Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2017.0215

58 (2017) 267–273
January

Removal of methylene blue on soil: an alternative to clay

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Received 21 February 2016; Accepted 29 May 2016

ABSTRACT

The present study exploits the adsorption capacity of locally available soil from Rajasthan, India. Removal of methylene blue (MB), a cationic dye, from aqueous solution by the batch adsorption technique under different conditions of initial dye concentration, adsorbent concentration, contact time and solution pH was studied. In order to analyse the chemical composition of soil, zeta potentials, FTIR and XRF were conducted. The results showed that the adsorption reached to equilibrium within 10 min of contact time. It was found that the amount of methylene blue adsorbed per unit mass of adsorbent dose decreases with increasing adsorbent dosage but increases with the increase in initial dye concentration. Adsorption capacity increases with increasing pH in a range of 9 to 11. The equilibrium data for methylene blue adsorption well fitted to Freundlich equation. The results indicate that locally available soil from Rajasthan, India, could be employed as a low cost alternative in textile wastewater treatment for removal of methylene blue dye.

Keywords: Dyes; Soil; Clay; Textile effluent; Low cost adsorbent

1. Introduction

Water contamination from dyeing and finishing in the textile industry is a major concern. The colour produced by the synthetic dyes not only harms the aesthetic nature of the environment but these dyes are also toxic to aquatic life also [1,2]. Therefore, removal of these dyes from wastewater is indispensible. In recent years, stricter directives coupled with increased enforcement concerning wastewater discharges have been established in many countries. To meet the effluent discharge limits set by the legislation, various biological and chemical methods have been employed. However, non-biodegradable nature of most of the dyes makes them difficult to treat [3].

Adsorption is known to be a promising technique to produce good quality effluents. The ease of operation and comparable low cost of application in decolouration process [4] makes this process more suitable for small-scale industries which cannot afford to treat their wastes due to their limited resources [5].

Although activated carbons have been most widely used for the adsorption of dyes, clay minerals have been increasingly gaining attention because they are cheaper than activated carbons and they usually have chemical and mechanical stability, high surface area and structural properties [4,6]. Presence of net negative charge and exchangeable cation on their surface make them more efficient for colour removal. A number of studies have been reported on clay and various clay minerals for MB adsorption from its aqueous solution such as montmorillonite clay [2], kaolinite [7], bentonite [8], etc.. However, its low particle size creates separation/filtration problems after adsorption [9].

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1944-3994/1944-3986

Table 1
Adsorption capacity and particle size of various clays

Adsorbent	Particle size	Adsorption capacity	Dye	Reference
Montmorillonite clay	53–105 μm	86.32–348.89 mgg^{-1}	Methylene blue	[2]
Organo-attapulgite	<200 μm	189.39 mgg^{-1}	Congo red	[4]
Calcined pure kaolin	<230 μm	8.88 mgg^{-1}	Methylene blue	[7]
Calcined raw kaolin	<230 μm	7.59 mgg^{-1}	Methylene blue	[7]
Bentonite	80–125 μm	500 mgg^{-1}	Methylene blue	[10]
Acid-Activated	<63 μm	416.3 mgg^{-1}	AR57	[11]
Bentonite		119.1 mgg^{-1}	AB294	
Activated clay	<38 μm	208–54.3 mgg^{-1}	BR81	[12]
Activated clay	<38 μm	7.29–58.2 mgg^{-1}	AB9	[12]
Clay	<= 53 μm	6.93 mgg^{-1}	Methylene blue	[13]

From the Table 1, the competence of various clays for dye removal is evident. Therefore, the clays in soil can also act as a natural scavenger in removing colour from the textile effluents. Since Rajasthan has leading position in printing and dyeing of low cost, low weight fabric, it generates a substantial amount of textile wastewater. As the State has an abundance of soil, this study was undertaken to assess the potential of adsorption capacity of locally available soil in removal of dyes, if any. This paper reports the removal of methylene blue (MB) from aqueous solution onto locally available soil from Rajasthan, India.,

Some of the reported studies show that illite is the dominant clay mineral with about ten percent kaolinite in the arid region of Rajasthan. The geomorphic features of Rajasthan area are the result of both fluvial and eolian action. The geology is further influenced by the Aravalli range [14]. These soils are found in slopes along the foothills of the Aravalli ranges. They are reddish to yellowish red in colour and sandy loam to loamy sand in texture [15].

The data above in Table 2 show that soils of Rajasthan contain convincingly good amount of clay minerals. Considering this characteristic of the soil, the present study for MB removal by adsorption on local soil was undertaken. The MB dye removal was examined as a function of adsorbate concentration, adsorbent concentration, contact time, solution pH and adsorbent particle size.

Table 2
Mineralogical content of sandy soil

Mineral	In percentage [14]
Montmorillonite	30
Chlorite	22
Illite	26
Kaolinite	15
Quartz+Felspar	11
Surface Area (m^2/g)	398
CEC (meq %)	44.3

2. Materials & methods

2.1. Adsorbate

MB (basic dye, C.I. 52015; chemical formula: $C_{16}H_{18}ClN_3S{\cdot}3H_2O$; MW: 373.90 $gmol^{-1}$) was used as the adsorbate in this study. It was used without further purification. The chemical structure of MB is shown in Fig.1. This structure shows decentralized positive charge on the organic framework, which could play a major role in keeping the dye species on the surface of the soil. It has a maximum visible absorbance at a wavelength of 663 nm. MB stock solution was prepared by dissolving an accurately weighed amount of MB in distilled water to achieve a concentration of 500 mgL^{-1}, and subsequently diluted to the required concentrations. All chemicals used in this study were of analytical-laboratory grade, being purchased from Merck.

2.2. Adsorbent

Adsorbent used for the present study is locally available sandy soil. It was collected from the premises of Malaviya National Institute of Technology, Jaipur. The collected soil was then washed and oven dried at 100°C. The dried soil was crushed and sieved to give particle sizes in the range of 300–150 μm and 150–75 μm using ASTM Standard sieves.

2.3. Characterization

Chemical analysis of the soil was carried out using X-ray fluorescence spectrometer (PANalytical, PW 2404) as shown

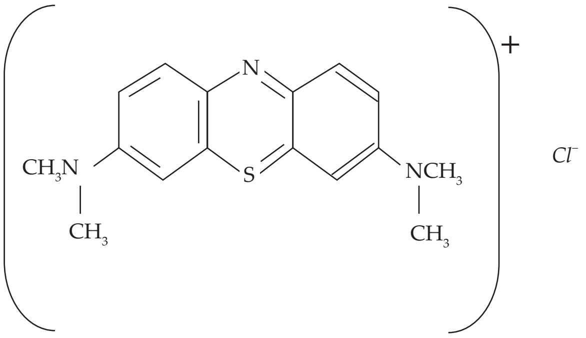


Fig. 1. The chemical structure of methylene blue.

in Table 3. Zeta potential measurements (Fig. 2) were made using Malvern Zetasizer Nano ZSP (ZEN5600). Infrared spectroscopic measurements were done on Perkin Elmer FT-IR Spectrum2 in the spectral range of 4,000–400 cm^{-1}using KBr pellet method (Fig. 3(a) & 3(b)). Particle size distribution was determined by Malvern Mastersizer 2000E (Fig. 9).

2.4. Adsorption studies

The batch tests were carried out in 500 ml capped-Erlenmeyer flasks. A given amount of soil was added with 100 ml of dye solution to the flask. The contents of the flasks were then agitated at a constant speed of 150 rpm for one hour in incubator shaker (Model MSW-232) at 30°C. At regular intervals, an accurately measured content was taken out and centrifuged in a centrifuge (Model Remi R8C-BL) at 2,000 rpm for 20 min. The supernatant solution was pipetted out and monitored instantaneously on a spectrophotometer (Model UV-1800 Shimadzu UV-Vis Spectrophotometer) for absorption values. The influence of pH (1, 3, 5, 7, 9, 11), time (3, 6, 10, 20, 30, 40, 50, 60 min) adsorbent dose (12.5, 25, 50, 75, 100 g) and initial dye concentration (125, 250, 500, 1000 mg/l) on dye removal was studied.

Table 3
Composition of soil

Component	Weight (%)
SiO^2	15.86
Al_2O^3	3.72
Fe_2O^3	2.33
CaO	0.73
MgO	0.13
Na_2O	0.65
K_2O	0.53

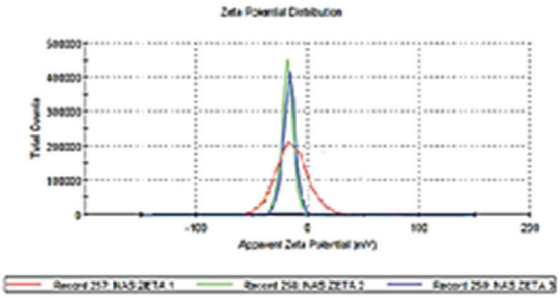


Fig. 2. Zeta potential distribution on soil.

2.5. Calculation

The percent removal (%) and the amount of MB (q_t) adsorbed by the soil in each time interval t, was calculated using the following equation:

$$\text{Removal (\%)} = \left[\frac{(C_o - C_t}{C_o}\right] \times 100 \tag{1}$$

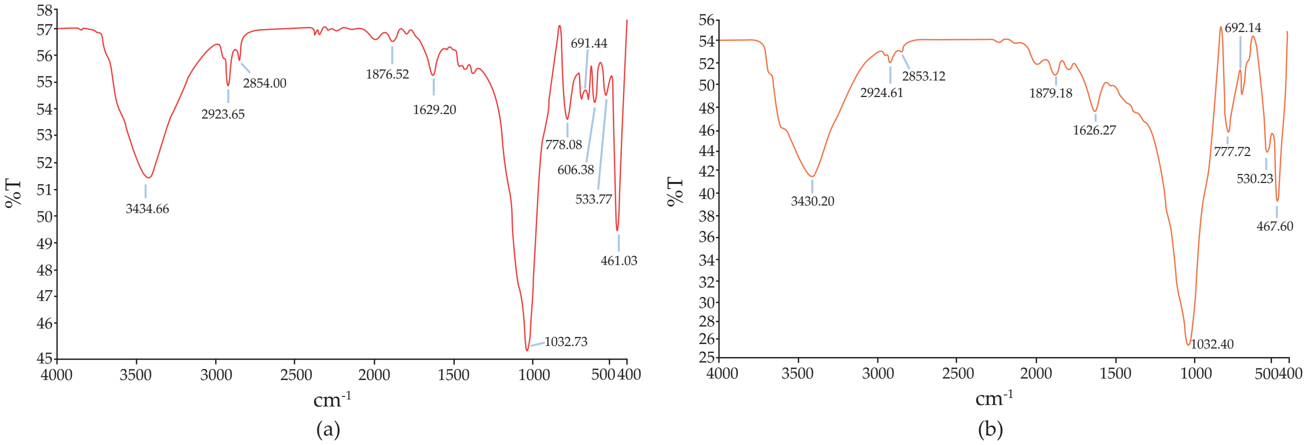
$$qt = (C_o - C_t)\frac{v}{m} \tag{2}$$

where q_t (mgg^{-1}) is the amount of MB adsorbed per unit weight of soil at any time t; C_o and C_t are, respectively, the initial and liquid-phase concentrations of the dye solution at any time t (mgL^{-1}); V is the volume of the MB solution (0.1 L); m is the mass of the soil. For the experiments with varying soil concentrations, the amount of soil was in the range of 12.5 g to 100 g.

3. Results and discussion

3.1. Characterization

Table 3, illustrates that SiO_2 and Al_2O_3 are the major oxide constituents of the soil, which are also the major constituents of clay [2], with other oxides being present in much smaller fractions. Hence, it can be anticipated that either the major oxides present in the adsorbent or their combined influence is mainly responsible for the adsorption of MB. It can be seen from the Fig. 2, that the presence of negatively charged surface lattice (–17 mV), which is again the property of clay [4], provides a greater capacity to adsorb a cationic dye, MB. Fig. 3(a) and (b) shows the infrared spectra of soil. It helps



(a)

(b)

Fig. 3. Infrared spectra of (a) soil and (b) MB adsorbed soil.

in the identification of functional groups on the surface of the adsorbents which are responsible for adsorption of dye molecule [16]. The peak at 3434.66 cm^{-1} is assigned to stretching vibration of hydrogen bonded silanol group υ(≡Si–OH) and υ(–OH) of physisorbed water molecule [17]. A pair of strong bands at 2854.00 and 2923.65 cm^{-1} can be assigned to the symmetric and asymmetric stretching vibrations of the methylene group ($-CH_3$) [18]. The peak at 1629.20 cm^{-1} is attributed to O–H bending vibration because the deep band at 3434.66 cm^{-1} suggests the presence of some interlamellar water [17]. The strong band observed at 1876.52 cm^{-1} and 1033.8 cm^{-1} indicated the stretching of the many C–OH and C–O–C bonds [19].

The bands appearing between 800 and 500 cm^{-1} are associated with the tetrahedral vibrations formed by what are known as secondary building units (SBU) and fragments of the alumino silicate system. These bands are typical characteristic of the double or single rings (depending on the structure of the material) and/or the TO_4 (T = Si, Al) tetrahedral bonds [20]. The adsorption peaks at 778.08 cm^{-1} and 606.38 cm^{-1} lies in the region of stretching vibration for Si–O symmetric and asymmetric bond vibration respectively [16,21]. Absorption bands at 521.27 cm^{-1}, 693.91 cm^{-1} are related to the bending vibration of Si–O group in asymmetric and symmetric vibration [16]. The band at 461.03 cm^{-1} is due to Si–O–Al (octahedral) and Si–O–Si bending vibration [22,20].

Thus, the result from the FTIR data supports the presence of negative charge on the soil surface which would help in the fixation of cationic dye.

On comparing spectrum Fig. 3(a) of soil with the spectrum Fig. 3(b) of MB adsorbed soil, it could be seen that there are small shifts in some bands (3430.20 cm^{-1} and 1626.27 cm^{-1}) and some bands disappeared (606.38 cm^{-1}). The intensity of the some peaks was minimised (1879.18 cm^{-1}, 1032.40 cm^{-1}, 692.14 cm^{-1}, 530.2 cm^{-1}, 467.60 cm^{-1} and 777.72 cm^{-1}) as shown in the Fig. 3. These changes observed in the spectrum indicated the possible involvement of these functional groups on the surface of soil in adsorption process.

3.2. Effect of contact time

The contact time between adsorbate and the adsorbent is of significant importance in the wastewater treatment by adsorption. A rapid uptake of adsorbates and establishment of equilibrium in a short period indicate the efficiency of the adsorbent for its use in wastewater treatment. In physical adsorption, most of the adsorbate species get fixed on the adsorbent within a short interval of contact time. However, strong chemical binding of the adsorbate with adsorbent requires a longer contact time to attain the equilibrium. The uptake of adsorbate species is fast at the initial stages of the contact period, and thereafter, it becomes slower near the equilibrium. In between these two stages of the uptake, the rate of adsorption is found to be nearly constant [24]. The results for the effect of contact time on adsorption of MB onto soil given in the Fig. 4 are for initial dye concentration 1g/l at 30°C and solution pH 11. The graph shows two adsorbent doses (100 g and 12.5 g); however, all the five adsorbent doses were studied. Dye solution was kept in contact with soil for 60 min and MB removal was checked at regular intervals of time. The contact time curve from Fig. 4, shows that MB uptake was rapid in first 10 min and then it becomes almost constant. This could possibly be because a large number of vacant surface sites are available for adsorption during the initial stage, and after adsorption, the remaining vacant surface sites may be difficult to be occupied due to repulsive forces between the solute molecules on the solid and bulk phases [24]. The percent removal increased from 38% to 81% with increase in adsorbent dose from 12.5 g to 100 g (not shown). However, the adsorption capacity decreased from 3.04 mgg^{-1} to 0.81 mgg^{-1} with increase in adsorbent concentration (Fig. 4).The noteworthy feature here is the speed with which adsorption took place at both the adsorption doses. This feature was common for all the adsorbent doses. Since 100 g of adsorbent dose gave lesser amount of MB removal than 12.5 g of adsorbent dose, it could be said that saturation of the adsorbent could be achieved over 60 min of the contact time. The reports indicate that a relatively larger contact time is required for the adsorption of MB with classical adsorbents like sepiolite? [24], montmorillonite clay [2], etc. and it is one of the most difficult dyes to remove from aqueous solutions. Thus, the high affinity shown by the local soil for its adsorption can result in a very good low cost alternative for treating the wastewater containing MB. Since a contact time of 10 min can be considered as optimum time for MB adsorption, so further tests in the next sections were performed at this contact time only.

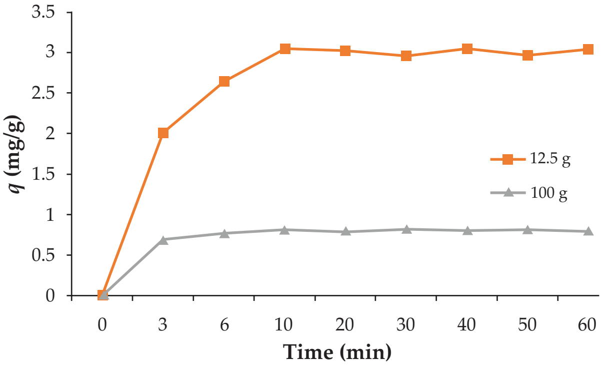


Fig. 4. Effect of contact time on MB adsorption onto adsorbent (pH: 11, adsorbent concentration: 12.5 g & 100 g, MB concentration: 1 g/l).

3.3. Effect of pH

The pH is an important parameter controlling the quantity of the dye fixed on the adsorbent. Experiments in a broad range of pH values have been carried out in order to examine the behaviour of adsorption of the dye with respect to various ionic forms present in solution [25]. Fig. 5 illustrates the effect of pH on adsorption of MB onto the soil. MB dissociates into molecular cations when dissolved in aqueous solution. The negative surface charge on the soil influences the adsorption of the dissociated cationic MB molecules. As can be seen from the Fig. 5, adsorption of the dye increases as the pH increases and maximum adsorption can be observed at pH 11. The increasing pH causes deprotonation of silica ($Si-O^-$) and silanol (Si–OH) groups on the soil surface. This results in more number

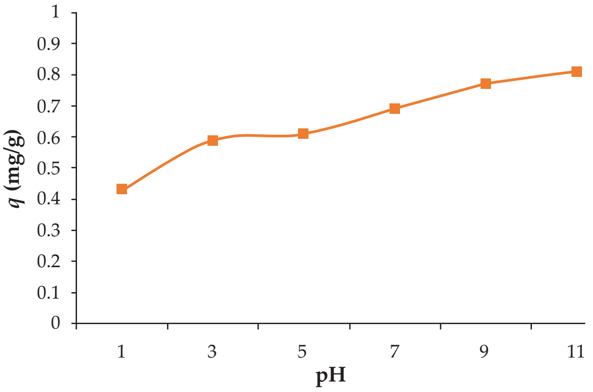


Fig. 5. Effect of pH on MB adsorption onto adsorbent (contact time: 10 min, adsorbent concentration: 100 g, MB concentration: 1 g/l).

of negatively charged adsorbent sites. At acidic pH, protonation of silanol groups to Si–OH$_2^+$occurs, which do not favour adsorption of the cationic dye due to electrostatic repulsion. Also, H$^+$ ions compete with positively charged MB dye for adsorption sites [2,4]. Since pH 11 can be considered as the optimum pH for the MB dye removal, in the next sections of the present study this pH was used for further tests.

3.4. Effect of MB concentration

The more concentrated the dye solution, the higher the adsorption capacity. This is probably due to a high driving force for mass transfer in high dye concentration [4]. Equilibrium adsorption studies were conducted to define the adsorption competence of the adsorbent. The effect of initial MB concentration on the adsorption onto soil in terms of amount adsorbed per unit mass of adsorbent is shown in Fig. 6. It could be seen from the graph that the amount of MB adsorbed onto the soil increased as the initial dye concentration increased. This could be because at lower MB concentration, the ratio of the initial number of dye molecules to the available surface area was low. However, at high concentration, large number of dye molecules came in contact with the available adsorption sites, hence, higher number of dye molecules gets adsorbed [23].

3.5. Effect of adsorbent concentration

The dependence of the MB dye adsorption on soil (300 μm–150 μm) concentration was studied at 30°C, 10 min contact time and pH of 11by varying the soil doses from 12.5 g to 100 g while keeping the dye solution constant (1 g/l). The results are shown in Fig. 7. The percentage removal of dye increased from 38% to 81% (not shown) with the increase in adsorbent dose from 12.5 g to 100 g, whereas the MB uptake decreased as the per unit mass of the adsorbent dose increased (Fig. 7). The plausible reason for this observation could be that though with increased dose of adsorbent, there is a greater presence of exchangeable/adsorption sites a higher fraction of the adsorbent sites remains unsaturated during the adsorption process [26]. Similar results have been reported for the adsorption of MB onto clay by other researchers [26–28].

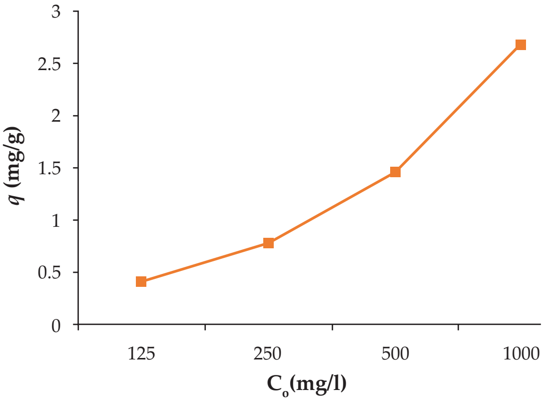


Fig. 6. Effect of MB concentration on the adsorption onto adsorbent (contact time: 10 min, pH: 11, adsorbent concentration: 25 g).

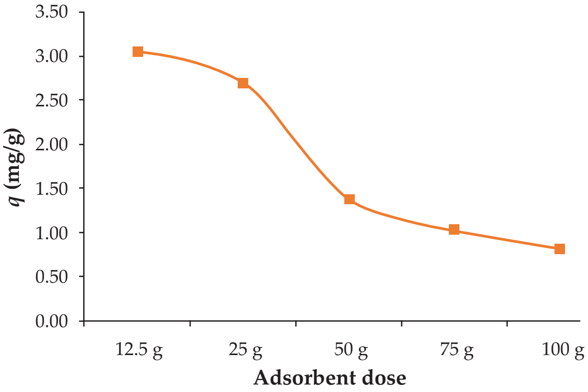


Fig. 7. Effect of adsorbent concentration on MB adsorption (contact time: 10 min, pH: 11, MB concentration: 1 g/l).

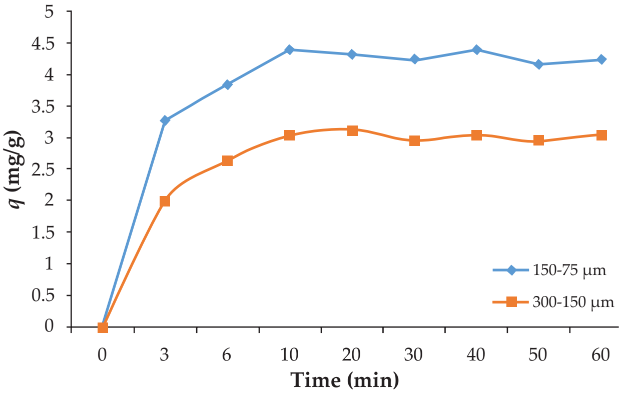


Fig. 8. Effect of particle size of adsorbent on MB adsorption (contact time: 10 min, pH: 11, MB concentration: 1 g/l, adsorbent concentration: 12.5 g).

3.6. Effect of particle size

Fig. 8 shows the effect of the particle size of soil on MB adsorption. Two different particle size ranges were analysed for this purpose. When the particle size range was decreased from 300–150 μm to 150–75 μm, the adsorption capacity of soil was increased from 3.04 mgg^{-1} to 4.4 mgg^{-1}. This is

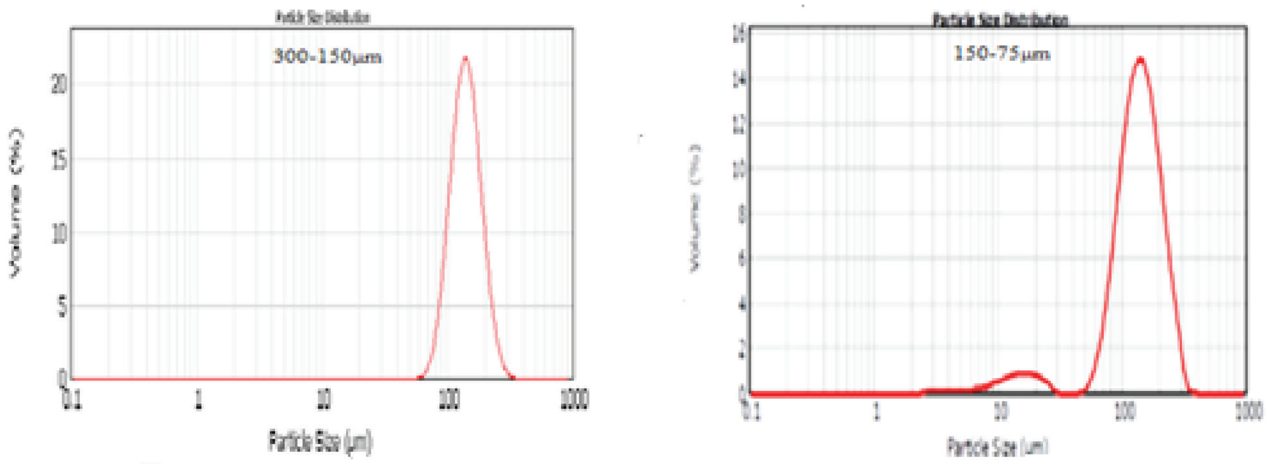


Fig. 9. Particle size distribution curve of soil.

because the external surface increases as the particle size reduces resulting in the exposure of more active sites for adsorption. This increase by almost 50% was substantial and therefore, particle size 150–75 µm is recommended for higher adsorption capacity. Fig. 9 and Table 4 represent the particle size distribution curve. It is important to note that this size range is coarser than the clay sizes used by other researchers earlier (Table 1), which is prone to early clogging of the adsorbent bed.

3.7. Equilibrium adsorption isotherms

Equilibrium data are important for designing any adsorption system. The equilibrium data were analysed using two well-known isotherms; Langmuir and Freundlich. 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 (MB) in the plane of surface [31].

Table 4
Particle size distribution of soil

	300–150 µm	150–75 µm
Uniformity	0.2	0.35
Specific surface area	0.0441 m^2/g	0.0783 m^2/g
Surface weighted mean	135.948 µm	76.655 µm
Volume weighted mean	146.194 µm	140.556 µm
D(0.1)	99.690 µm	74.173 µm
D(0.5)	140.532 µm	135.622 µm
D(0.9)	200.237 µm	122.907 µm

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \left(\frac{1}{Q_o}\right) C_e \tag{3}$$

where, *Ce* is the equilibrium concentration of the adsorbate (MB) (mg/l), *qe* the amount of adsorbate adsorbed per unit mass of adsorbate (mgg^{-1}), and Q_o and *b* are Langmuir constants related to adsorption capacity and rate of adsorption, respectively. The Langmuir constants '*b*' and 'Q_o' can be obtained from the plot C_e/q_e versus C_e.

Freundlich isotherm model assumes heterogeneous surface energies, in which the energy term in Langmuir equation varies on a function of the surface coverage.

$$\log q_e = \log K_f + \left(\frac{1}{n}\right)\log C_e \tag{4}$$

where q_e is the amount of MB adsorbed at equilibrium (mgg^{-1}), C_e is the equilibrium concentration of the adsorbate (MB) and K_f and *n* are Freundlich constants, *n* giving an indication of how favourable the adsorption process and K_f ($mgg^{-1}(lmg^{-1})^n$) is the adsorption capacity of the adsorbent. K_f can be defined as the adsorption or distribution coefficient and represents the quantity of MB onto soil for a unit equilibrium concentration. The slope $1/n$ ranging between 0 and 1 is a measure of adsorption intensity or surface heterogeneity, becoming more heterogeneous as its value gets closer to zero. A value for $1/n$ below one indicates a normal Langmuir isotherm while $1/n$ above one is indicative of cooperative adsorption. The plot of log *qe* versus logC_e gives the Freundlich constants (K_f and *n*) [31].

The applicability of the isotherm equation is compared by judging the correlation coefficient R^2. It was found that

Table 5
Isotherm constants for MB adsorption onto soil

Isotherm	Parameter	Adsorbent dose				
		12.5 g	25 g	50 g	75 g	100 g
Fruendlich	*n*	0.864	0.671	0.649	0.634	0.599
	K_f	9.690	8.023	10.237	12.672	14.849
	R^2	0.999	0.994	0.999	0.999	0.996

Langmuir model gave R^2 values 0.9622, 0.9847, 0.9369, 0.8811, 0.9587 for adsorbent dose 12.5 g, 25 g, 50 g, 75 g, and 100 g, respectively. However, Freundlich model gave somewhat better fit (Table 5), indicating that the adsorption of MB on soil follows the Freundlich isotherm.

4. Conclusion

The present study was carried out to examine the efficacy of soil for the removal of cationic dye, methylene blue from the aqueous solution. As the sandy soil of Rajasthan, India, contains clayey minerals as its constituent, this feature was exploited for the present study. FTIR and zeta potential results showed the presence of negative charge on the soil surface, which is also the characteristic of the clayey particles. This charge helped in effective removal of cationic dye, MB. The equilibrium data also fitted well to the Fruendlich equation. This study showed that 3.04 mgg^{-1} and 4.4 mgg^{-1} of the MB can get fixed onto two different particle sizes of soil within 10 min. From the literature above, the small size of the clay particle makes it difficult to use at filtration/separation stage. However, the adsorbent in the present study showed good colour removal at reasonably larger size than clay minerals. Thus, it can be suggested that this locally available soil can be used for the removal of cationic dyes as a low cost alternative to the classical adsorbents.

Acknowledgement

The authors wish to thank RSPCB for financial assistance. Material Research Centre at MNIT, Jaipur, and SAIF at IIT, Bombay for chemical analysis of the soil sample.

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