POLYETHYLENE TEREPHTHALATE BASED ACTIVATED CARBON PRODUCTION: PRELIMINARY STUDY ON KOH ACTIVATION WITH MICROWAVE ASSIST

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ABSTRACT: Since the beginning of the plastic age, drinking-bottle waste has been increasing worldwide. Petroleum plastic waste is composed mainly of Polyethyleneterephthalate (PET), which is remained in the environment for a long period, thereby causing significant harm to humans, flora, and fauna. Waste recycling is one of the promising mitigation strategies, being used to reduce the amount of final disposal. This research, a preliminary investigation of activated carbon production conditions from PET bottle waste, was carried out using Fractional Factorial Design (FFD) with 95% confidence level. Starter material PET waste was obtained from a local recycling factory. Crusted and sieved PET was carbonized at 700°C for 1 hr. (called PET-char), then impregnated with potassium hydroxide (KOH) at a designed ratio and heated in a microwave. The production factors included PET-char:KOH ratio (1 and 3 by weight), microwave power (540 and 900 watts), and microwave heating time (10 and 30 mins). It was found that all production factors were significant contributors to the characteristics of PET-based activated carbon (PET-AC). The highest iodine adsorption capacity was found at the condition of 3 by weight, 900 Watts, and 30 mins for ratio, microwave power, and heating time, respectively. PET-AC shown highest iodine adsorption capacity with 1,125.34 mg/g and Brunauer Emmett Teller (BET) surface area with 1,345 m²/g. It was confirmed that PET bottle-based waste can be converted to a highly valuable nanoporous adsorbent.

Keywords: Activated carbon, Polyethyleneterephtalate, Design of experiment, Microwave

1. INTRODUCTION

Activated Carbon (AC) is a black material with high surface area, porosity, and high volume. It can be used as environmental purification, electrochemical energy storage, capacitors, catalyst supporter, separation/purification of gas and liquids, and removal of color and toxic substances in water and drinking water [1-6]. The activated carbon can be made from various raw materials that contain low ash content, high fixed carbon, and low cost, such as coal, coconut and palm shell, wood, bamboo, agricultural residues, PVC, and PET bottle waste [7-16]. In case of the increasing capacity of polymer waste, PET is one of the most abundant wastes in multiple levels namely municipal, industrial areas, and ocean [17]. To minimize the issue of PET waste, an interesting solution is converting PET waste, that is negative valued waste, into valuable materials such as activated carbon. It can be used as the starter material for activated carbon production due to its low organic content [18]. PET-based Activated Carbon (PET-AC) provided higher surface area than the commercial activated carbon [19].

Activated carbon preparation can be performed by utilizing two different methods: physical activation (CO₂ or steam) and chemical activation (KOH, ZnCl₂, HNO₃, and NaOH) [20-23]. Typically, the surface area of PET-based activated carbon by chemical activation is higher than physical activation. Conventional heating using physical activation (CO₂ and steam) provided a surface area of 1,100 - 2,831 m²/g [19,24-27], while for chemical activation, it was $1,200 - 2,900 \text{ m}^2/\text{g}$ [28-31]. Using a convention furnace at high temperature, for a long duration of heating of about 1 to 5 h [15,24,29,32], and high energy consumption resulted in non-uniform porosity of the activated carbon [5,33]. This is a result of the heat transferred from the surface to the inside of the materials, with thermal gradient from high temperature surface to the interior until steady state [34-36]. Recently, a method that has been prominent is microwave irradiation because of its shorter activation time (10-20 mins) [37-40] and low energy consumption [41]. Moreover, this method is able to reduce the non-uniform porosity to solve the present problems. The microwave heats the molecular level of materials by dipole rotation and ionic conduction [42,43].

This research aimed to preliminarily investigate the optimum production conditions of PET-based activated carbon by KOH activation using microwave heating.

2. METHODOLOGY

2.1 PET-char Preparation

The PET bottle's waste was washed and crushed to 1 - 3 mm and dried at 80°C for 24 h. PET was converted to char to remove volatile matter and increase carbon content before activation. Twenty grams of sieved PET was carbonized at 700°C with a 10°C/min heating rate under nitrogen atmospheres for 1 h. Then, it was cooled at room temperature (25 - 30°C). The remaining black solid material was called PET-char, which was ready for the activation process.

2.2 Experimental Design

Design of Experiment (DOE) via Fractional Factorial Design (FFD) has been used for the investigation of the optimum condition of PET-AC synthesis. FFD optimized the number of experiments, time, and overall process cost [44,45].

The three production factors were PETchar:KOH ratio (1:1 and 1:3 by wt.), microwave power (540 and 900 watts), and microwave heating time (10 and 30 mins) with two levels are showing in Table 1. The production factors were A-ratio, Bmicrowave power, and C-microwave heating time. The experiment was performed in triplicate with totally 12 experiments.

Table 1 PET-AC production factors and levels

Factors	Code	Level	
		-1	+1
Ratio (by weight)	А	1	3
Microwave power (Watt)	В	540	900
Microwave heating time (min)	C	10	30

2.3 PET-AC Production

The mixture of PET-char and KOH at the designed ratio was placed in a 32 mm diameter vertical quartz tube container and heated in a microwave oven (Magnetron OM75S (31), 2450 MHz) under a nitrogen flow rate of 200 ml/min at the given power and the microwave heating time. Afterward, the activated char was rinsed with hydrochloric acid and washed with hot distilled water until the pH reached neutral. The solid adsorbent was dried at 105°C overnight. The produced activated char was then named PET-based activated carbon (PET-AC). All PET-AC were implemented for its iodine adsorption capacity, in order to determine the optimum production condition using analysis of variance.

2.4 PET-AC Characterization

The iodine adsorption capacity of the obtained PET-AC from all experiments were characterized according to the American Society Testing and Materials (ASTM D4607-86). The surface area, the pore volume and the pore size distribution of the PET-AC analvzed using were nitrogen adsorption/desorption isotherms at -196.15°C (Micro Active for ASAP 2460, Norcross, USA). The surface area was calculated with the Brunauer Emmett Teller theory, and the total pore volume was calculated using the adsorbed N₂ volume at high relative pressure. The surface morphology of PET-AC was taken by scanning electron microscopy (Hitachi SU8030, Tokyo, Japan). The surface function group was presented with the Fourier Transform Infrared (FT-IR, Perkin Elmer System 2000). The carbon, hydrogen, nitrogen, and sulfur contents were determined by the elemental analyzer (LECO CHN628 Series Sulfur, USA).

3. RESULTS AND DISCUSSION

3.1 PET-AC Optimum Production Condition

It was found that at the higher level of all factors, the provided iodine adsorption capacity was at its highest level. The obtaining PET-AC provided high iodine adsorption capacity ranging from 759.38 - 1,125.34 mg/g. The relationship between iodine adsorption capacity and all factors was analyzed in a linear form. The fitness of the model and experimental data was in agreement (R²adjusted 0.9918 and R²-predicted 0.9866). The R²adjusted and the R²-predicted are high, indicating that there is a well equation in this model between the experiment data and the model prediction [7, 13,38, 43]. The iodine adsorption prediction model was obtained from the analysis of the results using Design Expert 11 as shown in Eq. (1). It was indicated that all observed factors, A, B and C are significant (P-value less than 0.05). The optimal condition factors were 3 by weight, 900 watts, and 30 mins for ratio, microwave power, and heating time, respectively.

$$\begin{aligned} Iodine \ adsorption &= +888.06 + 52.66A + 105.41B \\ &+ 71.67C \end{aligned} \tag{1}$$

The analysis of variance (ANOVA) of iodine adsorption capacity is shown in Table 2. The main effects of each factor show *P*-values < 0.05 representing significant factors. The sum of squares of each factor indicated that it was corresponding to the iodine adsorption capacity of the PET-AC.

The normal plot of residuals for iodine adsorption capacity is plotted in order to investigate

Source	Sum of	df	Mean square	F-value	P-value
	squares				
Model	2.282E+05	3	76082.71	445.33	< 0.0001
А	33271.64	1	33271.64	194.74	< 0.0001
В	1.333E+05	1	1.333E+05	780.42	< 0.0001
С	61643.37	1	61643.37	360.81	< 0.0001
Pure error	1366.78	8	170.85		
Cor total	2.296E+05	11			

Table 2 Analysis of variance (ANOVA) for fractional factorial design

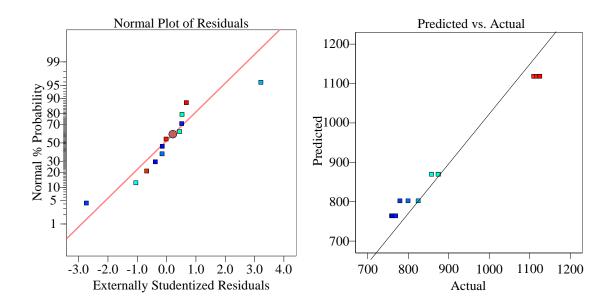


Fig.1 Normal plot of residuals for iodine adsorption capacity (left) and plot of actual response versus predicted response for iodine adsorption capacity (right)

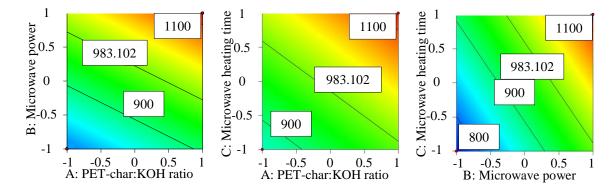


Fig.2 Iodine adsorption capacity contour plot of PET-AC

the normality of data [46-48]. Fig.1 shows well normal distribution residuals data and the predicted (obtained from Eq.1) versus actual plots of iodine adsorption capacity. It indicated that the predicted values agreed well with the actual data, thereby indicating high model ability [7,43,49,50].

The response surface of the combined effect of the overall factors are shown in Fig.2. The iodine adsorption capacity of PET-AC was enhanced with the increase of factor levels due to the improvement in the porosity of the activated carbon.

3.2 PET-AC Characteristics

3.2.1 N₂ adsorption-desorption isotherm

The N_2 adsorption-desorption isotherms of the produced PET-AC indicated type IV with the hysteresis loop (Fig.3) based on the classification isotherm of The International Union of Pure and Applied Chemistry (IUPAC) classification [51,52]. It was demonstrated that PET-AC was mainly mesoporous [15,51-53].

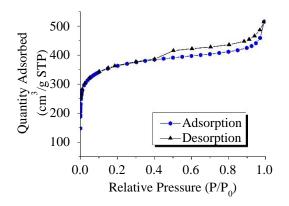


Fig.3 Nitrogen adsorption-desorption isotherm of PET-based activated carbon obtained from the optimum production condition

The pore size distribution of PET-AC is shown in Fig.4. PET-AC achieved the optimum condition with the average pores size of 2.03 nm, the BET surface area of 1,345 m²/g, and the total pore volume with 0.68 cm³/g. The obtained PET-AC had a relatively high surface area compared to the previous reports, as shown in Table 3.

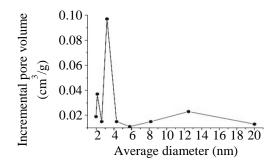


Fig.4 Pore size distribution of PET-AC obtained from the optimum production condition

3.2.2 Physical and chemical characteristics

The SEM morphology of the PET-char and PET-AC gained from the optimum production condition are shown in Fig.5. It can be seen that PET-AC exhibits higher pore distribution than PET-char after the chemical activation by the microwave heating process.

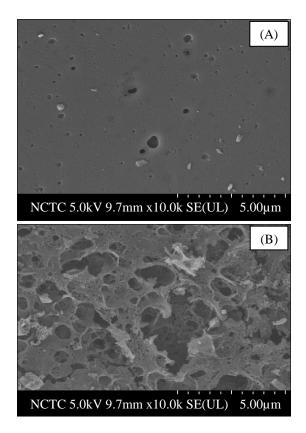


Fig.5 Scanning electron microscopy images of PET-char (A) and PET-AC (B)

The elemental analysis of PET, PET-char and PET-AC are shown in Table 4. It was found that the PET-AC shows a higher carbon content than the PET-char due to the elimination of some amount of oxygen from the materials [39].

The FT-IR spectra of the PET-char and PET-AC are shown in Fig.6. PET waste has large amounts of O-H stretching, CH₃, CH₂, CH groups, C=O, and aromatic groups [54,55], while this study shown the surface functional groups of PET-char have C=O, C-N and C-Cl bonds. Hence, PET-AC shown bands at 1,135 cm⁻¹ and 1,496 cm⁻¹, were observed for C=C and C=O in the aromatic and ether structures, respectively, because the others have been removed during the microwave heating process [15,51].

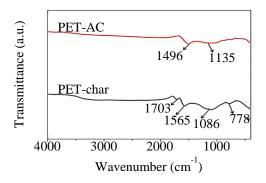


Fig.6 FT-IR spectra of the PET-char and PET-AC

Raw material	Activating agent	Heating method	$S_{BET}(m^2/g)$	$V_{T}(cm^{3}/g)$	Reference
PET	КОН	Microwave	1,345	0.68	This work
PET	Steam	furnace	1,170	0.63	[53]
PET	Steam	furnace	1,524	0.60	[26]
PET	CO_2	furnace	1,850	0.74	[26]
PET	CO_2	furnace	790	0.43	[15]
PET	CO_2	furnace	1,110	0.39	[52]

Table 3 Pore structure of different activated carbons

Table 4 Ultimate analysis of PET-based materials

Sample		Reference				
	С	Н	0	Ν	S	
PET	59.62	6.18	34.19	-	0.01	This work
PET-char	55.76	1.43	42.55	0.26	-	This work
PET-AC	80.08	1.45	17.94	0.52	-	This work
PET-50S	96.49	0.42	2.97	0.08	-	[26]
(steam activation)						
PET-50C	97.31	0.42	2.21	0.02	-	[26]
(CO ₂ activation)						

4. CONCLUSION

The optimum condition for the preparation of PET-AC was 3 by weight, 900 watts, and 30 mins for the ratio, the microwave power, and the heating time, respectively. The maximum iodine adsorption capacity was 1,125.34 mg/g, and surface area was 1,345 m²/g. It was completed that PET bottle waste can be converted to a highly valuable activated carbon by using KOH and the microwave activation.

5. ACKNOWLEDGEMENTS

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