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Adsorption of Methylene Blue Dye: Thermodynamics and Kinetics Studies of *Vigna unguiculata* L. Seed Shell Activated Carbon

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This study reports the efficient adsorption of methylene blue dye from aqueous solutions using activated carbon derived from *Vigna unguiculata* L. seed shells (VUSSAC). The activated carbon was prepared through a sulphuric acid impregnation process, followed by carbonization at 500 ºC. The adsorption efficiency of VUSSAC was evaluated under varying experimental conditions, including adsorbent dosage, pH, contact time and temperature. Optimal dye removal was achieved at pH 10, an adsorbent dosage of 100 mg and a contact time of 120 min. The adsorption process followed the pseudo-second-order kinetic model indicating that the adsorption rate was controlled by chemical interactions. Adsorption isotherms were analyzed using Langmuir, Freundlich and Temkin models, with Langmuir isotherm best describing the adsorption, suggesting monolayer adsorption on a surface with uniform adsorption sites. Thermodynamic parameters, including changes in enthalpy (ΔH^o), entropy (ΔS^o) and Gibbs free energy (ΔG^o), indicated that the adsorption was endothermic and spontaneous, with increased temperature enhancing the removal efficiency. Characterization of the adsorbent before and after adsorption was conducted using FT-IR, SEM, BET, XRD and AFM techniques. These analyses revealed the presence of functional groups and changes in the surface morphology of VUSSAC, supporting the efficient dye uptake. The results demonstrate that VUSSAC is a cost-effective, eco-friendly adsorbent with promising potential for the removal of methylene blue dye from aqueous solutions.

Keywords: Adsorption, Isotherms, Kinetics, Thermodynamic study, Activated charcoal, Methylene blue dye.

INTRODUCTION

Recent estimates indicate that approximately 12% of textile dyes are lost annually during manufacturing and processing, with nearly 30-35% of these dyes ultimately discharged into the environment through untreated or partially treated wastewater [1,2]. The extensive use of pigments and colorants in textile processing generates large volumes of dye-contaminated effluents, which pose serious environmental and health concerns due to their toxic, carcinogenic, and non-biodegradable nature. Human exposure to these dyes may result in skin irritation, ocular damage, respiratory ailments and carcinogenic effects through inhalation, ingestion or dermal contact. Once released into aquatic systems, dyes obstruct light penetration, inhibit photosynthetic activity, disrupt biological processes, elevate aquatic toxicity and deteriorate water aesthetics [2,3]. Predicting the persistence and behaviour of hazardous dyes in textile effluents remains complex, thereby hindering effective pollution control at the source. Nevertheless, assessing effluent toxicity using key dyeing parameters, such as fabric weight, shade depth and dye fixation efficiency, offers a practical approach for reducing dye losses and mitigating environmental impact [4].

Most dye removal techniques are employed to mitigate the environmental damage caused by industrial effluents and include processes such as chemical oxidation, ozonation, photooxidation, coagulation, photocatalysis, adsorption onto organic or inorganic matrices, and subsequent microbial degradation [5]. Among these methods, adsorption has proven to be one of the most effective and reliable techniques for removing dyes from the municipal and industrial wastewater. However, the widespread industrial applications of adsorption are limited by the high cost of commercially available adsorbents. To address this challenge, the development of low-cost and sust-

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ainable adsorption materials is essential. Consequently, considerable attention has been directed toward the utilization of inexpensive and readily available waste-derived materials including coir pith [6,7], rice flour [8], *Parthenium hysterophorus* [9], pine sawdust [10], wood sawdust and rubber waste [11], de-oiled soy residues and bottom ash [12], palm kernel shells [13] and crow feathers [14]. Based on this literature survey, *Vigna unguiculata* L. seed shell derived activated carbon (VUSSAC) was selected as an economical and abundantly available adsorbent, demonstrating strong potential for the efficient removal of methylene blue dye from aqueous solutions.

EXPERIMENTAL

All chemicals and reagents used in the study were of analytical grade and procured from E-Merck and SD Fine Chemicals (India). *Vigna unguiculata* L. seed shells were employed as an adsorbent for the removal of methylene blue dye from aqueous solutions and collected from the local garden of Thanjavur city.

Preparation of activated carbon: *Vigna unguiculata* L. seed shells were washed thoroughly with hot distilled water to remove adhering impurities, cut into small pieces and air-dried. The dried material was then chemically activated by impregnation with concentrated sulfuric acid using a weight-to-acid volume ratio of 0.5:1. After impregnation, the material was repeatedly washed with distilled water until a neutral pH was attained. The acid-treated sample was subsequently dried and carbonized in a muffle furnace at 500 ºC. The resulting activated carbon was ground and sieved to obtain a uniform particle size suitable for adsorption studies.

Adsorption studies: A stock solution of methylene blue dye (1000 mg L^{-1}) was prepared using double-distilled water and served as the source for all experimental investigations. Working solutions of desired concentrations were obtained by appropriate dilution of the stock solution. All adsorption experiments were conducted using double-distilled water. Batch adsorption studies were performed by adding 100 mg of adsorbent to 100 mL of methylene blue solution of known concentration in iodine flasks. The mixtures were agitated in a temperature-controlled water bath to ensure uniform contact between the adsorbent and the dye solution. At predetermined time intervals, the solutions were filtered and the residual methylene blue concentration was determined using a UV-visible spectrophotometer at a wavelength of 668 nm. Each experiment was repeated twice to ensure reproducibility of the results. The adsorption capacity was calculated from the following eqn.:

$$\text{Adsorption capacity } (q_e) = \frac{C_o - C_e}{W} V \tag{1}$$

where V represents the volume (mL) in methylene blue dye solution, W is the weight (g) of VUSSAAC (g), C_o is the starting point (mg/L) amount in the methylene blue dye solution and C_e represents the final (mg/L) concentration of the solution.

Adsorption isotherms: The adsorption of methylene blue dye molecules onto VUSSAC was analyzed using the Langmuir, Freundlich and Temkin isotherm models to elucidate the interaction between the adsorbate and adsorbent. The Langmuir model describes monolayer adsorption on a homogeneous surface, with the linearized form used to calculate the monolayer adsorption capacity (Q_o) and Langmuir constant (b_L), which represents the adsorption energy [15,16].

$$\frac{C_e}{q_e} = \frac{1}{Q_o.b_L} + \frac{C_e}{Q_o} \tag{2}$$

Based on the slope and intercept of the linear plot of C_e/q_e, the Langmuir isotherm constants Q_o and b_L were calculated. The linearized form of the Freundlich adsorption isotherm is expressed as follows [16]:

$$\log q_e = \log k_f + \frac{1}{n} \log C_e \tag{3}$$

The Langmuir constants Q_o and b_L represent the maximum monolayer adsorption capacity (mg g^{-1}) and the adsorption energy (L mg^{-1}), respectively. In contrast, the Freundlich isotherm is characterized by empirical constants that describe adsorption intensity and surface heterogeneity. The Freundlich constants indicate the adsorption capacity and strength. Here, C_e denotes the equilibrium concentration of methylene blue dye remaining in solution.

Temkin isotherm: This model assumes that owing to adsorbent-adsorbate interactions, the heat of adsorption of molecules in a given layer decreases linearly with increasing surface coverage. Unlike other isotherm models that assume a logarithmic dependence, the Temkin isotherm considers a linear variation of adsorption energy with coverage. The linear form of the Temkin isotherm is expressed as follows [17]:

$$q_e = \frac{RT}{b_r} \ln a_T + \frac{RT}{b_r} \tag{4}$$

where R denotes the universal gas constant (J K^{-1} mol^{-1}); T represents the absolute temperature (K); b_T is the Temkin constant related to the heat of adsorption (kJ mol^{-1}) and a_T is the Temkin equilibrium binding constant (L mg^{-1}).

RESULTS AND DISCUSSION

Characterization of VUSSAC as adsorbent before and after adsorption

FT-IR studies: FT-IR spectroscopy was employed to identify the surface functional groups of *V. unguiculata* L. seed shell derived activated carbon (VUSSAC) and to elucidate the adsorption mechanism of methylene blue dye molecules. The FT-IR spectrum of pristine VUSSAC (Fig. 1a) showed a broad band at 3783 cm^{-1} corresponding to O–H stretching of hydroxyl groups. Peaks at 3409 cm^{-1} and 2922 cm^{-1} were assigned to the N–H stretching of amines and C–H stretching of alkanes, respectively. The bands at 1704 cm^{-1} and 1591 cm^{-1} indicated C=O stretching and N–H bending vibrations, while the peaks at 1364, 1229, 1160, and 1116 cm^{-1} corresponded to C–H bending and C–N stretching of aliphatic amines (Fig. 1b). The presence of these oxygen- and nitrogen-containing groups confirms their active participation in dye adsorption.

SEM studies: SEM technique was used to study the surface morphology of VUSSAC before and after methylene blue adsorption. The pristine adsorbent exhibited a highly

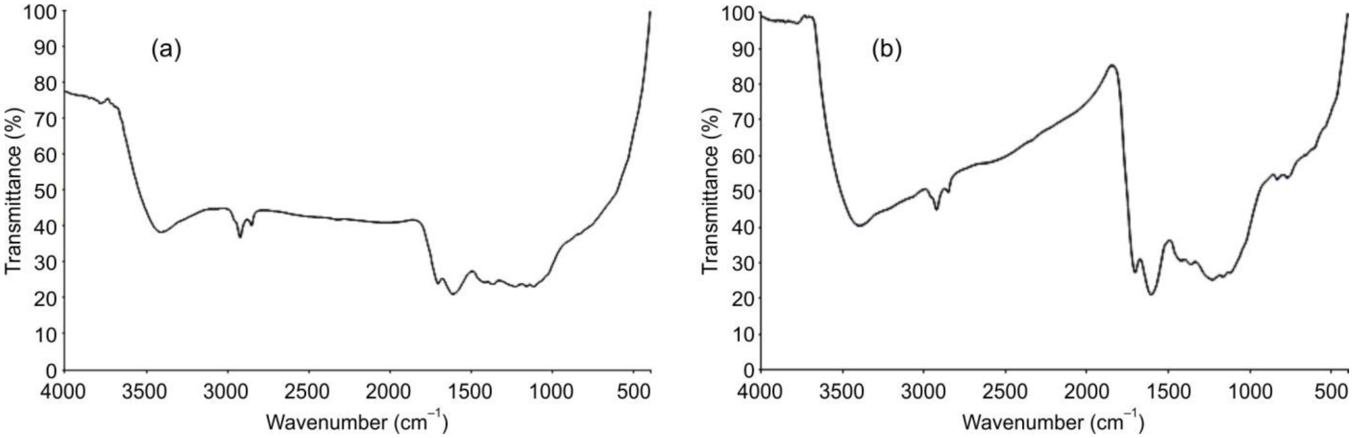

Fig. 1. FT-IR spectra of VUSSAC adsorbent before (a) and after (b) methylene blue dye adsorption

porous surface with abundant cavities, enhancing the surface area and accessibility of adsorption sites (Fig. 2a). After adsorption, the dye molecules were observed to occupy the pores and surface cavities, causing noticeable morphological changes (Fig. 2b). These images provide clear evidence of effective dye attachment and adsorption onto VUSSAC [18,19].

AFM studies: AFM technique was employed to examine nanoscale surface topography and roughness. The surface roughness (S_a) of pristine VUSSAC was found to be 10.273 nm. Post-adsorption, a significant reduction in surface roughness was observed (Sa = 3.688 nm, Fig. 3), indicating uniform coverage of the surface by methylene blue dye molecules. These findings validate the SEM results, confirming that adsorption occurs both on the surface and within the pores.

Brunauer-Emmett-Teller (BET) studies: BET analysis was conducted to quantify the specific surface area and porosity of VUSSAC. The nitrogen adsorption-desorption isotherms

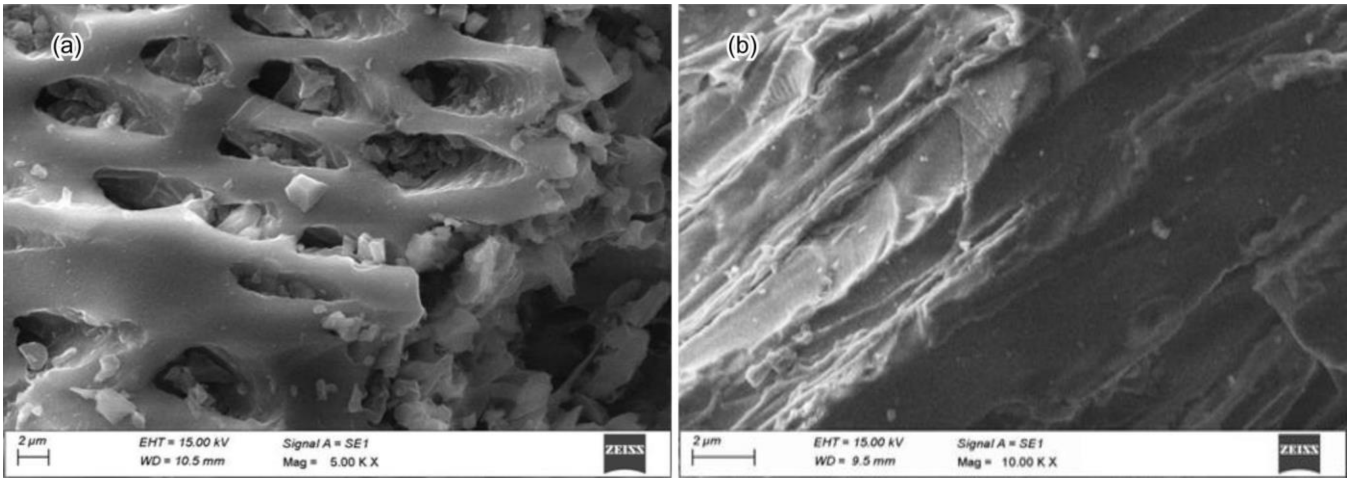

Fig. 2. SEM images of VUSSAC before (a) and after (b) methylene blue dye adsorption

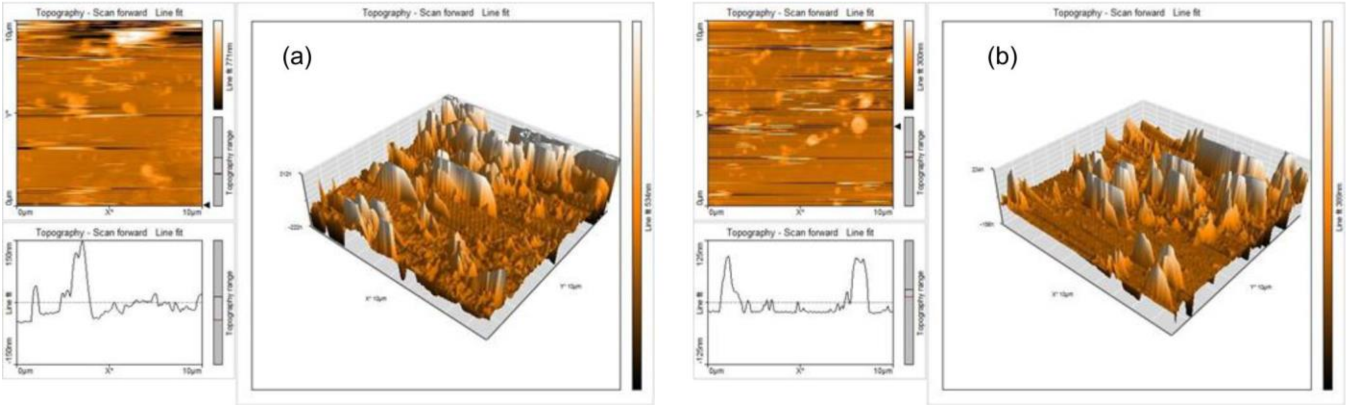

Fig. 3. AFM images of VUSSAC before (a) and after (b) methylene blue dye adsorption

(Fig. 4) displayed a Type-IV isotherm, characteristic of mesoporous materials. Such mesoporosity is favourable for dye adsorption, facilitating enhanced mass transfer and accessibility of active sites. The detailed BET surface area and pore parameters are summarized in Table-1.

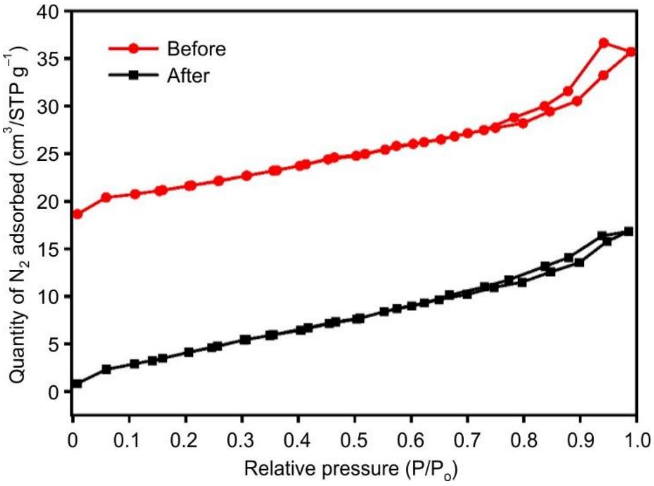

Fig. 4. Nitrogen adsorption-desorption isotherms of VUSSAC adsorbent for methylene blue dye adsorption

TABLE-1
TEXTURAL PROPERTIES DATA OF VUSSAC ADSORBENT OBTAINED FROM BET SURFACE AREA ANALYSIS

Sample	Surface area (m^2/g)	Pore volume (cc/g)	Pore diameter (nm)
VUSSAC	22.877	0.033	2.494

Powder X-ray diffraction (P-XRD) studies: P-XRD studies were performed to investigate the structural and crystallinity changes of VUSSAC before and after dye adsorption. The pristine VUSSAC displayed a broad peak at 2θ ≈ 24.97º, confirming its amorphous nature and disordered carbon structure formed during carbonization at 500 ºC. Minor peaks suggested the presence of functional groups contributing to adsorption (Fig. 5a). After methylene blue adsorption (Fig. 5b), peaks at 2θ ≈ 23.12º, 26.27º and 42.93º were observed, confirming dye attachment and partial coverage of adsorption sites [20]. The overall reduction in peak intensity further supports the successful adsorption of methylene blue dye onto VUSSAC.

Adsorption dynamics

Effects of adsorbent quantity, pH, time and contact: Batch adsorption experiments were conducted using adsorbent dosages ranging from 50 to 300 mg. A methylene blue dye solution (100 mL of 10 mg L^{-1}) was agitated with varying amounts of adsorbent to determine the optimal conditions. The results indicated that a dosage of 100 mg, a contact time of 100 min, and a solution pH of 10 achieved maximum dye removal [21]. The effect of initial dye concentration and temperature on adsorption was also investigated. Equilibrium time was determined independently of the initial concentration (C_o). The adsorption capacity (q_e) increased with higher initial methylene blue dye concentrations due to an enhanced driving force for mass transfer, effectively overcoming resistance in the solid phase [22]. Moreover, increasing the temperature from 30 to 50 ºC improved dye removal, confirming the endothermic nature of the adsorption process (Table-2).

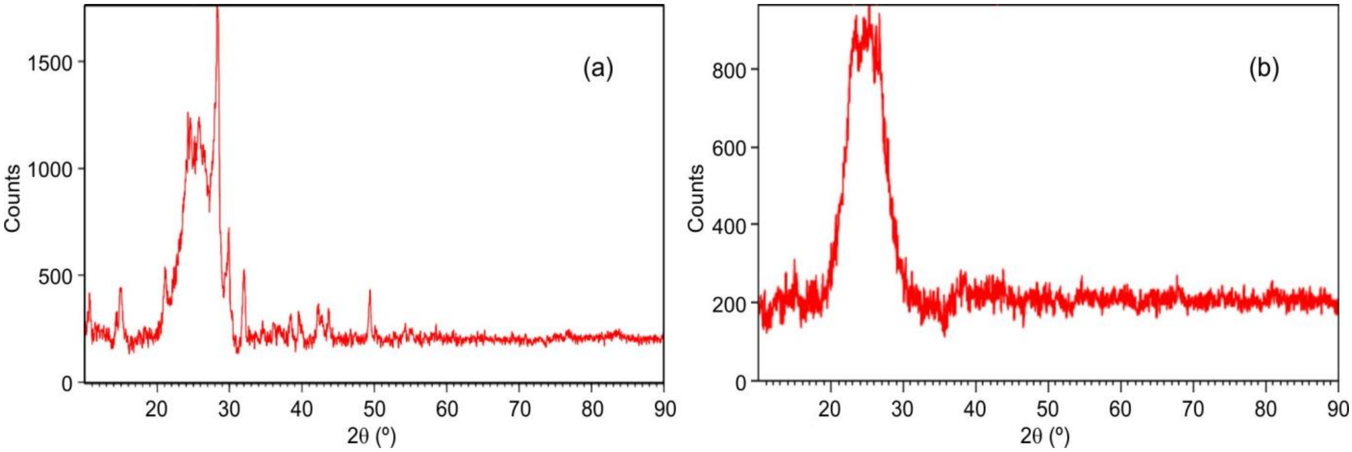

Fig. 5. Powder-XRD spectra of VUSSAC adsorbent before (a) and after (b) methylene blue dye adsorption

TABLE-2
ADSORPTION PERFORMANCE OF VUSSAC FOR METHYLENE BLUE DYE AT DIFFERENT INITIAL CONCENTRATIONS AND TEMPERATURES

Primary conc. of MBD (C_o) (mg/L)	Equilibrium conc. of MBD (C_e) (mg/L)			Amount of MBD adsorbed at equilibrium (q_e) (mg/g)			MBD removal (%) by VUSSAC		
	30 ºC	40 ºC	50 ºC	30 ºC	40 ºC	50 ºC	30 ºC	40 ºC	50 ºC
10	2.455	1.773	1.227	7.546	8.227	8.773	75.46	82.27	87.73
20	7.636	5.864	5.409	12.364	14.136	14.591	61.82	70.68	72.95
30	15.455	14.546	13.864	14.546	15.455	16.136	48.49	51.52	53.79
40	23.591	20.955	19.136	16.409	19.046	20.864	41.02	47.61	52.16
50	31.455	29.273	26.682	18.546	20.727	23.318	37.09	41.45	46.64

Adsorption isotherms studies: The equilibrium adsorption behaviour of methylene blue dye onto VUSSAC adsorbent was evaluated using Langmuir, Freundlich and Temkin isotherm models at three different temperatures (30, 40 and 50 ºC). The corresponding isotherm constants and correlation coefficients are summarized in Table-3, while the linearized plots are shown in Fig. 6.

Langmuir isotherm: The Langmuir isotherm model exhibited an excellent fit to the experimental data, with high correlation coefficients (r^2 = 0.979-0.996), indicating monolayer adsorption of methylene blue dye on a homogeneous VUSSAC surface. The linear relationship observed in Fig. 6a between C_e/q_e and C_e further confirms the applicability of this model. The maximum monolayer adsorption capacity (Q_o) increased from 23.009 mg g^{-1} at 30 ºC to 27.473 mg g^{-1} at 50 ºC, demonstrating enhanced adsorption at elevated temperatures. This increase suggests an endothermic adsorption process, likely due to increased mobility of methylene blue dye molecules and improved accessibility of active adsorption sites. Moreover, the Langmuir affinity constant (b_L) showed an increasing trend with temperature, reflecting stronger adsorbate-adsorbent interactions at higher thermal conditions.

Freundlich isotherm: The Freundlich isotherm model also adequately described the adsorption process, as indicated by high correlation coefficients (r^2 = 0.989-0.996). The linear plots of log q_e *vs.* log C_e (Fig. 6b) suggest multilayer adsorption on a heterogeneous surface. The Freundlich adsorption capacity constant (k_f) increased with temperature, confirming enhanced adsorption efficiency at higher temperatures. The heterogeneity factor (n) ranged from 2.789 to 3.339, with values greater than unity indicating favourable adsorption conditions. The increase in 'n' with temperature implies improved adsorption intensity and stronger interaction between methylene blue dye molecules and the VUSSAC surface.

Temkin isotherm: Temkin isotherm model provided reasonable agreement with the experimental data, with correlation coefficients ranging from 0.958 to 0.997. This suggests that adsorbate-adsorbent interactions play a role in the adsorption mechanism and that the heat of adsorption decreases linearly with surface coverage. The Temkin constant related to adsorption heat (b_T) was found to be in the range of 9.774-10.586 kJ mol^{-1}, indicating that the adsorption process is predominantly physical in nature. Furthermore, the increase in the Temkin binding constant (a_T) with temperature signifies enhanced binding affinity and surface activity of VUSSAC adsorbent at elevated temperatures.

Thus, comparative evaluation of the three isotherm models indicates that the Langmuir model best represents the adsorption of methylene blue dye onto VUSSAC, suggesting monolayer coverage on a relatively uniform surface. The Freundlich and Temkin models further support the presence of surface heterogeneity and adsorbate-adsorbent interactions. The observed increase in adsorption capacity and affinity constants with temperature confirms that the adsorption process is favourable and endothermic.

Kinetic studies: Various kinetic models, including the pseudo-first-order and pseudo-second-order models originally proposed by Lagergren, were applied to analyze the adsorption data obtained from batch experiments. The integrated linear form of the pseudo-first-order kinetic model is expressed as:

$$\log(q_e - q_1) = \log q_e - \frac{k_1}{2.303} t \qquad (7)$$

where q_e and q_t (mg g^{-1}) represent the amounts of MBD adsorbed onto VUSSAC at equilibrium and at time t, respectively, and K_1 (min^{-1}) is the pseudo-first-order rate constant. If the adsorption process follows pseudo-first-order kinetics,

TABLE-3
ISOTHERM PARAMETERS FOR THE ADSORPTION OF METHYLENE BLUE DYE ONTO VUSSAC AT DIFFERENT TEMPERATURES OBTAINED FROM LANGMUIR, FREUNDLICH, AND TEMKIN MODELS

Temp. (ºC)	Langmuir isotherm			Freundlich isotherm			Tempkin isotherm		
	r^2	Q_0 (mg/g)	b_L (L/mg)	r^2	k_f	n	r^2	b_T (kJ mol^{-1})	a_T (L/mg)
30	0.994	23.009	0.181	0.996	5.991	2.789	0.994	10.586	3.852
40	0.996	24.331	0.249	0.993	8.032	3.333	0.997	9.774	6.988
50	0.979	27.473	0.257	0.989	9.253	3.339	0.958	10.382	8.672

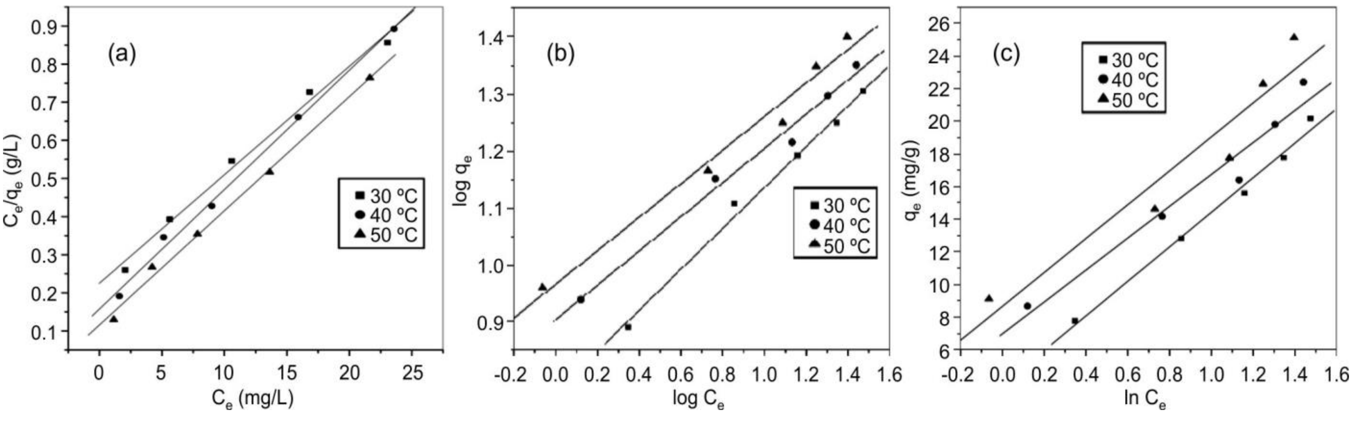

Fig. 6. Plots of Langmuir (a), Freundlich (b) and Temkin (c) isotherms for the adsorption towards methylene blue dye on VUSSAC

a linear relationship between log ($q_e - q_t$) and t is expected. The values of k_1 and q_e can be obtained from the slope and intercept of the corresponding linear plot. When the pseudo-first-order model fails to adequately describe the adsorption behaviour, the pseudo-second-order kinetic model is considered.

The linearized form of the pseudo-second-order kinetic model is given as:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{8}$$

The pseudo-second-order rate constant (K_2, g mg^{-1} min^{-1}), describes the kinetics of the adsorption process. The values of the equilibrium adsorption capacity (q_e) and the rate constant (k_2) were determined from the slope and intercept of the linear plot of t/q_t *versus* time (t). A good linear relationship in this plot indicates that the adsorption process follows pseudo-second-order kinetics.

The kinetic plots (Fig. 7) and the parameters obtained from the pseudo-first-order and pseudo-second-order models are shown in Table-4. Comparison between the experimental adsorption capacities and the calculated values shows that the pseudo-second-order model provides a superior description of the adsorption process compared to the pseudo-first-order model. This is supported by the high correlation coefficient (r^2 = 0.999) and the relatively low percentage relative error observed for MBD adsorption. Therefore, the adsorption kinetics of MBD onto VUSSAC are best represented by the pseudo-second-order model, which is consistent with earlier reported studies [23,24].

Thermodynamic studies: The changes in entropy (ΔS°), Gibbs free energy (ΔG°) and enthalpy (ΔH°) were determined using the thermodynamic equilibrium constant (K_o). According to the method reported in the literature [25], the consistency of these values confirms that the adsorption process attains equilibrium. Eqns. 5 and 6 are derived from the fundamental thermodynamic relationships and do not alter the basic principles of thermodynamics.

$$\ln K_o = \frac{\Delta S^o}{R} - \frac{\Delta H^o}{RT} \tag{5}$$

$$\Delta G = -RT \ln K_o \tag{6}$$

The values of ΔH° and ΔS° were determined from the slope and intercept of the van't Hoff plot of ln K_o *versus* 1/T, while the corresponding thermodynamic parameters are summarized in Table-5. The increase in the equilibrium constant (K_o) with temperature confirms the endothermic nature of the adsorption process. Negative ΔG° values obtained at all studied temperatures indicate the spontaneous nature of methylene blue dye adsorption, with higher initial dye concentrations leading to more favorable free energy changes. The enthalpy values, ranging between 15 and 45 kJ mol^{-1}, suggest that the adsorption process is predominantly physical. Furthermore, the positive ΔS° values (50-159 J K^{-1} mol^{-1}) reflect increased randomness at the solid-solution interface during adsorption,

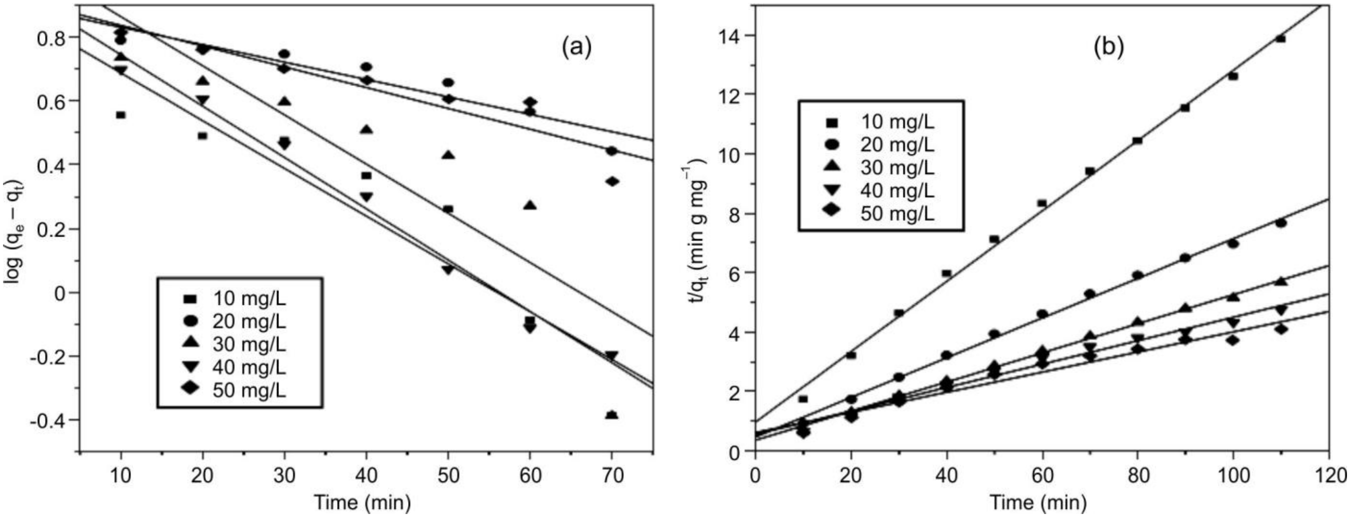

Fig. 7. Pseudo-first order (a) and pseudo-second order (b) kinetic plots for the adsorption of methylene blue dye on VUSSAC

TABLE-4
COMPARISON OF PSEUDO-FIRST-ORDER AND PSEUDO-SECOND-ORDER KINETIC MODELS FOR METHYLENE BLUE DYE ADSORPTION ON VUSSAC ADSORBENT

C_o (mg/L)	q_e (exp) (mg/g)	Pseudo-first order				Pseudo-second order			
		q_e (cal) (mg/g)	k_1 (min^{-1})	r^2	P	q_e (cal) (mg/g)	k_2 (g mg^{-1} min^{-1})	r^2	P
10	7.727	6.904	0.035	0.926	10.65	9.585	0.004	0.976	19.41
20	12.818	7.707	0.013	0.947	39.87	15.291	0.002	0.919	16.17
30	15.591	10.479	0.036	0.876	32.79	17.513	0.004	0.991	10.98
40	17.773	8.076	0.037	0.993	54.56	19.121	0.007	0.999	7.05
50	20.182	8.013	0.015	0.928	60.29	21.786	0.004	0.982	7.37

TABLE-5
THERMODYNAMIC PARAMETERS FOR THE ADSORPTION OF METHYLENE BLUE DYE ONTO VUSSAC AT DIFFERENT INITIAL CONCENTRATIONS AND TEMPERATURES

C_o (mg/L)	K_o			ΔG^o (kJ mol^{-1})			ΔH^o (kJ mol^{-1})	ΔS^o (JK^{-1} mol^{-1})
	30 °C	40 °C	50 °C	30 °C	40 °C	50 °C		
10	3.490	6.586	10.579	-3.148	-4.905	-6.335	45.170	159.659
20	1.785	2.437	2.729	-1.459	-2.319	-2.696	17.351	64.997
30	1.082	1.207	1.454	-0.199	-0.490	-1.004	11.972	62.341
40	0.800	0.982	1.262	-0.563	-0.047	-0.625	18.549	59.281

indicating significant interaction between ΔS^o molecules and the VUSSAC surface [26].

Conclusion

In conclusion, *Vigna unguiculata* L. seed shell activated carbon (VUSSAC) was found to be an effective, low-cost, and eco-friendly adsorbent for the removal of methylene blue dye solution from aqueous solution. The optimum adsorption occurred at pH 10.0 with a contact time of 120 min and an adsorbent dosage of 100 mg. Equilibrium data were well described by adsorption isotherm models, indicating favourable adsorption behaviour. FT-IR and SEM analyses confirmed the involvement of surface functional groups and porous morphology in the adsorption mechanism. Kinetic studies showed that the pseudo-second-order model provided a better fit than the pseudo-first-order model. Moreover, the increase in adsorption capacity with temperature (30-50 °C) confirmed the endothermic nature of the process. These results demonstrate the potential applicability of VUSSAC as a sustainable adsorbent for removal of dye from wastewater.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

DECLARATION OF AI-ASSISTED TECHNOLOGIES

During the preparation of this manuscript, the authors used an AI-assisted tool(s) to improve the language. The authors reviewed and edited the content and take full responsibility for the published work.

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