Removal of Colour Methylene Blue dye from contaminated water using satpuda region *Mangifera indica* fruit peel as a very low cost adsorbent

Shinde Anil V.[1*], Patil Bharat N.[2] and Beldar Anil G.[3]
1. MJPV Arts, Commerce and Shri V. K. K Science College Dhadgaon, Dist- Nandurbar (MS), INDIA
2. RFNS Senior Science College, Akalkuwa, Dist- Nandurbar (MS), INDIA
3. PSGVPM Arts, Commerce and Science College Shahada, Dist- Nandurbar (MS), INDIA
*anilshinde593@gmail.com

Abstract
The potential of Mangifera Indica fruit peel (MIFP) as an inexpensive adsorbent for the removal of methylene blue (MB) dye was examined in the current study. To determine the several parameters such as contact duration, initial dye concentration, adsorbent dose, pH and temperature, influenced adsorption, batch adsorption experiments were carried out. It was found that pseudo-second order kinetics worked best. The Langmuir, Freundlich and Temkin isotherm models were used to assess the experimental results.

The Langmuir isotherm adsorption isotherm was discovered to have the maximum adsorption capacity for monolayer coverage and it was determined best to describe the data, Results suggest that MIFP is a very effective low cost adsorbent for the removal of dyes from wastewater.

Keywords: *Mangifera Indica* fruit peel (MIFP), Dye removal, Methylene blue, Kinetics study and Adsorption isotherm.

Introduction
Water is essential for the survival of all living organisms. Today contamination of freshwater systems with a wide variety of organic pollutants is a subject of great concern. Out of all the contaminants present in industrial effluents, dyes are an important class of organic pollutants and can be identified even by human eye. Dyes are used as colouring agents in different industries such as textiles, food, paper, rubber, plastics, cosmetics, leather etc. The discharge of wastewater from these industries to water resources causes unavoidable problems due to the toxic and unpleasant nature of dyes. The presence of dyes in water in trace amount is undesirable because most of them are toxic, mutagenic and carcinogenic[1]. Dyes also prevent light penetration and thereby reduce photosynthetic activities of water streams and disturb aquatic balance and equipoise (equilibrium). Thus, removal of dyes from wastewater before discharge is a challenging task.

Methylene blue (MB), a cationic dye, is most commonly used as colouring agent for cotton, wool and silk. It is also used as a staining agent to make certain body fluids and tissues easier to view during surgery and diagnostic examinations. The medical applications of MB also include the treatment of methemoglobinemia and cyanide poisoning. In spite of several applications, this dye has a number of negative impacts on human beings and animals such as irritation of mouth, stomach, throat and oesophagus with symptoms of nausea, abdominal discomfort, vomiting and diarrhoea. Skin contact may cause mechanical irritation resulting in redness and itching. Thus, removal of MB dyes from wastewater is of great concern from human and environmental aspects.

Several techniques like flocculation, adsorption, oxidation, electrolysis, biodegradation, ion-exchange, photo catalysis have been employed for the removal of dyes from wastewater[10]. Amongst the various techniques, adsorption has received considerable attention due to its several advantages in terms of cost, ease of operation, insensitivity to toxic pollutants, flexibility and simplicity of design[15,39]. Among variety of adsorbents, activated carbon may be logically the most preferred adsorbent for the removal of dyes because of its excellent adsorption ability[41]. However, widespread use of activated carbon is restricted because of its high cost[4]. Thus, attention has shifted to find cheaper and efficient alternatives of activated carbon.

Natural materials, domestic, agricultural and industrial wastes and bio-sorbents represent potential alternatives. A number of non-conventional and low cost adsorbents have been proposed by many workers for the removal of dyes[9,20,42,43]. These include agricultural waste products such as saw dust[1], bark[34], orange peel[44], industrial waste products like metal hydroxide sludge[35], red mud[47], fly ash[33], clay materials like bentonite[19], diatomite[5], zeolites[7,37], siliceous materials silica beads[28], alunite[36], dolomite[46] and biosorbents such as chitosan[48], peat[6], biomass[3,31] and others (cyclodextrin[13,14], starch[17], cotton[11] etc.).

Many low cost adsorbents such as jack fruit peel[21], garlic peel[23], hazelnut shell[18], pine apple stem[24], longan shell[49], spent tea leaves[22], zeolite[26], corn cobs[40] etc. have been reported in literature for the adsorption of methylene blue dye. *Mangifera Indica* is a species of *Mangifera* family, commonly known as mango. Borneo, Java, Sumatra and the Malay Peninsula are home to the majority of the species of the genus Mangifera, which is native to tropical Asia. India and Myanmar are the birthplaces of the most widely grown Mangifera species, M. indica, or mango. In India, it is available throughout the year and abundantly during the

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months of April, May and June. It is generally taken as a fresh fruit or consumed as juice. In Satapuda region, amchur preparation is done from the waste of peel of mangifera indica fruit. The peels of *Mangifera Indica* are discarded as waste.

In view of the above facts, the present investigation was undertaken with the prime objective to explore the feasibility and utilisation of *Mangifera indica* fruit peel (MIFP) for the adsorptive removal of MB dyes. It has also been decided to optimize, wherever possible, the important variables which affect the adsorption capacity.

Material and Methods

Experimental: The batch equilibrium experiments were planned to determine the efficiency of MIFP for the removal of MB from aqueous solution. The adsorption experiments were carried out by taking 25 mL of adsorbate solutions with varying initial concentrations (25–250 mg/L for MB dye) in different conical flasks. For MB-MIFP system, the fixed quantity (0.05 g) of adsorbent was added to each flask kept on a shaker and equilibrated for 4 hours. After filtration, the residual concentration of MB in each flask was measured by a UV-Vis spectrophotometer at a pre-optimized λ_{max} of 665 nm. The effects of various parameters affecting adsorption process i.e. contact time, adsorbent dose, pH, particle size and temperature were studied and optimized.

The desorption experiments were conducted in different desorbing media. 0.5 g adsorbent was treated with 50 mL of 50 mg/L dye solution for 3 hours. The dye-loaded adsorbents were filtered and thoroughly washed several times with deionized doubly distilled water (DDDW) to remove any un adsorbed dye present. The dye-loaded adsorbents were then agitated with 25 mL of different desorbing solutions and equilibrated for 4 hours. The desorbed dye concentration was measured and percent desorption was calculated.

Results and Discussion

Effect of contact time and initial dye concentration: The effects of contact time and initial dye concentration on the adsorption capacity of MIFP at different initial concentrations of MB are shown in fig. 1. The figure shows that adsorption capacity increases with contact time and reaches to equilibrium after 3 hours. However, the increase is relatively higher during initial 30 minutes. Rapid increase in adsorption during the initial stage may presumably be due to the availability of vacant active sites on the surface of the adsorbent. The slow increase at the later stage is due to the diffusion of dye into the pores of the adsorbent because the external. sites are completely occupied. It is also inferred from the figure that the adsorption capacity increases with increasing initial dye concentration at any dye-adsorbent contact time.

Effect of adsorbent dosage: The effect of adsorbent dose on the equilibrium adsorption capacity and removal efficiency for MB is shown in fig. 2. It has been observed that as the concentration of MIFP adsorbent is increased from 0.4 o 2.0 g/L, removal efficiency increased rapidly from 92.6% to 93.1%. Thereafter, no significant change has been observed. As the adsorption saturation reached at adsorbent dose above 2.0 g/L, this concentration was chosen as optimum dose for further studies. The increase in removal efficiency in this range of adsorbent (0.4 – 2.0 g/L) is due to increase in total surface area and the availability of more binding sites for adsorption. The decrease in adsorption capacity with increase in adsorbent dosage might be due to interaction of adsorbent particles like aggregation or agglomeration which resulted in decrease of effective surface area per unit weight of the adsorbent.

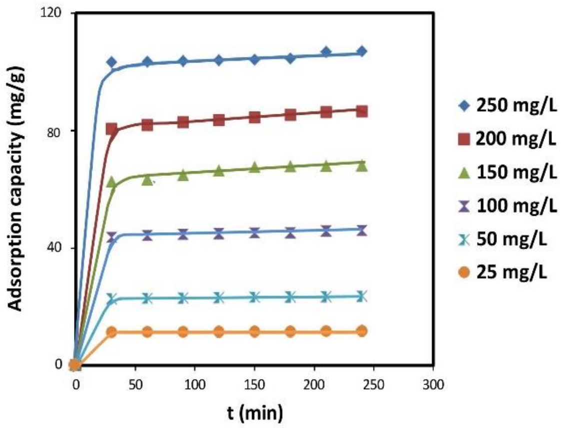

Fig. 1: Effect of contact time on the adsorption of MB onto MIFP at different concentrations

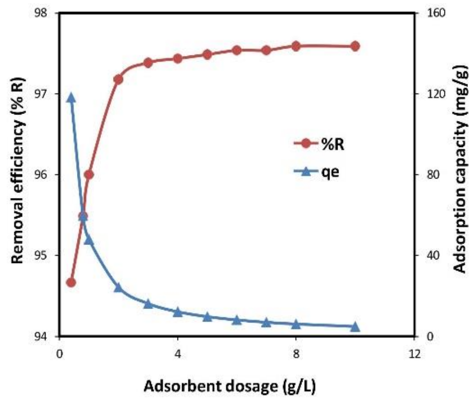

Fig. 2: Effect of adsorbent dosage on removal efficiency and adsorption capacity for the adsorption of MB onto MIFP

Effect of pH: The effect of pH on removal efficiency of MB dyes by MIFP adsorbent is depicted in fig. 3. The adsorption capacity has been observed to increase drastically with increase in pH of the solution (up to a value of 4.0 for MB) after which it remains almost constant with the further increase in pH up to 12.0. In acidic condition i.e. at lower pH, the surface of adsorbent absorbs H^+ ions and acquires positive charge resulting in an electrostatic repulsion between positively charged adsorbent and cationic dyes (MB) which reduce the adsorption. Also there exists

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competition between cationic dye and H^+ ions for the adsorption sites on MIFP adsorbent.

At higher pH, increased adsorption is observed because of the electrostatic attraction between cationic dyes and negatively charged adsorbent (because of the absorption of OH^- ions in alkaline medium). The result is in agreement with the existing hypothesis of increasing negatively charged sites/decreasing positively charged sites of adsorbent with increase of pH[29].

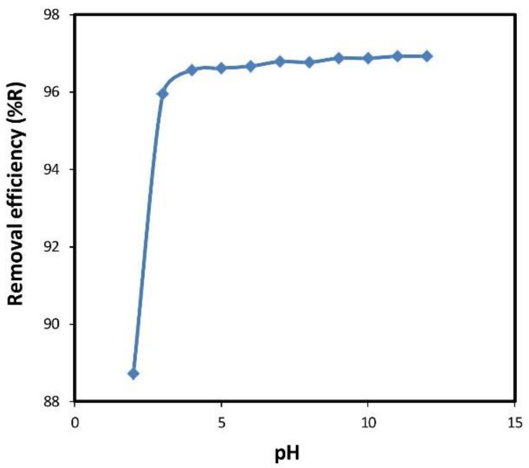

Fig. 3: Effect of pH on adsorption of MB on to MIFP

Point of zero charge: The point of zero charge (pH_{pzc}) value of MIFP was found to be 8.0 (Fig. 4). This indicates that the adsorbent surface has positive charge at pH < 8.0, net zero charge at pH = 8.0 and negative charge at pH > 8.0. Therefore, point of zero charge study reflects that the favorable condition for adsorption of cationic dye by MIFP adsorbent is the medium having pH greater than 8.0. Thus a safer pH value of 9.0 was selected for the adsorption studies of MB onto MIFP adsorbent.

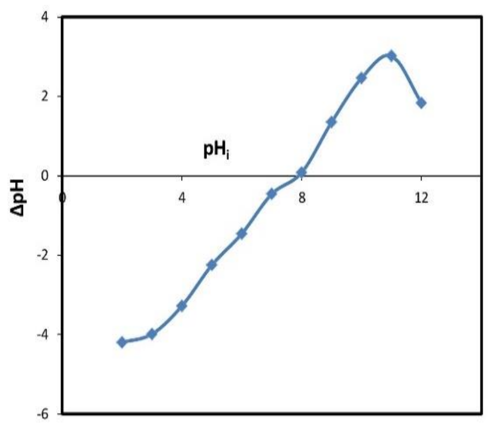

Fig. 4: Point of zero charge for MIFP

Effect of particle size: Adsorption of MB dyes on MIFP adsorbent was studied using three different size ranges of adsorbent particles (80–150, 150–200 and >200 BSS mesh) showing the effect of particle size on the removal efficiency of MB. It has been observed that as the particle size is decreased, removal efficiency increased from 93% to 97.2% for MB. It is due to the fact that smaller particle size provides large surface area for adsorption which results in the higher removal of dye. Maximum removal efficiency is associated with mesh size >200. However, because of handling problem accompanying with small particles and for easy separation of solid and liquid phases, particles of 80–150 BSS mesh size range have been chosen throughout.

Effect of Temperature: The Adsorption of MB dyes on MIFP adsorbent dye increases rapidly with an increase in temperature from 30^0 C to 60^0 C. The increase in adsorption capacity was attributed to the enlargement of pore size and activation of the sorbent surface with temperature. Further rise in temperature increases the mobility of the large dye ions and reduces the swelling effect, thus enabling the large dye molecule to penetrate further.

Adsorption kinetics: The kinetic data have been analyzed in the light of different kinetic models, namely, pseudo-first order and pseudo-second order. The kinetic parameters associated with these models so obtained are summarized in table 1. On comparing the values of correlation coefficient, one can infer that the pseudo-second order kinetic model is best to represent the adsorption results of both dyes i.e. MB. From the linear plots shown in fig. 5, following mathematical equations have been obtained for the pseudo-second order kinetics for the adsorption of MB by MIFP.

Pseudo-second order kinetics have also been observed to obey the adsorption of MB onto different adsorbents such as wheat straw[51], cotton stalk[16], parsley stalk[2], rice hull ash[12], shaddock peel[30], Arthrospira platensis[32] and coconut bunch waste[25]. The present values of pseudo-second order rate constant and $q_{e,cal}$ are incorporated in table 1.

$$\frac{t}{q_t} = \frac{t}{23.255} + \frac{1}{30.960} \qquad (R^2 = 0.9999)$$

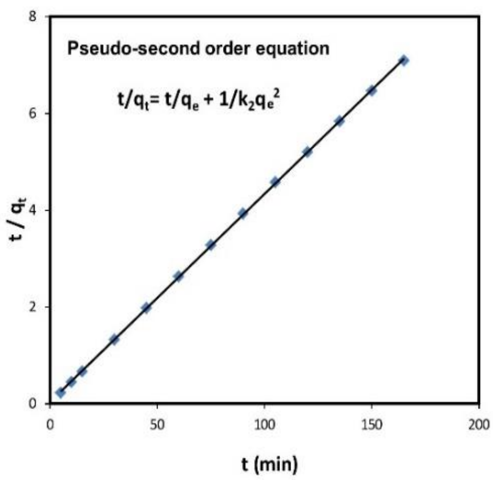

Fig. 5: Pseudo-second order kinetic model for the adsorption of MB onto MIFP

Table 1
Isotherm parameters for the adsorption of MB onto MIFP

Isotherm models	**Parameters**	**MB dye**
Pseudo-first order	k_1 (min^{-1}) $q_{e,\ exp}$ (mg/g)R_L $q_{e,\ cal}$ (mg/g) R^2	0.013 23.333 1.092 0.9022
Pseudo-second order	k_2 (g/mg min) $q_{e,\ exp}$ (mg/g)R_L $q_{e,\ cal}$ (mg/g) R^2	0.057 23.333 23.255 0.9999

Table 2
Pseudo-second order rate constant for the adsorption of MB onto different adsorbents

System	**Pseudo-second order model**		**R^2**
	k_2 (g/mg min)	**$q_{e,cal}$ (mg/g)**	
MB-Shaddock peel[30]	0.00005	131.19	0.9998
MB-Coconut bunch[25]	0.041	34.36	0.991
MB-*Mangifera indica Fruit* peel*	0.057	23.255	0.9999

* Present study

Table 3
Kinetic parameters for the adsorption of MB onto MIFP

Isotherm models	**Parameters**	**MB dye**
Langmuir	q_m (mg/g) K_L (L/mg) R_L R^2	227.3 0.0289 0.4089 0.9787
Freundlich	n K_F ($mg^{1-1/n}L^{1/n}$/g) R^2	1.438 8.758 0.9676
Temkin	b (J/mol) K_T (L/g) R^2	83.084 0.648 0.9756

Adsorption isotherms: The experimental results have been fitted on the Langmuir, Freundlich and Temkin isotherm models. For this purpose, the linear mathematical forms of these models were used. The values of different isotherm constants for the adsorption of MB onto MIFP are listed in table 3. In the light of relative values of correlation coefficients (R^2), table 3 clearly indicates that the isotherm data for MB can be best represented by Langmuir adsorption isotherm model. The linear plot of this model illustrated in fig. 6 has generated following solved equation:

$$\frac{1}{q_e} = \frac{0.1527}{C_e} + \frac{1}{227.3} \qquad (R^2 = 0.9787)$$

It is worth to mention here that adsorptive removal of MB dye by other adsorbents obtained from agricultural waste such as wheat straw[51], cotton stalk[16], cucumber peels[2], rice hull ash[12], shaddock peel[30], cottonseed hull[50], banana leaves[27] and Bacillus subtilis[8] and clay[38] has also been found to obey Langmuir isotherm. The values of Langmuir constants have been compared in table 3. This table clearly indicates that the results obtained in the present study fall within the ranges comparable to those reported earlier.

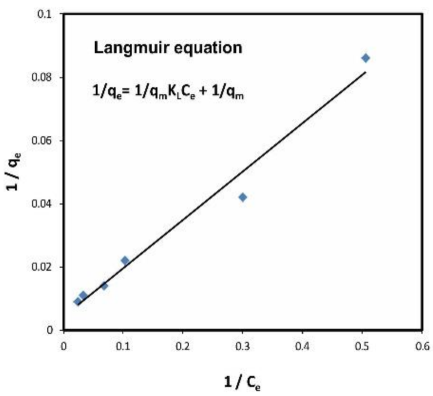

Fig. 6: Langmuir adsorption plot of MB onto MIFP adsorbent

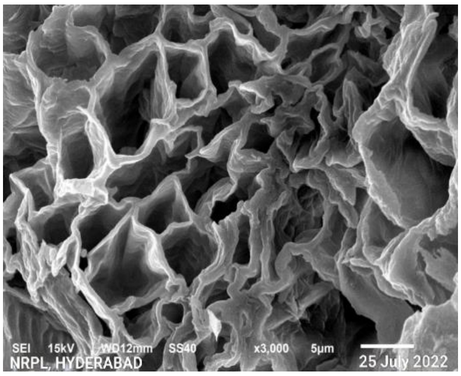

Fig. 7a: SEM image of MIFP adsorbent

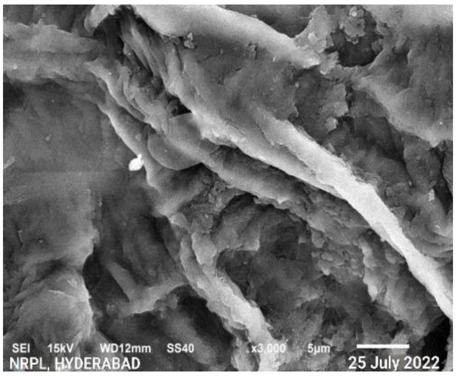

Fig. 7b: SEM image of MB-loaded MIFP

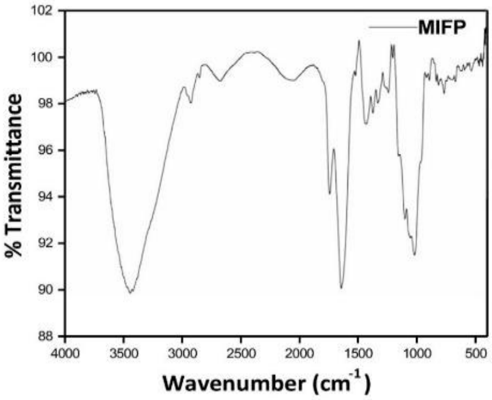

Fig. 8a: FTIR spectrum of MIFP adsorbent

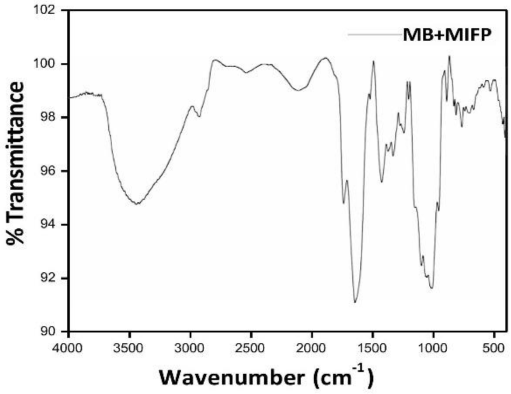

Fig. 8b: FTIR spectrum of MB-loaded MIFP

Material characterization: The morphological characteristics of MIFP adsorbent before and after adsorption with MB dye were analyzed by Scanning electron microscopy and the micrographs are presented in figures 7a and b. It can be seen in the micrograph that the surface of MIFP adsorbent is highly irregular with cracks and crevices. But after adsorption, the pores of MIFP adsorbent were filled with MB (Fig. 7b) dye molecules.

Presence of different functional groups was determined by employing FTIR spectrophotometric method. The FTIR spectra of MIFP before and after adsorption with MB are shown in figures 8a and b respectively. The band at 3442 cm^{-1} might be due to O–H stretching of carboxyl group. The band at 2925 cm^{-1} might be due to antisymmetric and symmetric stretching of C–H bond of methyl and methylene groups. Sharp band at 1742 cm^{-1} is due to C=O stretching and strong band at 1643 cm^{-1} may be attributed to C=O stretching of carboxylic acid. In MB-loaded MIFP, the decrease in peak intensity at 3442 cm^{-1} and 1643 cm^{-1} indicates the involvement of hydroxyl group and carboxyl group respectively, in binding with MB during adsorption. Minor changes in other frequencies have also been observed.

Conclusion

In the present study, the efficiency of MIFP as an adsorbent was investigated. It was found that adsorption is affected by various parameters like contact time, initial dye concentration, adsorbent dosage, pH and temperature. The equilibrium time for the adsorption of MB onto MIFP was found to be 3 hours. The adsorption study also revealed that the optimum dosage for MB-MIFP system was 2.0 g/L. In acidic conditions, there is a drastic increase in uptake of dye up to pH = 4.0 for MB.

Thereafter, no significant change was observed. Kinetic study showed that the adsorptive removal by MIFP follows pseudo-second order kinetics. Adsorption isotherm study indicated that the equilibrium data were in agreement with Langmuir model for MB-MIFP. Consequently, it was concluded that MIFP is an effective adsorbent for the removal of MB dye from aqueous solution.

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