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Development of Cockleshell Filter as Adsorbent in Palm Oil Mill Effluents

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ABSTRACT

The Malaysian palm oil industry has experienced significant growth, contributing to a substantial increase in processed waste, particularly palm oil mill effluent (POME). POME contains highly concentrated compositions that contribute to its brownish colour, posing environmental and health risks. The adsorption method, particularly using cockleshells (Anadara granosa), has emerged as a promising and environmentally friendly approach. The study aims to assess the performance of cockleshells in treating POME, focusing on reducing pollutants and colour to ensure compliance with effluent discharge standards for crude palm oil mills. Cockleshells specific size with size of 425 µm-1.17 mm, 1.18-2.36 mm, and greater than 10 mm adsorbent produced. Characterization of the adsorbent was done utilizing Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR) analysis to observe the surface morphology and functional groups. The preparation process for cockleshells as an adsorbent involves several phases. It operates with a column filter model by layer with medium sand, cockleshell, and fine sand with sizes of 1 mm-2 mm and 0.5 mm-1 mm. The parameters measured for the untreated and treated POME are temperature (°C), pH, turbidity (TUR), colour (ADMI) and total suspended solid (TSS). The best results were achieved by cockleshell size of 425µm-1.17mm where removal efficiencies are Colour=56.32%, TUR=81.89 %, and TSS=62.20% while temperature =23.92°C and pH=8.56. The cockleshell adsorbent's functional groups, such as alkene, are essential to the adsorption process. The surface structure of cockleshells that are porous and uneven surface makes the adsorption process effective.

1. Introduction

The palm oil industry currently is one of the agro-based industries that have a high contribution to the global economy including Malaysia [1]. This high-demand industry that is usually found in equatorial climate nations has also been identified because of its various by-products and their usage [2]. As environmental awareness grows, natural materials with properties like biodegradability and

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eco-friendliness are being developed, and palm oil byproducts are increasingly used to create sustainable solutions [3]. In Southeast Asia, Malaysia's palm oil industry plays an important part in the Malaysia economy and presently becomes the global second largest palm oil producer of commodity after their neighboring country, Indonesia [4]. Despite the advancement in the palm oil industry being great for the economy, there is a negative impact on the environment caused by the production of by-product palm oil, which results in high waste pollution known as palm oil mill effluent (POME). Every tonnes production of crude palm oil will generate about 2.5 tonnes of POME. Therefore, as the number of production increases then the amount of POME generated increases [4,5]. The characteristic of POME is brownish due to the colloidal mixture, gives off an unpleasant smell, and usually shows temperatures from 80-84°C. It also contains 95-96% of water, 4-5% of total solids and 0.6-0.7% of oil [2]. The high quantity of carotene, pectin, tannin, phenolic compounds and lignin contributes to the brownish colour [6]. Decolorizing water before discharge is necessary to avoid this environmental concern [7]. Depending on the industry, organic substances, microorganisms and other sources frequently contaminate industrial wastewater. In this instance, the release of contaminants from industrial sources has severely surpassed other environmental issues in severity [8].

POME pollutants prevent sunlight from penetrating water bodies, which lowers photosynthetic activity. Additionally, the presence of organic materials reduces the amount of dissolved oxygen consumed by aquatic creatures and speeds up bacterial development. Therefore, POME must be treated before being released into the environment. On the other hand, the physical and chemical processes have limitations as well. A membrane system for the physical treatment of wastewater with an organic component is not recommended. Instead, as the organic matter accumulates on the membrane's surface, a fouling layer will form. As a result, treatment effectiveness is reduced and frequent maintenance is required [9].

Currently, a variety of technologies, including reverse osmosis, ion exchange, electrolysis and membrane filtering, are utilized to remove heavy metals in industrial effluent. Most of these techniques, however, can be highly complicated, expensive, time-consuming and sludge-heavy. Due to its large surface area and porosity the adsorption method, a physicochemical process is frequently utilized in various research. Additionally, compared with other conventional methods, the adsorption process is highly effective, has a low operating cost, and is also very environmentally friendly [8]. In Malaysia, especially on the west coast of West Malaysia, cockleshells (Anadara granosa), which live behaviorally buried in the sediment, have gained prominence as a valuable ecological marine product. They have demonstrated excellent effectiveness in the elimination of both inorganic and organic matter in contaminated water and can be employed as an economical adsorbent material. Calcium oxide (CaO) is created from calcium carbonate ($CaCO_3$) through calcination or thermal breakdown. CaO is an alkaline earth oxide that can be used to cleanse sewage and industrial effluent [10].

The primary goal of this research is to assess the performance of the cockleshell to clarify the effluents of POME. In addition, the adsorption technique is highly eco-friendly and has excellent efficiency compared to other conventional approaches. The complaints were obtained due to the low clarity of the water and the turbidity of the water. The health of the flora and fauna is impacted by the large number of organic contaminants in wastewater that are dangerous for the aquatic environment. This colored POME significant effect on water bodies when it releases wastewater, the adverse effects of untreated color effluents are aquatic ecosystems by reducing sunlight penetration, increasing water temperature and disrupting the natural balance of the ecosystem [11,12].

Adsorption treatment has a higher ability to treat contaminants at low concentrations than other wastewater treatment techniques, which is thought to be the main constraint of currently used conventional treatments [11]. Therefore, this research explores the potential of utilizing cockle shells as an innovative natural adsorbent for treating palm oil mill effluents, aiming to provide a sustainable and cost-effective alternative to conventional methods. Cockleshell filters as an absorbent offer a viable solution because cockleshells are numerous and renewable, and they have shown promising outcomes in adsorbing colour.

2. Methodology

2.1 Collection and Parameter of POME

The effluent of POME was collected at the final discharge from Kilang Kelapa Sawit Bukit Pasir Sdn Bhd as a wastewater sample for the experiment. POME samples were kept in the cool rooms of the UTHM Environmental Laboratory, although the temperature was verified that same morning to ensure reliable data. The initial parameters such as pH value, dissolved oxygen (DO), turbidity (TUR), biochemical oxygen demand (BOD), total suspended solids (TSS) and ammoniacal nitrogen (AN) of untreated POME were measured and recorded. Then, the POME will go through the treatment and final parameters were measured and recorded to compare with the initial parameters. Both initial and final parameters also were compared with the effluent standard discharged of POME stated by DOE Malaysia.

2.1.1 Preparation of cockleshell

Cockleshells (Anadara granosa) were collected near Pantai Parit Penyegat, Muar Johor. In general, the preparation process consists of many phases. First, the selected cockleshells have been gathered and cleaned to remove any pollutants or contaminants. This guarantees that the filtration material was in the absence of contaminants that could disrupt the adsorption process. Then, the cockleshell dried directly under sunlight. Next, cockleshells were dried in a drying oven at 110 °C for 1 h to ensure complete drying before being crushed with a pestle and mortar [13]. Depending on the intended size of the adsorbent particles, this involves grinding and pounding the cockleshells into granules form. Concerning the scope of the study, grinding can be accomplished mechanically or manually with a mortar and pestle. Subsequently, the resultant size powder was sieved using a stainless-steel laboratory sieve with an aperture of 12 mm for raw cockleshell, 425 µm-1.17 mm, 1.18 mm-2.36 mm to get respective sizes. Finally, the cockleshell powder was stored in a Ziplock bag. The use of various sizes of cockleshells is to study the adsorption effectiveness of each size and to find the best adsorption process. Figure 1 shows the process of preparation of cockleshells from the cleaning to the sieving process.

Fig. 1. The process of preparation of cockleshell (a) Cleaning (b) Drying (c) Grinding (d) Sieving (e) Packing [14]

2.2 Characterization of Cockleshell
2.2.1 Fourier Transform Infrared (FTIR) spectroscopy

The FTIR spectrum analysis offers information about the molecular structure of the functional groups present in the samples under research. Simple spectra are typically derived from substances with few IR active covalent bonds, whereas complex spectra contain many adsorption bands. Furthermore, the magnitude of the peaks in the spectrum indicates the amount of material present. Infrared is a useful tool for quantitative analysis when combined with modern software algorithms.

2.2.2 Scanning Electron Microscopy (SEM)

SEM pictures can recreate views of a sample's almost three-dimensional surface and depict the sample's morphology. Consequently, the technique's primary application is to provide a high-resolution image of a surface feature from which judgments regarding the distribution of various chemical elements within the sample can be drawn. A resolving power of better than one nanometer can be achieved by modern SEMs. The entire operation takes place in the vacuum chamber. The cockleshells can be analyzed in an SEM to visualize the porous structure.

2.3 Preparation of Cockleshell Filtration

The layering of the cockleshell filter is shown in Figure 2 [15] and the functions for each layer are shown in Table 1. The first segment contains an intake reservoir into which treated effluents are poured. The second segment was made up of medium sand with particle sizes ranging from 1 mm to 2 mm, packed to a height of 6 cm. Medium sand acts as an additional layer in the filter bed, enhancing the overall effectiveness of the cockleshell filter in conjunction with the cockleshell medium. Medium sand serves as a supporting medium for cockleshell granules, which was one of its principal roles. The sand within the filter creates a stable and consistent bed, ensuring proper distribution of the effluent flow and preventing the formation of preferential flow routes and the cockleshell layer in the filter. The sand media maximizes contact between the effluents and the cockleshell granules by distributing the flow evenly, allowing for good adsorption of color compounds and total suspended solids. The third segment includes cockleshells with a height of 12 cm as the primary adsorber of POME. The fourth segment was made up of fine sand of particle sizes ranging from 1 to 0.5 mm. The fine sand layer is the last segment before the treated effluent runs into the outlet tube.

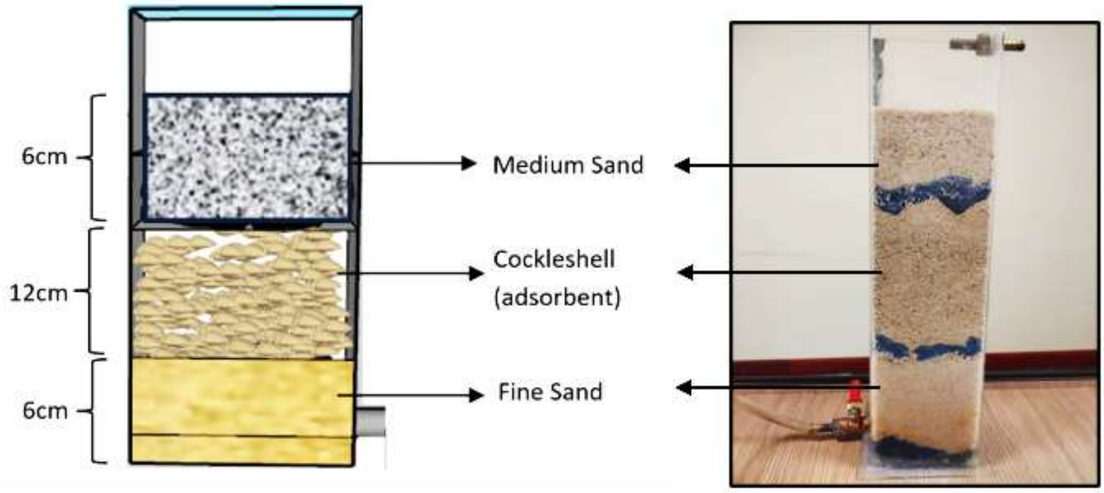

Fig. 2. The layering of cockleshell filter

Table 1
The components of the cockleshell filter and the functions

Components	Functions
Medium sand	Removes the pathogens and suspended solids.
Cockleshell	Acts as an adsorbent and holds the capacity of water.
Fine sand	Supports the cockleshell granular to prevent it from going into the drainage layer and outlet tube.

2.4 Analysis of Removal Percentage

The removal percentages of Colour, turbidity (TUR) and total suspended solid (TSS) were measured using Eq. (1) to examine the performance of the cockleshell filter [14,15]. Removal percentages obtained by treatment of cockleshell filter compared to the initial result and effluent standard discharged of POME stated by DOE Malaysia.

$$\text{Removal (\%)} = \frac{\text{Initial} - \text{Treated POME}}{\text{Initial POME}} \times 100 \quad (1)$$

2.5 Mechanism Adsorption Process of Cockleshell Filter

Figure 3 illustrates the adsorption mechanism that occurs between the pollutant particles in 2L POME and particles of cockleshell adsorbent. Cockleshell adsorbent attaches at the adsorbate in the POME and traps pollutant particles of POME followed by attached at the surface of the cockleshell, through the cockleshell layer. The existence of the aliphatic ether (C-H) group and alkene group increases the rate of absorption and bonding that assist in this mechanism [16,17]. The porous and uneven structure of the cockleshell surface also contributes to the effectiveness of this adsorption mechanism.

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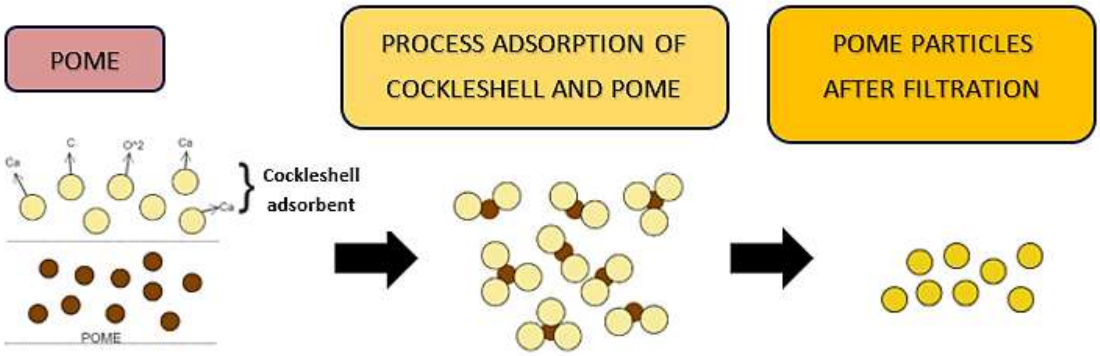


Fig. 3. Mechanism adsorption process of cockleshell filter

3. Results and Discussion

3.1 Initial POME Parameters

The initial parameters of collected POME from the factory are shown in Table 2 with a comparison to standard discharge limits for POME. Based on Table 2, all the parameters comply with the standard discharge limit set by the Department of Environment Malaysia (DOE) except colour.

Table 2
Initial value for raw POME and DOE discharge limits [18]

Parameters	Raw POME	DOE wastewater discharge limit
Temperature (°C)	25.32	40
pH	8.28	5.5-9.0
Color (ADMI)	1702.4	200
Turbidity	25.4	400
TSS (mg/L)	328	400

3.2 Fourier Transform Infrared (FTIR) Analysis

The structural and compositional details of the functional groups contained in the samples are provided by FTIR spectroscopy. The peaks located at 589, 704, 854, 1081 and 1450cm^{-1} observed in Figure 4 indicate the presence of the C-O and C-H group in the cockleshell. The peak location at 589cm^{-1} shows often corresponds to compounds like aliphatic nitrite. Next, the peak of 704cm^{-1} for cockleshell was assigned to the presence of the aliphatic acid halide. In addition, the peak located at 854cm^{-1} indicated the presence of the aliphatic ether (C-H) stretching group while 1081 cm^{-1} corresponded to the aliphatic alkoxy. The highest peak in Figure 4 located at 1450 cm^{-1} shows the presence of stretching of aliphatic isonitrile. The presence of functional groups such as alkene in the cockleshell adsorbent plays a major role in the adsorption process [19].

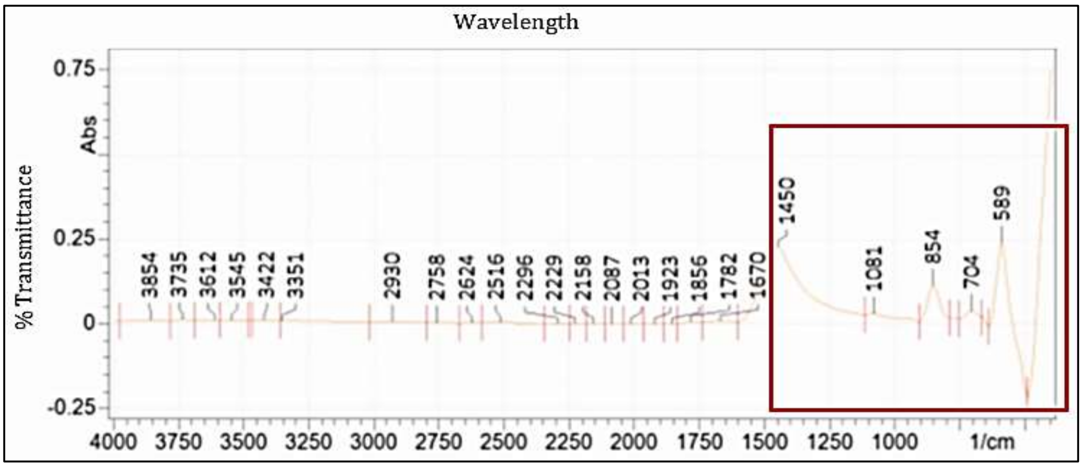
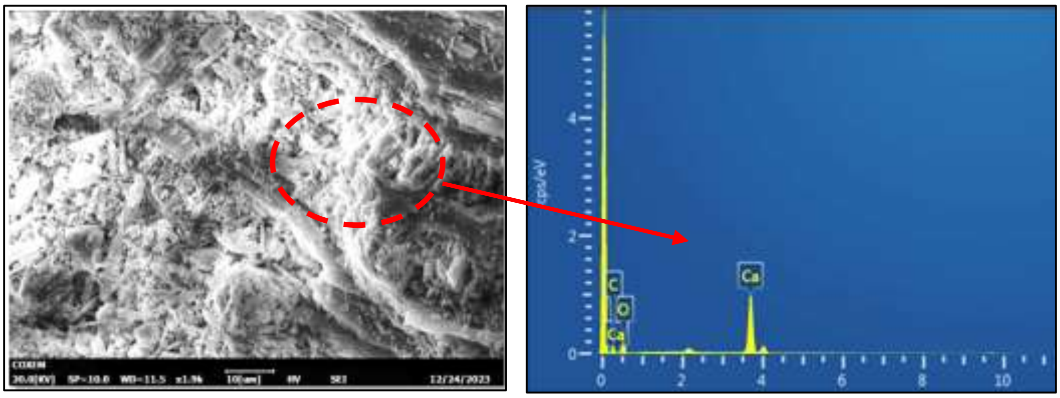


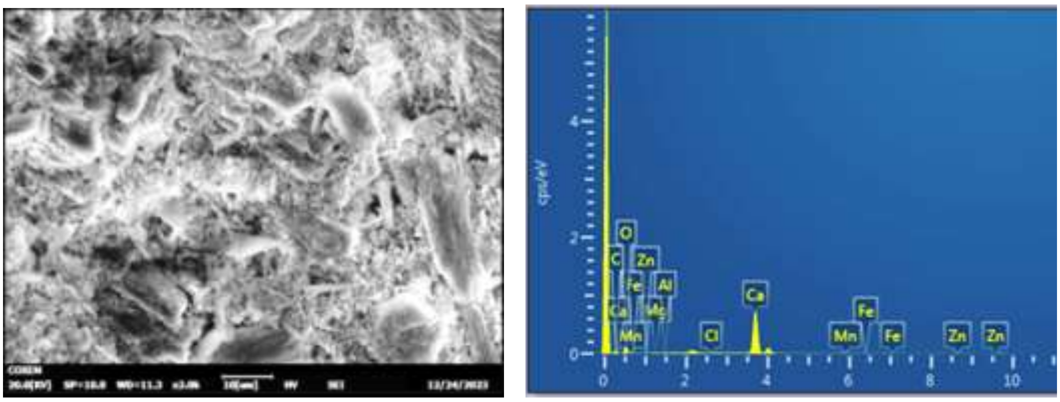
Fig. 4. FTIR spectrum of cockleshell

3.3 Scanning Electron Microscopy (SEM) Analysis

Figure 5 shows SEM morphology of cockleshell adsorbent (a) before and (b) after filtration experiment at 2,000 x magnification respectively. Furthermore, for effective trapping of the pollutant in the adsorbent's pores, porous and uneven surface shape is preferred [15]. The cockleshell surface in Figure 5(a) shows the surface structure of cockleshells that are porous and uneven surface and free from adsorbed substances. The porous and uneven surface makes the adsorption process effective in column filter [18]. However, following the filtration experiment, the adsorbent surface displays a denser structure with the attached element. Figure 5(b) shows that the attached element in the cockleshell surface reduces the palm oil mill effluent colour and turbidity. This might be the result of particles binding to the adsorbent's surface and depositing.



(a) (b)



(c) (d)

Fig. 5. SEM morphology of cockleshell adsorbent (a) Before (b) After filtration experiment

Figure 5(a) displays the chemical components of the cockleshell before the filtration process 29.43% calcium, 55.18% oxygen and 15.39% carbon represent the majority of the adsorbent's elements. The other chemical elements found in Figure 5(b) are Cl, Mg, Zn, Fe, Mn and Al with 0.05, 0.14, 0.11, 0.48, 0.17 and 0.08 of the element percentage of the cockleshell after the filtration experiment. The presence of another contaminant as shown in Figure 5(b) shows the efficiency of the cockleshell as an adsorbent and reduces the impurity in the Palm oil mill effluent (POME).

3.4 Discussion

In summary, the investigation into the initial parameters and their variations with different cockleshell sizes yields crucial insights for the efficiency of the cockleshell filtration process. The analysis of POME effluent after filtration process are shown in Table 3. The initial temperature of the POME was recorded at 25.32°C. Intriguingly, with the introduction of cockleshells, a trend is observed wherein larger sizes (>10 mm) marginally elevate the temperature to 25.86°C, while the smaller sizes (1.18 mm-2.36 mm and 425 µm-1.17 mm) exhibit a favourable reduction to 23.8°C and 23.92°C, respectively. This nuanced temperature modulation is pivotal for process optimization, emphasizing the significance of carefully selecting the cockshell sizes. The initial pH average of 8.28 for POME with the introduction of a variety cockleshell sizes. Larger cockleshells (>10mm) showed a marginal elevation, reaching an average pH of 8.77, whereas smaller size categories, particularly the 1.18 mm-2.36 mm and 425 µm-1.17mm ranges, showed a more favourable reduction to 8.56.

Table 3
Analysis of POME after filtration with DOE discharge standard

Parameters	Initial average value	Cockleshell sizes for average parameters			DOE discharge limit
		>10 mm	1.18 mm-2.36 mm	425 µm-1.17 mm	
Temperature (°C)	25.32	25.86	23.8	23.92	45
pH	8.28	8.66	8.77	8.56	5.0-9.0
Color (ADMI)	1702.4	1654	1620.2	743.6	200
Turbidity (NTU)	25.4	17.22	5.10	4.60	400
TSS (mg/L)	328	248	204	124	400

Moreover, the investigation into the impact of the various cockleshell sizes on crucial parameters such as Colour revealed nuanced trends with major implications for the efficiency of the cockleshell filtering system in treating POME. The initial state, marked by a Colour value of 1702.4 ADMI, was notably diminished with the introduction of cockleshells. This reduction was most pronounced in the smaller size categories, with the 1.18 mm-2.36 mm and 425 µm-1.17 mm ranges demonstrating the greatest efficacy, resulting in average color values of 1620.2 ADMI and 743.6 ADMI, respectively. The treated effluents became yellow after the adsorption because of cockleshell adsorbent sizes and the sand filtration improved the water quality and was more suitable for aquatic life as shown in Figure 6. The reduction in coloring indicates cockleshell adsorption potential, particularly in finer-size fractions [20].

Fig. 6. POME before and after the filtration process

Furthermore, the report indicates that the cockleshell filtration process is effective in reducing the turbidity and total suspended solids (TSS) of the POME. The initial turbidity of 25.4 NTU witnessed a substantial decline across different cockleshell sizes, reaching an average of 5.10 NTU for the 1.18 mm-2.36 mm range and 4.60 NTU for the 425 µm-1.17 mm range. This demonstrates the effectiveness of smaller cockleshells in clarifying the POME, aligning with expectations of enhanced filtration efficiency with finer particles. Moreover, total suspended solids (TSS), initially at 328 mg/L, exhibited a notable reduction, with the 1.18 mm-2.36 mm and 425 µm-1.17 mm cockleshell sizes yielding averages of 204 mg/L and 124 mg/L, respectively. This underscores the potential of cockleshells, especially in finer-size fractions, to mitigate suspended solid content in the wastewater.

Given that the proportion of adsorption increases with the increasing surface area of the cockleshell [21,22]. The adsorbent has an excellent capacity for adsorption of color and turbidity as shown in Table 4. The evaluation of the cockleshell adsorbent performance was shown by percentage removal by calculation of initial and final parameters. The best results were achieved by cockleshell size of 425 µm-1.17 mm where removal efficiencies are Colour (56.32%), TUR (81.89%), and TSS (62.20%) while temperature (23.92°C) and pH (8.56).

Table 4
Percentage removal of colour, turbidity and TSS with different sizes of cockleshell

Percentage removal (%)	Cockleshell sizes		
	>10 mm	1.18 mm-2.36 mm	425 µm -1.17 mm
Colour	2.84	4.83	56.32
Turbidity	32.20	80.28	81.89
TSS	24.39	37.80	62.20

4. Conclusions

The study was conducted to assess the capability of cockleshell as an adsorbent in palm oil mill effluents. The adsorbent characteristic was analyzed by FTIR and SEM which confirm the presence of an aliphatic group of hydrocarbons and an alkaline functional group as an adsorbent. Several parameters with three different sizes of cockleshells were studied to investigate the performance of cockleshells as adsorbents like pH, temperature, color, turbidity and TSS. The studies prove that the cockleshell is an efficient low-cost adsorbent for the removal of color and turbidity from palm oil mill effluents as shown in the removal percentage in Table 4. This is supported by SEM analysis, which revealed a decrease in empty spaces on the cockleshell surface following adsorption as compared to pre-adsorption. The surface morphology changed and contained contaminated material in the form of needles. This was because the cockleshell had bound the metal in the effluent sample. The unique properties of cockleshells, ultimately contribute to more effective processes, enhanced product quality, and create sustainable natural material production. Future studies could evaluate the performance of cockle shells with sizes smaller than 425 µm to determine their potential for further enhancing adsorption efficiency.

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