



UNIVERSITI PUTRA MALAYSIA

***ACTIVATED CARBON FROM OIL PALM MESOCARP FIBER FOR THE
TREATMENT OF FINAL DISCHARGE OF PALM OIL EFFLUENT***

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FBSB 2016 30



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By

IZZUDIN IBRAHIM

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

June 2016

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

ACTIVATED CARBON FROM OIL PALM MESOCARP FIBER FOR THE TREATMENT OF FINAL DISCHARGE OF PALM OIL EFFLUENT

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June 2016

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Malaysia palm oil industry produced abundant amounts of solid and liquid biomass annually. The current disposal practice is not environmentally friendly, with viable biomass for production of value added product being wasted. It is desirable to reuse the biomass as a feedstock for production of activated carbon and use it to treat palm oil mill final discharge to achieve zero emission system. This study aimed to reduce the COD and suspended solids content of palm oil mill final discharge using oil palm mesocarp fiber activated by steam and phosphoric acid.

The oil palm mesocarp fiber activated carbon was successfully produced either using steam and phosphoric acid as activating agents in a two-step process at a temperature of 600°C for 30 minutes. The resulting activated carbon BET surface area was found to be 494 and 1090 m²/g, respectively, which are comparable to commercial activated carbon. The activated carbon also shows pore development compared to raw material from SEM analysis. Oil palm mesocarp fiber activated carbon was used to treat palm oil mill final discharge and the effect of adsorbent dosage, treatment time, adsorbate concentration and consecutive treatments was studied.

The oil palm mesocarp fiber activated carbon were able to reduce the COD and suspended solids content of the final discharge with a COD percent removal of 70% and 42 % for phosphoric acid and steam activated mesocarp fiber at 10 g/L dosage, respectively. For suspended solids, the removal percent was 85% and 81%, respectively. The amount of pollutant reduced increased as the adsorbent dosage increase and remained constant after 10 g/L. In terms of treatment time, as the treatment time increased, the amount of pollutants that were removed increased until a certain amount of time, which was found to be 6 hours for both COD and suspended solids content. For adsorbate concentration, as the COD

initial concentration increased, reduction of pollutants for phosphoric acid activated carbon maintained, while steam activated carbon shows a higher reduction at lower concentration. For suspended solids, a similar effect was observed. The effect of consecutive treatment was also studied, with the increasing number of treatments resulting in a higher total reduction of pollutants. Consecutive small dosage treatment also proves to be more efficient compared to a single large dosage treatment.

It can be concluded that oil palm mesocarp fiber is a viable feedstock for activated carbon production, with resulting activated carbon able to reduce the pollutants in palm oil mill final discharge.



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

KARBON TERAKTIF DARIPADA SERAT MESOKARP KELAPA SAWIT UNTUK MERAWAT SISA BUANGAN AKHIR KILANG MINYAK SAWIT

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Industri minyak sawit negara menghasilkan sisa biomas yang banyak sepanjang tahun. Kebanyakan biomas yang boleh dijadikan produk baru dibuang begitu sahaja dengan cara yang tidak mesra alam. Biomas itu boleh digunakan sebagai bahan asas untuk penghasilan karbon teraktif yang boleh digunakan untuk merawat sisa buangan akhir kilang minyak sawit untuk mencapai tarap system pelepasan sisa sifar. Kajian ini dijalankan untuk mengurangkan kandungan COD dan pepejal terampai didalam sisa buangan akhir kilang minyak sawit dengan menggunakan karbon teraktif dari serat mesocarp yang dihasilkan menggunakan pengaktifan wap air dan asid fosforik.

Karbon teraktif daripada serat mesokarp kelapa sawit berjaya dihasilkan dengan menggunakan wap air atau asid fosforik menggunakan proses dua langkah pada suhu 600°C selama 30 minit. Karbon teraktif yang terhasil mempunyai luas permukaan seperti karbon teraktif komersil, masing-masing mempunyai luas permukaan BET sebanyak 494 dan 1090 m²/g. Perkembangan liang juga dapat dilihat melalui analisis SEM keatas serat mesocarp yang teraktif berbanding asal. Serat mesokarp kelapa sawit telah digunakan untuk merawat sisa buangan akhir kilang minyak sawit dan kesan dos penjerap, masa rawatan, kepekatan bahan terjerap dan rawatan berturutan turut dikaji.

Serat mesokarp kelapa sawit teraktif boleh mengurangkan kandungan COD dan pepejal terampai didalam sisa buangan akhir sebanyak 70% dan 42%, masing-masing untuk serat mesokarp yang diaktifkan menggunakan asid fosforik dan wap air pada dos 10 g/L. Untuk pepejal terampai, peratus pengurangan adalah masing-masing sebanyak 85% dan 81%.Peningkatan dos bahan penjerap dapat meningkatkan pengurangan bahan pencemar, dan kekal sama dengan penambahan dos melebihi 10 g/L.Ini adalah kerana pertindihan tapak penjerapan oleh partikel penjerap. Dari segi masa rawatan, semakin lama masa

rawatan, semakin tinggi pengurangan bahan cemar, dengan masa rawatan maksimum selama 6 jam untuk kedua-dua jenis pencemar. Ini adalah kerana tapak penjerapan pada bahan penjerap yang akan dipenuhi selepas satu tempoh masa. Dari segi kepekatan bahan terjerap, didapati bahawa kepekatan asal COD tidak memberi kesan yang signifikan terhadap pengurangan bahan pencemar bagi serat mesokarp diaktifkan dengan asid fosforik, manakala serat mesokarp diaktifkan dengan wap air menunjukkan pengurangan yang lebih tinggi dalam julat kepekatan rendah. Bagi pepejal terampai, kesan yang sama diperhatikan, dengan peningkatan kepekatan awal meningkatkan pengurangan pepejal terampai. Kesan rawatan berturutan turut dikaji, dengan peningkatan jumlah rawatan mengakibatkan jumlah pengurangan bahan pencemar menjadi lebih tinggi. Rawatan berturutan menggunakan dos yang kecil secara berturutan terbukti lebih baik berbanding rawatan menggunakan satu dos yang besar.

Dapat disimpulkan bahawa serat mesokarp kelapa sawit adalah bahan mentah yang sesuai untuk pengeluaran karbon teraktif, dengan karbon teraktif yang dihasilkan dapat mengurangkan bahan pencemar dalam sisa buangan akhir kilang minyak sawit.

ACKNOWLEDGEMENTS

Alhamdulillah, with the blessings of Allah S.W.T., I manage to complete the thesis entitled "Activated Carbon From Oil Palm Mesocarp Fiber For The Treatment Of Final Discharge Of Palm Oil Effluent" as a requirement for the Degree of Master of Science. Here, I would like to thank everyone who had helped me in any way during my research period here in Universiti Putra Malaysia.

First of all, I would like to thank my main supervisor, Prof.Dr. Mohd Ali Hassan for his guidance during the period of my study. His advice, critics, and encouragements throughout the years had pushed me through thick and thin in completing the research and finishing up the thesis presented here.

I would also like to thank the supervisory committee members, Prof. Dr, Suraini Abd-Aziz and Prof. Dr. Yoshihito Shirai for their insights, ideas and meaningful comments that helped in shaping the research progress into what it is today. In addition to that, I would like to express my thanks to all Environmental Biotechnology (EB) group members for their help and advice. I would also like to thank Universiti Putra Malaysia and Malaysian Education Ministry in supporting my study financially. Special thanks to Prof. Haruo Nishida and Associate Prof. Yoshito Ando for their help during my attachment in Japan, and Japan International Cooperation Agency for the financial support during the attachment. Last but not least, I would like to express my gratitude to my all my family member, especially my mother, Naaimah Khamis, my father, Ibrahim Yunus, and my brother Muhammad Iqbal for their never ending support.

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LIST OF ABBREVIATIONS

ATR	Attenuated Total Reflectance
BET	Brunauer-Emmett-Teller
BOD	biological oxygen demand
CEFIC	The European Chemical Industry Council
COD	Chemical Oxygen Demand
CPO	Crude Palm Oil
DTA	Differential Thermal Analysis
DTG	Differential Thermogravimetric
FELDA	Federal Land Development Authority
FESEM	Field Emission Scanning Electron Microscopy
FTIR	Fourier Transform Infrared
GLC	Government Linked Company
HCl	Hydrochloric Acid
H ₃ PO ₄	Phosphoric Acid
KBr	Potassium Bromide
KOH	Potassium Hydroxide
MDF	Medium Density Fiberboard
MMT	Million Metric Tonnes
MPOB	Malaysian Palm Oil Board
MPOC	Malaysian Palm Oil Council
NGO	Non-Governmental Organization
OPEFB	Oil Palm Empty Fruit Bunch
OPF	Oil Palm Frond

OPMF	Oil Palm Mesocarp Fiber
OPT	Oil Palm Trunk
OPS	Oil Palm Shell
PAC	Polyaluminium Chloride
PKO	Palm Kernel Oil
POME	Palm Oil Mill Effluent
POMFD	Palm Oil Mill Final Discharge
POMS	Palm Oil Mill Sludge
Proton	Perbadanan Otomobil Nasional
PSD	Pore Size Distribution
SEM	Scanning Electron Microscopy
SS	Suspended Solids
STP	Standard Temperature and Pressure
TG	Thermogravimetric
ZnCl ₂	Zinc Chloride

CHAPTER 1

INTRODUCTION

Since the 1980s, Malaysia had become one of the largest producers and exporters of palm oil and its derivatives in the world, producing 19.79 million metric ton (MMT) of crude palm oil, and exporting 17.31 MMT of it to the world (Malaysian Palm Oil Board, 2014). Consequently, continuous mass production of palm oil creates by-products in the form of solid and liquid waste. Solid waste produced feasible trunks, fronds, empty fruit bunches, mesocarp fiber, kernel shell and press cake from oil extraction process while liquid waste produced are in the form of palm oil mill effluent (POME) and palm oil mill sludge (POMS).

The increasingly large amount of waste biomass produced every year leads to disposal problem. Currently, palm oil mills all around the country burned the waste biomass for energy in boilers (Ali *et al.*, 2015), or recycled as mulching in the palm oil plantation (Yoshizaki *et al.*, 2013). Older practices such as burning the empty fruit bunch to produce oil palm ash have been banned by the government due to its high pollution risks (Abdullah and Sulaiman, 2013). For liquid waste, aerobic treatment using large consecutive ponds in the palm oil mill was chosen due to its low cost and ability to reduce a significant amount of pollutant in the form of suspended solids (SS) and chemical oxygen demand (COD). This process produced palm oil mill final discharge (POMFD) or biologically treated palm oil mill effluent, which is then discarded to the main water body, usually rivers nearby (Othman *et al.*, 2014).

The current practice by the palm oil mills in Malaysia focused on the most economical method, or in other words, cheapest alternative available while at the same time adhering to the rules and regulations set by the government. The downside of this mentality is that the bad effects of such practice towards the environment are often neglected. For solid waste, the current practice promotes seepage of nutrients into the underground water and rivers from uncontrolled uses of mulch from palm oil biomass (Yoshizaki *et al.*, 2013) such as mesocarp fiber and empty fruit bunch. Uses of boilers to produce electricity by burning waste biomass increase the risk of pollution while inefficient boilers produce less electricity than is theoretically possible. On the other hand, discharge of effluent by palm oil mills is governed under the Environment Quality Act 1974 and its subsidiary legislation, where standard discharge limits for liquid effluent were outlined. There is no current federal legislation that governed the disposal of solid waste by the palm oil mills. Even though palm oil mill final discharge is safe to be disposed of according to the discharge limit, the high value of COD and visible color of the POMFD will increase the risk of eutrophication and reduce the aesthetics

of the river water. It is evident that greener, more environmental-friendly method of disposing palm oil mill solid and liquid waste is needed.

The best solution for the problem at hand is to simultaneously dispose of both solid and liquid waste while reducing the effect on the environment. Better yet, treatment of the liquid waste reduces the amount of pollutant to the extent that it can be recycled back to the mill as feed water, while the disposal process of the solid waste produces value-added product that can be used to treat the liquid waste, in this case, POMFD. This concept was proposed by Othman *et al.*, (2014) where palm oil mill final discharge was treated until it reached river water quality so that it can be recycled back as feed water to the mill. Although the treatment was successful, the process uses commercial activated carbon and coagulants. This instead creates additional problems such as large amount of sludge to dispose and additional cost of using expensive activated carbon. The logical next step is to produce activated carbon from oil palm biomass, and use it to treat palm oil mill final discharge. Both disadvantages from previous research are solved while new value added product in the form of activated carbon is produced.

Presently, activated carbon is mass produced from coal, lignite, petroleum pitch or biomass such as coconut shells, via activation using physical or chemical activation agents. Biomass feedstock is first pyrolyzed at 700-1100°C under inert atmosphere (nitrogen or argon). Carbonized feedstock and other carbon precursor such as coal is then exposed to oxidizing atmosphere such as oxygen, carbon dioxide or steam at elevated temperature (600-1200°C). Another method of activation using chemical agents proceeds with impregnation of chemical agents onto the surface of the feedstock. Chemicals used are usually acid (phosphoric acid, H_3PO_4), strong base (potassium hydroxide, KOH) or salt (zinc chloride, $ZnCl_2$). Impregnated feedstock is then carbonized at temperature of about 450-900°C, where carbonization and activation happened simultaneously (Yahya *et al.*, 2015).

Researchers have been investigating the feasibility of using biomass as feedstock for biochar and activated carbon production. A study by Agirre *et al.*, (2013) uses fruit cuttings from Extremadura as feedstock for production of charcoal using screw pyrolysis reactor. The resulting charcoal produced was determined to be suitable for use as reducing agent in metal production due to excellent high carbon content. Another research by Rashidi *et al.*, (2012) activates palm kernel shell, palm mesocarp fiber, rice husk, coconut shell and coconut fiber using carbon dioxide. Activated samples show high porosity through field emission scanning electron microscopy (FESEM) analysis with a yield of about 20%. Even though the research shows the feasibility of oil palm biomass in terms of palm kernel shell and oil palm mesocarp fiber as a precursor for activated carbon production, there is a few problems that needs to be addressed. In terms of biomass, palm kernel shell

is a good precursor due to its high carbon content but its high current price with large reduction of final product weight after activation, will results in loss of revenue to the palm oil mills. The activation agents chosen which is carbon dioxide, adds to the cost of the process compared to using readily available resources in the mills such as excess steam from the sterilization process. Uses of fibers such as oil palm mesocarp fiber and steam as its activating agents can effectively reduce the cost of production, in which will increase the revenue that can be made from the activated carbon.

Although multiple types of biomass have been studied, due to the diverse method of activation used, no significant comparison can be made between each research. In addition to that, no large scale application of previous research has been done, inhibiting progress towards implementation of biomass activated carbon production technology in the industry.

This research is expected to be able to produce activated carbon from oil palm biomass with comparable quality to commercially activated carbon. Activated carbon produced are expected to have Brunauer-Emmett-Teller (BET) surface area of 500 m²/g or above for wastewater treatment utilization. It is also expected that the activated carbon produced is able to treat palm oil mill final discharge effectively, by reducing the residual pollutants in the final discharge down, until it reaches river water quality for use as recycled water in the palm oil mill.

The overall objective of this research is to produce activated carbon with a minimum surface area of 500 m²/g from oil palm biomass using carbonization and activation. This research also targeted that the activated carbon produced can treat palm oil mill final discharge effectively.

Specific objectives:

- To produce activated carbon using oil palm mesocarp fiber via carbonization, physical and chemical activation with at least 500 m²/g surface area.
- To characterize the activated carbon produced in terms of BET surface area, pore size and volume, surface functional groups, and its thermogravimetric properties.
- To determine the performance of activated carbon produced in treating palm oil mill final discharge.

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