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Synthesis of Magnetic Biochar Derived from Oil Palm Frond for Waste Cooking Oil Treatment

Nur Izzah Mohamad Ketar @ Mokhtar[1], **Hasnida Harun**[1]*

[1]Department of Civil Engineering Technology, Faculty of Engineering Technology, Universiti Tun Hussein Onn Malaysia, 84600 Pagoh, Johor, MALAYSIA

*Corresponding Author Designation

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Abstract: Nowadays, oil frying method is widely used causing the accumulation of waste generated from cooking oil. Improper cooking oil waste management leads to the discharge into the environment causing land and water pollution. This study investigates the potential of magnetic biochar from oil palm fronds as a low-cost bio-adsorbent. It was characterized by FTIR, and SEM. Pyrolysis and co-precipitation method was used. Batch experiments of the magnetic biochar were conducted with different dosages, oil concentration and contact time to find out the optimum condition for the water and oil separation. The magnetic biochar's well-cyclability is also observed. The characterization of magnetic biochar shows strong peak at 2341 cm^{-1} that represents the symmetric stretch of vibration of strong O=C=O stretching of carbon dioxide and 2113 cm^{-1} that represents strong N=C=S stretching of isothiocyanate. By using SEM, it can be clearly observed, the surface of the magnetic biochar oil palm frond consists of shining bulky particles and whitish patches structure. From the batch experiment's results, the optimum efficiency of magnetic biochar was 120 seconds of contact time in 1.00 % oil concentration by using 1.5 g magnetic biochar with separation efficiency of 95.00 %. In addition, after 3 cycles of the adsorption process, the separation efficiency of oil waste was still around 94.00 %. Therefore, this study revealed that the magnetic biochar from oil palm fronds is a good candidate for application in the clean-up of waste cooking oil that being spill in drainage

Keywords: Magnetic Biochar, Oil Palm Frond, Pyrolysis, Co-precipitation, Carbonized, Waste Cooking Oil

1. Introduction

The access to safe, protected water is an important issue in many developing countries. Disposal of oily water is becoming an urgent issue. Excessive waste of oil generated from human daily basis activities such as cooking have given out many adverse impacts to the environment. Oil pollution can have a devastating effect on the water environment. Therefore, it needs to be treated properly with high efficiency. The objectives of this study are to synthesis a low-cost magnetic biochar from agricultural

**Corresponding author: hasnidah@uthm.edu.my*

waste which is oil palm and to analyze the performance of the magnetic biochar in treating waste cooking oil. The usage of cost-effective materials such as waste from agricultural sector is one of the best methods to improve water treatment. This study is beneficial towards the environment, community and industry by investigating a more desirable and excellent way to treat wastewater using a biomaterial waste.

1.1 Waste cooking oil

Huge quantities of waste cooking oils and animal fats are available throughout the world, especially in the developed countries. Management of such oils and fats pose a significant challenge because of their disposal problems and possible contamination of the water and land resources. In the fast-food business alone, a single branch which serves fried food can produce as much as 15 liters of used cooking oil per day. Considering that there are hundreds of these outlets in Malaysia, the total amount generated can reach several thousand liters per day. Properties of degraded used cooking oil after it gets into sewage system are conductive to corrosion of metal and affects installations in wastewater treatment plants.

1.2 Oil Palm Waste

Malaysia has been recognized for being one of the world's top manufacturers and exporters of palm oil products in past decades. Oil palm plantations take up Malaysia's largest agricultural sector with planting area of approximately 5.7 million hectares in 2016. The subsequent process unwittingly produces vast quantities of oil palm waste such as mesocarp fibers, palm kernel shells, empty fruit bunches and oil palm frond. According to the Malaysia Palm Oil Board (MPOB), gross crude palm oil (CPO) output in Malaysia was 3.4 million tons in 2016, resulting in approximately 25.5 million tons of OPW while 75.00 % of solid waste was generated from 10.00 % of CPO [1]. However, all these agricultural wastes can turn into wealth if being managed with the right way. A large profit can be achieved if the oil palm waste turn into beneficial products that can be used by human.

1.3 Magnetic biochar

One of the beneficial products that had been invented is oil palm frond biochar for water treatment by using pyrolysis method to treat water. Biochar refers to the black carbon generated from biomass, which is commonly formed by burning biomass in a minimal amount of oxygen and transforms it into char [2]. Some additional elements such as magnetic properties can be added to the biochar in order to cultivate the separation process in waste cooking oil water. It has some benefits compared to traditional treatment techniques, such as lower demand, smaller occupied area and shorter processing period [3]. Higher efficiency to treat oil in waste cooking oil water is expected as the result of the added elements. The incorporation of this biomass into something much more valuable and beneficial can also help to put down the problems of waste disposal without many troubles [4].

1.4 Adsorption process

Adsorption is a process of separating substances from two phases such as oil-liquid, gas-liquid, gas-solid, and oil solid at the interface. Adsorption may be divided into two classes; physisorption and chemisorption, based on the interactions between the adsorbents. The main benefit of adsorption technique is the recycling or regeneration of the adsorbent materials which makes it economical. The adsorbent recycling depends upon the adsorbent's desorption or regeneration capacity. The adsorption of surfactants is based on the adsorbent, adsorbent and solution properties [5]. The properties of adsorbate, adsorbent and solution play vital roles in the adsorption of surfactants. Recent progress in the development of adsorbents for surfactants removal. Many adsorbents have been developed for the removal of surfactants from wastewater. The adsorbents can be categorized into organic and inorganic. Selected adsorbents and their adsorption performance are described below in relation to their characteristics [6].

2. Materials and Methods

2.1 Pretreatment of Oil Palm Frond

The oil palm frond was collected at Muar, Johor's oil palm plantations near the residential area. The quantity of the oil palm fronds collected was estimated to be 500 grams. The oil palm frond was then kept in airtight container for the pretreatment process. The collected biomass was dirty and contains moisture. Therefore, preliminary preparation of the oil palm residues by mechanical and physical processing was required before being used in the pyrolysis process. After the leaves and bark are removed, they were cut into smaller sizes. Then, palm fronds were left to dry under the sun for a few hours until they were dry to reduce moisture content.

2.2 Preparation of waste cooking oil

Waste cooking oil was used for oil removal experiment. The sample of waste cooking oil is held in a storage container before it is used. In the first batch, 2.5 ml waste cooking oil, it was stained with 0.1 g red oil o and was stirred until the solution is homogeneous. After that, the solution was mixed with 1000ml distilled water and shaken until mixed as shown in Figure 1. Another second and third batch of waste cooking oil were made with 10.0 ml and 50.0 ml waste cooking oil with 0.4 g and 2.0 g of red oil o mixed with 1000.0 ml of distilled water.

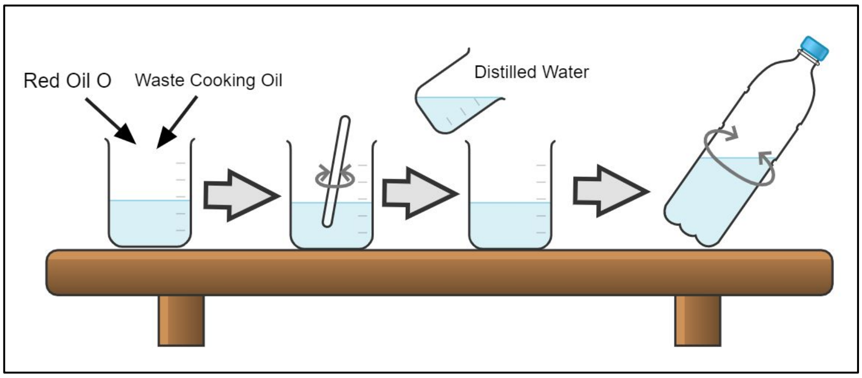

Figure 1: Procedure for preparation of synthetic waste cooking oil

2.3 Preparation of magnetic biochar

The pyrolysis method was used in the preparation of biochar where the biochar was carbonized at 400 ºC for 1 hour after drying at 120 ºC. Then, the carbonized product was impregnated with $ZnCl_2$ and exposed to 700 ºC for 2 hours and washed several times with HCl (0.1 mol L^{-1}) followed by deionized water until the range of pH was 6.0–6.5. After that, the biochar was sieved after being dried and through 100–160 micrometer mesh carbon. The as-prepared biochar (20.0 g) was then suspended in a beaker with 200 mL of deionized water. $FeCl_3 \cdot 6H_2O$ (20.0 g) and $FeSO_4 \cdot 7H_2O$ (11.1 g) was mixed with 600 mL of deionized water and stirred until they were dissolved completely at room temperature (20–25 ºC) for 20 min. Thereafter, 10 M of NaOH was added drop wise into the mixed suspension until the pH was 10–11. After being stirred for 1 hour, the suspension was boiled for 1 hour and filtered. The filtrate was washed with deionized water and ethanol for several times and dried at 70 ºC for 12 hours in a drying oven. Figure 2,3,4 and 5 shows the illustration process preparation of magnetic biochar oil palm frond performed in this study.

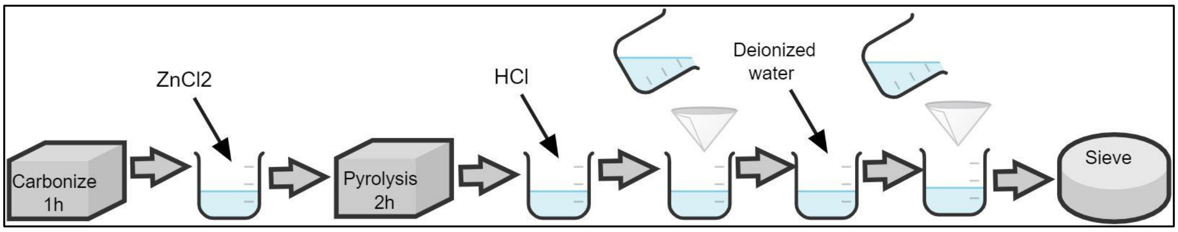

Figure 2: Procedure for preparation of biochar

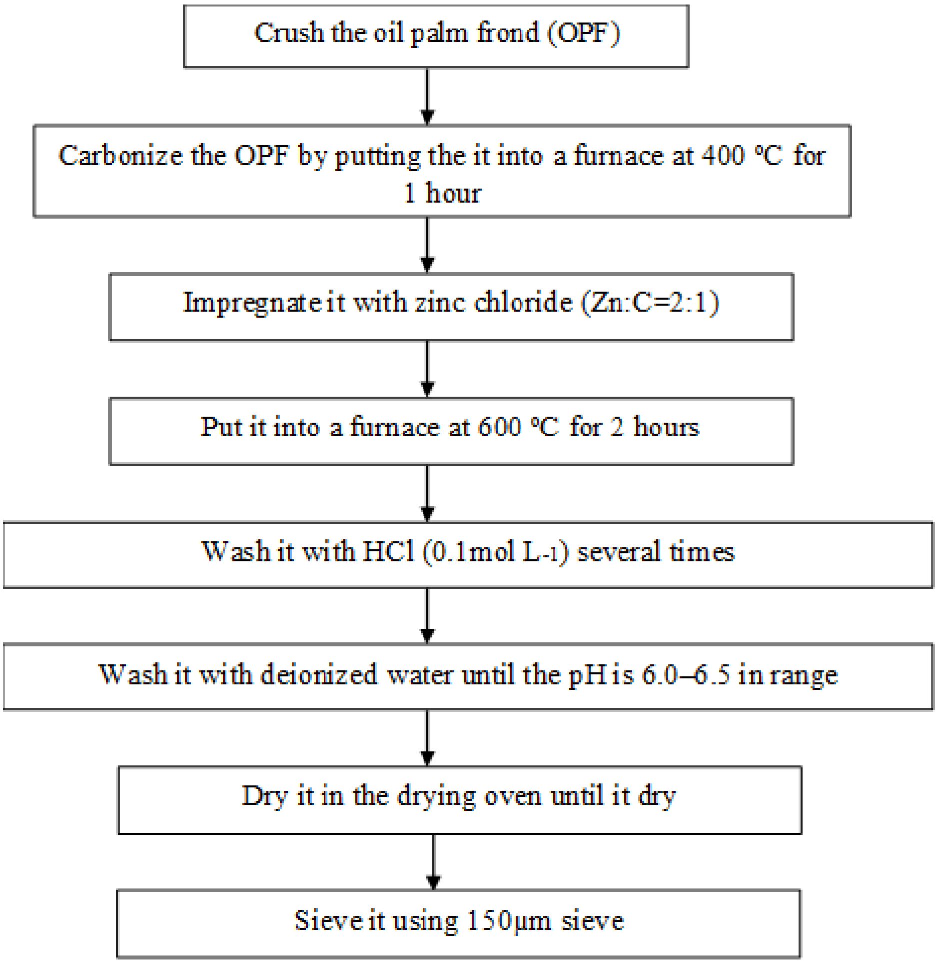

Figure 3: Flowchart preparation of biochar

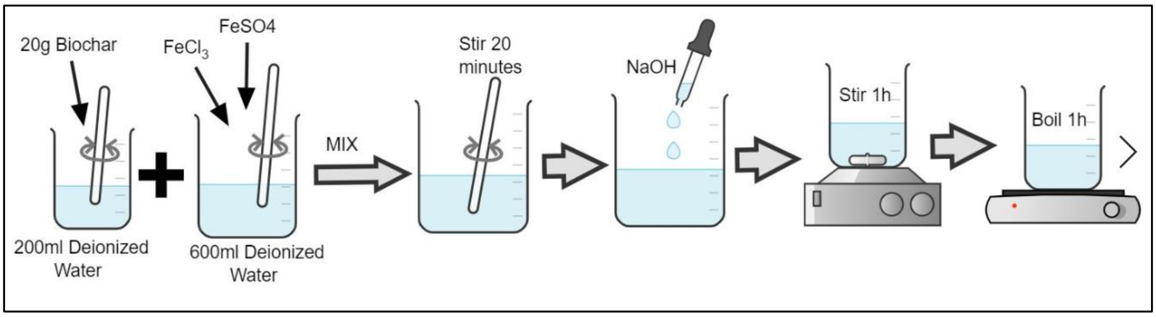

Figure 4: Procedure for preparation of oil palm frond magnetic biochar

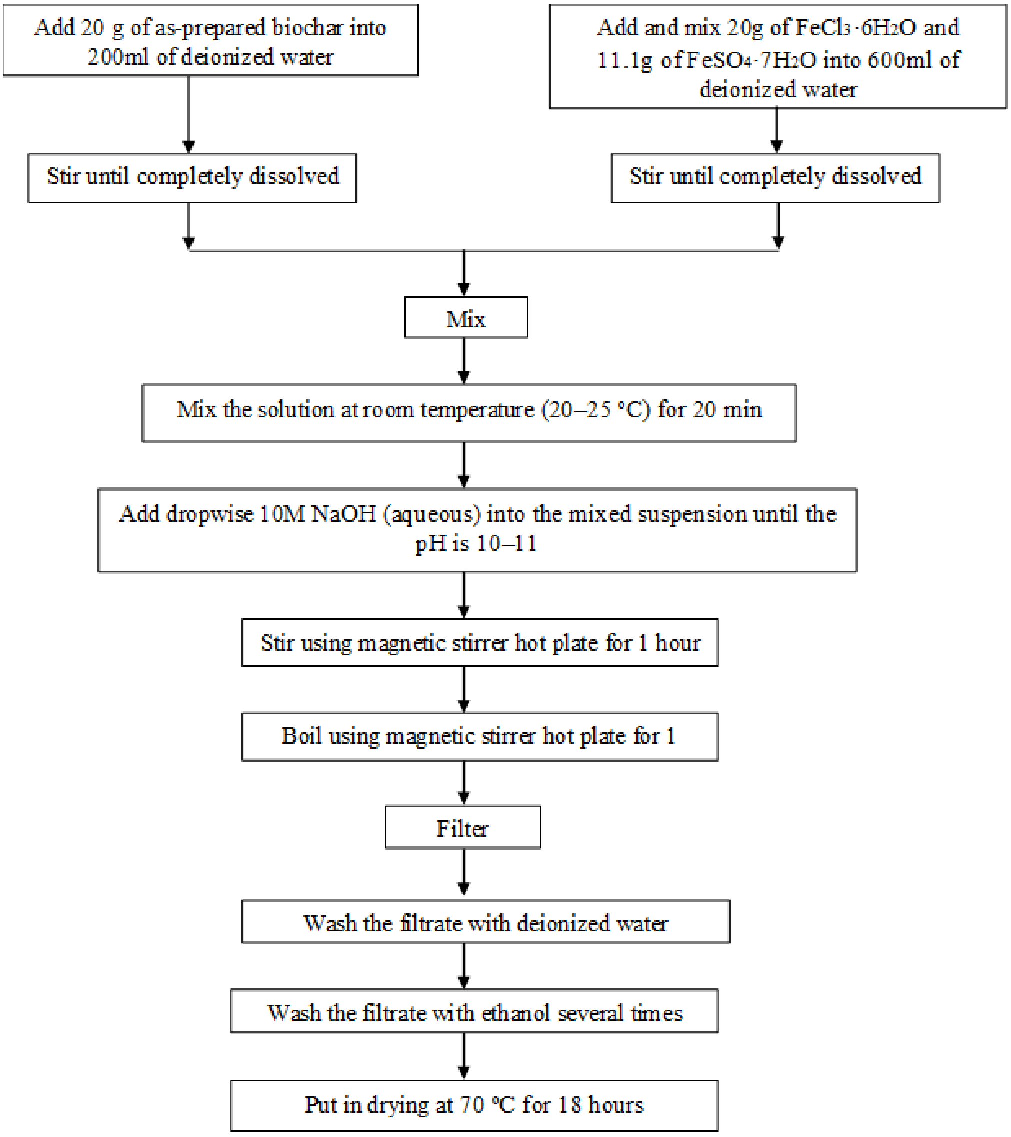

Figure 5: Flowchart preparation of magnetic biochar

2.4 Adsorption Study

Batch experiments were carried out to determine the effect of magnetic biochar dosage, contact time and concentration of oil on the adsorption performances. The amounts of dosage used in the batch experiment were 0.5, 1.0 and 1.5 g. The value of pH used were ranging from 6.5 to 7.5. The adjustment of the pH of solution was done by using analytical reagent grade such as hydrochloric acid (HCl) and sodium hydroxide (NaOH). Contact time between the magnetic biochar and the waste cooking oil water in this experiment started with 10 seconds. After that, it increased into 60 seconds and the longest contact time was 120 seconds. The amount of concentration of waste cooking oil water used were 0.25, 1.00 and 5.00 %. At the end of the experiment, the magnetic biochar was separated from water with a magnet and the removal percentage of the oil was measured.

The percentage of removal are calculated using the following equation:

$$R\ (\%) = \quad \times 100\% \qquad \textit{Eq. 1}$$

Where Co and Ct are the initial concentration and equilibrium concentration of oil at time, t in solution respectively (mg/L) and R is the removal percentage (%).

2.5 Recyclability test

The magnetic biochar derived from oil palm frond that showed a higher adsorption performance was selected for recyclability test. Such recycle adsorption of waste cooking oil on magnetic biochar was conducted three times to evaluate the recycle efficiency.

3. Results and Discussion

3.1 Characterization of magnetic biochar

3.1.1 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) identifies chemical bonds in a molecule by producing an infrared absorption spectrum. The spectra produce a profile of the sample, a distinctive molecular fingerprint that can be used to screen and scan samples for many different components. To have a low-cost, rapid, and easy to use method for non-destructive screening of biochar, FTIR spectroscopy could be a possible option. Not only has it been used to obtain highly structural information on various biochar and starting materials but has also been applied to investigate the structural changes of biochar as a function of pyrolysis temperatures [7].

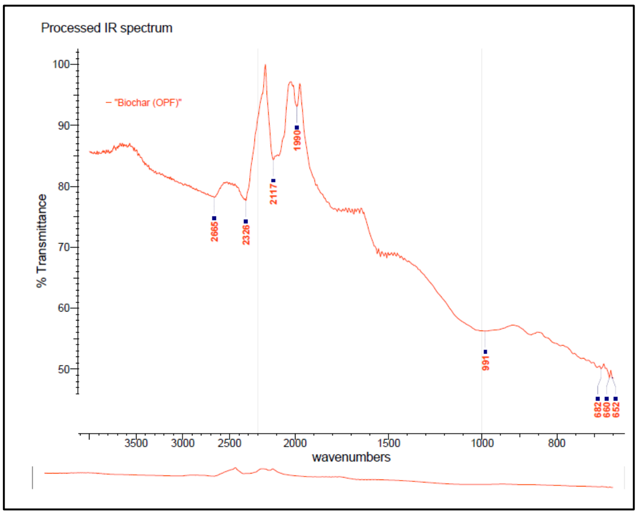

Figure 6: FTIR Spectra of Biochar

Figure 6 shows the characteristics of functional groups of oil palm frond biochar. With unique absorptions in the region from 4000 to 600 cm^{-1}, FTIR spectra represent the fundamental absorbance of functional groups in biochar [7]. The adsorption peaks indicate the structure of the adsorbent. The figure shows a strong peak at 2665 cm^{-1} that represents the symmetric stretch of vibration of medium C-H stretching of aldehyde and 2117 cm^{-1} that represents strong N=C=S stretching of isothiocyanate.

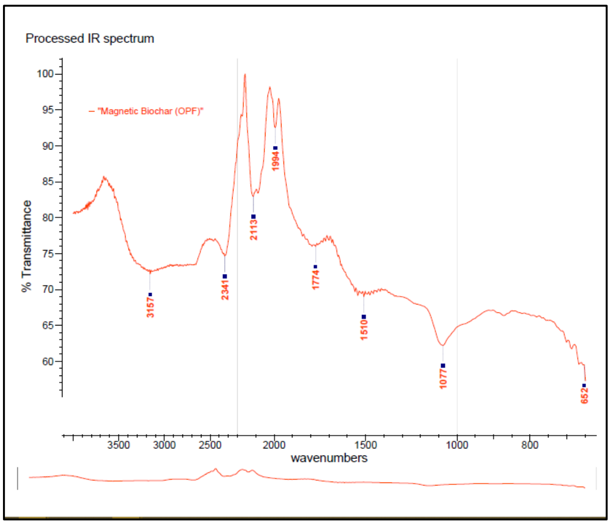

Figure 7: FTIR Spectra of Magnetic Biochar

Figure 7 shows strong peak at 2341 cm-1 that represents the symmetric stretch of vibration of strong O=C=O stretching of carbon dioxide and 2113 cm-1 that represents strong N=C=S stretching of isothiocyanate. The adsorption behavior of novel magnetic biochar is also influenced by the chemical reactivity of the surface, especially in the form of chemisorbed oxygen in various forms of functional groups [8].

3.1.2 Scanning Electron Microscope (SEM)

The surface morphology and developed pores for the carbonized samples were analyzed using scanning electron microscope (SEM). SEM generate a variety of signals at the surface of magnetic biochar. The signals that derive from electron-sample interactions reveal information about the sample's surface topography and morphology. Figure 8 revealed the porous structure of the palm frond bio-char surface. It shows there are large number of pores at the surface of magnetic biochar. It can be clearly observed, the surface of the palm frond biochar consists of shining bulky particles and whitish patches structure.

The pores structure of biochar is developed within a certain range of temperature. When the temperature is high, the quantity of microporous and large hole increased due to microporous collapse therefore it will lead to increasing of specific surface area [9]. After being disposed by FeCl3, the surface of magnetic biochar formed smaller pore channels. Metal clusters were clearly observed on both surface and within the pore channel of magnetic biochar. This indicated that the magnetic particle had been assembled on the surface of magnetic biochar [10]. A porous structure can be found in porous biochar which has a smooth surface. After mixing with the iron and calcination, a lot of crystals grew on the surface of the porous biochar, indicating that the porous biochar can be well combined with magnetic particles by calcination [11].

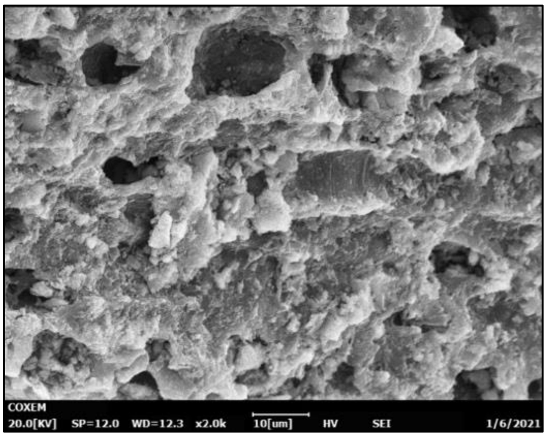

Figure 8: Magnetic biochar (2000x magnification)

3.2 The Performances of Magnetic Biochar

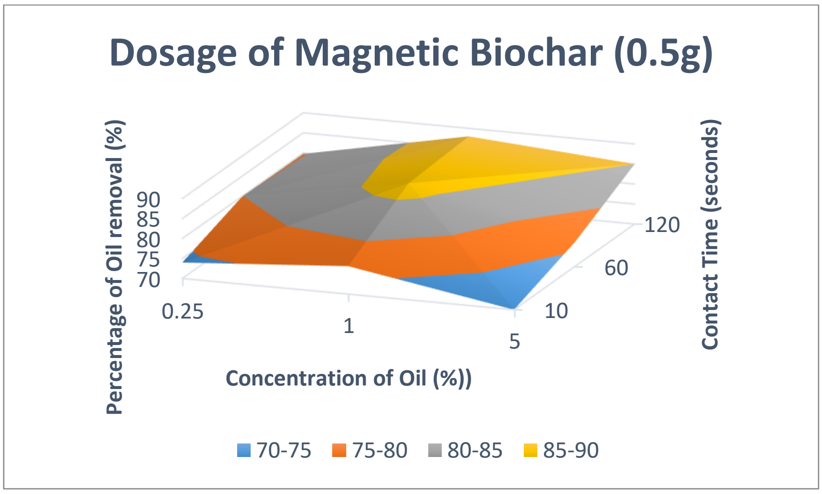

Figure 9: Percentage removal of 0.5 g magnetic biochar

Based on the graph in Figure 9, the highest percentage of oil removal is during 1.00 % concentration of oil with 120 seconds contact time where the 88.00 % of oil was removed. The lowest percentage of oil removal is during 5.00 % concentration of oil with 10 seconds contact time where only 43.00 % of oil was removed.

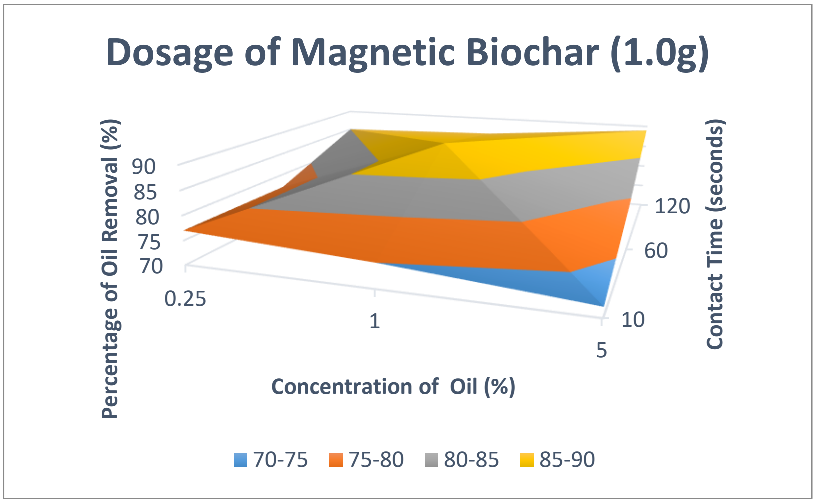

Figure 10: Percentage removal of 1.0 g magnetic biochar

Based on the graph in Figure 10, the highest percentage of oil removal is during 1.00 % concentration of oil with 60 seconds contact time where the 90.00 % of oil was removed. The lowest percentage of oil removal is during 5.00 % concentration of oil with 10 seconds contact time where only 72.00 % of oil was removed.

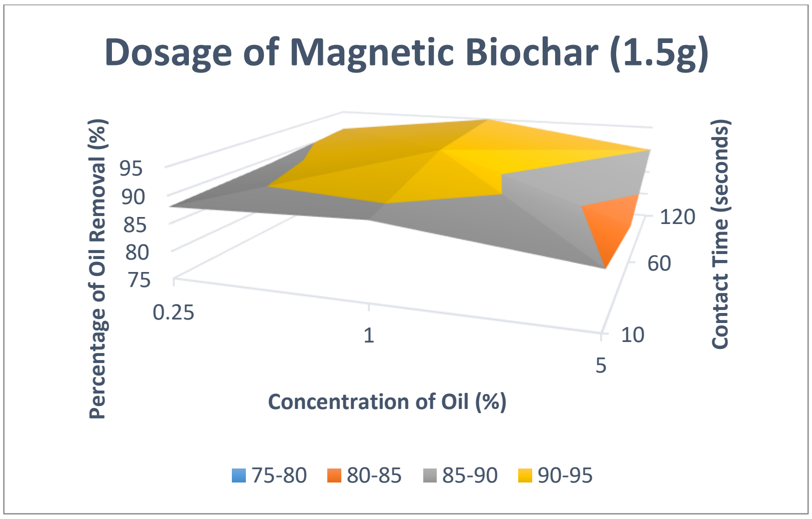

Figure 11: Percentage removal of 1.5 g magnetic biochar

Based on the graph in Figure 11, the highest percentage of oil removal is during 1.00 % concentration of oil with 120 seconds contact time where the 95.00 % of oil was removed. The lowest percentage of oil removal is during 5.00 % concentration of oil with 60 seconds contact time where only 82.00 % of oil was removed.

The performances of the magnetic biochar under several parameters which are dosage of magnetic biochar, contact time and concentration of oil are as shown in the 3-dimensional graph above. As dosage of the adsorbent increase, the efficiency removal also increases [12]. The dose of the adsorbent is an important factor that should be considered for effective removal [13].

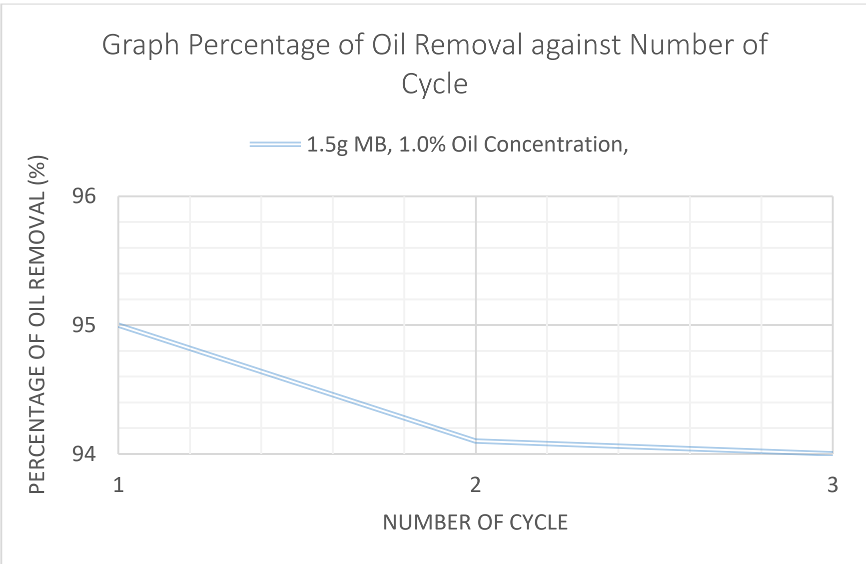

Figure 12: Graph percentage of oil removal against number of cycles

Based on the graph percentage of oil removal against number of cycles, the optimum condition for magnetic biochar to have highest efficiency in treating waste cooking oil is during 1.5 g dosage of magnetic biochar with 1.00 % oil concentration in the first cycle with 95.00 % of oil removal.

4. Conclusion

Waste cooking oil spills in Malaysia is one of the problems that need to be addressed as it brings out various adverse effects. Thus, the outcome of the experiment conducted in this study should be able to help in treating waste cooking oil. The percentage of oil removal increased with the increase the amount of magnetic biochar used and contact time taken. The optimum condition of magnetic biochar for high removal of oil is 120.0 g of magnetic biochar, 120 minutes and 1.00 % of oil concentration where the percentage removal of oil is 95.00 %. From the results achieved, it is proven that high dosage of magnetic biochar with high contact time taken will lead to high oil removal oil percentage. The magnetic biochar derived from oil palm frond have high potential in treating waste cooking oil water. This is due to the characteristics of the magnetic biochar which are large specific surface area, porous structure and surface functional group of magnetic biochar which had promote high removal efficiency of contaminants.

Acknowledgement

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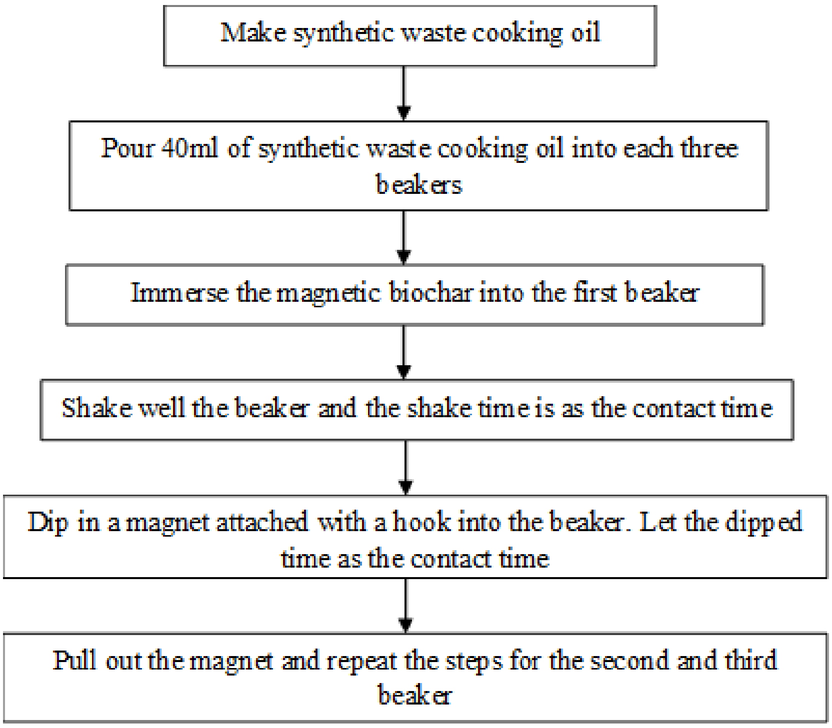
Appendix A

Experimental Procedure of Oil Removal



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