Iraqi Journal of Chemical and Petroleum Engineering
Iraqi Journal of Chemical and Petroleum Engineering
Vol.15 No.1 (March 2014) 33- 42
ISSN: 1997-4884
University of Baghdad
College of Engineering

Optimization of Activated Carbon Preparation from Date Stones by Microwave Assisted K_2CO_3 Activation

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Abstract

The preparation of activated carbon (AC) from date stones by using microwave assisted K_2CO_3 activation was investigated in this paper. The influence of radiation time, radiation power, and impregnation ratio on the yield and methylene blue (MB) uptake of such carbon were studied. Based on Box-Wilson central composite design, two second order polynomial models were developed to correlate the process variables to the two responses. From the analysis of variance the significant variables on each response were identified. Optimum coditions of 8 min radiation time, 660 W radiation power and 1.5 g/g impregnation ratio gave 460.123 mg/g MB uptake and 19.99 % yield. The characteristics of the AC were examined by pore structure analysis, and scanning electron microscopy (SEM).The BET surface area and total pore volume were indentified to be 1144.25 m²/g and 0.656 m³/g, respectively.

Keywords: Activated Carbon, Methylene blue, Microwave, Optimization, Adsorption

Introduction

Activated carbon (AC) is a carbonaceous material possessing a higher porosity due to which it is commonly used in variety of application, concerned principally with the removal of chemical species by adsorption form the liquid or gas phase [1]. However, AC is expensive which limits its large-scale application. A potential method to reduce its cost is to produce it from low-cost material such as agricultural by-products, which has attracted an increasing research interest in recent years [2]. From the literature, many studies have been carried out to prepare low cost AC from agricultural wastes such as cotton stalk [3], grapevine rhytidome [4], sewage sludage [5], mangosteen peel [6], and pineapple peel [7]. The preparation of AC involves of two stages, namely pyrolysis and activation [8]. In the first stage, suitable carbon precursors are carbonized under inert atmosphere at moderate temperature to release volatile matters and produce chars with rudimentary pore structures. Subsequently, the resulting chars are subjected to partial gasification at higher temperature (usually above 900˚C) with oxidizing gases, to produce activated carbons with well-developed and accessible internal porosities [9].Nevertheless, in some cases, the thermal process may take long processing time, involves high energy consumption, requires larger equipment size and generates improper heating rate, thereby resulting in a detrimental effect on the quality of the prepared activated carbons [10]. Furthermore, there is a considerable risk of overheating or even thermal runaway of local sample, leading to the complete combustion of the carbon [11]. Therefore, it is necessary to find a rapid and easy route for the preparation of AC. Although microwave heating is today a mature technique which finds wide applications in the area of material science, food processing and analytical chemistry [12], there are relatively few studies in this field. Microwave heating has the advantages of rapid temperature rise, uniform temperature distribution and saving of energy over conventional thermal method [13].

Methylene blue, the most commonly used substance in the dying process was chosen as the model adsorbate in this study, due to its potential risk towards the environmental pollution and ecosystems.

Currently no study has been done on optimization of the production of activated carbon from Date stones using the response surface methodology (RSM) approach. RSM has been found to be a useful tool to study the interaction of two or more factors [14]. A standard RSM design called Box-Wilson central composite design is suitable for fitting a quadratic surface and it helps to optimize the effective parameters with a minimum number of experiments, as well as to analyze the interaction between the parameter [15]. RSM has just recently been used for the optimization of activated carbon production from rattan sawdust [14], Tamarind wood [16] and Turkish lignite [17] by chemical activation whereas oil palm empty fruit bunch [18] and coconut husk [19] by physiochemical activation method. The goal of this work is to optimize the preparation conditions of AC from Date stones for the removal of MB dye from aqueous solution. The effects of radiation time, radiation power and impregnation ratio are also studied simultaneously to obtain a high MB uptake using the Box-Wilson central composite design.

Materials and Method

Materials

Date stones were used as the precursor for the preparation of AC. The date stones as received were first washed with water to get rid of impurities, dried at 110 ˚C for 24 h, crushed using disk mill, and sieved. Fraction with particle size of 300-600 μm was selected for the preparation. Potassium carbonate (purchased from Didactic company, Espana) of purity 99.9 % was used as an adsorbate. MB has a chemical formula of $C_{16}H_{18}N_3SCl$, with molecular weight of 319.86 g/mol. All other chemical used such as hydrochloric acid were from analytical grades.

Preparation of Activated Carbon

A weighed amount (2g) of dried date stones was mixed with 10 ml of K_2CO_3 solution at various impregnation ratios (0.5-2.5 g/g) for 24 h at room temperature. The samples were next placed in an oven (Model IH-100, England) at 110 ˚C until completely dried and stored in desiccators. The dried samples were activated by using a quartz glass reactor (2.5 cm diameter x 12.5 cm length). The reactor was sealed at bottom and open from the top end to allow for the escape of the pyrolysis gases. A modified microwave heating oven (MM717 CPJ, China) was used for preparation as shown in Fig. 1. The upper surface of oven had a removable cover connected to a stainless steel pipe of 5 mm inside diameter from which pyrolysis gases were exit. The reactor was placed inside the oven and held at different radiation powers (540-700 W) for different radiation times (4-12 min). After activation, the sample was withdrawn from the oven and allowed to cool. For isolation of residual K_2CO_3 activator, the sample was mixed with 0.1M HCl solution at 10 mg/L solid to liquid ratio. The mixture was left overnight at room temperature, and then filtered and subsequently the sample was repeatedly washed with distilled water to remove residual organic matters and alkalis, until the PH of filtrate reached 6.5-7.0. After that, the sample was dried at 110 ˚C for 24 h, and subsequently was weighed. Finally the sample was stored in tightly closed bottles.

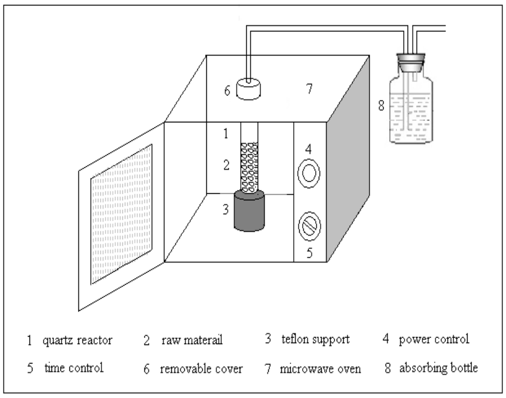

Fig. 1, Schematic diagram of microwave unit for preparation of activated carbon

Process Performance

The performance of chemical activation process was determined by the product yield, along with its uptake for MB. The yield and MB uptake are determined as follows.

Yield

The yield is defined as the ratio of final weight of the obtained product after washing and drying to the weight of dried precursor initially used. The yield of AC was calculated based on the following equation:

$$\text{Yield}\ (\%) = \frac{W_f}{W_o} \times 100 \qquad \ldots(1)$$

Where W_f and W_o are the weight of final AC product (g) and the weight of dried date stones (g), respectively.

MB Uptake

The MB uptake or adsorption capacity of prepared AC was determined by performing batch adsorption tests in 100 mL Erlenmeyer flasks where 50 mL of MB aqueous solutions with initial concentration of 250 mg/L was placed in each flask. The pH of the solution was 6.85 without any pH adjustment. 0.005 g of each of the prepared AC, with average particle size of 0.25 mm, was added to each flask and kept in a shaker of 200 rpm at room temperature for 24 h to reach equilibrium. Aqueous samples were taken from the solutions and the concentrations were analyzed. All samples were filtered prior to analysis in order to minimize interference of the carbon fines with the analysis. The concentrations of MB in the supernatant solutions were determined using UV-Visible Spectrophotometer (Shimadzu UV-160A) at its maximum wave length of 664 nm. The MB uptake at equilibrium, q_e (mg/g), was calculated by the following equation:

$$q_e = \frac{(C_o - C_e)\,V}{W} \qquad \ldots(2)$$

Where C_o and C_e are initial and equilibrium concentrations of the MB (mg/L), respectively, V is the volume of the aqueous MB solution (L), and W is the weight of AC used (g).

Experimental Design

In order to organize the experiments of AC preparation from date stones, a standard RSM design, known as Box-Wilson central composite design was adopted. This design can reduce the number of experimental trails needed to evaluate multiple parameters and their interactions [20]. Generally, the

Box-Wilson design consists of 2^n factorial points, 2n axial points, and one center point, where n is the numbers of variables. In order to design the experiments, the operating range of variables must be specified, thus: radiation time X_1 (4-12 min), radiation power X_2 (540-700 W), and impregnation ratio X_3 (0.5-2.5 g/g). The total number of experiments N is computed according to the following equation:

$$N = 2^n + 2n + 1 \qquad \ldots(3)$$

The relationship between the coded variable and the corresponding real variable is as follows:

$$X_{coded} = \left(\frac{X_{actual} - X_{center}}{X_{center} - X_{min}} \right) * \sqrt{n} \qquad \ldots(4)$$

Characterization of Activated Carbon

The surface area, pore volume, and average pore diameter of AC prepared under optimum preparation condition were determined by using Micromeritics ASAP 2020 Volumetric adsorption analyzer which based on the continuous flow method originally developed by Nelsen &Eggertsen. For BET surface area analysis, a mixture of nitrogen balance helium is passed through the reference channel of the TCD detector to the sample housed within a flow-through glass cell and finally into the TCD analytical channel. The signal produced by the TCD detector is collected by the microprocessor control board, integrated and stored in the memory file.

Microscopic appearance of AC and date stones was studied by scanning electron microscope (SEM). Microscopic image of AC and precursor were obtained by SEM (VEGA3 TESCAN).

Results and Discussion

Model Fitting and Statistical Analysis

The experimental values of yield and MB uptake of AC are fitted with a second order polynomial mathematical model. The general form of this model as a function of X_1, X_2, and X_3 is represented by the following equation:

$$y = B_0 + B_1X_1 + B_2X_2 + B_3X_3 + B_4X_1X_2 + B_5X_1X_3 + B_6X_2X_3 + B_7X_1^2 + B_8X_2^2 + B_9X_3^2 \qquad \ldots(5)$$

A nonlinear least-squares regression program based on Gauss-Newton method was used to fit Eq. 5 to the coded data and experimental responses. The fitted response surface of Eq. 5 is:

$$y_1 = 15.80434 - 4.69503X_1 - 3.89331X_2 - 3.20738X_3 + 0.26816X_1X_2 + 0.24511X_1X_3 - 0.18184X_2X_3 + 1.310993X_1^2 + 1.189653X_2^2 + 0.775462X_3^2 \qquad \ldots(6)$$

$$y_2 = 451.4909 + 1.774731X_1 + 4.220162X_2 + 2.151651X_3 - 0.21509X_1X_2 + 0.000389X_1X_3 - 0.17579X_2X_3 - 11.4624X_1^2 - 2.14966X_2^2 - 34.1106X_3^2 \qquad \ldots(7)$$

The analysis of variance (F-test) was used for testing the significance of each effect in Eqs. 6 And 7. An estimate of the variance S_b^2 is obtained by dividing the experimental error variance S_r^2 by the sum of squares of each effect ΣX^2, as follows:

$$S_b^2 = \frac{S_r^2}{\Sigma X^2} = \frac{(\Sigma e^2)/(N-n)}{\Sigma X^2} \qquad \ldots(8)$$

The significance of effects may be estimated by comparing the values of the ratio (B^2/S_b^2) with the critical value of the F-distribution at 95 % confidence level ($F_{0.95}$=6.61). If the ratio B^2/S_b^2 > 6.61 then the effect is significant. From this result, it appears that the interaction effects are not significant and the radiation time has the greatest effect on the yield while radiation power has the greatest effect on MB uptake of prepared AC, 744.051 and 274.209, respectively. The best response functions in terms of actual variables are then conveniently written as follows:

$$y_1 = 388.2609 - 6.22825X_1 - 0.80217X_2 - 12.9309X_3 + 0.252326X_1^2 + 0.000579X_2^2 + 2.463893X_3^2 \quad \ldots(9)$$

$$y_2 = -430.879 + 35.776387X_1 + 1.5131206X_2 + 313.42911X_3 - 2.184567X_1^2 - 0.001146X_2^2 - 103.2372X_3^2 \quad \ldots(10)$$

Eqs. 9 And 10 were used to construct graphical representations of yield and MB uptake versus each variable within the variables ranges used in forming the models.

Process Optimization

At optimum conditions, the prepared AC should have a high yield and high MB uptake. However, it is difficult to optimize both these responses under the same condition because the interest region of variables is different, when MB uptake increases, carbon yield decrease and vice versa. Therefore, the optimum conditions have been determined depending on achieving high MB uptake. The optimum operating conditions has been determined by differentiating both sides of Eq. 10 for each independent variable and equating the derivative to zero [21]. Thus, the optimum conditions corresponding to a maximum MB uptake are 8 min radiation time, 660 W radiation power, and 1.5 g/g impregnation ratio. At these conditions, 460.12 mg/g MB uptake with 19.99 % yield were reported experimentally.

Effect of Process Variables

Effect of Radiation Time

The effect of radiation time on yield and MB uptake of prepared AC at different radiation powers and optimum impregnation ratio is shown in Figs. 2 and 3, respectively.

Fig. 2 Shows that the yield of AC decreases with increasing radiation time. An increase in radiation time from 4 to 12 min at optimum conditions of 660 W radiation power and 1.5 g/g impregnation ratio leads to a decrease in yield of AC from 26.26 to 8.69 %. A steep decrease occurs after 10 min, this is probably due to rapid evolution of volatile materials to form stable compounds as explained by Foo and Hameed [22]. They showed that a steep decrease in yield occurs after 7 min for production of AC from coconut husk by microwave KOH activation.

MB uptake of AC increases with radiation time and reaches at maximum of 452.66 mg/g at 8 min and optimum conditions of 660 W and 1.5 g/g. thereafter it decreases, as shown in Fig. 3.

The decrease in MB uptake with activation above 8 min is probably due to turn off mesopores to macropores which are not effective for MB adsorption, as explained by Foo and Hameed [23]. Figs. 2 and 3 show that there are no interactions between radiation time and radiation power.

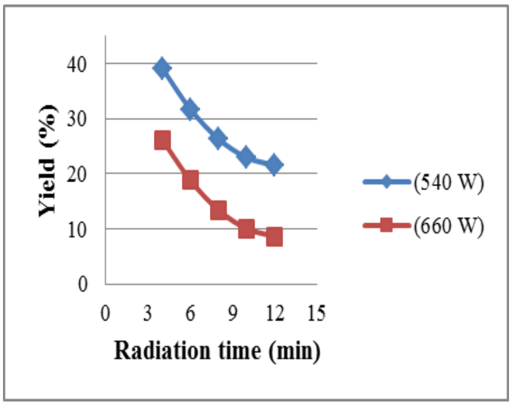

Fig. 2, Effect of radiation time on yield (impregnation ratio of 1.5 g/g)

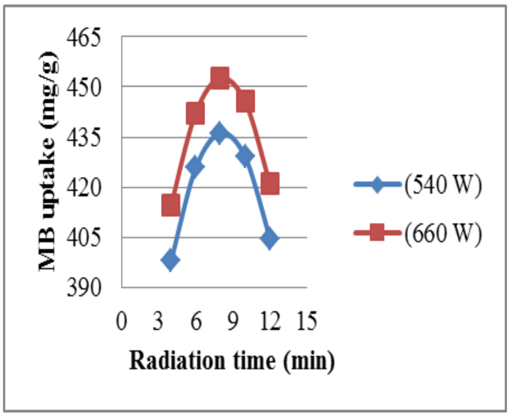

Fig. 3, Effect of radiation time on MB uptake (impregnation ratio of 1.5 g/g)

Effect of Radiation Power

The yield and MB uptake of prepared AC versus radiation power at different impregnation ratios and optimum radiation time is shown in Figs. 4 and 5, respectively. It can be seen from Fig. 4 that, as the radiation power increases from 540 to 700 W at optimum conditions of 8 min radiation time and 1.5 g/g impregnation ratio, the yield decreases from 26.33 to 12.81 %. This may be due to the loss of the volatile materials with increasing power. Beyond 660 W a lower rate of yield decrease was noticed where a stable structure is formed. Fig. 5 Shows that the MB uptake increases with radiation power up to 660 W, then decreased. An increase in power from 540 to 660 W at optimum conditions of 8 min radiation time and 1.5 g/g impregnation ratio causes an increase in MB uptake from 436.17 to 452.66 mg/g. The decrease in adsorption ability with further increase in power might be due to the sintering effect at high power, followed by shrinkage of the char, and realignment of the carbon structure which resulted in reduced pore areas as well as volume, as explained by Deng et al. [23] for MB adsorption on AC prepared from cotton stalk by microwave assisted KOH activation, who reported an optimum radiation power of 680 W.

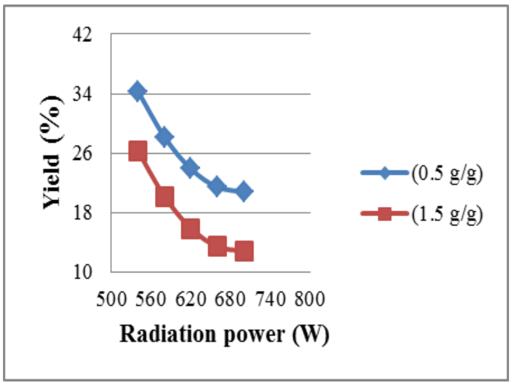

Fig. 4, Effect of radiation power on yield (radiation time of 4 min)

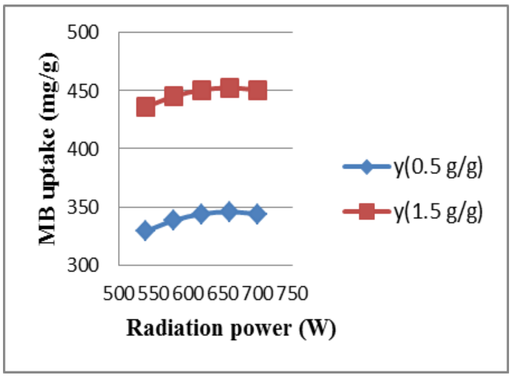

Fig. 5, Effect of radiation power on MB uptake (radiation time of 4 min)

Effect of Impregnation Ratio

Figs. 6 and 7 show the effect of impregnation ratio on yield and MB uptake of prepared AC, respectively at different radiation times and optimum radiation power.

It is noticed that, as the impregnation ratio increases the yield decreases, as shown in Fig. 6. An increase in impregnation ratio from 0.5 to 2.5 g/g at 8 min radiation time and 660 W radiation power leads to a decrease in yield from 21.42 to 10.34 %. This decrease is due to the continuous removal of tar material from the pores. The decreasing rate of yield is lowered beyond an impregnation ratio of 1.5 g/g where a stable structure is formed. This behavior agrees with results obtained by Sudaryanto et al. [25] for AC production from cassava peel by chemical activation with potassium hydroxide. Fig. 7 shows that the MB uptake of AC increases with impregnation ratio up to 1.5 g/g, then decreased. The increase in impregnation ratio from 0.5 to 1.5 g/g at 8 min and 660 W leads to an increase in MB uptake from 345.70 to 452.66 mg/g. More increase in the concentration of K_2CO_3activator perhaps leads to the excessive dehydration and destruction of mesopores and turning them to larger pores which reduces the adsorption efficiency, as explained by Foo and Hameed [26] for adsorption of MB on AC prepared from oil palm empty fruit bunch by microwave assisted KOH activation. They reported an optimum impregnation ratio of 1 g/g.

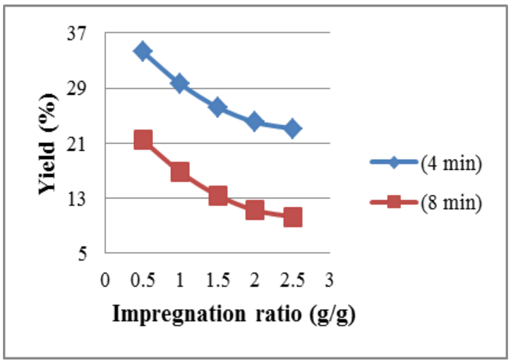

Fig. 6, Effect of impregnation ratio on yield (radiation power of 660 W)

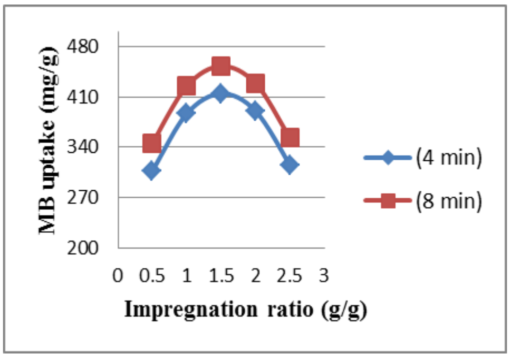

Fig. 7, Effect of impregnation ratio on MB uptake (radiation power of 660 W)

Characterization of Activated Carbon

BET Surface Area and Pore Volume

The BET surface area, total pore volume and average pore diameter of AC prepared at optimum conditions are found to be 1144.25 m^2/g, 0.656 m^3/g and 3.004 nm, respectively. These values higher than that reported by Foo et al. [28] who show the BET surface area and total pore volume of activated carbon from date stones by KOH activation were 865 m^2/g and 0.468 m^3/g, respectively. The average pore diameter of 3.004 nm indicates that the AC prepared is in the mesopores region according to the IUPAC classification [27]. The chemical activation process has contributed to the high surface area and total pore volume of the prepared AC. Table 1 shows the comparison between the other precursors of prepared AC of structure textural.

Surface Morphology

Figs. 8a and b show SEM images of date stones and AC. It can be found that the surface of date stones is dense, planar, constricted and blocked by deposited tray substance. However, the microwave irradiation sample demonstrated a well-developed and uniform surface, forming an orderly pore structure. The development of porosity is associated with gasification according to the following reduction reactions [29]:

$$K_2CO_3 + 2C \rightarrow 2K + 3CO$$

$$K_2CO_3 \rightarrow K_2O + CO_2$$

$$K_2O + 2C \rightarrow 2K + CO$$

It was assumed that metallic potassium K formed during the gasification process would diffuse into the internal structure of carbon matrix widening the existing pores and created new porosities. Fig. 4b also shows that the surface of prepared carbon contains some cavities which are resulted from the evaporation of impregnated K_2CO_3 derived compounds, leaving the space previously occupied by the reagent. Theses cavities provide channels for the adsorbate molecules to access the micropores and mesopores inside a carbon particle.

Table 1, Comparison of pore structures of Carbons prepared from various agricultural wastes by microwave heating

Precursor	Time (min)	Power (W)	Ratio (g/g)	Chemical agent	S_{BET} (m^2/g)	V_t (cm3/g)	Ref.
Date stones	8	660	1.5	K_2CO_3	1144.25	0.656	This work
Date stones	8	600	1.75	KOH	856	0.468	[28]
Oil palm empty fruit bunch	15	360	0.75	KOH	807.54	0.450	[26]
Cotton stalk	8	400	50(vol%)	H_3PO_4	652.82	0.476	[3]

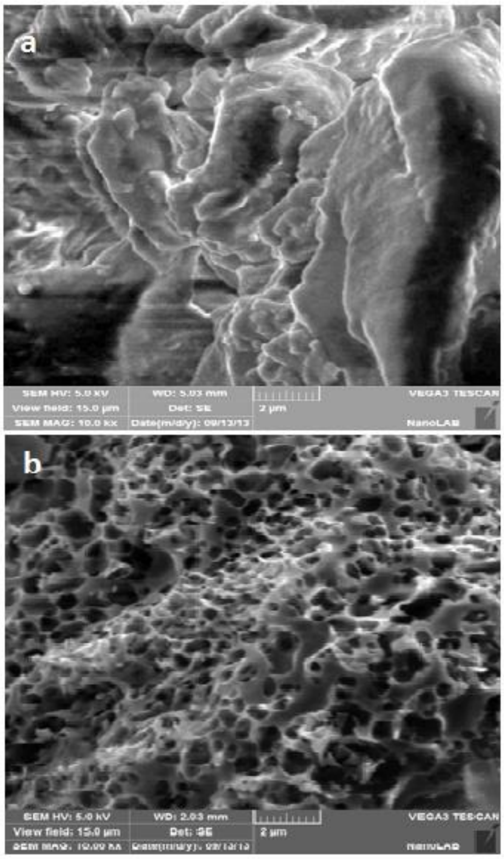

Fig. 8, SEM micrographs (10 kx) of date stones (a) and AC (b)

Conclusion

Activated carbons were prepared from Date stones by microwave assisted K_2CO_3 activation. 460.1234 mg/g MB uptake with corresponding yield of 19.99 % were obtained at optimum conditions of 8 min radiation time, 660 W radiation power, and 1.5 g/g impregnation ratio. Also, the surface area, total pore volume and average pore size of AC were 1144.25 m^2/g, 0.656 m^3/g and 3.004 nm, respectively .Box-Wilson central composite design was adopted for arrangement of preparation experiment. Two second order polynomial models were successfully used to correlate the process variables to the two responses.

Acknowledgement

We gratefully acknowledge Department of Chemical Engineering and University of Baghdad for assist and support of this work.

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