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RESEARCH ARTICLE

Preparation of activated carbon from residues coffee by physical activation: using Response surface methodology

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Abstract

The intention of this work is to realize a planned study of the preparation conditions of the activated carbon from residues coffee based physical activation under steam (CAC). The influence of activation temperature, activation time, and heating rate (HR) on the carbon yield, iodine number and methylene blue (MB) adsorption capacity of such carbons were studied. Based on a central composite design (CCD) was used to optimize the preparation conditions of activated carbons from residues coffee. For CAC, an optimum condition of 732.5 °C activation temperature, 57.68 min activation time and 25.11°C/min heating rate gave 50.29 % yield, 608.50 mg/g iodine number and 221.98 mg/g MB uptake. The optimum activated carbon showed a BET surface area of 772.49 m^2/g and average pore 24.7 Å. It was observed that the experimental values obtained were in good agreement with the values predicted from the models, with relatively small errors between the predicted and the actual values.

INTRODUCTION

In current research, the Response surface methodology (RSM) is a common and widely applied technique to optimize the processes and evaluate the relative significance of the parameters and their interactions. RSM benefits, the researchers get optimum condition using the first or second order polynomial equations to the experimental responses obtained in the experimental design. The principal advantage of RSM is the reduced number of experimental trials needed to evaluate multiple parameters and their interactions (A.A. Ahmad et al, 2009,Alexandro M.M. Vargas et al, 2010, Marcos Almeida Bezerra et al, 2008, Deniz Bas, Ismail H. Boyacı ,2007).

In this paper, RSM optimization has been used to the synthesis of activated carbon. Different materials are used to produce activated carbon such as coconut shell, palm shell, Rice Husk and olive-waste (M.K.B. Gratuito et al, 2008, Donni Adinata et al, 2007, Nasehir Khan E M Yahaya et al, 2010, A. Baçaoui et al, 2001).

Basically, two methods for preparing an activated carbon: physical activation and chemical activation. In the physical activation, there are two steps: carbonization and activation by steam or carbon dioxide. In the chemical activation, a raw material is impregnated with an activating reagent (such as $ZnCl_2$, H_3PO_4, KOH, K_2CO_3 and NaOH) and the impregnated material is heat-treated under an inert atmosphere.

Activated carbons have been extensively used as adsorbents processes for aqueous or gaseous solution system (K. Ravikumar et al, 2006, S. Sumathi et al, 2009, Mohd. Rafatullah et al, 2010). The adsorption compared with other processes appears to be a preferable method in view of its efficiency and easily available with which it can be applied in the treatment of organic compound containing wastewater (Mohd. Rafatullah et al, 2010).

The objective of this research was to optimize the preparation conditions of activated carbon from residues coffee by physical activation under steam. The preparation of activated carbon is influenced by three factors such as temperature activation, activation time, and heating rate and its consequent application to remove methylene blue based on the RSM experimental design approach. Additionally, the nitrogen adsorption isotherm of the activated carbon prepared under optimum conditions was analyzed to characterize the porous structures.

2. Materials and Methods

2.1 Preparation of activated carbon

Residues coffee used for preparation of activated carbon (CAC), and was first washed with distilled water to remove the impurities and surface adhered particles and then dried at 105 °C for 24h to remove the moisture. The carbonization was carried out. A quantity of residues coffee (20g) was introduced into a muffle furnace which was then heated to the desired temperature 400°C and conserved for 1h at the final temperature in a nitrogen flow. The activation process was carried out. The precursor was obtained heating the mixtures in a flow of steam add a flow of nitrogen (N_2), the flow rate of (N_2) and steam was kept constant, in all experiments. The condition experimental (temperature activation, time activation and heating rate) of preparation the activated carbon (CAC) are representing in table 1. The final products after activation washed with hot deionized water until the pH of the washing solution reached 6–7, and then dried at 110 °C for 24h.

The product is subject to characterization of carbon yield and iodine number. The activated carbon yield (Y_1) was calculated based on Eq. (1). The iodine adsorption capacity is represented as an iodine number (Y_2) which indicates milligrams of iodine adsorbed by a gram of activated carbon (mg/g) (Eun Jung Im et al, 2008).

$$\text{Yield } (\%) = (W_c/W_o) \times 100 \quad (1)$$

Where W_c is the dry weight (g) of final activated carbon and W_o is the dry weight (g) of precursor.

2.2 Design of experimenters

The parameters for preparing the activated carbon was studied with a standard response surface methodology (RSM) design called a central composite design (CCD). This method is suitable for fitting a quadratic surface and it helps to optimize the effective parameters with a minimum number of experiments and also to analyze the interaction between the parameters (V. N. Ganvir, Atul P. Dwivedi, 2012). RSM is a collection of mathematical and statistical techniques that are useful for modeling and analysis of problems in which a response of interest is influenced by several variables (J.N. Sahu et al, 2010). Generally, the CCD consists of a 2^n factorial runs with 2n axial runs and n_c center runs (Eq.2).

$$N= 2n +2n+ n_c = 2^3+2(3) + 6 = 20 \quad (2)$$

In this study, the activated carbons were prepared using chemical activation method of varying the preparation variables using the CCD. The variables studied were (i) x_1, activation temperature; (ii) x_2, activation time and (iii) x_3, heating rate. These three variables together with their respective ranges were chosen based on the literature and some preliminary studies.

Activation temperature, activation time and heating rate were found to be important parameters affecting the characteristics of the activated carbons produced (Arash Arami-Niya et al, 2012). For each categorical variable, a 2^3 full factorial central composite design for the three variables, consisting of 8 factorial points, 6 axial points and 6 replicates at the center points were employed, indicating that altogether 20 experiments were required, as calculated from (Eq.3)

$$Y_i = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j \quad (3)$$

Where Y_i is the predicted response; x_i to x_j are the coded values of the activated carbon preparation variables; β_0, the constant coefficient; β_i, the linear term coefficients; β_{ij} the interaction coefficients and β_{ii}, the quadratic coefficients. The complete design matrices of the experiments performed, together with the results obtained, are shown in table 2. The program Minitab16 was used to develop.

2.3 Batch equilibrium adsorption

The adsorption isotherms of methylene blue (MB) (C_{16} $H_{18}ClN_3S$) onto the activated carbon samples were obtained by adding 0.1g of carbon to flasks containing 100 ml of aqueous solutions with different initial concentrations of either methylene blue. These flasks were kept in a thermostat shaker for 24 hours. When the equilibrium time was reached, the suspensions were filtered and the equilibrium concentrations were determined. In the case of methylene blue, the concentrations were determined by UV-vis spectrophotometer (Schimadzu-1700) at the maximum absorbance wavelength of MB (664 nm). The adsorption capacities Q_e (mg/g) of the carbon samples for methylene blue (Y_3) were calculated by applying the Langmuir Eq. (4).

$$Q_e = (C_i - C_e)\, V/W \tag{4}$$

Where C_i and C_e (mg/l) are the initial and equilibrium concentration of MB, respectively. V (L) is the volume of solution and W (g) is the weight of adsorbent used.

2.4 Physical Characterization

The surface area and pore volume of the optimum CAC was determined by N_2 adsorption isotherms at 77K using an automatic adsorption unit (ASAP 2020). The surface area was determined by the application of Brunauer–Emmett–Teller (BET) and the total pore volumes (V) were estimated by the volume of N_2 adsorbed at high relative pressures near unity (≈0.99) (F. Brouers et al (2005)).

3. Results and discussion

3.1 Preparation of activated carbons

Central composite design (CCD) was used to develop a correlation between the activated carbon preparation variables to the carbon yield, iodine number, and removal of methylene blue. Table 2 shows the complete design matrixes together with both the response values obtained from the practical experiment. The CAC yield (Y_1) obtained ranged from 20 to 73%, the iodine number (Y_2) was found to range from 302 to 769 mg/g and adsorption of MB (Y_3) get it 135 to 306 mg/g. The performance of the model developed was evaluated based on the correlation coefficients (R^2), which were 98.9% for carbon yield, 99.56% for iodine number and 98.1% for MB. These R^2 values were relatively high, indicating a good agreement between the experimental data and the model prediction. The final empirical formula models for the CAC yield (Y_1), iodine number (Y_2) and adsorption of MB (Y_3) in terms of code factors are represented by Eq (5), (6) and (7), respectively.

$$Y_1 = 126.319 - 0.134336x_1 - 0.365919x_2\ 1.28223x_3 + 0.0000736x_1^2 + 0.00180395x_2^2 + 0.0617995x_3^2 - 0.000292x_{12} - 0.000250x_{13} + 0.00555556x_{23} \tag{5}$$

$$Y_2 = -135.436 + 1.67309x_1 + 1.56337x_2 - 2.34781x_3 - 0.00125041x_1^2 - 0.0177157x_2^2 - 0.336066x_3^2 + 0.00358333x_{12} + 0.0135x_{13} + 0.0207407x_{23} \tag{6}$$

$$Y_3 = 40.9614 + 0.0360367x_1 + 1.22704x_2 + 4.47645x_3 + 0.00010735x_1^2 + 0.00316815x_2^2 - 0.131077x_3^2 - 0.000222\, x_{12} + 0.00516667x_{13} - 0.0688889x_{23} \tag{7}$$

3.2 Activated carbon Yield

The yield of activated carbon is also an important parameter as it quantifies the amount of final product. The Heating rate shows the most significant effect on activated carbon yield. Table 2 represents the yield of activated carbon for each of the experiments. Fig. 1 a, shows the effect of activation temperature and activation time on the CAC yield, activation time fixed at zero level (x_3 = 12.5 min/°C), while Fig. 1b, demonstrates the effect of activation time and heating rate on the same response, with activation temperature fixed at zero level (T = 600°C). The carbon yield was found to decrease with increasing temperature activation and activation time. A maximum carbon yield is obtained at low temperature activation and activation time (A.A. Ahmad et al, 2009, Mohammed Danisha et al, 2013). The ANOVA of the quadratic model of activated carbon yield is shown in Table 3. An F-value of 99.57 and Prob>F less than 0.000 prove that the model is significant. The value of the model terms Prob>F less than 0.05 indicates the three process parameters x_1,x_2 ,x_3 and interaction parameters of x_1^2,x_2^2,x_3^2,x_{12}, x_{23} are significant model terms.

3.3 Iodine adsorption

For iodine adsorption (Y_2) in the other hand, activation temperature was found to have the greatest effect on it, with the highest F value of 251.98 in Table 3, whereas activation time and heating rate showed almost similar effects on the response, which were less significant compared to activation temperature. Fig.2a and Fig.2b illustrate the three-dimensional response surface which is constructed to show the interaction effects of temperature activation, time activation and heading rate on iodine number. The iodine number rapidly increased with the increase of temperature activation and time activation (Duan Xin-huia et al, 2012, A. Baçaoui et al, 2001).

3.4 MB adsorption

The ANOVA for the quadratic model of MB adsorption is shown in Table 3, where the F-value of 57.4 and Prob > F less than 0.000 prove that the model is significant. Generally, the value of model terms Prob > F less than 0.05 indicates that the model terms are significant. In this case x_1, activation temperature, x_2, activation time, x_3, heating rate and interaction parameters of x_2^2,x_3^2, x_{13}, x_{23} are significant model terms. As can be seen from fig.3 (a-b), MB adsorption capacity increases with increase in activation temperature and activation time. The highest adsorption capacity value was obtained when both the variables were at the maximum point within the range studied. Among all the three parameters chosen in the present study, heating rate is found to have the most significant influence on the MB adsorption capacity (A. Baçaoui et al, 2001, B.H. Hameed et al, 2008, O. Ioannidou, A. Zabaniotou, 2007, I.A.W. Tan et al, 2008). Indeed, MB molecule has a minimum molecular cross-section of about 0.8 nm, and it has been estimated the minimum pore diameter it can enter is 1.3 nm (S.S. Barton, 1987). This meant that when more mesopores were developed. More MB molecules could be adsorbed by the activated carbons. therefore enhancing the adsorption capacity of the activated carbons.

3.5 Process Optimization

The main reason of this paper is to determine the experimental time, activation temperature and heating rate required to prepare activated carbons from residues coffee. According to the calculation method used, to obtain this ideal carbon. To optimize all responses under the same conditions is difficult because the interest region of factors is different, thus, when Y_1 decreases all the other responses increase; therefore, in order to find a coperation, we have resorted to the 'function of desirability' using the software Minitab. The optimal point indicated by the model corresponds to an activation time of 57.38 min, an activation temperature 732.50 °C and heating rate 25.11°C/min. In order to test the validity of this method, we have prepared an activated carbon sample under the above experimental conditions. The characteristics of this sample are shown in Table 4 together with those calculated from the model. Good agreement between the predicted and experimental results validates the optimized conditions and reflects the existence of an optimal point.

3.6 Characterization of optimization activated carbon

The pore structure of the activated carbon is characterized by nitrogen adsorption at 77 K with an accelerated surface area and porosimetry system**.** Fig. 4 shows typical N_2 adsorption-desorption isotherms of optimization activated carbon obtained through the steam activation. The developer of micropores and mesopores can be clearly confirmed by the shape of the isotherms. The surface properties analyses were tabulated in Table 5. The Specific surface area using the BET method was 772.49m²/g, the average pore size was 24.70Å and pore volume was 0.477cm³/g

Table 1. Experimental factors and their levels

Variables	Symbol	Levels				
		- α (-1.682)	-1	0	1	+α (+1.682)
Activation temperature, (°C)	x_1	263.64	400	600	800	936.35
Activation time, (min)	x_2	-0.68	30	75	120	150.68
heating rate, (°C/min)	x_3	-0.11	5	12.5	20	25.11

Table 2. Experimental design matrix and results

Run	Levels			Activated carbon preparation variables			Yield, Y_1 (%)	iodine number, Y_2 (mg/g)	MB uptake, Y_3 (mg/g)
	x_1	x_2	x_3	Activation temperature, x_1 (°C)	Activation time, x_2 (min)	heating rate, x_3 (°C/min)			
1	1	1	-1	800	120	5	20	723	306
2	1	1	1	800	120	20	30	769	263
3	0	0	0	600	75	12.5	38	637	210
4	-1	1	-1	400	120	5	50	460	256
5	0	α	0	600	150.68	12.5	33	630	279
6	0	0	0	600	75	12.5	40	636	211
7	α	0	0	936.35	75	12.5	19	703	301
8	0	0	0	600	75	12.5	39	635	212
9	1	-1	1	400	30	20	71	338	156
10	1	-1	-1	800	30	5	47	553	207
11	0	0	0	600	75	12.5	38	635	212
12	-1	1	1	400	120	20	62	407	170
13	0	0	0	600	75	12.5	39	637	210
14	0	0	-α	600	75	-0.11	40	603	190
15	0	0	α	600	75	25.11	55	578	180
16	-α	0	0	263.64	75	12.5	73	302	135
17	0	-α	0	600	-0.68	12.5	63	455	169
18	-1	-1	-1	400	30	5	67	401	137
19	0	0	0	600	75	12.5	39	633	211
20	1	-1	1	800	30	20	50	553	245

Table 3. Analysis of variance (ANOVA) for responses

	for activated carbon yield					iodine number					for MB uptake				
Source	DF	SS	MS	F	P	DF	SS	MS	F	P	DF	SS	MS	F	P
Regression	9	4577.47	508.61	99.57	0.000	9	315866	35096	251.98	0.000	9	45811.2	5090.1	57.4	0.000
x_1	1	2750.63	2750.63	538.49	0.000	1	203333	203333	1459.84	0.000	1	24732.4	24732.4	278.89	0.000
x_2	1	1115.98	1115.98	218.47	0.000	1	47842	47842	343.48	0.000	1	13855.5	13855.5	156.24	0.000
x_3	1	215.32	215.32	42.15	0.000	1	919	919	6.6	0.028	1	577.6	577.6	6.51	0.029
x_1^2	1	125.07	125.07	24.48	0.001	1	36052	36052	258.84	0.000	1	265.7	265.7	3.00	0.114
x_2^2	1	192.31	192.31	37.65	0.000	1	18547	18547	133.16	0.000	1	593.1	593.1	6.69	0.027
x_3^2	1	174.15	174.15	34.09	0.000	1	5150	5150	36.97	0.000	1	783.4	783.4	8.83	0.014
x_1x_2	1	55.12	55.12	10.79	0.008	1	8320	8320	59.74	0.000	1	32	32	0.36	0.561
x_1x_3	1	1.13	1.13	0.22	0.649	1	3281	3281	23.55	0.001	1	480.5	480.5	5.42	0.042
x_2x_3	1	28.13	28.13	5.51	0.041	1	392	392	2.81	0.124	1	4324.5	4324.5	48.77	0.000
Residual	10	51.08	5.11	-	-	10	1393	139	-	-	10	886.8	88.7	-	-

Table 4. The optimum conditions for preparation of activated carbon from residues coffee and the predicted results for prepared activated carbon.

Optimization Condition	Activation Temperature, (°C)	Activation time, (min)	heating rate, (°C/min)	Carbon Yield (%)	Iodine number (mg/g)	MB uptake (mg/g)
Prediction	732.50	57.38	25.11	50.29	608.50	221.98
Experimental	732.50	57.38	25.11	45.30	617.57	246.21

Table 5. Physical characterization of the optimum activated carbon CAC.

activated carbon	BET surface area (m^2/g)	Pore volume (cm^3/g)	Average pore size (Å)
CAC (732.5/57.38/25.11)	772.49	0.477	24.70

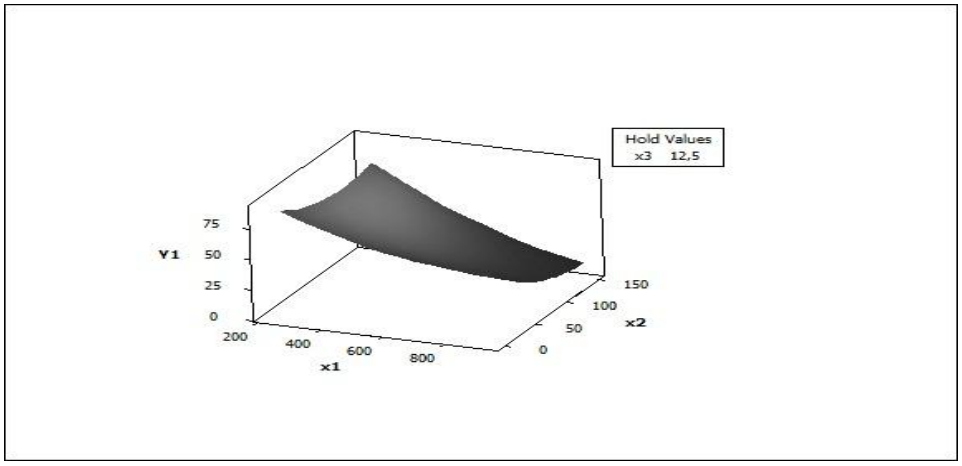

Fig. 1 a. Three-dimensional response surface plot of CAC yield; effect of activation temperature and activation time, HR=12.5°C/min.

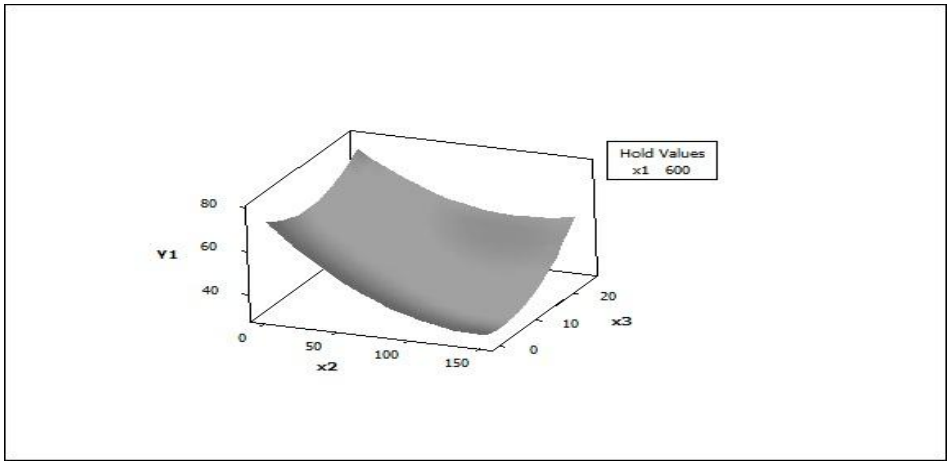

Fig. 1 b. Three-dimensional response surface plot of CAC yield; effect of activation time and Heating rate, T= 600 °C.

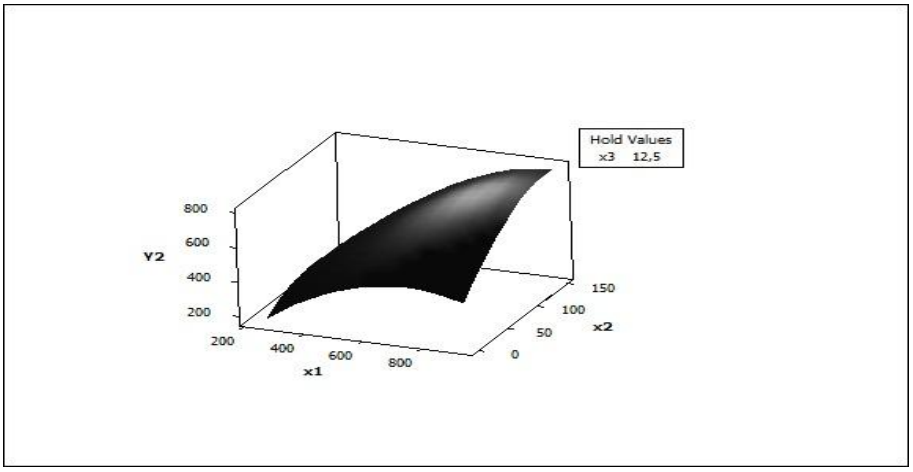

Fig. 2 a. Three-dimensional response surface plot of Iodine number; effect of activation temperature and activation time, HR=12.5°C/min.

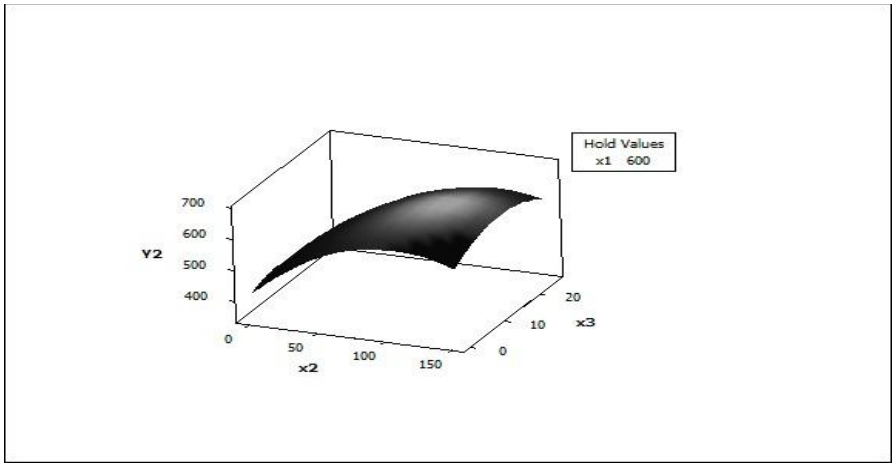

Fig. 2 b. Three-dimensional response surface plot of Iodine number; effect of activation time and Heating rate, T= 600 °C.

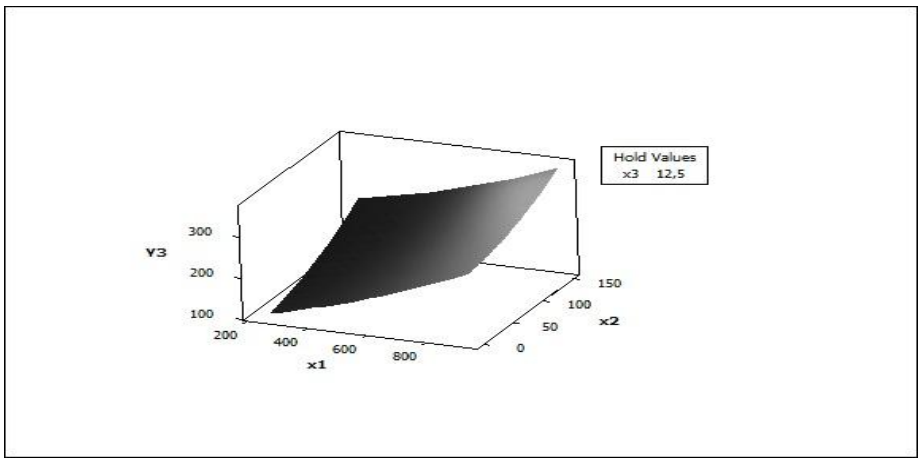

Fig. 3 a. Three-dimensional response surface plot of MB uptake, effect of activation temperature and activation time, HR= 12.5 °C/min.

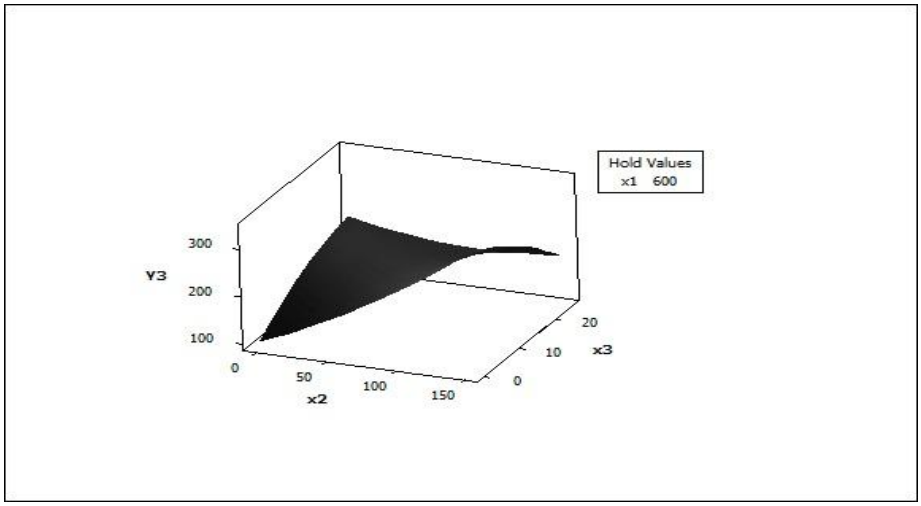

Fig. 3 b. Three-dimensional response surface plot of MB uptake, effect of activation time and Heating rate, T= 600 °C.

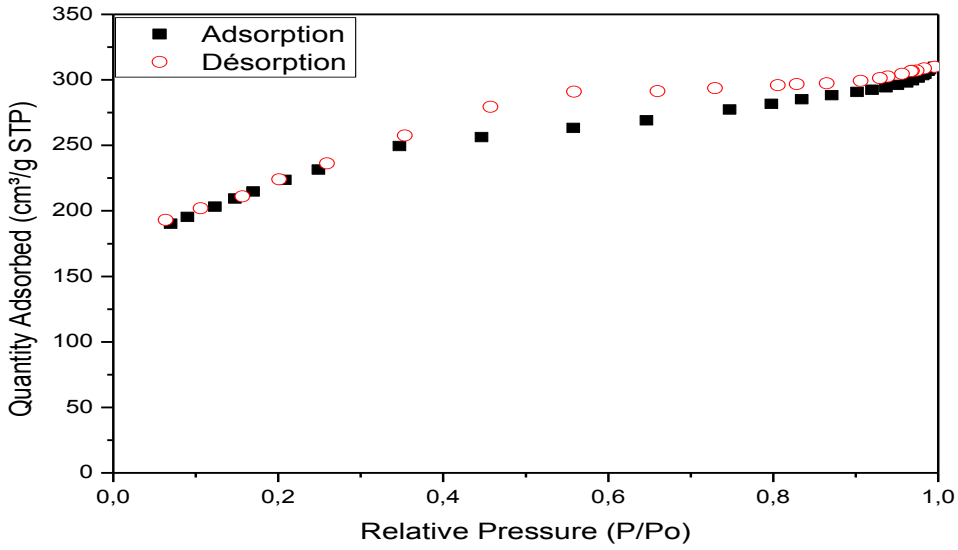

Fig. 4 . N_2 adsorption isotherm of the optimum activated carbon CAC.

4. Conclusion

Residues coffee is a good precursor for the production of activated carbons with greater adsorption capacity. The response surface methodology using central composite design is an appropriate tool to study optimization of the activation process to prepare activated carbons to be used in a given technological process. In the present paper, this optimization was carried out to obtain activated carbons from Residues coffee suitable for use in water treatments. The experimental parameters analyzed were activation temperature, activation time and heating rate and the optimal values obtained were 732.5°C, 57.38 min and 25.11°C/min, respectively.

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