UNIVERSITI PUTRA MALAYSIA

SURFACTANT-ASSISTED AQUEOUS EXTRACTION OF RESIDUAL OIL FROM PALM-PRESSED MESOCARP FIBRE

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SURFACTANT-ASSISTED AQUEOUS EXTRACTION OF RESIDUAL OIL FROM PALM-PRESSED MESOCARP FIBRE

By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

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SURFACTANT-ASSISTED AQUEOUS EXTRACTION OF RESIDUAL OIL FROM PALM-PRESSED MESOCARP FIBRE

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Surfactant-assisted aqueous extraction process (SAAEP) has been proposed as a green alternative for the extraction of oil from various plant materials. While most oil is extracted through hexane-based technique, concern about environmental and health effects from hexane extraction has led to an increased interest in the development of green SAAEP for edible oil application. Therefore, the aim of this study is to explore the potential of SAAEP using Tween 80, a food-grade surfactant, for the recovery of residual oil from palm-pressed mesocarp fibre (PPMF). First, the phase behaviour of Tween 80 solution and crude palm oil (CPO) were studied by analysing the interfacial tension (IFT), droplet size, viscosity and phase inversion temperature (PIT). The IFT value was evaluated by varying Tween 80 concentration (0.001-30w/v %), NaCl concentration (0.5-6w/v%), temperature (50-70°C), and fructose concentration (1-10w/v%). The lowest IFT of 0.253 mN/m was obtained using at least 0.5w/v% Tween 80, 6w/v% NaCl, and temperature of 60°C while the addition of fructose does not affect the IFT. The emulsion obtained has a droplet size of 4183.8 nm and viscosity of 7.38 cp. At high concentration of Tween 80 (30w/v%), the emulsion size was determined in nano-size-range but the viscosity of emulsion is very high (232.15 cp). Furthermore, emulsion of CPO and Tween 80 did not show the presence of phase inversion temperature (PIT) indicating that ultra-low level IFT (10^{-3} mN/m) was not achieved with increase in temperature. Microscopic study by TEM and light microscopy reveals that the oil in PPMF resides inside the broken cells of fibrils with a small amount of oil attached on the surface of the fibres.

Application of the Tween 80 solution in the extraction of oil from PPMF shows that the highest oil extraction (47.4 %) was obtained at 1 % w/v of Tween 80, 6w/v% of NaCl, solid to liquid ratio of 8g of PPMF: 200 mL of Tween 80 solution, 30 min of extraction time at 60°C of extraction temperature. To improve the extraction yield further, attempt was made to mix Tween 80 with other green surfactants and interfacial properties was

studied. Tween 80 was mixed with Emereen 1018 and Pluronic F108 in a binary surfactant system that theoretically can enhance the interaction with CPO. However, Tween 80/Emereen 1018 mix and Tween 80/Pluronic F108 mix can only produce the lowest IFT value of 0.64 mN/m and 0.69 mN/m respectively. As the binary system did not enhance the IFT reduction, Span 20 and Span 80 were added into the binary mixtures as lipophilic linkers. However, these lipophilic linkers also cannot experimentally enhance the IFT reduction further. Therefore, it can be concluded that single surfactant system using Tween 80 can be proposed as the potential extraction system for residual oil from PPMF. However, the studied mixed surfactant system did not assist in further reduction of IFT and thus is not expected to contribute into further increase of oil extraction efficiency.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

PENGEKSTRAKAN SISA MINYAK DARIPADA HAMPAS MESOKARP KELAPA SAWIT MENGGUNAKAN PENGEKSTRAKAN AKUEUS DIBANTU SURFAKTAN

Oleh

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Pengekstrakan akueus dibantu surfaktan telah dicadangkan sebagai alternatif hijau bagi mengestrak minyak daripada pelbagai jenis tumbuh-tumbuhan. Ketika kebanyakan minyak diekstrak menggunakan teknik berasaskan heksana, kesedaran tentang kesan kepada persekitaran dan kesihatan daripada pengekstrakan menggunakan heksana membawa kepada kecenderungan dalam mengembangkan SAAEP yang hijau ini untuk aplikasi minyak makanan. Oleh itu, tujuan kajian ini adalah untuk meneroka potensi SAAEP menggunakan Tween 80, satu surfaktan gred makanan untuk mengekstrak semula minyak sisa daripada hampas mesokarp kelapa sawit (PPMF). Pertamanya, perilaku fasa antara larutan Tween 80 dan minyak sawit mentah (CPO) telah dikaji dengan menganalisis ketegangan permukaan (IFT), saiz titisan, kelikatan dan suhu fasa penyongsangan (PIT). Nilai IFT dinilai dengan mengubah kepekatan surfaktan Tween 80 (0.001-30w/v%), kepekatan NaCl (0.5-6w/v%), suhu (50-70°C) dan kepekatan fruktosa (1-10w/v%) menggunakan CPO. Nilai IFT yang rendah iaitu 0.253mN/m telah dihasilkan menggunakan sekurang-kurangnya 0.5w/v% Tween 80, 6w/v% NaCl, dan suhu 60°C manakala penambahan fruktosa tidak memberi kesan kepada IFT. Emulsi yang dihasilkan mempunyai saiz titisan 4183.8nm dan kelikatan 7.38cp. Pada kepekatan Tween 80 yang tinggi (30w/v%), saiz emulsi didapati dalam julat saiz nano tetapi kelikatan emulsi tersebut sangat tinggi (232.15cp). Tambahan pula, emulsi antara CPO dan Tween 80 tidak menunjukkan kewujudan PIT yang menandakan nilai IFT ultra rendah tidak akan dicapai dengan pertambahan suhu. Kajian mikroskopi menggunakan mikroskop elektron penghantaran (TEM) dan mikroskop cahaya mendedahkan yang minyak dalam PPMF berada didalam fibril sel yang pecah dengan sedikit minyak terlekat pada permukaan gentian hampas.

Aplikasi larutan Tween 80 pada pengekstrakan minyak daripada PPMF menunjukkan bahawa pengekstrakan paling tinggi (47.4%) dihasilkan pada 1w/v% Tween 80, 6w/v% NaCl, nisbah pepejal kepada cecair 8g PPMF: 200 mL larutan Tween 80, 30 min masa pengekstrakan pada 60°C suhu pengekstrakan. Bagi meningkatkan hasil pengekstrakan yang lebih banyak, inisiatif diambil untuk mencampurkan Tween 80 dengan surfaktan hijau yang lain dan sifat antara permukaan dikaji. Tween 80 dicampurkan dengan Emereen 1018 dan Pluronic F108 didalam sistem surfaktan binari yang secara teorinya dapat menambahkan interaksi dengan CPO. Walaubagaimanapun, campuran Tween 80/Emereen dan campuran Tween 80/Pluronic F108 hanya menghasilkan nilai IFT yang paling rendah iaitu masing-masing 0.64mN/m dan 0.69mN/m. oleh kerana campuran binari tidak dapat membantu pengurangan IFT, Span 20 and Span 80 ditambah ke dalam campuran binari sebagai penyambung lipofilik. Walaubagaimanapun, penyambung lipofilik ini juga secara eksperimennya tidak dapat menurunkan nilai IFT kepada lebih rendah. Oleh itu, dapat disimpulkan bahawa sistem satu surfaktan menggunakan Tween 80 boleh dicadangkan sebagai sistem pengekstrakan yang berpotensi mengekstrak minyak sisa daripada PPMF. Walaubagaimapun, sistem surfaktan campuran yang dikaji tidak membantu dalam pengurangkan IFT kepada nilai yang lebih rendah dan seterusnya tidak dijangka akan menyumbangkan kepada pertambahan kadar pengekstrakan minyak.

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I certify that a Thesis Examination Committee has met on _____ to conduct the final examination of Nor Hakimah binti Ramly on her thesis entitled "Surfactant-Assisted Aqueous Extraction of Residual Oil from Palm-Pressed Mesocarp Fibre" in accordance with the Universiti Putra Malaysia [P.U (A) 106] 15 March 1998. The committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

CPO	Crude palm oil
PPMF	Palm-pressed mesocarp fibre
FFB	Fresh Fruit Bunches
DAG	Diacylglycerol
MAG	Monoacylglycerol
SAAEP	Surfactant-assisted aqueous extraction process
IFT	Interfacial tension
SDS	Sodium dodecyl sulphate
CMC	Critical micelle concentration
PIT	Phase inversion temperature
POE	Polyoxyethylene
CμC	Microemulsion concentration
CPKO	Crude palm kernel oil
TAG	Triacylglycerol
FFA	Free fatty acids
AEP	Aqueous Extraction Process
EAAEP	Enzyme-Assisted Aqueous Extraction Process
APG	Alkyl polyglucosides
TEM	Transmission Electron Microscopy
DIW	Departmentof Industrial Works

CHAPTER 1

INTRODUCTION

1.1 Overview of Research

Crude palm oil (CPO) is usually obtained by mechanical screw pressing. After this mechanical pressing, there is still about 5-11% residual oil per dry weight retained in the palm-pressed mesocarp fibre (PPMF), depending on the efficiency of the pressing process. Previous study reported that the residual oil from PPMF contains higher vitamin E of 1700-2600 ppm and higher carotenes of 1400-1600 ppm than common CPO which are 600-1000 ppm and 500-700 ppm respectively (Subramaniam et al., 2013). PPMF comprises of palm mesocarp fiber, kernel shell, crushed kernel and debris making the oil recovered from the fibre having a collective characteristic of all these components. These precious substances left in the residual oil from PPMF make it significant to be extracted (Neoh et al., 2011). The residual oil which is still retained in this pressed fibre is around 5% - 11% as a ratio to the dry matter, representing 0.25% - 0.55% of oil loss per tonne of Fresh Fruit Bunches (FFBs) processed (Subramaniam et al., 2013).

Until now, palm oil mills in Malaysia only burn the fresh fibre in the boiler for electricity generation. The combustion of PPMF together with the shells inside the boiler can create pollution caused by the emission of boiler ash and flue gases. This is because the combustion of solid biomass usually needs proper pretreatment to ensure efficient combustion to occur. Hence, palm oil mills still have to move towards better alternatives of energy sources such as biogas which is a more efficient and cleaner technology. Furthermore, other than precious minor constituents present in the residual oil from PPMF, the waste mesocarp after oil recovery which comprised of lignocellulosic material can be used to produce other valuable products such as biocomposite, or ethanol (May and Aziz, 2012).

Recently, surfactant-assisted aqueous extraction process (SAAEP) has been proposed as an environmentally friendly alternative to hexane extraction for the recovery of oil from plant materials such as corn germ and canola seeds (Do and Sabatini, 2010; Kadioglu, Phan, & Sabatini, 2010). Surfactant reduces the high interfacial tension (IFT), the surface energy exerted by intermolecular interactions, between the immiscible phases of oil and water. SAAEP using extended-type surfactant has been shown to effectively extract the oil from peanut and canola seeds with 95% and 93% of oil extraction efficiencies and the quality of extracted oil via this method in terms of free fatty acids are better than hexane extraction (Do and Sabatini, 2010).

An important characteristic of an efficient aqueous surfactant-extraction system is its ability to reduce the IFT value between the oil and the extracting solution to an ultralow level ($<10^{-3}$mN/m) (Do and Sabatini, 2010). At this ultralow IFT, a Winsor Type III microemulsion, also known as a middle-phase microemulsion, can be produced, whereby the oil is solubilized in the surfactant's micelle. When ultralow IFT is reached, the oil

can be efficiently detached from the plant cells without strong agitation and since the surfactant concentration is low, the detached oil will not be solubilized or emulsified in the micelle to a great extent, rather it is liberated as free oil and floats to the surface (Do and Sabatini, 2010). This is important since it prevents additional tedious process from separating the extracted oil from the emulsion.

Ultralow IFT can be achieved when there is a proper balance between the hydrophilic interaction and the lipophilic interaction. The required balance can be achieved using a single surfactant with a proper structure or a combination of surfactant and co-surfactants, and with the aid of an electrolyte addition at optimum salinity for anionic surfactants or increasing the temperature to the phase inversion temperature (PIT) for non-ionic surfactants (Rosen, 2004). More recently, extended surfactants, were developed to achieve the proper balance to reach an ultralow IFT (Do and Sabatini, 2010). However, the health and safety aspects of the extended surfactants in food processing are uncertain, leading to non-food applications of the extracted oil such as to biodiesel or other industrial products. Given the high nutritional values of the residual oil from PPMF, a surfactant that can be safely used in food processing should be evaluated to allow for the extracted oil to be used as food ingredients.

Tween 80 is a commercial food-grade non-ionic surfactant that has a relatively low toxicity, low irritation potential and is widely used in pharmaceutical microemulsion preparation (Cho et al., 2008). The structure of Tween 80 comprised polyoxyethylene (20) and sorbitan monooleate. Compared with other surfactants in the Tweens family such as Tween 20 and Tween 40, Tween 80 is less viscous and possesses a lower critical micelle concentration (CMC), which means that the system will use less surfactant to achieve minimum IFT (Hameed et al., 2012). In this study, the phase behavior of Tween 80 solution and CPO was investigated and the potential of Tween 80 in the recovery of residual oil from palm-pressed mesocarp was evaluated.

Formulating microemulsion between surfactant and vegetable oils in the form of triglycerides is quite challenging compared to hydrocarbons. Combination of bulky hydrophobic fatty acid chains and high polar ester in the molecule of triglyceride cause the solubilisation by the surfactant become difficult (Do et.al, 2008). In the case that Tween 80 alone was not able to reduce the interfacial tension between surfactant solution and the vegetable oil to an ultra-low value, combining proper types of surfactants have been shown to result in synergy such that it improves the performance of each of the separated components. When a surfactant with a large tail and a small head is combined with a surfactant with a large head and a small tail, the surfactant pair extend further on both the water and oil phase. This is expected to increase the IFT performance while preventing the surfactant from precipitating out from the solution. Therefore, in this study, mixtures of Tween 80 with several appropriate food-grade surfactants at different ratio was also studied to observe its effect on the interfacial properties of CPO and surfactant solution.

1.2 Problem Statement

Nowadays, the method of extraction that is used by all palm oil mills in Malaysia is by pressing the oil out from the mesocarp using mechanical approach, commonly known as mechanical screw presses. However, this approach is not able to completely squeeze out the CPO from mesocarp. Generally, the most efficient method to extract the vegetable oil is by using organic solvent such as hexane. However, hexane has been identified as hazardous solvent because of the risk to safety and health. Hexane can contribute to high emission of volatile organic compounds (VOCs) in the atmosphere which is harmful to the human health and ozone as well as can cause fire. Then, research moves towards a safe method which using water as extraction medium. However, as water and oil are naturally immiscible, water alone cannot efficiently work as an extraction medium. In previous literature, enzyme was introduced to assist the function of water as enzyme can degrade the cell wall that prevent the coalesced oil to mobilize to the bulk phase. However, enzymes are very specific to certain components and certain conditions. The cells usually comprise of a few different components which need specific enzyme to degrade them. Different from enzyme, introduction of surfactant focuses on reducing IFT between oil and water and it is easier to be handled since surfactant does not require very specific condition like enzyme. Several studies on surfactant-assisted aqueous extraction have been done but most studies use surfactants which are not suitable for edible oil application.

1.3 Objectives

1. To characterise crude palm oil (CPO) O/W emulsion produced with Tween 80 and to evaluate potential in residual oil recovery of palm-pressed mesocarp fibre (PPMF).
2. To analyse the synergism of mixtures of Tween 80 and several co-surfactants in reducing the IFT between CPO/surfactant system.

1.4 Scope of Research

In this study, the potential of Tween 80 in the recovery of residual oil from palm–pressed mesocarp was evaluated. The effect of surfactant concentration, electrolyte (NaCl), temperature, and fructose was studied to determine whether a Winsor Type III CPO microemulsion can be produced by using this single commercial surfactant. The IFT, droplet size and viscosity were then correlated to the oil extraction efficiency. In improving the interfacial properties of the surfactant system also, mixture of Tween 80 and few surfactants (alkyl polyglucosides, block copolymer and lipophilic linkers) were formulated and analysed to observe the synergism between them and the contribution in reducing the IFT value. The mixing ratios of surfactants were also varied at various surfactant concentration.

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