# SYNTHESIS AND CHARACTERIZATION OF TEXTILE SLUDGE BIOCHAR FOR OIL ADSORPTION

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# ABSTRACT

Oil contamination such as oily waste from industries is considered as one of the major problems to the sea and also public watercourse. It is also detrimental to marine life and the overall aquatic ecosystem. Thus, this study focuses on the oily wastewater treatment by adsorption process and investigated the potential of textile sludge waste from textile factory which is considered to be abundant in landfills as precursor for biochars. Textile sludge biochars (TSB) was prepared by carbonization in the laboratory tube furnace at a high temperature of 700 $^0$C and held for 60 minutes, with 10 ˚C/min heating rate and 0.2 L/min of $N_2$ gas flow. Biochar yields and moisture content had been reduced while ash and carbon content had been increased after carbonization. Then, TSB was characterized using Fourier transform infrared (FTIR), field emission scanning electron microscopy (FE-SEM), Carbon Hydrogen Nitrogen Oxygen (CHNO), pH zero point charge and Brunauer-Emmet-Teller (BET) surface area. The effects of adsorbent type, pH, initial concentration of simulated oily wastewater, contact time, adsorbent dosage, and temperature towards adsorption capacity of simulated oily wastewater were investigated. The optimal adsorption process condition obtained was at pH 7.4±2, 200 ppm initial concentration of simulated oily wastewater, 60 minutes contact time, 1 mg/ml dosage of biochar 700 $^0$C (BC700), and temperature of 313K with maximum adsorption capacity of 173.4± 0.589 mg/g. The BET study showed that the BC700 has a surface area of 195 $m^2$/g and has a rough surface from the results of FE-SEM which shows that BC700 has the ability to attract oil. The kinetics of adsorption is well described by pseudo-second order while the adsorption equilibrium is well represented by Langmuir isotherm. A regeneration study was carried out and it shows that TSB has the potential for oily wastewater treatment and can be regenerated until five cycles. The findings provided an alternative for low cost and effective adsorbent for oily wastewater treatment.

# ABSTRAK

Pencemaran minyak seperti air sisa berminyak dari industri dianggap sebagai salah satu masalah utama kepada laut dan juga saluran air awam. Ia memudaratkan kehidupan marin dan keseluruhan ekosistem akuatik. Untuk itu, kajian ini menumpukan kepada rawatan air sisa berminyak melalui proses penjerapan dan menyiasat potensi enapcemar tekstil dari kilang tekstil yang banyak dijumpai di tapak pelupusan sebagai sumber arang. Arang enapcemar tekstil (TSB) disediakan melalui karbonisasi pada suhu 700 $^{0}$C selama 60 minit, dengan kadar pemanasan 10 ˚C/min dan aliran gas $N_2$ 0.2 L/min. Selepas karbonisasi, hasil arang dan kandungan air telah berkurang manakala kandungan abu dan karbon telah meningkat. BC700 dicirikan menggunakan Inframerah transformasi Fourier (FTIR), mikroskop elektron pengimbas pancaran medan (FE-SEM), Karbon Hidrogen Nitrogen Oksigen (CHNO), titik sifar cas pH dan luas permukaan Brunauer-Emmet-Teller (BET). Kesan jenis penjerap, pH, kepekatan awal air sisa berminyak simulasi, masa penjerapan, dos penjerap, dan suhu terhadap keupayaan penjerapan air sisa berminyak simulasi telah disiasat. Keadaan optimal bagi proses penjerapan ialah pada pH 7.4±2, 200 ppm kepekatan awal air sisa berminyak simulasi, 60 minit masa penjerapan, 1 mg/ml dos penjerap arang 700 $^{0}$C (BC700) dan pada suhu 313K dengan keupayaan menjerap maksimum sebanyak 173.4±0.589 mg/g. Keputusan BET menunjukkan BC700 mempunyai keluasan permukaan sebanyak 195 $m^2$/g dan keputusan FE-SEM menunjukkan BC700 mempunyai permukaan yang kasar yang berupaya untuk menjerap minyak. Kinetik penjerapan sangat berpadanan dengan model kinetik pseudo-tertib-kedua, manakala, isotherma sangat mematuhi model isotherma Langmuir. Kajian penjanaan terhadap penjerap turut dilakukan dan menunjukkan BC700 berupaya sebagai penjerap minyak dan boleh dijana semula sehingga lima kitaran. Penemuan ini memberikan satu alternatif untuk bahan penjerap yang berkos rendah dan efektif untuk rawatan air sisa berminyak.

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# LIST OF SYMBOLS

| | | |
|---|---|---|
| $Q_m$ | - | Maximum monolayer adsorption capacity |
| $Q_e$ | - | Amount of adsorbate adsorbed at equilibrium per unit Mass sorbent |
| $K_L$ | - | Free adsorption energy constant |
| l | - | Litre |
| $C_0$ | - | Initial concentration |
| $C_t$ | - | Concentration at a time |
| m | - | Mass of sorbent |
| $M_b$ | - | Mass of raw textile sludge before |
| $M_a$ | - | Mass of raw textile sludge after |
| $R_L$ | - | Separation factor |
| $V_{sample}$ | - | Volume of analyzed wastewater sample |
| $C_e$ | - | Equilibrium concentration of solute |
| $K_F$ | - | Constant for elative adsorption capacity of the adsorbent |
| µm | - | Micrometer |
| mg | - | Milligram |
| 1/n | - | Intensity of the adsorption |
| b | - | Heat of sorption |
| A | - | Binding constant |
| $K_{RP}$ | - | Peterson constant |
| $\alpha_{RP}$ | - | Peterson constant |
| $q_e$ | - | Equilibrium amount of solute adsorbed per unit mass of adsorbent |
| $k_1$ | - | Constant for pseudo first order |

| | | |
|---|---|---|
| $k_2$ | - | Constant for pseudo second order |
| $\Delta G^\circ$ | - | Free Energy Change |
| $\Delta H^\circ$ | - | Enthalpy Change |
| $\Delta S^\circ$ | - | Entropy Change |
| $K_c$ | - | Equilibrium constant of adsorption |
| R | - | Gas constant (8.314 J/mol K) |
| $k_{id}$ | - | Intra-particle diffusion rate constant |
| $X_i$ | - | Coded value |
| Y | - | Dependent variable |
| WAS | - | Waste Activated Sludge |
| TS | - | Textile sludge |
| TSAC | - | Textile sludge activated carbon |
| $Z_i$ | - | Actual value |
| $Z_i$* | - | Step change |

# LIST OF ABBREVIATIONS

| | | |
|---|---|---|
| TS | - | Textile Sludge |
| TSB | - | Textile Sludge Biochars |
| BC200 | - | Biochar 200˚C |
| BC400 | - | Biochar 700˚C |
| BC700 | - | Biochar 700˚C |
| FTIR | - | Fourier Transform Infrared |
| FE-SEM | - | Field Emission Scanning Electron Microscopy |
| CHNO | - | Carbon Hydrogen Nitrogen Oxygen |
| BET | - | Brunauer–Emmett–Teller |
| BJH | - | Barrett-Joyner-Halenda |
| PPM | - | Part Per Million |
| NAD | - | Nitrogen Adsorption Desorption |
| O/W | - | Oil/Water |
| UV | - | Ultra Violet |
| PAHs | - | Polycyclic aromatic hydrocarbons |
| COD | - | Chemical Oxygen Demand |
| MF | - | Microfiltration |
| UF | - | Ultra-filtration |
| RO | - | Reverse Osmosis |
| A-PAM | - | Anionic Polyacrylamide |
| PISS | - | Zinc Silicate |
| POME | - | Palm Oil Mill Effluent |
| MSW | - | Municipal Solid Waste |
| HTC | - | Hydrothermal Carbonization |
| EBC | - | European Biochar Certificate |

# LIST OF APPENDICES

# CHAPTER 1

# INTRODUCTION

## 1.1 Research Background

Oil contamination is one of the major problems either to the sea or any public watercourse for the last three decades. Oil contamination is caused by the different types of sources and occurred in various forms. Since oil is one of the major sources of energy and is essentially needed for daily usage, its consumption has increased over the time. Thus, petrochemical and petroleum refinery as well as oil based product manufacturing industries has increased in number. The usage of cooking oil in the restaurant has also led to the production of huge amount of oily wastewater. To eliminate the waste oil, the restaurant owners usually took the easy way by throwing those wastes into the river without any proper treatment. The adverse impact from the direct discharge of oily wastewater into the drains and rivers is burdensome to the wastewater treatment provider. It is a challenge for them to provide clean water to the residents and industries. Therefore, this study focuses on the treatment of huge amount of oily wastewater by the restaurants and food manufacturing industries by using biochars.

Oily wastewater can affect the aquatic ecosystem, vegetation, and marine birds life (Kingston, 2002). The common problems that are caused by this oily wastewater pollution are unpleasant odors and dirty appearance. The performance of purification process is also affected by the oily wastewater pollution. Thus, it is detrimental to environment. Due to the serious issue of oily wastewater pollution for the last three decades, it is necessary to conduct research in finding the environmentally friendly and efficient method to treat the oily wastewater.

Many years beforehand, a lot of studies have been conducted with regards to the oil contamination problem. Various treatment methods were proposed for example adsorption (Hussein *et al*., 2008), membrane filtration (Hang *et al*., 2003), electroflotation (Hosny, 1996), flocculation and coagulation (Renault *et al*., 2009) and biological treatment. All of these methods have advantages and their own flaws.

Biological treatment for oily wastewater is effective but the operational cost and the size of reactor make its application difficult in most small city restaurants (Zulaikha *et al*., 2014) whereas the coagulation process was found not applicable to remove finely dispersed oil particles and there is possibility that the water is contaminated by the chemicals coagulant. Moreover, it was also reported that under general operating conditions, the removal of oil and grease and suspended solids by electrocoagulation and electroflotation processes is not effective (Yang *et al*., 2012). The electrocoagulation and electroflotation processes are also unable to produce effluent that meets the acceptable level especially when the COD of wastewater are abnormally high which is higher than 400 mg/l (Qin, 2011).

Meanwhile for membrane technology, there is membrane fouling which includes small emulsified oil particles and contaminants. After considering the advantages and the flaws of each method, adsorption is presumed to be the best wastewater treatment method due to its universal nature, inexpensiveness and ease of operation (Ali *et al*., 2012; Ibrahim *et al*., 2009). Adsorption is a method that applies an adsorbent for oil removal. An adsorbent must have all appropriate characteristics in order to make it effective and can be regenerated. The best adsorbent to treat oily waste water must be the one that has the characteristics of being environmentally friendly, hydrophobic, oleophilicity, porosity, have an interaction site, biodegradable and easy to use.

From the previous research, a natural adsorbent is considered as the best adsorbent for adsorption process (Hussein *et al*., 2008; Abdullah *et al*., 2010). The natural adsorbents are produced from kapok, barley straw, coconut husk, cotton grass, sugarcane bagasse, palm oil shells, chitosan and wool fibers (Wahi *et al*., 2013). The utilization of wastes and residue waste products from industries and agriculture also has led to a high interest among researchers due to the economical alternative source materials of adsorbent for environmental protection. Mohan *et al*. (2014) had claimed that activated carbon was normally thought as a universal adsorbent for water treatment which was normally synthesized from biomass.

However, activated carbon is costly to produce even if it is ideal for removing contaminants from water. On the other hand, ‘‘sustainable’’ biochar requires less investment. In comparison, biochar has more hydrogen and oxygen which stay in its structure together with the ash originating from the biomass. This is due to the fact that biochars are less carbonized than the activated carbon. Biochar could replace coal, coconut shell, and wood-based activated carbons as a low cost sorbent for contaminants and pathogens. Biochar is also used for removing contaminants from water while also being loaded with nutrients for subsequent use as a soil amendment, providing long-term sorption capacity and fertilizer.

Textile sludge (TS) is one of the alternative materials to produce biochars. This material is also known as one of the contributors to landfills abundance. In this study, an adsorbent from textile sludge was synthesized. Based on the data of textile sludge (TS) sewage obtained from Anfi Industries Sdn. Bhd., Batu Pahat, Johor, textile sludge has a potential to be a precursor for biochar due to the percentage of carbon it possesses, which is 24%. Although TS has a lower carbon content as compared to other precursor such as wood and coal, it is feasible to be used as biochar's source. These biochars are synthesized through carbonization process with the absence of oxygen.

## 1.2 Problem Statement

Nowadays, the world is facing with a serious oil contamination problem either to the sea or public watercourse and this problem has caused harmful effects to the environment and living things especially to the marine life as well as to the humans. Many studies have been conducted in order to obtain the best treatment of oil contamination. At the same time, strict legislations had been enacted in order to overcome the serious oil pollution incidents. According to several studies done on the oil contamination treatment, adsorption is a treatment that has been considered to be easily operated, cheap, can change from oil in liquid phase to semi-solid or in solid phase, high sorption capacity, did not use other equipment and the availability of the materials (Abdullah *et al*., 2010). Furthermore, little attention has been paid to the exploration of using adsorption process for restaurant's wastewater and this can be reflected by the limited number of technical papers available in the literature.

A good adsorbent must be cost effective and environmentally friendly yet effective. Low cost is considered the favourable criterion in the selection of adsorbent. According to the study by Gupta (2009), low cost alternative adsorbent comes from precursors like natural materials and industrial or domestic wastes. Textile sludge sewage is one of the by-product wastes of textile industry that has potential to be used as an alternative source of adsorbent synthesis. Moreover, the utilization of textile sludge sewage to biochars will reduce the amount of abundant textile waste in landfill and can be converted into something that is more valuable and useful for the environment. The utilization of waste or any industrial by-product also leads to the cost reduction of environmental protection. It can be said that this study has a double benefit, first it is economical due to the useful product that can be produced from a waste at lower cost. Second, it is environmentally friendly because the waste can be disposed effectively by utilizing the waste into useful products.

Therefore, this study investigates the synthesis of biochars from textile sludge sewage. In addition, no research has been conducted on biochars based on textile sludge for treatment of oily wastewater. Thus, this study also aims to explore the potential of biochars from textile sludge as an adsorbent for oily wastewater treatment.

## 1.3 Hypothesis of Research

The hypothesis for this research is the textile sludge from textile waste can be used as a precursor of biochar. The biochar produced from the textile sludge can give a high adsorption capacity. A high carbonization temperature is necessary to produce biochar with a large BET surface area. The hydrophobic, oleophilic, rough surface and microporous material are essential criteria for the adsorption of oily wastewater. The adsorbent from the textile sludge biochar is easy to use, environmentally friendly, has high adsorption capacity and low cost. The performance of biochars as oily wastewater adsorbent can be regenerated for five treatment cycles. Moreover, the findings can provide an alternative for more effective adsorbent for oily wastewater treatment by adsorption process.

## 1.4 Objectives of Research

This research embarks on the following objectives:

1) To synthesize and characterize textile sludge biochars.
2) To determine the optimum adsorption conditions in terms of pH, contact time, initial concentration of simulated oily wastewater, and dosage of biochars for adsorption process.
3) To study the adsorption mechanism of biochars as an adsorbent in the treatment of simulated oily wastewater.
4) To study the regeneration capacity of biochars as adsorbent for oily waste water adsorption treatment.

## 1.5 Scope of Research

The scopes of this research are:

- The synthesis of biochars was done by carbonization process. Preliminary studies were conducted in choosing the best adsorbents (different carbonization temperature) based on adsorption performance of oil. The carbonization was done at temperatures between 105 -700˚C for 1 hour with nitrogen flow of 0.2 L per minute. The textile sludge and biochars produced were characterized first by several tests like percentage yield, moisture content and ash content analysis. Then, the optimum adsorbent was characterized through Fourier Transform Infrared (FTIR) Spectroscopy, Field Emission Scanning Electron Microscopy (FE-SEM) Analysis, CHNO analysis, pH zero point charge and BET method.

- The performance of the best carbonization temperature adsorbent was tested by batch adsorption by varying several parameters. The four independent parameters (adsorption contact time in min, amount of dosage of the biochars, initial concentration of simulated oily wastewater, pH and temperature of the simulated oily water) were used in this test. The range for the design conditions was 1 min to 120 min for contact times, 50 to 500ppm for range of initial concentration, 0.6 to 4 mg/ml for adsorbent dosage and 3 to 11 for pH and temperature in range 303, 313, 323, 333 and 343 K.

- The mechanism of the adsorbent from textile sludge biochars was obtained by using several kinetic models (pseudo-first order, pseudo second order and diffusion model), isotherms (Langmuir, Freudlich, Temkin) and thermodynamics equation.

- The regeneration efficiency of biochars as oily wastewater adsorbent was studied by using iso-propanol as desorption agent.

## 1.6 Significance of Research

This study was proposed based on the need of having an effective, low cost, simple and environmentally friendly sorbent for oily wastewater treatment. This study also proposes to provide an alternative of biochars by using textile sludge waste as the precursor. This research also led to the exploration of new biochars from textile sludge for oily wastewater treatment. In local textile industries, there are many different steps in dyeing and finishing processes which produced great volumes of wastewater. The wastewater from printing and dyeing is treated before draining and eventually producing abundant sludge which is thrown away to landfills. Thus, the research was also proposed based on the need of sludge reduction that had been produced from local textile industries. The study will also be beneficial for the textile industry to comply with zero waste.

Furthermore, the research was carried out in view of the need to reuse and utilize the sludge for valuable products like biochars production. Moreover, by using textile sludge waste as a precursor of biochars for removal of oil is an economical and ecological approach. It is because no chemical agents are emitted out of the river, sea and other watercourse during the adsorption process. It is hoped that this research can be a reference for commercial and industrial application and also as a solution for the problem of getting an effective sorbent for oil pollution.

## 1.7 Thesis Outline

There are five chapters in this thesis and each chapter explains the sequence of this research.

**Chapter 1** presents a brief overview of oily wastewater, its effect and common methods used to treat oily wastewater. This chapter also presents the problem statement, hypothesis, research objectives, scopes and significance of study.

**Chapter 2** explains the in-depth view of related knowledge about oily wastewater and the available treatment methods nowadays, reasons why adsorption was a preferable method, the advantages and flaws of available treatment, biochars, textile sludge and the isotherms, kinetics and thermodynamic equation of adsorption process used in this research. Moreover, this chapter also focuses on biochars; its sources, synthesis of biochars and its application to treat oily wastewater.

**Chapter 3** refers to the material and methods that explained the experimental procedure that were used in this research for the preparation of biochars from textile sludge as adsorbent. This chapter also shows the analysis of the synthesis of biochars, the characterization, oil adsorption and regeneration study.

**Chapter 4** presents the obtained results covering optimum conditions of oil adsorption process by textile sludge biochar, physical study on biochars including Fourier Transform Infrared (FTIR) spectroscopy, Field Emission Scanning Electron Microscopy (FE-SEM) analysis, pH Zero Point Charge, CHNO analysis and Brunauer–Emmett–Teller (BET) surface area analysis. The analysis of isotherm, kinetics and thermodynamic studies are also covered in the chapter.

**Chapter 5** presents the summary of the research findings and some practical recommendations for future works.

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