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Sorption Study of Methylene Blue on Activated Carbon Prepared from *Jatropha curcas* and *Terminalia catappa* Seed Coats

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Abstract: This research work targets the effectiveness of the prepared activated carbon from *Jatropha curcas* and *Terminalia catappa* seed coats for adsorption of methylene blue (MB) from aqueous solution. The prepared *Jatropha* activated carbon (JAC) and *Terminalia* activated carbon (TAC) were characterised using Fourier Transform Infrared Spectroscopy (FTIR), Scanning electron microscopy (SEM), and Brunauer- Emmett-Teller (BET) surface area analysis. Effects of initial concentration, pH, contact time, adsorbent dose and temperature on the adsorption experiments were studied and the concentration of methylene blue was monitored using spectrophotometry. The adsorption capacities of these adsorbents were found to be 37.84 mg/g and 17.44 mg/g for methylene blue uptake by JAC and TAC, respectively. The experimental data were analysed using Langmuir, Freundlich, and Dubinin-Radushkevich isotherms. The data fitted best into Langmuir isotherm for Methylene blue-JAC and Methylene blue-TAC systems. The kinetic studies fitted into pseudo second order kinetics model. The process chemistry was exothermic.

Keywords: Adsorption; activated carbon; methylene blue; kinetics; thermodynamics.

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INTRODUCTION

Organic dyes are used for textile dyeing and other purposes. Wastewaters emanating from these activities are discharged into aquatic environment as industrial wastes. These industrial effluents form part of the major environmental pollutants which in turn affect the survival of living organisms in the ecosystem [1]. Therefore, wastewaters from dyeing activities have to be treated for the protection of life and remediation of environmental pollution. Activated carbon is a known adsorbent of widely applied in the purification of polluted environment, treatment of gaseous effluents discharged from industry, and has additionally been effective for the removal of coloured substances in wastewater [2]. To improve the effluent quality before discharging into the environment, physical and/or chemical treatments comprising adsorption, photocatalytic degradation, electrochemical methods or reverse osmosis are necessary. Adsorption using activated charcoal is simple, cost effective, and efficient in removing organic and inorganic contaminants [3].

Activated carbon can be prepared from various wellsprings of biomaterials. The nature of the raw material utilised, activation process adopted, and processing conditions decide the properties and effectiveness of the activated carbon. The activation processes also have an influence on the surface properties of the activated charcoal [4]. Chemical activation uses chemical agents usually acids, bases or salts as activating agents while physical activation involves the use of carbon dioxide and steam as activating agents [4].

This research focuses on determining the potential of prepared activated carbon from seed coat of *Terminalia catappa* and *Jatropha curcas* for the removal of methylene blue (MB) in aqueous medium.

MATERIALS AND METHODS

Preparation of adsorbents

Termilania catappa and *Jatropha curcas* fruits were collected from University of Ilorin campus and a private estate at Oke Oyi, Kwara state respectively. The seed coats were appropriately washed, air-dried, and broken to pieces. The seed coats were then charred at 500 °C for 1h 30 minutes. The carbonised materials were ground and sieved with mesh that permits the entry of particles size < 90 μm [5]. The char was soaked in 0.5 M NaOH for 24 hours and then separated, washed to neutral pH, and air-dried. The subsequent material was later heated for 1 hour at a temperature of 200 °C to complete the process. The prepared activated carbons JAC and TAC were cooled and stored [6].

Characterization of the adsorbents

Proximate analysis

Moisture content: 0.20 g of the sample was heated at 105 °C for 2 hrs. The mass of the sample was determined after heating. *Ash content*: 0.20 g sample was heated at 750 °C for 1.5 h. The mass of the ash was determined after heating. *Bulk density*: 5.00 g of sample was weighed into a measuring cylinder and tapped until a steady volume is obtained. The mass was obtained by weighing [7].

Instrumental analysis

Morphological characterisation of the prepared JAC and TAC was done using SEM magnifying instrument (ASPEX 3020). 8400S Fourier transform infrared spectrometer was utilised to ascertain the functional groups on JAC and TAC. An ASAP 2020 BET instrument was employed to decide the BET surface area, pore volume and diameter of JAC & TAC [8].

Preparation of adsorbate solutions

Stock solution of 500 mg/L of methylene blue (see Figure 1 for the structure) was prepared and the required concentrations were obtained from the stock via serial dilution using deionised water. The concentration of MB was monitored using UV-Visible spectrophotometry (Beckman Coulter DU 730) at 664 nm. A calibration curve with the concentration extending from 5 to 100 mg/L was obtained [9].

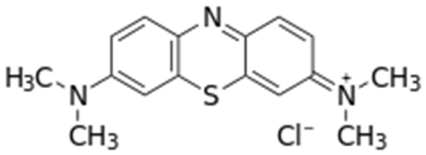

Figure 1: The structure of methylene blue.

Batch adsorption experiment

The influence of initial concentration, pH, contact time, adsorbent dose, and temperature on the adsorption of MB onto JAC and TAC were examined. A series of 20 mL MB solutions of different concentrations was prepared at controlled temperature of 28±2 °C. A 0.02 g of activated carbon was contacted with each solution and agitated in a mechanical shaker for 2 hours at 200 rpm. After centrifuging for 2 min, the final MB concentration in solution was monitored by UV- Vis spectrophotometer at the maximal absorbance wavelength of 664 nm. The amount of MB adsorbed was determined using Equation 1.

$$q_e = \frac{C_0 - C_e}{M} X\, V \qquad \text{(Eq. 1)}$$

where q_e denotes the quantity of MB adsorbed (mg/g), C_0 represents initial concentration of the MB (mg L^{-1}), C_e is the equilibrium concentration of the MB (mg L^{-1}) at time t, V is the

volume of the solution used (in litres, L) and m is the mass of the adsorbent utilised (g) [10, 11].

Adsorption isotherms

Freundlich isotherm

The linearised form of the equation is given by:

$$\log q_e = logK_f + \frac{1}{n}\, logC_e \qquad \text{(Eq. 2)}$$

q_e is the quantity adsorbed at equilibrium (mg/g), C_e is the equilibrium concentration of adsorbate (mg/L), 1/n is a heterogeneity factor, K_f is Freundlich's constant (mg/g) [12].

Langmuir isotherm

The linearised form of the Langmuir equation and the separation factor, R_L are given by Equations 3 and 4, respectively.

$$\frac{c_e}{q_e} = \frac{1}{k_L q_{max}} + \frac{1}{q_{max}} c_e \quad \text{....} \qquad \text{(Eq. 3)}$$

$$R_L = \frac{1}{1+K_L C_0} \qquad \text{.....} \qquad \text{(Eq. 4)}$$

Where C_e is the equilibrium concentration of the adsorbate (mg/L), q_e the amount adsorbed per adsorbent mass (mg/g), K_L is the Langmuir's constant and q_{max} is the maximal adsorption efficiency (mg/g). $0 < R_L < 1$ the adsorption is feasible [13].

Dubinin-Radushkevich isotherm

The D-R isotherm equation describes adsorption through multi-layer condensation [14, 15]. The linearised form is given by Equation 5:

$$In\ q_e = In\ q_d - \beta\ \varepsilon^2 \quad \text{.....} \qquad \text{(Eq. 5)}$$

where q_d is the D-R constant which relates to maximum adsorption efficiency (mg/g), β is a constant corresponding to free energy (mol^2/J^2) and ε is the Polanyi's potential which is defined in Equation 6 as:

$$\varepsilon = RT\ In\left[1 + \frac{1}{C_e}\right] \quad . \qquad \text{....(Eq. 6)}$$

where R is the gas constant and T is the absolute temperature. The mean free energy E is given by Equation 7 [14, 15].

$$E = \frac{1}{\sqrt{2\beta}} \quad \text{...} \qquad \text{..(Eq. 7)}$$

Adsorption kinetics

Kinetics study of the sorption experiment determines the adsorption efficiency and its potential applications. Pseudo first-order [16] and pseudo second-order [17] kinetics were used to assess the data obtained. The integrated forms of the equations are given by Equations 8 and 9, respectively:

$$log(q_e - q_t) = logq_e - \frac{k_1 t}{2.303} \quad \text{(Eq. 8)}$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad \ldots \quad \text{.(Eq. 9)}$$

k_1 (min^{-1}) and k_2 (g/mg.min) are rate constants of pseudo first-order and pseudo second order respectively. q_e and q_t are the amount of dye adsorbed (mg/g) at equilibrium and at time t, respectively.

Adsorption thermodynamics

The thermodynamics parameters [18,19] were evaluated using Equations 10 and 11.

$$\Delta G^0 = -RT\ In\ K \quad \text{(Eq. 10)}$$

$$In\ K = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad \text{(Eq. 11)}$$

K is given as q_e/c_e, T is the temperature and R is the gas constant.

RESULT AND DISCUSSION

Characterization of the adsorbent

The results of the proximate analysis of JAC and TAC (see Table 1) uncover that the adsorbents could be utilised for adsorption purpose due to their low ash content (n=3, 0 ≤ %E ≤ 0.88). The SEM micrographs of prepared JAC and TAC (Figure 2a & 2b) reveal that the surface of the prepared activated carbons possesses cavities, pores, and rough surface structure. The surface is also hollowed and fragmented [20,21]. The results of BET surface area, pore volume and pore size of JAC are 50.29 m^2/g, 0.0265 cm^3/g and 21.1 Å, respectively, while that of TAC were found to be 75.70 m^2/g, 0.0476 cm^3/g and 25.1 Å, respectively. This indicates that the surface of JAC and TAC are overwhelmed with mesopores [22].

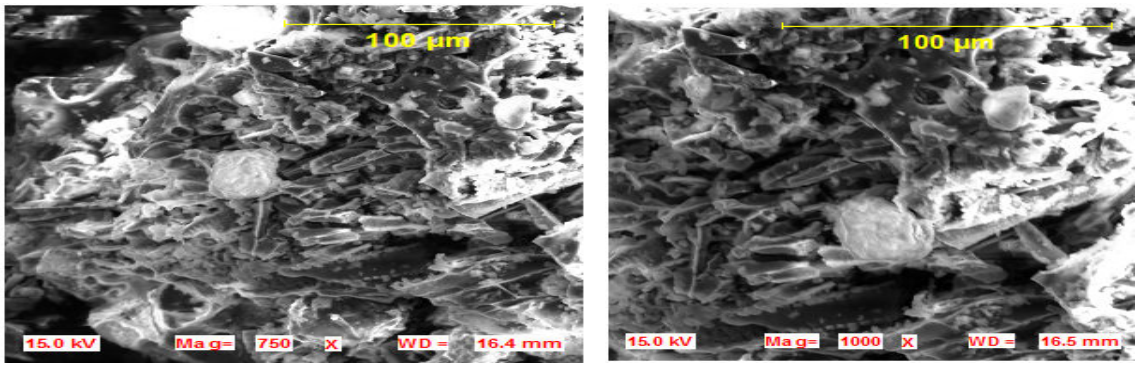

Figure 2(a): SEM image of JAC (left: mag=750; right: mag=1000).

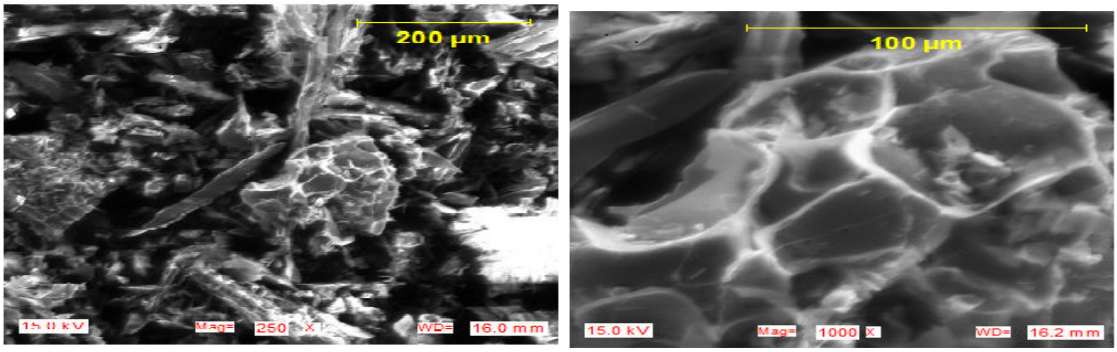

Figure 2(b): SEM image of TAC (left mag= 250; right: mag=1000).

Table 1: Proximate analysis of adsorbents and isothermal parameters for adsorption of MB onto JAC and TAC.

	Parameters	Values (JAC)	Values (TAC)
Proximate			
% Moisture Content		34	31.05
% Ash content		9.9	8.01
Bulk density (gcm^{-3})		1.33	2.00
Isotherms			
Langmuir	Q_{max} (mg/g)	44.05	23.26
	R_L	0.0182	0.172
	R^2	0.9916	0.9899
Freundlich	K_f (mg/g)	21.95	4.18
	n	2.62	2.24
	R^2	0.6935	0.9879
Dubinin-Radushkevich	β (mol^2/J^2)	$1x10^{-7}$	$2x10^{-6}$
	E ($Jmol^{-1}$)	$2.2x10^3$	500
	R^2	0.9627	0.8937

The FTIR spectra of JAC and TAC (Figure 3a & 3b) show a prominent signal at ~1570 cm^{-1} and 1370 cm^{-1} credited to graphitic structures that are expected to dominate activated carbons. The broad signal above 3400 cm^{-1} is attributed to OH stretching frequency in adsorbed water but also to surface OH groups not surprisingly for phenols, lactones, and carboxylic acid groups known to exist at the surface of activated carbon. The OH bending mode is usually observed around 1600 cm^{-1} and evidently adds to the broadening of the graphitic band. The signal at ~1200 cm^{-1} is allocated to the C-O stretching mode [23].

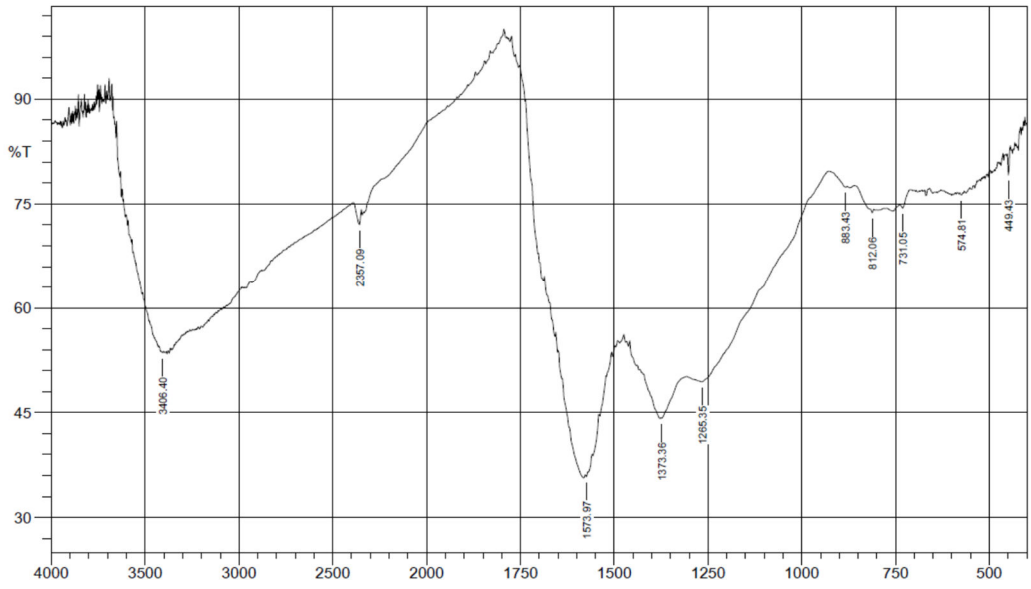

Figure 3(a): FTIR spectrum of JAC.

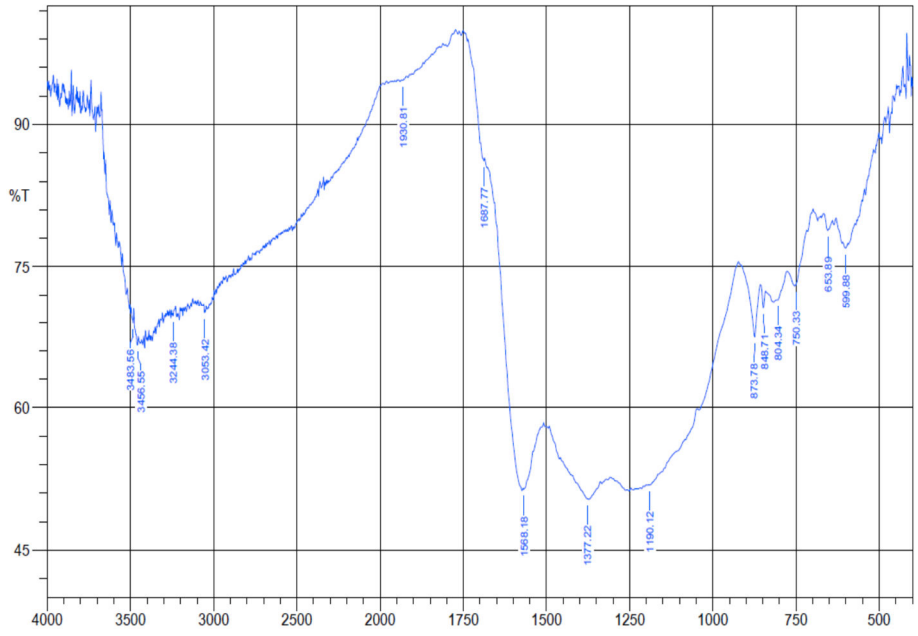

Figure 3(b): FTIR spectrum of TAC.

Effect of Initial Concentration

The quantity of MB adsorbed on JAC and TAC rises with increase in initial concentration of dye until saturation is reached (see Figure 4a). The adsorption limit of JAC is observed to be higher than that of TAC [24].

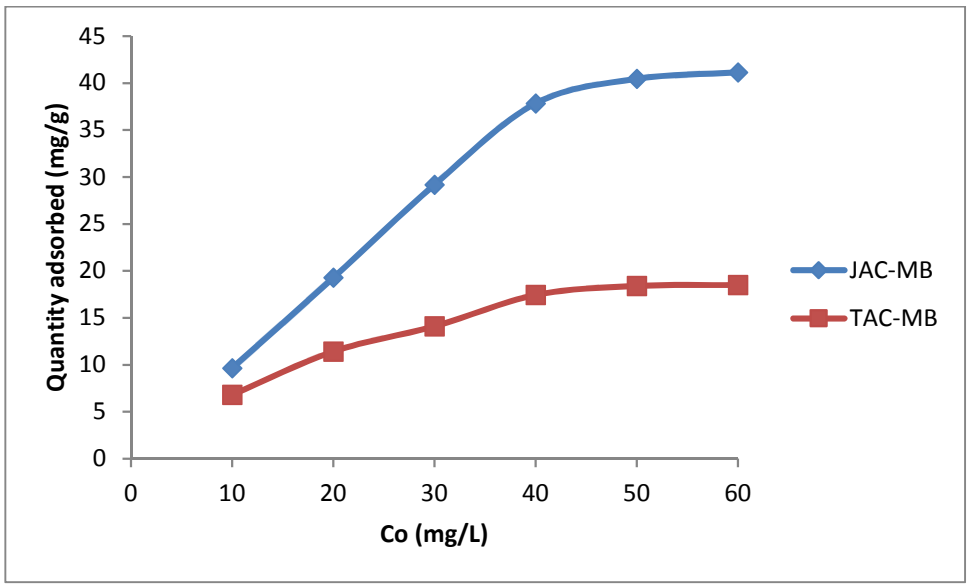

Figure 4(a): Effect of initial concentration (Conditions: pH=6.8, adsorbent dose=0.02 g, T=28±2°C, agitation time=120 min, agitation speed=200 rpm, n=3, 0 ≤ %E ≤ 0.90).

Effect of pH

The influence of pH on sorption of MB onto JAC and TAC was analysed (Figure 4b). The adsorption of MB under these conditions increases significantly as the pH increases from 2 to 4. However, at higher pH, the adsorption of dye molecules increases inconsequentially in JAC-MB system but diminishes due to the repulsive force between the adsorbent and adsorbate in TAC-MB system [25].

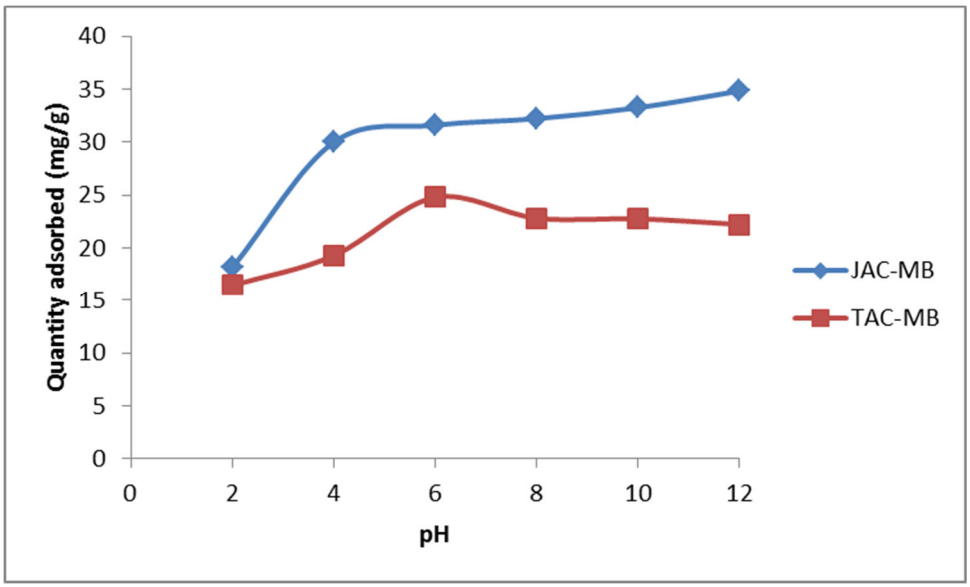

Figure 4(b): Effect of pH (Conditions: concentration= 40 mg/L, adsorbent dose=0.02 g, T=28±2°C, agitation time=120 min, agitation speed=200 rpm, n=3, 0 ≤ %E ≤ 0.92).

Effect of contact time

The progress of MB adsorption demonstrates a fast take-up at lower contact time (see Figure 4c). The quantity adsorbed increases thereafter until equilibrium was reached and no change in the quantity adsorbed could be seen beyond this point [26].

Effect of adsorbent dose

The influence of adsorbent dose on sorption experiment is presented in Figure 4d. The quantity of MB adsorbed diminishes with increase in dosage which shows that increasing the dose of adsorbent makes adsorbate molecules circulated over a larger amount of adsorbent [27].

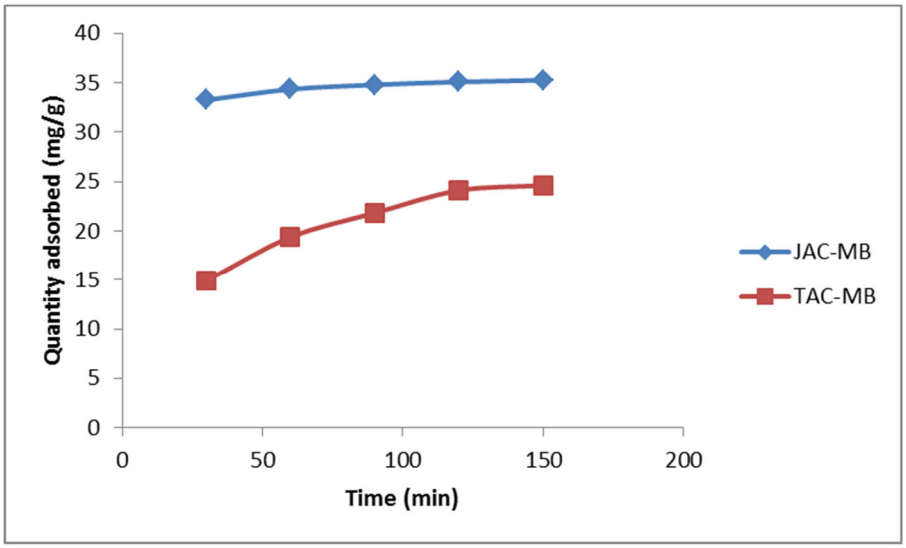

Figure 4(c): Effect of contact time (Conditions: concentration=40 mg/L, T=28±2°C, adsorbent dose=0.02 g, agitation time=120 min, agitation speed=200 rpm, n=3, 0 ≤ %E ≤ 0.90).

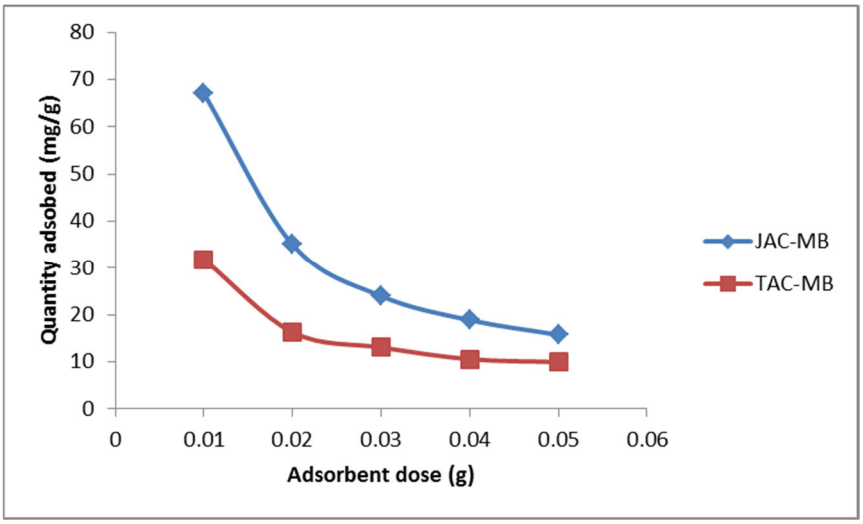

Figure 4d: Effect of adsorbent dose (Conditions: concentration=40 mg/L, pH=6, T=28±2°C, agitation time=120 min, agitation speed=200 rpm, n=3, 0 ≤ %E ≤ 0.24).

Effect of temperature

Figure 4e presents the impact of temperature on the sorption experiment. The quantity of MB adsorbed on JAC and TAC diminishes with a rise in temperature. This observation suggests that the force which binds the MB molecule to the activated carbon surface becomes weaker as the temperature of the medium is raised [28,29].

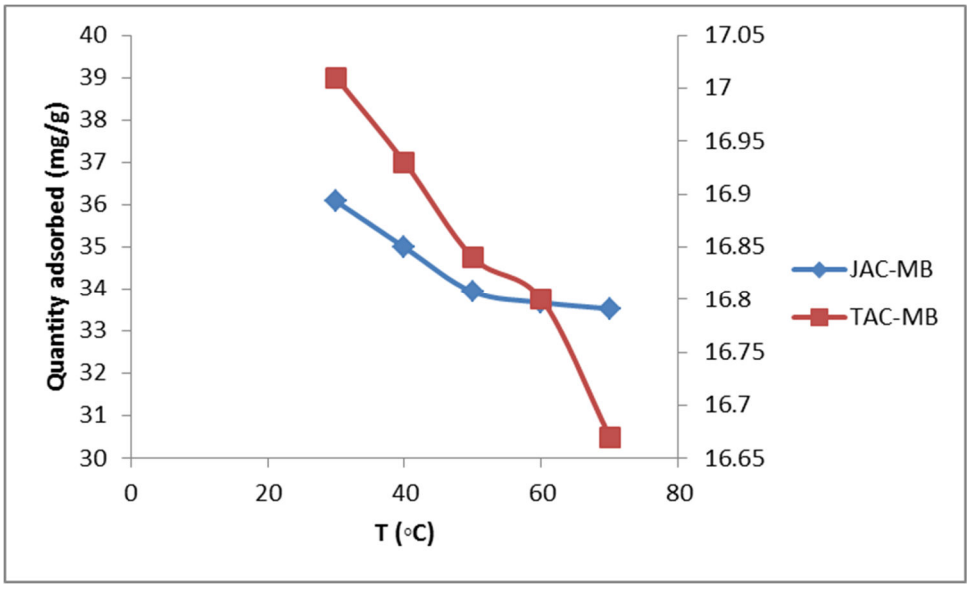

Figure 4e: Effect of Temperature (Conditions: concentration=40 mg/L, pH=6, adsorbent dose=0.02 g, agitation time=120 min, agitation speed=200 rpm, n=3, 0 ≤ %E ≤ 0.36).

Adsorption isotherms

The plots of Freundlich, Langmuir, and D-R isotherms model are shown in Figure 5. The R^2 values (see Table 1) of various isotherms reveal that Langmuir model fits the data well in both JAC-MB and TAC-MB systems [25]. Although D-R isotherm fits into JAC-MB system while Freundlich isotherm also describes adsorption data in TAC-MB system.

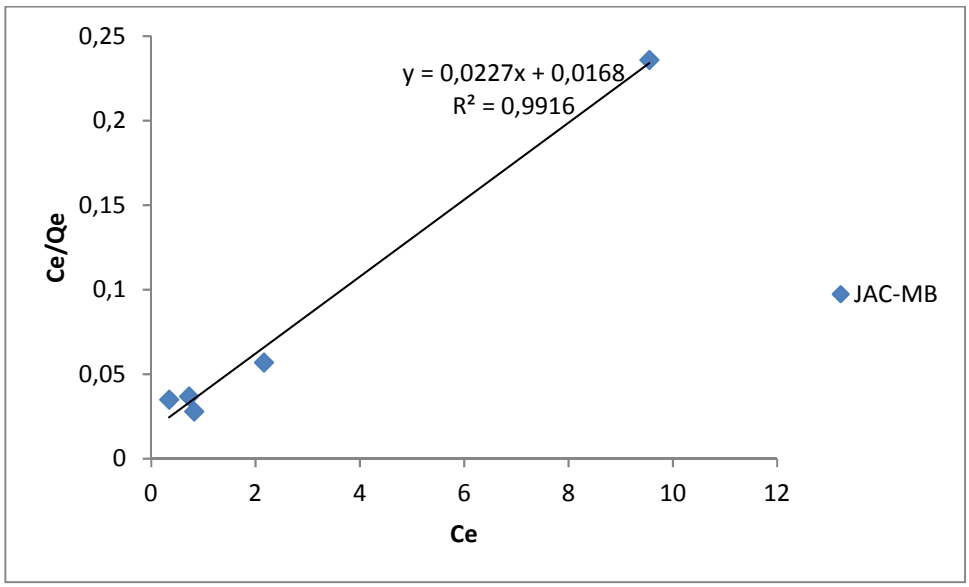

Figure 5(a): Langmuir plot of MB on JAC.

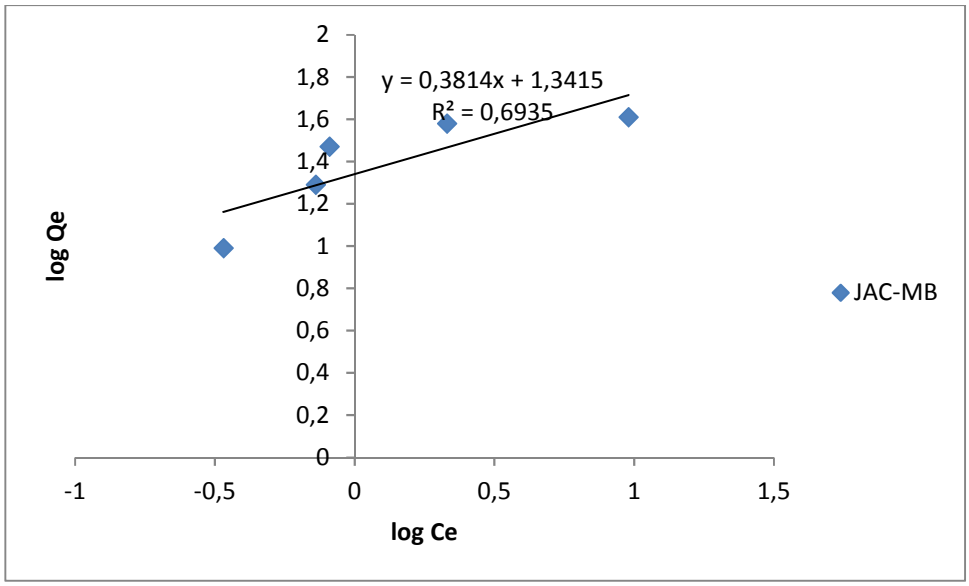

Figure 5(b): Freundlich plot of MB on JAC.

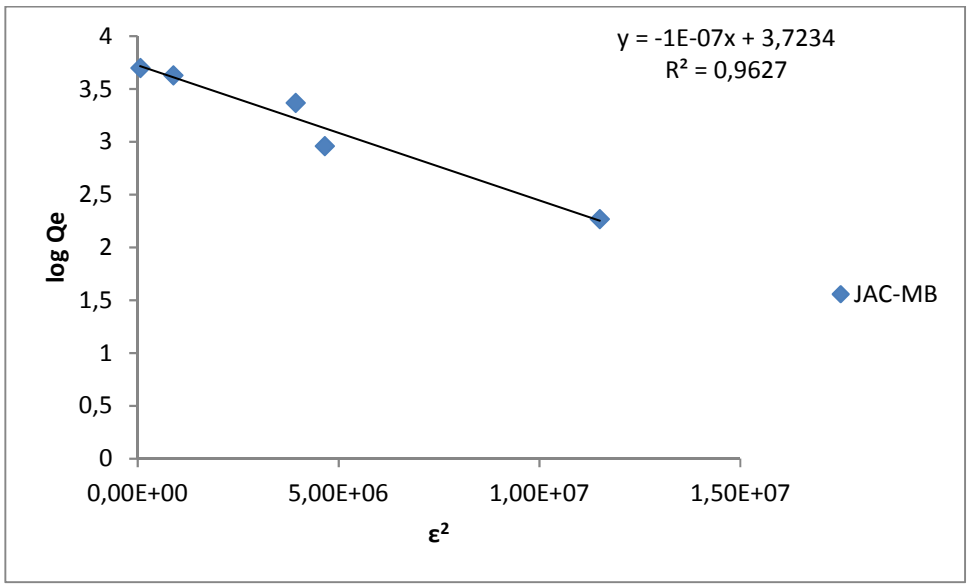

Figure 5(c): D-R isotherm plot of MB on JAC.

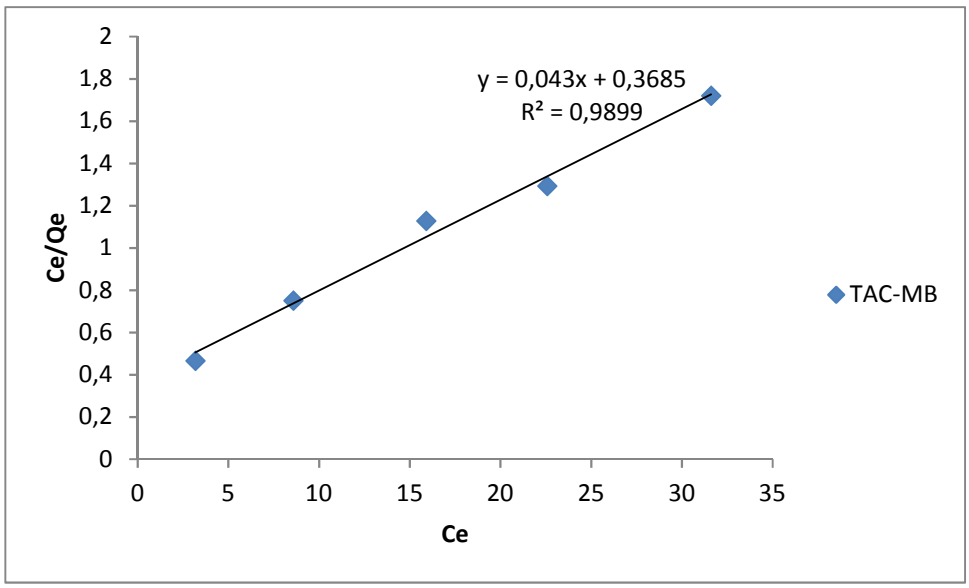

Figure 5(d): Langmuir plot of MB on TAC.

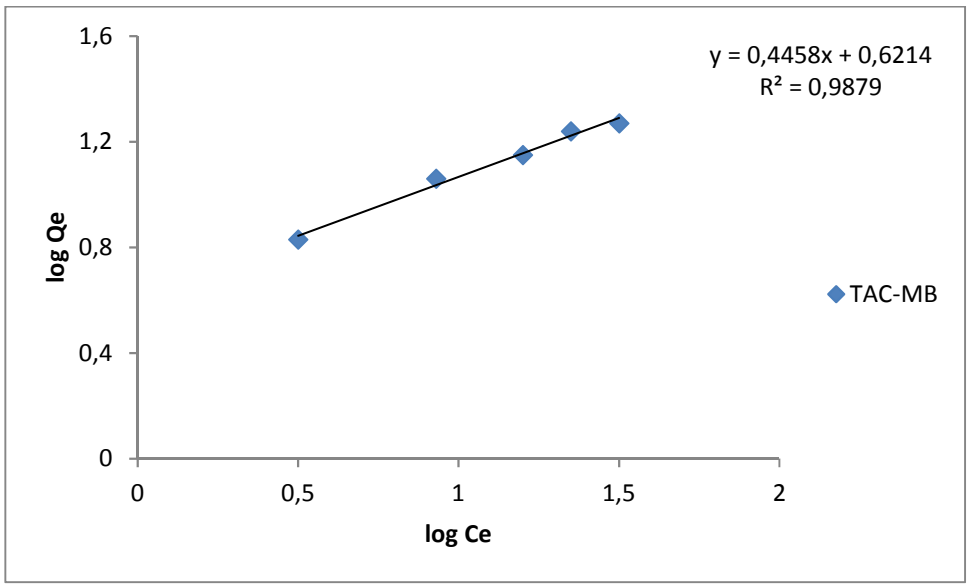

Figure 5(e): Freundlich plot of MB on TAC.

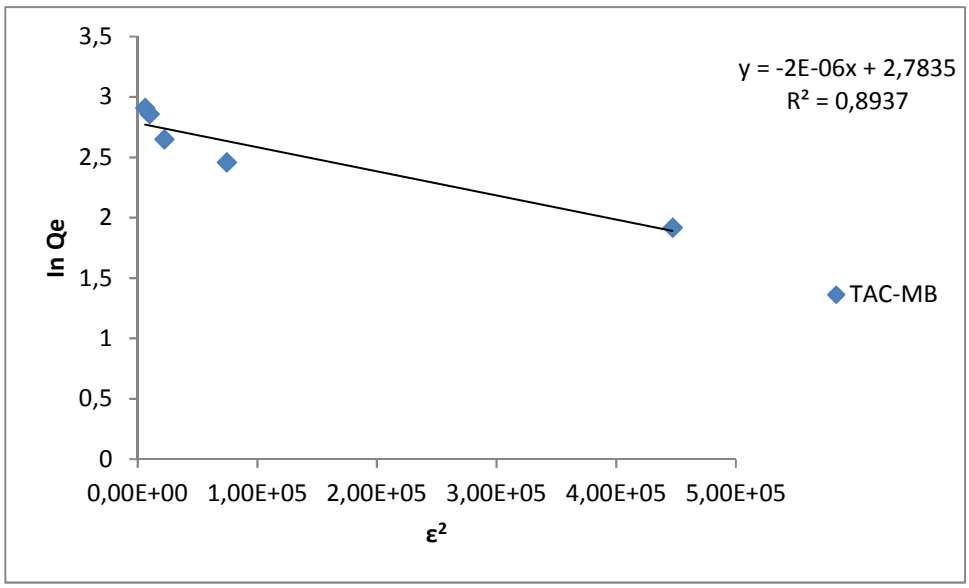

Figure 5(f): D-R isotherm plot of MB on TAC.

Adsorption kinetics

The pseudo first order and pseudo second order plots for MB-JAC and MB-TAC are presented in Figure 6. The correlation coefficients, R^2 observed is summarised in the Table 2 which suggests that pseudo second order fitted best for the sorption experimental data [30].

Table 2: Kinetics and Thermodynamic parameters for adsorption of MB onto JAC and TAC.

	Parameters	**Values (JAC)**	**Values (TAC)**
Kinetics Model			
Pseudo-first order	K_1 (min^{-1})	0.011	$8.75x10^{-3}$
	R^2	0.9485	0.9897
Pseudo-second order	K_2($gmg^{-1}min^{-1}$)	0.0112	$9.2x10^{-4}$
	R^2	1.000	0.9976
Thermodynamics			
	ΔH ($KJmol^{-1}$)	-12.68	-0.71
	ΔS ($Jmol^{-1}K^{-1}$)	-24.06	-4.85
	T (K)	ΔG (J/mol)	ΔG (J/mol)
	303	-5592	758
	313	-5048	804
	323	-4619	856
	333	-4623	894
	343	-4562	955

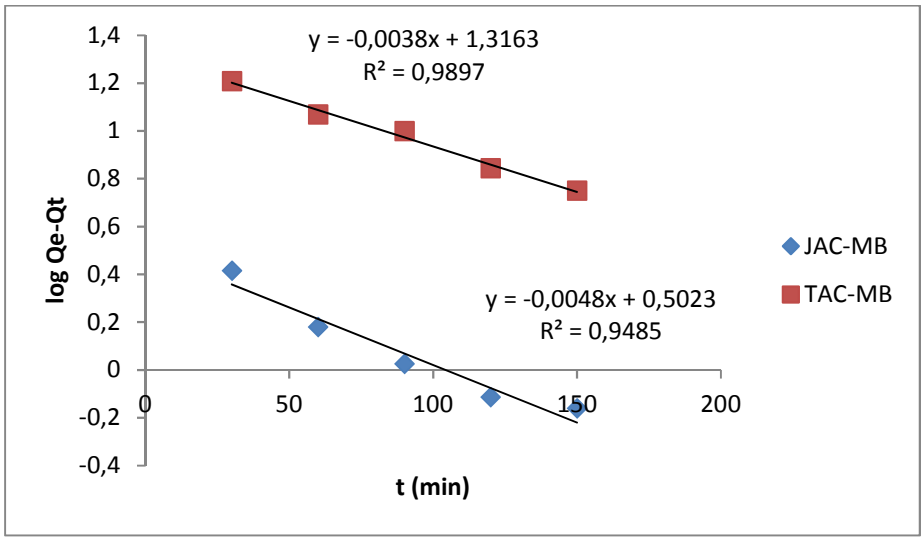

Figure 6a: Pseudo first order plots for the adsorption MB onto JAC and TAC.

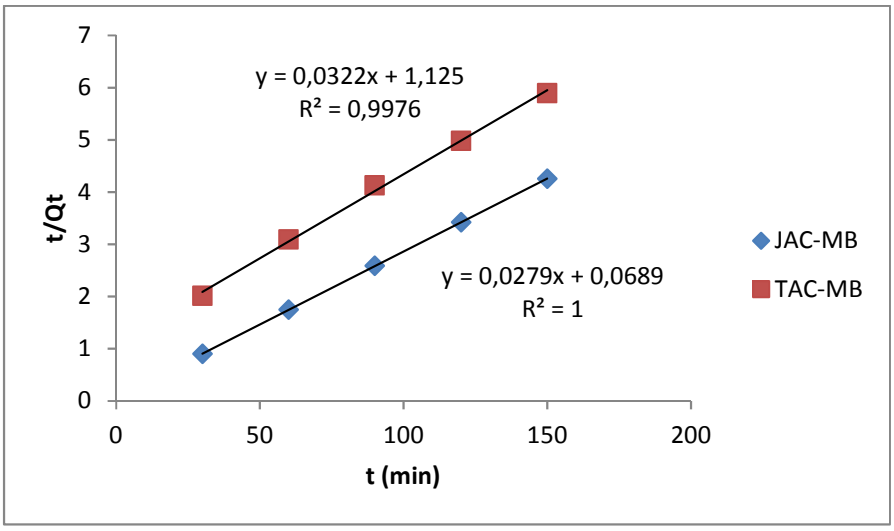

Figure 6b: Pseudo second order plots for the adsorption MB onto JAC and TAC.

Thermodynamics of adsorption

The thermodynamic parameters, free energy (ΔG^o), enthalpy (ΔH^o), and entropy (ΔS^o) assessed from the experimental data are presented using Van't Hoff's plot (see Figure 7). The values of ΔG^o, ΔH^o and ΔS^o acquired from the plots are introduced in Table 2. The values of ΔG^o obtained show that the adsorption process is more spontaneous at lower temperature. The negative value of ΔH^o was an indicative of the exothermic nature of the process. The negative value of ΔS^o proposes a diminished disorderliness at the solid-solute interface systems. This also proposes that the gain of entropy resulted from water molecules dislodged was not as much as that lost by the MB molecules [8].

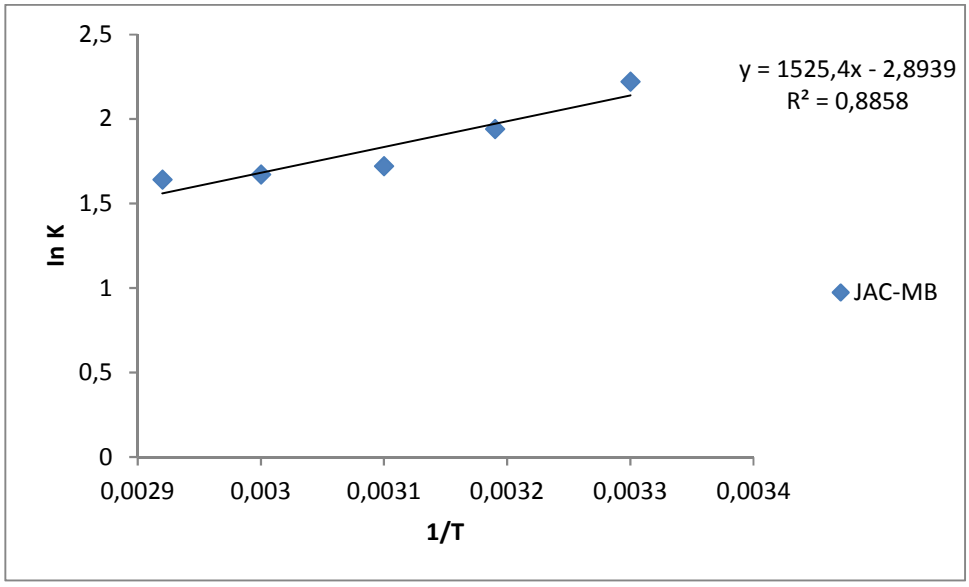

Figure 7a: Van't Hoff's plots for the adsorption of MB onto JAC.

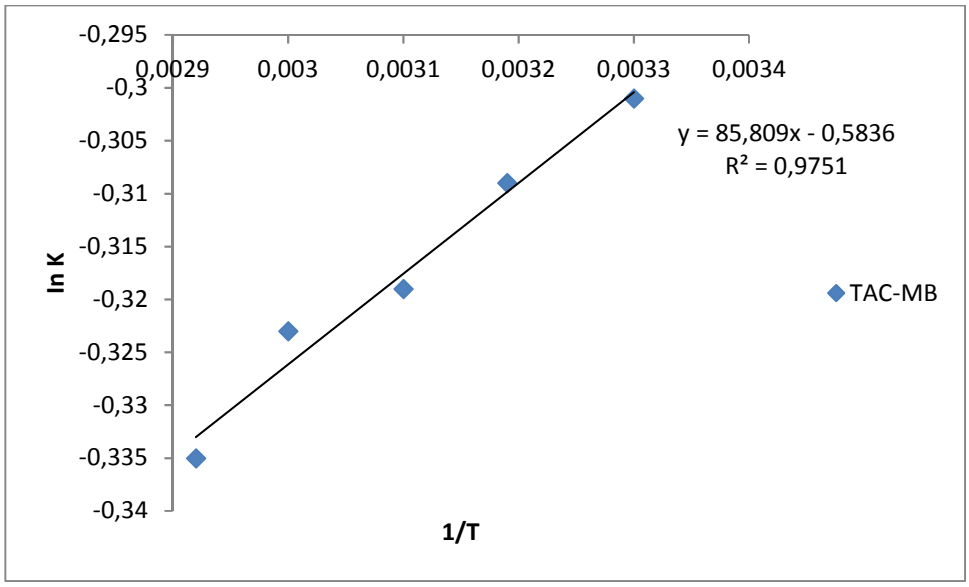

Figure 7b: Van't Hoff's plots for the adsorption of MB onto TAC.

CONCLUSIONS

The activated carbons (JAC and TAC) prepared were effective in the removal MB from aqueous medium. The sorption capacity of the prepared activated carbons was found to depend on the nature of the biomaterial used, experimental condition (initial concentration, pH, contact time, adsorbent dosage & temperature) adopted and the kinds of interaction that exist between the adsorbate and the adsorbent. The adsorption of MB on prepared JAC fitted into Langmuir and D-R isotherms; but Langmuir and Freundlich model fitted successfully for MB-TAC system. The kinetics studies best fitted into pseudo second order model. The process chemistry was found to be exothermic. The preferred disposal method of the spent adsorbent is by drying and burning or through landfill.

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Türkçe Öz ve Anahtar Kelimeler

Jatropha curcas ve *Terminalia catappa* Tohum Kabuklarından Hazırlanan Aktif Karbon üzerinde Metilen Mavisinin Tutulma Çalışması

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Öz: .Bu araştırma *Jatropha curcas* ve *Terminalia catappa* tohum kabuklarından üretilen aktif karbonun sudan metilen mavisi (MB) giderimi için etkinliğini bulmayı hedeflemiştir. *Jatropha* aktif karbonu (JAC) ve *Terminalia* aktif karbonu (TAC) Fourier Dönüşüm Kızılötesi Spektroskopisi (FTIR), taramalı electron mikroskopisi (SEM) ve Brunauer-Emmett-Teller (BET) yüzey alan analizi ile karakterize edilmiştir. Başlangıç derişimi, pH, temas süresi, adsorban dozu ve sıcaklığın adsorpsiyon deneyleri üzerindeki etkisi çalışılmıştır ve metilen mavisinin derişimi spektrofotometri ile takip edilmiştir. Bu adsorbanların adsorpsiyon kapasiteleri JAC ve TAC için sırasıyla 37,84 mg/g ve 17,44 mg/g olarak bulunmuştur. Deneysel veriler Langmuir, Freundlich ve Dubinin-Radushkevich izotermleri kullanılarak analiz edilmiştir. Veriler en iyi şekilde metilen mavisi-JAC sistemi ile metilen mavisi-TAC sistemine Langmuir izotermi üzerinden uyum göstermiştir. Kinetik çalışmaları yalancı ikinci derece kinetic modeline uyum sağlamıştır. Üreç kimyası eksotermiktir.

Anahtar kelimeler: Adsorpsiyon; aktif karbon; metilen mavisi; kinetik; termodinamik.

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