



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

**INVESTIGATION OF EXTRACTING OIL FROM WASTE PALM FRUIT
FIBRE**

This report submitted in accordance with requirement of the Universiti Teknikal
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by

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DECLARATION

I hereby, declared this report entitled “Investigation of extracting oil from waste palm fruit fibre” is the results of my own research except as cited in references.

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Manufacturing Process) (Hons.). The member of the supervisory committee is as follow:

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ABSTRAK

Fiber perahan kelapa sawit ditekan adalah tertakluk kepada pengekstrakan soxhlet (SE) dan pengekstrakan superkritikal karbon dioksida (SC-CO₂) pada 80 ° C dan 100bar untuk menyiasat peratusan sisa minyak kelapa sawit. Kualiti sisa minyak kelapa sawit dibandingkan dengan minyak sawit mentah (CPO) diekstrak dengan mesin Press. Menurut hasil eksperimen, SE menghasilkan nilai perahan tertinggi OER dengan 11,423% berdasarkan berat kering sampel membandingkan SC-CO₂ dengan 1,789%. Kecekapan teknik pengekstrakan SC-CO₂ bagi mendapatkan OER daripada PPF adalah 15.66% pada 100bar dan 80 ° C berbanding SE . Walau bagaimanapun dari segi kualiti secara keseluruhan, kualiti minyak dari teknik perahan SC-CO₂ adalah lebih baik dari perahan Soxhlet dengan nilai DOBI adalah 2.565, PV pada 1.58 meq O₂/kg, kandungan kelembapan adalah 0.039%, peratusan FFA dengan 0,628% dan bendasing pada 0.004%.

ABSTRACT

Fresh palm-pressed fiber was subjected to soxhlet extraction and supercritical carbon dioxide (SC-CO₂) extraction at 80 °C and 100bar to investigate the residue fiber oil yield percentage. The residue oil quality compared to the crude palm oil (CPO) extracted with press machine. According to the results, SE resulted in the highest value of OER with 11.423% based on sample dry weight comparing to the SC-CO₂ extraction with 1.789%. The efficiency of SC-CO₂ extraction techniques for recovery of OER from PPF was 15.66% at 100bar and 80°C of those found with SE, respectively. However in term of overall quality. SC-CO₂ extraction techniques far surpass Soxhlet extraction with DOBI of 2.565, PV of 1.58 meq O₂/kg, moisture content of 0.039%, FFA percentage of 0.628% and impurities of 0.004%.

DEDICATION

To my beloved parents

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I would like to thank all my colleagues and others who have provided assistance at various occasions. Their views and tips are useful indeed. Unfortunately, it is not possible to list all of them in this limited space. I am also very thankful to my father, Mr. Shahipurullah, my mother, Mrs. Paizah and family members for their advice and motivations. I am grateful to all my members in University Teknikal Malaysia Melaka. My sincere thanks to all those involved directly and indirectly in helping me completing this report.

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LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

Bp	-	Boiling Point
CH ₄	-	Methane
C ₂ H ₆	-	Ethane
C ₃ H ₈	-	Propane
C ₂ H ₄	-	Propylene
C ₃ H ₆	-	Ethylene
CH ₃ OH	-	Methanol
C ₂ H ₅ OH	-	Ethanol
CO ₂	-	Carbon Dioxide
CPO	-	Crude Palm Oil
DOBI	-	The Deterioration of Bleachability Index
EFB	-	Empty Fruit Bunches
FFA	-	Free Fat Acid
FFB	-	Fresh Fruit Bunch
H ₂ O	-	Water
K	-	Distribution Coefficient
OSI	-	Oxidative Stability Index
PPF	-	Palm Pressed Fibre
PV	-	Peroxide Value
SC-CO ₂	-	Supercritical Carbon Dioxide
SCF	-	Supercritical Fluid
SFE	-	Supercritical Fluid Extraction
SPE	-	Solid-Phase Extraction

CHAPTER 1

INTRODUCTION

In this chapter contain the introduction of the research. This research has been carried based on the residue from Sime Darby Nordanal Palm Oil Mill that situated at Muar, Johor. Problem statement, objectives, scopes of this research and the theory also discussed in this chapter. There are also consisting of chapter organization that explains further about overall chapter in this report.

1.1 Research Background

The palm oil tree (*Elais guineensis*) originated from West Africa and was introduced in Malaysia in early of 1870's. Palm oil plant covers 67% of Malaysia's total agricultural land with 3 million hectares of the trees under cultivation, and 500,000 people in Malaysia are engaged in the sector. Today Malaysia is the largest producer and exporter of palm oil in the world. According to Malaysian Palm Oil Council currently we are producing 50% of world palm crude edible oil and account 62% of world exports.

The palm oil produces two types of oils; crude palm oil (CPO) from the fibrous mesocarp and crude palm kernel oil from the kernels. Besides that, the industry also produces large amounts of by-products such as palm kernel cake, palm

oil sludge (decanter cake), empty fruit bunches, palm shell, and palm pressed fiber as residues.

An oil palm fiber is one of residue that has been produced after processing of crude palm oil take place. Oil Palm Fibres can be used by manufactures to make various fibre composites such as furniture, infrastructures, and mattress. The oil palm fibre is produced from empty fruit bunches (EFB) that are considered as waste after the extraction oil palm fruits. However there are some problems regarding to this waste. Laboratory testing finds that there still has oil in the fibre range at 5 - 7%. Therefore, in this research, there are some suggestions that can reduce oil loss in the oil palm fibre.

To become the useable fibre, the empty fruit bunches goes through process which involve empty fruit bunches to be shredded, separated, refined and dried. No chemicals were involved in the production of oil palm fibres. High quality oil palm fibres are clean and toxic free. After the process, the end product of high quality oil palm fibre can be used by manufactures to make various fibre composites such as furniture, infrastructures, mattress, erosion control, paper production and also landscaping.

1.2 Problem Statement

Mechanical extraction is often considered more reliable than other extraction process when processing palm oil. The facility capital cost is low and the process can be set as lower as 5 ton per day per machine (100 ton per day per machine). Although it have such advantages, the machine also have some disadvantages where there still some residue oil in the fibre after the pressing process called oil loss that will effect the palm oil mill throughput. The oil loss will be difficult to control and larger if the the press capacity are bigger.

1.3 Objectives

The aim of this research is to propose some technique to reduce the oil lost by extracting the residue oil from the pressed palm fibre. Hence the specific objectives of the research are:

- To investigate the percentage of oil in palm pressed residue.
- To extract residue oil from pressed palm oil fibre with the Soxhlet extraction method.
- To compare oil quality.

1.4 Scope of Work

Based on the objectives, the scope of the research included study about the process of processing crude palm oil that produces oil palm fibre and collecting sample. Then, drying the sample to remove moisture and preparing the sample for extraction. The dry weights of sample can determine the maximum extracted oil. Then the solvent extraction method and supercritical fluid extraction (SFE) method will be carried on using selected reagent. There will also be a comparison between solvent extraction method and supercritical fluid extraction method. This is to determine the percentage of extracted oil and the extraction efficiency will be investigated through this work.

1.5 Theory

1.5.1 Solution Extraction

Solvent extraction is a process whereby two immiscible liquids or substance are added up and vigorously shaken in an attempt to disperse one in the other so that solutes can migrate from one solvent to the other. When the two liquids are not shaken the solvent to solvent interface area is limited to the geometric area of the circle separating the two solvents. However as the two liquids are vigorously shaken the solvents become intimately dispersed in each other. In general, a solution will be distributed, or partitioned between both available phases. An equilibrium distribution will be reached, and the equilibrium constant for this process is called the distribution coefficient, or sometimes the partition coefficient.

1.5.1.1 Distribution Coefficient

The distribution coefficient is a measure of the tendency for the solute to reside in one phase versus the other and, to a very good approximation, is equal to the ratio of solubilities for **A** in the respective solvents. Thus, if **A** is much more soluble in water than in the organic solvent, the distribution coefficient will be small (<1), while a large distribution coefficient implies that **A** is much more soluble in the organic than in water. You should note that it is the concentration in each phase which is important, so that the quantity of **A** which is found in each phase will also depend on the volumes of solvent involved.

$$K = \frac{A_{Organic}}{A_{Water}} (g/100ml)$$

K : Distribution Coefficient

A : Solubilities

Those compounds with very large (say, >20) or very small (<0.05) distribution coefficients will be found nearly totally in one of the phases. In contrast, for compounds with intermediate values for the distribution coefficient, the distinction between phases is not so clear-cut. Significant amounts of material will be found in each phase.

1.5.1.3 Choosing a Solvent System

One important aspect when choosing a solvent system for extraction is to pick two immiscible solvents. Some common extraction solvent pairs are water-dichloromethane, water ether, and water-hexane. Most extractions involve water because it is highly polar and immiscible with most organic solvents. In addition, the compound you are attempting to extract must be soluble in the organic solvent, but insoluble in the water layer. An organic compound like benzene is simple to extract from water, because its solubility in water is very low. However, solvents like ethanol and methanol will not separate using liquid/liquid extraction techniques, because they are soluble in both organic solvents and water.

There are also practical concerns when choosing extraction solvents. Cost, toxicity, flammability should be considered. The volatility of the organic solvent is important. Solvents with low boiling points like ether and hexane are often used to make isolating and drying the isolate material easier. If ether is used (bp = $35\text{ }^{\circ}\text{C}$) then evaporation to collect the solid is fast.

1.5.1.4 Identifying the Layers

One common mistake when performing an extraction is to mix-up the layers and discards the wrong one. The densities of the solvents will predict which solvent is the top or bottom layer. In general, the density of nonhalogenated organic solvents are less than 1.0 g/mL and halogenated solvents are greater than 1.0 g/mL . One common solvent pair is dichloromethane and water. The density of dichloromethane

is 1.325 g/mL and water is 1.000 g/mL. Dichloromethane is more dense than water; therefore, dichloromethane will be the bottom layer and water will be the top layer. Table 1 lists the densities of some extraction organic solvents.

Table 1.1: Common Extraction Solvent Listed by Densities (Solution Extraction, page 76)

Solvent	Density (g/mL)
hexane	0.695
ether	0.708
toluene	0.867
water	1.000
dichloromethane	1.325
chloroform	1.492

1.5.2 Super Critical Fluid Extraction

Supercritical Fluid Extraction (SFE) is the process of separating one component from another using supercritical fluid as the extracting solvent. Extraction is usually from a solid matrix, but can also be from liquids. SFE can be used as a sample preparation step for analytical purposes, or on a larger scale to either strip unwanted material from a product or collect a desired product.

Table 1.2: Critical properties of various solvent (Reid et al, 1987)

Solvent	Molecular weight(g/mol)	Critical temperature(K)	Critical pressure (Mpa;atm)	Critical density (g/cm ³)
Carbon Dioxide (CO ₂)	44.01	304.1	7.38;72.8	0.469
Water (H ₂ O)	18.015	647.096	22.064;217.755	0.322
Methane (CH ₄)	16.04	190.4	4.60;45.4	0.162

Ethane (C ₂ H ₆)	30.07	305.3	4.87;48.1	0.203
Propane (C ₃ H ₈)	44.09	369.8	4.25;41.9	0.217
Ethylene (C ₂ H ₄)	28.05	282.4	5.04;49.7	0.215
Propylene (C ₃ H ₆)	42.08	364.9	4.60;45.4	0.232
Methanol (CH ₃ OH)	32.04	512.6	8.09;79.8	0.272
Ethanol (C ₂ H ₅ OH)	46.07	513.9	6.14;60.6	0.276

There are two essential steps to SFE, diffusion of solution with the solid particles to the surface, and dissolution in the supercritical fluid. Clifford, Tony. (1999) say Figure 1.1 shows the stages during extraction from a spherical particle where at the start of the extraction the level of extracting is equal across the whole sphere shown on Figure 1.1a. As extraction started, the material is initially extracted from the edge of the sphere, and the concentration in the center is unchanged as shown on Figure 1.1b. As the extracting diffuses towards the edge of the sphere, the concentration in the center of the sphere drops as shown on Figure 1.1c.

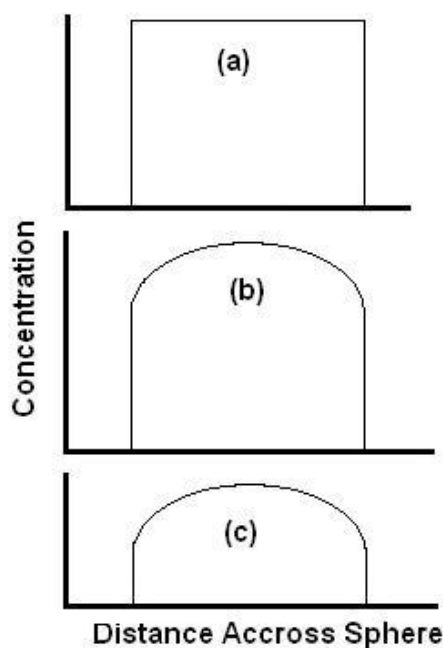


Figure 1.1: SFE extraction concentration profile (Clifford, Tony. 1999).

Clifford, Tony. (1999) also say Figure 1.2 is illustrating two extreme cases of the relative rates of diffusion and dissolution. Dissolution is fast relative to diffusion case shown on Figure 1.2a. The concentration at the edge drops to zero caused by the rate of the material is carried away from the edge is faster than it can diffuse from the center. The material is carried away as fast as it arrives at the surface, and the extraction is completely diffusion limited. Figure 1.2b shows a case where solubility is low relative to diffusion. The extracting is able to diffuse to the edge faster than it can be carried away by the solvent, and the concentration profile is flat.

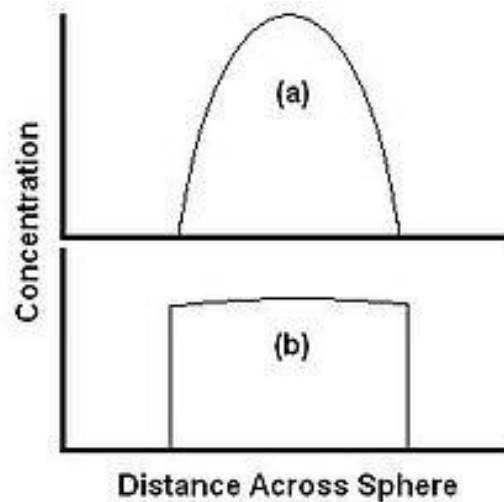


Figure 1.2: Concentration profile (a) Limited diffusion (b) Limited solubility (Clifford, Tony. 1999).

Supercritical fluid extraction in comparison with liquid extraction are relatively faster because of the low viscosities and high diffusivities associated with supercritical fluids. The extraction can be selective to some extent by controlling the density of the medium and the extracted material is easily recovered by simply depressurizing, allowing the supercritical fluid to return to gas phase and evaporate leaving no or little solvent residues.

Table 1.3: Density, Diffusivity and Viscosity for Liquids, Gases and Supercritical Fluids

	Density (kg/m ³)	Viscosity (μPa.s)	Diffusivity (mm ² /s)
Gases	1	10	1-10
Supercritical Fluids	100-1000	50-100	0.01-0.1
Liquids	1000	500-1000	0.001

1.5.3 Solid Phase Extraction

Solid-phase extraction (SPE) is nonequilibrium, exhaustive removal of chemical constituents from a flowing liquid sample via retention on a contained solid sorbent and subsequent recovery of selected constituents by elution from the sorbent. M.J.M. Wells et al, (2000) say analytical laboratories use solid phase extraction to concentrate and purify samples for analysis. Solid phase extraction can be used to isolate analytes of interest from a wide variety of matrices, including urine, blood, water, beverages, soil, and animal tissue.

1.5.3.1 Normal Phase SPE

Normal phase SPE procedures typically involve a polar analyte, a mid- to nonpolar matrix and a polar stationary phase. Polar-functionalized bonded silicas and polar adsorption media typically are used under normal phase conditions. Retention of an analyte under normal phase conditions is primarily due to interactions between polar functional groups of the analyte and polar groups on the sorbent surface. These include hydrogen bonding, pi-pi interactions, dipole-dipole interactions, and dipole-induced dipole interactions, among others. A compound adsorbed by these mechanisms is eluted by passing a solvent that disrupts the binding mechanism, usually a solvent that is more polar than the sample's original matrix.

1.5.3.2 Reversed Phase SPE

Reversed phase separations involve a polar or moderately polar sample matrix in mobile phase and a nonpolar stationary phase. The analyte of interest is typically middle to nonpolar. Several SPE materials, such as the alkyl- or aryl-bonded silicas are in the reversed phase category. Retention of organic analytes from polar solutions (e.g. water) onto these SPE materials is due primarily to the attractive forces between the carbon-hydrogen bonds in the analyte and the functional groups on the silica surface. These nonpolar-nonpolar attractive forces are commonly called

