

ADSORPTION OF HYDROGEN SULFIDE USING PALM SHELL ACTIVATED CARBON: AN OPTIMIZATION STUDY USING STATISTICAL ANALYSIS

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Abstract

Removing H_2S from biogas that is produced from anaerobic digestion of palm oil mill effluent is a crucial step in order for the biogas to be utilized as a source of energy. In this study, palm shell activated carbon (PSAC) prepared by steam activation was used to adsorb H_2S from simulated biogas. The parameters studied were H_2S concentration, adsorption temperature and space velocity. The effect of these parameters towards breakthrough adsorption capacity was studied using statistical analysis with Design Expert Software. H_2S concentration and space velocity were found to be significant in affecting the breakthrough adsorption capacity. Adsorption temperature on its own was found not to have significant effect on the breakthrough adsorption capacity but its interaction with other parameters was found to be significant. Characterization of fresh and spent PSAC confirmed and provided further information on the adsorption of sulfur species on PSAC pore surface.

Keywords: Activated carbon; Biogas; Hydrogen sulfide; Adsorption

1. INTRODUCTION

Palm oil mill effluent (POME) is the pollutant generated from the oil palm industries in Malaysia and must be treated before discharge. One of the possible way to treat POME is by subjecting it to anaerobic digestion that produces biogas. Biogas can be combusted directly as fuel. Ng et al. reported that 1,688 million m^3 of biogas is generated from palm oil mill in 2009 [1]. Biogas concentration largely varies from 13.5–49.0% methane (CH_4) and the remaining carbon dioxide (CO_2) [2]. Typically, trace amount of hydrogen sulfide (H_2S) between 0–1% is also produced in biogas as a result from the anaerobic digestion of POME [3]. Biogas should not be emitted directly to the atmosphere because H_2S is corrosive and CH_4 is a greenhouse gas. Instead, biogas can be utilized as a source of energy by combusting biogas in a gas engine to generate electricity or steam. However, H_2S must be removed prior to combustion or it can cause severe corrosion [4]. For biogas scrubbing technology, H_2S adsorption with activated carbon appears to be a promising method. Activated carbon is a cheap adsorbent that is widely utilized in air pollution abatement technology due to favorable surface properties that allows it to have high adsorption capacity and fast reaction kinetic even at ambient temperature. Activated carbon can be produced from various lignocellulosic biomass such as coconut shell, corncob and palm shell. The use of palm shell to synthesize activated carbon for H_2S removal is more economical because both palm shell and H_2S are by-products

from the oil palm industries. Thus, the use of palm shell activated carbon (PSAC) is a sustainable process because a waste from the oil palm industry is utilized to remove acidic gas generated within the same industry. PSAC is conventionally prepared using a two-step process; carbonization and activation. During carbonization, volatile compound in palm shell will be removed in the inert atmosphere. Carbon is retained due to the absence of oxygen that avoids the formation of carbon dioxide. The product after carbonization process is palm shell char that contains mostly carbon material with a surface area of about $100m^2/g$. After that, the char will be activated using steam or carbon dioxide. Thermal treatment using steam or carbon dioxide will distort the structure of char to form porous carbon that has high surface area and good adsorption capability.

In this research, several reaction parameters; H_2S concentration, adsorption temperature and space velocity that affect breakthrough adsorption capacity using steam activated PSAC were studied. These parameters were studied using statistical analysis by employing Software Design Expert 6.0. Design of experiment is generated by the software in order to minimize the amount of experiments required to study the parameters thoroughly. In addition, the importance of each parameter in affecting activated carbon adsorption capacity can be identified. Interaction between reaction parameters can

be also analyzed so that detailed understanding between H2S adsorption capacity and reaction parameters can be obtained.

2. EXPERIMENTAL

2.1 Chemicals

Steam activated PSAC used in this study was obtained from Victory Element SdnBhd, Malaysia. The mesh size of the PSAC was 8 x 12. In the adsorption test, PSAC was used as received without any further treatment. Three types of gases (99.99% CH4, 99.99% CO2 and 1% H2S, balance CH4) in cylinders supplied by WellgasSdnBhd, Malaysia were used to simulate industrial biogas for the adsorption test.

2.2 Adsorption Test

H2S adsorption test was carried out using a packed bed reactor test rig as shown in Figure 1. The composition of simulated biogas was adjusted by controlling the flow rate of gases using Aalborg AFC26 mass flow controllers. CO2 and CH4 were passed through a humidification system to give approximately 25% relative humidity to the biogas inlet stream. H2S/CH4 was not passed through the humidification system because H2S is soluble in water. The path of the biogas after the humidification system to the reactor is insulated to avoid condensation of moisture. The diameter of the stainless steel tubular reactor used is half inch. The flow rate of the biogas was fixed at 450 mL/min with 50% CO2, 1000–5000 ppm H2S and balance CH4. This biogas composition was simulated to mimic the data obtained from a collection pond located at FeldaBesout Palm Oil Mill. The height of PSAC was adjusted corresponding to space velocity between 3375 to 13500 h-1 and placed in the middle of the tubular reactor and supported with approximately 0.05g glass wool. Adsorption temperature was controlled using a Linberg/Blue M tube furnace. Concentration of H2S at the outlet stream was analyzed using an IMR 6000 gas analyzer via electrochemical sensor calibrated for 0–5000 ppm H2S. The workspace of the test rig was ventilated as a safety procedure just in case there is a leakage of hazardous H2S.

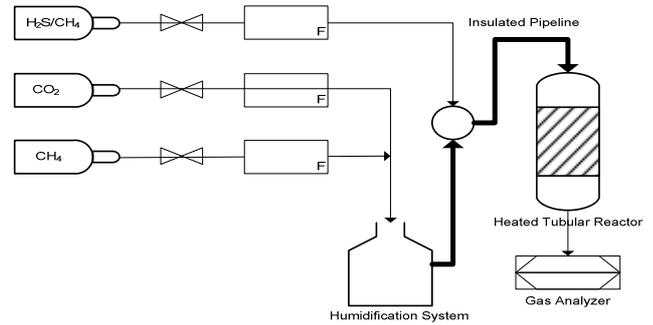


Figure1. H2S adsorption test rig.

2.3 Characterization

Several characterizations such as N2 adsorption-desorption, scanning electron microscopy–energy dispersive X-ray spectroscopy (SEM–EDX), thermogravimetric analysis (TGA) and elemental analysis were performed on fresh PSAC and spent PSAC to analyze its surface properties, functional groups and morphologies/composition. N2 adsorption-desorption was carried out with an Micromeritics ASAP 2020 Surface Area and Porosity Analyzer. Prior to analysis, the samples were degassed for 5 hours. SEM–EDX analysis was carried out by a Quanta FEG 450 at accelerating voltage of 3kV in order to obtain the sorbent surface morphologies and determine localized chemical composition. TGA was performed using a Perkin Elmer TGA7 with a heating rate of 100C/min until 6000C under a 100mL/min flow of purified air [5]. Elemental analysis was performed using a Perkin Elmer 2400 CHNS Analyzer to analyze the sulfur content of the sample.

3. RESULTS AND DISCUSSION

3.1 Development and analysis of regression model

In this study, adsorption capacity of activated carbon was determined at breakthrough point, in which the ratio of the outlet to inlet H2S concentration at 0.05. The adsorption experimental run was conducted based on the design of experiment and the complete design matrix with results are shown in Table 1.

Table1. Design Matrix and Results for Adsorption Capacity At Different Experimental Conditions

Run Order	x1: H2S concentration (ppm)	x2: Temperature (oC)	x3: Space velocity (h-1)	y: Adsorption capacity (mg/g)
R1	3000	50	3375	28.9
R2	5000	30	3375	30.3
R3	3000	50	5400	21.8
R4	3000	50	5400	21.0

R5	1000	50	5400	93.6
R6	3000	50	5400	23.0
R7	3000	70	5400	18.4
R8	3000	50	5400	24.5
R9	3000	50	5400	23.0
R10	3000	50	5400	22.6
R11	5000	50	5400	16.6
R12	5000	70	3375	23.9
R13	1000	70	13500	5.1
R14	1000	70	3375	38.1
R15	3000	30	5400	19.1
R16	5000	30	13500	1.6
R17	1000	30	3375	116.5
R18	1000	30	13500	26.1
R19	5000	70	13500	3.2
R20	3000	50	13500	3.8

Six center points (R3, R4, R6, R8, R9, and R10) are incorporated and spread out in the experimental matrix in order to calculate experimental error and reduce other errors due to unaccounted parameters. Experimental error was calculated to be 4.8%. Using the results shown in Table 1, adsorption parameters (x1: H2S concentration, x2: adsorption temperature and x3: space velocity) were correlated with breakthrough adsorption capacity (y) using Design-Expert 6.0.6 software. The model selected by this software is Response Surface Reduced Cubic Model and the equation in coded factors is shown in Eq. (2). The graph for the predicted versus actual breakthrough adsorption capacity is shown in Figure 2.

$$\log_{10} y = 1.132 - 0.423x_1 + 0.005x_2 - 0.438x_3 + 0.228x_1^2 - 0.081x_2^2 - 0.111x_3^2 + 0.174x_1x_2 - 0.079x_1x_3 + 0.022x_2x_3 - 0.129x_1^2x_2 - 0.022x_1^2x_3 + 0.147x_1x_2^2 + 0.078x_1x_2x_3 \quad (2)$$

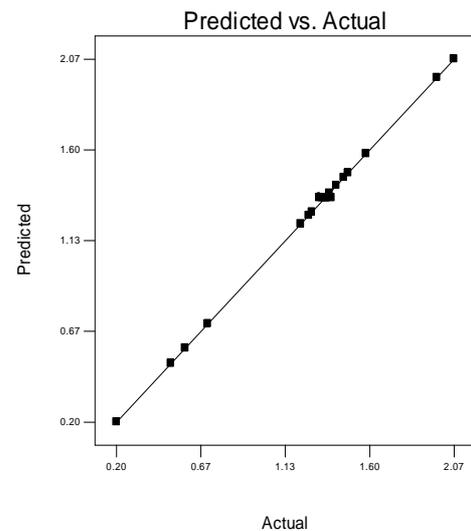


Figure 2. Predicted versus actual log breakthrough adsorption capacity.

The R2 value for Eq. (2) was found to be 0.9994 and the predicted R2 value of 0.9990 is in reasonable agreement with the adjusted R2 value of 0.9980. On the other hand, adequate precision of 109.0 indicated an adequate signal to noise ratio for the model to be used to navigate the design space. These results verify that the developed model can be used to predict the PSAC breakthrough adsorption capacity within the range of adsorption parameters studied.

An analysis of variance (ANOVA) that shows the significance of the model and each adsorption parameter is shown in Table 2. Value of Prob> F less than 0.05 indicates that model term is significant. Therefore, the model with Prob> F value less than 0.0001 implies that the model is significant. The model F-value is 717.7 and there is only a 0.01% chance that a model F-value this large could occur due to noise. The lack of fit F-value of 0.00 implies the lack of fit is not significant relative to the pure error. There is a 98.70% chance that a "Lack of Fit F-

value" this large could occur due to noise. Non-significant lack of fit indicates that the model fit the experimental result very well. From the ANOVA result, significant terms with high F value are x1, x3, and x1x2. Other significant terms with lower F values are not discussed in this paper because their effect on breakthrough adsorption capacity is less significant as compared to those with higher F values.

Table 2. Analysis of Variance of the Response Surface Reduced Cubic Model

Source	Sum of Squares	DF	Mean Square	F Value	Prob> F
Model	3.89	13	0.2996	717.7	< 0.0001
x1	0.32	1	0.3286	787.1	< 0.0001
x2	0.00	1	0.0000	0.1	0.7707
x3	0.41	1	0.4124	987.8	< 0.0001
x12	0.11	1	0.1165	279.1	< 0.0001
x22	0.01	1	0.0169	40.5	0.0007
x32	0.01	1	0.0115	27.7	0.0019
x1x2	0.24	1	0.2427	581.3	< 0.0001
x1x3	0.04	1	0.0498	119.3	< 0.0001
x2x3	0.00	1	0.0040	9.6	0.0210
x12x2	0.02	1	0.0249	59.6	0.0002
x12x3	0.00	1	0.0008	2.0	0.2053
x1x22	0.03	1	0.0324	77.6	0.0001
x1x2x3	0.04	1	0.0492	118.0	< 0.0001
Residual	0.00	6	0.0004	–	–
Lack of Fit	0.00	1	0.0000	0.0003	0.9870
Pure Error	0.0025	5	0.0005	–	–

3.2 Effect of Significant Terms

Significant term with highest F value is space velocity. Highest F value denotes that space velocity has the greatest effect towards breakthrough adsorption capacity among the adsorption parameters studied. From Table 1, it can be seen that experiments conducted with high space velocity (13500 h⁻¹) generally resulted to low breakthrough adsorption capacity. The major reason for this observation is related with the residence time of the biogas in the adsorption zone. At higher space velocity, residence time of biogas in the adsorption zone is shorter. Adsorption studies R13, R16, R19 and R20 that utilized 13500 h⁻¹ space velocity showed low breakthrough adsorption capacity, ranging only from 1.6–5.1 mg/g. However, although R18 was also conducted using 13500 h⁻¹ space velocity, but it recorded relatively high breakthrough

adsorption capacity of 26.1 mg/g that could be mostly attributed to the low H₂S concentration used (1000 ppm). When the space velocity was lowered to 5400 h⁻¹, residence time of the gas was increased and the PSAC breakthrough adsorption capacity rose significantly to the range 16.6–24.5 mg/g. Again, R5 that utilized 5400 h⁻¹ space velocity achieved a relatively high breakthrough adsorption capacity of 93.6 mg/g due to the low H₂S concentration used (1000 ppm). Further increase in breakthrough adsorption capacity ranging from 23.9–38.1 mg/g was obtained when using lower space velocity of 3375 h⁻¹. Once again, R17 (116.5 mg/g) is an exceptional case because of the low H₂S concentration (1000 ppm). The effect of low H₂S concentration will be discussed in the next section. Higher residence time increases the frequency of molecule collision that results to a successful adsorption process [6]. On the contrary, low residence time

resulted to insufficient time for all H₂S molecules to adsorb on the PSAC surface. This is especially more prominent when more surface sites are taken up as adsorption process progresses and the remaining H₂S molecules have to diffuse deeper into the pores of PSAC. This denotes a longer mean free path for the H₂S molecule to travel before it can be adsorbed on the PSAC pore surface. Thus, the finding on the significance of this term provides an insight towards the importance of mass transfer in the adsorption process.

Another significant term with a high F value of 787.1 is x₁ (H₂S concentration). H₂S concentration has antagonistic effect towards adsorption capacity, as indicated from a minus sign for the term x₁ in Eq. (2). From the previous discussion on space velocity, lower H₂S concentration resulted to a higher breakthrough adsorption capacity. Adsorption study using 1000 ppm H₂S concentration gives adsorption capacity in the range of 26.1-116.5 mg/g for run R13 which recorded merely 5.1 mg/g, due to the high space velocity used. When using higher H₂S concentration at 5000 ppm, breakthrough adsorption capacity within the range of 1.6 to 30.3 mg/g was obtained. At higher H₂S concentration, the diffusion of H₂S molecules into the pores of PSAC for adsorption to occur is not fast enough to facilitate complete removal of all H₂S molecules from the gaseous stream. Therefore, the breakthrough occurs faster, i.e., lower adsorption capacity. At lower H₂S concentration, there will be less H₂S molecules diffusing into the PSAC pore structure per unit time. Thus on the average, the H₂S molecule will have a shorter mean free path before reaching an active site due to lower competition with other molecules for adsorption. However, these findings contradict the finding reported by Xue and Liu [7]. Breakthrough capacity was found to increase with higher H₂S concentration. Nevertheless, it is worthwhile to note that the concentration used in their study was between 210-630 ppm, which is far lower than this study. At very low concentration region, driving force for the H₂S diffusion will increase with higher concentration and there will be no significant competition for active sites. Therefore, both studies are expected to have different results. In order to have a clearer view on the effect of H₂S concentration towards breakthrough adsorption capacity, diffusion coefficient and adsorption rate constant should be obtained in order to determine the rate limiting step of this adsorption process.

The effect of temperature (x₂) was also investigated in this study. It was found to have insignificant effect towards PSAC breakthrough adsorption capacity. However, this does not mean that temperature has no effect on breakthrough adsorption capacity, but merely denotes that there is no linear relationship between temperature and adsorption capacity. Based on R16 and R19, increasing temperature increases adsorption capacity. On the contrary, increasing temperature reduces adsorption capacity as observed from R14 and R17. On the other hand, R7, R8 and R15 show an optimum temperature appearing at 50°C. These three sets of

data were conducted at different concentration and space velocity, indicating that the effect of temperature on breakthrough adsorption capacity is dependent on other parameters. Temperature was found to have significant interaction effect with H₂S concentration on breakthrough adsorption capacity as indicated by the high F value for the term x₁x₂ (581.4). This significant interaction term could be used to explain the three sets of data mentioned earlier which also indicates that the effect of temperature toward breakthrough adsorption capacity is dependent on the level of H₂S concentration and space velocity. This finding is again different from those reported in the literature. Xiao et al. studied the effect of temperature and reported that higher temperature reduces adsorption capacity of activated carbon [8]. They explained that physical adsorption is an exothermic process that favors lower temperature.

The synergistic effect of x₁x₂ can be visualized in Figure 3. From Figure 3, increasing temperature at 1000 ppm H₂S and 3375 h⁻¹ space velocity resulted to a decrease in breakthrough adsorption capacity. However, when experiments were conducted at 5000 ppm H₂S and 3375 h⁻¹ space velocity, increasing temperature would slightly increase the breakthrough adsorption capacity. This phenomenon could be due to the extra kinetic energy obtained at higher temperature. This extra kinetic energy affects the diffusion of molecules in the pores and the bulk gas motion in the main stream. At lower H₂S concentration, the results from the previous section shows that high adsorption capacity can be easily achieved. If temperature is raised, H₂S molecules will gain extra kinetic energy and therefore reduces the residence time in the adsorbent zone. Shorter residence time does not allow all H₂S molecules to diffuse deep into the pores especially after most of the outer pores are saturated with H₂S. Thus, more H₂S molecules will pass through the adsorbent without being adsorbed at higher temperature and therefore gives a lower adsorption capacity. However, at higher H₂S concentration, breakthrough adsorption capacity is generally lower. Therefore, the mean free path for H₂S diffusion is still considerably low. Higher temperature will improve the diffusion of H₂S molecules deeper into the pores for adsorption to occur. Although higher temperature will also increase the bulk motion of H₂S molecules through the adsorbent zone, the positive effect of H₂S molecule diffusing deeper into the pores of the adsorbent will be more prominent. Thus the adsorption capacity will slightly increase at higher temperature.

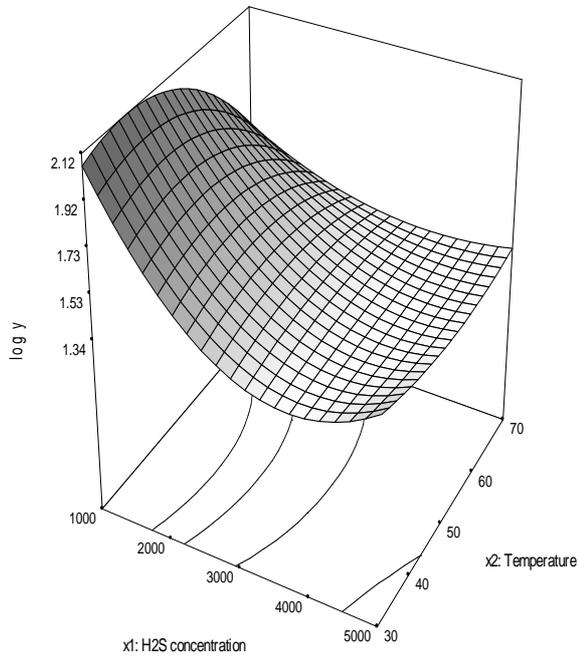


Figure 3. Surface graph of interaction between x1 x2 at x3 = 3375 h-1.

3.3 Optimization Study

Using Design Expert Software, optimization was performed in order to search for the highest breakthrough adsorption capacity within the range of parameters studied. Ten sets of solution with predicted breakthrough adsorption capacity were generated and tabulated in Table 3. Set Number 1 was identified as the optimum conditions for the adsorption process because it gave the highest predicted breakthrough adsorption capacity. The optimum operating parameters are 1006 ppm H2S, 43.60C and 3926 h-1 space velocity. Three identical experiments were performed using the predicted parameters and the breakthrough adsorption capacity obtained was 121.9-130.0mg/g, which verifies the prediction of Design Expert Software. Comparing the result from the optimization process with the Runs in Table 1, H2S concentration has to be kept as low as possible in order to achieve high breakthrough adsorption capacity. However, for practical operation, H2S concentration is generally not consistent and fluctuates significantly depending on the anaerobic digestion condition, unless the digestion is performed in a bioreactor. On the other hand, optimum temperature of 43.60C on the other hand is beneficial to actual plant operation because trapped biogas from POME anaerobic digestion generally range from 40–500C due to heating by sunlight. Although in the initial part of

this paper, the breakthrough adsorption capacity was found to increase with lower space velocity, but the optimization result shows that there is an optimum value for space velocity. Utilization of lower space velocity does not necessarily guarantee higher breakthrough adsorption capacity. Therefore, for practical application, optimized space velocity should be used in order to obtain higher breakthrough adsorption capacity of PSAC.

Table 3. Prediction of Optimum Adsorption Capacity at Different Conditions

Number	H2S concentration (ppm)	Temperature (0C)	Space Velocity (h-1)	adsorption capacity (mg/g)
1	1006	43.6	3926	122.18
2	1000	37.3	5544	109.90
3	1000	43.6	5792	103.27
4	1000	30.0	5427	101.62
6	1000	44.1	6198	97.72
6	1000	41.3	6729	94.84
7	1000	57.4	3375	85.11
8	1000	30.0	8698	68.54
9	1502	30.0	3375	66.98
10	1577	30.0	3375	62.37

3.4 Characterization of Fresh and Spent PSAC

The N2 adsorption desorption of fresh PSAC enables the derivation of adsorption and desorption isotherm. Langmuir correlation was found to fit the adsorption capacity of nitrogen at various pressures as shown in Figure 4. Other surface properties analysis were tabulated in Table 4. Since the t-plot micropore surface area is about 74.2% of BET surface area for fresh PSAC, it can be concluded that pore structure of PSAC is predominantly micropore (<2nm).

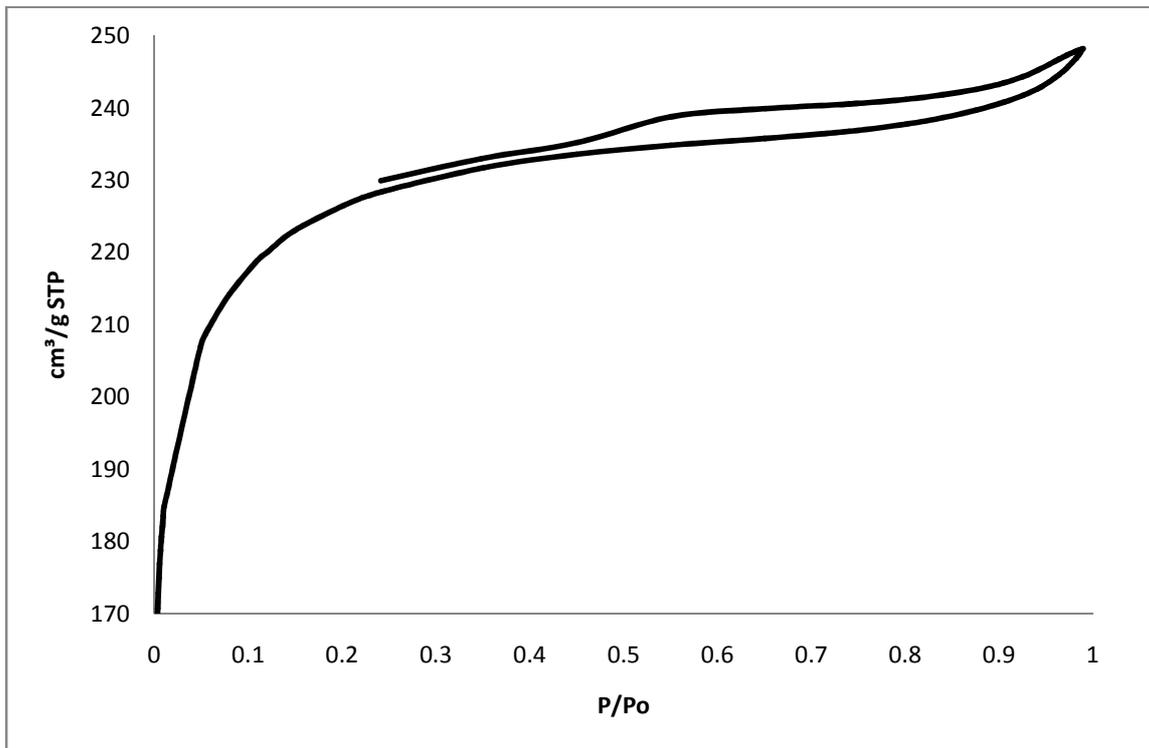


Figure4. N2 adsorption-desorption isotherm of fresh PSAC.

Table4. Characteristic of Fresh PSAC from N2 Adsorption Desorption

	Fresh PSAC
BET surface area (m ² /g)	736.8
Total pore volume (cm ³ /g)	0.382
t-plot micropore area (m ² /g)	546.9
t-plot external surface area (m ² /g)	189.9
Average pore width (Å)	20.8

The adsorption of H₂S and water molecules were determined from the result of Thermal Gravimetric Analysis (TGA). TGA was performed for both fresh and spent PSAC and the results are shown in Figure 5. The derivative form of TGA is also shown in Figure 6. The weight of fresh PSAC was reduced rapidly to 80% when temperature was increased to 1000C. After that, the weight change of fresh PSAC was not significant until 6000C. Spent PSAC, on the other hand, had a

weight reduction to 85% at 1000C, and the second cycle of weight change occurred between 200-4000C. In addition, total weight change of spent PSAC is larger than fresh PSAC within the temperature range of the analysis, denoting spent PSAC contained larger amount of adsorbed substances than fresh PSAC. As shown in Figure 6, fresh PSAC only have one peak formed. Spent PSAC shares the same peak with fresh PSAC but possess additional peak at 200-4000C. The first peak denotes to the desorption of water molecules. In the H₂S adsorption operation, moisture was added to the gas stream because it plays an important role in the adsorption process. Therefore, spent PSAC could contain up to 15 wt% of moisture, especially due to the long duration of the operation. The second peak of DTG for spent PSAC denotes the release of SO₂ because oxidative atmosphere was used in the analysis [5]. In this study, the peak that denotes to elemental sulfur was not detected at 450-4700C [5], therefore strengthening the claim that the adsorption of H₂S was mostly physisorption process. Apart from that, approximately 10% weight reduction in the 200-4000C region can be used to compare with the adsorption capacity of PSAC (122.2 mg/g). The value of the adsorption capacity showed that PSAC could adsorb about 12.22% H₂S. Therefore, if adsorbed H₂S were removed, weight reduction of PSAC will be 12.22%, which is similar with the results from TGA. This confirms the second peak as H₂S desorption.

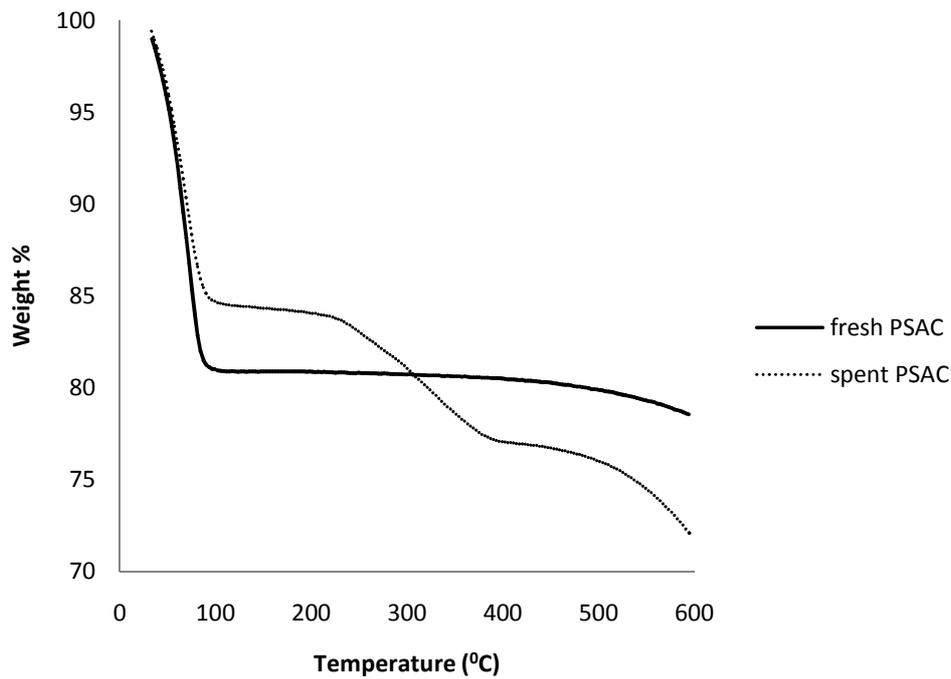


Figure 5. Weight % versus temperature.

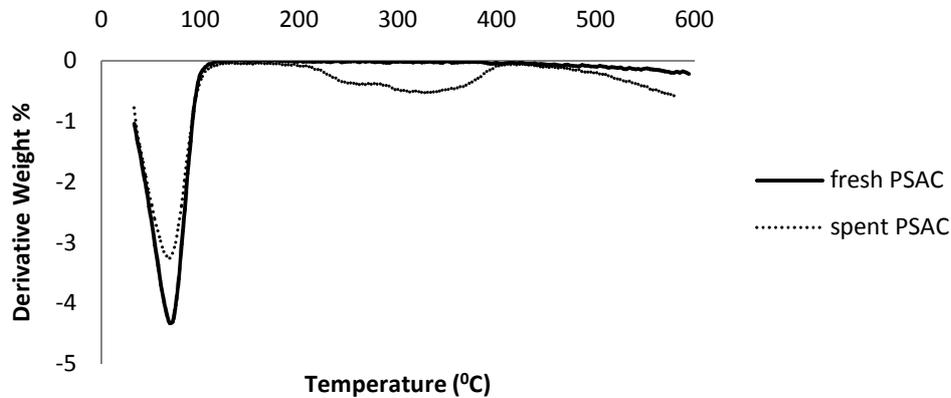


Figure 6. DTG curves of fresh and spent PSAC.

Figure 7 and 8 shows the SEM micrographs of fresh and spent PSAC, respectively at various magnifications. The surface of fresh PSAC was found to be cratered. The diameter of the craters is in the magnitude of 30µm. Further magnification on the crater shows a porous surface. The pores are generally between 1-2µm. This kind of porous structure is the main reason PSAC possesses high surface area, as determined from N₂ adsorption desorption analysis. Spent PSAC on the other hand, possess different structure with fresh PSAC. Bone-like structure was found and it is believed to be the product of H₂S

adsorption process. EDX result shows that it contains 3.35% sulfur, which agree well with a 2.94% sulfur detected in a CHNS elemental analyzer. Deposit of white agglomerates was also found in fresh and spent PSAC, as shown in Figure 9. The deposit was identified with EDX and it contains about 4% copper and trace amount of magnesium, potassium, calcium, silicon and iron. This could be the inert remains that did not burn off during the preparation of PSAC from palm shell. The effect of these inert remains towards H₂S adsorption was not studied.

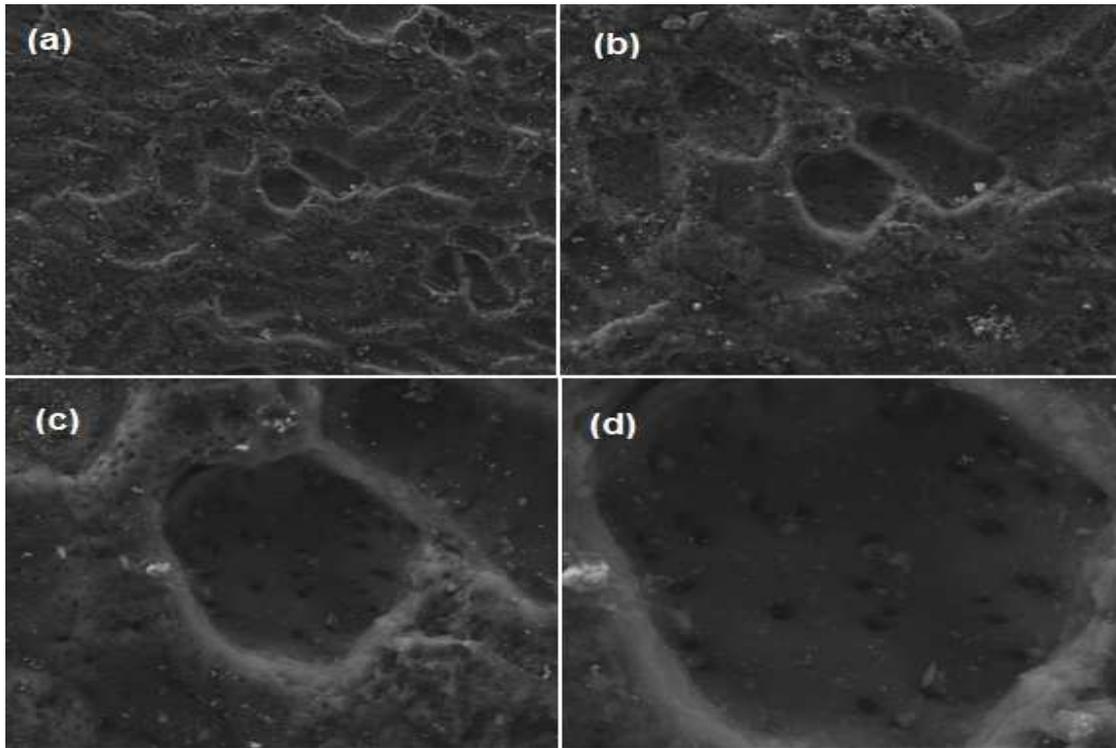


Figure7. SEM of fresh PSAC at various magnifications (a) 1000X, (b) 2000X, (c) 5000X and (d) 10000X.

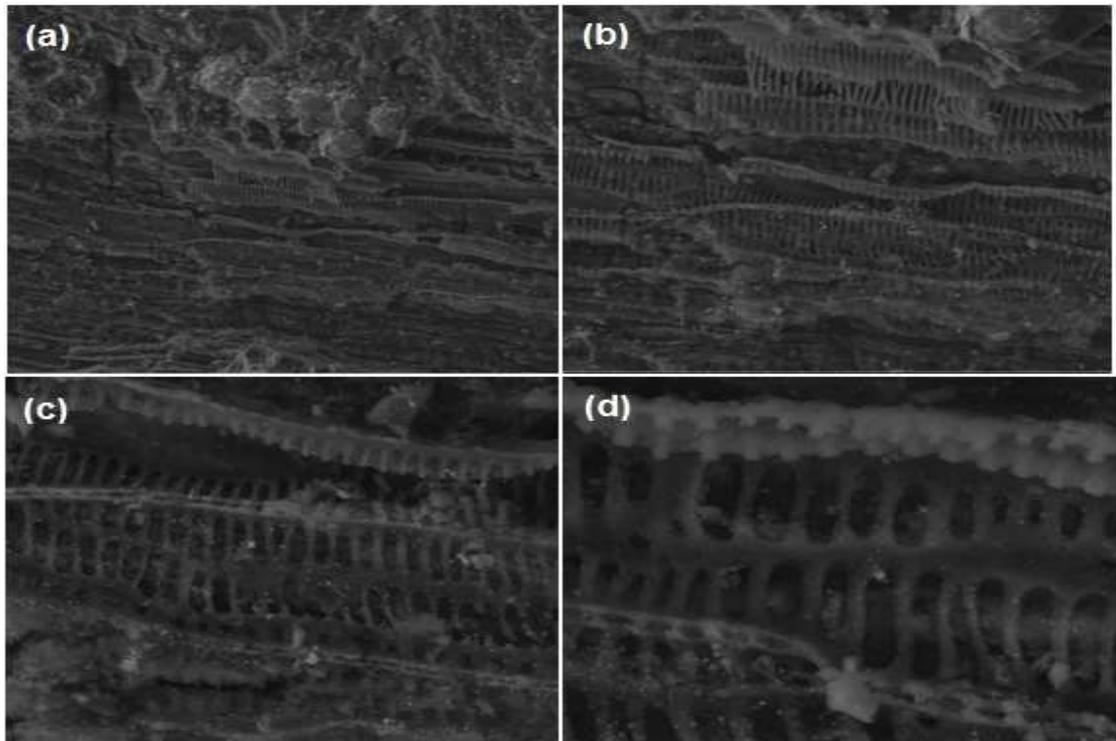


Figure8. SEM of spent PSAC at various magnifications, (a) 1000X, (b) 2000X, (c) 5000X, (d) 10000X.

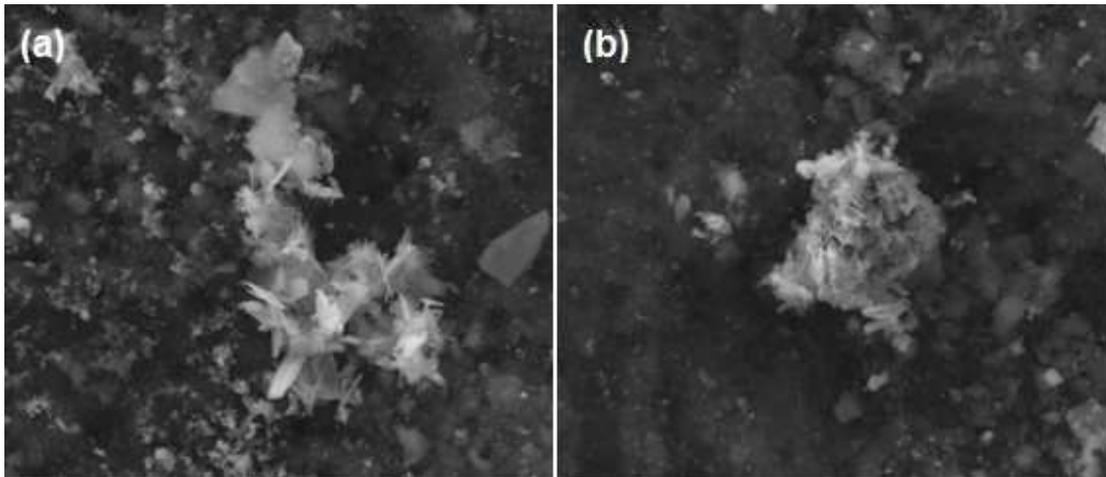


Figure 9. Magnification of agglomerates found on the spent PSAC surface.

CONCLUSIONS

H₂S adsorption has been successfully performed using palm shell activated carbon. In this study, reaction parameters such as H₂S concentration, adsorption temperature and space velocity were studied using a statistical analytical method via Design Expert Software. H₂S concentration and space velocity were found to be significant in affecting the adsorption capacity, indicated by the generated mathematical model. Interaction terms involving temperature were also found affecting the adsorption capacity. Characterization of fresh and spent PSAC confirm and provide further information on the adsorption of sulfur species on PSAC pore structure.

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