Adsorption of Contaminants from Palm Oil Mill Effluent Using Agricultural Biomass Wastes as Adsorbents

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Abstract. Palm oil mill effluent (POME) has become critical issue among the oil palm industries due to its high organic contents and other contaminants which results in dark colour, turbid and bad smell. POME that is not treated effectively will result in significant wastewater issues as it disposes to the environment. Adsorption is a promising technique to purify the wastewater and has a wide variety of potential adsorbents. It works by adhering the pollutants on the high porous of activated carbon. Therefore, activated carbon has been widely used due to the effectiveness to adsorb pollutants and easy to produce. Previous studies proved that adsorption using activated carbon has been an effective method for the treatment of POME. This paper presents the utilization of activated carbon from agricultural biomass wastes for POME treatment. The materials are renewable, low-cost and viable as precursors of the activated carbon. The adsorption efficiencies and mechanisms of the activated carbon are discussed and summarized for easy reference. Development of methods for synthesis may become great challenges in the future, as well as the regeneration of activated carbon.

1. Introduction
Malaysian palm oil industry has developed gradually by years and impacted on the economic growth of the country prominently. The industry has exported large amount of crude palm oil (CPO) and it has been increasing currently due to the higher demands of higher population worldwide. In 2018, there were 451 palm oil mills producing 19.52 million tonnes of CPO followed by 64 million tonnes of palm oil mill effluent [1,2]. According to the data, the amount of POME being generated was higher than the main product, therefore, the effluent has become the major contribution of water pollution coming out from palm oil industry.

Palm oil mill effluent (POME) is wastewater generated by palm oil mills, mainly contains organic matters, oil, grease, and dirt. The organic matter parameters, such as Chemical Oxygen Demand (COD), Biological Oxygen Demand (BOD) and dirt parameter or Total Suspended Solids (TSS) are the highly concentrated compositions affecting to the brownish colour and bad smells of POME. The discharge of POME without appropriate treatment has resulted in high concentrations of contaminants and exceeded the standard of POME disposal into the environment. As a result, POME contaminants block the sunlight entering the water bodies, which lead to the reduction of photosynthesis activity. Moreover, the presence of organic matters accelerates bacteria growth and decrease the dissolved...
oxygen consumed by aquatic animals. Thereby, it is essential to treat POME before being disposed to the environment.

There have been many studies about POME using methods with different processes, such as biological, physical and chemical treatments [3]. The biological process like anaerobic treatment needs large area of land and takes a long time due to the mechanism of microorganisms to remove the pollutants. On the other side, the physical and chemical processes also have their limitations. Physical treatment using membrane system would not be suitable for effluent with organic composition. The organic matter will deposit on membrane surface and form fouling layer instead [4]. Hence, it reduces the efficiency of treatment and requires frequent maintenances.

Adsorption is a physical or chemical method in treating effluents. One popular method is adsorption using activated carbon (AC) which has great efficiency and simplicity of the process [5] due to the high adsorptive capacity of the pores [6]. However, the limitation of AC is the high cost of raw materials. Commercial ACs (CAC) are mainly made from lignin and coal which are non-renewable. Therefore, some researches were conducted by utilizing low-cost and renewable materials from agricultural biomass wastes. The capacity to adsorb contaminants in POME is comparable to those using CAC. In this study, the adsorption applications and mechanisms of AC derived from the agricultural biomass wastes in the treatment of POME are reviewed.

2. POME treatment using agricultural biomass wastes as adsorbents

2.1. Chemical oxygen demand removal

Chemical Oxygen Demand (COD) is one of the parameters for assessing quality the water or wastewater. COD analysis measures the total amount of all chemicals, like organic and inorganic matters present in water bodies. COD generally has higher values than BOD due to the higher amount of compounds that can be oxidized chemically rather than the compounds that can be degraded biologically [7]. The oxidation of matters is done by the strong oxidizing agents such as potassium dichromate (K₂Cr₂O₇) in polluted water bodies (wastewater) and potassium permanganate (KMnO₄) in clean water bodies [8,9]. The high amount of COD potentially harms the environment and the living creatures.

COD level in POME comes from the matters contained in oil palms and the processes to produce the final products of palm oil. Raw POME contains around 50,000 mg/L of COD [3] and 1730 mg/L of COD in bio-treated POME [10]. According to Malaysian Department of Environment (DOE) 1986, the discharge limit for COD is 1000 mg/L [11]. Therefore, it is necessary to proceed to the more effective treatment of POME since a lot of oil palm industries have used biological treatment like ponding system.

| Agricultural biomass wastes | Dosages (g/100mL) | Contact time | pH | S_BET (m²/g) | COD | References |
|-----------------------------|-------------------|--------------|----|--------------|-----|------------|
| Banana peel                 | 30                | 30 h         | 2  | 875          | 100%| [12]       |
| Empty fruit bunch           | 9                 | 24 h         | -  | 937          | 98% | [13]       |
| Oil palm kernel shell       | 15                | 120 h        | 8  | 566          | 94% | [14]       |
| Oil palm kernel shell       | 1                 | 36 h         | -  | 1320         | 89% | [15]       |
| Oil palm kernel shell       | 12.5              | 120 h        | 5  | -            | 83% | [16]       |
| Oil palm mesocarp fiber     | 1                 | 24 h         | 9  | 494          | 70% | [17]       |
| Coconut shell (CAC)         | 5                 | 48 h         | 8  | 744          | 70% | [6]        |
| Oil palm kernel shell       | 4                 | 24 h         | 9  | 987          | 68% | [18]       |
| Oil palm kernel shell       | 0.02              | 35.9 min     | 3.9| -            | 42% | [19]       |

Adsorption using activated carbon is such a promising method to treat POME. Table 1 shows some studies regarding POME adsorption by using activated carbon derived from agricultural biomass.
wastes. Mohammed and Chong (2014) conducted experiment on COD removal from POME using banana peel as adsorbent [12]. It was explained the lower pH obtained higher uptakes in COD, TSS, and colour removals due to the electrostatic attractions and hydrogen bonding between POME and banana peel AC. Besides, Wafti et al. (2017) evaluated the application of empty fruit bunch as precursor of AC in POME treatment [13]. It resulted that the adsorption capacities of AC were comparable to the CAC. The dosages used were 0.5, 1, 2, 3, 5, 6, 7, 8, and 9 w/v%. The adsorption capacities increased sharply with dosages up to 2 w/v% then increased slowly with larger dosages until the maximum reduction of COD occurred. The increased COD removal was influenced by the availability of sites and larger surface area in AC [20].

2.2. Biological oxygen demand removal

Biological Oxygen Demand (BOD) is an indicator of organic matter in water bodies. BOD level shows the amount of oxygen needed by microorganisms to degrade the organic matter. BOD analysis takes up to 5 days for measuring the biodegradable compounds compared to COD analysis which takes less time and sufficiently indicates both biodegradable and non-biodegradable matters [8,21]. BOD measurement works by calculating the difference of dissolved oxygen (DO) concentrations before and after incubation of samples in a dark space at a given temperature of 20°C [22].

Raw POME contains around 25,000 mg/L of BOD [3], which is considerably higher than the limit of discharge (50 mg/L) set by Malaysian DOE 1986 [11]. It contains the complex form of organic acids which produces the low pH after being out of mill processes [23]. The high amount of organic matter in POME consumed a lot of DO which are required for the biodegradation. Thus, there will be adverse effects on aquatic life as it depends on the adequacy and availability of DO. The removal of organic pollutants has been applied using effective method like adsorption [24].

Table 2. Analysis of BOD removal from POME

| Agricultural biomass | Dosages (g/100mL) | Contact time | pH | S_BET (m²/g) | BOD | References |
|---------------------|------------------|--------------|----|--------------|-----|------------|
| Banana peel         | 30               | 30 h         | 2  | 875          | 97% | [12]       |
| Empty fruit bunch   | 9                | 24 h         | -  | 937          | 88% | [13]       |
| Oil palm kernel shell | 1               | 36 h         | -  | 1320         | 85% | [15]       |
| Oil palm kernel shell | 4               | 24 h         | 9  | 987          | 83% | [18]       |
| Oil palm kernel shell | 0.02           | 35.9 min     | 3.9| -            | 63% | [19]       |

Table 2 shows the studies of BOD removal in POME treatment. Zainal et al. (2018) investigated the BOD removal from POME [18]. It was observed that the maximum removal of BOD was achieved within the first 24 h. After 30 h, the adsorption equilibrium was reached due to the saturation of the AC sites. According to the table, other studies also used the same precursor as the aforementioned study. Azmi and Yunos (2014) conducted study of POME treatment using adsorption as pre-treatment stage and ultrafiltration membrane as treatment stage [19]. In the adsorption stage, the dosages varied from 0.2 g/L to 0.6 g/L with operation time from 20 min to 50 min. The optimum removals were obtained using regression equations, with values of 63% BOD removal, 0.2 g/L (0.02 g/100mL) and 35.9 min of operation time. The results present that the rapid removal of BOD in shorter time can be achieved by using agricultural biomass wastes as precursors of AC in POME treatment.

2.3. Total suspended solids removal

Total suspended solids (TSS) is sedimentation of particles which contribute to the turbid visual and low water quality in water bodies. TSS analysis measures the weight of suspended particles larger than 2µm using glass fibre filter [22]. The suspended solid influences colour in wastewater, including dark visual of POME. Raw POME contains 40,500 mg/L of TSS, resulting in adverse effects to the aquatic ecosystems. TSS affects the environment by blocking the sunlight and reducing photosynthesis [25].
TSS increases the water temperature, depletes the DO in water, carries heavy metals and organic matters which are harmful to the living creatures.

Table 3. Analysis of TSS removal from POME

| Agricultural biomass wastes | Dosages (g/100mL) | Contact time | pH | \(S_{\text{BET}}\) (m²/g) | TSS | References |
|-----------------------------|-------------------|--------------|----|--------------------------|-----|------------|
| Banana peel                 | 30                | 30 h         | 2  | 875                      | 100%| [12]       |
| Empty fruit bunch           | 9                 | 24 h         | -  | 937                      | 96% | [13]       |
| Oil palm kernel shell       | 12.5              | 120 h        | 5  | -                        | 92% | [16]       |
| Oil palm kernel shell       | 4                 | 24 h         | 9  | 987                      | 90% | [18]       |
| Oil palm mesocarp fibre     | 1                 | 24 h         | 9  | 494                      | 88% | [17]       |
| Oil palm kernel shell       | 0.02              | 35.9 min     | 3.9| -                        | 71% | [19]       |
| Coconut shell (CAC)         | 5                 | 48 h         | 8  | 744                      | 70% | [6]        |
| Oil palm kernel shell       | 1                 | 36 h         | -  | 1320                     | 57% | [15]       |

Table 3 represents the studies of TSS removal using agricultural biomass wastes as source of AC. According to the table, the surface area obtained by Liew et al. (2019) was much greater than other studies but found much lower in TSS percentage removal [15]. The study used Al or Ni metals in impregnating AC (metallic AC), which resulted in pore volume decreasing and surface area blocking towards the large suspended particles. Another study of AC derived from oil palm mesocarp fibre was investigated by Ibrahim et al. (2017) in treating POME [17]. The surface area obtained was the smallest one compared to other studies on the table. However, the TSS removal was quite high, reaching 88% with relatively low dosages of 10 g/L in 50 mL POME. This due to the 6 consecutive treatments used, in which the decrease of contaminants in POME occurred per repeated treatment. Thus, this method requires lower adsorbent dosages and has more contribution in zero-emission system for AC regeneration compared to the common single treatment method used in adsorption.

2.4. Colour removal

Colour is the indicator of water quality which is easy to observe. The dark colour of wastewater indicates the high level of BOD, COD, and TSS that need to be reduced. Colour of POME comes from the sterilization process of fresh fruit bunches. The brownish colour and unpleasant odour are the results of the complex organic compositions in POME. As the fresh fruit bunches are sterilized, lignocellulosic components are degraded; the organic matters such as anthocyanin, lignin, tannin, carotene pigment, polyphenol compounds, polyalcohol, and melanoidin are then formed and result in the oily, viscous texture and the brownish colour of POME [12,26,27]. There is no standard limitation for POME colour according to DOE of Malaysia. However, the turbid and dark colour of wastewater increases the public concerns towards the low water quality, in which supporting the researchers to keep conducting studies regarding colour removal of wastewater.

Table 4. Analysis of colour removal from POME

| Agricultural biomass wastes | Dosages (g/100mL) | Contact time | pH | \(S_{\text{BET}}\) (m²/g) | Colour | References |
|-----------------------------|-------------------|--------------|----|--------------------------|--------|------------|
| Oil palm kernel shell       | 5                 | 12 h         | 2  | 1252                     | 100%   | [27]       |
| Oil palm kernel shell       | 15                | 120 h        | 8  | 566                      | 100%   | [14]       |
| Oil palm kernel shell       | 4                 | 24 h         | 9  | 987                      | 97%    | [18]       |
| Banana peel                 | 30                | 30 h         | 2  | 875                      | 96%    | [12]       |
| Coconut shell (CAC)         | 5                 | 48 h         | 8  | 744                      | 70%    | [6]        |
Previous studies of colour removals in treating POME were represented in Table 4. Jalani et al. (2016) investigated colour removal in POME treatment [14]. The maximum colour removal (100%) was obtained during 120 h of contact time, while the optimum colour removal was 92% and achieved at 72 h. The essential parameter that has large effects on removal of the colour was pH. Mohammed (2013) reported that the alteration to lower pH has direct influence on the protonation of adsorbent surface which attracts the anionic functional groups, such as hydroxyl (OH\(^-\)) in POME [27]. The electrostatic attraction was also supported by Mohammed and Chong (2014), as the pH used in both studies produced the best removals at 2 [12].

3. Adsorption mechanism

Adsorption takes place due to the mass transfer of organic and inorganic molecules from liquid bulk to active sites within adsorbent. The mechanisms involve two forces, such as physical adsorption and chemisorption [28]. The physical forces like van der Walls forces and chemical bonds influence the steps of mechanisms. Basically, the steps consist of external diffusion, internal diffusion, and adsorption on active sites [29]. First, the external diffusion happens when the concentrations between liquid bulk and adsorbent surface are different, causing mass transfer of molecules on the boundary layer which surrounds the adsorbent particles. Some of the stick molecules on the adsorbent surface become adsorbed into the pores, while some others rebound back to the liquid bulk. This process takes place at equilibrium, where concentrations of the latter are equal to each other. The equilibrium relations are provided by using isotherm, as it is important for data prediction and interpretation [30–32]. Second, the internal diffusion takes place when the adsorbed molecules pass through the pores. The transport mechanism can be investigated by using internal diffusion models, such as Weber-Morris Intra Particle Diffusion (IPD) and Boyd’s model. Third, when the molecules reach the active sites within adsorbent particles, the process is very fast; thus, the investigation becomes complicated. The binding process of molecules involves physical adsorption and chemisorption. The active functional groups are essential for the chemisorption as chemical bonding agents towards the molecules [12]. Thereby, the adsorption mechanisms depend on both adsorbate and adsorbent properties [33].

The rate of adsorption in the aforementioned steps can be determined by using common kinetic models (Pseudo-First-Order, Pseudo-Second-Order, Elovich models) and mass transfer models (Weber-Morris IPD, Boyd’s model). The mechanism studies of adsorption kinetic and isotherm in POME treatment has been summarized in Table 5. Pseudo-First-Order (PFO), Pseudo-Second-Order (PSO), and Elovich models are used for determining rate constant steps, while IPD model is used for determining rate controlling step [34]. However, PFO and PSO are empirical models and have inadequate specific physical meanings [29]. Therefore, PFO and PSO models are not suitable to investigate the diffusion mechanism, which can be applied by IPD model [35,36].
Table 5. Mechanism supported-models in POME adsorption studies

| Parameters          | Kinetic models          | Best-fitted models | Isotherm models          | Best-fitted models | References |
|---------------------|-------------------------|--------------------|--------------------------|--------------------|------------|
| COD, NH₃-N          | PFO         | PSO                | Weber-Morris IPD         | PSO                | Langmuir   | [31]       |
|                     |             |                    |                          |                    | Freundlich  |            |
|                     |             |                    | PSO                      |                    | Langmuir    | [31]       |
|                     |             |                    | Freundlich               |                    |            |            |
|                     |             |                    | Temkin                   |                    | DR          | [10]       |
|                     |             |                    | Dubinin-Radushkevich (DR)|                    |            |            |
| COD                 | PFO         | PSO                | Elovich                  | PSO                | Langmuir   | [36]       |
| Manganese H₂S       |             |                    | Weber-Morris IPD         |                    | Freundlich (without magnetic field) |            |
|                     |             |                    | Elovich                  | PSO                | Freundlich (with magnetic field) |            |
|                     |             |                    | PSO                      |                    |            | [36]       |
| COD, Colour, TSS    | PFO         | PSO                | Weber-Morris IPD         | PSO                | Langmuir   | [12]       |
|                     |             |                    | Elovich                  |                    | Freundlich |            |
|                     |             |                    | PSO                      |                    | Redlich-Peterson |            |
|                     |             |                    | Second order kinetic     |                    | Sips       |            |
|                     |             |                    | PSO                      |                    |            |            |

4. Conclusions and future challenges
Agricultural biomass wastes have successfully been modified into highly porous AC. The performance of adsorption removals was greatly satisfied, specifically measuring parameters in COD, BOD, TSS, and colour in the treatment of POME. It is therefore the future studies of this area will keep challenging, as precursors are renewable and environmentally-friendly. It is also envisaged that the synthesis method of AC will be developed deeply in various parameters, such as carbonisation and activation. However, the regeneration of AC has become main topic in this research area since there have been many industries prefer to dispose of AC due to the higher cost of recycling. Thereby, it is highly recommended to find efficient and low-cost regeneration of AC for better industrial economics and green environmental sustainability.

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