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Nitrogen Tailored Activated Carbon via Microwave Synthesis Method for High Removal of Hydrogen Sulfide

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Abstract In this work, commercialized palm shell activated carbon was modified into nitrogen-functionalized carbon via urea impregnation method, followed by heat treatment via microwave irradiation at 950 °C. The efficiency of nitrogen-functionalized palm shell activated carbon (N-PSAC) towards hydrogen sulfide (H_2S) removal was compared to raw palm shell activated carbon and sodium hydroxide impregnated palm shell activated carbon (NaOH-PSAC). These adsorbents were tested for H_2S adsorption using simulated POME biogas composition. The composition of simulated POME biogas contains 3000 ppm of H_2S, 40% of CO_2 and 59.7% of CH_4. Experimental data showed that N-PSAC adsorbent performed better, which obtained comparable adsorption capacity (264.3 mg/g) with NaOH-PSAC (240.87 mg/g). The chemical and physical properties of the adsorbents were tested using elemental analysis, nitrogen adsorption desorption analysis, FTIR, and SEM analysis. Higher sorption capacity of H2S using N-PSAC showed that the introduction of basic nitrogen functionality via thermal treatment on palm shell activated carbon, significantly improved the performance of palm shell activated carbon as H_2S adsorbent.

Keywords: Hydrogen sulfide, activated carbon, biogas, nitrogen functionalized.

Introduction

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Palm oil industry in Malaysia has grown rapidly over the years and is accounted as one of the largest oils and fat producer in the world. Even though the industry has significantly contributed to the country's economy, increase in worldwide demand of oil palm will also worsen the environmental pollution. In 2017, about 51.19 million tonnes (Mt) of oil palm waste was generated mainly from oil palm fronds (OPF), oil palm trunks (OPT), empty fruit bunch (EFB), mesocarp fibre (MF), palm kernel shell (PKS) and palm oil mill effluent (POME) [1]. To reduce the waste generation from palm oil industry, POME has been utilized to produce biogas via anaerobic digestion process and it has been applied in some of palm oil mills in Malaysia as a feedstock for biogas production [2]. In general, biogas composition produced from POME is typically contains 50 – 70 % of methane (CH_4), 30 – 40 % of carbon dioxide (CO_2) and traces (1000 – 3000 ppm) of hydrogen sulfide (H2S) [2-3]. These gases have been identified as greenhouse gas (GHG) source, which could contribute to global warming effect. The GHG potential of CH4 is approximately 20 times of CO_2 and it would be equivalent to 7.2 million tons of CO_2 emission [4]. Since CH4 has physical and chemical properties that is close to natural gas, it would be a great advantage to integrate CH4 as a renewable energy and at the same time eliminating its contribution towards global warming. However, to utilize CH4 as a renewable energy, traces of H2S need to be removed.

H2S is an extremely toxic and hazardous gas with unpleasant smell. The presence of H2S in biogas could cause corrosion to the gas engine and metal parts via emission of sulfur dioxide (SO2) during combustion, especially when the gas engine is not operated continuously. Apart from that, H2S and SO2 concentrations emitted to the workplace would be toxic for workers as well [5]. This has been reported that, even at a very low concentration of H2S (5 ppm) it is still hazardous to human health [6]. To cater this problem, there are various technologies that are available to remove traces of H2S from biogas, such as absorption, adsorption on a solid material (such as iron oxide-based materials or activated carbon), and biological conversion [5-6]. Among these technologies, adsorption on activated carbon/impregnated activated carbon has been widely used due to its effectiveness in removing gaseous pollutant [5].

Activated carbon is a low-cost adsorbent where it could be easily modified to obtain good surface properties that is capable for high adsorption capacity and fast reaction kinetic. The use of lignocellulosic biomass to be converted into activated carbon precursor is relatively economic and easy because it is abundantly available. The utilization of palm shell biomass into activated carbon adsorbent for H2S removal in this study could establish a 'carbon neutral cycle', since both are by-products produced from palm oil industries [7]. The adsorption capacity of modified activated carbon will depend on the type of chemicals used, due to the changes of surface chemistry and physical characteristics. Previous researchers reported, to enhance the adsorption capacity of H2S, the activated carbons need to be modified via chemical activation, which promotes high oxidation of H2S to elemental sulfur [8-10]. This includes introducing oxidative agent via impregnation method (KOH, NaOH, NaCO3, KI, KMnO4) and alteration of surface chemistry of the carbon matrix and incorporation of heteroatom such as nitrogen functionality (urea, melamine, NH3). The introduction of nitrogen onto activated carbon surface could enhance the adsorption of H2S. The existence of nitrogen on activated carbon surface can increase the basic groups, thus the oxidation of acidic gas such as H2S into sulfur can occurred easily [11-13].

The objective of this paper is to study the effect of nitrogen enhanced adsorbent by using urea as a source of nitrogen onto palm shell activated carbon (PSAC). The synthesis method will be done via microwave (MIW) irradiation heating method. Previously, researchers have conducted study related to the effect of nitrogen functionality on adsorption of H2S by using various type of waste materials [11-13]. Up to date, nitrogen-modified activated carbon has been produced via conventional heating method by using external heat source which are very slow and inefficient, due to the transfer of energy into the system. However, in this study MW heating method will be introduced, where it could offer efficient heating mechanism since it requires short reaction time. Moreover, activated carbon that undergo MW heating is a great absorbers of MW energy and it could convert the energy easily into heat [14-16]. This study is conducted for the purpose of improving the physicochemical properties of PSAC. For this purpose, the best selected synthesis parameter from previous research work [11] have been followed to study the effect of nitrogen functionality on the PSAC. Raw PSAC and NaOH impregnated PSAC (NAOH-PSAC) were used to compare the adsorption capacity of H2S.

Materials and Methods

Materials
A commercial palm shell based activated carbon (KD Carbon Sdn. Bhd., Malaysia) was used in this study. The raw sample is designated as PSAC. In the NaOH impregnation step, NaOH pellet (Merck, ≥97.0%) was used. In the urea impregnation step, nitric acid (HNO_3) (Merck, 65%), urea (Merck, 99%), ethanol (Merck, 96%) and purified nitrogen (N_2) gas (99.99% N_2) used for purging was supplied by Linde (M) Sdn Bhd. In the H_2S adsorption test, 99.99% N_2 and 1% H_2S in N_2 supplied by Linde (M) Sdn. Bhd. were used. The modified PSAC samples were referred as NaoH-PSAC and N-PSAC, in which both were impregnated using NaOH and urea, respectively.

PSAC Impregnation
NAOH-PSAC. The sample was prepared by mixing 100 mL of 1wt% NaOH solution with 5 g of raw PSAC. The solution was stirred at 180 rpm, 25°C for 3 h. Then, the mixture was heated at 100°C with constant stirring until all liquid evaporated. The NaOH-PSAC was then dried in an oven at 120°C for 6h [9].

N-PSAC. PSAC was pre-oxidized using 250 mL of 50wt% HNO_3 for 4 h. Then, it was rinsed with distilled water to remove excess acid and oxidized water-soluble products prior to nitrogen functionalization. Nitrogen species was introduced to the pre-oxidized PSAC via urea impregnation. The solution containing urea (20 g), oxidized carbon (30 g) and ethanol (100 mL) was stirred (180 rpm) at room

temperature for 5 h. Then, the mixture was boiled to evaporate the alcohol, before continuing with the drying of the impregnated sample at 110 °C [11]. The impregnated sample was then heated using MW under 500 ml/min of N_2 flow at 950 °C for 0.5 h. The N-PSAC was washed with boiling water to remove excess of urea product and was dried in an oven at 120 °C for 6 h.

H₂S Adsorption Study

A custom designed H_2S scrubber rig was used to test the performance of the adsorbent samples prepared. A 1.0 g of the adsorbent samples (size: 0.5 – 1 mm) was weighed and placed in the middle of stainless-steel column (length 450 mm, internal diameter 12.5 mm), supported with approximately 0.5 g of borosilicate glass wool. In this work, a simulated biogas mixture containing 59.7% of CH_4, 40% of CO_2 and 3000 ppm of H_2S was passed through the column under a fixed flow rate of 300 ml/min. The biogas composition used was simulated to mimic the actual composition of digestion pond located in Felda Besout Palm Oill Mill, Malaysia. The simulated biogas composition was adjusted by controlling the gas flow rate by using Aalborg AFC26 mass flow controllers. To achieve 25% of biogas relative humidity, CH_4 and CO_2 were passed through the humidification system first before entering the column. H_2S was not passed through the humidification system due to it is soluble in water [10]. The outlet concentration of H_2S was monitored by using a continuous biogas analyzer (IMR500) via electrochemical sensor calibrated for 0-5000 ppm of H_2S range. To ensure the safety of the workstation, the ventilation system of the rig was installed in case there is a leakage of hazardous H_2S gas.

pH of the Carbon Surface

The value of acidity/basicity of the adsorbent samples were tested. Each of 0.4 g of dry adsorbent sample was added to 20 mL of distilled water and the suspension was stirred overnight to reach equilibrium. Then, the sample was filtered, and the pH of the solution was measured by using pH meter (Eutech Instruments pH700).

CHNS/O Analysis

The elemental analysis was performed using a Perkin Elmer 2400 CHNS Analyzer to analyze the content of carbon, hydrogen, nitrogen, sulfur and oxygen in the sample.

N₂ Adsorption/Desorption

Nitrogen adsorption/desorption was carried out using Micromeritics ASAP 2020 surface area and porosity analyzer. Before the experiment, the samples were heated at 120 °C and degassed to constant vacuum (10^{-4} kPa) for 5 hours. Nitrogen isotherms were used to calculate the specific surface area (S_{BET}), micropore volume (V_{mic}), and total pore volume (V_t).

FESEM

The analysis of Field-emission Scanning Electron Microscopy (FESEM) was carried out using Quanta FEG 450 at accelerating voltage of 3kV, to obtain the sorbent surface morphologies.

FTIR

Surface functional groups of the samples were analyzed using Fourier-transform infrared spectroscopy (FTIR). The analysis was carried out using Nicolet 6700 and the adsorbents samples were analyzed in the range of 400–4000 cm^{-1} spectrum.

Results and Discussion

H₂S Adsorption

In this study, palm shell activated carbon that undergoes nitrogen modification (N-PSAC) was compared with raw palm shell activated carbon (PSAC) and NaOH impregnation palm shell activated carbon (PSAC-N). The performance of each carbon sample was analyzed using H_2S breakthrough curve, H_2S adsorption capacity, and the length of mass transfer zone (L_{MTZ}). Figure 1 shows the H_2S breakthrough curve for PSAC, NAOH-PSAC and N-PSAC adsorbents, which emphasized on the fixed bed adsorption type. It was observed that the breakthrough curve for both NaOH-PSAC and N-PSAC showed a longer trend compared to PSAC.
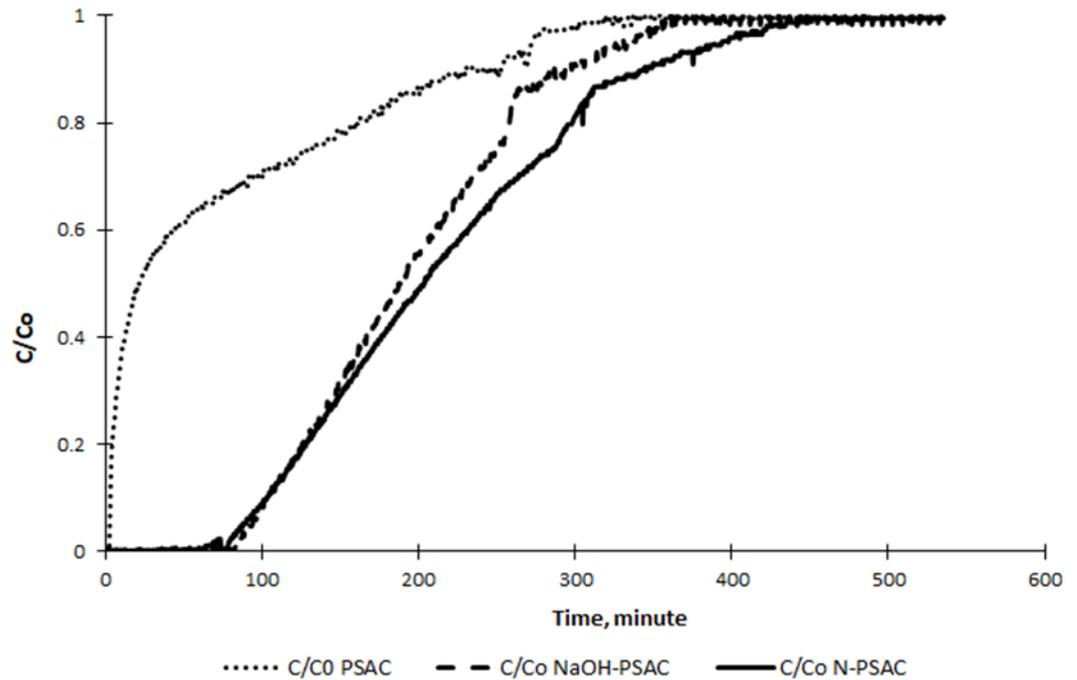
Figure 1. Breakthrough curve for raw and modified palm shell activated carbons.

Based on the breakthrough curve, the adsorption capacity for each carbon sample was calculated, where the breakthrough capacity was calculated at breakthrough time (where C/Co = 0.05) and the total adsorption capacity was calculated after the adsorbent reach equilibrium or saturated (where C/Co = 0.95). The H_2S breakthrough-curve and characteristics of all adsorbent samples are tabulated in Table 1. As expected, for NAOH-PSAC and N-PSAC, a significant increase in the adsorption capacity was found compared to PSAC. The highest H_2S breakthrough capacity was obtained using NAOH-PSAC followed by N-PSAC. However, the total H_2S adsorption capacity exhibited a reversed outcome, where the N-PSAC was capable to adsorb more H_2S with the total adsorption capacity of 264.29 mg/g, which is slightly higher than the NAOH-PSAC (240.87 mg/g).

The length of mass transfer zone (L_{MTZ}) has been studied to find out the active part of the bed where the adsorption is actually takes place. Mass transfer zone (MTZ) is the part in breakthrough curves that display a gradient in adsorbent concentration from zero to equilibrium. As the saturated part of the bed increases, the MTZ travels downstream and eventually exits the bed. The L_{MTZ} is calculated as in equation (1) [17]:

$$L_{MTZ} = \frac{L(t_{0.05} - t_{0.95})}{(t_{0.05} + t_{0.95})/2} \tag{1}$$

Where L is length of fix bed (cm), $t_{0.05}$ is breakthrough time at C/C$_o$ = 0.05 (s), and $t_{0.95}$ is saturated time at C/C$_o$ = 0.95 (s).

Table 1. H_2S breakthrough-curve and characteristics of raw and modified PSAC adsorbents.

Activated carbon	Breakthrough time, $t_{0.05}$ (min)	Saturated time, $t_{0.95}$ (min)	H_2S breakthrough capacity (mg/g)	Total H_2S adsorption capacity (mg/g)	Length of MTZ, L_{MTZ} (cm)
PSAC	5.0	312.0	6.16	91.28	0.97
NAOH-PSAC	93.5	388.0	115.27	240.87	0.61
N-PSAC	87.0	423.5	107.25	264.29	0.66

From Table 1, NAOH-PSAC shows better dynamic adsorption performances due to longer breakthrough

time, prolonged saturated time, and relatively shorter L_{MTZ} [9] compared to the PSAC and N-PSAC. Thus, this results in higher adsorption capacity. Competitively, N-PSAC also developed a very short L_{MTZ} (0.66 cm) where it took a little bit longer than the L_{MTZ} of NAOH-PSAC (0.61cm). This explains the difference in H_2S breakthrough capacity for both NAOH-PSAC and N-PSAC. Again, both the L_{MTZ} and adsorption capacities show that the N-PSAC is comparable to NAOH-PSAC. To understand better, details characteristics study was done in this work, to identify the factors that influence the H_2S adsorption by using these adsorbent samples.

N₂ Adsorption Desorption

Even though N-PSAC and NAOH-PSAC could adsorb H_2S very well compared to PSAC, the surface characteristics shown in Table 2 for these two adsorbent samples give divergence results. Increase of pH and surface area was found in NAOH-PSAC while decrease of pH and surface area was found in N-PSAC. As for NAOH-PSAC, with strong basic surface (pH 9.43) due to the presence of NaOH could easily enhance the oxidation of acidic H_2S to sulfur [10]. Under this circumstance, existing chemical reaction between H_2S and NaOH results in high adsorption capacity of H_2S, where the breakthrough capacity per unit pore volume calculated was 217.49 mg/cm³. Higher in surface area of NAOH-PSAC also provides better space to store H_2S, thus enhance the H_2S adsorption capacity. As reported in Adib et al. [18], the pH value for nitrogen-modified activated carbon is typically within pH 6 – 8. In this study, the pH value obtained for N-PSAC is pH 7.02, where N-PSAC's condition is less basic compared to NAOH-PSAC. However, the pH value for N-PSAC is still in agreeable range. The existence of basicity factor in N-PSAC, altogether with other factors such as surface area and pore volume offers a great help for oxidation of H_2S to happen.

Table 2. Physical and surface properties of raw and modified palm shell activated carbons.

Sample	pH @ 27°C	Surface area, S_{BET} (m²/g)	Micropore volume, V_{mic} (cm³/g)	Total pore volume, V_t (cm³/g)	Micropore surface area, S_{mic} (m²/g)	Total surface area, S_t (m²/g)	Breakthrough capacity per unit pore volume (mg/cm³)
PSAC	8.48	947.32	0.40	0.51	827.84	957.00	12.07
NAOH-PSAC	9.43	1085.97	0.39	0.53	798.90	1107.55	217.49
N-PSAC	7.02	886.75	0.30	0.47	611.27	896.30	228.19

As suggested by Bagreev and Bandosz, high H_2S adsorption of NAOH-PSAC is basically caused by higher surface area, the effect of surface functional group, and the effect of chemical reaction [19]. Even though the surface properties of N-PSAC shown opposite results compared to NAOH-PSAC, the adsorption capacity of N-PSAC is comparable to NAOH-PSAC (an established good alkaline impregnation agent). The reason is due to the changes of porosity, especially small micropore size (Figure 2) and surface chemistry of N-PSAC via introduction of nitrogen functionality on N-PSAC. From Table 2, it is clearly seen that after undergoes thermal treatment via MW at 950°C, there is some reduction in the physical and surface characteristic of N-PSAC compared to PSAC. This trend agrees with previous studies where the impregnation of urea had been done via conventional thermal treatment [20]. A clearer view of pore sizes and their contribution to porosity has been illustrated in Figure 2, where the pore width for PSAC is in the range of 30 – 40 Å and the pore width for both N-PSAC and NAOH-PSAC are in the range of 10 – 30 Å.

Seredych et al. [20] indicated that adsorbent samples that have small micropores are more effective. Smaller pores could immobilize H_2S gas inside the pores whilst avoiding the gas from escaping out of the adsorbent sample, given more binding time for H_2S to react with available functional groups. H_2S is a small molecule, thus small pores are more favorable for its physical adsorption due to increase the adsorption potential via trapping the gas molecule into small pores. Thus, compared to PSAC, shrinkage of pores width in the N-PSAC provides better structure where it could trap and adsorbs more H_2S molecules. Adib et al. [18] reported that thermal treatment at high temperature (950 °C) causes the carbon grains to shrink 30% of original size because of the increase in the adsorption energy characteristics. Thus, this explains why the surface area of N-PSAC is decreased by 10% compared to the PSAC. The shrinkage of carbon grains also affected the N-PSAC's pore width, where it decreases significantly. In this study, the breakthrough adsorption capacity has been divided by total pore volume, to calculate the normalized capacity, as shown in Table 2. The results show that the N-PSAC has higher normalized capacity followed by NAOH-PSAC and PSAC. This might be the reason why the total adsorption capacity of NAOH-PSAC is slightly lower than N-PSAC (Table 1).
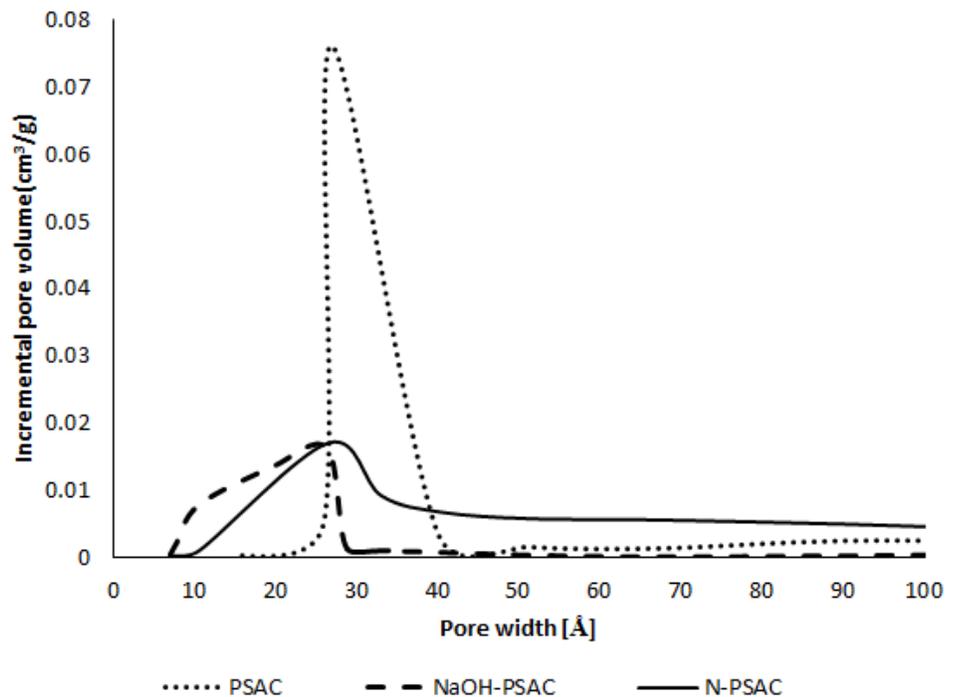
Figure 2. Pore size distribution (DFT) FTIR spectrum of PSAC, NaOH-PSAC, N-PSAC

Surface Functional Groups and Elemental Analysis

Figure 3 illustrates the FTIR spectra for all adsorbent samples. The PSAC spectrum show significant bands at 800 – 1600 cm^{-1} (C-H bond, C-O-C functional groups), 2800 – 3200 cm^{-1} (-COOH functional groups), and 3600 – 4000 cm^{-1} (O-H stretching vibration) wavelengths [21]. The bands of the N-PSAC were observed at 1200 cm^{-1} (C – N stretching vibration), 1400 – 1600 cm^{-1} (C – N – vibrations in secondary aromatic amines and N=N arrangement), and 3600 – 4000 cm^{-1} (O-H or N-H stretching vibration). The modification of N-PSAC removed some surface oxygen-containing groups and resulted in the incorporation of nitrogen to the carbon structure. As for the NaOH-PSAC, some functional groups were removed, and new bands were identified. The new bands are observed at 1200 – 1400 cm^{-1} (C=O, carbonyl compound), 2800 – 3000 and 3600 - 4000 cm^{-1} (O-H stretching bond). This confirmed the presence of a large amount of O-H functional groups on the surface of NaOH-PSAC, which resulted in high adsorption of H$_2$S. According to Wang et al., the active -OH functional groups produced by the NaOH impregnation played a substantial role in increasing the adsorption performance of H$_2$S [9]. Therefore, this can be concluded that the performance of these adsorbent samples can be related with the functional groups that were crafted on the surface of the adsorbents.

The discussion of functional groups can be supported with the elemental analysis presented in Table 3. From the table, a significant amount of nitrogen was found in the N-PSAC. The existence of nitrogen element in N-PSAC is due to most of nitrogen elements are easily incorporated into the carbon matrix as a component of aromatic ring at high temperature [11]. This finding is supported by Adib et al. [18], where the adsorption capacity of H$_2$S for adsorbent sample that undergo low temperature (450 °C) was not comparable to the adsorption capacity of H$_2$S for adsorbent sample that undergo high temperature (950 °C) treatment. Hence, the use of 950 °C treatment for N-PSAC helps in binding the nitrogen element in the carbon matrix. Apart from the effect of high temperature treatment, the PSAC's high surface area (947.32 m^2/g) is also one of the factors that contribute to the accessibility of nitrogen to be incorporated into the crystallite edges. This increased the carbon content accompanied by the decrease in hydrogen content for the N-PSAC compared to the PSAC. The changed in the elemental composition happened due to the degree of carbonization itself that has been changed, since it undergoes high temperature treatment twice (the first one is before impregnation). The existence of nitrogen functionality in the N-PSAC assists in the oxidation of H$_2$S where the chemical reaction will take place during the immobilization of H$_2$S because of its smaller pores. This circumstance justifies the higher value of total adsorption capacity of N-PSAC compared to NAOH-PSAC. Apart from that, in the NaOH-PSAC a significant oxygen composition was observed, which supported the finding from FTIR's spectrum discussed earlier.
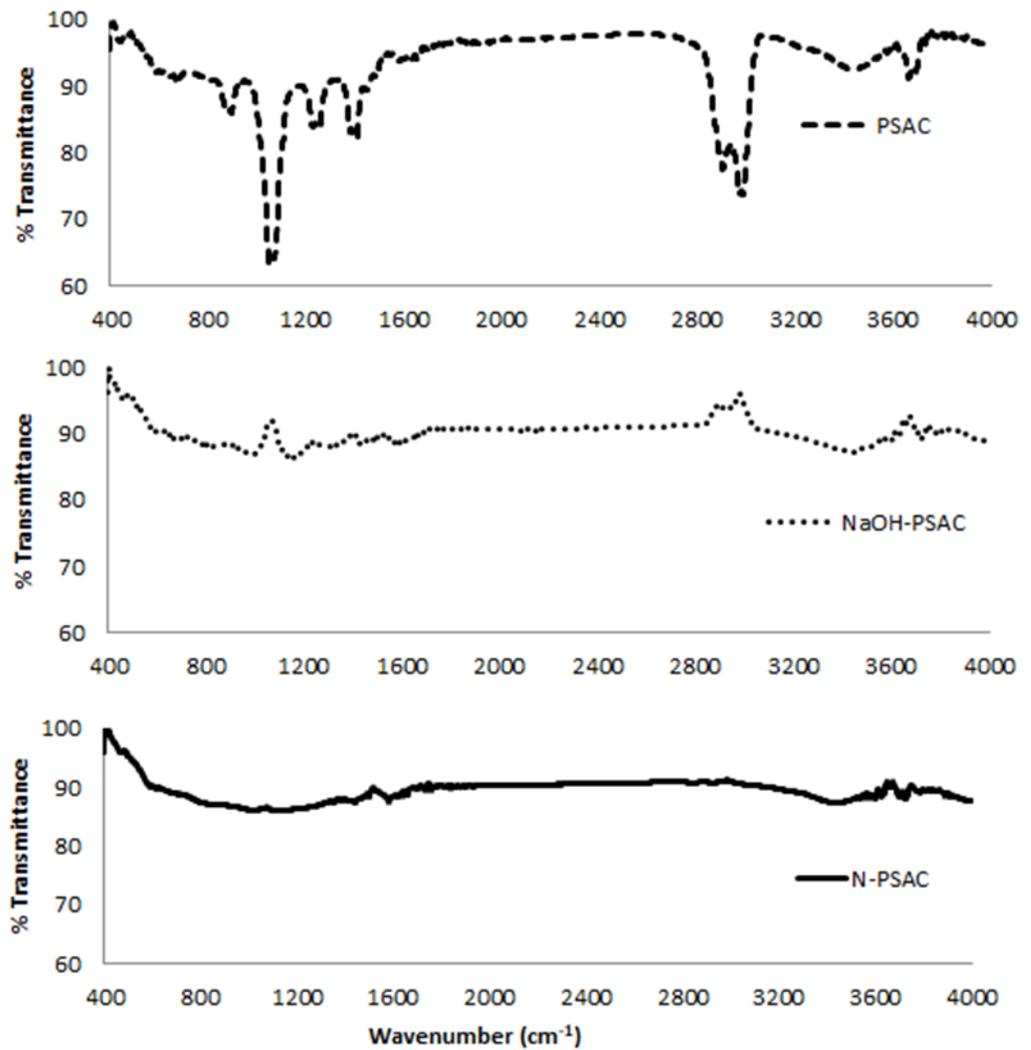
Figure 3. FTIR spectrum of PSAC, NaOH-PSAC, N-PSAC

Table 3. Elemental analysis of raw and modified PSAC adsorbents (before and after H$_2$S adsorption)

Element	%C	%H	%N	%S	O%
PSAC	61.61	1.48	-	-	36.99
Spent PSAC	62.27	0.59	-	6.47	30.67
NAOH-PSAC	59.98	0.45	-	-	39.57
Spent NAOH-PSAC	62.44	0.14	-	9.39	28.03
N-PSAC	79.92	0.90	0.49	-	18.69
Spent N-PSAC	61.71	0.76	0.32	13.49	23.72

Surface Morphologies

Figure 4 shows the images of before and after H_2S adsorption of all adsorbent samples that were characterized using FESEM analysis at magnification of 5000X. The elemental sulfur was detected by the EDX analysis that is illustrated in Figure 5. It can be clearly seen that the elemental sulfur has been deposited on the surface of all spent adsorbent samples in Figure 5. From the images, as for the PSAC, the micropores seem has been clogged by char and were not well developed, thus this resulted in low adsorption capacity of H_2S. On the other hand, for both NAOH-PSAC and N-PSAC, the surfaces of the adsorbent samples were showing the existence of NaOH and urea respectively as detected by EDX analysis. Thus, this suggests that both of NAOH-PSAC and N-PSAC undergo chemical adsorption during H_2S adsorption process. Based on the FESEM images, conventional thermal method (PSAC) and MW irradiation heating method (N-PSAC) have an identical effect onto the inner structure and the morphology of active carbon. However, by using MW heating method could change drastically the amorphous active carbon, where the cavities were intensified, and the inner surface of the sample are significant. In this study, N-PSAC developed a good amorphous active site where it formed narrow pores size since the broken carbon surface had a minimal expansion. Thus, this indicated that the production of N-PSAC via MW irradiation method could enhance the adsorption of H_2S, and the results will be greater if certain parameters such as MW temperature, flow rate of nitrogen gas, and holding time during MW heating method were optimized.
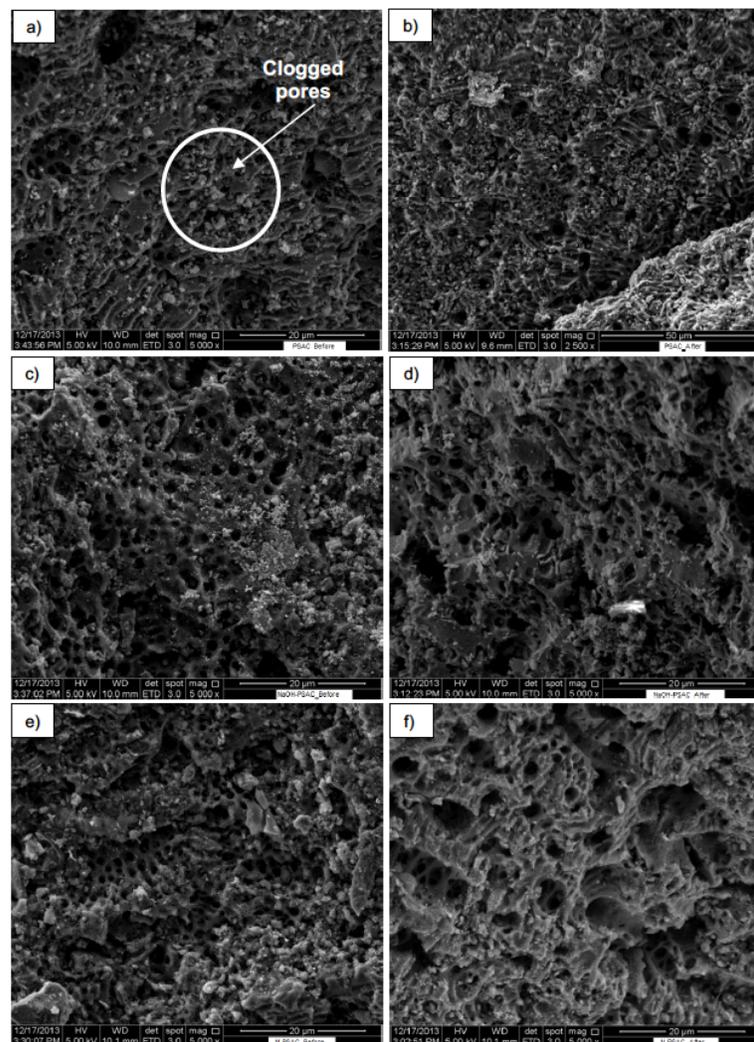
Figure 4. FESEM images of raw and modified palm shell activated carbons before and after H_2S adsorption at magnification of 5000x: a) PSAC_before, b) PSAC_after, c) NaOH-PSAC_before, d) NAOH-PSAC_after, e) N-PSAC_before, and f) N-PSAC_after
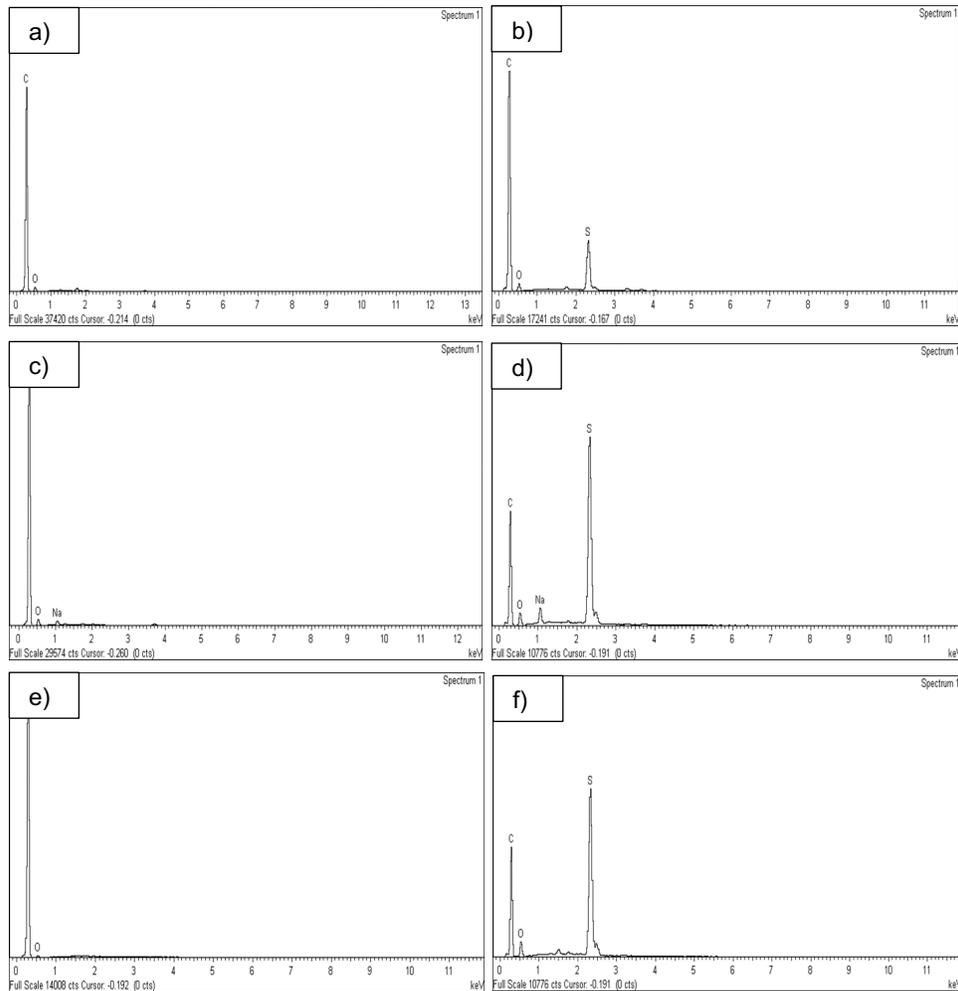
Figure 5. EDX spectrum of raw raw and modified palm shell activated carbons before and after H₂S adsorption at magnification of 5000x; a) PSAC_before, b) PSAC_after, c) NaOH-PSAC_before, d) NAOH-PSAC_after, e) N-PSAC_before, and f) N-PSAC_after.

Conclusions

The results presented in this study clearly showed the significant contribution of nitrogen in palm shell activated carbon (PSAC) impregnated using urea via MW irradiation for H2S adsorption. There are various factors that affecting the performance of adsorbent for H2S adsorption such as type of impregnation agent, surface chemistry and porosity. In fact, if the parameters of MW irradiation for N-PSAC have been optimized, it will have a better chance to become a better adsorbent than NAOH-PSAC which is well known as a good adsorbent. Based on the H2S breakthrough capacity, the performance of both NAOH-PSAC and N-PSAC is 100% better than PSAC. This resulted from the development of physical and chemical characteristics of the adsorbents such as high surface area, smaller micropore size, and availability of oxygen and nitrogen surface functional groups. Thus, the impregnation of urea could be a benchmark to further study the nitrogen functionality in palm shell activated carbon. The unique features of N-PSAC show important improvement in the application of H2S adsorption. This study will provide great momentum to develop an effective method via MW irradiation in producing a great adsorbent.

Conflicts of Interest

The authors declare that there is no conflict of interest regarding the publication of this paper.

Acknowledgment

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