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Research Article

REMOVAL OF METHYLENE BLUE DYE FROM AQUEOUS SOLUTIONS BY MOSAMBI FRUIT PEEL ACTIVATED CARBON

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ABSTRACT

This paper explains about the mosambi fruit peel activated carbon which adsorbs methylene blue dye from aqueous solutions. The physico – chemical key parameters such as dye concentration, P_H, agitation time, adsorbent dosage has been determined in batch test methods. The equilibrium data obtained were tested using two adsorptions isotherm models, Langmuir and Frendlich adsorption models and two kinetic models – pseudo-first and second order models.

INTRODUCTION

Industrial effluents are foremost causes of environmental pollutions because effluents discharged from dyeing industries such as textile, leather, food processing, dyeing, cosmetics, and paper manufacturing etc. are highly coloured with organic solids. Methylene blue dye is the most commonly used substance for dyeing (1). It is harmful to human which causes increased heart rate, vomiting, shock, jaundice (2). They increase oxidation demand and lower vital oxygen levels in water (3) and (4). So it is very important to remove such dyes from aqueous solutions. It is difficult to remove the dyes by conventional and biological waste water system methods. Many physico – chemical methods such as coagulations, ultrafiltration, electrochemical adsorption, photo – oxidation methods were studied for colour removal but the synthetic dyes are highly resistant to these techniques (5) and (6). Among these techniques, adsorption process has shown better efficiencies and the activated carbons with high adsorption capacity were widely used as absorbent in absorption process to protect the environment. These adsorbents have been readily available and inexpensive and this is an alternative and attractive treatment for adsorption of dyes from effluents (7). Many researchers have prepared activated carbons for using raw materials such as the peel of Cucumis sativa (8), Babul seed (9), Sunflower stalks (10), Barley husk (11), orange and lemon peel (12) as low-cost adsorbents. Even though there are many researchers used these raw materials for preparation, very limited researchers have studied about absorbent nature of activated carbon prepared from the major municipal wastage namely mosambi peel. That few researchers were also studied only the removal of dyes such as Erichrome black T, Yellow 2G dyes (13) and (14) from aqueous solutions at various operating condition Taking these into consideration, the present study has been proposed to study about absorbent nature of activated carbon prepared from mosambi fruit peel. These fruit peel were easily available in abundance which is disposed of as a waste material. They were collected and processed at different operating conditions to convert it to an effective low cost adsorbent, namely the activated carbon. The main aim of this study is to investigate the removal of methylene blue dye from aqueous solutions using activated carbon from mosambi fruit peel and H2SO4, The various parameters such as pH, adsorbent dose, initial dye concentration, time were investigated.

MATERIALS AND METHODS

Adsorbate

Stock solution (1000 mg/L) of MB dye was prepared by dissolving 1 gm of the dye in 1000 mL of distilled water. The stock solutions were diluted to obtain required standard solutions. At room temperature, the batch adsorption studies were preferred. The Table 1 and fig 1 given below represents the properties and structure of Methylene Blue dye respectively.

Preparation of Adsorbent

The peels of mosambi fruit were obtained after extracting the juice from the households and juice shops. Those peels were washed in distilled water and dried in the sunlight for several days.

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Table 1 Properties of MB Dye

Sl. No.	Chemical Properties	
1.	Chemical formula	$C_{16}H_{18}N_3Scl$
2.	Molar mass	319.85 gm / mol
3.	Melting point	100 to 110°C
4.	Boiling point	Decomposes
5.	Water solubility	43,600 mg/Lit at 25°C
6.	Density	1.757 gm/cm^3
7.	Vapour density	13
8.	P_{ka}/P_{kb}	3.8(P_{ka})

Fig.1 Structure of Methylene Blue

Then it is crushed into small pieces and soaked with the chemical activating agent H_2SO_4, in the ratio of 1:1 for twenty-four hours. The resulting black product was kept in hot air oven for about three hours at 110°C and then it is carbonized by keeping it in a muffle furnace for 90 mins in 350°C. This activated part of the carbon is washed several times with $NaHCo_3$ followed by distilled water to remove the excess acid present. Then the resulting products is once again kept in hot air oven at 110°C ± 5°C for more than 3 hours to obtain a complete fine black activated carbon. Finally, it is preserved in desicator for further use. The prepared activated carbon has some specific characteristics on the removal of methylene blue dye from aqueous solution and it is determined by batch adsorption studies. The experimental data was analyzed according to the linear form of the Langmuir and Freundlich isotherms. To investigate the mechanism of adsorption, kinetics models namely the pseudo-first and second orders were used.

Batch Adsorption Studies

For batch adsorption studies, the adsorption experiments were performed at room temperature. To know the effect of initial dye concentration, 0.1 gm of adsorbent was added with 100 ml of MB solutions with known initial dye concentration (50-500 mg/lit) and shaken for 2hrs to reach equilibrium and then it is centrifuged. The adsorbent and the adsorbate were separated by filtration and the filtrate was analyzed for residual methylene blue concentration spectrophotometrically using UV-visible spectrophotometer (Shimadzu UV1800) at λ max = 665 nm. The percentage removal of MB dye from solutions was calculated by the following equation 1,

$$\%\ of\ removal = \left(\frac{Co \quad Ce}{Co}\right) \times 100 \qquad (1)$$

where
Co – Initial dye concentration (mg/lit)
Ce – Equilibrium dye concentration (mg/lit)
The amount of dye adsorbed on adsorbent (q_e) was determined by eqn 2,

$$q_e = (Co \quad Ce)\frac{V}{W} \qquad (2)$$

where
V – Volume of dye solution (lit)
W – Mass of adsorbent (gm)

Similarly, Influence of each parameter (Time, Adsorbent dosage, pH) was evaluated in an experiment by varying the parameter under evaluation, while all other parameters in the experiment were maintained as constant. The experimental data were analysed for adsorption isotherm and kinetics.

Adsorption Isotherm

The adsorption isotherm represents the relationship between the amount absorbed by a unit of adsorbent and the amount of adsorbate remaining in the solution at equilibrium. The experimental data were analyzed according to the linear form of the Langmuir and Frendlich Isotherms. According to Langmuir adsorption theory, monolayer adsorption takes place on homogeneous sorption sites of equal energy (15). It is represented by the following equation 3.

$$(C_e/q_e) = \frac{1}{Q_0 b} + \frac{Ce}{Q_o} \qquad (3)$$

Where,
C_e – Equilibrium concentration of dye (mg/lit)
q_e – Amount adsorbed at equilibrium (mg/gm)
Q_0,b – Langmuir constants

The linear plots of C_e/q_e versus C_e suggest the applicability of the Langmuir isotherms. The values of Q_0 and b were determined from slope and intercepts of the plots.

The essential characteristic of the Langmuir isotherms can be expressed in terms of a dimensionless constant separation factor R_L which is given by equation (4),

$$R_L = \frac{1}{(1 + bC_0)} \qquad (4)$$

The value of R_L indicates the type of isotherms, $0 < R_L < 1$ – Favourable, $R_L > 1$ – Unfavourable, $R_L = 1$ – Linear, $R_L = 0$ – Irreversible.

By assuming a heterogeneous surface with a non-uniform distribution of heat of adsorption over the surface is determined by Frendlich isotherm. It is represented by equation (5),

$$Logq_e = Logk_f + \left(\frac{1}{n}\right) LogC_e \qquad (5)$$

Where,
q_e – Amount adsorbed at equilibrium (mg/gm)
k_f and n - Frendlich constants
C_e – Equilibrium concentration of dye (mg/lit)

The linear plot of log q_e verus log C_e suggests the applicability of the Frendlich isotherm. The constant values of k_f and n were determined from the graph.

Adsorption Kinetics

The kinetic studies were conducted to estimate the contact time required for the attainment of equilibrium between the dissolved and solid bound adsorbate. Fitting the experimental data into different kinetic models enables to study the adsorption rate, and predict information about adsorbent/adsorbate interaction (physisorption or chemisorption) [16]. In this study, two different models were used such as the pseudo-first-order [17] [18] [19] and the pseudo-second-order [20] [21].

Pseudo-First-Order Equation

To describe the kinetic process of a solid – liquid phase adsorption, lagrangian obtained a first order rate equation in 1898. It represents the adsorption capacity rate (22), (23) which is given by the pseudo first order equation (6),

$$Log(q_e \quad q_t) = Log\ q_e \quad \left(\frac{k_1}{2.303}\right)t \qquad (6)$$

Where,
q_e – Amount adsorbed at equilibrium (mg/gm)
q_t –Adsorption capacity at time t (minutes) (mg/gm)
k_1 – Rate Constant

Pseudo-Second-Order Rate Equation

The pseudo second order kinetic model (24) is expressed by equation (7).

$$\frac{t}{q_t} = \frac{1}{(k_2 \times q_e^2)} + \frac{t}{q_e} \qquad (7)$$

q_e – Amount adsorbed at equilibrium (mg/gm)
q_t –Adsorption capacity at time t (minutes) (mg/gm)
k_2 – Rate Constant

RESULTS AND DISCUSSION

Investigation of sorption parameters

The Effects of Initial Dye Concentration, Adsorbent dosage, contact time, and P_H on the removal of Methylene blue dye by citrus limetta (mosambi) fruit peel activated carbon was studied.

Dye Concentration Variation

The amount of dye adsorbed highly depends on the initial dye concentration. To study the effects of different initial concentration of Methylene blue dye, concentrations were varied from (50 mg/lit – 500 mg/lit) with constant adsorbent dosage (0.1 gm/lit) value, P_H (7) and contact time (120 minutes), the adsorption behaviour is determined which given in Table 2. The percentage adsorption of dye is maximum (95.25) at 50 mg/lit and slowly decreased to (46.30) at 450 mg/lit. This is because the increase of initial dye concentrations, the dye molecules gets highly resistant to the surface of the adsorbent which is clear from Figure (2).

Table 2 Effects of Different Dye Concentration on MB Removal, (Absorbent Dosage = 0.1 mg/lit, initial P_H = 7, Time = 120 minutes

Dye Concentration (mg/lit)	Percentage of Dye Removal
50	95.25
100	92.46
150	88.91
200	80.03
250	73.42
300	69.24
350	62.91
400	57.32
450	50.39
500	46.30

Time Variation

For the evaluation of adsorption as a function of time, 50 ml of 100 mg/lit of dye concentration with 0.1 gm/lit of adsorbent dosage at constant P_H (7) is taken and the agitation time period is varied from 15 minutes to 150 minutes.

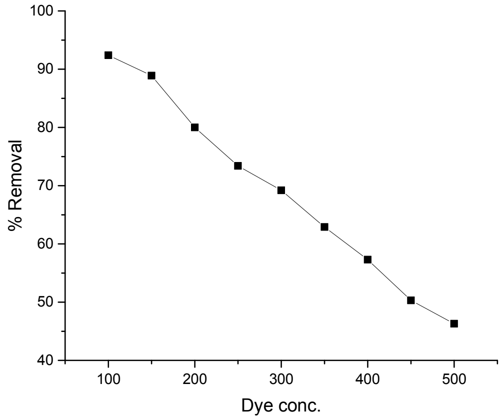

Fig 2 Effects of Different Dye Concentration on MB Removal

From the Figure 3, the adsorption of dye increased slowly and attained equilibrium at 120 minutes further agitation time showed no change in the adsorption process. The percentage of adsorption increased from (46.87) to (96.24), which is shown in Table 3.

Table 3 Effects of Different Time Periods on MB Removal, (Absorbent Dosage = 0.1 mg/lit, initial P_H = 7, Dye Conc = 100 mg/lit

Time Periods (minutes)	Percentage of Dye Removal
15	46.87
30	55.52
45	63.18
60	72.01
75	76.87
90	80.27
105	85.40
120	92.73
135	94.71
150	96.24

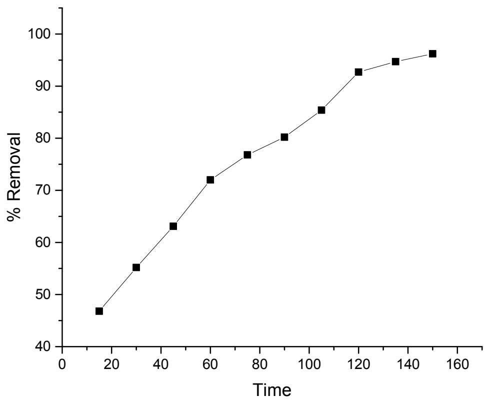

Fig 3 Effects of Different Time Periods on MB Removal

Adsorbent dosage variation

The adsorbent dose is an important parameter in adsorption studies because it determines the capacity of the adsorbent for a given initial concentration of dye solution. By varying the adsorbent dosage value from 0.05gm/lit to 0.5 gm/lit keeping constant P_H value (7), contact time (120 minutes), dye concentration (100 mg/lit), the percentage of adsorption is minimum (48.15) for an adsorbent dose value of 0.05 gm/lit and reaches maximum (98.25) for a dose value of 0.50 gm/lit which is given in Table 4. Due to the availability of large surface area, the percentage of adsorption increases with increase in adsorbent dosage. It is clear from Figure 4.

Table 4 Effects of Different Absorbent Dosage on MB Removal, (Initial P_H = 7, Dye Conc = 100 mg/lit, Time = 120 mins)

Absorbent dosage (mg/lit)	Percentage of Dye Removal
0.05	48.15
0.10	57.44
0.15	60.84
0.20	68.22
0.25	76.72
0.30	79.48
0.35	84.44
0.40	92.31
0.45	96.48
0.50	98.25

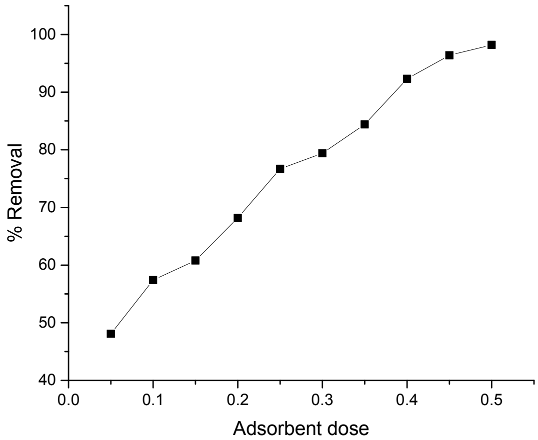

Fig 4 Effects of Different Absorbent Dosage on MB Removal

P_H Variation

The pH of the dye solution is an important factor in the adsorption processes, especially on the adsorption capacity. It influences not only the surface charge, and the degree of ionization of the functional groups of the adsorbent, but also the dye chemistry. An effective change in P_H values causes changing adsorption.

Table5 Effects of Different P_H on MB Removal, (Absorbent dosage =0.1 mg/lit, Time = 120 mins, Dye Conc = 100 mg/lit

P^H	Percentage of Dye Removal
1	65.49
2	73.48
3	81.20
4	83.81
5	90.69
6	93.18
7	95.40
8	91.14
9	85.64
10	82.10
11	79.84
12	76.39

The surface properties of the adsorbent and the degree of ionization of the dye get affected by the hydrogen ion. Experiments were carried out by keeping constant values of contact time (120 minutes), dye concentration (100 mg/lit), adsorbent dosage (0.1 gm/lit) and P_H is varied from 2 to 14. For the P_H value 2 to 7, the percentage of removal of dye increases and after P_H 7, there is a decrease in percentage removal of dye. This can be seen from the Figure 5. The optimum P_H value for the mosambi fruit peel activated carbon reaction with Methylene blue dye is 7. It is clear from the Table 5.

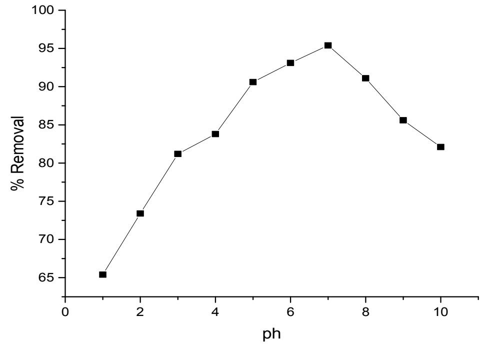

Fig: 5 Effects of Different PH on MB Removal

Adsorption Isotherms

This explains about the distribution of adsorbate molecules on the adsorbent surface, when equilibrium stage is reached during the adsorption process. This explores about the specific relation between the adsorbent and the adsorbate. The Langmuir and Freundlich isotherms models were selected for adsorption isotherms studies.

Langmuir Isotherm Model

The Langmuir model is valid for monolayer adsorption The Langmuir isotherm model is based on the assumption that there is a finite number of active sites which are homogeneously distributed over the surface of the adsorbent These active sites have the same affinity for adsorption of a monomolecular layer and there is no interaction between adsorbed molecules [25]. The data were fitted to Langmuir Isotherm Model and the results were shown in fig 6 and presented in table 6.

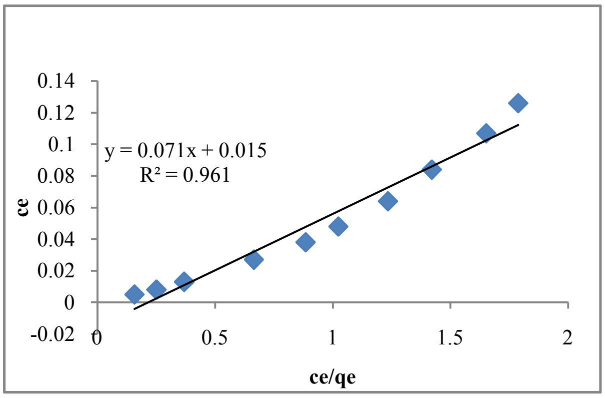

Fig 6 Langmuir Isotherm for the removal of MB Dye

Freundlich Isotherm Model

The Freundlich isotherm model applies to adsorption on heterogeneous surfaces with the interaction between the adsorbed molecules, and is not restricted to the formation of a monolayer. This model assumes that as the adsorbate concentration increases, the concentration of adsorbate on the adsorbent surface also increases and, correspondingly, the sorption energy exponentially decreases on completion of the sorption centres of the adsorbent. The datas were fitted to

Freundlich Isotherm Model and the results were shown in fig 7 and presented in table 6.

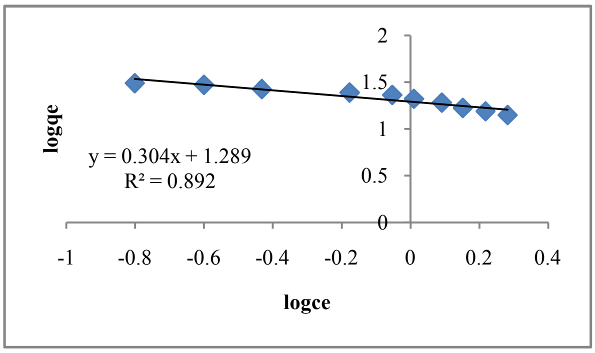

Fig 7 Freundlich Isotherm for the removal of MB Dye

Table – 6 Calculated values of Langmuir and Frendlich Constants

#	**Langmuir Model**	**Q0** = 14.08, **b** = 4.73, R_L = 0.0147, **R2** = 0.961
2	**Frendlich Model**	**n** = 3.289, K_f = 0.0514, R^{2} = 0.892

The results of Langmuir isotherm model reveals that, when Ce/q_e were plotted against Ce, a straight line was obtained with slope $1/Q_0$ which indicates that the adsorption of MB on mosambi peel activated carbon follows the Langmuir isotherm. The calculated Langmuir constant values 'b' and 'Q_0'' are 14.08 mg/g and 4.73 L/mg, respectively and they were given in table 6. The results shows the homogenous nature of mosambi peel activated carbon and also the outer surface of the prepared activated carbon is covered by monolayered dye molecule. The dimensionless equilibrium factor (R_L) value was 0.0147 which was less than unity showing that the adsorption of dye molecules onto mosambi peel activated carbon was favorable. The results of Frendlich isotherm model reveal that when log q_e were plotted against log C_e a straight line was obtained with slope $\left(\frac{1}{n}\right)$ which shows that the adsorption of MB dye molecules on the MPAC adsorbent has several different types of adsorption sites and follows the Frendlich isotherm. The constant values such as k_f and n are 0.0514mg/g and 3.289 respectively and they were shown in table 6. According to [26], the n values between 2 and 10 represent good adsorption. The calculated n value of 3.289 indicates good adsorption of MB dye molecules on MPAC. The value of 1/n is 0.304 which ranges between 0 and 1 represents the heterogenous nature of MPAC. i.e the multilayer adsorption of dye molecules by mosambi peel activated carbon.

Adsorption Kinetics

This explains the adsorption process, the uptake rate of adsorbate molecules by the adsorbent. It explores about the controlling mechanism of the adsorption process, and the experimental datas were fitted to the pseudo-first-order and second-order models.

Pseudo First Order Model

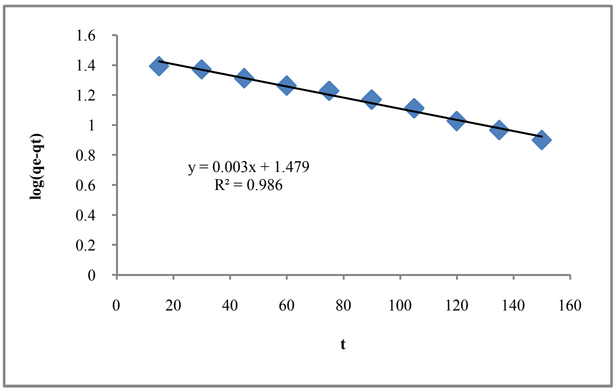

Fig 8 Pseudo First Order for the removal of MB Dye

Pseudo Second Order Model

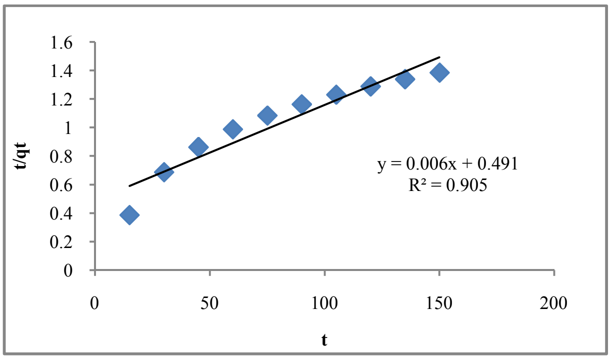

Fig: 9 Pseudo Second Order for the removal of MB Dye

Table – 7 Calculated values of Pseudo First Order and Second Order constants

S:no	Kinetic model	Constant Values
1	**Pseudo First Order Model**	qe = 1.006, k1= 3.406, R2 = 0.946
2	**Pseudo Second Order Model**	q_e = 2.036, k_2= $4.02*10^{-1}$, R^{2} = 0.905

Table – 7 represents the rate constants and correlation coefficients (R^2) of the two kinetic models. For the pseudo-first-order kinetic model, the plot versus log ($q_e - q_t$) and t gives a linear form which represents that when the adsorption takes place through boundary by diffusion method the kinetics follow the pseudo first order equation. The correlation coefficient (R^2) for the pseudo first order value is 0.946.

For the pseudo-second-order kinetic model, a straight line is obtained for the plots t/q_t versus t. This explains the adsorption process is controlled by chemisorption which involves the valency forces through the exchange of electrons between the adsorbate and the adsorbent. The value of (R^2) for this model is $R^{2=}$ 0.905.

CONCLUSION

The activated carbon prepared from natural waste, peel of mosambi fruits is an efficient and effective adsorbent in the removal of Methylene blue dye from aqeous solutions. The adsorption of Methylene blue dye depends on an optimum condition such as contact time, dye concentration, adsorbent dosage, P_H value. Experimental results reveal that the adsorption characteristic of this adsorbent obeyed both linear form of Langmuir, frendlich isotherms, Pseudo first and second Order kinetic models. It indicates that this adsorbent is having a comparable advantage over other adsorbents which obey only one isotherm. The important characteristic parameter Methylene blue dye adsorption shows that this adsorbent acts as a major economic tool for solving the problems of dye solution and also helps in the disposal of natural waste converted to a value product.

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