Activated Carbon from *Fenugreek* Seed: Characterization and Dyes Adsorption Properties

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ABSTRACT

In this study the preparation of activated carbon from agricultural product were reported. The fenugreek seeds were activated by chemical and microwave activation methods. The characterization of carbon materials were carried out using different techniques like Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscopy (SEM), Thermo Gravimetric Analysis (TGA), X-Ray Diffraction (XRD). Adsorption experiment was carried out for the adsorption of two different basic dyes, Methylene Blue (MB) and Crystal Violet (CV) under varying conditions such as concentration of dye, mixing time and adsorption temperature. The data acquired from concentration variation were found to fit well with Langmuir and also Freundlich isotherms. Kinetic studies revealed the fact that the system follows second order. Thermodynamic parameters like $\Delta H \neq$, $\Delta S \neq$ and $\Delta G \neq$ were calculated from the study on adsorption at different temperature. The adsorbent preparation via microwave technology is a desired methods which is resulted in conservation of energy for an efficient production of activated carbon.

INTRODUCTION

Adsorption, a surface phenomenon occurs in most of the physical, biological and chemical systems. The term adsorption refers to the existence of a higher concentration of any particular component at the surface of a liquid or solid phase than is present in the bulk [1, 2]. Adsorption has many applications in industries particularly in decolourisation, drying gas, catalysis, water softening [3] etc. Activated carbon is the most widely used adsorbent around the world with application in diverse areas due to its adsorption capacity, variable surface chemistry characteristics and high chemical resistance [4]. Adsorption of organic substances by activated carbon results in specific interactions between functional group on the adsorbent and on the surface of the adsorbents [5]. This distinctive feature makes activated carbon as an excellent adsorbent in food industry, pharmaceutical, chemical, petroleum, nuclear, automobile and vacuum industries. These are also used for the removal of organic pollutant like phenols [6], metal ions from water [7], decolourization [8], dechlorination [9], filtration of gases and liquids [10-12] etc. Physical and chemical activation are the two most extensively used methods for the synthesis of activated

carbon. These days microwave heating technology emerging as a promising method in the production of activated carbon, owing to the fact that this method is very effective compared to other methods [13, 14].

Consumption of refined sugar (refined from bone char) or direct ingestion of activated carbon (made from wood, coir husk, fossil fuels, etc.) in the form of dietary supplement may lead to serious health problems. Therefore work on agricultural related charcoal has been carried out which could be an alternative to commercial carbon. In this current study fenugreek seed, an agricultural product which is cheap and abundant was used as raw material for the preparation of activated carbon. Fenugreek (*Trigonella foenum - graecum*) belongs to family *fabaceae*. Fenugreek seeds (Fig. 1) are yellow-brown coloured which are mainly used for food and medicinal uses. The chemical constituent of fenugreek seed are proteins, amino acids, fatty acids, flavonoids, alkaloids, carbohydrates, saponins, sapogenins, fibres, etc. [15-18]. Present study includes the preparation of activated carbon by two methods namely, chemical activation and microwave treatment and its characterization. The prepared activated carbons were used as an adsorbent for the study of adsorption of methylene blue and crystal violet. Validation of two adsorption isotherms such as Freundlich and Langmuir

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isotherm has been studied. In addition, to know the feasibility of the reaction, evaluation of thermodynamic parameters has also been conducted.

Figure 1. Fenugreek seed

MATERIAL AND METHODS

Materials
All chemical reagents used in the experiments were of analytical grade.

Preparation of activated carbon
The fenugreek seeds were washed with water to remove dirt and dust. Later it was dried in an oven at 110ºC. Two-step activation procedure was adopted for chemical activation method. Firstly fenugreek seed powder was pre-carbonized at 250ºC for 2 hours in a hot air oven. The pre-carbonized sample was treated with 10% $ZnCl_2$ solution and agitated for 1 hour on a hot plate. Later it was filtered, dried; carbonization was carried at 400ºC for 1 hour in a muffle furnace. The sample obtained was washed with hot water as well as distilled water to eliminate remnant impurities, dried in an oven at 110ºC and resulted activated carbon is designated as ZFC. For the preparation of microwave treated carbon, the sample was directly subjected to microwave treatment (90 W of power) for 3 hours and the obtained carbon was labelled as MFC.

Characterization of activated carbon
Fourier Transform Infrared spectrum of activated carbon was recorded over the frequency range of 4000 to 400 cm^{-1} using IR Prestige-21 Fourier Transform infrared spectrometer, Shimadzu (Japan). Thermogravimetric study was carried out using the instrument SDT Q600 V20.9 (Japan) at a heating rate of 10ºC/ min under a nitrogen flow of 10ml/min. X - Ray Diffraction study was performed by Rigaku Miniflex 600 (Japan). Scanning Electron Micrographs were obtained using Sigma Series Field Emission Scanning Electron Microscope (Ziess, Germany).

Adsorption studies
Prepared activated carbons (ZFC and MFC) were used as an adsorbent for the adsorption of two different dyes, methylene blue and crystal violet under various conditions such as temperature, mixing time and concentration of the dye. For the adsorption studies, a definite amount of adsorbent of particle size 100-50 μm was added to a 250-ml conical flask containing 50 ml of the dye solution and agitated for fixed time span. Later the dye solution was filtered and its concentration was determined by spectrophotometer while observing corresponding λ_{max} values (664nm for Methylene Blue and 588nm for Crystal Violet). The amount of methylene blue adsorbed, q_e (mg/g), by carbon at time t, was calculated by the following equation:

$$q_e = \frac{(Co - Ce)V}{W} \tag{1}$$

where Co and Ce are initial and equilibrium concentration (mg/l), V is volume of solution (l) and W is the weight of activated carbon(g).

RESULTS AND DISCUSSION

Characterization of activated carbon

FTIR analysis
The functional groups present on activated carbon were identified by the observed peaks in an FTIR spectrum obtained for activated carbons. The FTIR spectrum obtained for raw fenugreek seed (RFS) powder and prepared activated carbons (MFC and ZFC) are shown in Fig. 2. The absorption bands for activated carbons are listed in Table 1 with their structural assignments.
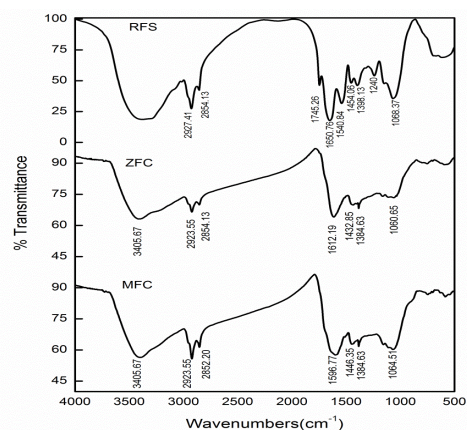
The broad but intense peak in the range 3400 - 3300 cm^{-1} may be due to conjunction of O–H and N–H stretching. The bands at 2927.41 cm^{-1} and 2854.13 cm^{-1} are attributed to the asymmetric and symmetric stretching of C – H bonds respectively. The peak formed at 1745.26 cm^{-1} is due to C = O stretch for esters. The bands at 1650.76 cm^{-1} and 1540.84 cm^{-1} indicates N–H bending of primary amines and secondary amines respectively. The weak bands occurred at 1454.06 cm^{-1} may be the result of vibrations of C–O–H bonds. The band formed at 1398.13 cm^{-1}may be due to bending mode of CH_2 groups. The band at 1240 cm^{-1} is due to C–N stretching in amines and the band at 1068.37 cm^{-1} may be due to C–O stretching of saponins.

A notable difference was observed when the study was further extended to correlate the spectra of both the raw fenugreek seed powder and the synthesised activated carbons. The peaks which are accountable for vibrations of N–H, O–H, C–H, C–O–H and C–O bonds are retained in the spectra of both MFC and ZFC where as the bands which are responsible for carbonyl stretching, N–H bending (Secondary amines) and C–N stretching are

absent in the spectrum of activated carbons (MFC and ZFC). The disappearance of bands may be attributed to the release of CO_2 by carbonyl group and decomposition of C–N bond, at higher temperature (400^0C). MFC and ZFC showed similar spectrum with varied intensities and this variation in intensities may be due to impregnation of activated carbon with zinc chloride. El-Bahy [19] studied the composition of fenugreek seeds through FTIR and FT Raman spectra measurements. The obtained FTIR frequency, v values are well compared (see Table 1). Peaks present in IR spectra indicated the presence of hetero atoms (N, O) and some π bonds on the surface of activated carbon, and they are believed to be are very active towards the adsorption process and hence the prepared carbons can be used as adsorbents.

Figure 2. FTIR spectrum of raw fenugreek seed and activated carbon (ZFC &MFC)

XRD Analysis

The crystallographic characters of the raw sample and activated carbon were analysed by XRD with Cu-Kα radiation. The XRD pattern obtained is shown in Fig. 3. Considerable difference was noticed in the obtained XRD patterns. Raw seed sample exhibited amorphous state where as few peaks were spotted in the X-ray patterns of activated carbon and the difference in the XRD patterns could be due to the activation process. Presence of few sharp peaks in the midst of some weak peaks confirms that the activated carbons exhibit most likely, a mixture of single crystal and mixture of crystallites. Based on the intensity of existing peaks and 2θ values inter-planar distance d, hkl values and cell volume, a^3 were determined and obtained data are shown in Table 2. The interlayer spacing d is determined by using Bragg's equation:

TABLE 1. Spectrum assignment of fenugreek seed and synthesised activated carbons

RFS	MFC	ZFC	Assignment
3400-3300	3405.67	3417.24	N–H stretching, O–H stretching
2927.41	2923.55	2923.55	Asymmetrical – CH_2- stretching
2854.13	-	-	
1745.26	2852.20	2854.13	Symmetric –CH_2- stretching
1650.76	1596.77	1612.19	
1540.84	-	-	C=O stretching
1454.06	1446.35	1432.85	N–H bending (primary amines)
1398.13	1384.63	1384.63	N–H bending (Secondary amines)
1240.00	-	-	C–O–H bending
1068.37	1068.37	1060.65	(C–H_2) bending
			C–N stretching in amines
			C–O stretching

TABLE 2. XRD data for MFC and ZFC

Sample	2θ (degree)	d (A°)	N	hkl	A (A°)	Cell volume a^3(A°)	Crystallite size D(A°)
MFC	19.74	4.51	1	100	4.51	91.73	0.075
	20.88	4.27	1	100	4.27	77.85	0.075
	21.00	4.24	1	100	4.24	76.76	0.075
	21.55	4.14	1	100	4.14	71.47	0.075
	21.82	4.08	1	100	4.08	68.41	0.075
	22.44	3.97	1	100	3.97	63.04	0.075
	24.06	3.71	1	100	3.71	51.06	0.076
	28.06	3.19	2	110	4.51	91.73	0.076
	40.30	2.24	4	200	4.49	90.51	0.079
	17.56	5.07	1	100	5.07	130.47	0.066
ZFC	23.02	3.87	2	110	5.48	165.06	0.067
	24.32	3.67	2	110	5.19	140.30	0.067
	25.1	3.56	2	110	5.03	127.82	0.067
	26.16	3.42	2	110	4.83	113.37	0.067

TABLE 3. Langmuir and Freundlich isotherm constants and correlation coefficients for adsorption capacity

Sample	Dye	Freundlich Isotherm			Langmuir Isotherm			
		n	K_f	R^2	Q_o	*b*	R_L	R^2
MFC	MB	2.20	2.28	0.98	35.08	0.13	0.27 – 0.07	0.99
	CV	2.53	2.54	0.97	37.39	0.17	0.21 – 0.05	0.98
ZFC	MB	1.56	2.12	0.97	64.43	0.06	0.42 – 0.13	0.97
	CV	1.46	1.89	0.97	64.64	0.05	0.48 – 0.15	0.99

. TABLE 4. Second order rate constants for the adsorption

Sample	Dye	q_e	k_2	R^2
MFC	MB	21.82	0.184	0.99
	CV	25.12	0.065	0.99
ZFC	MB	25.92	0.122	0.99
	CV	27.67	0.059	0.99

TABLE 5. Thermodynamic parameters for the Adsorption of MB and CV on activated carbon

Sample	Dye	Temperature (^0C)	K_L (L/mol)	$\Delta G^{\neq}$ (kJ/mol)	$\Delta H^{\neq}$ (kJ/mol)	$\Delta S^{\neq}$ (kJ/mol/K)	Ea (kJ/mol)
MFC	MB	30	11.36×10^3	- 23.52			
		40	20.41×10^3	- 25.82	57.87	0.268	60.39
		50	47.29×10^3	- 28.90			
	CV	30	14.04×10^3	- 24.05			
		40	24.04×10^3	- 26.25	50.86	0.246	53.46
		50	49.15×10^3	- 29.01			
ZFC	MB	30	22.98×10^3	- 25.29			
		40	44.33×10^3	- 27.84	49.15	0.245	51.75
		50	76.88×10^3	- 30.21			
	CV	30	60.88×10^3	- 27.75			
		40	10.55×10^4	- 30.09	42.11	0.230	44.71
		50	17.13×10^4	- 32.36			

$$d = \frac{\lambda}{2 Sin\theta} \qquad (2)$$

where $\lambda = 1.5418 A^o$ (CuKα) and Θ is the scattering angle. The value of 'a' (Table 2) appears to be the same at different Θ values which indicates the cubic pattern for the system. The crystallite size D, was determined from the Scherrer equation:

$$D = \frac{K\lambda}{B cos\theta} \qquad (3)$$

where K is the Scherrer constant value from 0.9 to 1, λ is the wavelength of the X – ray radiation and B is the width at half maximum [20].

Among the two synthesised activated carbons (Table 2) the higher cell volume and lower crystallite size of ZFC indicates likely higher adsorption capacity compared to MFC.

Thermo gravimetric analysis

The TG curve of raw fenugreek seed (RFS), MFC and ZFC obtained and are shown in Fig. 4. A considerable difference was observed in the pyrolysis behaviour of the raw fenugreek seed and synthesised activated carbons (MFC and ZFC). Weight loss in the range 20 - 200ºC was the result of evaporation of moisture or loosely bound water molecules. RFS showed rapid degradation in the range of 200-500ºC which slowed down there after, whereas steady decomposition was observed for MFC (200 -700 ºC) and ZFC (200- 800 ºC). Decrease in mass beyond 200ºC may be due the loss of water of crystallization or degradation of sapogenins, alkaloids and fibers present in the activated carbon. Retaining of the weight till about 500ºC and later decrease probably indicates the increase in the pores of the activated carbon which helps in the increase of adsorption.

Scanning electron microscopic (SEM) analysis

Surface morphologies, before and after adsorption were examined by SEM. Figs. 5a and 5b show the scanning electron micrographs of MFC and ZFC, respectively. It is evident from the images that both of the activated carbons show different surface structure and occurrence of large number of cavities, which plays key role in the process of adsorption. Figs. 5c, 5d, 5e and 5f are the post adsorption images; indicate blocking the surface cavities by the dye molecules. That is to say the interaction between the dye and the active centres of the activated carbon has taken place.

Characterization study indicates that the activated carbon prepared in the present study is a good adsorbent. Hence the adsorption study was carried out.
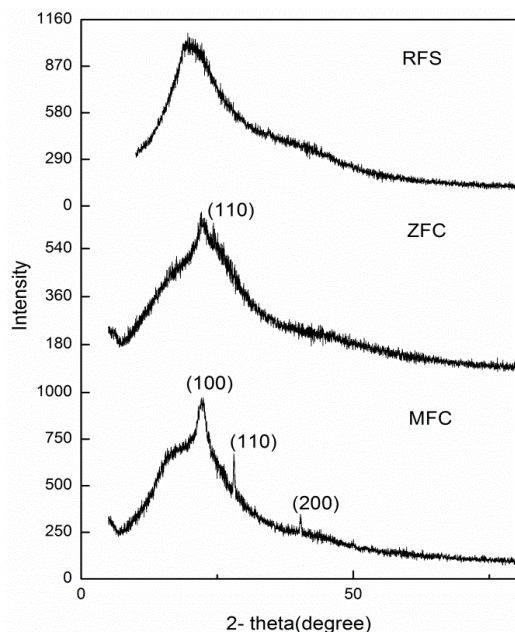

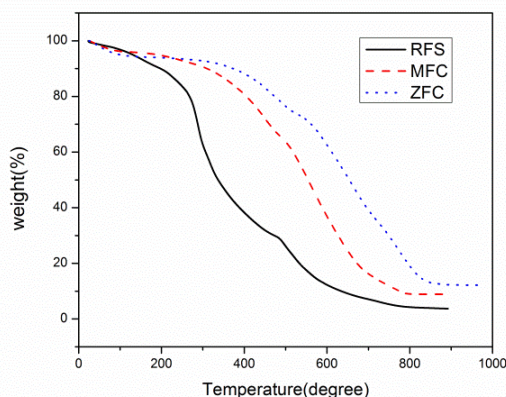**Figure 3**. XRD of RFS and activated carbon (ZFC and MFC)

Figure 4. Thermo gravimetric curve of RFS, MFC and ZFC

Adsorption studies
Adsorption isotherms
Langmuir and Freundlich adsorption isotherm models were employed to describe the adsorption process. The linear form of the Freundlich Isotherm is given as [21]

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \qquad (4)$$

where n shows the adsorption intensity in the Freundlich equation and K_f is the Freundlich adsorption capacity $[(mol/g)(L/g)^{1/n}]$. The values of n and K_f were calculated by the slope and intercept obtained from the plot of $\log q_e$ vs. $\log C_e$ (Figs. 6(a) and 6(b))

The Langmuir equation is expressed by the following expression [22].

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \qquad (5)$$

where Ce (mg/L) is the equilibrium concentration of the solute, q_e is amount of solute adsorbed at equilibrium (mg/g) and Q_o (mg/g) and b (L/mg) are constants related to the adsorption capacity and energy of adsorption respectively. The values of Q_o and b were calculated from the slope and intercept from the plot of C_e/q_e vs. C_e. (Figs. 7(a) and 7(b)). The separation factor, R_L is calculated as:

$$R_L = \frac{1}{1 + bC_0} \qquad (6)$$

where C_0 is the initial concentration of the adsorbate. R_L value shows the form of the isotherm to be either linear ($R_L=1$), irreversible ($R_L= 0$), favourable ($0< R_L <1$) or unfavourable ($R_L >1$) [23].

The results (Table 3) indicate that both the Langmuir and Freundlich models are equally applicable for the present system. R_L values lies between 0 and 1, implies that the adsorption process is favourable and also the values of n (>1) are supportive to the adsorption process.

Effect of contact time
The variation in the adsorption of the dyes with contact time (15 – 120 min) is shown in Fig. 8. The result signifies the sharp increase in adsorption in the beginning (from 15-60 min) with later decline (from 60 – 120 min). In the initial stage, rate of adsorption is high due to availability of the active sites on the surface of the adsorbent. The maximum adsorption is found to be at 60 minutes for both the dyes. After 60 minutes the decrease in the adsorption is likely to be due to scarcity of vacant sites on the adsorbent. So the adsorption appears to decrease and it continued till the end of the study. Rate of adsorption appear to be more in case of ZFC than MFC for both the dyes.

Adsorption kinetics
In this investigation, the adsorption mechanism was studied by fitting first and second order rate expression to the experimental data. The first order kinetic model can be expressed as follows [24]:
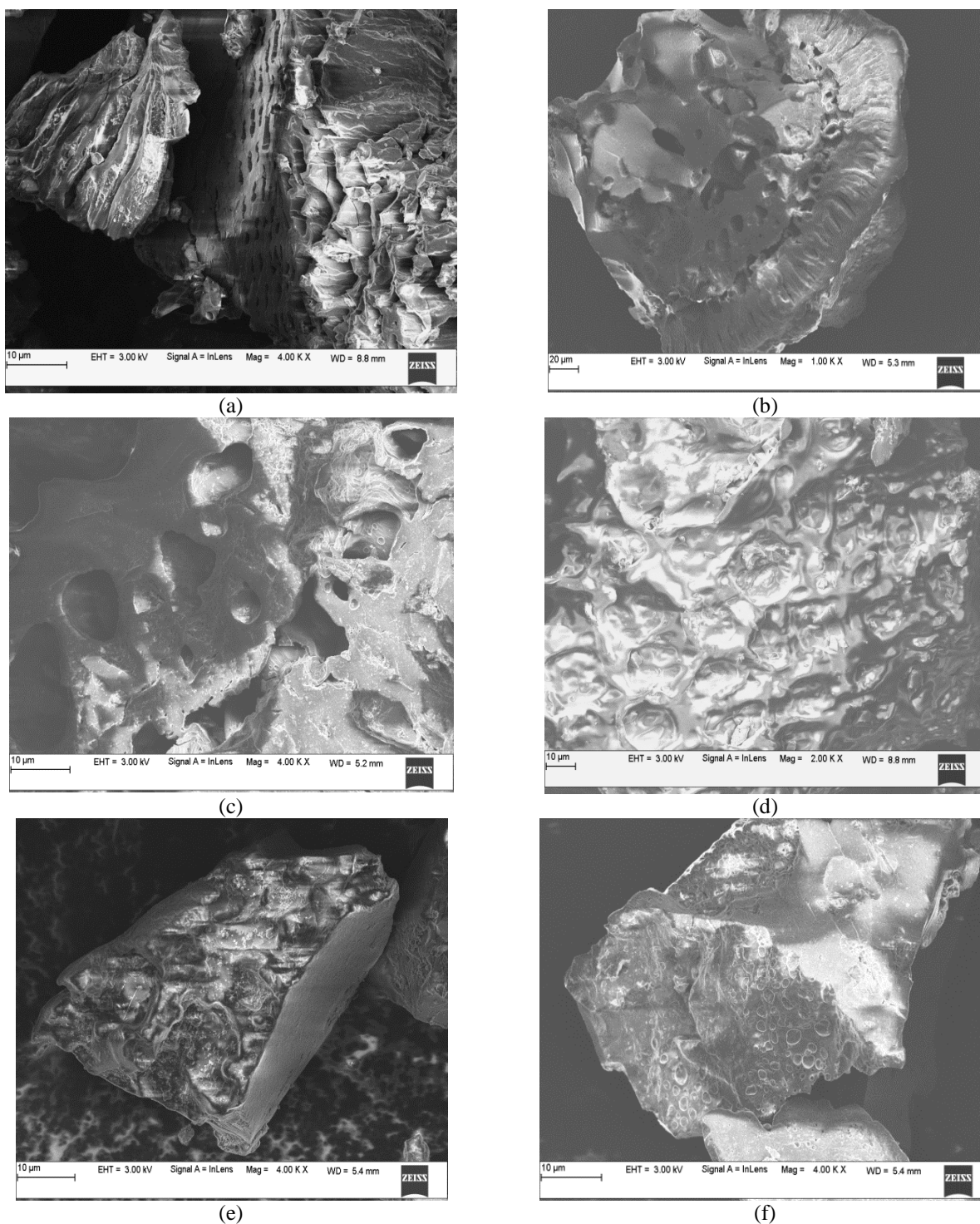
Figure 5. Scanning Electron Micrographs of activated carbon (5a), (5b) ZFC and MFC before adsorption, (5c) ZFC after adsorption of MB, (5d) MFC after adsorption of MB, (5e) ZFC after adsorption of CV, (5f) MFC after adsorption of CV.

$$\ln(q_e - q_t) = \ln q_e - k_1 t \qquad (7)$$

where k_1 is the equilibrium rate constant of the first order adsorption, and q_e and q_t refer to amount of dye adsorbed (mg/g) at equilibrium and at any time, t (min), respectively. The data obtained in this experimental

work, did not fit well for the first order model. The linear form of the second - order kinetic model is represented as follows [25]:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \qquad (8)$$

(a)

(b)
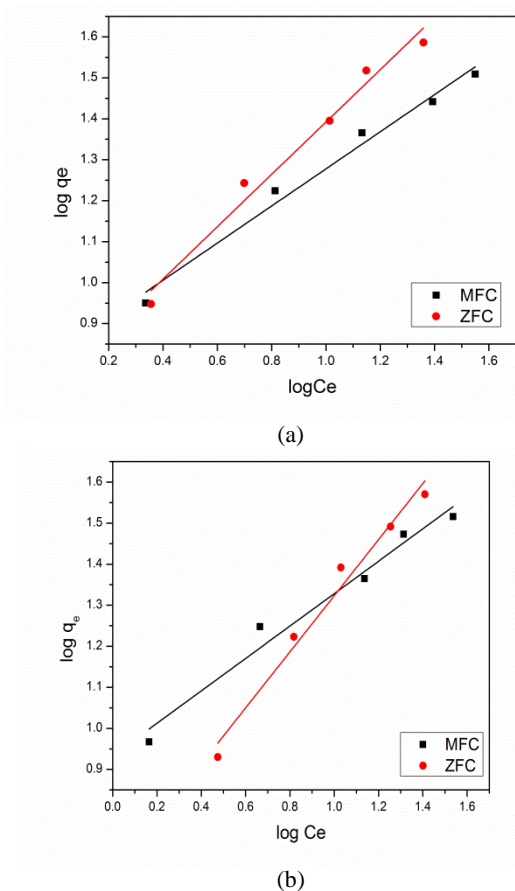
Figure 6. (a) Freundlich Isotherm for MB adsorption (b) Freundlich Isotherm for CV adsorption

where q_e and q_t are the adsorption capacities at equilibrium and time t respectively and k_2 is the second order rate constant for adsorption ($(mg/L)^{-1}$ min^{-1}). The rate constant k_2 and q_e were computed respectively from the intercept and slope of the linear plot of t/qt vs. t (Fig 9). The value of R^2 (Table 4) confirms that second - order kinetic model fits best for the adsorption process.

Effect of temperature
Effect of temperature on the adsorption was carried out in the temperature range 20 – 70 $^\circ$C. The result indicated that with the increase in temperature there is increase in the adsorption capacity of the carbon up to a certain temperature (50^{0}C) with a later decrease in adsorption (Fig. 10). Increase in the adsorption capacity may be due to increase in the mobility of the dye molecules with an increase in their kinetic energy as well as increase in the pore volume of an adsorbent [26]. The decrease in adsorption capacity with increase in temperature is due to weakening of the intermolecular attractive forces between the active sites on the adsorbent and dye molecules, and also between the adjoining dye molecules on the adsorbed phase [27].

Adsorption thermodynamic
From the study on the variation of temperature (30 - 50 ^{0}C), thermodynamic parameters $\Delta H^{\neq}$, $\Delta S^{\neq}$ and $\Delta G^{\neq}$, for the adsorption were calculated. For a dilute solution of charged adsorbate, $\Delta G^{\neq}$ can be determined by using Langmuir equilibrium constant [28, 29]:

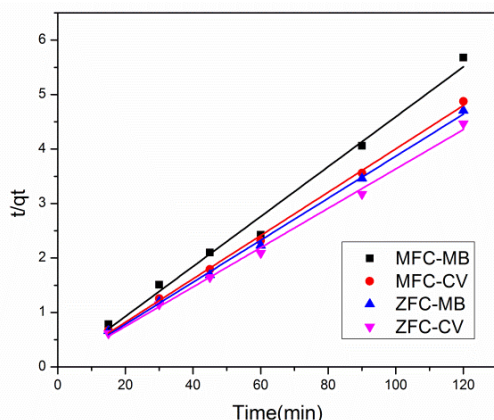$$q_e = q_{max} \frac{C_e K_L}{C_e K_L + 1} \qquad (9)$$
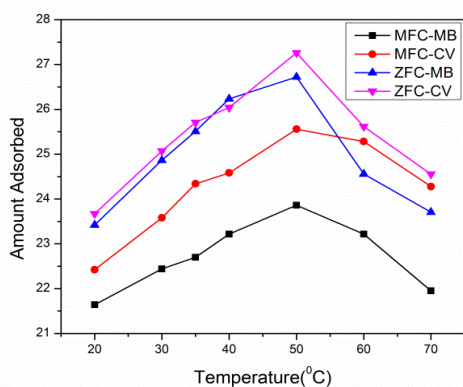

(a)

(b)
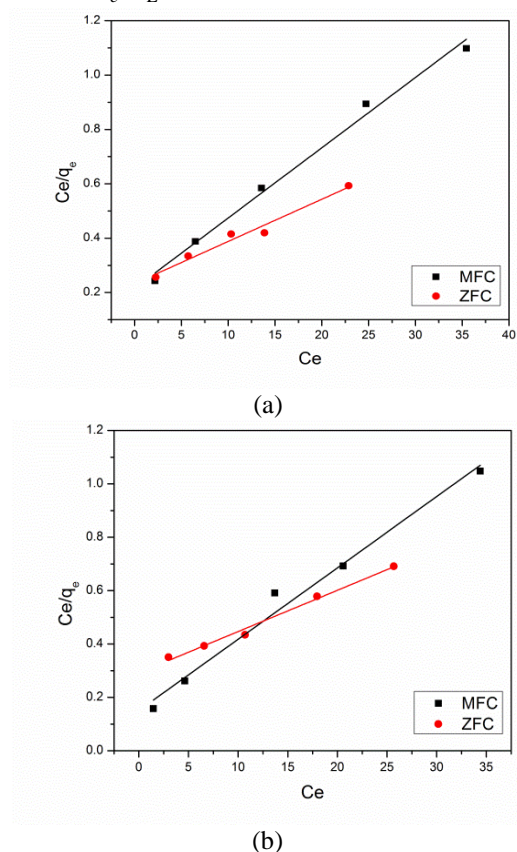
Figure 7. (a) Langmuir Isotherm for MB adsorption (b) Langmuir Isotherm for CV adsorption
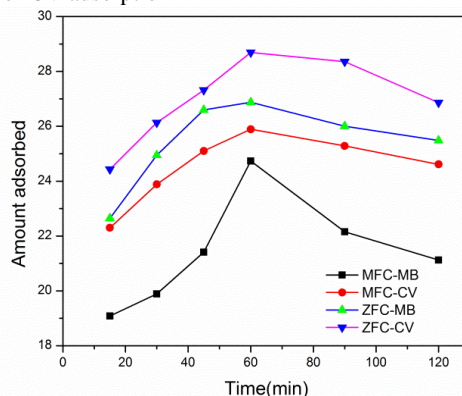
Figure 8. Effect of contact time for MB and CV adsorption

Figure 9. Plot of second – order kinetic modelling of the adsorption process

Figure 10. Effect of temperature for MB and CV adsorption

where q_e and q_{max} are the adsorption capacity of adsorbent at equilibrium (milligrams per gram) and its maximum value, C_e is the equilibrium concentration of adsorbate in solution (moles per liter) and K_L is the Langmuir equilibrium constant of adsorption (litres per mole). Change in free energy, $\Delta G^{\neq}$ is calculated as

$$\Delta G^{\neq} = -RT \ln K_L \qquad (10)$$

where R is the gas constant, T is the absolute temperature. Enthalpy and entropy of the adsorption process can be calculated from the van't Hoff equation:

$$\ln K = \frac{-\Delta H^{\neq}}{RT} + \frac{\Delta S^{\neq}}{R} \qquad (11)$$

Activation Energy, E_a was determined by using the following equation:

$$E_a = \Delta H^{\neq} + RT \qquad (12)$$

The values of $\Delta H^{\neq}$ and $\Delta S^{\neq}$ were calculated from the slope and intercept of the linear plot of lnK vs. $1/T$ (Fig. 11) and the results obtained are given in Table 5.

Positive values of $\Delta H^{\neq}$ and Ea indicate that the adsorption is an endothermic process and absorb necessary energy from the surroundings. The low values

of $\Delta H^{\neq}$ also specifies that the system experiences physisorption. Negative value of $\Delta G^{\neq}$ indicates the spontaneous nature of adsorption, which decreased with the increase in temperature. The change in $\Delta G^{\neq}$ with temperature suggests that adsorption is a favourable process and the positive values of $\Delta S^{\neq}$ state the involvement of slight disorderliness in the adsorption process.
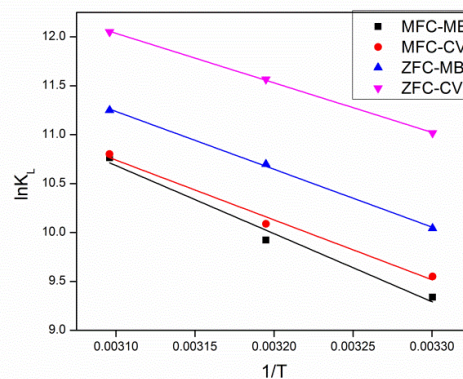
Figure 11. van't Hoff equation for MB and CV adsorption

CONCLUSION

Activated carbons were prepared from fenugreek seed, characterized by FTIR, XRD, SEM and TGA, and utilized for adsorption study. Results from the concentration variation indicated that both Langmuir and Freundlich Isotherm models could be used to fit the data. Kinetic studies revealed that the system follows second order rate. Thermodynamic parameters were calculated and the adsorption process was found to be endothermic and spontaneous.

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Persian Abstract

چکیده

در این مطالعه تهیه کربن فعال از محصولات کشاورزی گزارش شد است . بذر های زردچوبه با روشهای شیمیایی و میکروویوفعال میشوند. مشخصه های ماده کربن با روش های مختلفی مثل FTIR، SEM، TGA وXRD مشخص می‌شود .آزمایش جاذب برای جذب دو رنگ مختلف مهم، متیلن آبی و بنفش کریستالی تحت شرایط مختلف مانند غلظت رنگ، زمان مخلوط کردن و دمای جذب انجام شد. داده های بدست امده از تغییرات غلظت به خوبی با ایزوترم‌های لانگمویر و فرندلیچ مناسب بود. مطالعه سینتیکی این واقعیت که سیستم از درجه دو تبعیت میکند را نشان داد. پارامترهای ترمودینامیکی مانند ΔH، ΔS و ΔG از مطالعه جذب در دمای مختلف محاسبه شده است. آماده سازی جاذب از طریق تکنولوژی مایکروویو یک روش مطلوب است که منجر به حفظ انرژی برای تولید کارآمد کربن فعال می شود.