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Preparation of activated carbon from Jatropha hull with microwave heating: Optimization using response surface methodology

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ABSTRACT

Preparation of activated carbon has been attempted using steam as the activating agent by microwave heating from Jatropha hull. The response surface methodology (RSM) technique is utilized to optimize the process conditions. The influences of the three major parameters, activation temperature, activation time and steam flow rate on the properties of activated carbon are investigated using analysis of variance (ANOVA), to identify the significant parameters. The optimum conditions for the preparation of activated carbon has been identified to be an activation temperature of 900 °C, activation time of 19 min and steam flow rate of 5 g/min. The optimum conditions resulted in an activated carbon with an iodine number of 988 mg/g and a yield of 16.56% respectively, while the BET surface area evaluated using nitrogen adsorption isotherm correspond to 1350 m^2/g, with the pore volume of 1.07 cm^3/g. The activated carbon is hetero porous with the micropore volume contributing to 40.8%.

1. Introduction

Activated carbons are one of the most important adsorbent materials in modern society. They have a very board range of applications in different industries which include gas purification [1], reduction of organic pollutants from drinking as well as waste waters [2–4], several medical applications [5] and as catalyst support [6] owing to their surface area, pore structure, thermal stability and low acid/base reactivity [7]. One of the main challenges in the commercial manufacture of activated carbons is to identify new precursors that are cheap, accessible and available in abundant quantity that has potential for significant economic benefits. A number of raw lignocellulosic materials such as wood, fruit shells and stones, have been utilized for the preparation of activated carbons with varied degree of success over the years.

With the growing concern on effect of pollutants on the environment and stringent environmental regulations in practice today, increased attention has been paid on the natural materials like waste biomass for the extraction of energy. Energy from alternative sources becomes imperative to overcome the depletion of the fossil fuel sources and to counter the release of green house gases. Most promising among alternative sources of energy could be either biodiesel or waste biomass. Fuels derived from biomass are sustainable and environment friendly which augurs the global interest to enhance its contribution to the total energy needs. As an effective way to solve the energy crisis, the biological energy is getting widespread attention all over the world. Jatropha curcas is widely accepted to be a promising plant for production of bio diesel which has the potential to reduce our dependence on the fossil fuel, as well as a renewable source of energy. Widespread cultivation of Jetropha curuas is initiated globally, and it is being currently cultivated in China in area in excess of 30,000 ha. With the estimated yield of plant being 65 t per hectare, the total output is estimated to be approximately around 2 million t. With an optimistic estimate of 30% waste biomass generation, the projected Jatropha hull can be 0.6 million t [8–11]. As Jatropha hull is rich of lignin, it has the potential to be good precursor for the production of activated carbon which forms the basis of the present work.

The conventional heating methods, typically heated from the hearth wall, do not ensure a uniform temperature of the material inside the furnace. This generates a temperature gradient from the hot surface of the sample particle to its interior and impedes the effective removal of gaseous products to its surroundings, thereby resulting in long reaction time and higher energy consumption. Microwave heating is increasingly utilized in various technological and scientific fields for variety of applications, due to its advantage of faster and uniform heating rate as compared to conventional heating method. The energy transfer is not based on the effect of conduction or convention, but is readily transformed into heat inside the particles by

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dipole rotation and ionic conduction. When high frequency voltages are applied to a material, the response of the molecules with a permanent dipole to the applied potential field, is to change their orientation in the direction opposite to that of the applied field, which causes the molecules to agitate and generate heat [12–14]. The tremendous temperature gradient from the interior of the char particle to its cool surface allows the microwave induced reaction to proceed more quickly and effectively, resulting in energy saving and shorter reaction time.

Although microwave heating has been utilized to regenerate activated carbon, the relevant literature is very limited. The studies pertaining to preparation of activated carbon has been limited to Guo and Lua [15], Williams and Parker [16]; while the regeneration studies are limited to Ania et al. [17] and Price and Schmidt [18]; Coss and Cha [19], Nabais et al. [20] and Menendez et al. [21], who have reported the surface chemistry modification of activated carbon fibers by means of microwave heating.

The present paper utilizes waste Jatropha hull and attempts to optimize the process parameters such as the activation temperature, activation time and flow rate of the activating agent (steam), for the preparation of activated carbon using microwave heating. The experimental design and process optimization has been conducted using the statistical process optimization tool, Response Surface Methodology (RSM) [22,23].

2. Materials and methods

2.1. Materials

Jatropha hull received from ShenYu Company, Kunming, Yunnan Province of China is washed with deionized water to remove the foreign materials and dried in an air oven at 105 °C. The dry materials are sieved to the particle size of 2 to 5 mm and stored in airtight container for further experiment. The proximate analysis of Jatropha hull represented in weight percent, shows a moisture content of 9.5% a volatile matter of 60.78%, fixed carbon of 25.48% and an ash content of 3.8%.

2.2. Experimental methods

The carbonization of Jatropha hull is carried out by loading 100 g of dried material into a muffle furnace, under N_2 gas flow (100 cm^3/min) and heated up to a carbonization temperature of 600 °C, at a heating rate of 10 °C/min. Upon reaching 600 °C the samples is held at the same conditions for 1 h. After carbonization the sample is cooled to room temperature under N_2 flow (100 cm^3/min). The yield of char is found to be around 40%.

The carbonized product is activated using a self-made microwave tube furnace, which utilizes a single-mode continuous controllable power for the experiments and is shown in Fig. 1. The microwave frequency is 2.45 GHz, while the output power could be set to a maximum of 3000 W. The activation temperature is controlled by the input microwave power during the activation process, which is measured by nichrome–nickel silicon armor type thermo-element, placed such that it touched the material. The thermo element has the dimension of 8 mm diameter and a length of 450 mm, with the temperature range of 0–1250 °C and a measurement precision of ± 0.5 °C.

The carbonized material is placed into reactor and set to the desired temperature along with the N_2 flow rate at 200 $cm^3\ min^{-1}$. Upon reaching the desired temperature the steam is allowed into the reactor at a desired flow rate to initiate the activation process.

It takes about 7–10 min, depending upon the set temperature to reach the desired temperature. It should be noted that the microwave heating is very effective and fast as the heating rate corresponds approximately in excess of 150 per minute. All the experiments are conducted under similar activation conditions. The activation time reported in Table 1, corresponds to the time the material is left in the reactor upon reaching the desired temperature. The completion of activation process under the set of desired activation conditions is marked by terminating the steam flow, by switching to the nitrogen flow until the activated carbon is cooled to the room temperature. The experiments are conducted covering the process parameters of activation temperature (800–1000 °C), the steam flow rate (1–6 g/min) and the activation time (15–30 min).

The product is subject to the characterization for iodine number, yield and the BET surface area. The yield is defined as grams of activated carbon per gram char utilized for activation. The iodine adsorption capacity is represented as iodine number which indicates milligrams of iodine adsorbed by a gram of activated carbon (mg/g), by using the Standard Testing Methods of PR China (GB/T12496.8-1999) [24] for testing acting activated carbons. The BET surface area, average pore size distribution are estimated using the surface area analyzer, Autosorbe 1-C made by Quantachrome Instruments, USA.

2.3. Design of experiments

RSM is a statistical technique for modeling and analysis of problems in which a response of interest is influenced by several factors [25]. A central composite design (CCD) is utilized to optimize the effective parameters with a minimum number of experiments, as well as to analyze the interactions between the parameters [26].

The dependant variables selected in the present study are activation temperature (X_1), activation time (X_2) and steam flow rate (X_3). A full factorial CCD for the three variables, consisting of 8 factorial points, 6 axial points and 6 replicates at the center points are employed, with a total of 20 experiments as calculated from the following equation [26],

$$N = 2^n + 2n + n_c = 2^3 + 2 \times 3 + 6 = 20 \quad (1)$$

N is the total number of experiments, while n is the number of dependent variables. The experimental design matrix is provided in Table 1.

The experimental data are analyzed using statistical software Design Expert software version 7.1.5 (STAT-EASE Inc., Minneapolis, USA) for regression analysis and to evaluate statistical significance of the equation.

3. Result and discussion

The activated carbon is prepared under the conditions as shown in Table 1 covering the parameters such as activation temperature, activation time and steam flow rate. The experimental conditions are as generated by the Design Expert software, which generates the minimum number of experiments with repeat runs, provided the range of the each of the parameters. The activated carbons are characterized for the yield, iodine absorption capacity and results are compiled as well in Table 1. The activated carbon with highest iodine number is characterized for BET surface area and pore size distribution.

3.1. Iodine adsorption

The most important characteristic of an activated carbon is its adsorption capacity, which is characterized by the surface area and pore size which are highly influenced by the process of preparation. All three parameters chosen in the present study are found to have significant effect on iodine adsorption capacity. Fig. 2 shows the three-dimensional response surfaces of the combined effect of activation temperature and activation time on the iodine number, while the steam flow rate is maintained at 3 g/min. The iodine

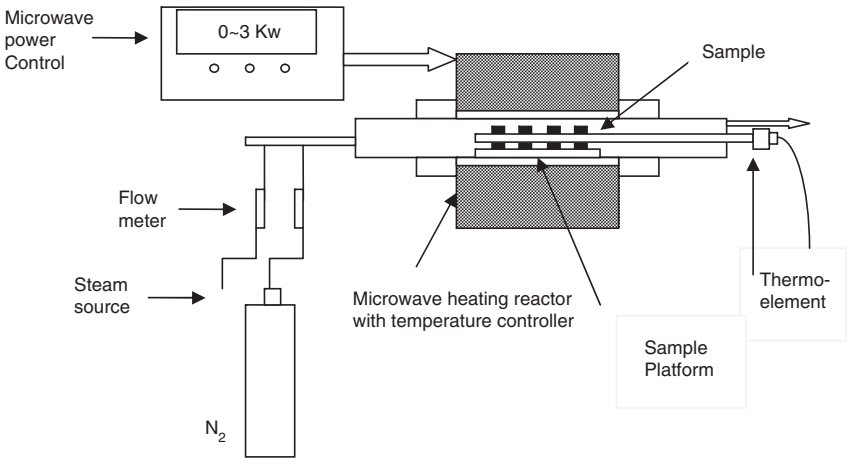

Fig. 1. Schematic of microwave heating equipment.

number increases with an increase in the activation temperature and activation time, with the maximum iodine number of about 990 mg/g, corresponding to the maximum of activation temperature and activation time, covered in the present study. An increase in the activation temperature leads to a reduction in the activation energy for the carbon-steam reaction, which increases the rate of reaction. The higher rate of reaction results in higher conversion or in other words a better porous carbon. Similarly an increase in the activation time would increase the extent of the carbon-steam reaction, which in turn would increase the porosity of the activated carbon. An attempt has been made to compare the iodine number due to the present study with the typical values reported in literature. Zhang et al. [27] have reported a maximum iodine number of 1018 mg/g for a microwave irradiated tobacco stems activated using steam, and additionally reported the porous structure to resemble honeycomb-shaped microscopic crevices. A similar study due to Xia and Peng [28] using tobacco stems as precursor with steam and microwave heating have reported a maximum iodine number of 1061 mg/g and the surface area of 1109 m^2/g, further to highlighting the reduction in activation time using microwave heating, owing to the faster and uniform heating rate due to microwave. Li and Peng [7] have made an identical remark to Xia and Peng [28] on the advantages of microwave heating and they have reported an iodine number of 1085 mg/g for coconut shell precursor, activated using steam and microwave heating. Fan and Peng [29] have reported a better porous structure of the activated carbon activated using microwave in comparison with the conventional heating for coconut shells with steam. The increase in iodine adsorption capacity was attributed to the deeper degree of carbon-steam reaction, which facilitates formation of new micropores and well developed pore structure.

Fig. 3 shows the three-dimensional response surfaces of the combined effect of activation temperature and steam flow rate on the iodine number, at an activation time of 15 min. As can be seen, the iodine number of activated carbon increases with the increase of steam flow rate and activation temperature, however the effect of steam flow rate is not as significant as the temperature. As discussed earlier the improvement in the porous nature of the activated carbon due to higher temperature could be attributed to the reduction in the activation energy, while the improvement due to the steam flow rate could be attributed to the improvement in the carbon-steam reaction ratio. The reduction in slope of the curve at higher steam flow rate could be attributed either to the flow in excess of stoichiometric ratio or to the mass transfer limitations of steam in the reactor due to non interaction of the steam with the carbon particles.

Table 1
Experimental design matrix and results.

Run	X_1(°C)	X_2(min)	X_3(g/min)	Y_1(mg/g)	Y_2 (%)
1	800	10.00	2.00	680	71.30
2	1000	10.00	2.00	823	54.24
3	800	25.00	2.00	879	32.70
4	1000	25.00	2.00	924	22.20
5	800	10.00	5.00	726	59.54
6	1000	10.00	5.00	911	29.40
7	800	25.00	5.00	939	24.34
8	950	25.00	5.00	997	15.10
9	731	17.50	3.50	749	70.32
10	1068	17.50	3.50	950	20.70
11	900	4.89	3.50	674	69.46
12	900	30.11	3.50	958	20.30
13	900	17.50	0.98	718	77.85
14	900	17.50	6.02	942	21.13
15	900	17.50	3.50	891	27.43
16	900	17.50	3.50	893	27.30
17	900	17.50	3.50	896	27.21
18	900	17.50	3.50	892	27.15
19	900	17.50	3.50	897	27.32
20	900	17.50	3.50	896	27.40

3.2. Activated carbon yield

The yield of activation carbon is an important parameter as it quantifies the amount of final product. Table 1 also compiles the yield

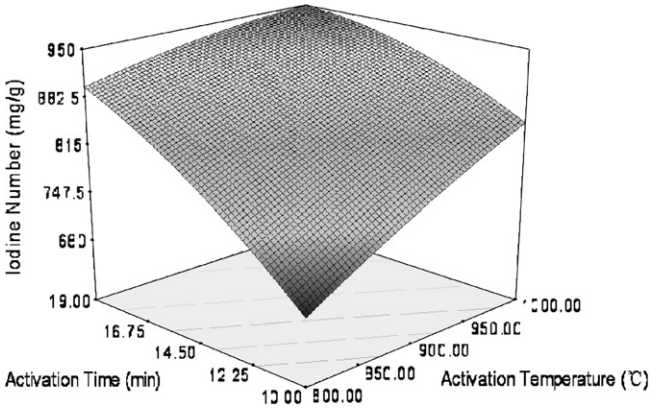

Fig. 2. Three-dimensional response surface plot of iodine uptake: effect of activation temperature and activation time on the iodine number (steam flow rate: 3 g/min).

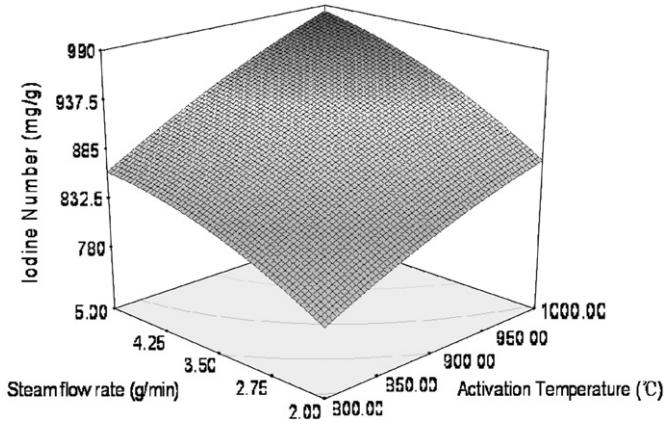

Fig. 3. Three-dimensional response surface plot of activated iodine uptake: effect of activation temperature and steam flow rate on the iodine number (activation time: 15 min).

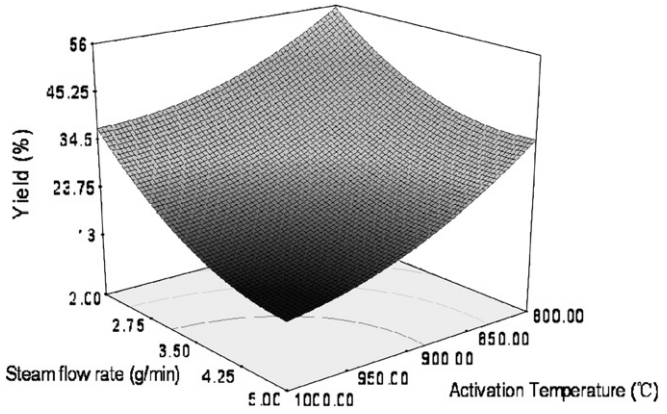

Fig. 5. Three-dimensional response surface plot of activated carbon yield: effect of activation temperature and steam flow rate on the yield (activation time: 15 min).

of activated carbon for each of the experiments performed. Fig. 4 shows the three dimensional plot of effect of activation temperature and activation time while Fig. 5 shows the effect of activation temperature and steam flow rate on the yield of activated carbon. The activated carbon yield is found to decrease with increase in activation temperature and activation time in Fig. 4, while it is found to decrease with increase in activation temperature and steam flow rate in Fig. 5. All the three parameters have significant effects on the yield of activated carbon and the highest yield corresponds to the lowest point of the all three parameters covered. However it should be noted that the maximum yield corresponds to activated carbon with lowest iodine number. An increase in activation temperature, activation time or the steam flow rate increases the extent of carbon-steam reaction and hence the yield of activated carbon decreases with increase in anyone of the parameters. The yield of activated carbon for iodine number in excess of 950 mg/g is found to range between 15 and 20%. Similar yields have been reported in the literature for other precursors with Zhang et al. [27] reporting a yield of 20.74% with 6 min of activation time using steam, while Xia and Peng [28] have reported a yield of 30.83% by microwave heating with relatively low steam flow rate. Similarly, Wang and Su [30] have reported a yield of 17% for activated carbon prepared using coconut shell using steam activation. Hu and Li [31] have reported a yield of 14% for the rice husk precursor activated using steam. In one of the recent publication Zabaniotou et al. [32] have reported a significantly increases the surface area and adsorption capacity with carbon burn-off. The amount of carbon removed from the material was found to depend on the extent of carbon-steam reaction, which was reported to depend on the activation temperature, activation time and the steam flow rate.

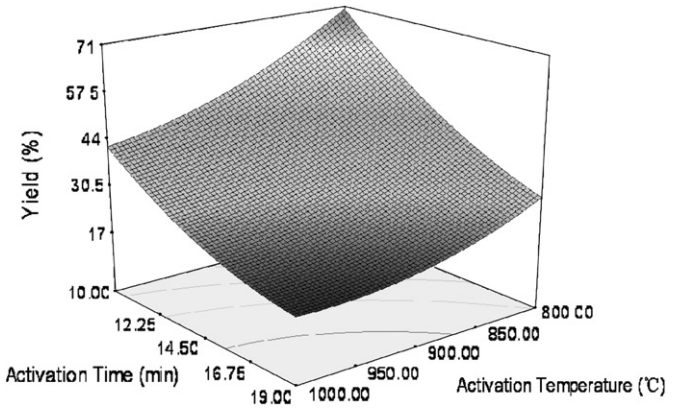

Fig. 4. Three-dimensional response surface plot of activated carbon yield: effect of activation temperature and activation time on the yield (steam flow rate: 3 g/min).

3.3. Development of regression model

A polynomial regression equation is developed using CCD to analyze the factor interactions and to identify the significant factors contribution to the regression model. The design together with the response values from the experiments are shown in Table 2. Runs 15–20 at the center point are repeated to determine the experimental error. The Iodine adsorption and carbon yield are utilized in the quadratic model according to the propositions of the software. The final empirical models in terms of coded factors (all the terms are included so better remove the striked out part) for iodine adsorption (Y_1) and carbon yield (Y_2) are shown in Eqs. (2) and (3) respectively as,

$$Y_1 = -1587.95 + 2.75X_1 + 102.60X_2 + 35.06X_3 - 0.062X_1X_2 + 0.047X_1X_3 + 0.026X_2X_3 - 7.99E^{-004}X_1^2 - 0.98X_2^2 - 6.62X_3^2 \quad (2)$$

$$Y_2 = +722.30 - 0.98X_1 - 18.11X_2 - 21.34X_3 + 7.91E^{-003}X_1X_2 - 0.011X_1X_3 + 0.39X_2X_3 + 4.39E^{-004}X_1^2 + 0.22X_2^2 + 2.59X_3^2 \quad (3)$$

The suitability of model equation is evaluated using the correlation coefficients (R^2), which are 0.9593 for Eq. (2) and 0.9028 for Eq. (3). The proximity of *R2* value to unity, indicate the suitability of the model equation. Both the *R2* values of Iodine adsorption and carbon yield are relatively high indicating, good agreement between experimental data and the model prediction.

Table 2
Analysis of variance (ANOVA) for response surface quadratic model for Iodine uptake.

Source	Sum of squares	Degree of freedom	Mean square	F-value	Pb>F
Model	1.739E^{+005}	9	19,326.94	26.16	<0.0001
X_1	43,365.36	1	43,365.36	58.70	<0.0001
X_2	85,043.14	1	85,043.14	115.12	<0.0001
X_3	30,220.09	1	30,220.09	40.91	<0.0001
X_1X_2	6407.12	1	6407.12	8.67	0.0147
X_1X_3	397.62	1	397.62	0.54	0.4800
X_2X_3	0.24	1	0.24	3.316E^{-004}	0.9858
X_1^2	921.00	1	921.00	1.25	0.2903
X_2^2	5732.49	1	5732.49	7.76	0.0193
X_3^2	3202.12	1	3202.12	4.33	0.0640

The analysis of variance (ANOVA) is carried out to justify the adequacy of the model. The ANOVA for the quadratic model of Iodine adsorption is shown in Table 2, where the F-value of 26.16 and Prob>F of 0.0001 prove that the model is significant. The value of model terms Prob>F less than 0.05 indicates that the model terms are significant. Checking the adequacy of model is an important part of the data analysis procedure, since it would result in poor or misleading results if the fit is inadequate [33]. In this case, X_1, activation temperature; X_2, activation time and X_3, steam flow rate are all significant model terms.

Fig. 6 shows the comparison of the predicted iodine number versus the experimental iodine number of activated carbon. The experimental iodine numbers are the measured data of a particular experimental run while the predicted values are evaluated from the model. As can be seen, the predicted values match well with the experimental values, indicating the ability of the model to successfully capture the correlation between the process variables and the iodine adsorption capacity.

The ANOVA of the quadratic model of activated carbon yield is shown in Table 3. An F-value of 10.32 and Prob>F of 0.0006 prove that the model is significant. The value of the model terms Prob>F less than 0.05 indicates that all the three process parameters, X_1, X_2 and X_3, are significant. The results show that the model is suitable to predict the carbon yield within the range of factors studied. The predicted value versus the experimental values for yield of activated carbon is shown in Fig. 7.

3.4. Process optimization

The commercial production of activated carbon augurs higher product yield for economic feasibility while the adsorption efficiency is critical for marketing. Hence it is desirable to prepare activated carbon with high yield and a high adsorption capacity.

It is established experimentally that the iodine uptake and carbon yield responds opposite to each other with the process parameter. In order to compromise these two values, the function of desirability is applied using Design Expert software version, 7.1.5 (STAT-EASE Inc., Minneapolis, USA). The experimental conditions with highest desirability are selected with the help of software. The optimum condition for preparation of activated carbon is found to be an activation temperature of 900 activation time of 18.82 min and steam flow rate of 5 g/min, with the Iodine adsorption of 979 mg/g and carbon yield of 16%. The repeat experiments are carried out to justify the accuracy of the predicted result which showed an average Iodine number of 988 mg/g and average carbon yield of 16.56%, with the relatively error of just 0.91% and 3.50%, which indicates the success of the process optimization exercise.

Table 3
Analysis of variance (ANOVA) for response surface quadratic model for activated carbon yield.

Source	Sum of squares	Degree of freedom	Mean square	F-value	Pb>F
Model	7269.37	9	807.71	10.32	0.0006
X_1	1645.00	1	1645.00	21.02	0.0010
X_2	3013.77	1	3013.77	38.52	0.0001
X_3	1551.09	1	1551.09	19.82	0.0012
X_1X_2	101.53	1	101.53	1.30	0.2812
X_1X_3	20.80	1	20.80	0.27	0.6173
X_2X_3	57.78	1	57.78	0.74	0.4103
X_1^2	278.81	1	278.81	3.56	0.0884
X_2^2	278.81	1	278.81	3.56	0.0884
X_3^2	489.19	1	489.19	6.26	0.0313

3.5. Pore structure and surface area analysis

The pore structure of the carbon char and the activated carbon is characterized by nitrogen adsorption at 77 K with an accelerated surface area and porosimetry system (Autosorb-1-C, Quantachrome). Prior to gas adsorption measurements, the carbon is degassed at 300 °C in a vacuum condition for a period of at least 2 h. Nitrogen adsorption isotherm is measured over a relative pressure (P/P_0) range from approximately 10^{-7} to 1. The BET surface area is calculated from the isotherms by using the Brunauer–Emmett–Teller (BET) equation. The cross-sectional area for nitrogen molecule is assumed to be 0.162 nm. The Dubinin–Radushkevich (DR) method is used to calculate the micropore volume. The micropore size distribution is ascertained using the Non-local Density Functional Theory (NLDFT) model. The total volume is estimated by converting the amount of N_2 gas adsorbed at a relative pressure of 0.95 to equivalent liquid volume of the adsorbate (N_2) [34]. The mesopore volume is estimated by the subtracting the micropore volume from the total volume.

The nitrogen adsorption isotherm estimated using the Autosorb instrument is shown Fig. 8. It can be seen from the figure that the volume adsorbed increases sharply (concave) at low relative pressure which indicate the filling of micro pores and reach a plateau. The adsorption capacity continues to increase with increase in the relative pressure up to a P/P_0 value of 1, which pertains to adsorption isotherm type II under the IUPAC classification of isotherms, based on the progressive increase in the adsorption capacity beyond the relative pressure of 0.1. The sharp rise beyond a relative pressure of 0.8 and formation of hysteresis loop, while desorbing indicate a type IV isotherm. This type of isotherm is a general characteristic of porous carbons that have large sized pores. The cumulative pore volume plot shown in Fig. 9 substantiates the amount of pores in the mesoporous range, with the average pore diameter estimated to be 3.1 nm. The

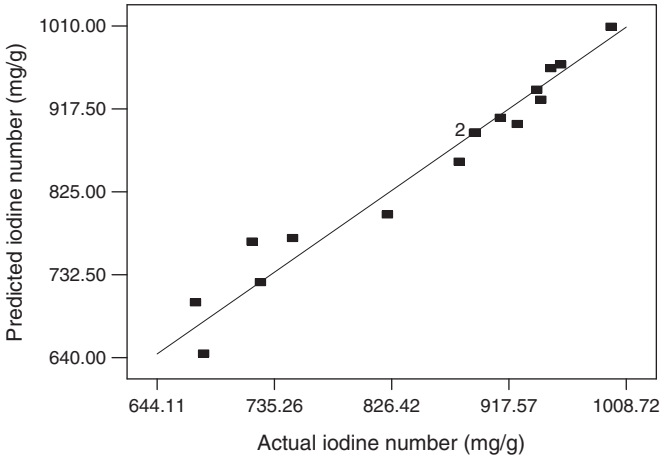

Fig. 6. Predicted vs. experimental adsorption uptake on iodine.

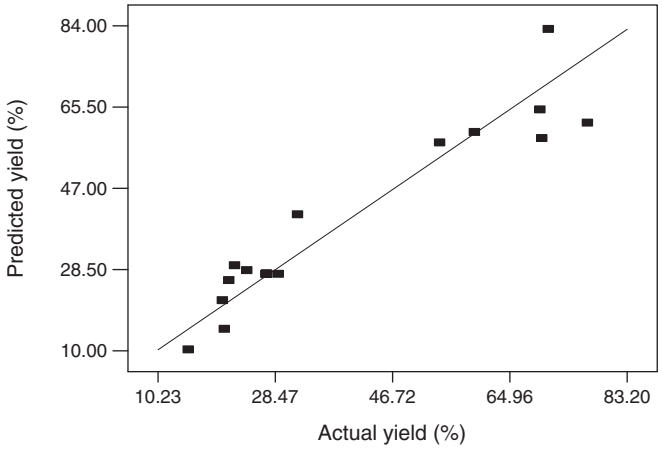

Fig. 7. Predicted vs. experimental activated carbon yield.

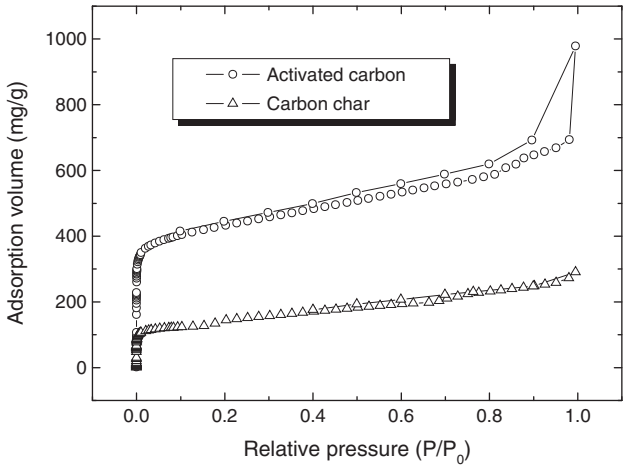

Fig. 8. Nitrogen adsorption isotherm of the activated carbon and char.

surface area of the activation is estimated to be 1350 m^2/g, while the total pore volume is 1.07 cc/g. Table 4 provides the pore volume corresponding to the micropore and mesopore along with average pore diameter of the activated carbon and pyrolysed carbon char utilized for activation. A comparison of the key parameters of the activated carbon with the pyrolysed char shows that the proportion of mesopore volume is much higher than the micropore volume in the pyrolysed char. A comparison of the quality of activated carbon with the carbonized char shows a significant increase in the pore volume and the surface area attributed to the activation process.

3.6. Microscopic structure analysis of the activated carbon

The microscopic structure of the jatropha hull char (before activation) and the activated carbon prepared using steam activation are shown in Fig. 10. As can be seen in Fig. 10a, the surface of the primary char utilized for activation is planar, comprising mainly macro and meso pores without deeper pore structure. This shows that the carbonization stage mainly creates macro and mesoporous carbon [35–38]. After activation the chars transform into activated carbon with a significant increase in the micro and mesopores. As can be seen in Fig. 10b, there are lots of small crevices on the surface, which are small pores having well developed pore network. During the process of activation, the reaction of carbon with steam generates large number of micropores which significantly increases the surface area of the activated carbon.

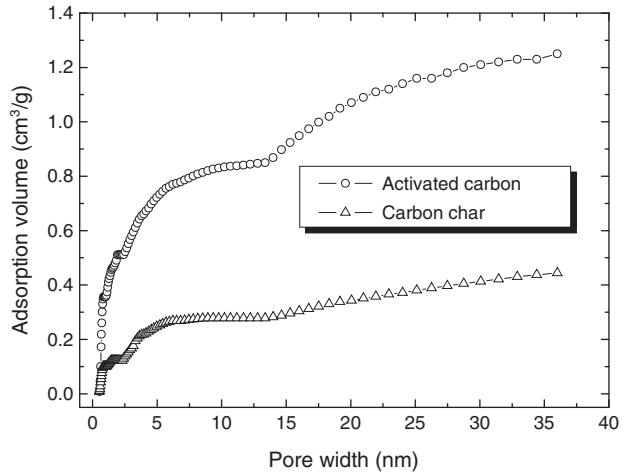

Fig. 9. Cumulative pore volume distribution chart for activated carbon and char.

Table 4
Details of pore structure of activated carbon vs. carbonized product.

Subject		Activated carbon	Carbonized char
Pore volume	(ml/g)	1.07	0.42
Average pore diameter	(nm)	3.10	3.50
Micropore volume	(%)	40.80	29.1
Mesopore volume	(%)	59.20	70.9
Surface area	(m^2/g)	1350	480

4. Conclusion

The response surface methodology (RSM) technique is used to optimize the process conditions, for the preparation of activated carbon from Jatropha hull. The influences of the three major parameters, activation temperature, activation time and steam flow rate on the properties of activated carbon are investigated using analysis of variance (ANOVA), to identify the significant parameters. The experimental data of the adsorption capacity and yield are found to agree satisfactorily with the model predictions.

The optimum conditions for preparation of activated carbon has been identified to be an activation temperature of 900 °C, activation time of 19 min and steam flow rate of 5 g/min. The optimum conditions result in an activated carbon with an iodine number of 988 mg/g and a yield of 16.56% respectively. The BET surface area evaluated using nitrogen adsorption isotherm for the optimal sample correspond to 1350 m^2/g, with the pore volume of 1.07 cm^3/g. The activated carbon is

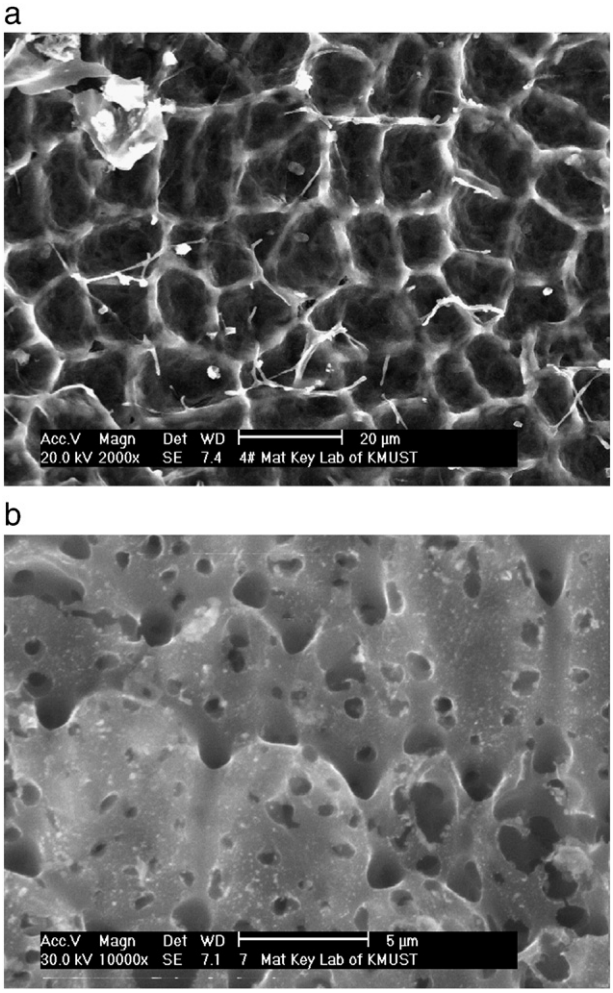

Fig. 10. (a) SEM image of the char prior to activation. (b) SEM image of the char after activation.

hetero porous with the micropore volume contributing to 40.8%. The results of this study indicate the potential of Jatropha hull to be a potential feedstock for production of activated carbon of significant quality, using steam and microwave heating.

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