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Modification of Agro-wastes Reinforced onto the PET Fabric for Decolorisation of Palm Oil Mill Effluent

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ABSTRACT

The abundance of agricultural wastes from various industrialized processes has become one of the significant contributors to water pollution, particularly in the color of effluents from industrial-based palm oil mills. Inventively, this research focused on three different agro-wastes: pineapple leaves (PL), rice straw (RS), and empty fruit bunch (EFB) reinforced onto PET fabric composite and its decolorization performances by using palm oil mill final effluent discharged (POME-FED). The calcinated agro wastes/polyvinylidene fluoride (PVDF) reinforced onto the polyethylene terephthalate (PET) fabrics were prepared by using the dip-coating technique and characterized via Scanning Electron Microscopy (SEM-EDS), spectroscopy (FTIR-ATR), turbidity and color of POME-FED. It was found that the calcinated PL/PVDF/Fabric displayed the best performance in the turbidity and decolorization by 12.02 NTU, 760 ADMI, and ~60% color removal efficiency as compared with raw POME-FED (~1800 ADMI). Nevertheless, the decolorization efficiencies of RS/PVDF/Fabric and EFB/PVDF/Fabric had increased by ~37 % and ~49 %, respectively. It shows that the formation of a reinforcing layer on the PET fabric surface has improved the transparency of POME-FED. The SEM micrographs and the change of peaks at regions 1650 cm^{-1}, 1450 cm^{-1}, 1210 $cm^{-1,}$ and 990 cm^{-1} in composites' spectroscopies demonstrate the different patterns of these calcinated samples are various patterns that impart the strength of the composite fabric surface functionality and hydrophobicity. The reduction of the color value of effluent showed the hydrophobicity of the integrated palm oil waste coated with PET, which enables to trap of the particles in the effluent, thus this composite has potential use in the filtration of water treatment.

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1. Introduction

Agricultural wastes are waste-based biomass derived from plants, including leaves and stems, also containing minerals [1]. Disposal of agro-waste directly to landfills leads to excessive environmental pollution, thus becoming a critical issue that must be tackled. Most researchers have shifted interest toward utilizing these agro-wastes due to their advantages such as sustainable availability, non-toxicity, lightweight nature, low density, cost-effectiveness and eco-friendliness. Many studies have also applied these kinds of agro-waste in the development or improvement of properties of materials such as supercapacitor separators, reinforcers in concrete, packaging, composites, and catalysts [2-9]. Nevertheless, these agro-waste are effectively applied for environmental mitigation. For instance, empty fruit bunch (EFB) and rice straw (RS) ashes act as activated carbon that eliminates the dyes [3,10,11], and pineapple leaves (PL) are applied for the removal of heavy metals and inorganic pigment [12]. With this regard, the modification and reinforcement with substrate materials have a good potential application in wastewater treatment.

Malaysia is considered one of the main producers of palm oil-based products, occupying about 77% of rural agricultural land. Consequently, the by-products from the palm oil industry have led to the generation of a significant amount of colored water discharged, known as the final discharge of palm oil mill effluent (POME)[13]. As reported in previous literature, more than 45 million metric tonnes per year (mt/y) of POME were released to the environment. While POME itself is non-toxic, it contains organic contaminants that compromise natural or synthetic organic compounds. Additionally, it releases an unpleasant odor under acidic conditions and exhibits a high concentration of carbon-oxygen demand (COD) ranging from 15,000–100,000 mg/L, biological oxygen demands (BOD) of about 10,000–25,000 mg/L, and color appearance between 3500 to 9900 ADMI [14-17]. Conventionally, biological treatment via ponding with aerobic and anaerobic treatments is used to treat POME [18-21]. However, the final discharge of POME still suffers from the presence of pigment contaminants, with an insignificant reduction of the brownish color of treated POME[22,23]. The brownish color of POME could be associated with the existence of lignin, tannin, and humic acids from the extraction process that could threaten aquatic life. Consequently, the consistent brownish color of the POME final discharged (POME-FED) has caused public concern about its impact on the water body.

Various approaches have been used in research to enhance the color quality of POME-FED. Physical methods include coagulation/flocculation, adsorption, and membrane filtrations. Although many types of filtration of the substrate are widely used, such as cotton [24], metal [25], fabric [26,27], and polymer membranes [28], the choice of the substrate in the separation process is a substantial challenge with some drawbacks due to overall separation efficiency, stability, and fouling effect. Hence, adding reinforcer onto porous materials-based polymer substrates could improve the properties such as substrate strength, separation efficiency, and color removal yield [29-35], and the dip-coating process is one of them [36,37]. Dip-coating is a simple and affordable method for depositing compounds onto any substrate, including ceramic [4], polymer films [5], and fiber materials [38]. The process could also be interpreted as an aqueous-based liquid phase coating solution deposited onto any substrate's surface [39].

PET fabric has become favored due to its porous structures, intrinsic hydrophobicity, rough surface, and high flexibility. However, conventional substrates still have some drawbacks, such as a foul tendency, easily rupture after processes and some materials not being produced due to their complicated process preparation. Thus, various efforts have been explored to improve the surface intrinsic properties of substrates. For instance, superhydrophobic filtration fabric integrated with fly ash or hydrogel composite has effectively separated oil/water mixture [40,41]. Huang *et al.,* have successfully investigated the hydrophobic fabric modified with hexadecyltrimethoxysilane (HDTMS), polymethylhydrosiloxane (PMHS) and hydroxyl-terminated polydimethylsiloxane (HTPDMS). It was found that the high efficiency in separate oil and water mixture. These studies have paved the way for developing new reinforced fabric for the decolorization of POME-FED.

Therefore, this study aims to prepare and characterize the unmodified and modified agro-wastes reinforced onto fabrics to decolorize the POME based on the different types of agro-wastes at a constant calcination temperature. The research was characterized in terms of the chemical structure and morphology of calcined agro-wastes. Furthermore, the performance of removal of color pigment by using modified agro-wastes onto PET fabric has not been studied yet. Hence, this study addresses the research gap by providing the separation efficiency of unmodified and modified reinforced fabrics by using palm oil mill final effluent discharged by decolorization and turbidity of POME-FED**.**

2. Methodology

2.1 Materials

All raw materials of agro-wastes; pineapple leaves (PL) and rice straw (RS) were obtained from local farmers from the community of Pagoh, Muar (Malaysia). Meanwhile, the empty fruit bunches (EFB) and the POME were supplied by KKS Pagoh (Sime Darby) Sdn Bhd. Polyethylene Terephthalate (PET) fabrics were purchased from Capital Resources Engineering Sdn Bhd (Malaysia). Chemicals such as natrium hydroxide (NaOH), and ethanol (analytical grade) were purchased from R&M Chemicals. Polyvinylidene fluoride (PVDF) and dimethylformamide (DMF) were supplied by Alfa Aesar, and Merck, respectively.

2.2 Methods

2.2.1 Preparation of composite agro-waste reinforced flat sheet fabric

Initially, three types of agro-waste (PL, RS, and EFB) were dried at 60 ℃ for 24 hours and then, calcinated at 500 ℃/min for 3 hours in a muffle furnace, Metrhom brand. After that, the calcinated agro-waste was crushed into powder form. In the secondary stage, the PET fabric samples were immersed in NaOH solution, 1N at 60°C for 2 hours. After that, the fabric sample was repeatedly washed with distilled water and soaked in ethanol for 10 minutes. The treated flat sheet fabric was dried at 60℃ for 24 hours and kept in the desiccator until further use.

To prepare the agro-waste/PVDF/Fabric, 0.3 g of PVDF was dissolved in 10 ml of DMF and stirred for 30 minutes. After the PVDF powder was dissolved entirely in the DMF solution, 0.5 g of agro-waste was slowly added into the PVDF/DMF solution and stirred to facilitate the reaction to reach a homogenous mixture of agro-waste/PVDF. Subsequently, the agro-waste/PVDF solution was poured into the glass petri dish and then PET fabric was immersed in the mixture solution. Finally, the composite agro-waste/PVDF/Fabric was dried at 60 ℃ for 7 hours and kept in the desiccator until further use.

2.2.2 Characterisation of calcinated agro-wastes and composite agro-waste/PVDF/fabric

The samples were analyzed in terms of their surface chemistry and morphology, which were conducted through Scanning Electron Microscope - Energy Dispersive Spectroscopy (SEM-EDS), operated at 20kV and high magnification and Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR) ranging from 700-4000 cm^{-1}. Meanwhile, the performance of POME-FED was evaluated based on the turbidity and decolorization effects, which are measured through the DR6000 spectrophotometer and Turbidity tester (TL2300), respectively. The magnification used in SEM-EDS was 2000 times, while the decolorization was referred to the standard program, 10mm of American Dye Manufacturers Institute (ADMI).

3. Results and Discussion

3.1 Functional Groups Determination of Calcinated Agro-Wastes and Agro-Wastes/PVDF/Fabrics

In this study, calcinated agro wastes were first used to identify the change in functional groups for unmodified and modified agro-wastes/PVDF/fabric. Figure 1(a-b) illustrates the ATR-FTIR spectra of calcinated agro-wastes (PL, RS, EFB) and the modified agro-wastes/PVDF/fabric at a temperature of 500 °C. Figure 1(a) shows a similar trend for all the agro-wastes, with a broad absorption peak showing a medium intensity at 3380-3800 $cm^{-1,}$ representing a hydroxyl (-OH) band. The band at 2125 cm^{-1} is attributed to C-H stretching, indicating the aliphatic nature of agro-waste. The existence of weak absorptions at 1950-2050 cm^{-1} and 1459-1560 cm^{-1} reflect the presence of C=C, C-C bonds in the aromatic ring and nitrogenous group (N-H group) due to the effect of the calcination process [8,33,42] The broad shoulder peak at 1650 cm^{-1} implies C=O stretching vibration and aromatic carbon (C-C). Additionally, 1410 cm^{-1} and 1200 cm^{-1} peaks were ascribed to carbonyl group (C-O). It is seen that overlapped broad absorption at regions 3300 cm^{-1} and strong absorption peaks of RS, compared PL and EFB at 1068 cm^{-1} and 899 cm^{-1}, corresponding to the silicone ions: silanol (Si-OH) and siloxane (Si-O-Si) bonds. The high difference peak at region 800-1200 cm^{-1} for RS possibly contains a high amount of Si-O, representing the silica compounds [32,43,44].

By contrast, Figure 1(b) shows a significant increase in intensity and broadened peaks at region 3300-3600 cm^{-1}. Moreover, the stretching vibration peaks at 1650 cm^{-1}, 1450 cm^{-1}, 1330 cm^{-1}, 1210 cm^{-1} and 990 cm^{-1} and 760 cm^{-1} are enhanced intensively which corresponds to the C-O, $-CF_2$ and CH_2, respectively [22,23]. It indicates that agro-wastes are reinforced significantly onto PVDF/PET fabric. After Modification, PL/PVDF/Fabric exhibits the highest intensity followed by RS/PVDF/Fabric and EFB/PVDF/Fabric.

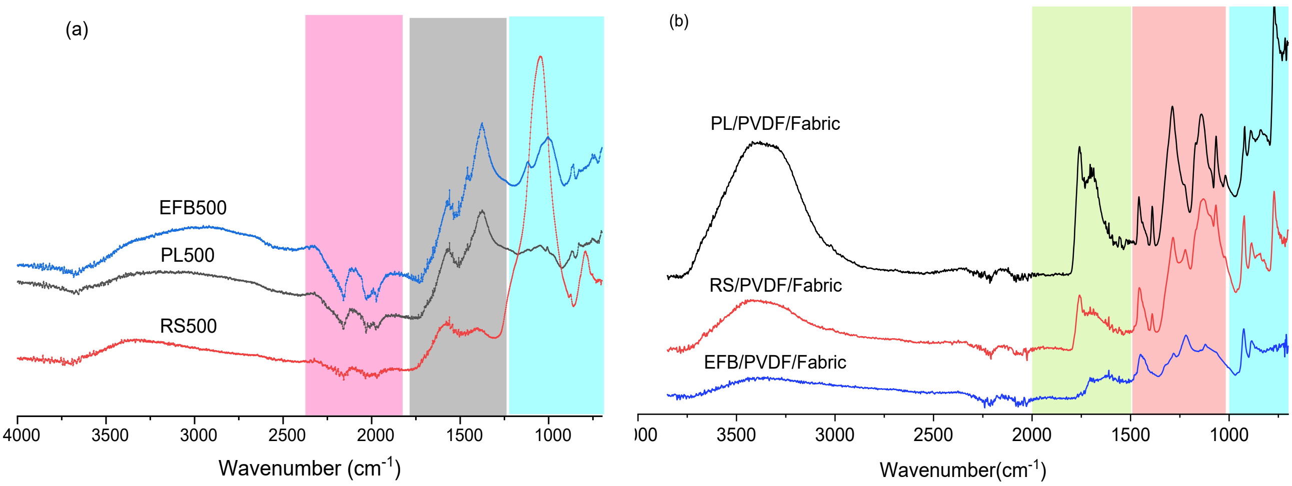

Fig.1. Fourier-Transformed Infrared (FTIR) of (a) calcinated agro-wastes (b) agro wastes/PVDF/Fabrics

3.2 Morphological Structure of Calcinated Agro-wastes and Agro-wastes/PVDF/Fabrics

Based on Figure 2, SEM was used to examine the surface morphology of PL, RS and EFB. There was a different surface morphology between them after undergoing the calcination at 500°C. It can be seen the distinct appearance of the particles can be found on the calcinated agro-waste ashes.

Figure 2a shows the surface of the PL where a small cluster shape was grouped, suggesting that the rosettes microparticles are bound preferentially by cellulose derivates because the based structure was maintained, and the particles stayed interconnected. Contradictory, Srikhaow *et al.* conducted the pyrolysis process on the pineapple leaves at 550 °C for 2 hours. They found that the morphology of pineapple leaf ash exhibited a tube-like structure containing pores. This is probably because different temperatures and calcination times of agro-waste could cause different patterns of agro-waste morphology.

Meanwhile, the RS surface morphology (Figure 2b) showed numerous small fragments that were aligned with the presence of pores that illustrated the surface of RS particles, suggesting that the rosettes microparticles are bound preferentially between themselves by cellulose derivates because the based structure was maintained, and the particles stayed interconnected[43].

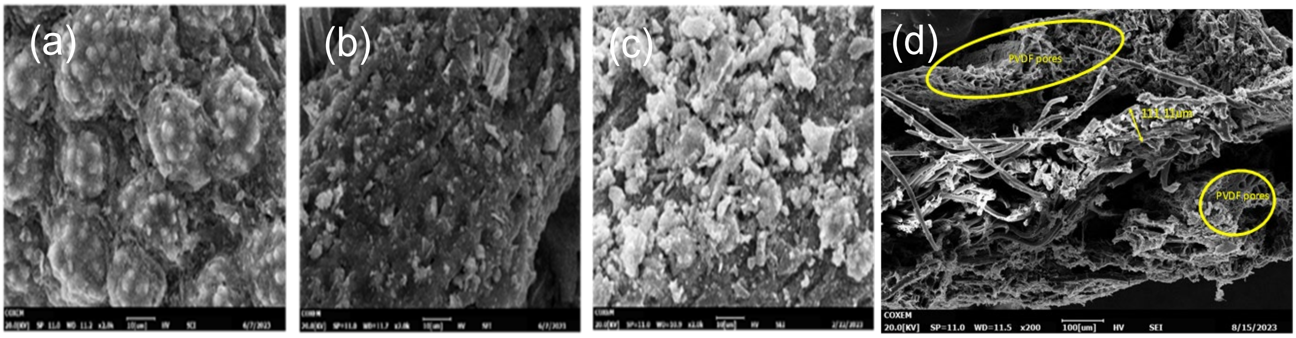

Fig. 2. Scanning Electron Microscopy (SEM) of (a) PL (b) RS (c) EFB at magnification 2000x (d) Example of modified agro-waste composite fabric (cross-sectional area) at magnification 200x

Besides, Figure 2c demonstrates there are many big fragments scattered around the EFB surface, in agreement with previous studies on irregular geometries such as cylindrical, flaky, angular porous, and spongy structures grouped into spherical clusters. However, there were low visible pores that could be seen on the surface of each type of agro-waste, which may be covered by the fragments. Nevertheless, a significant interaction between agro-waste with PVDF/Fabric can be found in Figure 2d. Figure 2d illustrates the layer formation and reinforcement of agro-waste after modification with PVDF/Fabric.

3.3 Physical Appearances of Unmodified and Modified Samples

The appearance of the samples before and after the modification can be seen in Figure 3. Figure 3(a) shows that the sample before modification is slightly hydrophobic with a whitish appearance. After the pretreatment and dip-coating process, it can be seen the rough black surface of the modified sample. (Figure 3(b)). This appearance indicated that the calcinated agro-waste was successfully reinforced onto the PVDF/fabric.

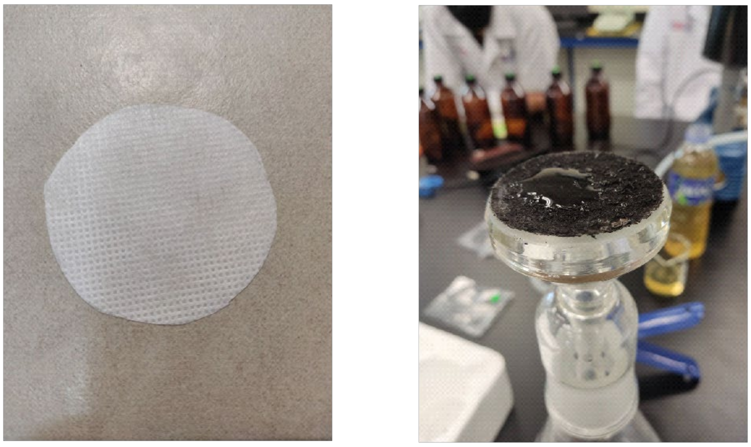

Fig. 3. Physical Appearances of the Sample (a) Before Modification (b) After Modification

3.4 The Performance of Agro-Wastes/ PVDF/Fabric: Turbidity and Decolorisation Efficiency

The appearance of POME-FED water was observed based on the transparency of water by using a turbidity test. Table 1 shows the PL/PVDF/Fabric exhibited the lowest value of turbidity, 12.02 NTU as compared to the RS/PVDF/Fabric and EFB/PVDF/Fabric. This result suggests that each type of agro-waste possessed a different amount of hydroxyl groups, resulting in the sample's hydrophilicity (Figure 1 (b)). In addition, Figure 4 demonstrates the reduction of color for POME-FED as compared with the original fabric filter. It can be seen that there is no change in color for the original fabric. The decolorisation results after filtration by using modified composite fabrics show there is a significant reduction in decolorisation of POME-FED with the value of color below 1000 ADMI as compared to initial POME-FED, 1800 ADMI.

Table 1
Turbidity of final POME-FED after filtration using different type of modified agro-wastes

Type of agro-wastes	Turbidity Value (NTU)	Standard deviation
PL/PVDF/Fabric	12.02	0.067
RS/PVDF/Fabric	15.00	0.055
EFB/PVDF/Fabric	14.68	0.091

The difference in efficiency for the modified sample can be seen in Figure 4, whereas the sample PL/PVDF/Fabric shows the lowest value compared to agro-waste based RS/PVDF/Fabric and EFB/PVDF/Fabric, with the reduction of color POME-FED is 760 ADMI and its color removal efficiency reached ~60 %. Both RS/PVDF/Fabric and EFB/PVDF/Fabric achieved the color reduction to 998 ADMI and 890 ADMI, with color removal efficiencies are ~37 % and ~49 %, respectively. The standard deviation that has been calculated was based on the average value, which is 0.707. The color of POME-FED could be due to the presence of organic compounds such as lignin and tannins which are hydrophobic in nature. These results reveal that a significant reinforcement PL onto PVDF/fabric has enhanced the hydrophilicity surface of a fabric, turning to surface attraction toward water molecules, thus leading to the susceptibility toward organic fouling such as lignin and tannins [22,23].

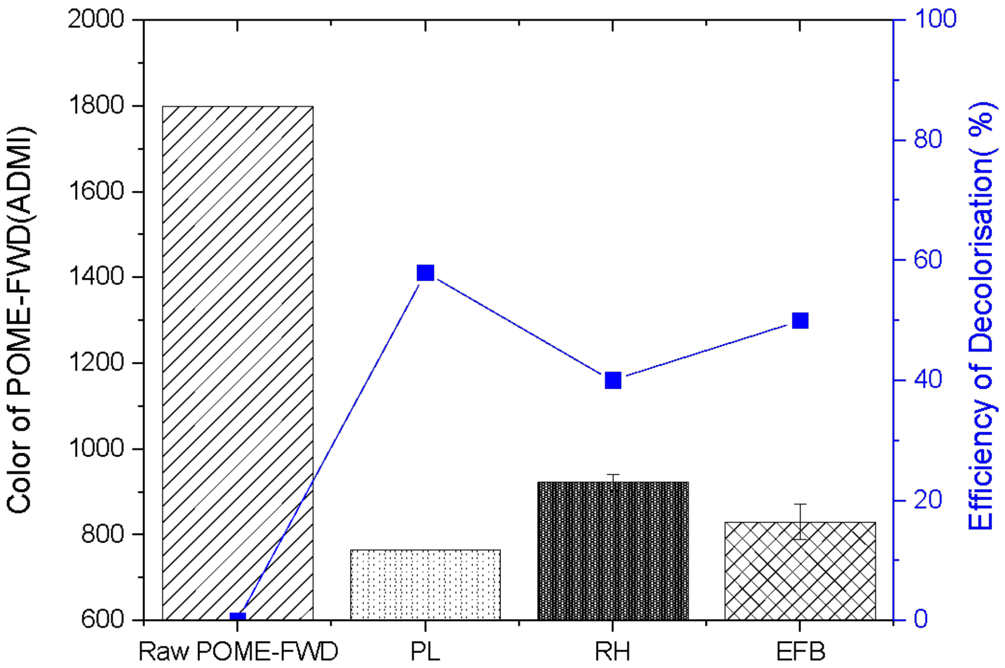

Fig. 4. Decolorising POME-FED using unmodified and modified agro-wastes reinforced onto PVDF/ fabrics (PL/PVDF/Fabric, RS/PVDF/Fabric, EFB/PVDF/Fabric)

4. Conclusions

The calcinated agro-wastes: PL, RS and EFB and modified agro-waste reinforced onto PVDF/fabrics presented a different intensity of peaks at certain regions and functional groups. The high silica contents appeared in the calcinated RS. Additionally, the agro-wastes successfully reinforced onto the PVDF/Fabrics with good interaction can also be seen via morphology results. Similar results in terms of the morphology of these agro-waste ashes, various patterns with multi-fragments on the surface of the agro-waste ashes after calcination, which may close the pores of the agro-waste ashes, thus these reinforcements of agro-wastes has improved the surface interaction and hydrophilicity of modified composites fabrics. The observation clearly shows that reinforcement of calcinated PL onto PVDF/Fabric exhibits the best performances, with a display turbidity value of 12.02 NTU and color removal efficiency of ~60%. This presented that the modification of agro-wastes with PVDF/Fabric has enhanced its hydrophilicity and surface attraction towards water, thus increasing color removal of POME-FED.

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