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Analytical Methods

Optimization of microwave-assisted extraction of cottonseed oil and evaluation of its oxidative stability and physicochemical properties

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ABSTRACT

Microwave assisted extraction (MAE) is a novel method, which can reduce the extraction time and solvent consumption. This study aimed to evaluate the influence of MAE on oxidative stability and physicochemical properties of cottonseed oil. We found that the optimum extraction conditions were: irradiation time 3.57 min; cottonseed moisture content 14% and cottonseed to solvent ratio 1:4, which resulted in an extraction efficiency of 32.6%, 46 ppm total phenolic content, 0.7% free fatty acids, peroxide value of 0.2 and 11.5 h of Rancimat oxidative stability at 110 °C. GC analysis for MAE cottonseed oil determined palmitic acid (23.6%), stearic acid (2.3%), oleic acid (15.6%) and linoleic acid (55.1%), which were not significant different ($P > 0.05$) than conventionally-extracted (control) cottonseed oil. MAE oil samples from whole cottonseed (without dehulling) had the greatest long-term stability, more than oil samples containing BHT.

1. Introduction

Microwave assisted extraction (MAE) is a modern process that has been studied extensively (Amarni & Kadi, 2010; Camel, 2000). The efficiency of MAE process depends on the time and temperature of extraction, sample ratio and nature of both the solvent and the solid matrix (Terigar et al., 2010). It has been well documented that microwaves destroy biological cell structure in plant tissues such as oilseeds resulting in better extraction (Azadmard, Habibi, Hesari, Nemati, & Fathi, 2010; Chemat, Amar, Lagha, & Esveld, 2005; Jun & Chun, 1998). This is because the heat generated by movement of the polar molecules, which denatures cellular proteins. Thus, the moisture of oilseeds before extraction has an important role in extraction efficiency.

Azadmard et al. (2010) investigated the effect of microwave pre-treatment of rapeseeds on cold press extraction efficiency with the aim of enhancing oil extraction yield. They concluded that microwave pre-treatment could increase oil extraction by 10%. In another study, Amarni and Kadi (2010) obtained oil from olive cake using microwave assisted extraction and hexane as the solvent. They concluded that MAE gave better yields in less time and used less solvent.

In a recent study by Taghvaei, Jafari, Nowrouzieh, and Alishah (2013) has been revelled that microwave can destroy the structure of oil cells during process and facilitate the oil extraction without any heat treatment before extraction. Hence, applying MAE, there is no need to cook the oilseeds before extraction, which could have a potential benefit for the industry.

Oxidation reactions are the main reason of deterioration in edible oils and fats during storage or heat treatments such as frying and cooking. Autoxidation, which is the most common oxidation phenomena in edible oils, happens through a reaction between oxygen and unsaturated fatty acids via an auto-catalytic process consisting of a free radical chain mechanism. This chain includes initiation, propagation, and termination stages, which may be cyclical once started (Shahidi, 2005). Antioxidants prevent autoxidation of oils and fats by giving their hydrogen to free radicals formed during initial stages of autoxidation. There are two main groups of antioxidants, natural and synthetic. There is still doubt about safety and the approval, usage level, type and application of synthetic antioxidants and they are regulated in most countries (Akoh & Min, 2008; Gunstone, 2011; Shahidi, 2005). In recent years, a global trend has been made toward the substitution of synthetic antioxidants with natural alternatives, which has been reviewed by Taghvaei and Jafari (2013). The main goal of these research studies was to reduce the application of synthetic compounds as antioxidants because of their potential negative health effects and as a result of consumer demand.

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It has been proved that natural antioxidants like phenolic compounds are more susceptible to microwave (Proestos & Komaitis, 2008), and much research has been undertaken to extract phenolic compounds such as isoflavones (Terigar et al., 2010), polycyclic aromatic hydrocarbons (Camel, 2000), carvone and limonene (Chemat et al., 2005), gallic acid, vannilic acid, catechin, p-coumaric acid, ferulic acid and many others phenolic compounds (Proestos & Komaitis, 2008), pigments (Jun & Chun, 1998), tea polyphenols and caffeine (Pan, Niu, & Liu, 2003), tocopherols and tocotrienol (Zigoneanu, Williams, Xu, & Sabliov, 2008), olive leaf polyphenols (Rafiee, Jafari, Alami, & Khomeiri, 2012; Taghvaei et al., 2014) and many others from various plant resources and most of them concluded that MAE decreased the extraction time, solvent usage and increased the amount of extracted phenolic compounds. Also, Taghvaei et al. (2013) concluded that phenolic compounds from cottonseed, such as gossypol, could be extracted more easily using MAE than conventional extraction methods. Therefore, MAE of edible oils can lead to extraction of more natural phenolic compounds, which can improve the oxidative stability of final products (Azadmard et al., 2010; Rafiee, Jafari, Alami, & Khomeiri, 2011). In a study by Azadmard et al. (2010), microwave pre-treatment of rapeseed caused an increase in the oil phytosterols (by 15%) and tocopherols (by 55%) and, as a result, the stability of rapeseed oil (analysed by Rancimat) increased from one hour (which was for untreated rapeseed) to eight hours after MAE.

The cottonseed oil, which contains around 30% saturated fatty acids (palmitic and stearic acid) is stable and is consumed mainly as frying oil formulations (Shahidi, 2005), hence it is subjected more frequently to high temperatures and moisture (during frying). The presence of food moisture, atmospheric oxygen and high temperatures could cause various chemical changes and loss of antioxidants such as steam distillation of antioxidants, oxidation of phenolic compounds, reduction of their pro-oxidative activity due to reaction with fried materials and polymerisation (Pokorny, Yanishlieva, & Gordon, 2000). Synthetic antioxidants such as butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) are more susceptible to steam distillation (Warner, Brumley, Daniels, Joe, & Fazio, 1986). Losses of natural antioxidants are comparatively smaller since their volatility is much lower than that of common synthetic antioxidants (Pokorny et al., 2000).

The objectives of this study were, firstly, to increase cottonseed oil extraction efficiency through MAE and optimize extraction time, solvent usage and moisture content of seeds using response surface methodology (RSM) to achieve the highest extraction efficiency, thermal and oxidative stability, and most natural phenolic compounds as well as the lowest free fatty acid and peroxide content. Secondly, to investigate the effect of natural phenolic compounds from cottonseed hull (extraction of cottonseed oil without de-hulling) on long-term oxidative stability of cottonseed oil compared with soybean oil with and without 100 and 200 ppm synthetic antioxidant.

2. Materials and methods

2.1. Sample preparations and reagents

The cottonseeds (pak variety) were obtained from Cotton Research Institute of Iran (2011). Pak is a variety of cotton, which has trace amounts of gossypol, and was selected to eliminate the effect of gossypol on stability and total phenolic content of the final oil. Gossypol is a phenolic compound with antioxidant and toxic effects, and should be removed from cottonseed oil during the refining process (Taghvaei et al., 2013). Solvents and chemicals were obtained from Merck (Darmstadt, Germany). Gallic acid, gossypol standards and Folin–Ciocalteu reagent were purchased from Sigma–Aldrich Co. (St. Louis, MO, USA)

2.2. Microwave-assisted extraction

Cottonseeds were de-hulled and powdered with a laboratory mill (Sunny, SFP-820). For evaluating the influence of cottonseed moisture content on MAE efficiency, three levels of moisture content (1%, 7% and 14% wet basis) were applied before extraction. The initial moisture content of cottonseeds was 7% wet basis. The cottonseed powders were placed as a uniform layer on an aluminum tray with 5 mm diameter and were placed in a vacuum oven (Memmert VO-200, Germany) under 10,000 Pa pressure at 50 °C for about 5 h until the moisture content was reached to 1%. For increasing the powders moisture content to 14%, sufficient amount of distilled water was sprayed on samples according to the following equation. In order to have a uniform diffusion of water, the moisturised powders were maintained at 4 °C for 24 h.

$$Q = W_i(M_f - M_i)/100 - M_f \quad (1)$$

Q: amount of water which must be added (kg).
W_i: initial weight of cottonseed powder (kg).
M_i: initial moisture content (% wet basis).
M_f: final moisture content (% wet basis).

A microwave oven (Samsung, model: CF3110N-5, Korea) was modified for oil extraction. The modified MAE system consisted of a volumetric flask (500 ml) coupled with a condenser at the top and a magnetic stirrer beneath. The microwave output was 900 W with 2450 MHz frequency and its inner cavity dimensions were 400 mm × 300 mm × 250 mm. For each extraction, 100 g cottonseed powder, 200, 300 or 400 ml of solvent (*n*-hexane) and a magnet were placed in microwave oven. After 1, 3.5 and 6 min of irradiation and simultaneous magnetic stirring, the extraction process was stopped. Then, the cottonseed meal removed from miscella by means of filtration followed by centrifugation (Sigma, 3K30, Germany) at 80,500*g* for 5 min. Also, the solvent removed under reduced pressure at 50 °C by a rotary evaporator (IKA RV 10 basic, Japan). The control (blank) oil was extracted by soaking of 100 g cottonseed powder in 200 ml of *n*-hexane at 50 °C for 30 min without any microwave treatment for comparison. Refined soybean oil without any additives was purchased from a local oil refining factory (Alia Golestan Co.) to compare its oxidative stability with our samples.

For evaluating the influence of phenolic compounds contained in cottonseed hull on oxidative stability of the final MAE oil, the whole cottonseeds (without dehulling) were milled and mixed with *n*-hexane by a ratio of 1:3 and the oil was extracted through MAE after 3.5 min.

2.3. Oil physicochemical properties

In order to comparison of the effect of various microwave treatments on physical properties of resulting oil, the following experiments were carried out: free fatty acid content (AOCS Ca 5a-40), melting point (AOCS Cc 3-25), smoke point (AOCS Cc 9a-48), refractive index (AOCS Cc 7-25) and the moisture content of cottonseed powder (AOCS Aa 3-38). The total oil content of cottonseeds was determined by Soxhlet apparatus for 16 h using *n*-hexane. The colour was evaluated by transferring cottonseed oil samples into a micro-plate cell (4 mm diameter) and analysing using a Lovibond Tintometer model cam-system 500 in the L, a, b, mode of CIE (L, a, b, indicate lightness, redness/greenness, and yellowness/blueness, respectively).