UNIVERSITI PUTRA MALAYSIA

SUB-CRITICAL WATER PYROLYSIS OF OIL PALM TRUNK FOR PRODUCTION OF BIO-OIL AND SOLID PRODUCT

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By

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

March 2018

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

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March 2018

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Oil palm tree is a commercial crop in Malaysia producing millions of tonnes of unused oil palm trunk (OPT) waste during replanting process which is done in large scale every 25 to 30 years. The inner region of OPT was unutilized due to high moisture content which make it easy to rot. In this study, water at sub-critical state was utilized to produce bio-oil and solid product from the inner region of OPT. The objectives for this study are to evaluate the effect of reaction temperature and time on the yield of bio-oil and solid product produced, to determine the optimum condition (reaction temperature and time) for the production of bio-oil and solid product, and to characterize the bio-oil and solid product obtained. In order to determine the best OPT for the production of bio-oil and solid product from the inner region of OPT, 21 and 35 year old OPTs were used. Each OPT were divided into two parts, namely top and bottom parts. OPTs were then cut into small size and loaded into a reactor. The reactor was then immersed in a molten salt bath for sub-critical water (Sub-CW) reaction. The reaction was carried out in a temperature range between 100°C-370°C with 10°C intervals and reaction time was varied between 0.1 to 20 minutes. It was found that, the best OPT for production of bio-oil and solid product is from the 21 years old OPT. In the 21 years old OPT, the optimum condition for the highest production yield of bio-oil was 27.5% kg/kg-dry OPT (OPT21T, 340ºC, 5 min) and 30.1% kg/kg-dry OPT (OPT21B, 330ºC, 5 min). The highest Higher Heating Value (HHV) of bio-oil was 33,200kJ/kg (OPT21T, 350ºC, 5 min) and 26,400kJ/kg (OPT21B, 340ºC, 5 min). The best HHV of solid product was 31,000kJ/kg (OPT21T, 370ºC, 5 min) and 32,400kJ/kg (OPT21B, 340ºC, 5 min). The particle size of solid product at optimum condition was 20m and 17m for OPT21B and OPT21T, respectively. The main chemical compound found in bio-oilwere phenol, 2-methoxy-phenol, 4-ethyl-2-methoxy-phenol, and 2,6-dimethoxy- phenol. These results show that Sub-CW reaction is technically feasible in producing bio-oil and solid product from OPT.

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

SUB-KRITIKAL AIR PIROLISIS BATANG KELAPA SAWIT UNTUK PENGHASILAN BIO-MINYAK DAN PRODUK PEPEJAL

Oleh

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Pokok kelapa sawit adalah tanaman komersil di Malaysia yang menghasilkan satu juta tan batang kelapa sawit yang tidak digunakan (OPT) semasa proses penanaman semula yang dilakukan secara besar-besaran setiap 25 hingga 30 tahun. Bahagian dalam OPT tidak digunakan kerana kandungan kelembapan yang tinggi menjadikannya mudah rosak. Dalam kajian ini, air dalam keadaan sub-kritikal digunakan untuk menghasilkan bio-minyak dan produk pepejal dari bahagian dalam OPT. Objektif kajian ini adalah untuk menilai kesan reaksi suhu dan masa terhadap penghasilan bio-minyak dan produk pepejal, untuk menentukan keadaan optimum (suhu dan masa reaksi) untuk pengeluaran bio-minyak dan produk pepejal, dan untuk mengambarkan sifat bio-minyak dan produk pepejal yang diperolehi. Untuk menentukan OPT terbaik bagi pengeluaran bio-minyak dan produk pepejal dari bahagian dalam OPT, 21 dan 35 tahun OPT digunakan. Setiap OPT dibahagikan kepada dua bahagian, yang dinamakan bahagian atas dan bawah. OPT kemudian dipotong ke dalam saiz kecil dan dimuatkan kedalam reaktor. Reaktor itu kemudiannya direndam dalam larutan garam cair untuk reaksi air sub-kritikal (Sub-CW). Reaksi dilakukan dalam julat suhu diantara 100°C-370°C dengan selang 10°C dan masa tindak balas berubah antara 0.1 hingga 20 minit. Telah didapati bahawa, OPT terbaik untuk pengeluaran bio-minyak dan produk pepejal adalah dari OPT berusia 21 tahun. Dalam OPT 21 tahun, keadaan optimum bagi penghasilan bio-minyak tertinggi ialah 27.5% kg/kg-kering OPT (OPT21T, 340ºC, 5 minit) dan 30.1% kg/kg-kering OPT (OPT21B, 330ºC, 5 minit). Nilai tertinggi Pemanasan Tinggi (HHV) bio-minyak adalah 33,200kJ/kg (OPT21T, 350ºC, 5 minit) dan 26,400kJ/kg (OPT21B, 340ºC, 5 minit). HHV terbaik produk pepejal adalah 31,000kJ/kg (OPT21T, 370ºC, 5 minit) dan 32,400kJ/kg (OPT21B, 340ºC, 5 minit). Saiz zarah bagi produk pepejal pada keadaan optimum adalah 20µm dan 17µm bagi OPT21B dan OPT21T. Komposisi kimia utama yang terdapat dalam bio-minyak adalah fenol, 2-metoksi-fenol, 4-etil-2-metoksi-fenol, dan 2,6-dimetoksi-fenol. Keputusan ini

menunjukkan reaksi Sub-CW secara teknikal boleh menghasilkan produk bio-minyak dan produk pepejal dari OPT.

ACKNOWLEDGEMENTS

I would like to express my deepest appreciation to my research supervisor Ir. Dr. Shamsul Izhar bin Siajam from the Department of Chemical and Environmental Engineering, Universiti Putra Malaysia for his valuable and resolute guidance, enthusiastic encouragement and beneficial critiques during the planning and development of this research work. His willingness to give his time so generously to ensure high quality work tremendously appreciated.

I also would like to thank my former supervisor, Professor Yoshida Hiroyuki for his valuable ideas and support in this study. Not forgetting my co-supervisor, Associate Professor Dr. Mohd Halim Shah bin Ismail, academic staffs and my supportive course mates who were always ready to lend a helping hand. Thank you for all the help, encouragement and strength that aided me in coping with those stressful moments. Not to forget my seniors who would always provide some ideas regarding the research work.

Next, I wish to thank my parents for their relentless support, prayers, love and non-stop encouragement for me to complete my study successfully. Special thank to my beloved husband and beautiful daughters.

Last but not least, thank you to Ministry of Malaysian Higher Education and Universiti Putra Malaysia for providing financial support throughout my study.

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

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LIST OF ABBREVIATIONS

Sub-CW	Sub-critical water
OPT	Oil palm trunk
HHV	Higher heating value
SEM	Scanning electron microscopy
CHNS/O	Carbon, Hydrogen, Nitrogen, Sulfur, Oxygen
GC/MS	Gas chromatography mass spectrometry
PSD	Particle size distribution

CHAPTER 1

INTRODUCTION

1.1 Introduction

Fuel from fossil sources has been used for a long time, and the demand for it keeps increasing every year. The continue usage of fossil fuel will result in continuing emission of CO_2 gas, causing global warming on earth. With a limited source of fossil fuel, the development for alternative energy has to be undertaken. A biomass-derived energy known as bio-oil has been receiving attention as an alternative energy to petroleum-derive fuel [1–11]. Woods are mentioned as a typical biomass which attracts attention as raw materials for this new energy [12–15]. Malaysia produces a large volume of wood mass in the region. However, only 60% to 65% of the residues harvested are used to generate energy [16]. The remaining percentage is left to rot or burn.

Oil palm biomass is the largest biomass produced in Malaysia which covers about 85.6% of the total biomass produced [17]. Oil palm trunk (OPT) waste is produce significantly during the replanting process where oil palm tree is replanted on a large scale at once (hundred million hectares)[18]. OPT consist of primary vascular bundles which is embedded in parenchymatous tissues [19]. The percentage of vascular bundle is decreases from the outer to the inner section of OPT and increases from the bottom to the top level. Parenchyma tissues are found associated with the vascular bundle where it was found at the inner tip of vascular bundle. The lignified parenchyma tissues which stiffen the cell wall of OPT was found most at the bottom part of OPT and the non-lignified cell walls of parenchyma are found in the upper part of OPT. These non-lignified parenchyma tissues are fragile and easily ruptured during the cutting process [20].

The technology for converting biomass into bio-oil and solid product can be carried out by two main processes namely pyrolysis and hydrothermal process. The hydrothermal processing has receiving attention from researchers worldwide due to numerous advantages including high separation and energy efficiencies, the suitability to utilized biomass with high moisture content and the ability to use mixed feedstock [21,22]. Hydrothermal liquefaction can be carried out in sub-critical water (Sub-CW) conditions [23].

1.2 Background of study

Oil palm tree (*Elaeis guineensis jacs.*) is one of the most valuables plants in the world. As the world's second largest oil palm producer, millions of hectares of land in Malaysia are covered with oil palm tree and the availability of oil palm biomass especially oil palm trunk (OPT) is very huge [24]. In recent years, OPT has attracted attention for having a high potential value for bio-fuel production. The presence of high sugar content in its juice that mainly comes from the inner part of OPT makes it a promising feedstock for bio-fuel production. Moreover, bioethanol fuel (yield: 0.510 g/g at 25°C, fermented for 72 hours) can be successfully produced from OPT juice with the assistance of *S. cerevisiae* as a fermentation agent [7]. Nazia Hossain and Rafidah Jalil [8] reported using the same fermentation agent with Alanine amino acid and Epsom salt as nutritional supplement, successfully producing 58.43% w/w of ethanol and 21.14% w/w of sugar content from OPT juice. OPT juice also has become a good feedstock for the production of lactic acid with a yield of 89.9% wt% [25]. OPT is also capable of producing black-liquor which can be used to produce methanol fuel and also for pulp and paper production [26]. In additional to that, OPT also shows the ability to produce bio-oil (18.7 wt%) and bio-char (30 wt%) at 600°C with the retention time of two (2) hours by pyrolysis reaction technology [27].

The common methods used for producing bio-oil and bio-char were normal pyrolysis, slow pyrolysis and fast pyrolysis. These methods require a pretreatment for the feedstock. The pretreatment of a feedstock includes drying, grinding, microwave irradiation, hot water pretreatment, enzymatic pretreatment and also chemical pretreatment. Prior to pyrolysis reaction, a high temperature and longer reaction time is needed around 400°C-700°C and 45-60 min [28]. A study on the pyrolysis of white pine shows that, the feedstock was air dried at 110°C for 1 hr before its pyrolysis and the reaction temperature is also high which is between 400°C-600°C with the highest yield of bio-oil of about 47.2 wt% at 500°C [29]. In the slow pyrolysis of *Spirulina Sp*. Algae to produce bio-oil and bio-char, the feedstock is first needed to be dried at room temperature for one (1) week and reaction was carried out at 500°C for 60 min to produce about 45 wt% and 32 wt% of bio-oil and bio-char, respectively [30].

Sub-CW refer to liquid water between its boiling temperature (370°C) and critical temperature (374°C) under sufficient pressure to maintain its liquid state [31]. At this condition, the interfaces between liquid and vapor phase become indistinguishable [32]. The most important properties of Sub-CW are dielectric constant and ion production constant. Sub-CW has low dielectric constant which causes it to behave as an extraction solvent [32–34] and also high ion production constant which provides an acidic medium for hydrolysis reaction [35]. Using water as solvent offers low operation cost, as well as environmental friendly, pollution free and non-toxic waste. Additional to that, Sub-CW technology requires a shorter time and milder operation temperature compared to normal conventional pyrolysis technology [29,36].

Yuki Katayama and Hiroyuki Yoshida [37], provide an evidence for the suitability of Sub-CW reaction for the production of useful substances such as organic acid, sugars and bio-oil from wood waste (Sawdust-like, Beitsuga).At low temperature range (200°C-400°C) and shorter time (0.1-15 min), Sub-CW reaction was able to produce 50 wt% bio-oil and 20 wt% total organic acids and 10 wt% residual solid. Another study by Omid Pourali et al. [33] utilizing rice bran (*Oryza sativa* L. japonica) shows that 27 wt% of rice bran oil (350°C) and 20 wt% (175°C) of soluble sugars were successfully extracted at reaction time 5 min. These studies give us some indication that Sub-CW reaction may be a better alternative for pyrolysis and SCWO methods in terms of time consumed temperature range and yield of product. Since, there are no latest research utilizing OPT using Sub-CW pyrolysis reaction to produce bio-oil and solid product, therefore this study was done to determine the effect of reaction temperature and time on the yield of bio-oil and solid product produced and also the characteristic of bio-oil and solid product produced from OPT by Sub-CW pyrolysis reaction was analyzed.

1.3 Problem statement

With the projected area of around 235,277 hectares in 2024 made up of old oil palm tree (25-30 years old tree), Malaysia will have about 32.94 million trees (140 trees/ha) available for replanting. With the assumption of OPT at about 75.5 tons/ha (dry matter), Malaysia will generate 17.76 million tonnes of dried matter of trunk [38]. In the early studies, OPT was used for nutrient enrichment in plantation and animal food [18]. The high yield of bio-oil (40.87 wt%) and bio-char (33.60 wt%) were reported obtained from the OPT via pyrolysis at lower temperature and faster retention time (500°C, 60 min) [39]. In addition, OPT is also widely used for the production of particle board, fiberboard, furniture and cement board [18,40].

Normal pyrolysis, slow pyrolysis and fast pyrolysis are the three common methods used for producing bio-oil and bio-char from wood waste. These conventional thermochemical methods require a pretreatment of the feedstock to enhance the efficiency of pyrolysis reaction. Pretreatment will cause a longer preparation time and increases the operation cost for those methods. These conventional methods also require high reaction temperature (400°C-700°C) and longer reaction time (45-60 min) [28]. To overcome these drawbacks, supercritical water oxidation (SCWO) method was developed to reduce the reaction time consumption and increase the production of bio-oil yield. The operating of SCWO is in the same range of pyrolysis reaction which is between 500°C-700°C and 24-50MPa [41,42]. Even the time consume was reduced (10 min) and the production of liquefaction yield was increased (almost 90%) [43], SCWO immerged new drawbacks such as high operation cost, severe corrosion and serious reactor plugging caused by salt precipitate. Due to the severe corrosion problem, an expensive material like nickel-based alloy and titanium must be used to build the reactor [41]. In addition to that, SCWO also uses carbon dioxide (CO_2) as its main solvent which is not environmental friendly [44]. Thus, another suitable method for producing bio-oil and solid product that addresses aforementioned problem is required.

Sub-critical water (Sub-CW) technology is a new promising method which is fast, safe and economical friendly compared to other methods [45]. Producing bio-oil and solid product using Sub-CW method uses water as a solvent which is environmental friendly and cheap [46]. Furthermore, Sub-CW requires significantly less reaction time (15 minutes) at 340°C to produce high quality and quantity of bio-oil (yield of bio-oil: 50wt%) [37].

As mention earlier, finding an alternative method for addressing drawbacks by pyrolysis and SCWO process would be useful for the production of bio-oil and solid product from biomass waste. Yet, to the best of the author's knowledge, there is no study on producing bio-oil and solid product from oil palm trunk (OPT) itself using Sub-CW technology that has been done this far. There are very limited studies on utilizing OPT into bio-oil (heavy oil) and solid product even through pyrolysis process [27,39]. The reaction temperature and time used in pyrolysis process are higher than in sub-CW process. More studies should be done on utilizing OPT into bio-oil and solid product using technology other than conventional process such as Sub-CW process. Product characterization of bio-oil and solid product obtained also should be done.

1.4 Research objectives

This study attempts to fill the mentioned gaps and focuses on the bio-oil and solid product produce from OPT using Sub-CW pyrolysis reaction. There are three main objectives for this research project asfollows:

1. To evaluate the effect of reaction temperature and reaction time on the yields of bio-oil and solid product produced by sub-critical water (Sub-CW) pyrolysis reaction.
2. To determine the optimum condition (reaction temperature and time) for the production of bio-oil and solid product by Sub-CW pyrolysis reaction.
3. To characterize the bio-oil and solid product produced by Sub-CW pyrolysis reaction

1.5 Scope of study

This study will cover only on the production of bio-oil by acetone recovery and solid product in the residual solid phase from the inner part of OPT using Sub-CW pyrolysis reaction in the temperature range between 250°C-370°C and reaction time of 0.1-20 minutes. The gas released after opening the reactor was not collected and analyzed due to the limitation of this type of batch reactor used. The recovered water soluble also not been analyzed and reported due to it being outside the topic of interest and there is no recover of oil in water soluble phase since there was no oil layer observed after transferring all reactor content into the centrifuge tube.

1.6 Thesis Layout

This thesis contains five chapters whereby each of the chapter is summarized as follows: Chapter 1 gives the background of study, the current oil palm biomass produced in Malaysia and problems arising from the biomass produced. The problem statement and research objectives are mentioned to give the reader the overall view of what this thesis is entails.

Chapter 2 begins with a brief description of oil palm tree and end with the potential product from sub-critical water technology. An explanation about composition in OPT and the applications of sub-critical-water as a new green method has been reviewed.

Chapter 3 deals with the materials and methods. The OPT sample preparation, reactor set up, the Sub-CW reaction, Sub-CW pyrolysis reaction and methods to extract bio-oil and solid product are shown, together with the analyzed products obtained.

Chapter 4 gives results and discussion. The effect of reaction temperature and the reaction time on the decomposition of lignocelluloses (hemicelluloses, cellulose and lignin) of OPTs into bio-oil and solid product are discussed. The optimum reaction temperature and reaction time to produce maximum amount bio-oil and solid product are presented. The effect of Sub-CW treatment to the heating values was evaluated. These results are discussed in details.

Chapter 5 summarized the overall finding from this study and recommendations for the study. All important results as well as recommendations for future improvement on the results or methodology are stated.

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LIST OF PUBLICATIONS

Muda, N. A., Yoshida, H., Ishak, H., Ismail, M. H. S., and IZHAR, S. (2019). Conversion of oil palm trunk into bio-oil via treatment with sub-critical water. *Journal of Wood Chemistry and Technology,* 0 (2017):1–17. http://doi.org/10.1080/02773813.2019.1578375. (Accepted January, 2019)

Muda, N. A., Yoshida, H., Ishak, H., Ismail, M. H. S., and IZHAR, S. (2019). Production and properties of solid biochar from oil palm trunk waste using sub-critical water technology. *Journal of Oil Palm Research*, (Accepted March, 2019)

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