

Adsorption and Kinetic Studies on the Removal of Methyl Red from Aqueous Solutions Using Low-cost Adsorbent: Guar Gum Powder

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Abstract—In this paper the potential of Guar gum powder (GG) a low cost and eco-friendly biosorbent was characterized and investigated for the removal of Methyl Red dye (MR) from an aqueous solution. The adsorption behavior of the biosorbent was investigated by performing both kinetic and equilibrium isothermal studies in batch conditions at 34°C. During study, the adsorption conditions for the adsorbent were calculated by changing different experimental parameters i.e. contact time, initial dye concentration, adsorbent dose, particle size and pH of the solution. The adsorption studies was best fit with Langmuir isotherm, and it gives monolayer adsorption capacity of 66.66 mg/g at pH 4.2, and temperature 34°C. The correlation coefficient value indicate a moderate fit for monolayer Langmuir model ($R^2 = 0.99$). The q_e experimental and calculated values for the pseudo-second-order kinetic model were in good agreement as compared to that by pseudo first order kinetics. The structural characterization of the GG was done by scanning electron microscopy (SEM).

Index Terms— Adsorption, Dye, Guar Gum Powder, Isothermal studies, Parameters, SEM, Kinetics

1. INTRODUCTION

In Rajasthan the textile industry is a growing industry. Various dyes have been used extensively in various branches of the textile industry. It is reported that more than 100,000 commercially dyes are available and approx 7×10^5 metric tonnes are produced per year². Most of the dyes are complex organic molecules and show resistance towards weather, action of detergents, etc.³. Many industries use dyes in order to colour their products and also consume substantial volumes of water⁴. A considerable amount of coloured wastewater is generated due to dyeing process through textile industries. The presence of very small amount of dyes in water (less than 1 ppm for some dyes) is highly visible and undesirable⁵. Dyes have been reported to cause carcinogenesis, mutagenesis, chromosomal fractures, teratogenicity and respiratory toxicity⁶. Azo dyes show good solubility nature and is one of the common water pollutants which may frequently be found in trace quantities in industrial wastewater. Due to increasingly stringent restrictions on the organic content of industrial effluents, it is necessary to eliminate these dyes from wastewater before it is discharged⁷.

The colour removal from effluent water had been extensively studied using different methods such as flocculation and coagulation⁸, ultra and nano filtration⁹, photo oxidation¹⁰, activated carbon¹¹ etc. which are quite expensive^{12,13}. Recently various attempts have been made by using green approach for the removal of dyes from wastewater using adsorption process¹⁴. Over the last few years adsorption has gained prime importance in industry and environment protection¹⁵. Different

adsorbents show environment friendly behavior, abundant in nature and are very much-cost effective and so is normally used in the process of adsorption¹⁶. Removal of dyes by using these bio-adsorbents follows the mechanism of reaction with proteinaceous cellular material¹⁷. Due to high affinity of the adsorbent for the adsorbate species, the latter is attracted and bound thereby different mechanism. The degree of adsorbent affinity for the adsorbate determines its distribution between the solid and liquid phases. Various sorbents had already been used for removal of dyes like mango peel¹⁸, banana peel¹⁹, coconut bunch waste²⁰, wheat husk²¹, tree bark²², orange peel²³, chitosan²⁴ etc.

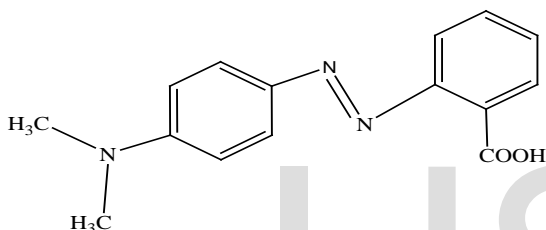
In the present study, attempts have been made to determine adsorption efficiency of one of the low cost biosorbent as Guar gum powder (GG) for removal of Methyl Red (MR) dye from aqueous solutions. The Guar or cluster bean (*Cyamopsis tetragonoloba*) is a source of guar gum and is also known as Gavar, Guwar or Guvar bean. Guar gum has wide applications in pharmaceutical like cosmetic, food, textile, paper, explosive and toiletries industries etc.²⁵. About 2.5- 3.5 million tons of guar is produced by India (mainly Rajasthan, Gujarat, and Haryana) and it makes it the largest producer with about 80% of world production²⁶. The objective of present study was to analyze the percentage of methyl red dye removal from aqueous solutions using guar gum powder, as a low cost biosorbent.

2. MATERIALS AND METHODS

The instruments used in this work include Spectrophotometer(Digital UV Spectrophotometer, Model UV 2300,Chemito Make),Digital pH meter(Electronic India ,EI Model 112),Digital electronic weighing balance(Citizen, Model no.100 C),Oven(220/230 AC supply, Tanco,QS 9001; 2008,WHO,GMP),Standard Test sieve(MSECO Maharani Scientific and Engineering Cooperation, Delhi),Orbital Shaker (Remi Instrument Ltd., IHB, 597), conical flask, burette, pipette, stop watch.

2.1. Adsorbate

All chemicals used were of analytical grade and solutions were prepared in doubly distilled water. The azo dye used in this study was Methyl Red of Merck Limited.The maximum absorption wavelength of MR dye is 525 nm at pH 4.2.The structure of MR is shown in Scheme 1.The physical and chemical characterization of dye is given in Table 1.



Scheme 1.Chemical Structure of methyl red

Table 1.Physical and Chemical properties of dye (MR)

Character	MR dye
Physical State	Solid
Melting Point	180 °C
Molecular weight	269.30 gm/mole
Chemical Formula	C ₁₅ H ₁₅ N ₃ O ₂
λ_{max}	525 nm
pH	4.2

2.2. Preparation and characterization of adsorbent

The guar seeds collected from the local vegetable market Jaipur, Rajasthan (India). These guar seeds were than washed with distilled water to remove dust and water soluble materials. Wetted guar seeds wiped and dried in sunlight for 4-5 hours per day up to three to four days. After crushing, seeds are converting into guar splits. It was then grinded unless smooth, fine guar gum (GG) powder obtained. No other chemical or physical treatment was used prior to adsorption experiments. Scanning electron microscopy (SEM) analysis was carried out to study its surface texture before and after MR dye adsorption.

2.3. Adsorption Studies

The batch sorption experiments were carried out in 250 ml Erlenmeyer flask where 0.10 g of the adsorbent and 30 ml of MR dye solution (100-500 ppm) at a pH 4.2 were added at 34°C. The pH of solution was adjusted by using 1N HCl and 1N NaOH. The Erlenmeyer flask were capped and agitated in an isothermal shaker at 110 rpm at 34°C for 4 hours to achieve equilibrium. The concentration of the dye in the solution after equilibrium adsorption was determined by UV-Visible spectrophotometer at 525 nm at pH 4.2. The amount of adsorption at equilibrium, q_e (mg/g), was calculated by Eq.(1)

$$q_e = \frac{(C_0 - C_e)V}{W} \quad (1)$$

Where C_0 and C_e (mg/L) are the liquid -phase concentration of dye at initial and equilibrium state respectively. V is the volume of the solution (L) and W is the mass of dry adsorbent used in grams (g).

Kinetic studies of adsorption were also carried out at various concentrations of the MR and the extent of adsorption was investigated as a function of time. The amount of adsorption at time t , q_t (mg/g), was calculated by Eq.(2)

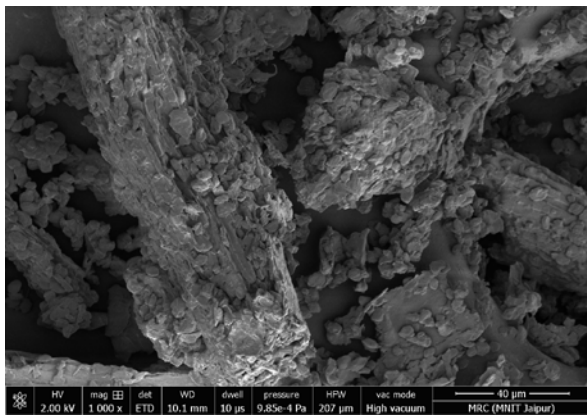
$$q_t = \frac{(C_0 - C_t)V}{W} \quad (2)$$

The adsorption studies were studied by various isotherms.

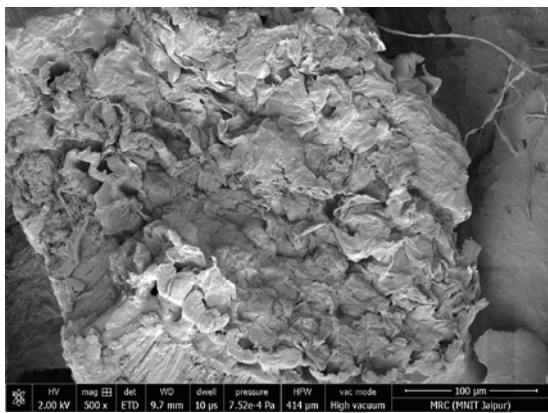
3. RESULTS AND DISCUSSION

3.1 Characterization of sorbent

Surface morphology of GG was determined by SEM. Fig.1 (a) and (b) shows the SEM micrographs of GG sample before and after adsorption of MR. The micrograph of GG shows uneven, cave like rough surface morphology. The surface of dye loaded adsorbent indicates that some of the surface of GG is covered with dye molecules and play important role in adsorption. The changes in the images show the possible involvement of rough surface during adsorption.



(A)



(B)

Fig.1 Scanning electron microscope of (a) Fresh GG and (b) MR adsorbed GG

3.2 Effect of initial dye concentration on dye adsorption

The effect of MR dye concentration (100 to 500 ppm) was studied by adding adsorbate of 0.10g at mixing rate 110 rpm. The effect was shown in Fig.2. It can be seen that the adsorption at different concentration is gradually decreases with the progress of adsorption until the equilibrium is reached. The amount of MR dye adsorbed at equilibrium q_e increased from 22.5 to 57.6 mg/g as the concentration of dye was increased from 100 to 500 ppm. Hence a higher initial concentration of dye will enhance the adsorption process. The MR removal decreased from 70% to 37.8% as the MR concentration was increased from 100 to 500 ppm. The equilibrium conditions were reached within 3 hour. Fig. 3 shows the plot between initial dye concentration and the percentage of dye removed. The negative slope of the plot indicates that the dye removed by the GG powder decreases with increasing concentration of the dye in an aqueous solution.

Similar results were shown by Paul Egwuonwu²² while studying the removal of MR by Neem bark powder (NBP). In the case of NBP, an increase in initial dye concentration from $1 \times 10^{-3} \text{ mol/dm}^3$ to $5 \times 10^{-3} \text{ mol/dm}^3$ leads to a decrease in the percentage of MR removal.

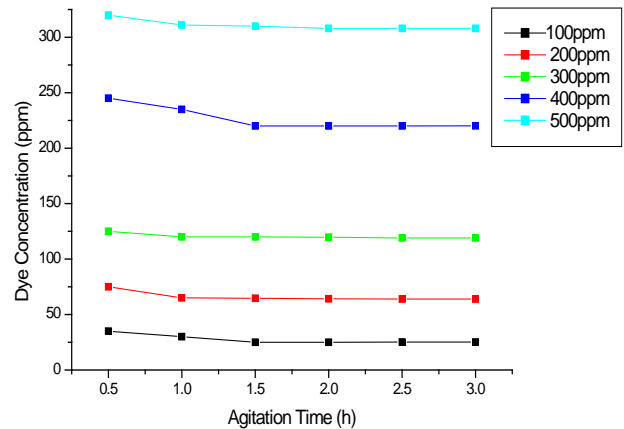


Fig.2 Effect of initial dye concentration on removal of MR by GG (conditions: sorbent dosage, 0.10 g; initial pH, 4.2; temperature, 34°C; particle size, 500 μ ; stirring rate, 110 rpm; V, 30 ml)

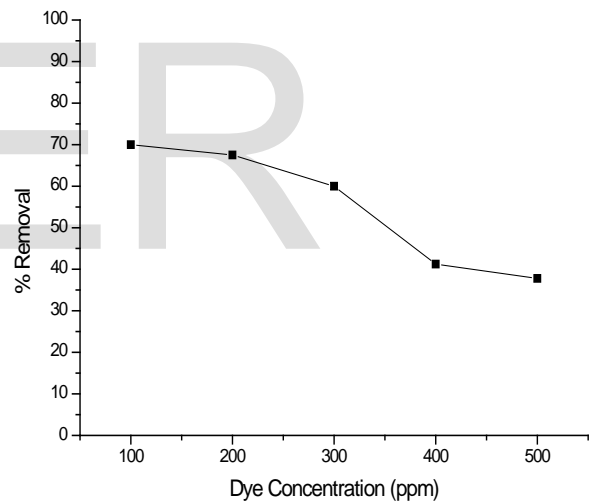


Fig.3 Percentage removal of MR by GG (conditions: sorbent dosage, 0.10 g; initial pH, 4.2; temperature, 34°C; particle size, 500 μ ; stirring rate, 110 rpm; V, 30 ml; dye concentration, 100-500 ppm)

3.3 Effect of pH on dye adsorption

Effect of pH on adsorption was studied using 100 ppm dye concentration, pH 2 -10 at 34°C as given in Fig. 4. MR removal increased from 5% to 85% as pH increases from 2 to 10. The lowest dye adsorption q_e was found to increase with increasing pH, it increases from 15 to 25.5 mg/g for an increase of pH from 2 to 10. It is found that this lower adsorption at acidic pH is probably due to presence of excess H^+ ions which are competing with cation groups on the dye adsorption sites. At higher pH, the surface of GG particles may get negatively charged, which enhance the positively charged dye cation through electrostatic forces of

attraction²⁷. This further indicates possible involvement of some functional groups of GG in sorption process. The percentage removal was optimum at pH 4.2.

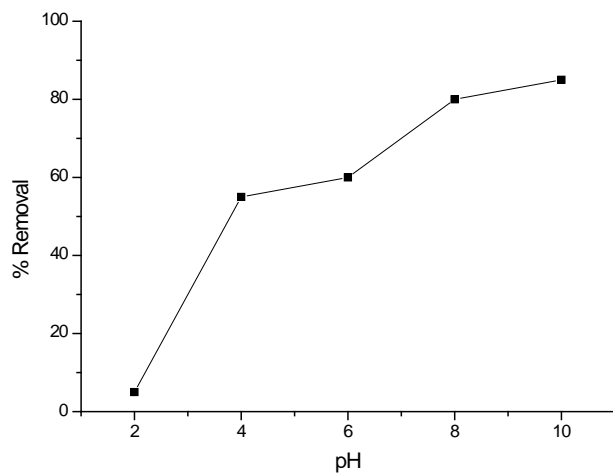


Fig.4 Effect of solution pH on percentage removal of MR GG (conditions: sorbent dosage ,0.10 g; initial pH, 4.2; temperature, 34°C; particle size, 500 μ ; stirring rate, 110 rpm; V, 30 ml; dye concentration, 100ppm)

Similar results were given by Paul Eqwuonwu DIM²². He carried out his work to determine the effect of pH on MR adsorption by NBP and found that the percentage removal of MR by NBP was optimum at pH 6.5 respectively. Similarly results were also reported by H.S.Ashoka¹¹.

3.4 Effect of biosorbent dose on adsorption

The biosorbent dose is also an important parameter because it determines the capacity of a biosorbent for a given initial concentration. To determine the effect of biosorbent dosage on adsorption, dye concentration of 100ppm and samples with different biosorbent dosages ranging from 0.10 to 0.25 g under constant temperature at 34°C and pH 4.2 were taken for 1h. The results shown in Fig.5 indicate that percentage of dye adsorption increased with increase of adsorbent dose. Dye removal of MR increases from 50 % to 76 % with the increase of sorbent dose from 0.05 to 0.20 g. This is mostly due to an increase in the adsorption surface area and the availability of more active and available sites. The plot shows positive slope which indicates that adsorption increases with an increase in adsorption dose. It is clear that a certain amount of dye is adsorbed by a fixed mass of the adsorbent. The increase in sorption dose at constant dye concentration and volume leads to unsaturation of sorption sites through the sorption process.

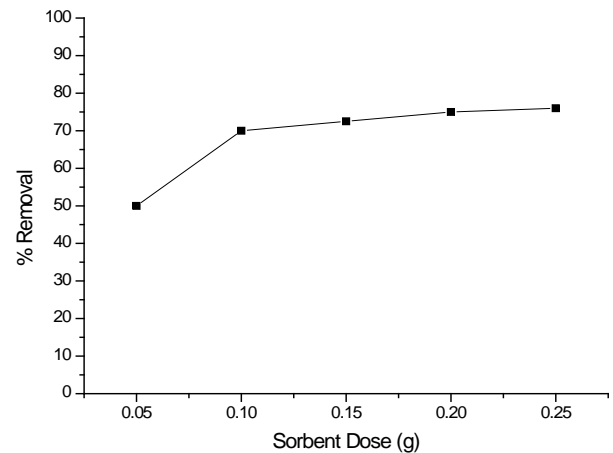


Fig.5 Effect of Biosorbent Dosage(g) on percentage removal of MR by GG (conditions: initial pH, 4.2; temperature, 34°C; particle size, 500 μ ; stirring rate, 110 rpm; V, 30 ml; Dye concentration, 100ppm, time, 1 h)

Similar results are obtained with Rosemal et.al²⁸. As the sorbent dose was increased from 0.4 to 1.2 g/ml, the percentage of MR sorbed on the modified banana trunk fibers was increased. Paul Egwuonwu DIM²² also observed an increase in the percentage removal of MR dye (35.5% to 85.4%) from aqueous solution, by an increase in the adsorbent dose of NBP from 0.5 to 2.5 g/50 ml. Mogaddasi et.al²⁹ also reported similar results in case of removal of methyl orange dye from aqueous solution.

3.5 Effect of Particle size on biosorption

Percentage removal of dye increases on decreasing particle size as shown in Fig.6. On reducing particle size from 500 μ to 150 μ , percentage removal increases from 75% to 90% as more fine powder increases the surface area for adsorption.

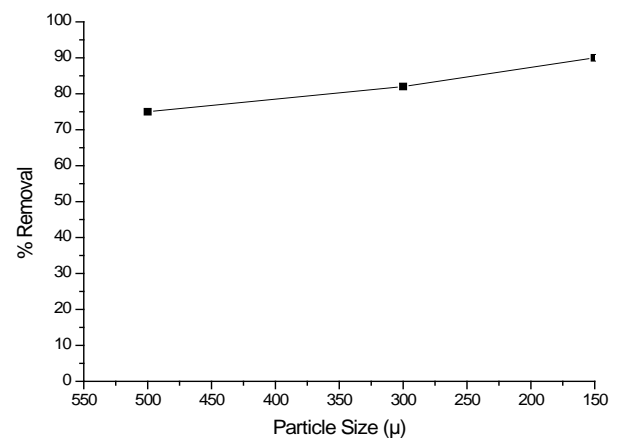


Fig.6 Effect of particle size on percentage removal of MR by GG (conditions: sorbent dosage ,0.10 g; initial pH, 4.2; temperature, 34°C; stirring rate, 110 rpm; V, 30 ml; Dye concentration, 100ppm)

3.6 Effect of Agitation Time on adsorption

Variation in agitation time plays key role in determining the efficiency of the adsorption. Batch equilibrium studies were carried out by adding fixed amount of sorbent 0.10 g to 100 ppm dye solution at 4.2 pH at 34°C. The flasks were agitated in shaker at 110 rpm for 3 h. Aliquots were taken at different constant time (0.5, 1, 1.5, 2, 2.5 and 3 h) and concentration was analyzed. The adsorption capacity increases till the contact time increases up to 2 h and then it attained saturation for 3 h. On increasing agitation time from 0.5 to 3 h, percentage removal of dye increases from 65% to 74.8% as shown in Fig.7. Initially there is an increased rate of percentage removal of MR dye due to availability of number of active binding sites on GG surface. After 2 h no significant adsorption takes place as sorption sites are already occupied and no further sites are available for adsorption. Similar results of effect of agitation time have also been reported by Ruchi Shrivastava¹⁴ and H.S.Ashoka¹¹.

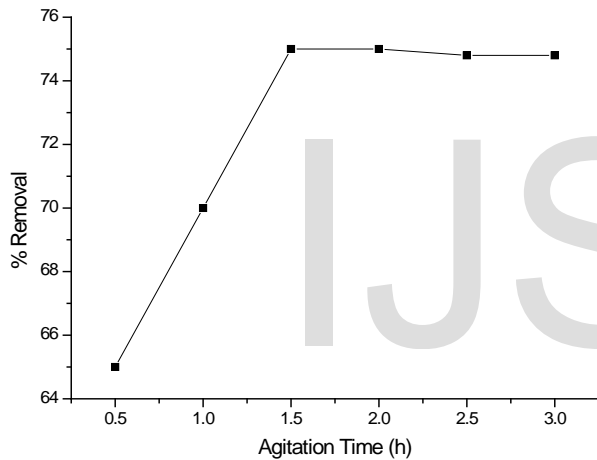


Fig.7 Effect of agitation time on percentage removal of MR by GG (conditions: sorbent dosage ,0.10 g; initial pH, 4.2; temperature,34°C; stirring rate, 110 rpm; V, 30 ml; Dye concentration, 100ppm)

3.7 Adsorption isotherms

The main purpose of studying adsorption isotherms is to relate the adsorbate concentration in the bulk and the adsorbed amount at the interface³⁰. In the present experimental work the isothermal results were analyzed using well known expression for Langmuir, Freundlich and Temkin isotherms. According to Langmuir adsorption model³¹ maximum adsorption corresponds to a saturated mono layer of solute molecules on the adsorbent surface. Expression of the Langmuir model is given by Eq. (3)

$$q_e = \frac{Q_o b C_e}{(1 + b C_e)} \quad (3)$$

Where q_e (mg/g) and C_e (mg/L or ppm) are the amount of adsorbed dye per unit mass of sorbent and unadsorbed dye concentration in solution at equilibrium, respectively. The maximum amount of the dye per unit mass of sorbent to form a complete monolayer on the surface bound at high C_e is denoted as Q_o and b is a constant related to the affinity of the binding sites (L/mg). The Langmuir equation can be described by the linearized form by Eq.(4)

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \quad (4)$$

On plotting a graph between specific adsorption (C_e/q_e) against equilibrium concentration C_e , the linear plot (Fig. 8) is obtained shows that the adsorption obeys the Langmuir model. The values of Langmuir constants Q_o and b were determined from the slope and intercept of the plot. According to Langmuir model, estimated value of monolayer adsorption capacity (Q_o) is 66.66 mg/g. One of the essential characteristic of the Langmuir isotherm can be expressed in term of a dimensionless constant separation factor R_L . The value of R_L indicates shape of the isotherms to be either unfavorable ($R_L > 1$), linear ($R_L = 1$), favorable ($0 < R_L < 1$), or irreversible ($R_L = 0$). The R_L values between 0 to 1 indicates favorable adsorption³². The expression for R_L is given by the following Eq. (5)

$$R_L = \frac{1}{(1 + b C_o)} \quad (5)$$

Here highest initial concentration of adsorbate and Langmuir constant is represented by C_o (mg/L or ppm) and b (L/mg) respectively. The value of R_L was found to be 0.55. at 34°C. This further indicates that the adsorption of MR on GG is favorable. The relation between Langmuir isotherm and experimental data was determined through correlation coefficient (R^2). The value of R^2 comes out to be 0.998 which further prove that the Langmuir isotherm fitted well with the experimental data. In case of removal of one of the azo dye (methyl orange) similar results were obtained by Mogaddasi et.al²⁹.

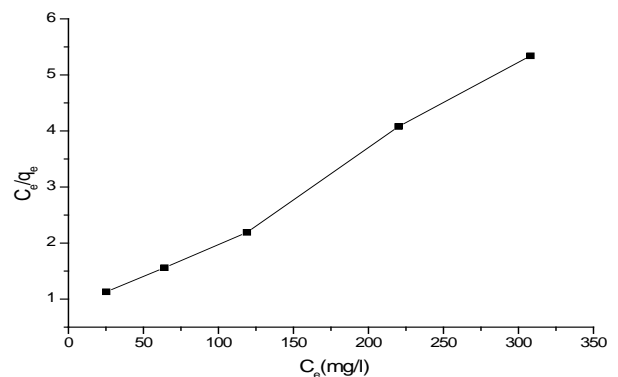


Fig.8 Langmuir isotherm for MR sorption onto GG at 34°C

For better understanding of the adsorption the Freundlich Isotherm³³ was also studied. The isotherm is given by an empirical equation which describes heterogeneous system. The Freundlich equation is given by Eq. (6)

$$q_e = K_F C_e^{1/n} \quad (6)$$

Where K_F and n are Freundlich constants. The value of K_F (mg/g (L/mg)^{1/n}) indicate the adsorption capacity of the sorbent and n gives an indication of favorable adsorption process. The magnitude of exponent, $1/n$, gives an indication of favorability of adsorption. Values of $n > 1$ represent favorable adsorption condition³⁴. On plotting a graph between $\ln(q_e)$ versus $\ln(C_e)$ as in Fig.9, constants K_F and n are determined. The value of K_F and n were calculated from the intercept and slope of the plot from the Eq. (7)

$$\ln q_e = \ln K_F + \left(\frac{1}{n}\right) \ln C_e \quad (7)$$

The value of maximum adsorption capacity (K_F) according to Freundlich model comes out to be 4.864 and that of n comes out to be 2.717. As value of $n > 1$ it further indicates favourable adsorption condition. The value correlation coefficient according to Freundlich isotherm comes out $R^2 < 0.96$. In case of NBP similar results were reported by Ashoka and Inamdar¹¹.

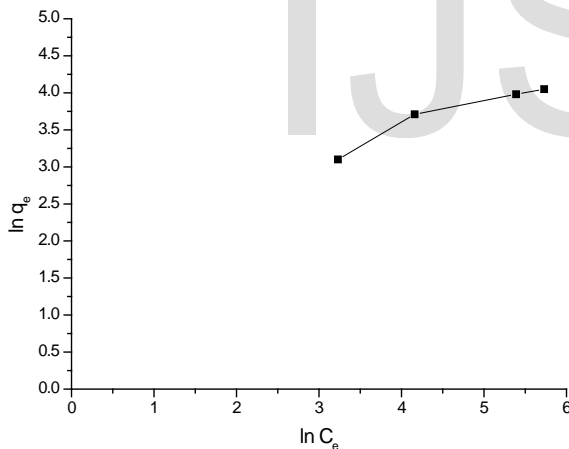


Fig.9 Freundlich isotherm for MR sorption onto GG at 34°C

One more adsorption isotherm model was studied as Temkin isotherm³⁵. According to Temkin isotherm model some indirect interactions between adsorbate and adsorbent were studied on adsorption isotherm. The Temkin isotherm is represented as in Eq. (8)

$$q_e = \left(\frac{RT}{b}\right) \ln(AC_e) \quad (8)$$

The above equation can be linearized as Eq. (9)

$$q_e = B \ln A + B \ln C_e \quad (9)$$

Where $B = RT/b$, b is the Temkin constant related to heat of

sorption (J/mol); A is the Temkin isotherm constant (L/g), R the gas constant (8.314 J/mol K) and T the absolute temperature (K). The values of constant A and B can be determined by plotting q_e against $\ln C_e$ (Fig.10). In present investigation the value of A comes out to be 5.22 and that of B comes out to be 13.9.

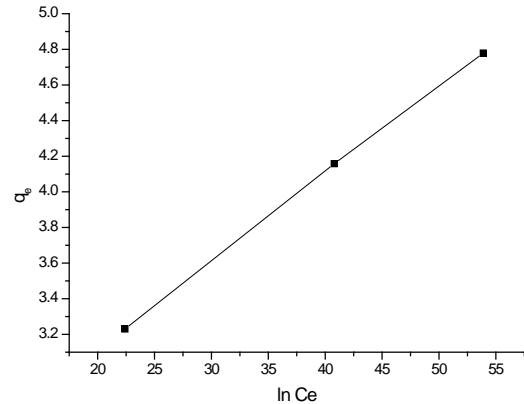


Fig. 10 Temkin isotherm for MR sorption onto GG at 34°C

According to Table 2, Langmuir isotherm fitted well with the experimental data as correlation coefficient R^2 is greater than 0.99. But in case of Freundlich and Temkin isotherm, the correlation coefficient R^2 is less than 0.96 which shows that these isotherms are not better and do not fit well with experimental data as compared to Langmuir isotherm model. The monolayer adsorption capacity according to Langmuir model was 66.66 mg/g. The Langmuir isotherm fits well because of homogeneous distribution of active sites onto GG surface. This finding was similar to other studies on the sorption of MR dye on different sorbents.

Table 2. Langmuir, Freundlich and Temkin isotherm model constants and Correlation Coefficient for biosorption of MR onto GG

Isotherm	Parameters	
Langmuir isotherm	Q_0 (mg/g)	66.66
	b (L/mg)	0.025
	R^2	0.998
	R_L	0.55
Freundlich Isotherm	K_F	4.864
	n	2.717
	R^2	0.842
Temkin Isotherm	A	5.22
	B	13.90
	R^2	0.905

3.8 Adsorption Kinetics

Adsorption Kinetics is done to determine the rate of adsorption according to Pseudo first order model and Pseudo second order model. Lagergren³⁶ explained a linear form of Pseudo-first-order model in a form of Eq. (10)

$$\log(q_e - q_t) = \frac{\log q_e - k_1 t}{2.303} \quad (10)$$

Pseudo first order rate constant k_1 was determined by plotting a linear graph between $\log (q_e - q_t)$ against time as shown in Fig.11. If the plot was found to be linear then the adsorption process is a pseudo first order process^{36,37}. Table 3 represents Pseudo first order rate constant k_1 and q_e determined from the model along with the corresponding correlation coefficients (R^2). It was observed that the pseudo-first-order model did not fit well as $R^2 < 0.99$. The calculated q_e values do not agree with the experimental q_e values which further suggest that adsorption of MR onto GG do not follow first-order kinetics. Similar results were shown by Ashoka and Inamdar¹¹

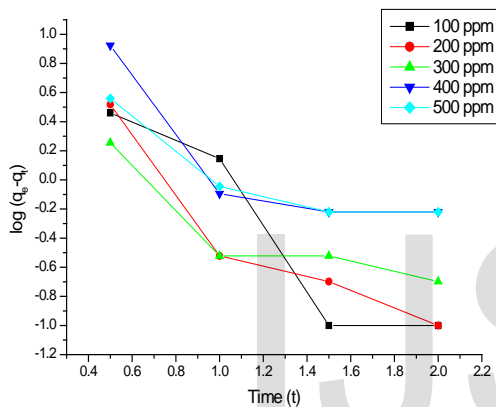


Fig.11 Pseudo-First-Order sorption Kinetics of MR onto GG

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e t} \quad (11)$$

On plotting a graph between t/q_t versus t , the equilibrium adsorption capacity (q_e) and the second order constants k_2 ($g/mg h$) was determined from the slope and intercept of plot respectively as shown in Fig.12. The values of k_2 and q_e determined from the model are presented in Table 3 along with the corresponding correlation coefficients. The calculated and experimental values of q_e are in good agreement for the Pseudo-second-order model with correlation coefficient $R^2 > 0.99$. Therefore it was concluded that the Pseudo-second-order model better represented the adsorption kinetics as compared to Pseudo first order model for adsorption of MR onto GG as biosorbent.

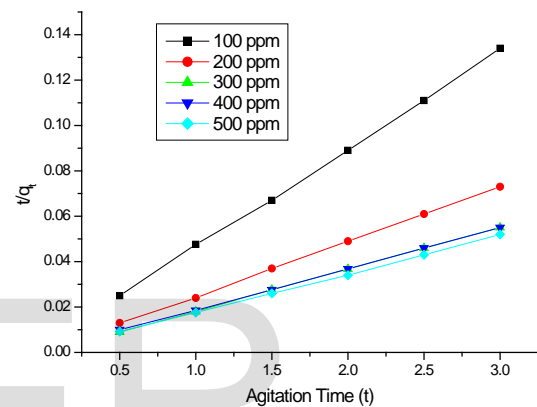


Fig.12 Pseudo-Second-Order sorption Kinetics of MR onto GG

The Pseudo-second-order kinetics^{31,38} may be expressed in a linear form as Eq. (11)

Table 3. Comparison of the Pseudo-first-order, Pseudo-second-order adsorption rate constant and calculated and experimental q_e values obtained at different initial MR concentration

Initial conc. (mg/L)	$q_{e,exp}$ (mg/g)	Pseudo-first order kinetic Model			Pseudo-second-order kinetic Model		
		k_1 (1/h)	$q_{e,cal}$ (mg/g)	R^2	k_2 (g/mg h)	$q_{e,cal}$ (mg/g)	R^2
100	22.4	2.5454	10.7930	0.8739	0.58268	23.1112	0.9995
200	40.8	2.1786	5.71671	0.8580	0.79145	41.5179	0.9996
300	54.3	1.3169	2.21334	0.7507	1.48104	54.1799	0.9999
400	54.9	1.6614	10.2726	0.7193	0.80930	55.0995	0.9999
500	57.6	0.3257	4.44239	0.7695	0.60489	58.6855	0.9996

4. CONCLUSION

In the present investigation, the adsorption ability of GG to remove MR dye from aqueous solution was investigated. During experimental work variation in various parameters were done like initial dye concentration, biosorbent concentration, pH of solution. Decolourization experiments were also done with respect to agitation time and particle size. Modelling of experimental data was done through

three isotherm models Langmuir, Freundlich and Temkin isotherm model. It is found that Langmuir adsorption isotherm fitted well with monolayer adsorption capacity as 66.66mg/g at pH 4.2, and temperature 34°C. Results for kinetic study are in well agreement for the Pseudo-second-order kinetic equation which further help us to conclude that GG could be used as one of the low cost biosorbent for removal of MR dye from aqueous solutions

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