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Optimization of preparation conditions for activated carbon from *Prosopis africana* seed hulls using response surface methodology

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ABSTRACT

Optimal conditions for the preparation of *Prosopis africana* seed hulls-based activated carbon (PASH-AC) for removal of 4-chloro-2-methoxyphenol (4C2MP) from aqueous solution were investigated. The PASH-AC was prepared using physiochemical activation method which involved sodium acetate (CH_3COONa) treatment and carbon dioxide (CO_2) gasification. Central composite design (CCD) was used to study the effects of three preparation variables which were activation temperature, activation time, and impregnation ratio (IR) on the 4C2MP removal and *Prosopis africana* seed hulls (PASH)-derived activated carbon yield (PASH-AC yield). Based on the CCD, two quadratic models were developed for both PASH-AC yield and 4C2MP removal. The optimum conditions for preparing activated carbon from PASH for the removal of 4C2MP were found as follows: activation temperature of 795°C, activation time of 62 min, and IR of 2.45 showing PASH-AC yield of 20.01% and 4C2MP removal of 92.89%. The experimental results obtained agreed satisfactorily with the model predictions. The activated carbon prepared under optimum conditions was mesoporous with BET surface area of 1085.92 m^2/g, total pore volume of 0.47 cm^3/g, micropore volume of 0.35 cm^3/g, and average pore size of 3.01 nm. The surface morphology and elemental composition of the activated carbon were determined by scanning electron microscopy and electron dispersive X-ray, respectively.

Keywords: *Prosopis Africana* seed hulls; Activated carbon; Central composite design; Response surface methodology; 4-chloro-2-methoxyphenol; Adsorption

1. Introduction

Substituted phenols which are generated by petroleum and petrochemical, coal conversion, phenol producing industries, and other chemical processes, are a class of phenolic compounds that are common contaminants in wastewater. Phenols as a class of organics are similar in structure to the more common herbicides and insecticides in that they are resistant to biodegradation [1]. Bad odor and taste indicate their presence in water supplies. In the presence of chlorine in drinking water, phenols form chlorophenols, which have a medicinal taste, which are quite pronounced and objectionable [1]. They are considered as priority pollutants because they are harmful to organisms at low concentrations and many of them have been classified as hazardous pollutants because of their

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possibility to harm human health. Because of their toxicity, the US Environmental Protection Agency and the European Union have designated phenols as priority pollutants [2,3]. Increasing concern for public health and environmental quality has led to the establishment of rigid limits on the acceptable environmental levels of specific pollutants [1]. Thus, the removal of phenols from waste streams is crucial as it became a major environmental problem.

Many treatment processes have been applied for the removal of chlorophenols from wastewater. Some of these processes include: adsorption [4–7], catalytic wet oxidation [8], and biodegradation [9,10]. Activated carbons (AC) are the most sought after adsorbents [11,12], widely utilized for a variety of industrial applications due to their versatile surface characteristics [13]. However, commercially available AC are usually not affordable [14], thus undesirable for large-scale treatment of industrial effluents [15], for that there is an increasing attention in using cheap and readily available materials as precursors for activated carbon production [16,17]. Waste materials such as lignin [18], date pits [2], inula viscosa waste [4], waste tea [19,20], pineapple leaf [21], and mango leaves [22] have been successfully converted into adsorbents on a laboratory scale.

An attempt was made in this work to prepare activated carbon from *Prosopis africana* seed hulls (PASH), a new agricultural material that has the potential of being a good precursor material for activated carbon production. *P. africana* is a perennial tree in the genus *Prosopis* that grows very well in Nigeria and other parts of West Africa. In many areas, its fermented seeds are used to prepare a food condiment called daddawa [23]. Daddawa is a locally fermented product that is widely used as a food condiment in Nigeria and many parts of West Africa so the seed hulls from this process lie as wastes and constitute environmental concern, therefore, good handling of the waste materials generated from this process could result in waste minimization and tremendous cost saving. Preliminary studies have shown the seed hulls of *P. africana* to contain high carbon and low ash content which makes it a good precursor material for preparing activated carbon as an adsorbent.

In evaluating the effect of treatments on quality attributes, the use of an adequate experimental design is imperative. Currently, no study has been done on optimization of the preparation conditions for the production of activated carbon from PASH using the response surface methodology (RSM) for the removal of 4-chloro-2-methoxyphenol (4C2MP).

Therefore, the innovative aspects of this work were to optimize the preparation conditions of activated carbon from PASH for the removal of 4C2MP and also introduce sodium acetate as a new activating agent. The effects of preparation variables; activation temperature, impregnation ratio (IR), and activation time were studied simultaneously to obtain a high activated carbon yield as well as high 4C2MP percentage removal from aqueous solution using central composite design (CCD).

2. Materials and methods

2.1. Adsorbate

The adsorbate (4C2MP) was supplied by Sigma–Aldrich (M) Sdn Bhd, Malaysia. Deionized water was used to prepare its solution. 4C2MP has a chemical formula of C_7H_7ClO with molecular weight of 158.58 g mol^{-1}. The chemical structure of 4C2MP is shown in Fig. 1.

2.2. Preparation of adsorbent (PASH-AC)

The seed hulls of *P. africana* were collected locally from Nigeria as discarded agricultural wastes. They were washed with distilled water, dried at 105℃ for 24 h, ground using a locally made grinder and sieved into particle size of 200–500 μm to generate the precursor. Analytical grade chemicals/reagents were used in this work.

The activated carbon preparation procedure was referred to our previous work [24] with little modification. The modification includes impregnating PASH with sodium acetate (CH_3COONa) as chemical activating agent with the IR computed using the following relationship:

$$\text{Impregnation Ratio} = \frac{\text{dried weight of } CH_3COON_a}{\text{dried weight of precursor(PASH)}} \tag{1}$$

The adsorbent preparation was strictly adhered to the DOE table as shown in Table 1.

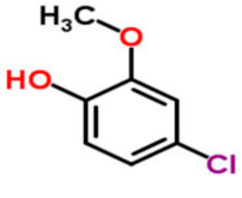

Fig. 1. Chemical structure of 4C2MP.

Table 1
Experimental design matrix for preparation of PASH-AC and responses

Run	Level			PASH activated carbon preparation variables				
				Temperature (℃)	IR	Time (min)	Y_{PASH} (%)	Y_{4C2MP} (%)
1	−1	−1	−1	658	0.97	52	21.75	26.43
2	+1	−1	−1	842	0.97	52	18.07	84.57
3	−1	+1	−1	658	3.23	52	16.80	90.52
4	+1	+1	−1	842	3.23	52	16.38	92.30
5	−1	−1	+1	658	0.97	148	20.99	60.44
6	+1	−1	+1	842	0.97	148	10.78	91.83
7	−1	+1	+1	658	3.23	148	16.58	89.98
8	+1	+1	+1	842	3.23	148	10.86	93.69
9	−1.682	0	0	595	2.10	100	20.36	82.29
10	+1.682	0	0	905	2.10	100	15.07	89.12
11	0	−1.682	0	750	0.20	100	23.61	33.00
12	0	+1.682	0	750	4.00	100	16.79	91.35
13	0	0	−1.682	750	2.10	20	21.90	89.87
14	0	0	+1.682	750	2.10	180	17.03	88.49
15	0	0	0	750	2.10	100	19.69	88.04
16	0	0	0	750	2.10	100	18.79	87.87
17	0	0	0	750	2.10	100	18.93	88.09
18	0	0	0	750	2.10	100	18.86	88.59
19	0	0	0	750	2.10	100	19.90	87.99
20	0	0	0	750	2.10	100	19.42	87.94

2.3. Design of experiments using RSM

A standard RSM design known as CCD was applied in this work to study the variables for preparing the AC from PASH in the same way as reported in our previously published work [24].

The variables studied were activation temperature (x_1), impregnation ratio (x_2), and activation time (x_3) with their respective ranges chosen based on the literature as given in Table 2.

2.4. Batch adsorption for the removal of 4C2MP

Batch adsorption experiments for the 4C2MP removal by PASH-based activated carbon (PASH-AC) were also conducted as reported in our formerly published work [25]. The percentage removal (% R) of 4C2MP was evaluated as:

$$\text{4C2MP removal}\,(\%) = \frac{C_o - C_e}{C_o} \times 100 \quad (2)$$

where the initial and equilibrium concentrations are denoted as C_o and C_e (mg/L), respectively.

The design expert statistical software (version 6.0.8 Stat Ease, Inc., Minneapolis, MN 55413, USA) was employed for model fitting and significance for the PASH-AC yield and percentage removal of the 4C2MP.

Table 2
Independent variables and their coded levels for the CCD of PASH-AC preparation

Variables	Code	Unit	Coded variable levels				
			$-\alpha$	−1	0	+1	$+\alpha$
Activation temperature	x_1	℃	595	658	750	842	905
Impregnation ratio (IR)	x_2	–	0.20	0.97	2.10	3.23	4.00
Activation time	x_3	min	20	52	100	148	180

2.5. Characterization of the prepared activated carbon

The BET surface area, micropore volume, total pore volume, and the pore size of the PASH activated carbon prepared under optimum preparation conditions were determined using Brunauer–Emmett–Teller (BET) equation and Quantachrome Nova Win2© 1994–2002. As a comparison, a sample of QRëC commercial activated carbon was also characterized under same conditions. The surface morphology of the samples was examined using a scanning electron microscope (FEI QUANTA PEG 60) while electron dispersive X-ray was used to determine the elemental composition of the samples (precursor and the prepared activated carbon). Proximate analysis was carried out using thermo gravimetric analyser (Perkin Elmer TGA7, USA).

3. Results and discussion

3.1. Development of regression model equation

Design matrix comprising the preparation variables, their ranges and the responses, which are percentage yield (Y_{PASH}) and adsorption of 4C2MP (Y_{4C2MP}) are presented in Table 1.

CCD was used to correlate the responses for the development of the polynomial regression equations which were all quadratic expressions as suggested by the software. The model expression was selected in accordance with sequential model sum of square that is based on the polynomial's highest order where the model was not aliased and the additional terms were significant.

Qualitative correlation between the predicted and experimental data was prominent as indicated by the model's R^2 values of 0.8673 (yield) and 0.9135 (4C2MP) which were within the desirability range [26]. The final empirical model's equations for the PASH activated carbon yield (Y_{PASH}) and removal of 4C2MP (Y_{4C2MP}) responses are given as Eqs. (3) and (4), respectively.

$$\begin{aligned} Y_{\mathrm{PASH}} = {} & 19.35 - 2.12x_1 - 1.64x_2 - 1.61x_3 - 1.09x_1^2 - 0.22x_2^2 \\ & - 0.48x_3^2 + 0.97x_1x_2 - 1.48x_1x_3 + 0.29x_2x_3 \end{aligned} \tag{3}$$

$$\begin{aligned} Y_{\mathrm{4C2MP}} = {} & 88.08 + 7.80x_1 + 14.74x_2 + 2.91x_3 \\ & - 0.79x_1^2 - 9.11x_2^2 + 0.44x_3^2 \\ & - 10.51x_1x_2 - 3.10x_1x_3 - 5.05x_2x_3 \end{aligned} \tag{4}$$

The positive and negative signs before the terms indicate synergetic and antagonistic effect of the respective variables. The appearance of a single variable in a term signified a uni-factor effect, two variables imply a double-factor effect, and a second-order term of variable appearance indicates the quadratic effect.

3.2. Statistical analysis

The results of the surface quadratic model in the form of analysis of variance (ANOVA) are given in Tables 3 and 4 for PASH-AC yield and 4C2MP removal, respectively. ANOVA is required to validate the importance and how adequate the models are. The mean squares were obtained by dividing the sum of the squares of each of the variation sources, the model, and the error variance, by the respective degrees of freedom [27]. If the value of Prob. > F is less than 0.05, the model terms are considered as significant. With respect to PASH-AC yield from Table 3, it can be seen that the model F-value and Prob. > F-value were 7.26 and 0.0023, respectively, indicating the model's significance. The significant model terms were x_1, x_2, x_3, x_1^2, and x_1x_3 with x_2^2, x_3^2, x_1x_2, and x_2x_3 denoting the insignificant terms.

From Table 4, the model F-value of 11.73 and Prob. > F of 0.0003 indicated that the model was significant. In this case, x_1, x_2, x_2^2 and x_1x_2 were the significant model terms whereas $x_3, x_1^2, x_3^2, x_1x_3$, and x_2x_3 were insignificant to the response. From the statistical results obtained, it was shown that both the models (Eqs. (3) and (4)) were adequate to predict the PASH-AC yield and the 4C2MP removal, respectively, within the range of variables studied. In addition, Figs. 2 and 3 show the predicted values vs. the experimental values for PASH-AC yield and the 4C2MP removal. It can be seen that the models developed were successful in capturing the correlation between the PASH-AC preparation variables and the responses when the predicted values obtained were quite close to the experimental values.

3.3. PASH-AC yield

From Table 3, it can be seen that activation temperature has the greatest effect on PASH-AC yield with the highest F-value of 22.44, whereas IR and activation time produced similar effects on this response with F-values of 13.50 and 12.95, respectively.

The quadratic effect of activation temperature was more pronounced with F-value of 6.33, while that of IR and time were low with F-values of 0.25 and 1.20, respectively. The interaction effects between

Table 3
The ANOVA for response surface quadratic model of PASH-AC yield

Source	Sum of squares	Degree of freedom	Mean square	*F*-value	Prob. > *F*
Model	178.51	9	19.84	7.26	0.0023
x_1	61.27	1	61.27	22.44	0.0008
x_2	36.87	1	36.87	13.50	0.0043
x_3	35.38	1	35.38	12.95	0.0049
x_1^2	17.28	1	17.28	6.33	0.0306
x_2^2	0.67	1	0.67	0.25	0.6298
x_3^2	3.27	1	3.27	1.20	0.2996
x_1x_2	7.51	1	7.51	2.75	0.1283
x_1x_3	17.49	1	17.49	6.41	0.0298
x_2x_3	0.67	1	0.67	0.24	0.6318
Residual	27.31	10	2.73	–	–

Table 4
ANOVA for response surface quadratic model of 4C2MP removal by PASH-AC

Source	Sum of squares	Degree of freedom	Mean square	*F*-value	Prob. > *F*
Model	6301.24	9	700.14	11.73	0.0003
x_1	830.62	1	830.62	13.92	0.0039
x_2	2968.68	1	2968.68	49.74	<0.0001
x_3	115.98	1	115.98	1.94	0.1935
x_1^2	8.98	1	8.98	0.15	0.7063
x_2^2	1195.60	1	1195.60	20.03	0.0012
x_3^2	2.78	1	2.78	0.05	0.8334
x_1x_2	882.84	1	882.84	14.79	0.0032
x_1x_3	77.00	1	77.00	1.29	0.2825
x_2x_3	204.22	1	204.22	3.42	0.0941
Residual	596.86	10	59.69	–	–

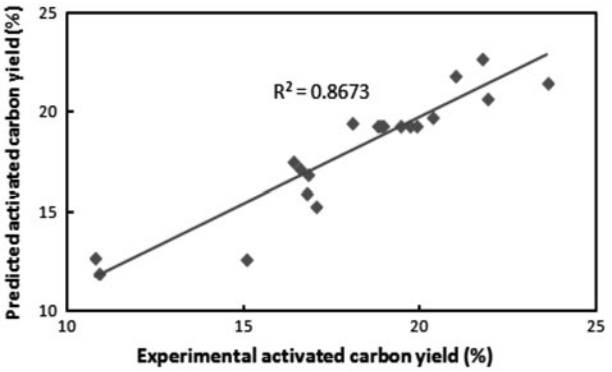

Fig. 2. Relationship between predicted and experimental data of PASH-AC yield.

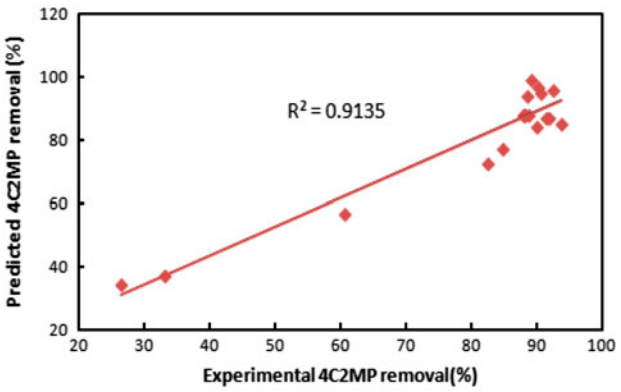

Fig. 3. Relationship between predicted and experimental data for 4C2MP removal.

IR-temperature and that of IR-time were slightly insignificant with *F*-values of 0.24 and 2.75, respectively, while that of interaction between temperature and time was more significant with *F*-value of 6.41.

Fig. 4((a) and (b)) shows the 3D response surface plots for the studied variables with Fig. 4(a) demonstrating the effect of time and temperature of activation with IR fixed at zero level (IR = 2.10), whereas Fig. 4(b) demonstrates the effect of temperature and IR with activation time fixed at zero level ($t = 100$ min), on the same response (Y_{PASH}). From both figures, the PASH-AC yield was also found to decrease with increase in activation temperature, activation time, and IR.

3.4. 4C2MP removal

From the ANOVA data generated in Table 4 for 4C2MP removal, it can be seen that the three variables (temperature, IR, and time of activation) have uneven impact on the removal of 4C2MP onto the prepared PASH-AC. Activation time had the least effect as its *F*-value was smaller and also its variation did not have a significant effect on the processes. The individual factors of IR and temperature and their interactions had the most significant effect on the 4C2MP adsorption as can be seen by their largest *F*-values as shown in Table 3. The *F*-values of 4C2MP removal by the PASH-AC for activation temperature (x_1), impregnation ratio (x_2), and activation time (x_3) were 13.92, 49.74, and 1.94, respectively.

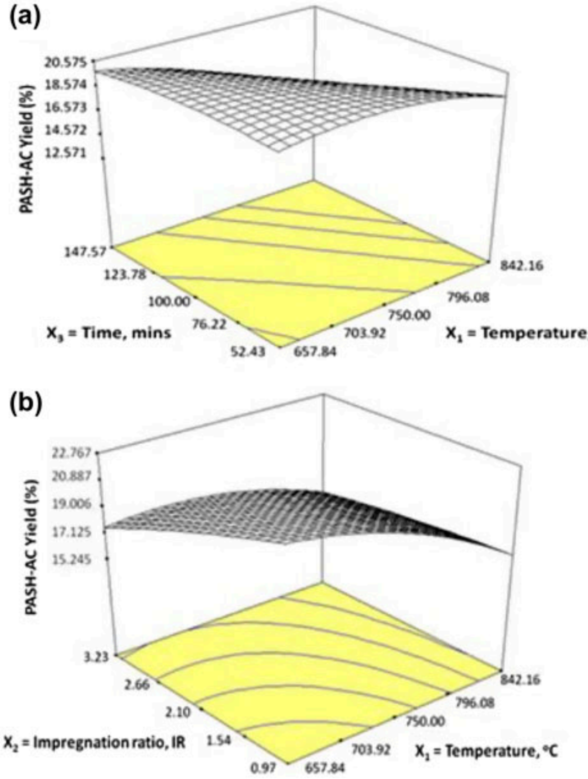

Fig. 4. Three-dimensional response surface plot of PASH-AC yield; (a) effect of activation temperature and activation time, IR = 2.10 and (b) effect of activation temperature and IR, t = 100 min.

IR showed the largest *F*-value of 49.74 among all the factors, indicating that this variable enacted the most significant effect on the 4C2MP removal by the PASH-AC. The effect of activation temperature was significant as well with *F*-value of 13.92. However, the effect of activation time on the response was relatively low (*F*-value of 1.94). As can be seen from Fig. 5, increase in IR and activation temperature was proportional to high adsorption of 4C2MP by the PASH-AC.

3.5. Process optimization

One of the main aims of this study was to find the optimum process parameters in which PASH-AC prepared should have a high carbon yield as well as high 4C2MP percentage removal. It is difficult to optimize both these responses under the same conditions because the interest region of factors is different. When adsorption performance increases, carbon yield will decrease and vice versa, therefore, the function of desirability was applied using the design-expert software, in order to compromise between these responses. In the optimization analysis, the target criteria were set as maximum values for the two responses. The predicted and experimental results of 4C2MP percentage removal and PASH-AC yield

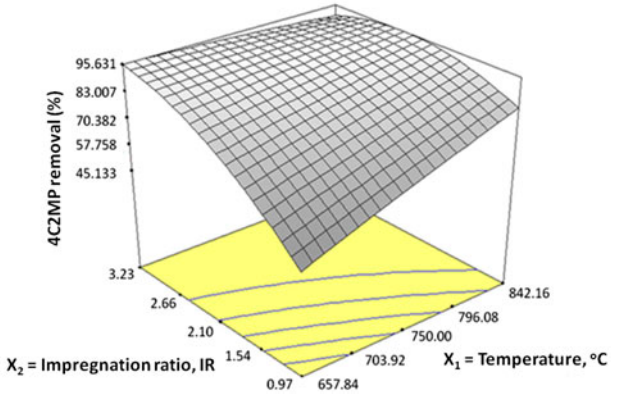

Fig. 5. Three-dimensional response surface plot of 4C2MP removal (effect of activation temperature and IR, t = 100 mins) on PASH-AC.

Table 5
The PASH-AC preparation parameters optimization

Parameters		PASH-AC
4C2MP removal (%)	Predicted	94.45
	Experimental	92.89
	Error	1.65
Activated carbon yield (%)	Predicted	19.43
	Experimental	20.01
	Error	2.99
Model Desirability		0.95
x_1 (℃)		795
x_2 (IR)		2.45
x_3 (min)		62

obtained at optimum conditions are listed in Table 5. The optimum PASH-AC was obtained using activation temperature of 795℃, activation time of 62 min, and IR of 2.45 showing PASH-AC yield of 20.01% and 4C2MP removal of 92.89%. It was observed that the experimental values obtained were in good agreement with the values predicted from the models, with relatively small percentage errors which were only 2.99 and 1.65% for PASH-AC yield and 4C2MP removal, respectively.

3.6. Characterization of the optimized PASH-AC

3.6.1. Surface morphology

Fig. 6(a) and (b) shows the scanning electron microscopy (SEM) images of the raw PASH and the PASH-AC, respectively. The precursor's surface textures were rough, uneven, undulating, and very little pores were present as observed in the other biomass [28–30]. From Fig. 6(b), it can be seen that almost homogeneous type pores structures were distributed on the surface of the PASH-AC. This result revealed that the combination activation process of CH_3COONa and CO_2 was effective in creating well-developed pores, resulting in large surface area activated carbon with a good mesoporous structure. Similar observations were reported by other researchers in their work of preparing AC from Borassus aethiopum shells [29], oil palm shell [30], and mangosteen peel [28].

3.6.2. Elemental analysis

The raw material of the PASH showed the presence of carbon, oxygen, sodium, chlorine, and silicon with 51.06, 47.14, 0.94, 0.39, and 0.47 wt.%, respectively, while the activated carbon prepared with CH_3COONa showed only three elements which are carbon, oxygen, and sodium with 83.23, 15.71, and 1.06 wt.%, respectively, as shown in Table 6. The raw PASH sample demonstrated lower carbon content and higher oxygen content compared to the PASH-AC. The carbon content increased from 51.06 to about 83.23 wt.% while the percentage of oxygen decreased to about 15.71 wt.% from 47.14% with the disappearance of chlorine and silicon after the activation process, this suggests that the chemical activating agent (CH_3COONa) has removed the interfering elements (chlorine and silicon) from the raw PASH sample.

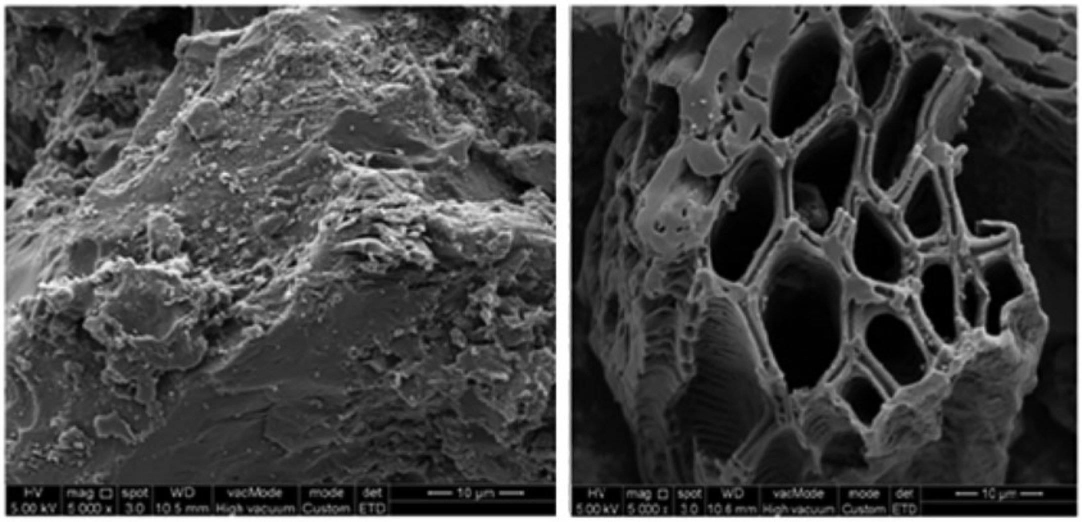
(a) (b)

Fig. 6. SEM micrograph of (a) raw PASH sample and (b) PASH-based activated carbon.

Table 6
Elemental contents of raw PASH sample and PASH-based activated carbon

Activated carbons	Element weight (%) C	O	Na	Cl	Si	Total wt.(%)
PASH sample	51.06	47.14	0.94	0.39	0.47	100
PASHAC–CH_3COONa	83.23	15.71	1.06	–	–	100

Table 7
Proximate analysis of raw PASH sample and PASH-based activated carbon

Activated carbons	Proximate analysis (%) Moisture content	Volatile matter	Fixed Carbon	Ash content
PASH sample	11.82	35.21	49.06	3.91
PASHAC–CH_3COONa	5.07	11.00	80.22	3.71

3.6.3. Proximate analysis

Table 7 presents the proximate analysis of precursor and the prepared activated carbon. The high carbon content of the precursor revealed its potential to be used for activated carbon preparation after evacuating the volatile components. The carbon content of this precursor is comparable with some carbonaceous materials [31]. A low ash content of carbonaceous material is a sign of a tendency that the precursor can withstand high temperature treatment during carbonization cum chemical activation. After undergoing carbonization and activation process, the volatile matter content of the precursor decreased significantly from 35.21 to 11.00%, whereas the fixed carbon content in the prepared activated carbon increased from 49.06 to 80.22% due to pyrolytic effect at high temperature where most of the organic substances have been degraded and discharged both as gas and liquid tars leaving a material with high porosity and carbon purity.

3.6.4. BET surface area and pore volume

The N_2 adsorption–desorption isotherms and the pore size distribution by Barret, Joyner and Halenda (BJH) method for PASH-AC are shown in Fig. 7(a) and (b). The sample presents an isotherm of I–IV hybrid shape from Fig. 7(a) according to the IUPAC classification. As the relative pressure increases, the adsorption isotherm of both the samples increases gently and a hysteresis loop appears, pointing to a vast majority of micropores associated with a considerable development of mesoporosity. The results obtained from analyzing the isotherms were as follows: BET surface area of 1,085.92 m^2/g, total pore volume of 0.47 cm^3/g, and micropore volume of 0.35 cm^3/g. According to the pore size distribution shown in Fig. 7(b), the presence of significant mesopores together with micropores can be found in the

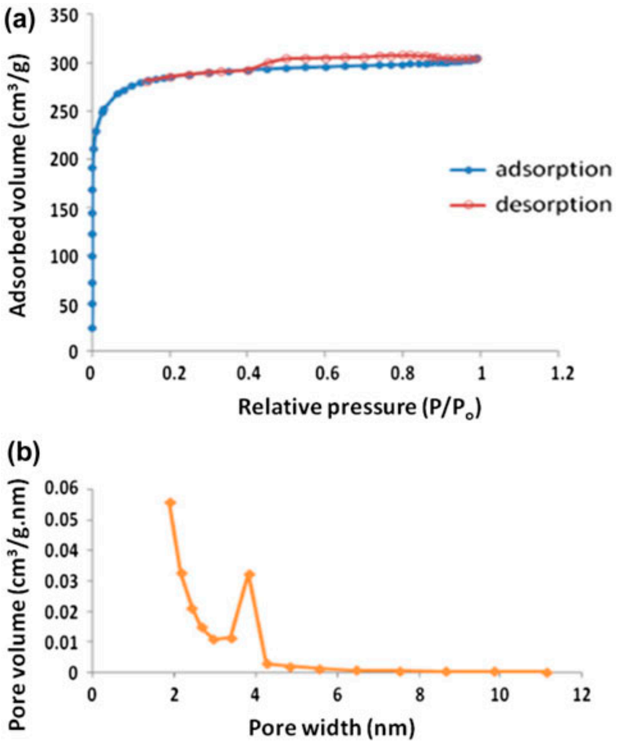

Fig. 7. (a) N_2 adsorption and desorption isotherms at 77 K and (b) BJH pore size distribution for the optimized PASH-AC.

Table 8
Comparing the surface areas and pore volumes of some adsorbents

Adsorbents	BET surface area (m^2/g)	Pore volume (cm^3/g)	Refs.
PASH-AC	1085.92	0.47	This work
Lignin-derived activated carbon	931.53	0.40	[18]
Waste tea activated carbon	232.95	0.08	[19]
Leather waste activated carbon	509.50	0.31	[5]
Borassus aethiopum activated carbon	795.21	0.48	[32]
Date pits activated carbon	425	0.17	[2]

PASH-AC, giving rise to an average pore size of 3.01 nm in the mesopores region according to the IUPAC classification.

The high BET surface area and total pore volume of the activated carbon were due to the activation process used which involved both chemical and physical activating agents (CH_3COONa and CO_2). Pore development during carbonization was important as this would enhance the BET surface area and pore volume of the activated carbon by promoting the diffusion of the activating agent molecules into the pores, and thereby increasing the CH_3COONa–carbon and CO_2–carbon reactions, which would then create more pores in the activated carbon [32]. The results obtained in this study were in agreement with the works found in the literature as shown in Table 8.

4. Conclusions

The effects of PASH-AC preparation variables (activation temperature, impregnation ratio, and activation time) on the PASH-AC yield and 4C2MP removal have been studied using CCD–a subset of RSM. Activation temperature was found to have the most significant effect on PASH-AC yield, while IR was found to impose the greatest effect on the 4C2MP removal. The optimum PASH-AC preparation conditions were obtained as activation temperature of 795℃, activation time of 62 min, and IR of 2.45 showed considerably high PASH-AC yield and 4C2MP percentage removal. The experimental values obtained were in good agreement with the values predicted from the models with relatively small errors. The optimum PASH-AC was mesoporous with significantly high BET surface area and demonstrated heterogeneous type of pore structure. The results confirmed sodium acetate and PASH to be a promising activating agent and low-cost precursor, respectively, for activated carbon production.

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