

Jackfruit (*Artocarpus heterophyllus*) leaf powder: An effective adsorbent for removal of methylene blue from aqueous solutions

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Batch sorption experiments were carried out using jackfruit leaf powder (JLP), for the removal of methylene blue (MB) from aqueous solutions. Effects of process parameters pH , adsorbent mass, concentration and contact time were studied. The amount of MB adsorbed per unit weight of the adsorbent increased with the increase of pH , concentration and contact time. The pH at the point of zero charge (pH_{pzc}) of the adsorbent was found to be 3.9. Adsorption of MB was found highly pH dependent. The FTIR of the adsorbent was done to find the potential adsorption sites for interaction with the cationic MB dye. Equilibrium data were fitted to Langmuir and Freundlich isotherms. The equilibrium data were best represented by both the isotherms. Maximum dye uptake was found to be 326.32 mg/g, indicating that JLP can be used as an excellent low-cost adsorbent for removal of MB dye. From experimental data it was found that adsorption of MB onto JLP followed pseudo second order kinetics. The desorption studies showed that most of the MB can be recovered by decreasing the pH of the solution. The experimental result inferred that electrostatic attraction between the surface and the dye is one of the major adsorption mechanisms for binding MB to JLP surface.

Keywords: Jackfruit leaf powder, Methylene blue, Adsorption, Isotherms, Kinetics

Many industries, such as cosmetics, leather, paper, printing, textile finishing etc. use dyes in order to colour their products and pour a lot of coloured wastewater into the environment. Dyes even in low concentrations affect the aquatic life and food web. The discharge of coloured wastes into streams not only affects the aesthetic nature but also interferes with transmission of sunlight into streams and therefore reduces photosynthetic activity. The removal of dyes from wastewater is of great concern, since some dyes and their degradation products may be carcinogens and toxic and, consequently, their treatment cannot depend on biodegradation alone¹.

Methylene blue (MB), which is the most common among all other dyes of its category, is generally used for dyeing cotton and silk. Though methylene blue is not strongly hazardous, it can cause some harmful effects. Acute exposure to methylene blue can cause increased heart rate, vomiting, shock, Heinz body formation, cyanosis, jaundice, quadriplegia, and tissue necrosis in humans². Therefore, the removal of such dye from effluents becomes environmentally important.

There are several reported methods for the removal of dyes from effluents. The technologies can be divided into three categories: biological, chemical and

physical. Biological treatment requires large land area and is constrained by sensitivity toward diurnal variation as well as toxicity of some chemicals, and less flexibility in design and operation³. Although many organic molecules are degraded, many others are recalcitrant due to their complex chemical structure and synthetic organic origin⁴. On the other hand chemical methods use huge amount of chemical and produce large volume of sludge which itself requires treatment. These methods are very expensive too. Different physical methods such as membrane filtration processes (nanofiltration, reverse osmosis, electro dialysis etc.) and adsorption techniques are widely used. The major disadvantage of the membrane processes is that they have a limited lifetime before membrane fouling occurs and the cost of periodic replacement must thus be included in any analysis of their economic viability. Liquid phase adsorption is one of the popular methods for the removal of pollutants from wastewater. Adsorption has been found to be superior to other techniques for water re-use in terms of initial cost, flexibility and simplicity of design, ease of operation and insensitivity to toxic pollutants. Adsorption also does not result in the formation of harmful substances. Due to its effectiveness and versatility, activated carbon is

widely employed in water and wastewater treatment. However, the operating cost of activated carbon adsorption is high. Problems of regeneration and difficulty in separation from the wastewater after use are the two major concerns of using this material. This has led many researchers to search for more economic adsorbents.

Many efforts, however, have been made to investigate the use of various low cost organic adsorbents^{5,6}. They should be cheap, easily available and disposable without regeneration. Some low cost botanic materials had directly been used as sorbent for dye adsorption from wastewater, which included apple pomace⁷, wheat straw⁷, orange peel⁸, banana peel⁹, water hyacinth roots¹⁰, water hyacinth¹¹, raw and activated date pits¹², rice husk², peanut hull¹³, palm fruit bunch^{14,15}, sawdust¹⁶, coir pith¹⁷ and neem leaf powder¹⁸.

In the present work, adsorption capacity of jackfruit leaf powder (JLP) was investigated, using methylene blue as a model basic dye. Jackfruit or *Artocarpus heterophyllus* is a tropical plant. It is common in backyards and waste places. During preliminary studies jackfruit leaf powder showed excellent adsorption capacity for methylene blue. Due to its abundant availability and low-cost it can also be disposed off after use without need for expensive regeneration. The objectives of the present study were, to examine the adsorption characteristics of jackfruit leaf powder, to study the feasibility of its use as low-cost adsorbent, to determine the kinetic and equilibrium batch adsorption parameters and to predict the maximum possible adsorption capacity.

Experimental Procedure

Materials

Jackfruit leaves were collected locally and were thoroughly washed successively with tap and distilled water to remove all the dirt and earthy materials and dried in an oven at 105°C overnight. The dried leaves were crushed and the crushed powder was boiled to remove lignin and colouring components. After filtration, the powder was dried at 105°C for 24 h and sieved to particle size 75-300 µm and stored in plastic bottle for use as an adsorbent.

Methylene blue (Fig. 1) was purchased from Merck and used as received without further purification. The stock solutions of MB (1000 mg/L) were prepared in distilled water. All working solutions were prepared by diluting the stock solution with distilled water to the desired concentration.

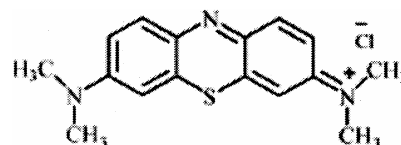


Fig. 1—Structure of methylene blue

Methods

The *pH* at the point of zero charge (pH_{PZC}) of the adsorbent in aqueous phase was analyzed with different system *pH* values by using the titration method¹⁹. 0.1 g adsorbent was taken in 50 mL of 0.1 M KNO_3 solution and agitated with magnetic stirrer. Then the *pH* of the solution was measured after an equilibrium time of 30 min. The titration was carried out with 0.1 M NaOH and 0.1 M HCl, respectively.

Fourier transform infrared spectroscopy (FTIR) of the adsorbent was done (and transferred to Microsoft Excel) by using an FTIR spectrophotometer (Model: FTIR 2000, Shimadzu, Kyoto, Japan). About 150 mg KBr disks containing approximately 2% of JLP samples was prepared shortly before recording the FTIR spectra in the range of 400-4000 cm^{-1} and with a resolution of 4 cm^{-1} . The resulting spectra were the average of 30 scans.

To study the effect of important parameters like *pH*, adsorbent mass, initial concentrations and contact time on the adsorptive removal of MB, batch experiments were conducted at 25±2°C. For each experimental run, 200 mL of MB solution of known concentration, amount of the adsorbent were taken in a 250 mL stopper plastic conical flask at desired *pH* and agitated using a flash shaker (Stuart Scientific Co. Ltd. Model SF1, U.K.) at constant oscillation of 500 osc/min. Samples were withdrawn at appropriate time intervals and were centrifuged and the concentration of dye left in the supernatant solution were analyzed using a UV/VIS spectrometry (Shimadzu Model UV-1601) by monitoring the absorbance changes at a wavelength of maximum absorbance 662 nm.

The effect of *pH* on dye removal was studied over a *pH* range of 2–10. The *pH* was adjusted by the addition of aqueous solutions of HCl or NaOH (0.10 M). The effect of adsorbent mass was studied by contacting 200 mL dye solution of initial concentration 100 mg/L with different amounts of JLP (0.1-0.8 g) till the equilibrium was attained. The kinetics of adsorption was determined by analyzing adsorptive uptake of the dye from the aqueous

solution at different time intervals. The adsorption density defined as the amount of MB adsorbed per unit weight of adsorbent (JLP) at time t , q_t (mg MB/g adsorbent), was calculated by:

$$q_t = \frac{(C_0 - C_t)V}{M} \quad \dots (1)$$

where C_0 and C_t (mg/L) are the liquid-phase concentrations of MB at initial and any time t , respectively. V is the volume of the solution (L), and M is the mass of dry adsorbent used (g).

For adsorption isotherms, 200 mL MB solutions of different initial concentrations (250–500 mg/L) were agitated with 0.25 g adsorbent till the equilibrium was attained. The adsorption density at equilibrium, q_e (mg/g), was calculated by Eq. (1) where C_t was replaced by C_e , the liquid-phase concentration at equilibrium.

Blank experimental runs, with only the adsorbent in 200 mL of distilled water, were conducted simultaneously at identical conditions to account for any colour leached by the adsorbent and adsorbed by glass containers.

Results and Discussions

Determination of pH_{PZC} of adsorbent

The surface charge Q of the adsorbent was calculated from the experimental titration data according to Eq. (2)¹⁹.

$$Q = \frac{1}{w}(C_A - C_B - [H^+] - [OH^-]) \quad \dots (2)$$

where, w - dry weight of adsorbent in aqueous system (g/L); C_A is the concentration of added acid in aqueous system (mol/L); C_B is the concentration of added base in aqueous system (mol/L); $[H^+]$ is the concentration of H^+ (mol/L); $[OH^-]$ is the concentration of OH^- (mol/L). The pH value at the point of zero charge was then determined by plotting Q versus pH .

Figure 2 shows the surface charge of the adsorbent as a function of pH . From Fig. 2 it is obvious that the surface charge of the adsorbent (JLP) around pH 3.9 is zero. Hence the pH_{PZC} at point of zero charge of the Jackfruit leaf adsorbent is 3.9.

Fourier Transforms Infrared Spectroscopy (FTIR) analysis

The FTIR spectrum of JLP (Fig. 3) displays a number of absorption peaks, indicating the complex nature of the

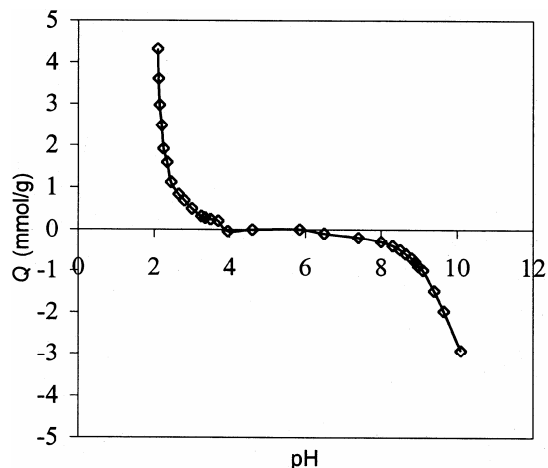


Fig. 2— Surface charge of adsorbent as a function of pH

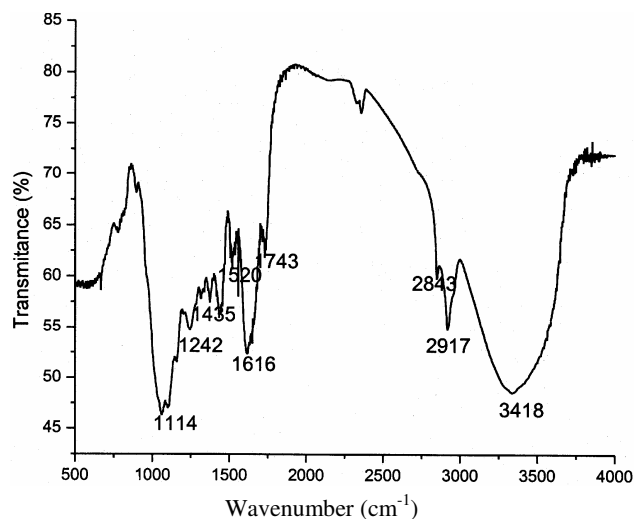


Fig. 3— FTIR spectrum of jackfruit leaf powder (JLP)

material examined. The FTIR spectroscopic analysis indicated broad bands at 3418 cm^{-1} , representing bonded $-OH$ groups. The bands observed at $2930\text{--}2880\text{ cm}^{-1}$ could be assigned to stretching of $C-H$ bond of methyl and methylene groups²⁰. The band observed at 1743 cm^{-1} was assigned to a carbonyl band ($C=O$) of unionized carboxylate stretching of carboxylic acid or pectin ester²⁰, while the peak at 1616 cm^{-1} was attributed to $C=O$ stretching of carboxylic acid with intermolecular hydrogen bond²⁰. The peak observed at 1520 cm^{-1} corresponds to secondary amine group. The band observed at 1435 cm^{-1} could be assigned to symmetric bending of CH_3 . The peaks observed at 1242 and 1114 cm^{-1} could be assigned to $-SO_3$ stretching and $C-O$ stretching of ether groups, respectively²¹. Thus, the FTIR analysis indicate that the JLP is represented by

functional groups such as OH, COOH, NH and CO, that could be potential adsorption sites for interaction with the cationic MB dye.

Effect of pH on adsorption

The initial pH of adsorption medium is one of the most important parameters affecting the adsorption process. Figure 4 shows the effect of pH on the adsorption of methylene blue onto JLP. When the pH of dye solution was increased from 2.4 to 10, the adsorption of methylene blue increased from 10.44 to 45.77 mg/g. It was seen that the adsorption of methylene blue is almost constant in the pH range of 7-10. The uptake of dye increased suddenly after pH of 4. This result could be explained considering the electrostatic interaction between the surfaces of the biosorbent, negatively charged, mainly due to COO⁻ species, since the pKa values of carboxylic acids range from 3.8 to 5.0²², with the cationic dye MB. At lower pH a possible protonation of COO⁻ occurs, precluding the electrostatic attraction with the MB dye, decreasing the adsorbate uptake. Thus, at pH values ranging from 7.0 to 10.0, the COO⁻ groups are available to adsorb the positively charged dye, increasing the removal of MB from aqueous solution. The pH_{PZC} of the biosorbent determined, whose value was 3.9, confirm these results. At pH values lower than pH_{PZC} the surface of JLP is positively charged, precluding the electrostatic attraction of MB, a cationic dye. On the other hand, for pH values higher than pH_{PZC} , the surface of the adsorbent becomes negatively charged and the MB adsorption is facilitated.

Effects of adsorbent dosage

Figure 5 presents the equilibrium adsorption density versus adsorbent mass plot. From the figure it was observed that, the amount of dye adsorbed varied with varying adsorbent mass and it decreased with increase in adsorbent mass. The adsorption density decreased from 169.2 to 28.86 mg/g for an increase in adsorbent mass from 0.1 to 0.8 g, whereas the percentage colour removal increased from 84.64 to 99.46 with an increase in adsorbent mass from 0.1 to 0.8 g. At higher JLP to solute concentration ratios, there is a very fast superficial sorption onto the adsorbent surface that produces a lower solute concentration in the solution than when the biomass to solute concentration ratio is lower. This is because a fixed mass of JLP can only adsorb only a certain amount of dye. Thus, the more the adsorbent mass, the larger the volume of effluent that a fixed mass of

JLP can purify. The decrease in equilibrium adsorption density q_e (mg/g) with increasing adsorbent mass is due to the split in the flux or the concentration gradient between solute concentration in the solution and the solute concentration in the surface of the adsorbent. Thus with increasing adsorbent mass, the amount of dye adsorbed onto unit weight of adsorbent gets reduced causing a decrease in equilibrium adsorption density.

Effect of contact time and concentration

To determine proper MB adsorption and equilibrium time, initial concentrations of MB solutions were changed and time intervals were assessed until no adsorption of adsorbate onto JLP

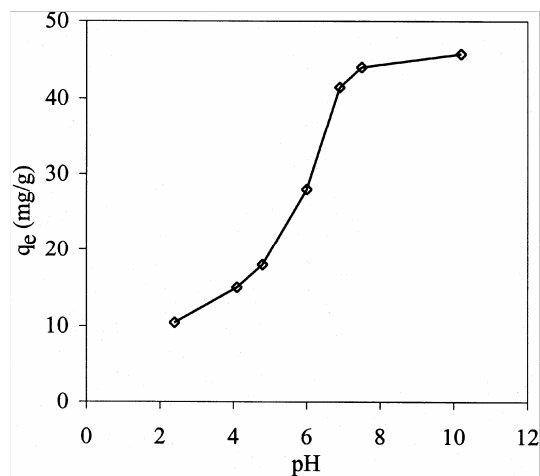


Fig. 4— Effect of pH on the equilibrium adsorption density of MB onto JLP ($C_0 = 100$ mg/L, $M = 0.25$ g, $V = 200$ mL, Temperature = $25 \pm 2^\circ\text{C}$)

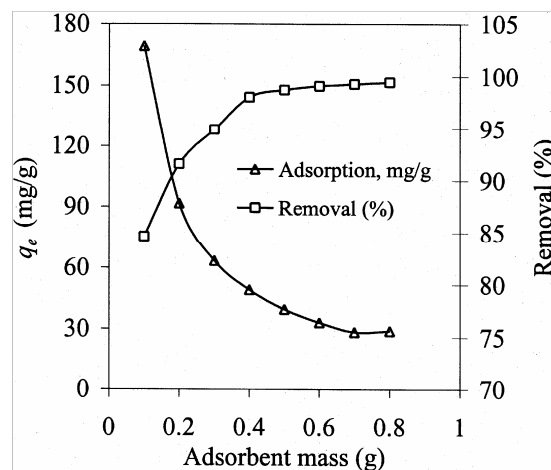


Fig. 5— Effect of adsorbent mass on the adsorption of MB onto JLP ($C_0 = 100$ mg/L, $pH = 6.8$, $V = 200$ mL, Temperature = $25 \pm 2^\circ\text{C}$)

took place. The adsorption data for the uptake of MB versus contact time at different concentrations (Fig. 6) indicate that the adsorption density increased with increase in MB concentration. This is due to increase in the driving force of the concentration gradient, as an increase in the initial dye concentration. The adsorption density for MB increased from 116.06 to 215.89 mg/g as the MB concentration increased from 150 to 300 mg/L. It is also evident from Fig. 6 that the contact time needed for MB solutions with initial concentrations of 150 to 300 mg/L to reach equilibrium was less than 5 h. The surface of JLP may contain a large number of active sites and the solute (MB) uptake can be related to the active sites on equilibrium time. Also up to 90-95% of the total

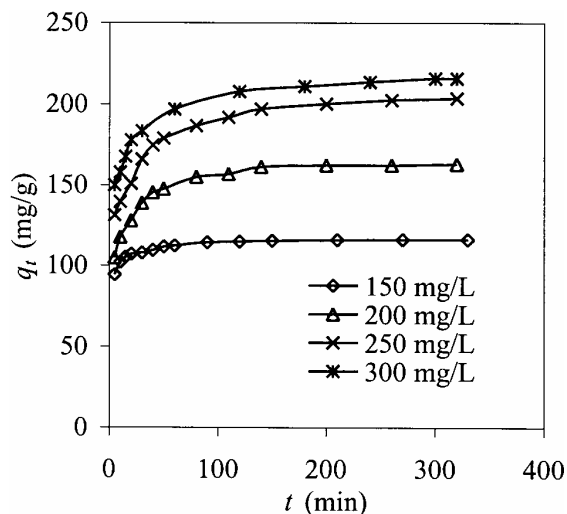


Fig. 6— Effect of contact time and initial concentrations on the adsorption of MB onto JLP ($C_0=150, 200, 250, 300$ mg/L, $M=0.25$ g, $V=200$ mL, $pH=6.8$, Temperature = $25\pm 2^\circ\text{C}$)

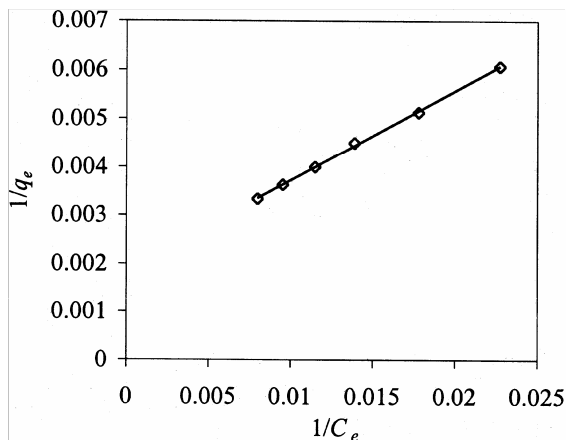


Fig. 7— Langmuir isotherm for methylene blue adsorption onto JLP ($C_0=250, 300, 350, 400, 450$ and 500 mg/L, $pH=6.8$, $M=0.25$ g, $V=200$ mL, Temperature = $25\pm 2^\circ\text{C}$)

amount of dye uptake was found to occur in the first rapid phase (120 min) and thereafter the sorption rate was found to decrease. The higher sorption rate at the initial period (first 120 min) may be due to an increased number of vacant sites available at the initial stage, as a result there exist increased concentration gradients between adsorbate in solution and adsorbate in adsorbent surface. This increased concentration gradients tends to increase in dye sorption at the initial stages. As time proceeds this concentration is reduced due to the accumulation of dye particles in the vacant sites leading to a decrease in the sorption rate at the larger stages from 120 to 320 min.

Adsorption isotherms

The adsorption isotherm indicates how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state. The analysis of the isotherm data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purpose²³. Adsorption isotherm is basically important to describe how solutes interact with adsorbents, and is critical in optimizing the use of adsorbents. Adsorption isotherm study is carried out on two well-known isotherms, Langmuir and Freundlich. The applicability of the isotherm equation is compared by judging the correlation coefficients r^2 .

Langmuir isotherm

Langmuir isotherm assumes monolayer adsorption onto a surface containing a finite number of adsorption sites of uniform strategies of adsorption with no transmigration of adsorbate in the plane of surface²⁴. The linear form of Langmuir isotherm equation is given as:

$$\frac{1}{q_e} = \frac{1}{q_0} + \frac{1}{q_0 K_L} \cdot \frac{1}{C_e} \quad \dots (3)$$

where q_0 and K_L are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. A plot of $1/q_e$ versus $1/C_e$ for MB adsorption onto JLP is presented in Fig. 7. The Langmuir constants K_L , q_0 calculated from Eq. (4) and the correlation co-efficient r^2 are shown in Table 1. The r^2 value of 0.999 indicated that the adsorption data of MB onto the JLP were best fitted to

the Langmuir isotherm model. From Table 1, it was observed that the maximum sorption capacity of JLP for MB was found to be 326.32 mg/g. Table 2 shows the sorption capacity of other low cost material to uptake methylene blue from aqueous solutions for comparison. From Table 2, it was observed that methylene blue sorption capacity of JLP was found to be relatively higher than the other low cost materials. Thus the low cost material JLP can be used as an adsorbent for the removal of dye bearing wastewaters.

Freundlich isotherm

Unlike Langmuir isotherm Freundlich isotherm assumes heterogeneous surface energies, in which the energy term in Langmuir equation varies as a function of the surface coverage²⁴. The linearized form of Freundlich isotherm can be written as:

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad \dots (4)$$

where K_F and n are Freundlich constants with n giving an indication of how favourable the adsorption process. K_F can be defined as the adsorption or distribution coefficient and represents the quantity of

Table 1—Equilibrium constants for methylene blue onto JLP

Freundlich isotherm parameters		Langmuir isotherm parameters			
K_F	n	r^2	q_0	k_L	r^2
(mg/g)/(mg/L) ^{1/n}			(mg/g)	(L/g)	
1.08	1.7476	0.996	326.32	0.010	0.999

Table 2 —Sorption capacities of several low cost adsorbents for the uptake of MB from its aqueous solutions

Adsorbent	Sorption capacity	Adsorbent	Sorption capacity
	q_0		q_0
	(mg/g)		(mg/g)
Rice husk ²	40.59	Silica ²⁹	11.21
Neem leaf powder ³	8.76	Activated sludge ³⁰	256.41
Banana peel ⁹	20.8	<i>Spirodela polyrrhiza</i> ³¹	144.93
Orange peel ⁹	18.6	<i>Aspergillus niger</i> ³²	18.54
Raw date pits ¹²	80.3	Fe(III)/Cr(III) hydroxide ³³	22.8
Peanut hull ¹³	68.03	Cotton waste ³⁴	277
Coir pith ¹⁷	120.43	Neem saw dust ³⁵	2.1–3.6
Fly ash ²⁵	5.57	Lemon peel ³⁶	29
Glass fibers ²⁶	2.24	Palm kernel fiber ³⁷	217.96
Clay ²⁷	6.3	Neem leaf powder ³⁸	8.76
Diatomite ²⁸	156.6	JLP (Present study)	326.32

dye adsorbed onto activated JLP for unit equilibrium concentration. The slope of $1/n$ ranging between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero³⁹. A value for $1/n$ below one indicates a normal Langmuir isotherm while $1/n$ above one is indicative of cooperative adsorption⁴⁰. The plot of $\ln q_e$ versus $\ln C_e$ gave a straight line with slope of $1/n$ with value of 0.57 (Fig. 8), indicating a normal Langmuir isotherm. Freundlich constants K_F and n were also calculated and are listed in Table 1.

Adsorption kinetics

The prediction of batch sorption kinetics is necessary for the design of industrial sorption columns. Kinetic models are used to examine the rate of the adsorption process and potential rate-controlling step. In the present work, the kinetic data obtained from batch studies have been analyzed by using pseudo-first-order and pseudo-second-order models.

The first order equation of Lagergren is generally expressed as follows⁴¹

$$\frac{dq}{dt} = k_1(q_e - q_t) \quad \dots (5)$$

where k_1 is the rate constant of pseudo-first-order sorption (min^{-1}).

The integrated form of Eq. (5) becomes

$$\ln(1 - \frac{q_t}{q_e}) = -k_1 t \quad \dots (6)$$

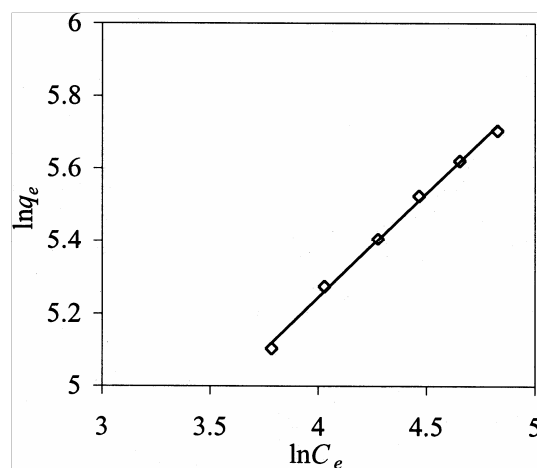


Fig. 8— Freundlich isotherm for methylene blue adsorption onto JLP ($C_0 = 250, 300, 350, 400, 450$ and 500 mg/L, $\text{pH} = 6.8$, $M = 0.25$ g, $V = 200$ mL, Temperature = $25 \pm 2^\circ\text{C}$)

A plot of $\ln(1 - \frac{q_t}{q_e})$ versus t should give a linear relationship with the slope k_1 .

The pseudo-second-order kinetic rate equation is expressed as follows⁴²

$$\frac{dq}{dt} = k_2(q_e - q_t)^2 \quad \dots (7)$$

where k_2 is the rate constant of pseudo-second-order sorption ($\text{g mg}^{-1}\text{min}^{-1}$). The integrated form of Eq. (7) becomes

$$\frac{t}{q} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad \dots (8)$$

If the second order kinetic equation is applicable, the plot of t/q versus t of Eq. (8) should give a linear relationship. The k_2 can be determined from the intercept of the plot.

Figures 9 and 10 show the pseudo-first-order [Eq. (6)] and pseudo-second-order [Eq. (8)] plots, respectively, for different initial dye concentration ranging from 150 to 250 mg/L. The linear lines of Figure 9 should have passed through the origin. Therefore, it is evident from Fig. 9 that the adsorption of MB did not follow the first order kinetic model.

The pseudo first-order and pseudo second-order rate constants and the corresponding correlation coefficients are shown in Table 3. From Table 3 it was noticed that the r_2^2 values were found to be higher than that of r_1^2 for all initial MB concentrations. In the view of these results, it can be said that the pseudo-second-order kinetic model provided a good correlation for the adsorption of methylene blue onto JLP in contrast to the pseudo-first-order model

Desorption studies

Desorption studies as a function of pH were conducted to explore the possibility of recovery of adsorbent and adsorbate. At first the adsorption was carried by increasing the pH of MB solution of initial concentration 50 mg/L from 2.4 to 10 (Figure not shown). The desorption was then carried out by decreasing the pH . Concentration of MB in solution at pH 10 was 4.53 mg/L. The concentration in solution increased from 4.53 to 36.12 mg/L with the decrease in pH from 10 to 2.4 (Fig. 11). These results show

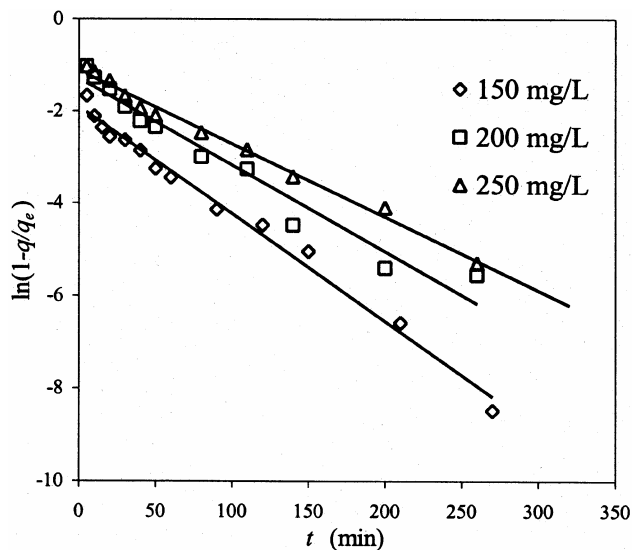


Fig. 9—Pseudo-first-order kinetics for the adsorption of MB onto JLP ($C_0=150, 200, 250$ mg/L, $M = 0.25$ g, $V = 200$ mL, $pH = 6.8$, Temperature= $25 \pm 2^\circ\text{C}$)

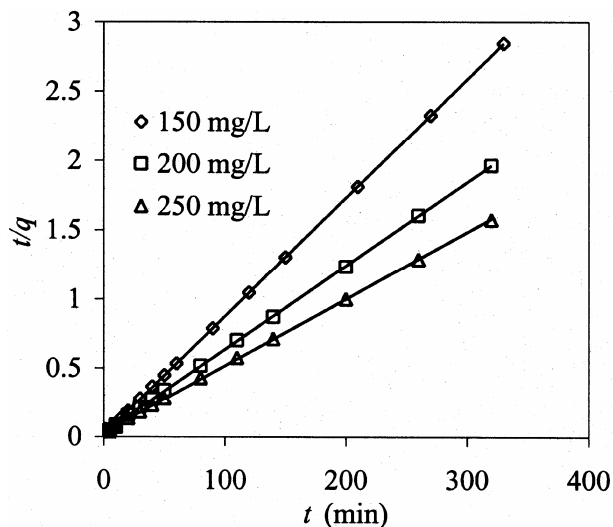


Fig. 10—Pseudo-second order kinetics for adsorption of MB onto JLP ($C_0=150, 200, 250$ mg/L, $M = 0.25$ g, $V = 200$ mL, $pH = 6.8$, Temperature= $25 \pm 2^\circ\text{C}$)

Table 3—Pseudo-first-order and Pseudo-second-order kinetic constants for the adsorption of MB onto JLP

C_0 (mg/L)	First-order kinetic model		Second-order kinetic model	
	k_1 (min^{-1})	r_1^2	k_2 ($\text{g/mg}\cdot\text{min}$)	r_2^2
150	0.0232	0.988	0.00437	1
200	0.0187	0.956	0.00118	0.9999
250	0.0159	0.989	0.00069	0.9997

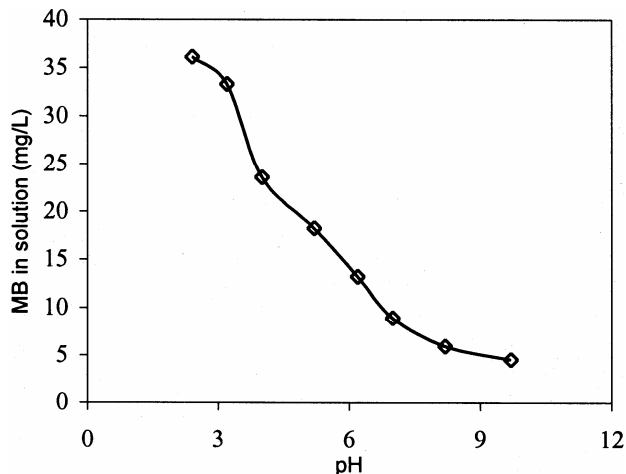


Fig. 11— Desorption of MB as a function of pH ($C_0 = 50$ mg/L, $M = 0.25$ g, $V = 200$ mL, Temperature = $25 \pm 2^\circ\text{C}$)

that the MB adsorbed by JLP can easily be desorbed by decreasing the pH values of the solution using HCl.

Conclusion

From the results of the study it can be concluded that JLP could be used effectively for the removal of MB from aqueous solutions. The amount MB adsorbed per unit mass of JLP was found to increase with increase in contact time, pH and adsorbent dosages. The adsorption process follows pseudo-second order kinetic model. Most of the dye can be desorbed by decreasing the pH. The Langmuir and Freundlich isotherm parameters showed that the adsorption of MB on JLP was favourable. The maximum adsorption capacity obtained from the adsorption of MB onto JLP has been found to be 326.32 mg/g, which is relatively high as comparison to some other similar materials tried earlier by other workers.

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