MICROWAVE-ASSISTED SOLVENT EXTRACTION OF CASTOR OIL FROM CASTOR SEEDS

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To my beloved husband, mother and father

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ABSTRACT

Castor oil has various uses in the field of cosmetics, plastics, manufacturing of biodiesel, lubricants and medicine. This study investigated the extraction of castor oil using microwave-assisted extraction (MAE). The extraction was performed at 5, 10, 20, 30 min extraction times, 160 W, 230 W and 330 W power intensities and 10, 20 and 40 solvent-to-feed (S/F) ratios (mL of solvent to gram of feed). The oils were characterized for yield, physico-chemical properties, dielectric properties and oxidation stability, and comparison was also made with oil extracted using Soxhlet method. Results show that the mixed solvent (5 % ethanol in hexane) was effective to extract castor oil with 36 % yield by MAE. The maximum oil yield of 37 % was obtained by MAE at 20 minutes with power intensity of 330 W and S/F ratio of 20. The density, refractive index and dielectric properties of oils were not affected by extraction methods and extraction parameters of MAE. However, the viscosity of oil increased, while the pH decreased when the power intensity and extraction time increased due to oil oxidation. The main fatty acid composition of castor oils is ricinoleic acid. The oil extracted by MAE experienced a higher oxidation compared to that by Soxhlet method. With extra caution on oxidation of castor oil, MAE can be an attractive and promising method of 86% less in processing time and higher yield compared to Soxhlet extraction.

ABSTRAK

Minyak kastor mempunyai pelbagai kegunaan dalam bidang kosmetik, plastik, pengeluaran biodiesel, pelincir and perubatan. Kajian ini meneliti pengekstrakan minyak kastor menggunakan pengekstrakan berbantu gelombang mikro (MAE). Pengekstrakan dilakukan pada masa 5, 10, 20 dan 30 min, pengamatan kuasa 160 W, 230 W dan 330 W serta nisbah pelarut terhadap suapan (S/F) (mL pelarut kepada gram suapan) 10, 20 dan 40. Minyak dicirikan terhadap hasil, sifat-sifat fiziko-kimia, sifat-sifat dielektrik dan kestabilan pengoksidaan, serta perbandingan dilakukan terhadap minyak yang diekstrak menggunakan kaedah *Soxhlet*. Keputusan kajian menunjukkan campuran pelarut (5 % etanol di dalam heksana) adalah berkesan untuk mengekstrak minyak kastor dengan hasil sebanyak 36 % menggunakan MAE. Hasil minyak maksimum diperoleh daripada MAE adalah 37 % selama 20 minit masa ekstrak dengan pengamatan kuasa 330 W dan nisbah S/F 20. Ketumpatan, indeks biasan dan sifat-sifat dielektrik minyak tidak dipengaruhi oleh kaedah dan parameter-parameter pengekstrakan MAE. Walau bagaimanapun, kelikatan minyak meningkat dan pH menurun apabila pengamatan kuasa dan masa mengekstrak meningkat disebabkan oleh pengoksidaan minyak. Komposisi utama asid lemak bagi minyak kastor adalah asid risinoleik. Minyak yang diekstrak menggunakan MAE mengalami pengoksidaan yang lebih tinggi berbanding kaedah *Soxhlet*. Dengan lebih berhati-hati pada kadar pengoksidaan minyak kastor, MAE boleh menjadi kaedah yang menarik dengan memberikan penjimatan masa mengekstrak sebanyak 86 % dan hasil yang lebih tinggi berbanding pengekstrakan *Soxhlet*.

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LIST OF ABBREVIATIONS

MAE	-	Microwave Assisted Extraction
S/F	-	Solvent-to-feed ratio
aq	-	Aqueous
SFME	-	Solvent-free microwave-assisted extraction
HO•	-	Hydroxyl radical
RO•	-	Alkyl radical
HOO•	-	Hydroperoxyl radical
THF	-	Tetra-hydrofuran
FFA	-	Free fatty acid
NaOH	-	Sodium hydroxide
VNA	-	Vector Network Analyzer

LIST OF SYMBOLS

GHz	-	Giga Hertz
MHz	-	Mega Hertz
W	-	Watt
ε^*	-	Permittivity
ε'	-	Dielectric constant
ε''	-	Dielectric loss factor
$\tan \delta$	-	Loss tangent
D_p	-	Penetration depth
λ	-	Wavelength
f	-	Frequency
c	-	Speed of light
δ	-	Dissipation factor

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CHAPTER 1

INTRODUCTION

1.1 Overview

This chapter briefly explains about the research background of microwave-assisted extraction and castor oil extraction from castor bean. Furthermore, the problem statement is formulated based on the comparison of conventional extraction methods with microwave extraction, and the properties of castor oil.

1.2 Research Background

A long time ago, castor oil has been used to against constipation and acts as medicinal oil. It is categorized as non-edible oil and possesses nauseous properties. Besides that, the plant has already been grown for its oil to be used in the wick lamps for lighting in Egypt about 6000 years ago (Scarpa and Guerci, 1982).

Nowadays, castor is cultivated in large scale for commercial purposes especially in Asia, where India has become the largest exporter of castor oil in the world, followed by China. In Malaysia, castor trees have been planted in order to fulfill the demand for castor seeds. Casa Kinabalu Sdn. Bhd. is one of the companies that are responsible in the production and planting the castor plant in Malaysia (Pin, 2010).

For human and animals, castor seeds are poisonous because of ricin, ricinine and certain allergens in the seeds that are toxic (Ogunniyi, 2006). However, castor oil is safe if it is taken in a recommended quantity. The extracted oil from castor bean seed has more than 700 uses in the field of cosmetics, plastics, manufacturing of biodiesel, lubricants and medicine (Weiss, 2005; Cosmetic Ingredient Review Expert Panel, 2007). Castor oil from the plants or seeds can be obtained through solvent extraction processes. The solvent used during the extraction can be separated from the extracted oil by distillation in order to get the pure oil (Kirk-Othmer, 1979). Usually, the average oil yield from castor seeds is between 46 and 55% oil by weight. Recently, search for extraction techniques with better extraction efficiency to improve the performance of conventional extraction methods has become a subject of considerable interest. The conventional solvent extraction technique has limitations, as it requires more than an hour to complete the extraction depending on the solvents rates of diffusion, and furthermore it is energy-intensive (Puri *et al.*, 2012).

Microwave-assisted extraction (MAE) is one of the modern extraction techniques that are suitable for the extraction of essential oils, fats, and oils from seeds due to its distinct benefits over solvent extraction techniques (Cintas *et al.*, 2013). Fast extraction ability with small amount of solvent consumption, and less risky for thermolabile or heat-sensitive constituents that easily destructed or decomposed by moderate heating are some of the attractive criteria of MAE technique (Vivekananda *et al.*, 2007). Besides that, MAE is comparable with other modern extraction techniques due to its simplicity and low cost of equipment compared to ultrasound-assisted extraction (UAE) and supercritical fluid extraction (SFE) (Danlami *et al.*, 2014; Zhang *et al.*, 2011).

Microwaves are usually applied in communication, energy vectors and conversion of electromagnetic energy to heat energy. There are many mechanisms of energy transfer during microwave heating, but typically dipole rotation and ionic conduction are the two main mechanisms in dielectric heating phenomena (Schiffmann, 1987). Whereas, in conventional heating, it depends on the conduction–convection mechanisms, and a finite period of time is required to heat the vessel before the heat is transferred to the solution. Microwave heating is totally different from conventional heating because it heats directly to the solution, and occurs in a selective manner. Hence, a decrease in extraction time of usually not more than 30 minutes as compared to that of Soxhlet (conventional heating) might be the consequence of this unique heating mechanism (Huie, 2002). Other than that, the heating principle using microwave is based upon its direct impact with polar materials or solvents (Letellier and Budzinski, 1999). The ability of a medium to absorb and convert the microwave energy into heat is the underlying principle towards the heating efficiency that is governed by its dielectric properties.

By using microwave, the oil quality may be differing from that of conventional extraction because the electromagnetic waves from microwave can change the cell structure of the sample (Veggi *et al.*, 2013). Despite many researches on MAE technique, there is still less detail and available data in literature regarding the dielectric properties and oxidative stability of oil obtained using MAE. Therefore, more research is needed to study the quality and physico-chemical properties of extracted oil using MAE, and their relationship with the dielectric properties and oxidation stability in order to obtain a better insight about the performance of this extraction method.

1.3 Problem Statement

Until recently, much effort has been put on developing the process for castor oil production. There are several methods to extract oil from seeds such as mechanical pressing, solvent extraction and supercritical fluid extraction (Radziah *et al.*, 2011). Soxhlet (solvent) extraction is a standard extraction technique where the fresh solvent is refluxed and in contact with the sample frequently (Wu *et al.*, 2011). It is a widely used technique because it is simple and easy to run. However, Soxhlet extraction requires the use of hazardous organic solvents and needs few hours to accomplish the extraction of oil.

On the other hand, MAE can produce similar or better yield within less than an hour. Less amount of solvent required, short extraction time and operation at relatively lower temperature are the added values of this technique (Danlami *et al.*, 2014). MAE is an original combination of microwave heating and extraction. Most of the past works on MAE were focusing on operating conditions, oil yield and its physico-chemical properties, but lack of knowledge on dielectric properties and oxidation stability are presented.

Dielectric properties material and/or mixture are important in microwave heating. The principle of heating using microwave is based upon its direct impact with polar materials/solvents (Letellier and Budzinski, 1999). The ability of a material to be heated under microwave field is referred to its dissipation factor or loss tangent (tan δ). The higher the tan δ of material, the easier the microwave energy is converted into heat (Menendez *et al.*, 2010; Atwater and Wheeler, 2003). Mostly, solvent that possesses high ε’ and tan δ strongly absorbs microwave energy and demonstrates excellent heating efficiency. Polar solvents will strongly absorb the microwave energy because of a permanent dipole moment... The molecules will rotate in an opposite direction in respond to the electromagnetic wave. This phenomenon causes the increment of solvent temperature. Meanwhile, the non-polar solvents such as hexane will not heat up when exposed to microwaves (Ibrahim and

Zaini, 2017; Zhou and Liu, 2006). Therefore, knowledge on dielectric properties of solvents and materials involved are crucial in microwave-assisted solvent extraction processes. To date, the study on dielectric properties of solvents in microwave-assisted extraction (MAE) is still lack in much of published literature.

Oxidation stability occurs by chemical reaction of oil and oxygen. The oxidation of polyunsaturated fatty acids in the oils normally results in lipid degradation, which later affects the final quality of the oil product. The rate of oil oxidation increased when the amount of polyunsaturated fatty acids in the oils is higher (Yoshida *et al.*, 1990; Lin *et al.*, 1999). This process brings undesirable changes in the oil quality, leading to the viscosity increment and sludge deposits. A low oil oxidation stability also limits the application in lubricant industry due to its impact on the lubrication performance. Plus, the use of thermal processes, such as microwave heating, frying, sterilization, and hydrolysis may as well accelerate the oxidation of oils due to the increase level of free fatty acids (Saldana and Martinez-Monteagudo, 2013; Yoshida *et al.*, 1992). Other factors that can also affect the oxidative stability of oil include thermal, light, oxygen, oil processing methods, fatty acid composition and the presence of minor components such as metal, mono- and diacylglycerols as well as phospholipids (Choe and Min, 2006). The oxidation of oil reduces the quality of oil and produces off-flavor compounds. Hence, it is also an important parameter for the quality assessment of castor oil obtained through MAE.

1.4 Objectives of Study

The research is aimed at studying the extraction of castor oil from castor seeds using MAE and to evaluate the quality of extracted oil.

In achieving these objectives, there are specific objectives that have to be fulfilled in this research include:

1. To evaluate the effect of operating conditions of MAE on the yield and physiochemical properties of castor oils.

2. To evaluate the dielectric properties of solvents and castor oils by open-ended coaxial probe technique.

3. To evaluate the oxidation stability of castor oils obtained by MAE and Soxhlet extraction methods.

1.5 Scope of study

This research covers extraction of castor oil using MAE at different operating conditions such as extraction time, solvent-to-feed ratio and power intensity. Then, the results were compared with the oil yield of conventional solvent extraction. Different solvents were used in the extraction in order to identify the best extraction solvent for castor oil using MAE. In addition, the characterization of physiochemical properties of the castor oil such as viscosity, acid value, free fatty acid composition, specific gravity, refractive index and pH value, by employing MAE and Soxhlet methods were studied. Moreover, the dielectric properties of solvents, oils and solid-solvent mixture were measured using an open ended coaxial probe technique at different microwave frequencies. The oxidative stability test of castor oil was evaluated based on oil viscosity and pH, and fatty acid composition at 70°C for 14 days. The oxidation rate was correlated and predicted with the pseudo-first-order, pseudo-second-order and rate law expression. Finally, modelling and optimization of the effect of independent variables which are extraction times and power intensities on oil yield were carried out using response surface methodology (RSM) to compare and verify with the experimental findings.

1.6 Hypothesis of study

The principle of heating using microwave is based upon its direct impact with polar materials. Microwaves energy is transmitted as waves penetrate into the materials and interact with polar molecules (such as water) inside the materials to generate heat. The ability of a medium to absorb and convert microwave energy into heat is governed by its dielectric properties. In solvent extraction, MAE can only be operative if polar solvent was used. Ethanol (polar solvent) is a good microwave absorber, while hexane (non-polar solvent) is a good extraction solvent, but not a good microwave absorber. Therefore, by manipulating the polarity of both solvents, the MAE of castor oil can be done with a possibly higher yield. Hence, it is believed that MAE can extract a similar or better yield than Soxhlet extraction but in a shorter time with a comparable oil quality.

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