Chapter 4

Optimization and Characterization Study of Preparation Factors of Activated Carbon Derived from Coconut shell to Remove of H_2S from Wastewater

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ABSTRACT

The main point of this work is to investigate the preparations variables of activated carbon derived from coconut shell (CSAC) for removal of hydrogen sulfide (H_2S) from wastewater. The CSAC was chemically modified with potassium hydroxide (KOH). The central composite design (CCD) under response surface methodology (RSM) was employed to prepare the CSAC. The three preparation variables impact on the removal efficiency (%) of H_2S were examined. The preparation parameters to the responses were correlated by developing a quadratic model. The analysis of variance shows the significant impact of variable on each experimental design responses. The results show that the temperature of $857°C$, chemical impregnation ratio of 3.4 wt% and activation time of 66 min were the optimum conditions for CSAC preparation of with removal efficiency of 88.8%.

Key Words: Coconut shell, Activated carbon, wastewater, hydrogen sulfide, Response surface methodology.

1.0   INTRODUCTION

Hydrogen sulfide (H_2S) is a very toxic and dangerous gas. There are many sources that contributed to the emission of H_2S to the environment. The petroleum refinery is considered as the main source contributor of H_2S. It is produced during the processing of crude oil to useful product. The crude oil normally contains sulfur component and to remove the sulfur, process

such as desulfurization, liquefied petroleum gases and distillation units in used in the crude oil refinery. Therefore, these processing procedures will generate many wastes which are dissolved inevitably in the water to produce hazardous sour water which consist of H_2S. Hence, it is a must the water to be treated before being discharge for reuse or discard to swamp (Kazmierczak-Razna et al. 2015). There are many methods to removeH_2S from wastewater, such as biological, chemical and physiochemical (adsorption) methods (Siefers et al. 2010). Amongst these methods, adsorption is the most effective method due to its excellent performance and low cost (Foo and Hameed 2010).

Activated carbons (ACs) from agricultural wastes, such as date stones (Sekirifa et al. 2013), sugar canes (Castro et al. 2000), nut shell (Hu and Vansant 1995), coconut shell (Yang et al. 2010), and sunflower straw (Foo and Hameed 2011) are the most widely used to remove the different types of pollutants from wastewater. ACs have large adsorption capacity due to its great permeability and large surface area which is created during the activation of carbonaceous substances and carbonization. ACs are commonly used in industries as well. The two common forms of ACs are granular forms with sizes ranging from 0.5 to 2.5mm to be used in adsorption and columns, while powder forms with a size predominantly less than 0.15 mm to be used in batch adsorption followed by filtration (Allen and Koumanova 2005) ; (Dabrowski et al. 2005). Adsorption using ACs has been reported to be an effective method for the removal of pollutants from water and air (Bansal and Goyal 2005). Availability, cost, low inorganic matter content, low degradation during storage and ease of activation are the criteria needed to be considered in the selection of the precursors (Dabrowski et al. 2005). Physical and chemical activation methods are normally used for activation of chosen precursor. Physical activation is used to activate the precursor to enhance the porosity, surface area and surface chemistry of materials. It consist of two steps: thermal carbonization and activation. The carbonization process of raw materials under high temperature is to increase the carbon content and reduced the other component in the materials used. The activation process is conducted under gasified with an oxidizing agent such as steam or carbon dioxide to enhance the porosity, surface area and functional groups on the carbon surfaces. Chemical activation is done by chemical agents such as alkaline hydroxides, zinc chloride, or phosphoric acid, and followed by carbonization under inert gas and high temperature. The impregnated product must be washed to remove excess chemical agent (Zhang, Shao, and Karanfil 2010). The chemical nature of ACs surface is very important to determine the textural properties and of the adsorption capacity ACs. The activated carbon surface is heterogeneous, consisting multiple faces/edges and layers of

graphite sheets. The located some of elements such as halogen hydrogen, nitrogen, and particularly oxygen on the edges are consisted of the chemisorbed foreign heteroatom (El-Sayed and Bandosz 2004). In the interior of the graphite sheets, the edge sites seem to be more reactive than the residing atoms. There are many processes that forms of surface chemical functional groups such as the activation process of precursor like heat treatment and chemical treatment (Moreno-Castilla 2004) ; (Derylo-Marczewska *et al.* 2008). There are two categories of surface functional groups. First, basic groups consisting of pyrone, chromene, ethers and carbonyls (Boehm 1994). Second, acidic groups which are mainly consisting of carboxylic, lactones and phenols.

Moreover, the caustic carbon surface could help to immobilize the H_2S species on the adsorbent surface and would allow for greater removal efficiency due to the H_2S is acidic. The caustic materials such as NaOH or KOH is capable to improve the pH level of the carbon surface which result an attractive adsorbent toward H_2S. In this study, the preparation factors of ACs produced from coconut shell are investigated. The design of experiment is implemented to optimize the preparations factors of ACs to remove of H_2S from wastewater. All the activities of experimental planning, conducting experiments, and fitting models to the out puts responses are involved in this method. Response surface methodology (RSM) is the useful tool for optimizing the factors using center composite design (CCD). The objectives on this work are to investigate the optimum preparation factors conditions needed to maximize the removal efficiency (RE) (%) to remove dissolved H_2S from wastewater.

2.0   METHODS AND MATERIALS

2.1.   Preparation of activated carbon

Coconut shell is used to produce CSAC. The details of raw material preparation method of precursors are detailed in our previous work (Habeeb *et al.*, 2016). In this study, the precursor was activated with KOH at different temperature and time with a constant heating rate of 10 °C/min. The KOH to precursor ratio was varied from 2:1 - 4:1. The precursor was blended with KOH at different fertilization proportion (IR), using equation (1):

$$IR = \frac{W_{KOH}}{W_c} \tag{1}$$

Where, W_{KOH} is the weight (g) of KOH pallets and W_C is the weight (g) of precursor.

The procedures for activation process are explained in details in our previous study (Omar Abed Habeeb, Ramesh Kanthasamy, Gomaa A.M. Ali and Yunus 2017). After activation process, the samples are rinsed with hot distillate water and 1.0 M hydrochloric acid until the pH of the washing solution achieved 6–7. The preparation factors ranges are shown in Table 1.

2.2. Modeling and optimization

2.2.1. Experiment design

Response surface methodology (RSM) is an accumulation of numerical and statistical methods that are important for illustrating, examination, demonstrating and investigation of problems in which a response of interest effected by a couple factors (Montgomery 2001). Central composite design (CCD), three-level factorial design and Box–Behnken design are considered the several classes of designs under RSM and they are the most common designs utilized by the scholars. However, in current study the CCD was employed to study optimization and the effect of factors toward their responses(Montgomery 2001). This method is suitable for fitting a quadratic surface and it spreads to enhance the influential parameters with a minimum number of experiments as well as examine the association between the parameters. The three parameters investigated were the activation temperatures (T), mass ratio of KOH to Precursor (IR) and the duration of time activation (t) and their related extends were 724– 1000℃, 2:1– 4:1 and 60-120 min, respectively with response of removal efficiency.

The residual error was estimated by replicating the central point and based on the extend of each elements (factor), the independent factors are coded to the (-1, 1) interval. The different levels, that is to say, high and low, are coded +1 and -1 separately. The axial points are situated at (0, 0, ±œ), (±œ, 0, 0), (0, ±œ, 0), what makes the design rotatable is the œ which represents the axial point from the center. In order to set up an empirical model the responses were used which in turn correlate the responses to three preparation elements using a second degree polynomial equation as given by equation (2) (Zainudin et al. 2005):

$$Y = b_o + \sum_{i=1}^{n} b_i x_i + \left(\sum_{i=1}^{n} b_{ii} x_i \right)$$
$$+ \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} b_{ij} x_i x_j \qquad (2)$$

where Y is the predicted response, bo the constant coefficient, bi the linear coefficients, bij the interaction coefficients, bii the quadratic coefficients and xi, xj are the coded values of the activated carbon preparation variables.

Model fitting and statistical analysis are conducted using ANOVA statistical techniques. A coefficient of determination (R-squared), Fisher value (F-value), probability (P value), and residual were used as a standard of significance of the model equations (Hassani et al. 2014). Finally, optimization and validation are also conducted to find the optimum preparation factors conditions to enhance the removal of H_2S from wastewater.

2.2 Characterization of adsorbent

The surface structure morphology of CSAC adsorbent are characterized using scanning electron microscopy (SEM). Moreover, the elements of the adsorbent before and after adsorption process are examined by Energy-dispersive X-ray spectroscopy (EDX).

3.0   RESULT AND DISCUSSION

3.1 Model analysis of central composite design (CCD)

The independent variables such as activation temperature (724-1000 ℃), activation mass ratio KOH: Precursor (2:1-4:1 w %), and activation contact time (60-120 min) are investigated. The factors names, coded, and ranges, are demonstrated in Table 1. The dependent response is the removal efficiency of H_2S from wastewater. The correlations between independent variables and dependent response are developed using quadratic polynomial model.

Table 1: The code, unit and ranges for optimization of independent variables of (CSAC)

Variables	code	Unit	Coded variable levels				
			-œ	-1	0	+1	+œ
Activation temperature	A	℃	630	724	862	1000	1094
KOH: Precursor ratio	B	Wt%	1.3	2	3	4	4.5
Activation contact time	C	min	39.5	60	90	120	140.5

3.2. Statistical Analysis

The interactions between three parameters (activation temperature, KOH: precursor ration, and activation contact time) for the removal efficiency of dissolved H_2S are analysed using response surface methodology (RSM). The central composite design results are allowed to develop a mathematic equation that can predict the response value. Moreover, the analysis of variance (ANOVA) is used to analysis obtained results as represented in Table 2. The goodness of fit and lack-of-fit are estimated from ANOVA Table. The ANOVA test is successfully conducted as seen in Table 2. The quadratic model is significant. While the lack of fit was not significant. The lack of fit describes the variation of the data around the fitted model. Thus, this is an indicated to prove the statistically corrected the relation between the factors and response. Moreover, the F-value of the quadratic model is (44.9). Consequently, the terms of significant model are A, B, C, A^2, B^2, C^2, AC and BC. While AB is the insignificant model terms. It can be observed that factors of KOH: Precursor ration has high contribution to the response of removal efficiency as seen in Table 2. This is attributed to the important of impregnation ratio that might be enhanced the surface chemistry of the adsorbent and it also provides the basic environment that could be attractive to acidic pollutant such H_2S. In addition, the residual value is very low that another indicator that shows the goodness fitted of the model.

Table 2: Analysis of variance (ANOVA) and lack-of-fit test for response surface quadratic model for removal efficiency (RE %).

Sources	Sum of squares	df	Mean square	F-value	P value	Comment
Model	110.89	9	12.32	44.95	< 0.0001	significant
A-activation temperature	2.83	1	2.83	10.31	0.0037	significant
B-KOH:AC ratio	11.12	1	11.12	40.57	< 0.0001	significant
C-Activation contact time	1.35	1	1.35	4.94	0.0359	significant
AB	2.500E-003	1	2.500E-003	9.121E-003	0.9247	insignificant
AC	7.29	1	7.29	26.60	< 0.0001	significant

BC	3.24	1	3.24	11.82	0.0021	significant
A^2	75.64	1	75.64	275.94	< 0.0001	significant
B^2	25.46	1	25.46	92.87	< 0.0001	significant
C^2	2.57	1	2.57	9.39	0.0053	significant
Residual	6.58	24	0.27			
Lack of Fit	2.12	5	0.42	1.80	0.1609	not significant

3.3 Development of the equation of regression model

In addition, the CCD software is implemented to improve the empirical model. The correlation coefficient and standard deviation were used to obviate the model developed fitness. The better the model in forecasting the response is the smaller the standard deviation and the closer the R^2 value is to unity (Alam *et al*. 2009). The statistical analysis of quadratic model for removal efficiency is demonstrated in Table 3. It can be seen clearly that the R^2 value of 0.944 with small standard deviation 0.52. Moreover, the predicted R^2 is in agreement with the adjusted R^2 with only small differences between them. Therefore, the quadratic model is successfully developed. The quadratic model equation suggested by the software can be written as shown in equation (3). This equation can be used to predict the value of response (removal efficiency).

Furthermore, predicted vs actual values and normal plot of residual graphs are shown in Figure 1 (a, b) respectively. Figure 1 (a) represents the predicted vs actual values. This figure shows a sufficient agreement between the predicted data from the mode and the ones obtained from experiments. It is observed that the values are very close to the line which indicated the experimental values are closed to predict one. Figure 1 (b) shows the normal plot of residual error which demonstrated the residual error deviation from the model.

Table 3: Regression statistics for removal efficiency RE (%) at equilibrium.

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	Comment
Linear	1.85	0.1302	0.0433	-0.0604	
2FI	1.84	0.2199	0.0465	-0.0027	
Quadratic	**0.52**	**0.9440**	**0.9230**	**0.9011**	**Suggested**
Cubic	0.48	0.9609	0.9355	0.9253	Aliased

Removal efficiency = $88.6+0.32A+0.638B-0.2226C-0.0125AB+0.675AC-0.45BC-1.83A^2-1.0625B^2-0.337C^2$ (3)

Predicted vs. Actual
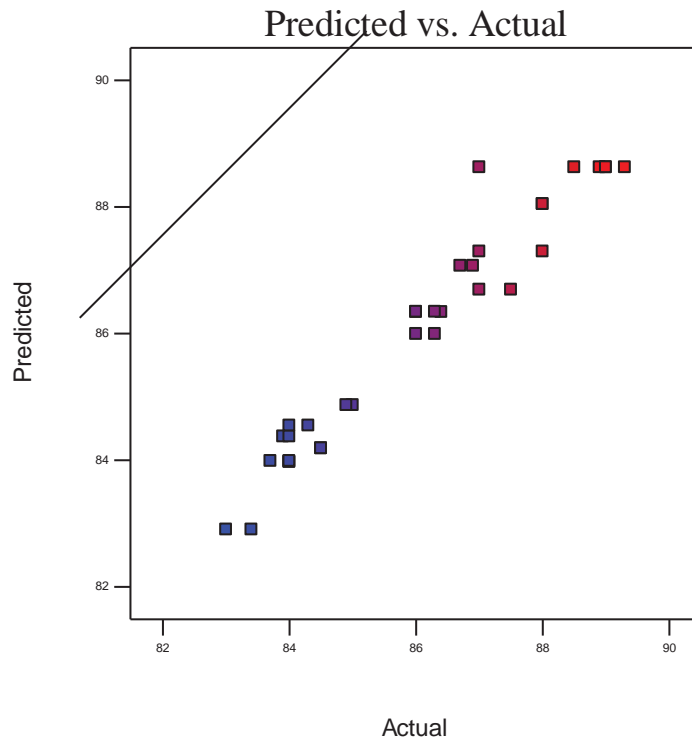
(a)
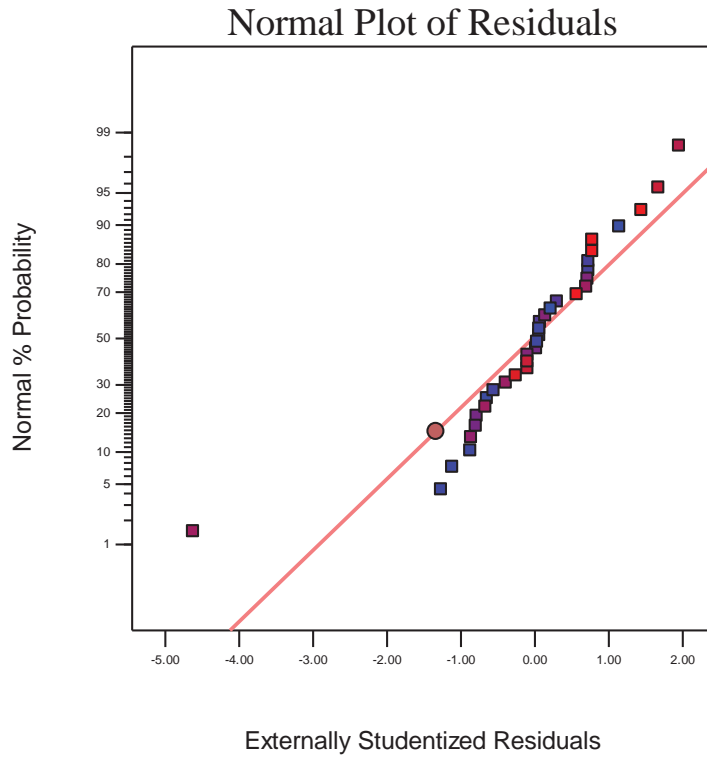
(b)

Figure1 (a) Actual and predicted curve. (b) Normal plot of residual

3.4 Effect of interaction factors

The effect of the factors interaction on the removal efficiency is demonstrated in Figure 2 (a, b, & c). The interaction between activation temperature and KOH: Precursor ratio are represented in Figure 2 (a). The two factors have affected the removal efficiency. It can be seen there is no interaction as the activated temperature increasing, the impregnation ration increased. However, the impact of the activation agent ratio has more significant effect on removal efficiency than activation temperature. While, the Figure 2 (b) represents the effect of interaction between activation temperature and activation contact time. The activation temperature has a significant effect on the removal efficiency of H_2S. The removal efficiency was low at lower temperature of 724 °C and higher activation contact time of 120 min. Then, it start increasing when the temperature increased and the time decreased until reach the optimum removal efficiency of 88.8 % at activation temperature of 857 °C and activation contact time of 70 min.
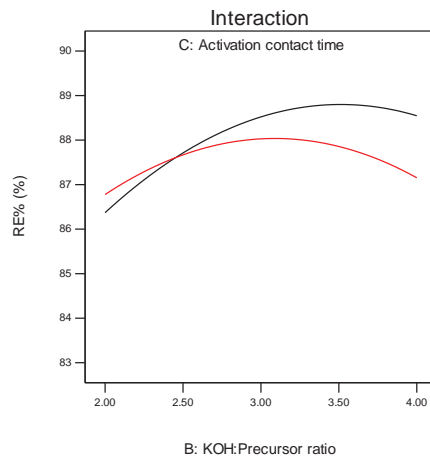
Figure 2 (c) shows the interactions between activation time and the impregnation ratio. The interaction plot shows the removal efficiency was very low at lower impregnation ratio and activation contact time. In fact, the graph demonstrated that high contact time of 120 min and low contact time of 60 min with low impregnation ratio are negatively affected the removal efficiency (as seen from Figure 2 (c)). After that, the removal of H_2S increasing with increased of impregnation ratio and activation contact time until reach the optimum removal efficiency of 88.8 % with impregnation ratio of 3.4 wt% and activation contact time of 70 min.
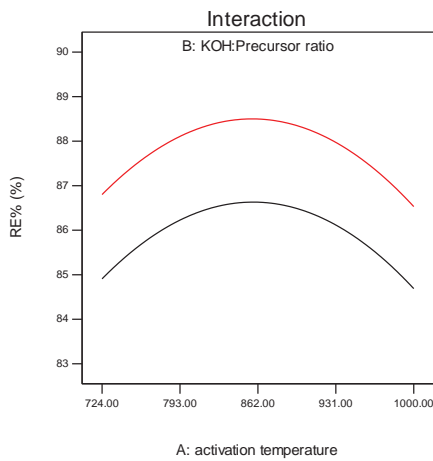
(a)
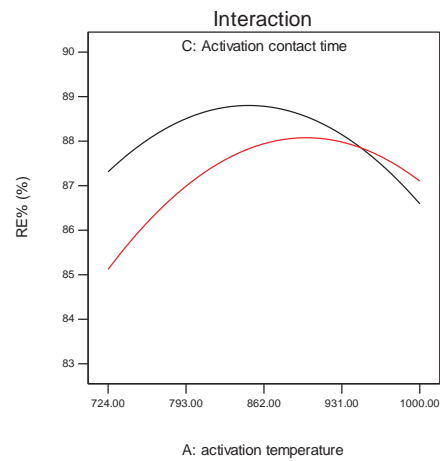
(b)

(c)

Figure 2. The effect of interactions between factors on the removal efficiency

3.5 Effect of combined factors

The effect of activation temperature and KOH: Precursor ratio on the removal efficiency show in Figure 3. The 3D plot presents impact of activation temperature with range between 724 to 1000℃ and activation agent mass ratio (KOH: Precursor) with range of (2:1-4:1 w%) on removal efficiency. From the trend of plot, it can be observed that, impregnation ratio and activation temperature have parallel contribution on removal efficiency. As increased in activation temperature and impregnation ratio, the removal efficiency increased. At the low activation temperature around 724℃, the value of removal efficiency was very low. After that, the removal efficiency start increased with increasing the activation temperature and impregnation ratio until reach the optimum requirement of activation temperature and impregnation ration for getting the optimum removal efficiency.

Moreover, the effect on the interaction between activation temperature and contact time on the removal efficiency are demonstrated in Figure 4. The activation temperature with range (724-1000℃) and activation contact time range (60-120 min) have a significant effect on response removal of H_2S. The temperature of activated of adsorbent has more effect on removal of H_2S than activation time as seen in Figure 4. From the trend of 3D plot was observed that H_2S experienced lowest residual at a preparation conditions of activation temperature of 857℃ and activation contact time of 66 min. Furthermore, the 3D plot of the interaction effect between activation time and KOH: Precursors on the removal efficiency are shown in Figure 5. It can be observed that the KOH: Precursors has a significant impact compared with the activation time which has less contribution on the removal of H_2S. The same trend was reported by Chaudhary (Chaudhary and Balomajumder 2014) As can be concluded that all the preparation factors of CSAC have a significant effect in different percentage that could contribute to enhance the removal of H_2S from synthetic wastewater.
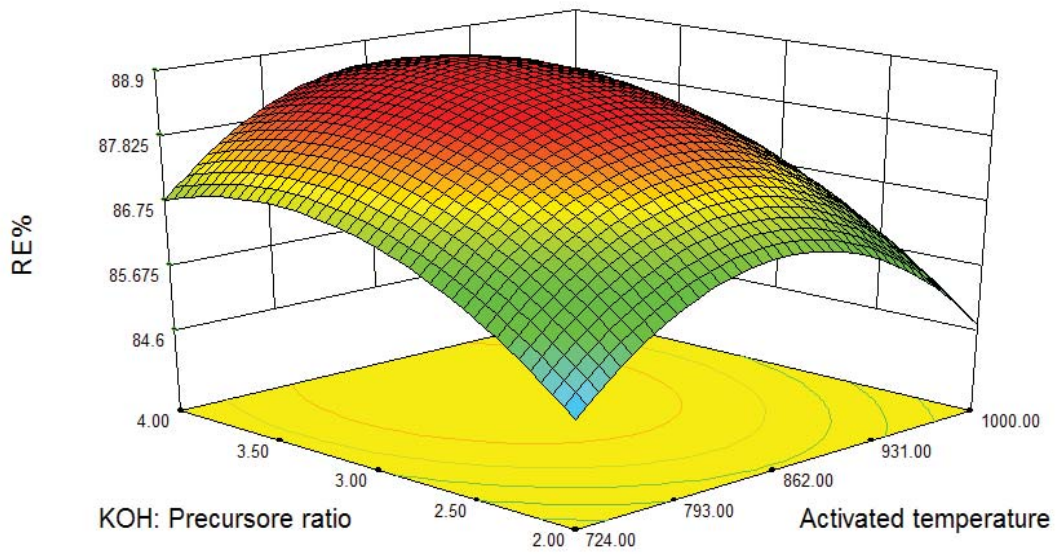
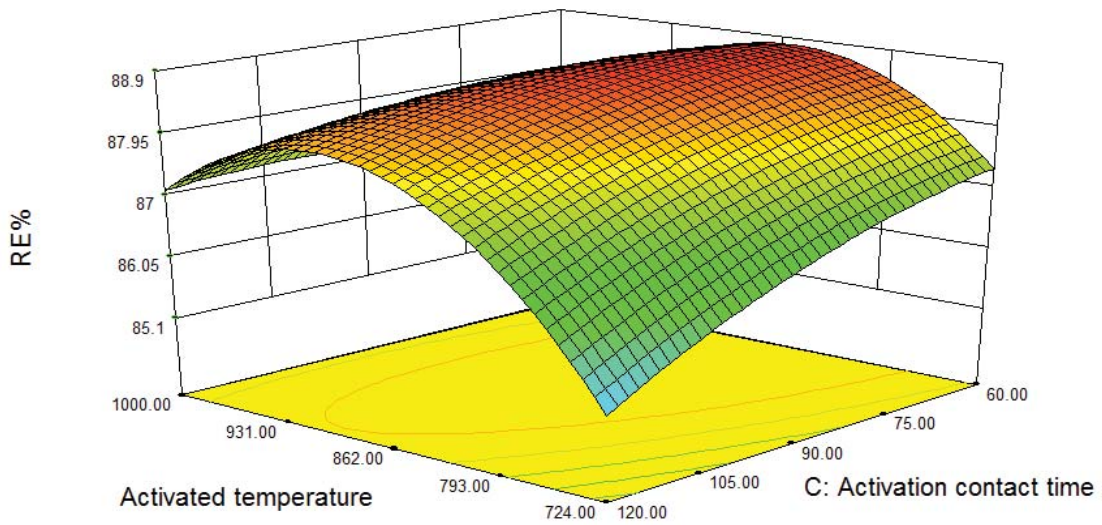Figure 3: Combined impact of activation temperature and KOH: Precursor ratio on RE (%).

Figure4: Combined impact of activation time and activation temperature of AC on of RE (%).
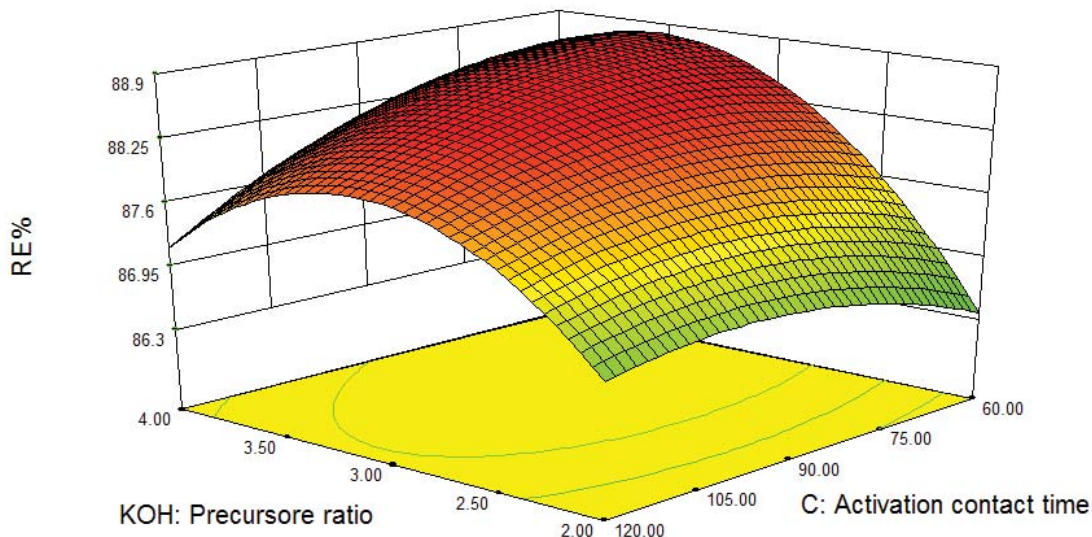
Figure 5: Combined impact of activation contact time and KOH: Precursor (IR) on RE (%)

3.6. Characterization of the optimally prepared activated carbon

3.6.1. Surface morphology (SEM / EDX)

Figure 6 (a, b) represent the SEM test for fresh and spent CSAC adsorbent respectively. As can be observed from Figure 6 (a) the chemical activation process with KOH was effective in creating well-developed pores on the surfaces of the activated carbon leading to produce activated carbon with a huge porous structure as seen in Figure 6 (a). The well-developed porosity on the surface of CSAC is attributed to the preparation factors such as activation temperature, activation contact time and KOH: Precursor ratio. The impregnation ratio with temperature have a significant contributed on developing the pore structure of the adsorbent due to during combustion of raw materials. It is attributed to chemical activations using of KOH. It possibly create more pores in CSAC structure due to the reaction between carbon and KOH as shown in equation (4). Pore generation is based on dehydration of KOH to K_2CO_3, which reacts with CO_2 produced by the water–shift reaction, affording K_2CO_3 as well as promoting the diffusion of KOH and CO_2 molecules into the pores, thereby increasing its porosity (Stavropoulos and Zabaniotou 2005). As seen in Figure 6 (a).

However, the image of SEM in Figure (b) indicating the morphology of adsorbent surface after adsorption process which could be seen that there is some blocked in porosity due to stick of

sulfur on the pore hole and also can be seen the some white particle on the surface which indicated that the sulfur already stick on porous area.

$$6KOH + 2C \longrightarrow 2K + 3H_2 + 2K_2CO_3 \tag{4}$$

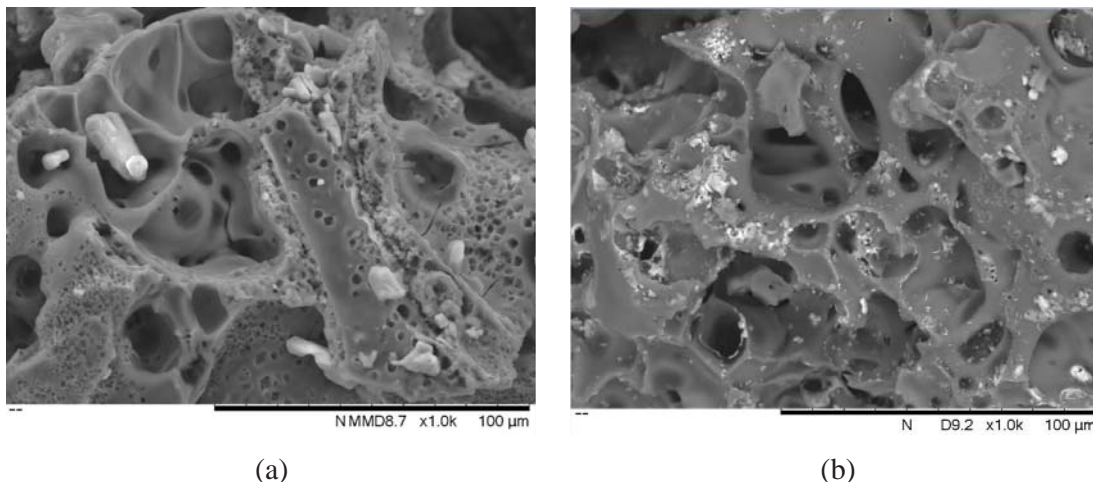(a)   (b)

Figure 6 SEM image of (a) fresh (b) spent CSAC adsorbent

Moreover, the EDX test for the CSAC before and after adsorption process are shown in Figure 7 (a, b). As can be seen the peaks for the fresh CSAC represents the adsorbent components which is (C, O and K). However, the peaks for the spent CSAC is representing the adsorbent components after adsorption process which is shown clearly that there more peaks which is represent the different components such as (C, O, K, and S). All composite components of the adsorbent before and after adsorption process shown in Table 4. It can be seen that the percentage of C, O, and K are decreased after the adsorption process. In fact, the most interesting finding was that the percentage of K decreased to around 0.93 % after adsorption, indicating that acid–base reaction occurs. Moreover, S content detected as 0.724, which is another indicator for the possible occurrence of oxidation. In fact, during the adsorption process, the acid/base reaction is occurred. The KOH provides a basic environment on the active sites on the adsorbent which attracted the acidic dissolved H_2S and its ions to produce the sulfide salt and water as seen in equation (5). The reactions occurred on the surface of CSAC. From that the percentage of C, O, and K are decreased as noticed in Table 4.

$$2KOH + H_2S \longrightarrow K_2S + 2H_2O \tag{5}$$
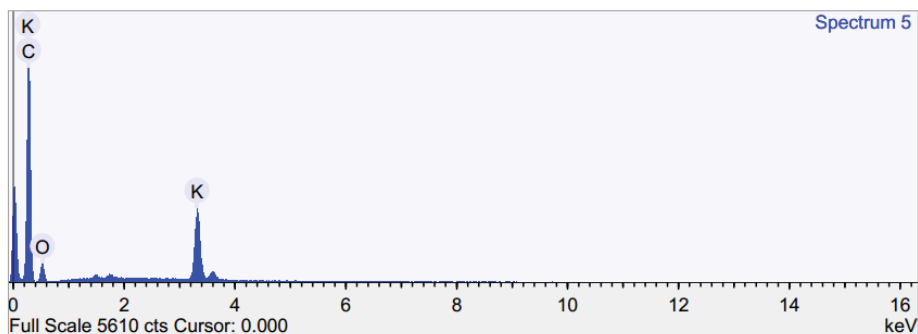
(a)
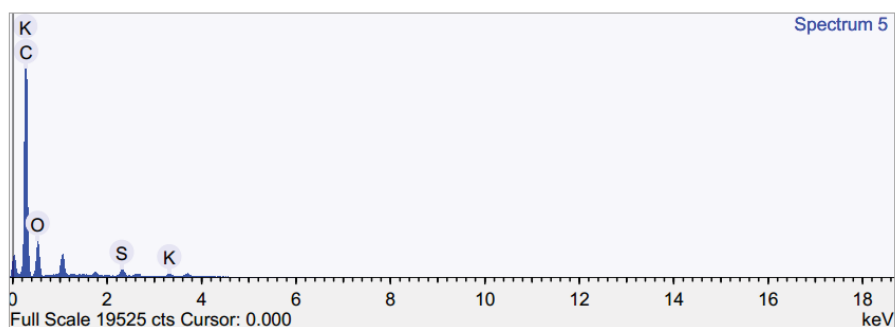
(b)

Figure 7 EDX test (a) Fresh. (b) Spent CSAC adsorbent.

Table 4: The components of activated carbon derived from coconut shell fresh and spent adsorbent.

Element	Component of fresh adsorbent (Weight %)	Components of spent adsorbent (Weight %)
Carbon (C)	76.575	73.24
Oxygen (O)	17.9	25.6
Potassium (K)	5.511	0.38
Sulfur (S)	0	0.724

4. 0   CONCLUSIONS

In this study the variables for preparations of CSAC for H_2S removal from wastewater was investigated. The optimum preparation variables conditions are examined. SEM and EDX

characterizations are conducted. The result shows that the optimum conditions of factors are temperature of 857 °C, chemical impregnation ratio of 3.4 wt% and activation time of 66 min with removal efficiency of 88.8 %.

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