UNIVERSITI PUTRA MALAYSIA

# *RECOVERY OF VALUABLE MATERIAL FROM OIL PALM EMPTY FRUIT BUNCH SPIKELETS USING SUB CRITICAL WATER TREATMENT*

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# RECOVERY OF VALUABLE MATERIAL FROM OIL PALM EMPTY FRUIT BUNCH SPIKELETS USING SUB CRITICAL WATER TREATMENT

By

**NOR AZRIN BINTI AHMAD KURNIN**

**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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**RECOVERY OF VALUABLE MATERIAL FROM OIL PALM EMPTY FRUIT BUNCH SPIKELETS USING SUB-CRITICAL WATER TREATMENT**

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**March 2018**

**Chair: Shamsul Izhar Siajam, PhD**
**Faculty: Engineering**

Oil palm empty fruit bunch (EFB) was one of the wastes produced in the palm oil mill. Whilst it still contains oil and can even be converted into valuable material, approximately 22.6 million tons is generated annually and treated as solid waste. Current method used by the industries in order to reduce and remove the EFB was by incineration process while some even dispose the EFB in the landfill. This process was known as mulching where the EFB was let to decompose naturally. Both methods were obviously wasting the advantage of fiber in the EFB. Thus, EFB were chosen to undergo sub-critical water (sub-cw) treatment to investigate its conversion into value added products as well as to evaluate the effects of process conditions (reaction temperature and time) of sub-cw treatment on value added products and sugar yield. Sub-cw is defined as water that lies between its boiling point, 100°C and its critical temperature, 374°C. At this condition, several physic-chemical properties of water have been enhanced. For instance, water has very low dielectric constant and higher ion product constant at high temperature (ex: 250°C). Therefore, sub-cw treatment was able to extract non-polar compound like oil and break the lignocelullosic component especially hemicellulose and cellulose into sugar, organic acid, tar and other derivatives. Highest yield of total sugar, 0.382 g/g-dry EFB was obtained at 220°C and reaction time of 5 minutes. Further analysis of the sample by using High Performance Liquid Chromatography (HPLC) detected pentoses and hexoses with yield of 0.008 to 0.032 g/g-dry EFB. Simple sugars which present in the sample at almost all reaction temperature were xylose, glucose, fructose and arabinose. Meanwhile, organic acids found were acetic and lactic acid in which lactic acid was most present in all reaction temperature compared to acetic acid. Highest yield of summation organic acid, 0.189 g/g-dry EFB was obtained at 330°C and reaction time of 5 minutes. Besides, tar was also produced from the reaction, yielded between 0.039 to 0.337 g/g-dry EFB. Phenolic compound were also detected in the tar produced. Highest yield of total phenolic content (TPC) obtained after the treatment was 89.51 mg/g-dry EFB at 330°C. Analysis of tar using Gas Chromatography Mass Spectrometer (GC-MS) indicate that Syringol (2,6-dimethoxy phenol), Guaiacol (2-methoxy phenol), and 4-Ethylguaiacol (4-ethyl, 2-methoxy phenol) were most present in samples at all reaction temperatures. This study showed that many valuable material can be recovered using water at sub-critical condition, and most attractive without the use of harmful organic solvent.

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

**PEROLEHAN PRODUK BERHARGA DARIPADA TANDAN KOSONG BUAH KELAPA SAWIT MENGGUNAKAN RAWATAN AIR SUB-KRITIKAL**

Oleh

**NOR AZRIN BINTI AHMAD KURNIN**

**Mac 2018**

**Pengerusi: Shamsul Izhar Siajam, PhD**
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Tandan kosong buah kelapa sawit (EFB) adalah salah satu bahan buangan yang dihasilkan oleh kilang proses kelapa sawit. Walaupun masih mempunyai sedikit minyak dan masih boleh diproses menjadi bahan berharga, lebih kurang 22.6 juta tan EFB dihasilkan setiap tahun sebagai sisa pepejal. Proses yang masih digunakan oleh industri untuk mengurangkan dan menghapuskan EFB adalah pembakaran dan pembuangan di tanah lapang. Proses ini juga dikenali sebagai sungkup atau pembajaan yang mana EFB dibiarkan mereput di tanah. Kedua-dua cara ini sememangnya tidak memanfaatkan kelebihan serat pada EFB. Oleh itu, EFB telah dipilih untuk menjalani rawatan air sub kritikal untuk menyiasat penukaran EFB kepada produk berharga, juga untuk menilai kesan suhu dan tempoh rawatan air sub kritikal yang pelbagai keatas produk berharga terutamanya gula. Air sub-kritikal didefinisikan sebagai air yang berada di antara suhu didih,100°C dan suhu kritikal, 374°C. Ketika ini, beberapa sifat fizik-kimia air telah dipertingkatkan. Sebagai contoh, air mengalami pemalar dielektrik yang rendah dan pemalar produk ion yang tinggi di suhu yang tinggi (contoh: 250°C). Oleh itu, rawatan air sub kritikal mampu untuk mengekstrak minyak dan memecahkan bahan liknosellulosa terutamanya hemiselulosadan selulosa kepada gula, asid organik, minyak tar dan lain-lain. Hasil tertinggi gula, iaitu 0.382 g/g-EFB kering telah diperolehi pada suhu 220°C dan waktu rawatan selama 5 minit. Analisa produk cecair menggunakan kromatografi cecair berprestasi tinggi (HPLC) telah mengesan pentosa dan heksosa dengan hasil sebanyak 0.008 ke 0.032 g/g-EFB kering. Gula yang dikesan di dalam hampir kesemua suhu rawatan adalah *xylose*, glukosa, fruktosa dan arabinosa. Sementara itu, asid organik yang diperolehi adalah asid asetik dan laktik dengan kehadiran asid laktik dikesan di dalam produk pada hampir kesemua suhu. Hasil tertinggi asid organik iaitu 0.189 g/g-EFB kering telah dicapai pada suhu 300°C dan waktu rawatan selama 5 minit. Selain itu, minyak tar juga terhasil, dengan hasil di antara 0.039 ke 0.337 g/g-EFB kering. Sebatian fenolik juga telah dikesan dalam minyak tar tersebut. Hasil tertinggi sebatian fenolik secara keseluruhan adalah 89.51 mg/g-dry EFB iaitu pada suhu rawatan 330°C. Analisa menggunakan kromatografi gas (GC-MS) mendapati Syringol (2,6-dimethoxy phenol), Guaiacol (2-methoxy phenol), dan 4-Ethylguaiacol (4-ethyl, 2-methoxy phenol) hadir di dalam minyak tar pada semua suhu rawatan. Kajian ini menunjukkan yang banyak produk berharga mampu dihasilkan menggunakan air sub kritikal, dan paling menariknya adalah ianya tanpa menggunakan pelarut yang merbahaya.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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This is to confirm that:

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| | |
|---|---|
| EFB | Empty fruit bunch |
| FFA | Free fatty acid |
| FFB | Fresh fruit bunch |
| GC-MS | Gas chromatography mass spectrometer |
| HMF | Hydroxymethyl furfural |
| HPLC | High performance liquid chromatography |
| HYSASE | Hydro solvent assisted steam extraction |
| MT | Metric ton |
| Sub-cw | Sub-critical water |
| TOC | Total organic carbon |
| TPC | Total phenolic content |
| UV | Ultraviolet |

## CHAPTER 1

## INTRODUCTION

### 1.1 Background of Study

There are many crops that were planted in the world and harvested for manufacturing of many products. Some are harvested to get the raw material to produce other different products. In the case of Malaysia, oil palm, coconut, banana and paddy are example of the main crops harvested here. *Elaeis Guineensis*, also known as oil palm tree is originated from West Africa and has been introduced in Malaysia in 1848 by Dutch planter. It is produced commercially in Malaysia starting from 1917. The ideal climate in Malaysia enhanced the growth of oil palm tree. They have a lifespan of 20-25 years before being cut and replanted. In 2009, about 51% of the world palm crude oil is produced by Malaysia and hence accounted for 62% of the world exports (Gopal, 2009). The commercial plantation areas in Malaysia have increased up to 5.1 million hectares which produced 18.8 million tonnes of palm oil in 2012 (Sime Darby, 2014).

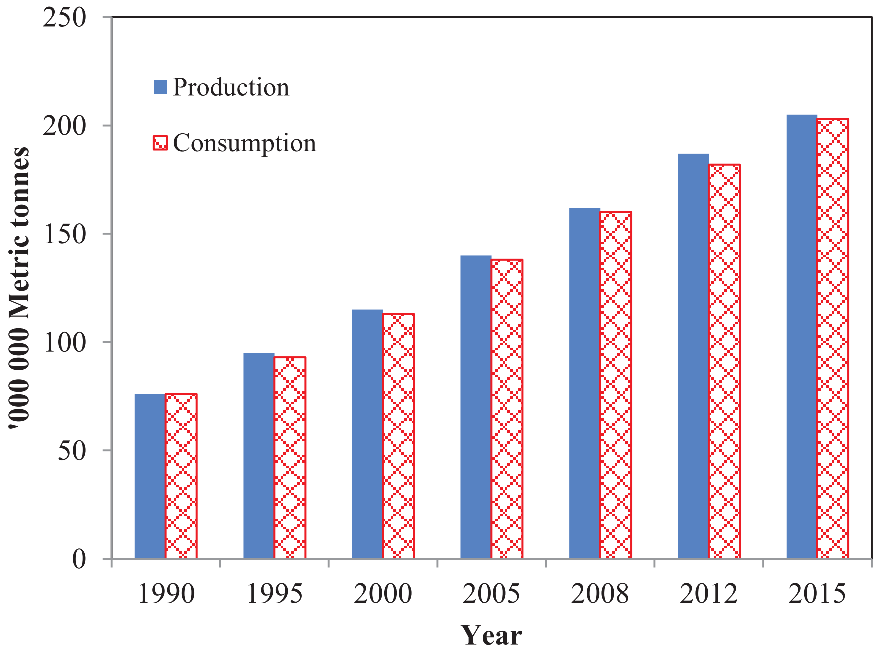


**Figure 1.1: Global oils and fats production and consumption 1990-2015** (Basiron, 2015)

It is reported that global oils and fats consumption increased in line with increasing in human population every year. Figure 1.1 shows the global oils and fats production and consumption from 1990 to 2015. In 2015, the production and consumption of oils and fats has doubled the one in 1990. The increasing demand on oils and fats every year has caused many countries especially Indonesia and Malaysia to focus on producing better quality palm fruitlets in terms of the oil extraction rate. This phenomenon explained the increase in land area reserved for palm oil plantation in Malaysia and Indonesia.

Figure 1.2 shows the percentage share of Malaysia Gross Domestic Product (GDP) for agriculture sector in Malaysia from 2006 to 2015. It can be observed that GDP contributed by forestry and logging, cocoa and fishing reduced year by year. None of the agriculture activities contributed GDP higher than oil palm did every year. As in 2015, oil palm industry contributed to the GDP of agriculture sector by 46.9%. Increment by 2.28 million tonnes of fresh fruit bunch (Department of Statistics Malaysia, 2016) from 96.07 million tonnes in 2014 (Department of Statistics Malaysia, 2015) showed that Malaysia are focused in the agriculture sector especially palm oil's production. Due to overwhelming global demand on palm oil, Malaysia has become one of the leading producer and exporter of palm oil besides Indonesia in the world. Thus, more palm waste like palm frond, empty fruit bunch, and palm pressed fiber are produced by these countries.

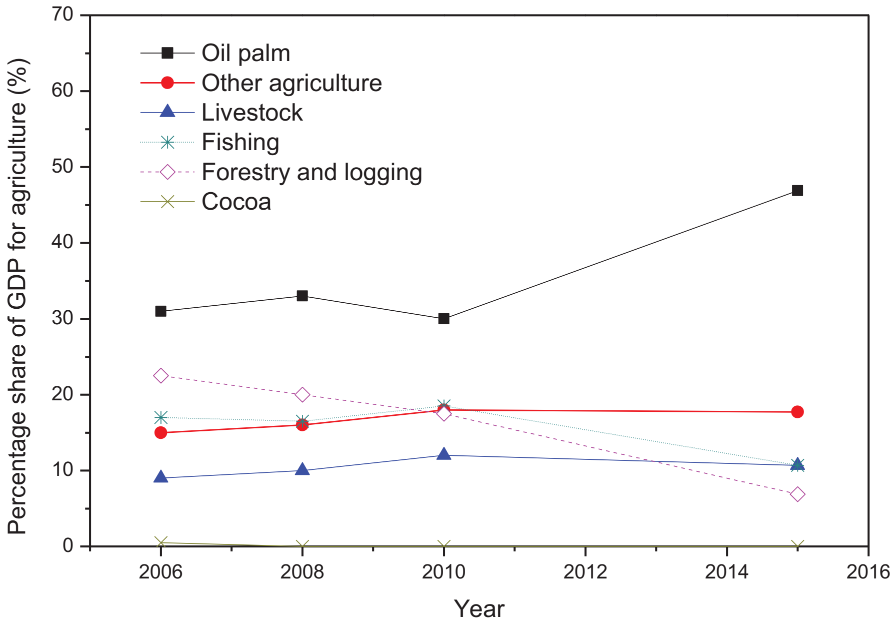


**Figure 1.2: Percentage share of GDP for agriculture sector, 2006-2015**
(Department of Statistics Malaysia, 2012, 2015)

## 1.2 Problem Statement

In Malaysia, palm oil industry is one of the major agricultural industries which provide edible oil domestically and exported to other countries too. However, palm oil mill produces many solid wastes such as the empty fruit bunch (EFB), oil palm fronds, trunks and palm oil mill effluent (POME). From 1 tonne of fresh fruit bunch collected, 22% is the EFB (Yusoff, 2006; Lim Meng Hon, 2010). In 2015, 21.64 million tonnes of EFB is produced from 98.35 million MT of fresh fruit bunch (FFB). Thus large area is required to allocate the EFB. To utilize these wastes, EFB are usually incinerated to produce energy (Udoetok, 2012)„ while some are used for mulching. These current methods applied by mill are obviously wasting the advantage of fibers in the waste and also cause environmental problem.

A study by Md Yunos et al., (2015) and Gomez et al., (2015) found that there is residual oil entrapped in the fiber of EFB which is worth to be recovered. Oil obtained from the EFB is valuable even with FFA higher than 5%, as it can be used in blending with the high purity crude palm oil. This will significantly help increase the oil extraction rate (OER) in the palm oil industry.

Other than oil, EFB contained lots of fiber which comprised of hemicellulose, cellulose and lignin that are able to be processed into other products. Many researches have been conducted in order to produce organic acid and sugar from EFB (Hamzah et al., 2011; Meilany et al., 2018; Nomanbhay et al., 2013) due to the high content of cellulose and hemicellulose in it. In addition, this sugar seem to be a good potential feedstock for fermentation to produce ethanol and butanol (Ibrahim et al., 2015; Kamoldeen et al., 2017; Lim Meng Hon, 2010). On the other hand, organic acid like lactic acid produced from EFB also is valuable as it can be used to make bioplastic. Hence, reliance on fossil fuels can be reduced.

Other than hydrolysis of cellulose and hemicellulose, attention has been given to the high amount of lignin in EFB that is suitable to be the feedstock for bio-oil and bio-char production (Chang, 2014; Chow et al., 2018). The bio-oil has potential to be used as fuel (Amin, (2011), however require several analysis test to identify its properties like heating value, acid content, and viscosity. Interestingly, EFB is also currently being exploited to produce hydrogen as biofuel (Ibrahim et al., 2015; Monir et al., 2018; Sivasangar et al., 2015).

Thus, many methods are being tested to helps reduce the waste production by converting it to new product. This is aligned with the advancement in technology which helps the positive growth in palm oil industry. An example of common method used nowadays is acid or alkaline hydrolysis. However, this method has several disadvantages like usage of strong acid, highly corrosive on the material used, require neutralization process and affect the edibility factor of the end-product.

Another method which is able to overcome the disadvantages of acid hydrolysis is enzymatic hydrolysis. This method is able to produce same products as acid hydrolysis does without affecting the edibility factor. However, this method requires special handling methods as it involve enzymes. In addition, enzymatic hydrolysis requires very long time to obtain the products. Both methods are also expensive as acid hydrolysis requires strong material for equipment and for safety purposes while enzymatic hydrolysis requires special care on enzyme and it cannot be recycle.

On the other hand, another method which is currently being exploited by many researchers is showing positive results with many different raw materials. This method is known as sub-critical water treatment (sub-CW) or hydrothermal treatment. Sub-CW method used water at high temperature between its boiling point (100°C) and critical temperature (374°C) where its physicochemical properties has been significantly altered. Water at temperature higher than 200°C has a very low dielectric constant which enables water to behave like solvent. At high temperature (i.e. 250°C) also, the ion product constant of water increased significantly by 1000 times than at room temperature which helps in hydrolysis. Thus, sub-cw can be used to breakdown polysaccharides into monosaccharides, oligosaccharides and organic acids. Besides, raw material can be directly used without drying using this method and therefore can save additional operating cost and time. In addition, this process uses only water as the solvent which is safe and easily available in Malaysia. Successful implication of this work will help reduce the amount of the empty bunch in the palm oil mill by utilizing it into other products in high amount.

### 1.3 Objectives of Studies

The objectives of this research are as follows;

i. To investigate the possibility for the conversion of oil palm empty fruit bunch into any value-added products.

ii. To evaluate the effects of process conditions (reaction temperature and time) of sub-critical water treatment on value added products and sugar yields.

### 1.4 Scopes of Study

This research covers the application of sub-critical water in an attempt to convert oil palm empty fruit bunch into valuable products. It involves sample collection from Sri Langat Mill in Dengkil and its preparation for drying purposes, sub-cw treatment, and Soxhlet extraction. Only spikelet is used in this study due to oil content. However, due to complicated procedure in segregating the midrib spine leaflets from the fiber, whole spikelet is used in this research.

The drying process takes place for few days in a dryer set at 80°C to measure the moisture content in EFB. Sample are cut into small pieces and prepared for sub-cw reaction at temperature range 180 to 360°C (reaction time: 5 minutes) and time range of 2 to 15 minutes (reaction temperature: 220°C). Size of the sample loaded in the reactor is not manipulated as it does not give significant effect on the hydrolysis process of a batch system (Prado et al., 2016). Soxhlet extraction is carried out to determine the total oil in the EFB's spikelet used. The oil yield is used as benchmark to know the efficiency of sub-critical water treatment in extracting oil.

All products are recovered using hexane, water and acetone. Recovered water phase is analyzed for its sugar content, organic acids and total organic carbon using Ultra-visible spectrometry (UV-Vis), High Performance Liquid Chromatography (HPLC) and Total Organic Carbon-Analyzer respectively. The water-insoluble tar is analyzed for individual and total phenolic content (TPC) using Gas Chromatography Mass Spectrometer (GC-MS) and UV-Vis. Besides sub-cw reaction, soxhlet extraction also is carried out to compare the effectiveness of sub-cw treatment in oil extraction.

### 1.5 Outline of Thesis

This thesis is comprised of five chapters. First chapter covers the brief introduction of sample, problems statement and objectives of this research. The second chapter described in detail about the raw material used and process involved throughout this research. Factors affecting the results and conventional method used are also described in this chapter. Chapter three is mainly describe in detail about the procedures and equipment involved like sub-critical water process and High Performance Liquid Chromatography equipment. Meanwhile, chapter four explained all results obtained throughout this research and what factors affecting the trend of yield obtained. Final chapter which is chapter five summarized all the findings according to the objectives and recommends some improvements suggestion for better result or future research.

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