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Preparation of Eupatorium Adenophorum Based Porous Carbon with Microwave Heating using Response Surface Methodology

Abstract: Eupatorium adenophorum was utilized as raw materials for the preparation of activated carbon via microwave assisted steam activation. Influences of the three vital process parameters – activation temperature, activation duration and steam flow rate – have been assessed on the adsorption capacity and yield of Eupatorium adenophorum activated carbon (EAAC). The process parameters were optimized utilizing the Design Expert software and were identified to be an activation duration of 45 min, an activation temperature of 950 °C and a steam flow rate of 0.7 ml/min, with the resultant iodine number and yield being 1,010 mg/g and 20.13% respectively. The validity of process model to optimize the process parameters was verified using the analysis of variance (ANOVA). The key parameters that characterize quality of the porous carbon such as the BET surface area, total pore volume and average pore diameter were estimated to be 1,142 m^2/g, 0.84 ml/g and 3.3 nm respectively, for the sample corresponding to the optimized process conditions. Additionally the pore structure is characterized using Scanning Electron Microscope (SEM). The present work strongly supports utilization of Eupatorium adenophorum as a potential precursor through microwave heating.

Keywords: Eupatorium adenophorum, activated carbon, microwave heating, response surface methodology

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1 Introduction

Activated carbons (AC) are important kind of porous material with abundantly developed pores having strong adsorption ability, high surface area and thermo stability. They are widely used in different industries for separation and purification processes [1–3], as catalyst supports [4], for removal of organic dyes and pollutants from industrial wastewater [5, 6].

The selection of an appropriate precursor plays an important role in deciding the characteristics of AC as well as the economics of manufacturing plant. To identify new precursors that are cheap, accessible and available in large quantity is one of the main challenges for adoption to commercial manufacture [7]. Towards which, different biomass based feedstock such as rice bran, coconut shell and waste materials were used as the raw materials since they are sustainable sources having high fixed carbon content [8–10].

Eupatorium adenophorum, a kind of global exotic weeds originated from Mexico, and which has spread extensively in many countries around the world such as America, Australia and the countries in Southeast Asia due to its strong ability to adapt to different environmental conditions [11]. Since the 1940s, Eupatorium adenophorum has spread extensively in south and western of China. Lots of the farm lands, pasture fields and forests have been destroyed causing huge economic losses. This has drawn attention of the society and many methods have been developed to control it, such as manual, chemical and biological means, however without any realistic means to eradicate it. In 2003, the Chinese ministry of environmental protection released a list of "The First Batch of Exotic Invasive Species" and Eupatorium adenophorum was rated the first [12, 13]. According to the published literatures, Eupatorium adenophorum can be used as bio-pesticide, organic fertilizer and feedstuff, feedstock for production of marsh gas [14, 15]. Although, Eupatorium adenophorum can be utilized as a biomass resource to prepare the AC, the relevant literature is very limited. The attempts pertaining to preparation of EAAC has been limited to Xia et al. and Wu et al. [16, 17].

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Activated carbons have been traditionally produced by the partial gasification of the char either with steam or CO_2 or a combination of both. The gasification reaction results in removal of most reactive carbon atoms and in the process simultaneously produce a wide range of pores (predominantly micropores), resulting in porous activated carbon. In general, the methods for AC production are divided into two classes: physical activation and chemical activation. Physical activation is essentially a two-step process, where the carbonization of a carbonaceous material forms the first step, while the second step involves the activation of the resulting char at elevated temperature in the presence of suitable oxidizing gases such as carbon dioxide, steam, air or their mixtures. Chemical activation involves the impregnation of a carbonaceous material with an activation agent and heat treatment of the impregnated material under inert atmosphere. Physical activation is widely adopted industrially for commercial production owing to the simplicity of process and the ability to produce AC with well developed micro porosity and desirable physical characteristics such as the good physical strength.

The conventional heating methods do not ensure a uniform temperature of the precursor owing to their variation in the size and shape as the mode of heating is through conduction and convection. This conventional heating mode 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, demanding higher processing time and energy consumption. Recently, microwave heating is being increasingly utilized for variety of applications, as heating is uniform, where the absorbed microwave readily transforms into heat inside the particles by dipole rotation and ionic conduction [18–20].

This urged research towards upgrading of the available biomass Eupatorium adenophorum using microwave heating. In recently years, microwave heating has been used to produce AC, the relevant literature is very limited [21–23]. The aim of present work is to explore the possibility of converting weed into value-added AC through process optimization technique, with the activation temperature, activation duration and steam flow rate as the variables utilizing the microwave. Moreover the influences of the variables on iodine number and yield of EAAC were investigated systematically.

2 Materials and methods

2.1 Materials

Eupatorium adenophorum (Fig. 1) was obtained from Kunming, Yunnan Province of China. The raw materials (stems of Eupatorium adenophorum) were crushed to a size of 5–7 mm and washed thoroughly with deionized water, then were oven-dried at 105 °C and stored in moisture free environment for utilization in the experiments. The proximate analysis of Eupatorium adenophorum is presented in Table 1. The carbonization of Eupatorium adenophorum is carried out at a carbonization temperature of 500 °C at the heating rate of 20 °C/min in muffle furnace, for duration of 60 min, under the nitrogen flow atmosphere. The char yield is estimated to be around 35% and the proximate analysis of char is also provided in Table 1. The carbon content of the char is found to have increased significantly upon carbonization.

Fig. 1: Photo of Eupatorium adenophorum

Table 1: The proximate analysis of precursor Eupatorium adenophorum and its chars

Content	Materials (wt%)	Chars (wt%)
Volatile matter	76.41	15.84
Ash	1.90	8.86
Fixed carbon	21.69	75.30

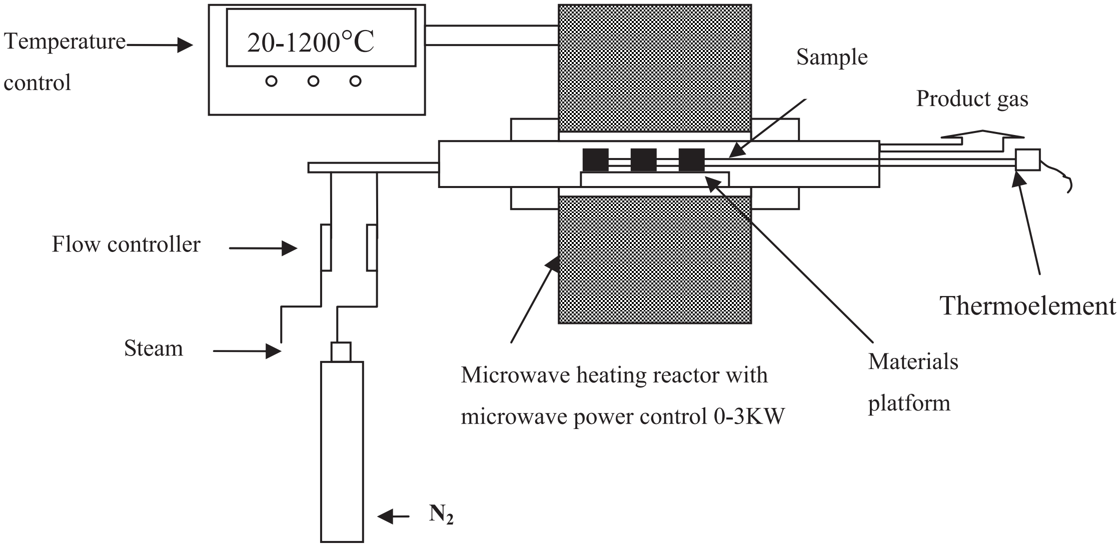

Fig. 2: The modified diagram for the preparation of microwave heating equipment

2.2 Preparation of EAAC

The activation experiments were carried out in a self-made microwave tube furnace as shown in Fig. 2. It consist of a microwave power control system (with the output power of 0–3 KW and the microwave frequency of 2.45 GHz), the temperature control system (temperature controlled by the input microwave power and measured by the thermoelement, with a measurement precision of ±0.5 °C), and flow control system (controlled by the flow meter with steam and N_2).

A known amount of carbonized materials of Eupatorium adenophorum were placed into the platform, and were heated from the room temperature to the desired temperature (850–1,000 °C) in a nitrogen atmosphere with N_2 flow at 100 cc/min. The desired temperature could be achieved in duration of approximately 10–12 minutes that correspond to heating rate of 120 °C per minute, which is significantly faster than the conventional heating. Once the carbonized materials reached the desired temperature the N_2 flow is terminated and replaced by steam into the reactor at the desired flow rate for desired activation duration. The variations in the flow rates of steam and the activation duration covered in the present experiments are reported in Table 2. The completion of activation process is marked by termination of the steam supply and starting with N_2 flow until the EAAC is cooled to the room temperature, which were then dried at 105 °C and stored for further characterization.

Table 2: Experimental design matrix and results by microwave heating with steam

Run	X_1 (°C)	X_2 (min)	X_3 (ml/min)	Y_1 (mg/g)	Y_2 (%)
1	850.00	15.00	0.30	735	79.20
2	950.00	15.00	0.30	836	57.88
3	850.00	45.00	0.30	822	40.35
4	950.00	45.00	0.30	908	30.74
5	850.00	15.00	0.7	778	62.03
6	950.00	15.00	0.7	841	38.36
7	850.00	45.00	0.7	796	35.55
8	950.00	45.00	0.7	1008	20.63
9	815.91	30.00	0.50	800	73.41
10	984.09	30.00	0.50	947	25.20
11	900.00	4.77	0.50	685	77.76
12	900.00	55.23	0.50	959	26.93
13	900.00	30.00	0.16	797	75.87
14	900.00	30.00	0.84	900	27.86
15	900.00	30.00	0.50	928	32.32
16	900.00	30.00	0.50	931	32.29
17	900.00	30.00	0.50	927	32.31
18	900.00	30.00	0.50	932	32.28
19	900.00	30.00	0.50	934	32.33
20	900.00	30.00	0.50	929	32.27

RSM is utilized to optimize the activation process as it is a popular statistical tool for modeling and analysis of multi parameter processes [24]. Influences of the three major parameters, activation temperature, activation duration and steam flow rate were experimentally assessed based on the three dimensional plots generated by the RSM. The experimental data were analyzed using the Design Expert software version 7.1.5 (Stat-Ease Inc., Minneapolis, USA).

2.3 Characterization of EAAC

The EAAC were characterized for iodine number, yield and the process optimization was performed based on these two response variables. The BET surface area and pore structure of the samples that exhibited highest of quality are reported. The iodine number was calculated using the Standard Testing Methods of PR China (GB/T12496.8-1999) [25], while the yield of EAAC is based on the following equation:

$$Y_2 = {}^{W_{AC}}\!\big/_{W_{CB}} \times 100\% \tag{1}$$

where W_{AC} is the weight of AC, W_{CB} is the weight of the carbonized materials before the activation placed in the platform. The BET surface area and pore structure of AC was characterized by the automated gas adsorption system (Autosorbe 1-C, USA) and the microstructures were captured using the scanning electron microscope (SEM, Philips XL30ESEM-TMP).

3 Results and discussion

Table 2 shows the experimental conditions for preparation of EAAC generated by the Design Expert software covering the parameters such as activation temperature, activation duration and steam flow rate. The EAAC are characterized for iodine number, yield and results are listed as well in Table 2. Experimental runs performed at the center point of all the variables (15–20 runs) were utilized to determine the experimental error.

3.1 Verification of the regression model

Table 3: Analysis of variance for the iodine number

Source	Sum of squares	Degree of freedom	Mean square	F-value	Pb > F
model	128200	9	14239.44	14.60	0.0001
X_1	36831.18	1	36831.18	37.75	0.0001
X_2	47428.28	1	47428.28	48.62	<0.0001
X_3	6381.97	1	6381.97	6.54	0.0285
X_1X_2	2244.50	1	2244.50	2.30	0.1603
X_1X_3	968.00	1	968.00	0.99	0.3427
X_2X_3	84.50	1	84.50	0.087	0.7745
X_1^2	6095.15	1	6095.15	6.25	0.0315
X_2^2	21665.79	1	21665.79	22.21	0.0008
X_3^2	12460.28	1	12460.28	12.77	0.0051

Central composite design (CCD) was used to develop correlation between the factors and to identify the significant factors contribution to the regression model. In this study, the independent variables selected are activation temperature (X_1), activation duration (X_2) and steam flow rate (X_3), while the dependent variables are iodine number (Y_1) and yield (Y_2). The iodine number was found to range from 685 mg/g to 1008 mg/g, while the yield obtained range from 18.93% to 80.41%. For the iodine number and yield, the quadratic models were chosen as they were found to be aptly representing the data. The final empirical models in terms of coded factors (excluding the insignificant terms) for iodine number (Y_1) and yield (Y_2) are shown in Eqs. (2) and (3), respectively:

$$Y_1 = -6064.30 + 14.63X_1 - 6.37X_2 - 179.30X_3 - 0.01X_1^2 - 0.17X_2^2 - 735.11X_3^2 \tag{2}$$

$$Y_2 = +1889.86 - 3.58X_1 - 5.99X_2 - 126.82X_3 + 0.01X_1^2 + 0.025X_2^2 + 137.32X_3^2 \tag{3}$$

The correlation coefficients (R^2) can be used for testing the quality of the model equation. The R^2 for Eqs. (2) and (3) were found to be 0.93 and 0.92, respectively. The R^2 value to closer to unity, indicate the suitability of the model equation and a good agreement between experimental data and the prediction using the model equation [26].

The appropriateness of the model can also be assessed based on analysis of variance (ANOVA), and to identify the significant model parameters. Validating the model adequacy is an import part of the data analysis, since it would lead to poor or misleading results if it is an inadequate fit. Table 3 presents the results of ANOVA for the quadratic model for iodine number. The model F-value of 14.60 and $P > F$ of 0.0001 indicated the accuracy and suitability of the model. Based on the ANOVA model parameters X_1, X_2, X_3 and the interaction terms (X_1^2, X_2^2, X_3^2) were found to be significant based on the low ‘p’ values (≤0.05). The ANOVA result of quadratic model corresponding to yield is shown in Table 4. A model F-value of 16.60 and $P > F$ of <0.0001 indicate the suitability of model. The model parameters X_1, X_2, X_3 along with the interaction parameters (X_1^2, X_2^2, X_3^2) were found to be significant.

Fig. 3 and Fig. 4 show the comparison of predicted iodine number and yield versus the experimental iodine number and yield of the EAAC, respectively. As can be seen, the experimental data are evenly distributed on the both sides of the model prediction, indicating the suitability of the model developed in capturing the correlation between the process and response variables.

Table 4: Analysis of variance for the yield

Source	Sum of squares	Degree of freedom	Mean square	F-value	Pb > F
model	6870.03	9	763.34	16.60	<0.0001
X_1	1660.71	1	1660.71	36.13	0.0001
X_2	2803.93	1	2803.93	60.99	<0.0001
X_3	1282.48	1	1282.48	27.90	0.0004
X_1X_2	52.33	1	52.33	1.14	0.3111
X_1X_3	7.33	1	7.33	0.16	0.6980
X_2X_3	59.30	1	59.30	1.29	0.2826
X_1^2	303.30	1	303.30	6.60	0.0280
X_2^2	462.06	1	462.06	10.05	0.0100
X_3^2	434.78	1	434.78	9.46	0.0117

3.2 Activated carbon iodine number

Adsorption capacity is one of the most important characteristic of AC which is highly influenced by the process of preparation. Three major factors: activation temperature, activation duration and steam flow rate chosen in the present study are found to have significant effect on the iodine number. Three-dimensional response surfaces are created to analyze the combined effect of the three factors on the iodine number. Fig. 5 shows the combined effect of activation temperature and activation duration on the iodine number while the third parameter steam flow rate is held constant at 0.5 ml/min. As can be seen, the iodine number of EAAC gradually increased with the increase in the activation temperature and activation duration, with the maximum iodine number of about 1,000 mg/g, corresponding to the maximum of activation temperature and activation duration. An increase in activation temperature is expected to reduce the activation energy for the carbon-steam reaction and would increase the rate of the reaction which contributes to the increase in porosity of the EAAC. However, rate of increase iodine number for both the parameters were found to reduce at higher temperature and duration, which could be due to the increase of iodine number gradually became slow after 900 °C, this phenomenon implied that the formation of new pores became less significant as the activation proceeded. Another possible reason for the loss of iodine number was that the micropores might be destroyed at the high temperature. Similarly an increasing in activation duration would increase the extent of the carbon-steam, indicating that the pores were formed and enlarged simultaneously. A similar tendency has been found in the preparation of AC from Jatropha hull [27].

Fig. 6 shows the combined effect of steam flow rate and activation temperature while the activation duration is held constant at 30 min. An increase in steam flow rate is found to increase the iodine number until an optimum steam flow rate beyond which contributing to reduction in iodine number, which is qualitatively similar to the trend observed with increase in activation temperature. The increase in iodine number with steam flow rate is not as significant as compared with the activation temperature. The increase in iodine number with increase in steam flow rate could be attributed to reduction in mass transfer

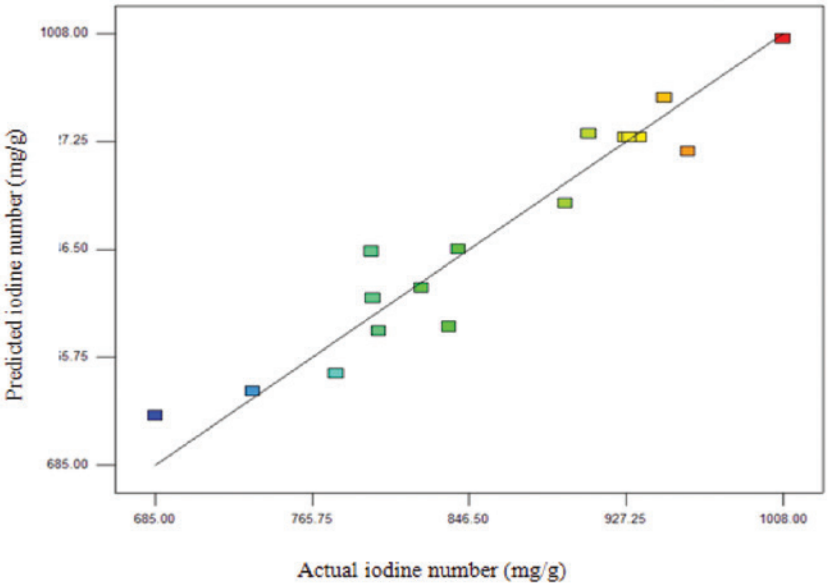

Fig. 3: Predicted vs. experimental iodine number of AC with microwave heating

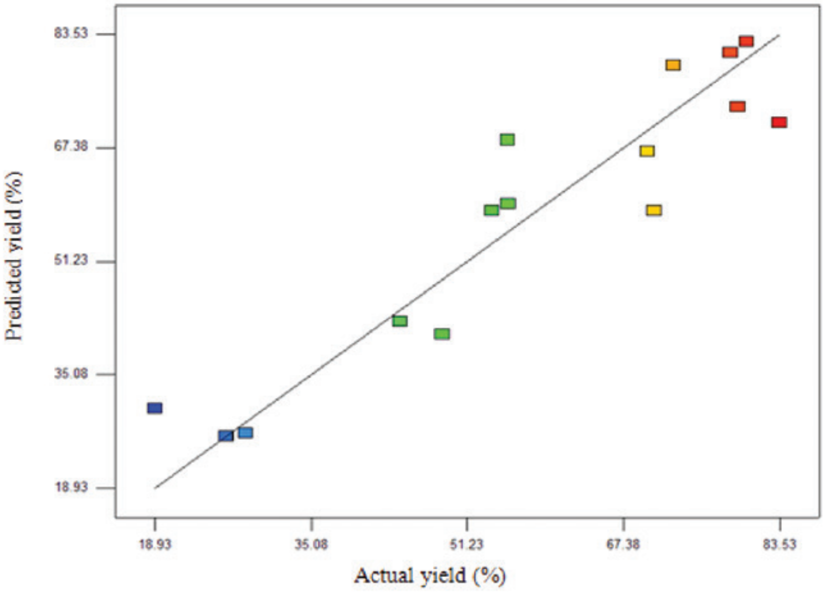

Fig. 4: Predicted vs. experimental yield of AC with microwave heating

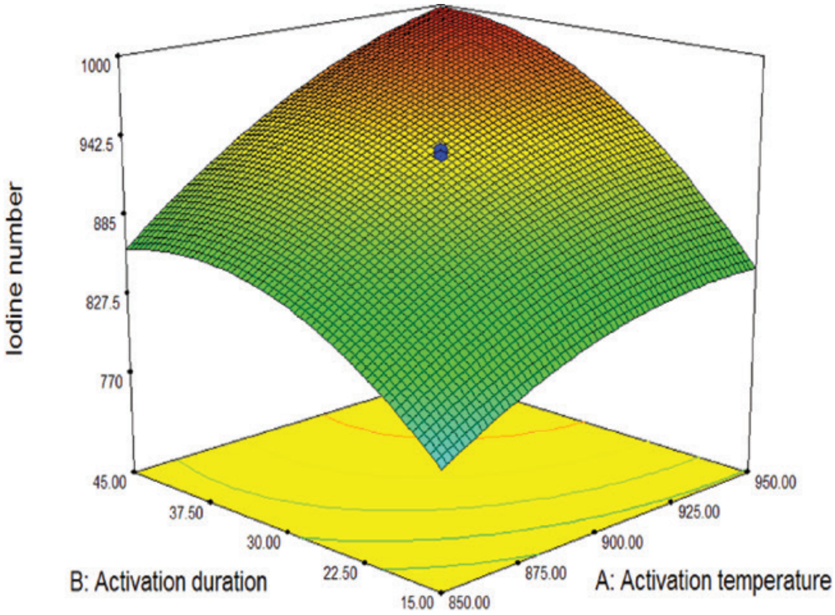

Fig. 5: Three-dimensional response surface plot of iodine number: effect of activation duration and activation temperature on iodine number (steam flow rate: 0.5 ml/min)

resistance on the steam side facilitating increase in the rate of reaction between the steam and carbon. A reduction beyond an optimum steam rate could be due to the higher extent of steam-carbon reaction contributing to pore merger as observed with the increase in temperature. Table 5 lists the comparison of maximum iodine number of various AC derived from different precursors reported in the literature. As can be seen, the EAAC prepared in the present work has relatively high iodine number as compared with the values reported in literature.

3.3 Activate carbon yield

The yield of AC is an important parameter, which is affected by the activation process and the activation con-

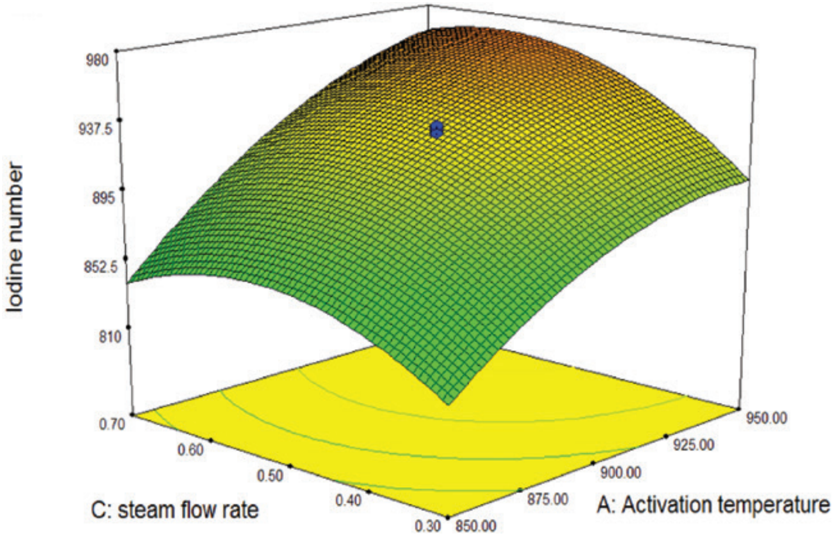

Fig. 6: Three-dimensional response surface plot of iodine number: effect of steam flow rate and activation temperature on iodine number (activation duration: 30 min)

Table 5: Comparison iodine adsorption of AC prepared from different biomass

Precursors	Heating method	Activating agent	Activation duration (min)	Iodine adsorption (mg/g)	Reference
Eupatorium adenophorum	Microwave heating	Steam	30	1,008	Present study
Edible fungi residue	Microwave heating	K_2CO_3	16	732	28
Phenolic resin	Conventional heating	Steam	60	1,050	29
Rice bran	Conventional heating	Steam	90	220	30
Olive-waste cakes	Conventional heating	H_3PO_4	120	583	31

ditions. Table 2 compiles the yield of EAAC for each experiment. Fig. 7 shows the combined effect of activation temperature and activation duration at a steam flow rate of 0.5 ml/min, while Fig. 8 shows the combined effect of activation temperature and steam flow rate at activation duration of 30 min. From both the figures, the yield of EAAC is found to decrease with the increasing of all the three parameters. Also the results indicate the minimum of yield corresponds to the maximum of the three parameters. An increase in any of the three parameters effectively contributes to an increase in the extent of carbon-steam reaction, contributing to the reduction in the yield of EAAC. Similar yield have been report in literatures for other precursors, namely for holm-oak sawdust (25.5%) and rockrose (20%) [32].

3.4 Process optimization

Industrial production of AC augur a high iodine number and yield as both of which contribute to improve the economics of commercial manufacture. However, both the response variables the iodine adsorption capacity (Y_1) and yield (Y_2) respond opposite to each other, demanding identification of an optimum combination of the parameters that maximize the iodine number and the yield of carbon. The optimum conditions was identified by invoking the optimization tool available with the Design Expert Software which correspond to an iodine number of 1,005 mg/g and a yield of 19.5%. In order to verity the reliability of identified optimized experimental conditions, three repeat runs were conducted and the average of the three runs are posted in Table 6. The proximity in iodine number and the yield between the repeat runs and the optimized conditions validate the success of the optimization process.

3.5 Characterizations of pore structure

The nitrogen adsorption isotherm of char and EAAC under the optimum condition estimated using the Autosorb

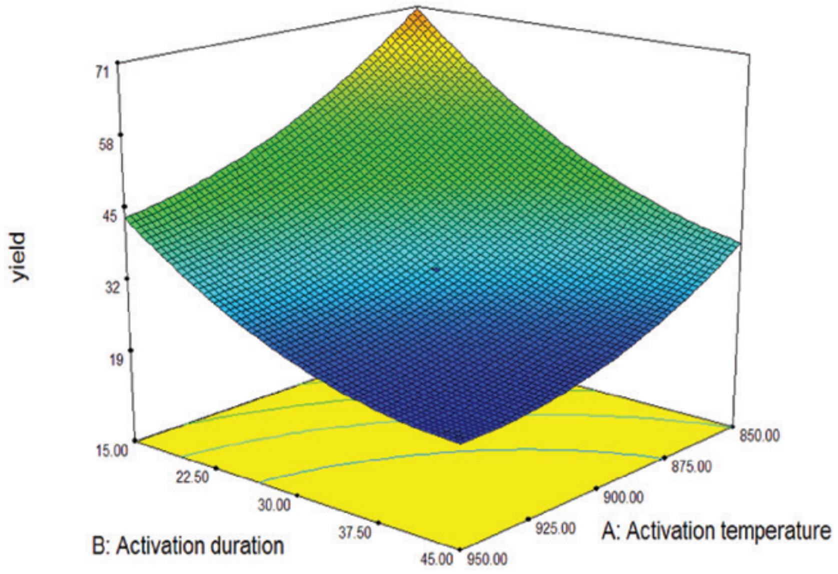

Fig. 7: Three-dimensional response surface plot of activated carbon yield: effect of activation temperature and activation time on yield (stem flow rate: 0.5 ml/min)

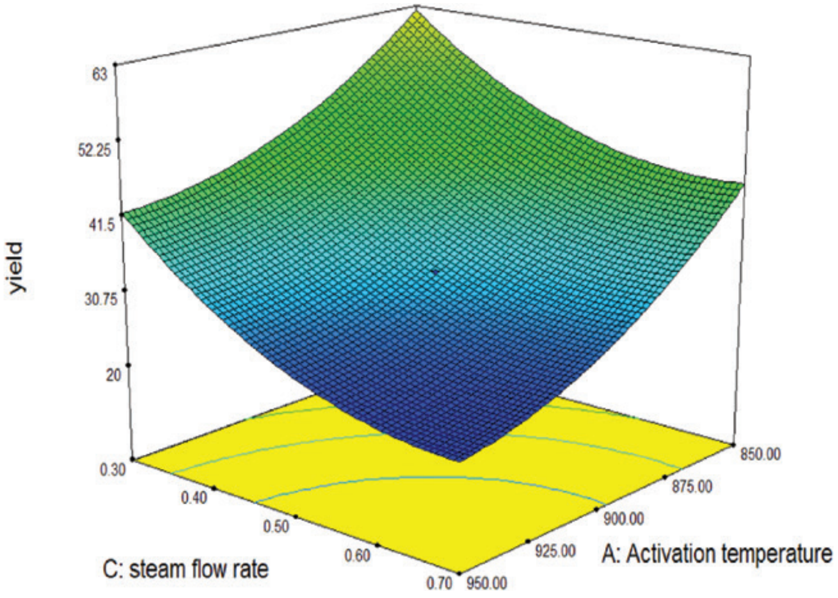

Fig. 8: Three-dimensional response surface plot of activated carbon yield: effect of stem flow rate and activation temperature on yield (activation duration: 30 min)

instrument at 77 K is shown Fig. 9. The shapes of the isotherm pertain to adsorption isotherm type II under the IUPAC classification [33], which indicates the filling of micro pores. Obviously the EAAC present a high adsorption capacity at the low pressures because of the well development pore structure. The cumulative pore volume plots shown in Fig. 10, while Fig. 11 shows the pore size distribution, both of which substantiate the amount of pores in the mesoporous range, with the average pore diameter estimated to be 3.3 nm. The surface area of the activation is estimated to be 1142 m^2/g, while the total pore volume is 0.84 ml/g.

Table 7 provides the pore volume corresponding to the micropore and mesopore along with average pore

Table 6: Validation of process optimization

Activation temperature X_1 (°C)	Activation time X_2 (min)	Steam flow rate X_3 (ml/min)	Iodine number (mg/g)		Yield (%)	
			Predicted	Experimental	Predicted	Experimental
950	45	0.7	1,005	1,010	19	20.13

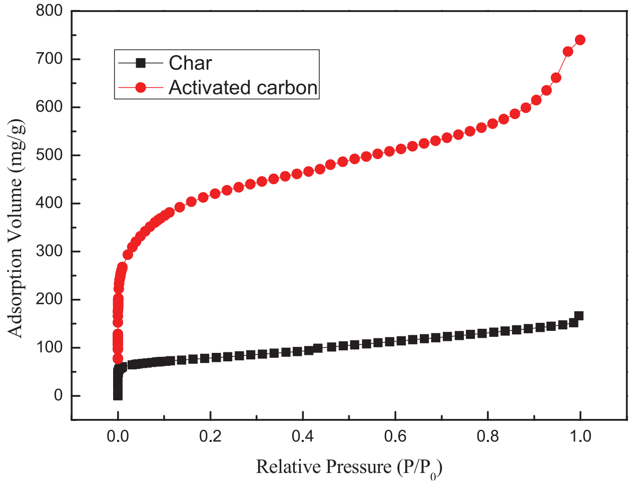

Fig. 9: Nitrogen adsorption isotherm of the activated carbons and char

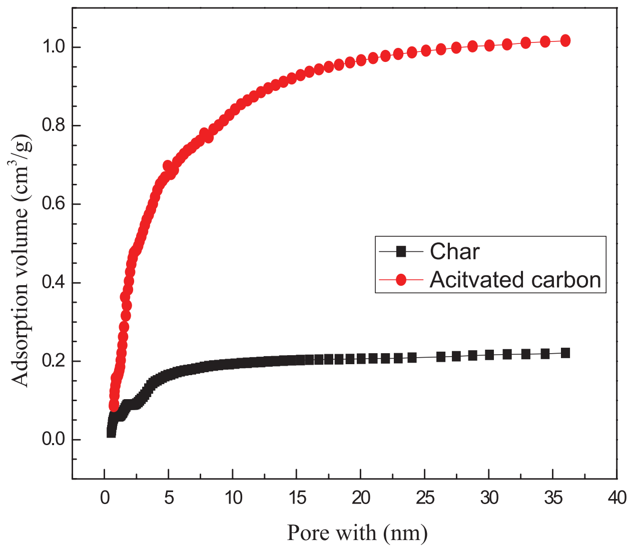

Fig. 10: Cumulative pore volume distribution chart for EAAC and precursors

diameter of the spent and regenerated activated carbon. A comparison of the quality of EAAC with the char exhibits a significant increase in the pore volume, the micropore volume and the surface area attributed to the activation process.

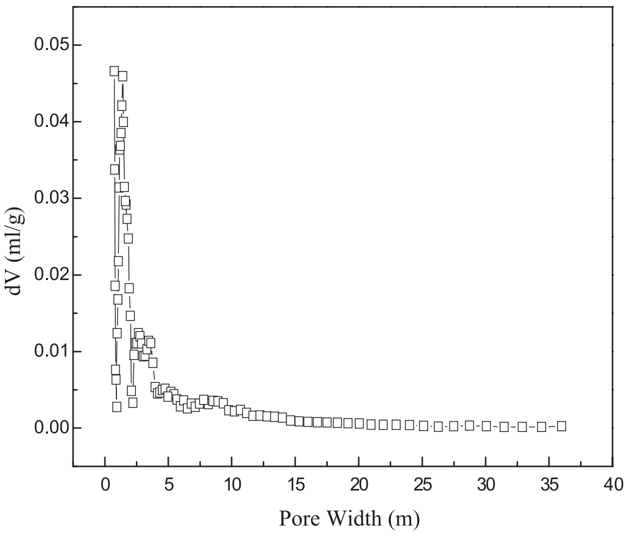

Fig. 11: Pore size distribution chart for EAAC

Table 7: Pore structural parameters of EAAC vs. chars

Properties	EAAC	Chars
Pore volume (ml/g)	0.84	0.23
Average pore diameter (nm)	3.3	3.6
Micropore volume (%)	38.7	27.1
Mesopore volume (%)	61.3	72.9
BET Surface area (m^2/g)	1142	260

3.6 SEM analysis of microstructure

Fig. 12(a) shows the SEM microstructure of precursors while Fig. 12(b) shows the SEM microstructure of regenerated the AC. The surface of the precursor is devoid of any tangible pores since it is covered by impurities. However, as shown in Fig. 12(b), the surface of EAAC has large number of pores of irregular and heterogeneous morphology, which attests a significant development of pore structure. A comparison of the microstructure of EAAC with the precursors indicates that the activation process plays an important role in removing surface impurities contributing to pore-formation.

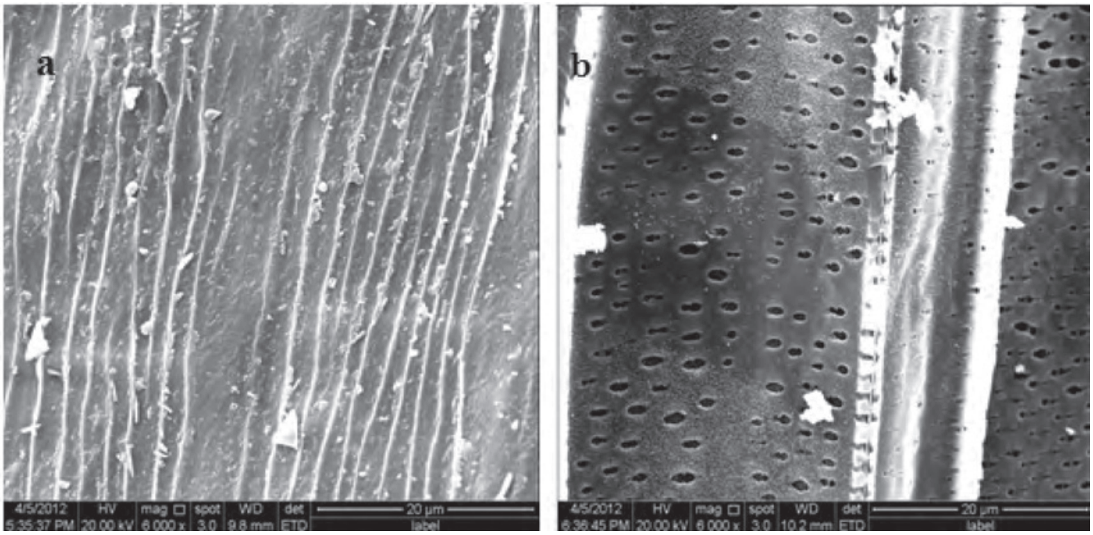

Fig. 12: SEM images of char (a) and EAAC (b)

4 Conclusions

Eupatorium adenophorum, a harmful biomass is utilized for preparing AC with microwave heating exhibit well developed pore structure. The effect of three vital process parameters, activation temperature, activation duration and steam flow rate have been assessed on the adsorption capacity and yield of AC. The process parameters were optimized utilizing the Design Expert software and were identified to be an activation duration of 45 min, an activation temperature of 950 °C and a steam flow rate of 0.7 ml/min, with the resultant iodine number and yield being 1,010 mg/g and 20.13% respectively. The key parameters that characterize quality of the porous carbon such as the BET surface area, total pore volume and average pore diameter were estimated to be 1,142 m^2/g, 0.84 ml/g and 3.3 nm respectively, for the sample corresponding to the optimized process conditions. Additionally the pore structure is characterized using Scanning Electron Microscope (SEM).

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