity in operation and high cost of equipment, $SC–CO_2$ extraction can not extensively be applied to practice (Mason et al., 2011). Therefore, it is important to explore a method of extracting TNO which can generate high efficiency and ensure the oil quality. Such a method is the use of microwave-assisted extraction (MAE) of TNO. Recently, MAE, a comparatively new method that combines microwave irradiation with organic solvent extraction, has attracted considerable attention due to its shorter extraction time, lower solvent consumption and higher extraction yield (Taghvaei et al., 2014; Nde et al., 2016; Yanık, 2017).

Up to our knowledge, the use of microwave to extract TNO has not been reported yet. Therefore, the aim of present study is to investigate

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the effects of operating condition, namely microwave power, temperature, liquid to solid ratio and time, on oil yields with response surface methodology (RSM). Subsequently, the physicochemical properties, fatty acid compositions and bioactive compounds of oil by MAE are also evaluated and compared with those of oil by Soxhlet extraction. Additionally, the microscopic structural changes of sample before and after extraction are observed to understand the characteristics of different extraction methods.

2. Materials and methods

2.1. Sample preparation and reagents

The tiger nut (*Cyperus esculentus*) was collected from wet market in Baoding district (Hebei Province, China) in October 2015. According to the retailer, the nuts were grown in the locality. The Samples were dried to moisture content of less than 5% with a vacuum drier at 75 °C and 25 kPa, pulverized by a disintegrator, and then sieved into homogeneous size (40 mesh). The powders were stored in the refrigerator before extraction.

Pure standards of Folin-Ciocalteu's reagent, α-tocopherol, *β*-carotene, *5α*-cholestane, and fatty acid methyl ester (FAME) standards were purchased from Sigma-Aldrich Co. (St. Louis, MO, USA). Other reagents from analytical to chromatographic grade were purchased from Chendu medicine group Co., Ltd (Chengdu, China).

2.2. Soxhlet extraction (SE) process

SE was implemented in accordance with the official method for named determination of oil content in oilseeds by the Association of Official Analytical Chemists (AOAC) Am 2-93. 20 g of tiger nut powder were extracted with 200 mL of petroleum ether (boiling range 60–90 °C) in a Soxhlet extractor at 80 °C for 6 h. Petroleum ether was removed at 50 °C under reduced pressure with a rotary evaporator. The oil was dried at 60 °C under vacuum until the weight remained constant. The amount of extracted oil was gravimetrically determined after collection. This method produced 30.05 ± 1.52 g of oil per 100 g of tiger nut powder. The oil was stored in a refrigerator at 4 °C for further analysis.

2.3. Microwave-assisted extraction (MAE) process

The microwave extraction apparatus (XO-SM50, Nanjing, China) is equipped with a multicolored liquid-crystal screen, a time controller, a temperature sensor, a power regulator and a circulating water-cooling system. The range of variables about microwave power was from 0 W to 700 W with 2450 MHz microwave frequency. During the experiment, power, temperature and time were controlled through an electronic control panel. The MAE was as follow: tiger nut powder (20 g) and different volume of extraction solvent (including petroleum ether, *n*-hexane, diethyl ether, ethyl acetate, ethanol, acetone and mixed solvent) were added into a flask. The flask was placed in the microwave oven cavity and connected to the cooling system through a hole at the top of microwave extraction apparatus. Then, the microwave extraction apparatus was turned on, and the experimental conditions including microwave power, extraction temperature and time were set by the digital panel. After extraction, the solvent was distilled with a rotary evaporator at 50 °C under reduced pressure. A small amount of residual solvent was removed by vacuum dryer at 60 °C until the weight of oil remained constant. The amount of extracted oil was quantified by gravimetry, and the extraction yield was calculated according to the equation given:

$$\text{Extraction yield of oil (\%)} = \left(\frac{\text{mass of extracted oil}}{\text{mass of dreid material}}\right) \times 100$$

2.4. Experimental design of MAE

RSM with a four-factor, three-level Box-Behnken design (BBD) was used to optimize variables in extraction of TNO. Based on the result of preliminary experiments (data not shown), the independent variables were microwave power (X1: 250–450 W), temperature (X2: 65–85 °C), liquid to solid ratio (X3: 4–8 mL/g) and time (X4: 40–60 min), and the response variable was the extraction yield of oil. The variables were transferred to a range between 1 and −1 for the appraisals of each factor. The independent variables were coded by the following equation:

$$Z_{0,j} = (Z_{1j} + Z_{2,j})/2$$

$$\varDelta_j = Z_{2,j} - Z_{0,j}$$

$$X_j = (Z_j - z_{0j})/\Delta_j, \quad j = 1, 2, 3, 4.$$

where Z_{1j} was the real value of the independent variable *j* at low level point. Z_{2j} was the real value of the independent variable *j* at high level point. Z_{0j} was the real value of the independent variable *j* at the center point. $\varDelta_j$ was the change interval of the real value of variable *j* at high level point and low level point. Z_j was the real value of the independent variable *j*. X_j was the coded value of the independent variable *j* by linear transformation. *j* was the experimental variable. The coded and actual levels of the independent variables used in the experimental design were shown in Table 1. A total of 29 randomized experiment including 24 factorial and 5 replicates at the center point tests were designed to fit the quadratic polynomial equation. The general equation was as follows:

Table 1
Results of BBD design for the microwave-assisted extraction of tiger nut oil[a].

Runs	Microwave power (X1, W)	Microwave temperature (X2, °C)	Liquid to solid ratio (X3, mL/g)	Microwave time (X4, time)	Extraction yield of oil (Y, %)
1	1(450)	1(85)	0(6)	0(50)	21.47 ± 0.18
2	1(450)	−1(65)	0(6)	0(50)	20.19 ± 0.10
3	1(450)	0(75)	1(8)	0(50)	22.86 ± 0.21
4	1(450)	0(75)	−1(4)	0(50)	17.68 ± 0.08
5	1(450)	0(75)	0(6)	1(60)	22.56 ± 0.26
6	1(450)	0(75)	0(6)	−1(40)	18.35 ± 0.14
7	−1(250)	−1(65)	0(6)	0(50)	19.25 ± 0.17
8	−1(250)	1(85)	0(6)	0(50)	18.23 ± 0.15
9	−1(250)	0(75)	−1(4)	0(50)	17.64 ± 0.23
10	−1(250)	0(75)	1(8)	0(50)	19.18 ± 0.21
11	−1(250)	0(75)	0(6)	1(60)	20.12 ± 0.19
12	−1(250)	0(75)	0(6)	−1(40)	16.91 ± 0.12
13	0(350)	1(85)	1(8)	0(50)	21.03 ± 0.22
14	0(350)	1(85)	−1(4)	0(50)	17.23 ± 0.09
15	0(350)	1(85)	0(6)	1(60)	20.39 ± 0.20
16	0(350)	1(85)	0(6)	−1(40)	17.15 ± 0.13
17	0(350)	−1(65)	0(6)	1(60)	20.84 ± 0.24
18	0(350)	−1(65)	1(8)	0(50)	20.47 ± 0.21
19	0(350)	−1(65)	−1(4)	0(50)	16.89 ± 0.14
20	0(350)	−1(65)	0(6)	−1(40)	16.27 ± 0.16
21	0(350)	0(75)	1(8)	1(60)	22.85 ± 0.25
22	0(350)	0(75)	1(8)	−1(40)	17.28 ± 0.18
23	0(350)	0(75)	−1(4)	−1(40)	15.46 ± 0.11
24	0(350)	0(75)	−1(4)	1(60)	17.53 ± 0.14
25	0(350)	0(75)	0(6)	0(50)	22.26 ± 0.19
26	0(350)	0(75)	0(6)	0(50)	22.03 ± 0.18
27	0(350)	0(75)	0(6)	0(50)	22.29 ± 0.21
28	0(350)	0(75)	0(6)	0(50)	22.10 ± 0.23
29	0(350)	0(75)	0(6)	0(50)	22.17 ± 0.14

[a] The results were obtained with Design Expert 10.0 software. Data were represented mean value ± standard deviation (n = 3).