

Rice Husk Modification via Esterification for Palm Oil Mill Effluent (POME)

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Abstract—Palm Oil Mill Effluent (POME) is a colloidal suspension originating from a mixture of sterilizer condensate, separator sludge and hydro-cyclone wastewater which is thick brownish colloidal mixture of water, oil and fine suspended solids when it is fresh. POME produced from industry contains higher amount of Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD), Suspended Solid (SS) and oil and grease which over limit the standard set by the Government Agency which is Department of Environment (DOE). POME is categorized as a serious problem towards the environment as it can cause the reduction of oxygen in water thus affected the river and aquatic life when there are excessive amount of untreated POME. Due to this case, many methods have been conducted in treating the POME such as membrane technology, adsorption, and single stage ponding system. Adsorption is considered as an effective method because it is simple, applicable and low cost technique in treating the POME. Thus, the adsorption was selected by using the natural adsorbent which is rice husk for POME treatment. The rice husk was chosen because of its simple accessibility and most abundant biodegradable. The rice husk was modified via esterification process by an effective mass of stearic acid, $\text{CH}_3(\text{CH}_2)_{16}\text{COOH}$ in order to improved its hydrophobicity character. The effects of percentage of calcium oxide, CaO added and ratio of rice husk to stearic acid on the capacity of oil adsorption were investigated. The parameters that have been varied during the experiment were the percentage of calcium oxide added (5 % and 10 %) and ratio of rice husk to stearic acid (1:1, 1:4 and 1:7). Oil adsorption test was carried out in order to characterize the esterified rice husk in removal of oil from POME thus increased in oil adsorption capacity. The optimum operating condition of esterified rice husk was obtained at ratio of 1:7 rice husk to stearic acid and 5 % of calcium oxide added into the reaction as it successfully increased the oil adsorption for the removal of oil from POME at 0.1992 g/g. In addition, the high adsorption capacity of modified rice husk was better than unmodified rice husk as a result of increasing from 0.0824 g/g (40 %) to 0.1992 g/g (96.7%).

Keywords— *Palm Oil Mill Effluent (POME), Adsorption, Rice husk, Esterification, Adsorption capacity*

I. INTRODUCTION

Palm Oil Mill Effluent (POME) is a colloidal suspension originating from a mixture of sterilizer condensate, separator sludge and hydro-cyclone wastewater. POME is a thick brownish colloidal mixture of water, oil and fine suspended solids when it is fresh [16]. POME is one of the wastes that are very difficult to manage by the mill operators because there are large amount generated at a time. Based on the findings, there are about 30 million tons of POME was generated from 381 palm oil mills in

Malaysia. The amount of POME in 2005 was 53 million tons and is expected to keep increasing due to the production and processing of palm oil continuous to rise to meet both domestic and global demand [16]. The demand for palm oil is estimated to increase as much as 78 million tons in 2020 [33].

Nowadays, POME is categorized as one of the environmental concerns. This is because POME produced from industry contains high amount of Biochemical Oxygen Demand (BOD), Chemical Oxygen Demand (COD), Suspended Solid (SS) and oil and grease which over limit the standard set by the government agency that has responsible towards the Environmental Quality Act (EQA) 1974 which is Department of Environment (DOE) [25]. POME could cause health hazards if it is improperly treat [25, 32]. Excessive amount of untreated POME can cause some problems towards the environment such as reduction of oxygen in water thus affected the rivers [16, 32]. The rivers degraded was proven when people from Keningau and Murat who keeply reported the case of pollution due to the palm oil mills that are pollute the Ongom, Ambual and Puntir rivers. The people from Murat complained that oil and the dirt pollution was coming from a mill had crippled the water source for the residents in Pegalan river [16]. Based on the researcher study by Igwe (2007), POME that is discharged from industry contains soluble materials that are harmful to the environment such as aquatic life due to the higher concentration that is above the threshold value [13, 23].

Current methods in treating the POME include membrane technology, single stage ponding system, adsorption, and lagoon system. These methods are focusing on reducing the pollutants in POME. However, it has disadvantages as it is required high operation cost, need professional skills to operate and take a long duration as well as releasing high amount of biogas in the atmosphere about 60 % methane gas, 36 % calcium dioxide, CO_2 and other gasses like hydrogen sulphide which become one of the sources of greenhouse effects [26].

Among these methods, adsorption is considered as a simple, applicable and low cost technique for oil treatment compared with other techniques [1]. Adsorption is a highly efficient method which more popular among the researchers due to the high surface area, high adsorption capacity and microporous structure. Despite of these advantages, the adsorption process is quite expensive due to the most of the adsorbent was high costly. Therefore, many researchers tried harder to found the more suitable, efficient, cheap and easily accessible type of adsorbent and finally found on the natural fibres [9].

One of the natural organic materials which are lignocellulosic fibres from agricultural residue has been notifying as one of the important options for oil sorbents. There are two (2) types of adsorbents which are natural adsorbent and commercial adsorbent. Normally, natural adsorbent such as kapok, barley straw, rice husk, sawdust and grass has the ability to absorb more oil rather than commercial adsorbent. Besides, the natural adsorbents are comparable density with a synthetic sorbent, chemical free and highly biodegradable [33]. Therefore, the engineer starts to trust the potentially of the natural fibres [19]. However, in natural

adsorbents have low hydrophobicity and low buoyancy. Hence, it is good for the natural adsorbent to undergo the chemical modification to improve the hydrophobicity first before increase the oil adsorption capacity.

In order to improve the hydrophobicity and oil adsorption capacity of natural adsorbent, the chemical treatment such as esterification, alkalization and acetylation must be conducted. Esterification is one of the widely used chemical modifications to increase the oil adsorption capacity of natural fibres. In the esterification, sago bark is used as adsorbent using stearic acid. From the study, it has successfully showed the increment in hydrophobicity of sago bark and oil removal efficiency in POME by 42.2 % and 50.2 %, respectively. The treatment conducted in a ratio of 1:1 for sago bark to stearic acid, percentage of catalyst at 15 % and refluxing time at 8 hours showed the maximum of oil removal efficiency which almost 96 % in POME [33]. The esterification also can be conducted on sawdust using oleic acid, stearic acid and decanoic acid. The functional group (-OH) that present in sawdust was replaced by C=O and C-O group after the esterification completed. As the resulted, the modified sawdust is one of the potential adsorbent for oil spillage since the oil adsorption capacity shows the increment from 3.5 g/g to 6 g/g of crude oil [6].

In this study, the natural adsorbent used to treat the POME was by using the rice husk. The rice husk has been selected because the amount produced from industry is quite high which about 80 million tons. The information from Malaysian Ministry of Agriculture states that 408000 metric tons of rice husk are produced in Malaysia each year [27]. 100 kg of paddy rice husk able to produce about 20 kg of rice husk [12]. The rice husk is a cellulose-based fiber consists mainly of 35 % cellulose, 35 % hemi-cellulose [8] and approximately 20 % of silica in a combination of lignin [12]. The outer surface of the rice husk contains silica which exists in the form of the silicon-cellulose membrane where acting as a natural protective layer against termites and other micro-organisms attack on the paddy. Meanwhile, the inner surface is smooth and contains wax and natural fats that provide good shelter for the grain [20]. Rice husk able to overcome the problem related to the wastewater as well as the problem related to POME at a reasonable cost. Due to the capability of the rice husk, many researchers have been focused on the utilization of unmodified or modified rice husk as an adsorbent for the removal of pollutants. Rice husk shows as a good adsorbent for many metals and basic dyes industry. According to Subki (2012), rice husk able to absorb the heavy metals in the wastewater such as lead, copper and zinc as well as to treat textile wastewater [27].

The aim of this work was to enhance the oil adsorption ability through the modification of rice husk using esterification process. On top of that, the efficiency of modified rice husk was compared to the unmodified rice husk.

II. METHODOLOGY

A. Materials

Rice husk was collected and raw POME was collected from palm oil mill located at Felda Sungai Tinggi, Kuala Kubu Bharu, Selangor. The sample was cooled at 4°C before used for further process. POME was filtered using filter paper by vacuum pump (Model: DOA-P504-BN) in order to remove all solid particles contain in POME before conducting the oil adsorption test. The chemical used in this experiment for the esterification process was namely as ethyl acetate, stearic acid and calcium oxide, CaO. Meanwhile, for the oil adsorption test the chemical used was n-hexane and hydrochloric acid, HCl. All the experiment was done in Multipurpose Laboratory at Faculty of Chemical Engineering.

Esterification of rice husk

The raw rice husk (5g) was placed in 500mL of round bottom flask that contains 100mL of ethyl acetate. The esterification process was conducted at different ratio of rice husk to stearic acid (1:1, 4:1 and 7:1). CaO at 5 % and 10 wt% of rice husk mass was added to the mixture as a catalyst and the flask was immersed in oil bath for heated at refluxing time (2h). The mixture was then filtered, washed with ethyl acetate to remove the unreacted stearic acid. Lastly, the sample was cooled to room temperature for the further process [33].

Oil adsorption test in POME

The adsorption process was conducted using 5g of esterified rice husk with 50mL of raw POME at room temperature. The esterified rice husk and POME mixture was stirred at 30 min before being filter. Then, the filtrate was analyzed for the oil adsorption capacity.

The n-hexane solvent extraction method was used to determine the oil adsorption capacity in POME. The raw POME (50 mL) was transferred to separatory funnel and required about 9 drops of hydrochloric acid, HCl to water ratio (1:1) prior to addition of 3 mL of n-hexane. Then, the separatory funnel was vigorously shaken at 2 min and left another 10 min to form two (2) layers. The combined organic and oil layer was collected. The oil sample was dried at 103 °C for 15 min and cooled at room temperature before weighed the sample as an oil content value [33]. The oil adsorption capacity was calculated using Equation 1 while adsorption efficiency was calculated using Equation 2 [1].

$$\text{Oil adsorption capacity (g/g)} = \frac{\text{Mass of oil removed}}{\text{Mass of adsorbent}} \quad (1)$$

$$\text{Adsorption efficiency (\%)} = \frac{(\text{Mass of oil removed} / \text{Initial mass of oil}) \times 100}{\quad} \quad (2)$$

III. RESULTS AND DISCUSSION

During the reaction, the functional groups that presence on the surface of the rice husk was totally changes and the specific surface area has been increased. The schematic diagram of esterification of rice husk by using stearic acid and calcium oxide to formed the esterified rice husk that has been shown in Figure 1.

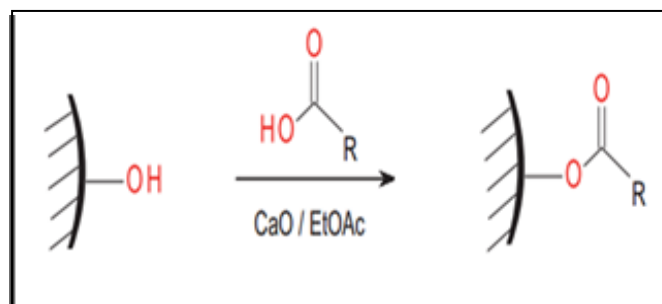


Fig. 1: Esterification of rice husk using stearic acid and calcium oxide to formed the esterified rice husk

In this reaction, calcium oxide was acted as a catalyst which helped to speed up the reaction and to deprotonate the abundance of hydroxyl group, OH that available in the rice husk. During the reaction, the cellulose content in the rice husk has increased due to the removal of lignin and hemicellulose solubilisation. Furthermore, the content of the cellulose crystallinity also reduced and therefore leads to an increasing of the surface area and the porosity [8]. Besides, the OH band intensity was slightly reduced during the reaction and thus improved the hydrophobicity and the

esterified rice husk become more thermally stable than unmodified rice husk [32].



Fig. 2: Layer of oil and POME

Figure 2 shows the layer of oil and POME that formed during the oil adsorption test.



Fig. 3: Sample of oil collected

Meanwhile, Figure 3 shows the oil that has been removed from POME where the left side showed the oil that has been collected before dried it in an oven whereas the right side showed the left POME.

A. The effects of percentage of CaO added into the reaction

After the rice husk was completely modified, the esterified rice husk was used for oil adsorption test of POME.

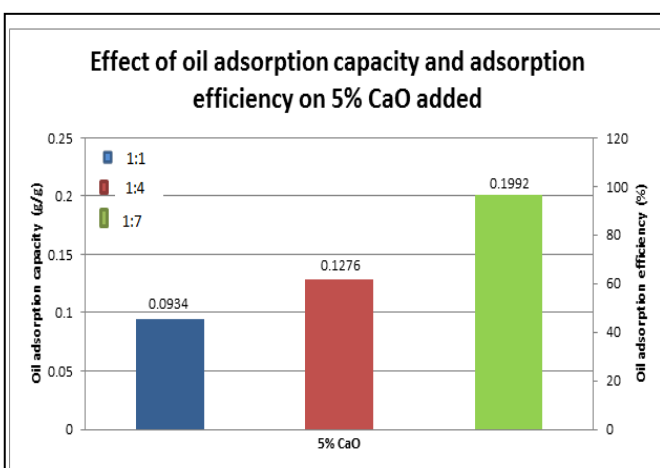


Fig. 4: Effect of oil adsorption capacity and adsorption efficiency on 5 % calcium oxide added

Figure 4 showed the oil adsorption capacity and its efficiency on the 5 % of calcium oxide added into the reaction by constant the mass of modified rice husk at 5 g. Ratio of 1:1 shows 5 g of stearic acid used in the test which give the result of oil adsorption capacity at 0.0934 g/g with the adsorption efficiency of 45.34 %. Then, it was continuous increased from 0.1276 g/g to 0.1992 g/g when the amount of stearic acid used higher from 20 g to 35 g, respectively. The oil adsorption efficiency also increased from 61.94 % to 96.7 %. Therefore, it can be concluded that the higher ratio of rice husk to stearic acid used, the higher the oil adsorption capacity and its efficiency.

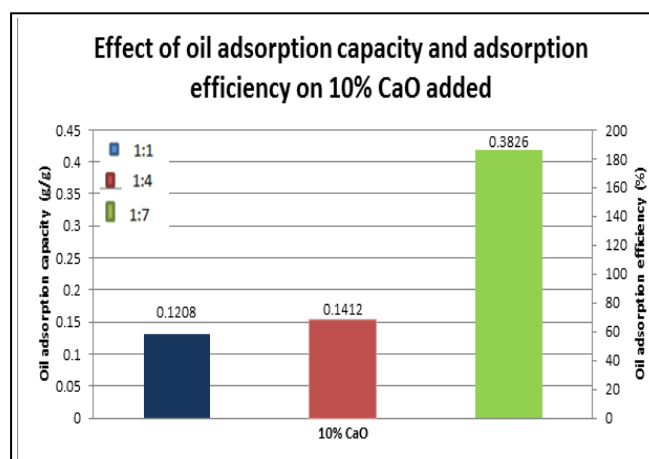


Fig. 5: Effect of oil adsorption capacity and adsorption efficiency on 10 % calcium added

Figure 5 shows the effect of oil adsorption capacity on 10 % of calcium oxide added into the reaction. From Figure 5, it is clearly shows the dramatically increased in oil adsorption capacity when it is reached the ratio of 1:7 which is 0.3826 g/g. For the ratio of 1:1 (5 g of stearic acid) and 1:4 (20 g of stearic acid), it is slightly increased from 0.1208 g/g to 0.1412 g/g. The adsorption efficiency was increased by 127.09 % from ratio 1:1 to 1:7. Therefore, the higher oil adsorption capacity was affected by the higher ratio of stearic acid used.

Figure 4 and 5 showed almost the same trend in the oil adsorption capacity result. The oil adsorption capacity increased when 10 % of calcium oxide was added into the reaction. However, in terms of efficiency the optimum condition was accomplished by 5 % of calcium oxide added with efficiency of 96.7 %. The increasing amount of calcium oxide helps the OH group that presence in the rice husk to transfer, hence gave rise to more reaction between stearic acid and rice husk [33].

B. The effects of different ratio of rice husk to stearic acid



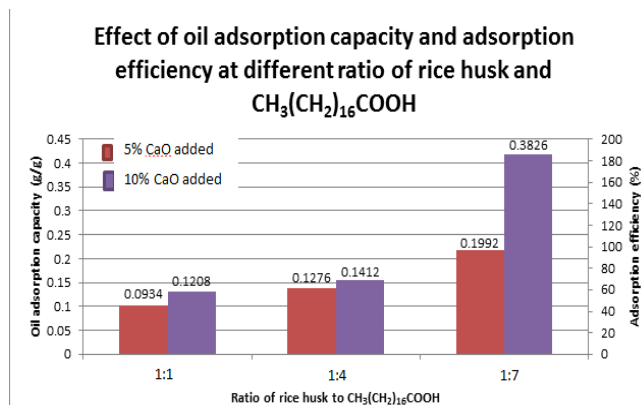


Fig. 6: Effect of oil adsorption capacity and adsorption efficiency at different ratio of rice husk to stearic acid

Figure 6 showed the effect of oil adsorption capacity and its efficiency at different ratio of rice husk to stearic acid. The result of oil adsorption capacity was steadily increased from 0.0934 g/g (45.34 %) to 0.1992 g/g (96.7 %) when calcium oxide added was constant at 5 %. For 10 % of calcium oxide added, the trend shows the dramatically increased in oil adsorption capacity whereby at ratio 1:1 shows 0.1208 g/g (58.64 %) whereas at 1:4 and 1:7 it shows 0.1412 g/g and 0.3826 g/g with 68.54 % and 185.73 % efficiency, respectively.

Hence, it can be concluded that ratio of 1:7 gave the best result on oil adsorption capacity (0.3826 g/g). This was because the higher ratio of rice husk to stearic acid used giving the rice husk more chances for esterification reaction to occurs and generated esterified rice husk with highest oil adsorption capacity.

Therefore, it can be concluded that the optimum condition to esterified the rice husk was obtained by using 5 % of calcium oxide with the ratio of 1:7 rice husk to stearic acid. This was accomplished by the adsorption capacity value of 0.1992 g/g with 96.7 % efficiency.

C. Effect of oil adsorption capacity between modified and unmodified rice husk

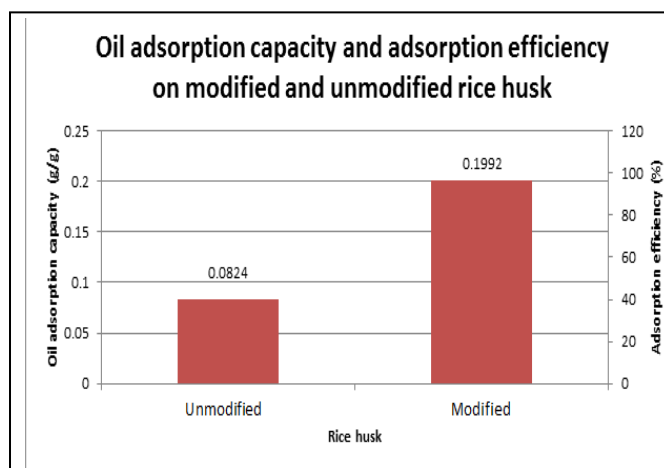


Fig. 7: Oil adsorption capacity between modified and unmodified rice husk

Figure 7 showed that the modified rice husk able to improve the amount oil adsorption capacity rather than the unmodified rice husk. Moreover, it has the potential to increase the oil adsorption efficiency. The oil adsorption capacity was compared between unmodified and modified rice husk. There was a sharp increased from 0.0824 g/g (40 %) for unmodified rice husk to 0.1992 g/g (96.7 %) for modified rice husk. Therefore, the trend was similar to

the previous study as in the Figure 7 the unmodified rice husk showed the smallest amount of oil adsorption capacity with the lowest adsorption efficiency (40 %).

Generally, the oil adsorption capacity was mainly depends on the functional group in the sorbent. In the unmodified rice husk, the high content of OH group [14] caused them to absorb more water [10]. Thus, it increased the surface polarity towards the hydrophilic effect which resulted in decreasing of oil adsorption capacity [32]. Consequently, it allowed the sorbent to catch many materials [14]. The increased in sorbent pore size decreased the oil adsorption capacity [32].

The esterification reaction studied by Banerjee et. al., (2006) showed that the result of modified sawdust was much better in oil adsorption capacity when there are increment by 2.5 g/g of crude oil. This was because the OH present in sawdust was replaced by the C=O and C-O group once the reaction completed [6]. In addition, the studied by Subki (2006) also stated that the modified rice husk exhibited higher adsorption capacities than unmodified rice husk [27].

IV. CONCLUSION

The modification of rice husk had caused the surface functional groups to change and the surface area of the adsorbent to increase. The surface functional groups have significant effect on the adsorption of stearic acid on the surface of adsorbent. In conclusion, a locally abundant waste material from rice husk has been successfully utilized for the removal of oil from POME. The esterification had successfully increased the oil adsorption capacity by 0.1168 g/g between the unmodified and modified rice husk. The optimum condition to esterified the rice husk was obtained by using 5 % of calcium oxide with the ratio of 1:7 of rice husk to stearic acid. This was accomplished by the adsorption capacity value of 0.1992 g/g with 96.7 % efficiency.

In order to obtain the better result in the future work, it is recommended to use the rotary evaporator in order to totally remove the n-hexane that has been added during the oil adsorption test. This is to ensure the n-hexane is not collected together with the oil removed from POME. In addition, it is better to use only one beaker to collect the oil sample so that the mass of beaker can be fixed during the experiment. The dried oil is weighed using the same beaker and the mass of oil removed can be taken after reduces the mass of beaker. This is to prevent the oil loss during transferring the oil to another beaker when weigh it to get the mass of oil removed from POME. Besides, the different size of adsorbent can be varied since the smaller size normally contains more available surface area as oil binding sites. Therefore, it gives the higher efficiency in oil adsorption capacity. The modification can be conducted by using different amount of rice husk. In addition, it can be tested by using other chemicals such as oleic acid or decanoic acid in order to improve the findings of this research.

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