

Research Article

Biomass Modification Using Cationic Surfactant Cetyltrimethylammonium Bromide (CTAB) to Remove Palm-Based Cooking Oil

Amira Satirawaty Mohamed Pauzan  and Normala Ahad

Faculty of Resource Science and Technology, Universiti Malaysia Sarawak, 94300 Kota Samarahan, Sarawak, Malaysia

Correspondence should be addressed to Amira Satirawaty Mohamed Pauzan; mpasatirawaty@unimas.my

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Adsorption based on natural fibre seems to widely used for oily wastewater recovery due to its low cost, simplicity, feasibility, easy handling, and effectiveness. However, oil sorbent based on natural fibre without modification has low adsorption capacity and selectivity. Thus, this paper proposes chemical modification of sago hampas to improve its adsorbent efficiency for the removal of palm-based cooking oil. The chemical modification was performed using a cationic surfactant, cetyltrimethylammonium bromide (CTAB). The chemical and surface properties of both unmodified and modified sago hampas were characterized by Fourier-Transform Infrared (FTIR) and Scanning Electron Microscopy (SEM). Parameters studied for the removal of cooking oil using modified sago hampas were sorption time, adsorbent dosage, and initial pH. The removal capacity was also compared using unmodified sago hampas. The results showed that additional functional groups were introduced on the surface of modified sago hampas. Modified sago hampas also showed a greater porosity than unmodified sago hampas. These properties enhanced the adsorption of palm-based cooking oil onto the surface of modified sago hampas. Modified sago hampas shows better removal of palm-based cooking oil than unmodified sago hampas, where 84.82% and 68.08% removal were achieved by modified and unmodified sago hampas, respectively. The optimum adsorption of palm-based cooking oil was identified at 45 min sorption time, pH 2, and 0.2 g adsorbent dosage.

1. Introduction

Oily wastewater is a big issue in the world since it is a persistent environmental pollutant [1]. Oily wastewater can come from a number of different sources such as waste cooking oil that enter the water from kitchen. River and lake contaminated with oil can have devastating effects on the water environment. Many methods have been introduced to reduce and minimize oily wastewater but the quality of the water seem to be far from satisfactory and contribute to serious physical effect to ecology. Oil is dispersed over the water surface and form oil layer. It could reduce oxygen supply to the water and eventually lead to death of aquatic life forms. There are a few methods that have been developed to reduce and minimize oil contaminant in water including physical, mechanical, biological and photochemical recovery, and filtration but these methods are not very efficient

in removing different types of oil and slow in removal of oil, and it is of high cost [2].

Adsorption process have been reported as the most efficient method since it does not require additional chemical and large amount of energy particularly adsorption by activated carbon. However, activated carbon is difficult to regenerate due to its strong interaction with adsorbed molecule [3].

A new method has been introduced which is adsorption using natural fibres for oily wastewater where the method is very effective, simple, and low cost [4]. Many natural fibres have been utilized as oil sorbents such as sugarcane bagasse, sawdust, barley straw, rice husk, wool, kapok, and grass [5–8]. These natural fibres are the best oil sorbent since it is low cost, sustainable, and abundant compared to the other sorbent.

Sago hampas is a natural fibre scientifically known as *Metroxylon sagu*. It is the main commodity crop of Sarawak, Malaysia, and potentially can be used to remove oil [9]. Sago

comes from genus *Metroxylon* and Palmae family [10]. Sago hampas is highly degradable, has good sorption capacity, chemical-free, and has comparable density with the synthetic sorbent. However, the disadvantages of natural sorbents are lack of hydrophobicity and low buoyancy [11]. This can cause reduction in effectiveness of oil sorption in the aqueous system. It is widely known that unmodified or raw sago hampas with lack of hydrophobicity can be improved by either chemical or physical modifications. The chemical modification of sago hampas by attaching various functional groups is easy to conduct since it contains lignin and cellulose with high amount of hydroxyl functional group that is prone to chemical modification [12].

Although in some recent studies good adsorption performance to remove various metal ions has been reported for chemical modification of biomass [13, 14] or dye removal [15], no studies reported on the chemical modification by cationic surfactant on sago hampas to improve its performance for removal of oily wastewater. This paper aims to investigate the adsorption capacity of cetyltrimethylammonium bromide (CTAB) surfactant-modified sago hampas on palm-based cooking oil at optimum parameters studied which are sorption time, adsorbent dosage, and initial pH. The used of chemically modified sago hampas for oil adsorption is one of the ways that has the potential to reduce oily wastewater in the environment.

2. Materials and Methods

The absorption capacity of palm-based cooking oil by surfactant-modified sago hampas involves two steps. In the first step, the surface properties of sago hampas were modified using CTAB surfactant. Next, optimum removal of palm-based cooking oil using modified sago hampas was studied at three different parameters: sorption time, adsorbent dosage, and initial pH. The detailed steps involved in this study are described next.

2.1. Modification of Sago Hampas. Collected sago hampas from Mukah, Sarawak, was washed repeatedly with distilled water to remove any impurities or dust. It was air-dried to remove excess water and then further dried at about 80°C for 24 h in an air circulating oven. The dried sago hampas was ground to a fine powder and sieved (500 μm) before modification to have reproducible result and uniform modification. The dried sago hampas was pretreated with 1 M KOH solution to improve its binding site. Five grams of sago hampas was immersed into 100 mL 1 M KOH. The mixture was stirred for 24 h at 30°C prior to filtration to achieve good penetration of chemical into the interior of the precursor [16]. Then, the pretreated samples were washed with hot water (80°C), mild acid (0.1 M HCl), and base (0.1 M NaOH) till the effluent water shows neutral pH. Then, the samples were dried at 70°C overnight. After pretreated by KOH, sago hampas was modified using CTAB solution. Two grams of pretreated sago hampas was immersed into 100 mL of 0.1 M CTAB solution and stirred for 24 h [17]. The mixture was filtered and washed repeatedly by distilled water

in order to remove unreacted surfactant. The sample was dried in an oven at 50°C, sieved, and used for characterization and batch adsorption tests.

FTIR (Thermo Scientific/Nicolet iS10) analysis was done on unmodified sago hampas and modified sago hampas to determine the functional groups that are present before and after the modification process. The functional group of pure CTAB and modified sago hampas loaded with oil at optimum condition also has been determined. The unmodified and modified sago hampas was ground with KBr and made into discs. The spectrum was recorded on FTIR in the spectral range 4000–500 cm⁻¹ under ambient condition.

The morphology of sago hampas before and after the modification by using cationic surfactant, CTAB, was observed by scanning electron microscopy (SEM) (JSM-63901A) at 500x and 1000x magnification. The sample was placed on an aluminium stub using double stick. Under a vacuum condition, a thick gold layer was sputtered on the surface by using sputter coater in order to make the sample surface conductive. SEM imaging was used to analyse the morphology of the sample. The difference in the surface structure and porosity was identified between unmodified and modified sago hampas.

2.2. Batch Adsorption Experiments. Batch adsorption experiments were performed, and the mass of palm oil before and after the sorption was measured. In adsorption experiments, 5 mL (4.45 g) of palm oil purchased from local supermarket was used. The initial mass of 250 μm size of adsorbent was measured. Each sample was agitated by a rotary shaker (Heidolph Rotamax 120) at 120 rpm at respective time to reach equilibrium of the solid-solution mixture. The samples were collected and filtered. The sample loaded with oil was air-dried until no further filtered oil was observed. Further drying was conducted at about 60°C for 30 min to evaporate water and *n*-hexane that might be trapped on sago hampas during adsorption process [18]. The final mass of sago hampas was recorded. All experiments were carried out in triplicate for each condition. The adsorption capacity was calculated using the following formula:

$$\text{Adsorption capacity (g/g)} = \frac{(S_t - S_o)}{S_o} \quad (1)$$

The percentage of oil removal will be determined using the following formula:

$$\text{Oil removal (\%)} = \frac{(m_{\text{oil}})}{(m_{\text{oil,o}})} \cdot 100, \quad (2)$$

where m_{oil} and $m_{\text{oil,o}}$ are the amount of palm oil extracted from sago hampas (g) and initial amount of palm oil (g), respectively. S_t is the total mass of absorb sample and S_o is the initial weight of sample. This procedure was applied to those of adsorption tests for modified sago hampas and unmodified sago hampas.

2.3. Effect of Sorption Time. Different sorption times which are 2 min, 5 min, 10 min, 15 min, 30 min, 45 min, and 60 min

were carried out for sorption experiment. The original pH of palm oil was not adjusted. Adsorbent dosage used was at 0.20 g, and it was spread evenly on top of 5 mL palm oil solution. It was agitated at 120 rpm. The highest adsorption capacity obtained was used to compare the efficiency between unmodified and modified sago hampas.

2.4. Effect of Adsorbent Dosage. Sorption experiment was studied at different dosages of modified sago hampas which are 0.1 g, 0.2 g, 0.3 g, and 0.4 g. The study was carried out for 1 h with 5 mL of palm oil solution. The original pH of palm oil was not adjusted. It was agitated at 120 rpm. The highest adsorption capacity obtained was used to compare the efficiency between unmodified and modified sago hampas.

2.5. Effect of Initial pH. The adsorption of palm oil by the modified sago hampas was carried over pH 0.5, 1, 2, 4, 6, 8, and 10 at room temperature for 1 h. Adsorbent dosage used was at 0.20 g and spread evenly on top of 5 mL palm oil. It was agitated at 120 rpm. A few drops of 0.1 M sodium hydroxide solution (NaOH) or 1 M hydrochloric acid solution (HCl) was added to each flask containing 0.20 g adsorbent and 5 mL palm oil to adjust the pH. A little amount of *n*-hexane was added in palm oil in order to solubilize the HCl in palm oil. The pH was measured by using pH meter. The highest adsorption capacity obtained was used to compare the efficiency between unmodified and modified sago hampas.

2.6. Effect of Modified and Unmodified Sago Hampas. The effect of modified and unmodified sago hampas on the adsorption of palm-based cooking oil was studied to compare the removal efficiency of palm oil by using both unmodified and modified sago hampas. Both experiments were carried out by using the optimum adsorption capacity on modified sago hampas. It was carried out with 0.2 g of adsorbent dosage with 250 μm size that was spread evenly on top of 5 mL palm oil solution. It was agitated at 120 rpm agitation speed for 45 min. It was conducted at room temperature with pH 2 of palm oil.

3. Results and Discussion

3.1. Scanning Electron Microscopy (SEM) Analyses. The morphology of sago hampas before and after modification by using cationic surfactant, CTAB, was observed by scanning electron microscopy (SEM) at 500x and 1000x magnification. At such magnification, the surface structures and porosity can be clearly seen. The surface morphology of unmodified sago hampas was different from modified sago hampas because the modification significantly alters the porosity and physicochemical properties of the materials [19]. After modification by using CTAB, the surface roughness was increased with the formation of pores throughout the structure. The SEM analyses revealed that modified sago hampas contained numerous pores, which can hold oil. The pore distributed is different in sizes. Figure 1 presents the SEM analyses of (a) unmodified sago

hampas and (b) modified sago hampas at 500x and 1000x magnification.

3.2. Fourier-Transform Infrared (FTIR) Analyses. Fourier-transform infrared (FTIR) analysis was used to investigate the effect of the modification on the surface structure of sago hampas. It was done upon unmodified sago hampas and modified sago hampas in order to determine the functional group that is present before and after modification by using CTAB. The functional group that is present in CTAB and modified sago hampas loaded with oil at optimum condition was also being observed.

FTIR spectrum of pure CTAB showed bands at 3020–2800 cm^{-1} attributed to C-H stretching bands of alkylammonium cations. The band at 1472.27 cm^{-1} is an indicative of high organization of the $-\text{CH}_2$ chain conformation. The FTIR spectrum of pure CTAB also shows the C-N stretching band at 950–900 cm^{-1} .

Both FTIR spectra of modified and unmodified sago hampas show C-H deformation of the methyl group stretching at 1325–1320 cm^{-1} . The C-O stretching vibration at 1000 cm^{-1} is due to the cellulose backbone [5]. Beyond the fingerprint region, a characteristic broadband in both modified and unmodified occurs in the range of 3600–3100 cm^{-1} , which corresponds to the stretching mode of O-H groups, hydrogen-bonded O-H, and chemisorbed water [16]. FTIR spectrum of modified sago hampas reveals more distribution peaks than that are found in unmodified sago hampas. Modified sago hampas have two sharp bands at 3000–2850 cm^{-1} that refer to the asymmetric and symmetric stretching vibrations of the $-\text{CH}_3$ and $-\text{CH}_2$ groups in CTAB [20]. The peak at 1700–1500 cm^{-1} shows the C=O of the carboxylic group of celluloses and lignin was seen in unmodified sago hampas but becomes weaker at modified sago hampas due to the alkali pretreatment [21]. These constituents cannot be completely removed in the dilute alkali solution [22]. Moreover, modified sago hampas underwent the $-\text{CH}_2$ vibration at 1366.03 cm^{-1} due to the modification by using CTAB [23]. Extra peak that is present at 900.36 cm^{-1} in modified sago hampas shows the presence of C-N stretching vibration support the modification of sago hampas by CTAB [24].

FTIR spectrum for modified sago hampas loaded with oil at optimum condition having peaks at 2920.35 cm^{-1} and 2851.53 cm^{-1} was observed to be stronger than modified sago hampas. It demonstrated the adsorption of palm oil to alkyl chain layer that contributed by CTAB on the surface of modified sago hampas [25]. The weaker peak at 1100–1000 cm^{-1} on modified sago hampas after adsorption of oil is probably because the interaction between the cellulose backbone (C-O) with palm oil at optimum condition. The extra peak between 1800 cm^{-1} and 1700 cm^{-1} on modified sago hampas after adsorption shows the present of C=O which was contributed by ester from palm oil [26].

3.3. Batch Adsorption Experiments. Batch adsorption experiments were carried out with fixed size of modified sago

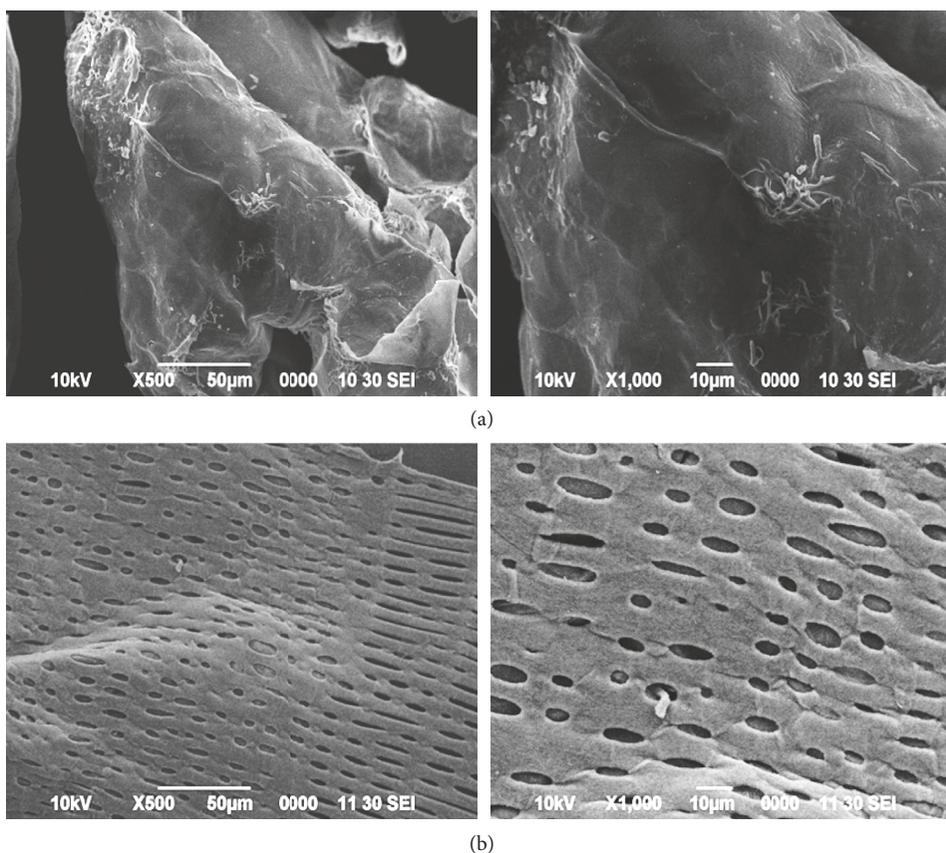


FIGURE 1: (a) 500x and 1000x magnified unmodified sago hampas. (b) 500x and 1000x magnified modified sago hampas.

hampas at $250\ \mu\text{m}$. It was agitated at 120 rpm in room temperature at respective time in 5 mL (4.45 g) of palm-based cooking oil. The effect of sorption time, adsorbent dosage, and pH were carried out. The optimum value for each parameter was taken into account to compare the efficiency of unmodified sago hampas and modified sago hampas to remove palm-based cooking oil.

3.4. Effect of Sorption Time. Figure 2 demonstrates the effect of sorption time on oil removal by modified sago hampas from 2 min to 60 min. Removal of palm oil increases when the sorption time increases. The adsorption capacity increases with the sorption time from the first 2 min. The maximum value for adsorption was reached at 45 min with 13.203 g/g of the adsorption capacity. This effect might be due to adsorption of palm oil on the surface of the sorbent which then starts to break through the inside microscopic voids [27]. There is large amount of available sites that causes a strong hydrophobic interaction between adsorbent and adsorbate. The results also showed the fast and stable nature of the process as only a little difference was observed between the initial and final contact time. When the sorption time is prolonged to 60 min, slow adsorption was observed. This is due to limited available sites of sorbent surfaces for oil entrapment [28]. The use of sorption time over 45 min has no significant effect on the sorption of the palm oils. Therefore, the optimum sorption time was 45 min.

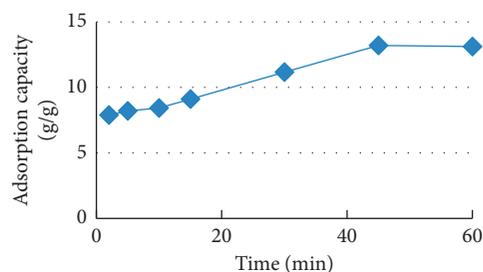


FIGURE 2: Effect of sorption time.

3.5. Effect of Adsorbent Dosage. The effect of adsorbent dosage on the removal of palm oil was carried out by using different dosages of modified sago hampas. The weight of modified sago hampas used was 0.1 g, 0.2 g, 0.3 g, and 0.4 g. The same size of modified sago hampas at $250\ \mu\text{m}$ was used. Figure 3 shows the adsorption capacity and percentage of oil removal of palm-based cooking oil that are plotted on the same axis against adsorbent dosage. It clearly shows that, with raise in adsorbent dosage of modified sago, hampas increases the percentage removal of palm oil and expressed that the existence of large surface is feasible for adsorption with decreasing the maximum amount of adsorbed palm oil per gram of adsorbent [29]. When sorbent dosage increases, the adsorption capacity of sago hampas decreases from 16.307 g/g to 7.647 g/g. This is mainly due to increase of unsaturated oil binding sites [25]. Moreover, the efficiency of

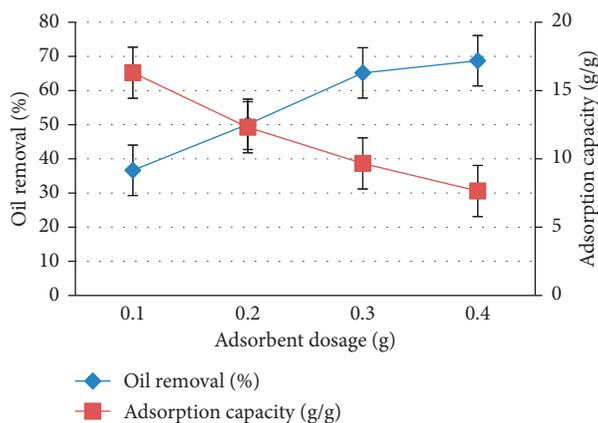


FIGURE 3: Effect of adsorbent dosage.

the adsorption of oil also decreased after maximum sorption capacity has been reached. This is because the saturation of sorbent has occurred [30]. Higher adsorbent dosage resulted in high percentage of oil removal of 68.734%. This is due to larger surface area and more adsorption sites available [31]. Considering the removal as well as the adsorption capacity, the intersection part of the plot is chosen as the optimum dosage of adsorbent [32]. Therefore, the adsorbent dosage at 0.2 g was chosen to be compared with unmodified sago hampas.

3.6. Effect of Initial pH. The effect of pH on oil adsorption is shown in Figure 4. The initial pH of the mixture is an important parameter in the adsorption processes since it influences the surface binding sites and surface properties of adsorbent by the concentration of proton or hydroxide ions in solution [33]. Figure 4 clearly presents that the palm oil adsorption process on modified sago hampas is highly dependent on pH.

The greatest adsorption capacities of modified sago hampas were obtained at pH 2. At pH 2, palm oil adsorption increased. Strong acidic condition intensifies oil to form unstable flocs, where modified sago hampas induce physicochemical effect which enhances the adsorption of oil. Adsorption of oil is unlikely to occur through ion exchange. However, spontaneous protonation and deprotonation of surface functional groups still can influence the adsorption. Oil droplets and hydrocarbon often carry negative charge in aqueous solution [34]. Therefore, at pH 2, due to the opposite charge hydrogen ions, the droplet repulsion forces decreased and coalescence occurs apparently serving to rapid demulsification. Moreover, the hydrogen ions could protonate the surface charge of sago hampas and their degree of ionization making it a better residual-oil adsorbent at this condition. However, the adsorption capacity decreased when pH is lower than 2. At pH 1 and 0.5, although hydrogen ions are helpful to induce droplet coalescence and protonization; an excessive charge concentration might delay the deposition and coalescence due to electrostatic repulsive forces between hydrogen ions [35]. Meanwhile, at pH 2, the hydrogen ion concentration is just enough for

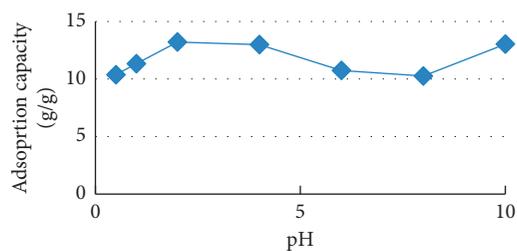


FIGURE 4: Effect of initial pH.

electrostatic forces act in favor of faster adsorption. When the pH increases from 2 to 6, the adsorption capacity decreased. This can be explained by the decrease in the dissociation of acid in the solution that reduce the concentration of palm oil available to interact with positively charged adsorbent sites [36].

Under basic condition, the adsorption of palm oil increased. However, this is not corresponding to palm oil removal efficiency since the addition of excessive NaOH in order to increase the pH could result in saponification process [37]. Addition of NaOH thus makes the palm oil react and undergo hydrolysis process that produce glycerol and fatty acid salt called soap, thus making adsorption of palm oil increased. Hence, inspection of Figure 4 indicates that the optimum pH is 2.

3.7. Effect of Modified and Unmodified Sago Hampas. The effect of modified and unmodified sago hampas on the adsorption of palm-based cooking oil was studied to compare the removal efficiency of palm oil by using both unmodified and modified sago hampas. Both experiments were carried out by using the optimum adsorption capacity that has been carried out on modified sago hampas. It was carried out with 0.2 g of adsorbent dosage with 250 μm size and agitated at 120 rpm agitation speed for 45 min. It was conducted at room temperature with pH 2 of palm oil (5 mL).

Modified sago hampas adsorbs large amount of palm oil compared to unmodified sago hampas. The adsorption capacity of modified sago hampas is 18.880 g/g while unmodified sago hampas is 15.147 g/g. The percentage of oil removal for modified sago hampas and unmodified sago hampas is 84.82% and 68.08%, respectively. The percentage of oil removal difference between both modified and unmodified sago hampas is 16.75%, clearly showing that modified sago hampas adsorbs palm-based cooking oil more efficient than unmodified sago hampas. The pretreatment using KOH increases the negatively charged site on the sago hampas, and modification by CTAB reverses the surface potential charge from negative to positive [19]. The modification of sago hampas by CTAB increases the performance to adsorb palm oil. Electrostatic interaction between the negative charge of palm oil at aqueous solution molecules and positive charge introduced by cationic surfactant (CTAB) on the surface of modified sago hampas can be considered as the significant reason for the improved adsorption capacity of the modified sago hampas. The poor

adsorption capacity for unmodified sago hampas for removal of palm-based oil might be due to the repulsive electrostatic interactions between the palm oil molecules and the negative sites on the surface of the sago hampas particles [31]. According to [16], the treatment process enhanced the porosity on the surface of sago hampas, thus increasing the number of adsorption site. Rough surface morphology sorbent surfaces are also needed to high adsorption capacity. The morphology and structure have more active sites for oil adsorption in modified sago hampas rather than smooth surfaces in unmodified sago hampas [9].

4. Conclusions

Modified sago hampas by chemical modification using cationic surfactant, cetyltrimethylammonium bromide (CTAB), was found to be an effective sorbent to remove palm-based cooking oil. The characterization of modified sago hampas using FTIR shows the presence of CTAB on the surface of sago hampas. In addition, scanning electron microscopy (SEM) shows that modified sago hampas has higher porosity in the surface morphology compared to unmodified sago hampas. This leads to higher adsorption efficiency of palm-based cooking oil using surfactant modified sago hampas compared to unmodified sago hampas.

Data Availability

The (percentage of oil removal) data used to support the findings of this study are included within the supplementary information file(s).

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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Supplementary Materials

The raw data for each parameter. (*Supplementary Materials*)

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