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Kinetic Modeling and Isotherm Studies on Removal of Methyl Orange from Aqueous Solutions Using Guar gum Powder

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In this paper, the potential of guar gum powder was investigated as a low cost and eco-friendly biosorbent for the removal of methyl orange dye from aqueous solutions. The adsorption behaviour of the biosorbent was investigated by performing 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 were best fit with Langmuir isotherm as compared to Freundlich and Temkin isotherms. According to Langmuir model the monolayer adsorption capacity found to be 45.45 mg/g at pH 4.2 at 34 °C. The correlation coefficient value indicate a moderate fit for monolayer Langmuir model ($R^2 = 0.96$). The q_e experimental and calculated values for the pseudo-second-order kinetic model was in good agreement as compared to that by pseudo first order kinetics ($R^2 > 0.99$). The structural characterization of guar gum powder was done by scanning electron microscopy.

Keywords: Kinetic modeling, Isotherm studies, Removal, Methyl orange, Guar gum powder.

INTRODUCTION

Textile industry is one of the growing industries in Rajasthan. Different types of dyes are used in various textile industries [1]. Main source of water pollution are dyeing and printing industries [2]. Dyes are basically chemical compounds that can attach themselves to fabrics or surfaces to impart colour [3]. Most dyes are complex organic molecules and are resistant to weather, action of detergents and can affect human health and interfere with industrial water use [4]. Many industries, such as dyestuffs, textile, paper and plastics, use dyes in order to colour their products and also consume substantial volumes of water [5]. Due to dyeing, they generate a considerable amount of coloured wastewater. Colour is the first contaminant to be recognized in wastewater [6]. The presence of very small amounts of dyes in water (less than 1 ppm for some dyes) is highly visible and undesirable [7].

Dyes have been reported to cause carcinogenesis, mutagenesis, chromosomal fractures, teratogenicity and respiratory toxicity [8]. Azo dye is one of the commonly used dye in textile and leather industries [9,10]. Their removal from waste water is an urgent challenge [11,12]. Various methods have been extensively used for colour removal from effluent water which is quite expensive [13-15]. Now a day's various attempts have been made to find inexpensive alternative treatment to remove

dyes from effluents. Over the last few years adsorption has gained paramount importance in industry and environment protection [16,17]. Recently, various approaches have been made for the development of low-cost and effective adsorbents. Removal of dyes by using these bio-adsorbents follows the mechanism of reaction with proteinaceous cellular material [11]. Various sorbent have already been used like mango peel [18], banana peel [19], coconut bunch waste [20], wheat husk [21], tree bark [22], chitosan [23], *etc.*

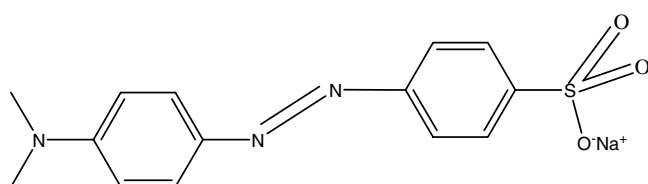
In this study, guar gum powder was used for adsorption of methyl orange dye from aqueous solutions. Guar gum has wide applications in pharmaceutical like cosmetic, food, textile, paper, explosive and toiletries industries *etc.* [24]. Guar gum is also called as guaran and is primarily the ground endosperm of guar beans. India (mainly Rajasthan, Gujarat, Haryana) produces 2.5-3.5 million tons of guar annually, making it the largest producer with about 80 % of world production. Industrial guar gum accounts for about 70 % of the total demand [25,26]. The objective of present study was to analyze the percentage of methyl orange removal using guar gum powder, as a novel adsorbent from aqueous solutions.

EXPERIMENTAL

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, thermometer, centrifuge, crusher, grinder.

Adsorbate: The azo dye used in this study was methyl orange of Merck Limited. The maximum absorption wavelength of methyl orange dye is 470 nm at pH 4.2. The structure of methyl orange dye is shown in **Scheme-I**. The physical and chemical characterization of methyl orange is given in Table-1.



Scheme-I: Chemical structure of methyl orange

TABLE-1
PHYSICAL AND CHEMICAL
PROPERTIES OF METHYL ORANGE DYE

Physical state	Solid
Melting point	300 °C
Molecular weight	327.33 g/mol
Chemical formula	C ₁₄ H ₁₄ N ₃ NaO ₃ S
λ_{max}	470 nm
pH	4.2

Preparation and characterization of adsorbent: The guar gum seed used was collected from the local vegetable market Jaipur, Rajasthan (India). These guar gum seeds were then washed with normal water and finally with distilled water to remove dust and water soluble materials. Wetted guar seeds wiped to remove extra adhered water drops on seed's outer surface. These were then spread on tray and dried in sunlight for 4-5 h per day. The process continues up to 3-4 days. After crushing, seeds are converting into guar splits. It was then grind to obtain guar gum powder (GGP). The process was repeated again and again. No other chemical or physical treatment was used prior to adsorption experiments. Surface morphology of guar gum powder was analyzed by using scanning electron microscopy (SEM) before and after adsorption of dye.

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 methyl orange dye solution (100-500 ppm) at a pH 4.2 were added at 34 °C. The pH was maintained by using 1 N NaOH and 1 N HCl solution. The Erlenmeyer flask were capped and agitated in an isothermal shaker at 110 rpm at 34 °C for 4 h to achieve equilibrium. The concentration of the dye in the solution after equilibrium adsorption was determined by UV-visible spectrophotometer at 470 nm for methyl orange dye at pH 4.2. The amount of adsorption at equilibrium, q_e (mg/g), was calculated by eqn. 1:

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

where C_o 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 methyl orange 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 eqn. 2:

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

The adsorption studies were studied by various isotherms.

RESULTS AND DISCUSSION

Characterization of sorbent: Fig. 1(a) show the SEM micrographs of guar gum powder sample before methyl orange adsorption and Fig. 1(b) show guar gum powder micrographs after adsorption of methyl orange. The guar gum powder exhibits uneven, cave like rough surface morphology. The surface of dye loaded adsorbent indicates that some of the surface of guar gum powder 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.

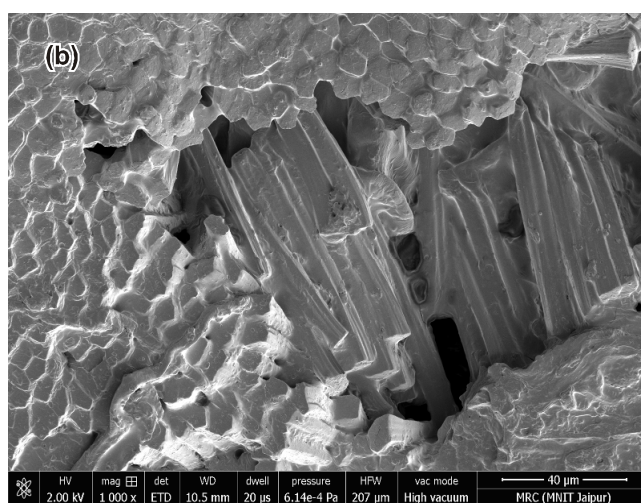
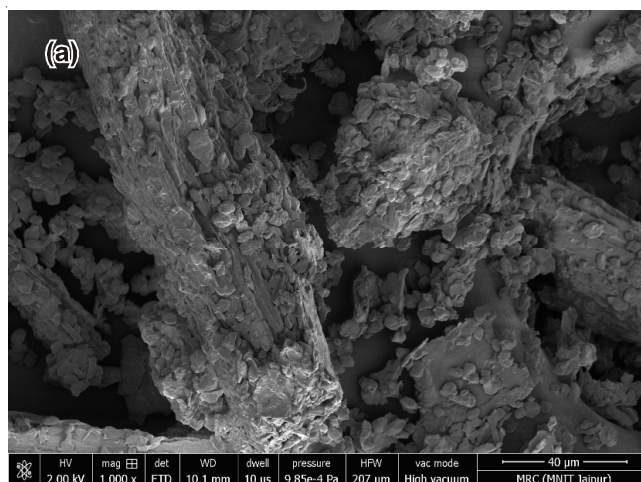


Fig. 1. SEM micrographs of (a) fresh guar gum powder and (b) methyl orange adsorbed guar gum powder

Effect of initial concentration of methyl orange: The effect of the initial concentration of methyl orange on the intake rate by guar gum powder adsorption at adsorbent dosage of 0.10 g and mixing speed of 110 rpm is 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 methyl orange adsorbed (q_e) increased from 18 to 36 mg/g as the concentration of dye was increased from 100 to 500 ppm. The initial dye concentration generates a driving force to overcome all mass transfer resistances of the dye between the aqueous and solid phase. Hence a higher initial concentration of dye will enhance the adsorption process. The methyl orange removal decreased from 55 to 20 % as the concentration of the dye increased from 100 to 500 ppm (Fig. 3). The equilibrium conditions were reached within 3 h. The negative slope of the plot indicates that the dye removed by guar gum powder decreases with increasing concentration in an aqueous solution.

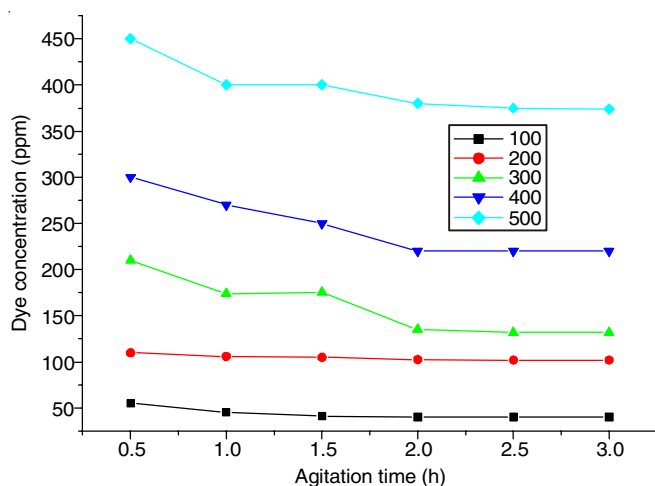


Fig. 2. Effect of initial dye concentration on removal of methyl orange by guar gum powder (conditions: sorbent dosage (W), 0.10 g; initial pH, 4.2; temperature, 34 °C; particle size, 500 μ ; stirring rate, 110 rpm; V, 30 mL; dye concentration, 100-500 ppm)

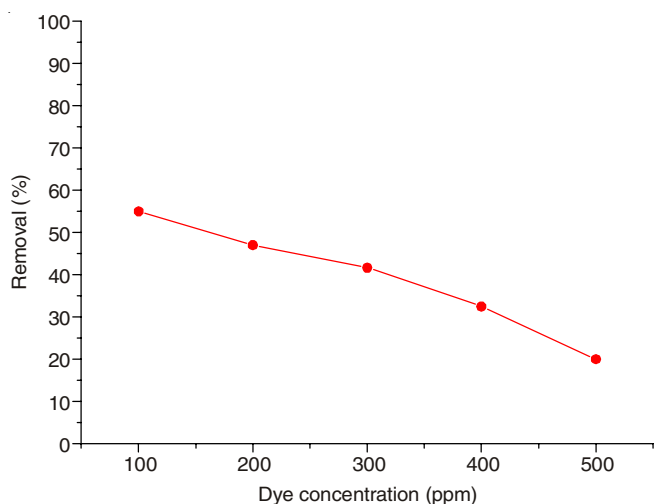


Fig. 3. Effect of initial dye concentration on percentage removal of methyl orange by guar gum powder (conditions: sorbent dosage (W), 0.10 g; initial pH, 4.2; temperature, 34 °C; particle size, 500 μ ; stirring rate, 110 rpm; V, 30 mL; dye concentration, 100-500 ppm)

Similar results were shown by Egwuonwu [22] while studying removal of methyl orange by neem bark powder. An increase in initial dye concentration from leads to a decrease in the percentage of methyl orange removal.

Effect of pH: Effect of pH on adsorption was studied using 100 ppm dye concentration and varying pH from 2 to 10 at 34 °C (Fig. 4). Methyl orange removal increased from 30 to 90 % over the same pH value from 2-10. The lowest dye adsorption (q_e) was found to increase with increasing pH. It increases from 9 to 27 mg/g for pH range 2-10. Lower adsorption at acidic pH is probably due to presence of excess H^+ ions competing with cation groups on the dye adsorption sites. At higher pH, the surface of guar gum powder may get negatively charged, which enhance the positively charged dye cation through electrostatic forces of attraction [20]. This further indicate possible involvement of some functional groups of guar gum powder in sorption process.

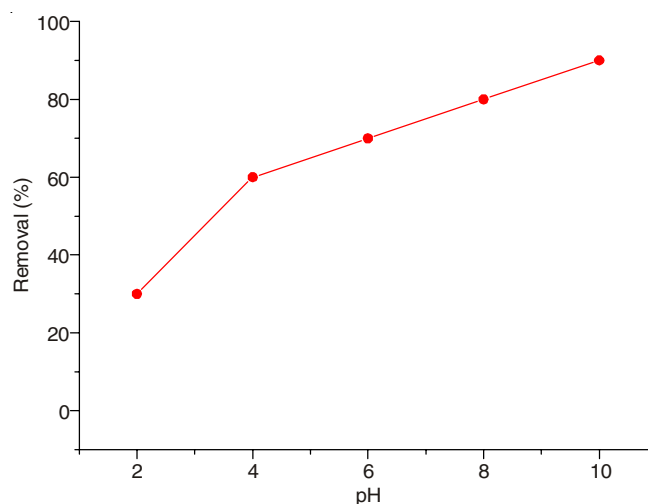


Fig. 4. Effect of solution pH on percentage removal of methyl orange by guar gum powder (conditions: sorbent dosage (W), 0.10 g; initial pH, 4.2; temperature, 34 °C; particle size, 500 μ ; stirring rate, 110 rpm; V, 30 mL; dye concentration, 100 ppm)

Similar results were given by Egwuonwu [22] who conducted the experiments to determine the effect of pH on methyl orange adsorption by neem bark powder and found that the percentage removal of methyl orange by neem bark powder was optimum at pH 4.

Effect of biosorbent dose on adsorption: The biosorbent dose is an important parameter because it determines the capacity of a biosorbent for a given initial concentration. To investigate the effect of biosorbent dosage on biosorption, the experiments were conducted with constant dye concentration (100 ppm) and samples with different biosorbent dosages ranging from 0.10 g to 0.25 g under constant temperature at 34 °C and pH 4.2 for 1 h. The results are shown in Fig 5. It was observed that percentage of dye adsorption increased with increase of adsorbent dose. The removal of methyl orange increases from 45 to 71 % 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.

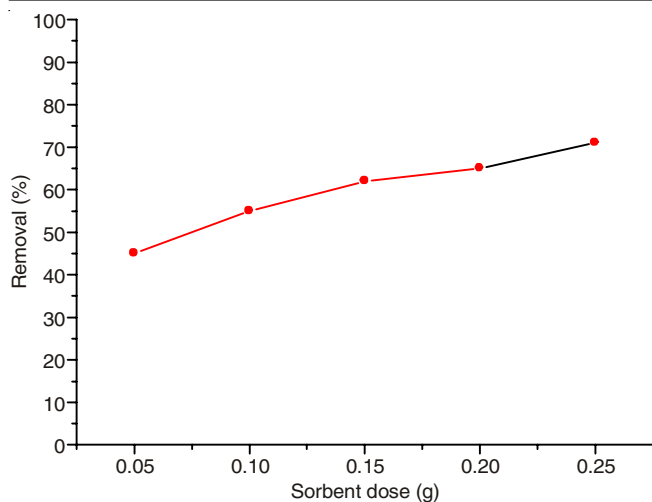


Fig. 5. Effect of biosorbent dosage (g) on percentage removal of methyl orange by guar gum powder (conditions: initial pH, 4.2; temperature, 34 °C; particle size, 500 μ ; stirring rate, 110 rpm; V, 30 mL; dye concentration, 100 ppm; agitation time, 1 h)

It is clear that 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.

Effect of particle size: Percentage removal of dye increases on decreasing particle size as shown in Fig. 6. It is due to the reason that more fine powder increases the surface area for adsorption. On reducing particle size from 500 to 150 μ , percentage removal increases from 65 to 80 % for methyl orange.

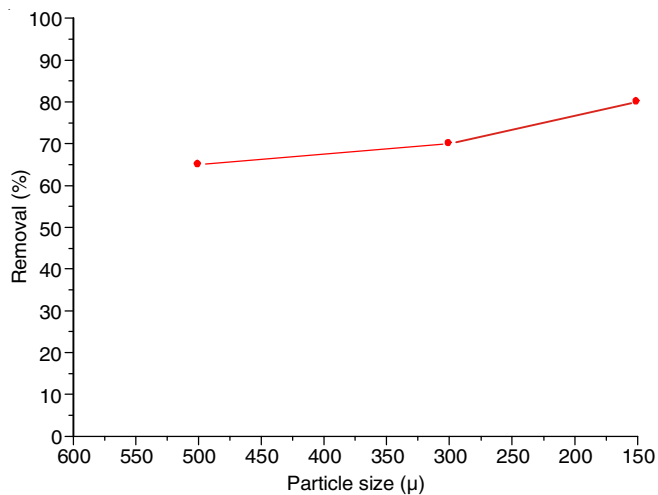


Fig. 6. Effect of particle size on percentage removal of methyl orange by guar gum powder (conditions: sorbent dosage (W), 0.10 g; initial pH, 4.2; temperature, 34 °C; stirring rate, 110 rpm; V, 30 mL; dye concentration, 100 ppm)

Effect of agitation time: Variation in agitation time also plays an important 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. Due to higher contact time, percentage removal of dye increases.

On increasing agitation time from 0.5 to 3 h, percentage removal of methyl orange dye increases from 45 to 60 % as shown in Fig. 7. Initially there is increased rate of percentage removal of methyl orange dye due to availability of number of active binding sites on guar gum powder 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 were reported by Srivastava and Rupainwar [27].

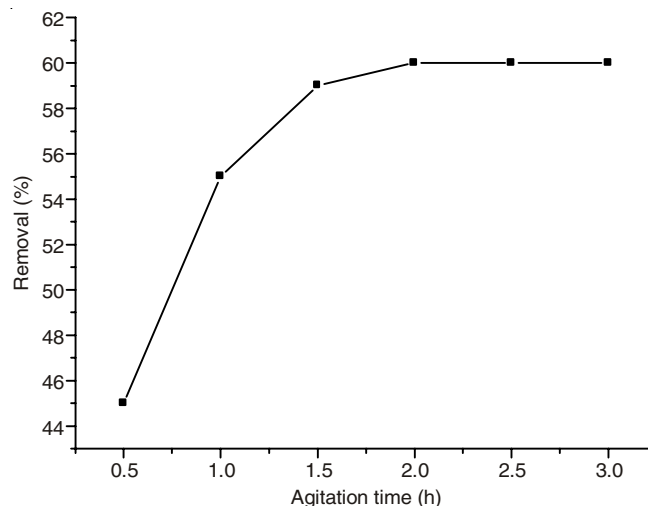


Fig. 7. Effect of agitation time on percentage removal of methyl orange by guar gum powder (conditions: sorbent dosage (W), 0.10 g; initial pH, 4.2; temperature, 34 °C; stirring rate, 110 rpm; V, 30 mL; dye concentration, 100 ppm)

Adsorption isotherms: The main purpose of the adsorption isotherms is to relate the adsorbate concentration in the bulk and the adsorbed amount at the interface [28]. The isotherm results were analyzed using the Langmuir, Freundlich and Temkin isotherms. The Langmuir adsorption model [29] is based on the assumption that maximum adsorption corresponds to a saturated mono layer of solute molecules on the adsorbent surface, with no later interaction between the sorbed molecules. The expression of the Langmuir model is given by eqn. 3:

$$q_e = \frac{Q_0 b C_e}{(1 + b C_e)} \quad (3)$$

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. Q_0 is the maximum amount of the dye per unit mass of sorbent to form a complete monolayer on the surface bound at high C_e 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 as in eqn. 4:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0} \quad (4)$$

when we plot a graph between specific adsorption C_e/q_e against equilibrium concentration C_e , the linear plot (Fig. 8) shows that the adsorption obeys the Langmuir model. The Langmuir constants Q_0 and b were determined from the slope and

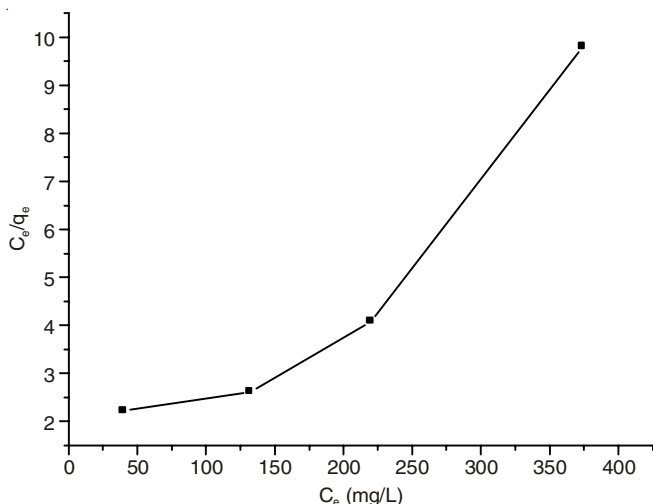


Fig. 8. Langmuir model curve for methyl orange sorption onto guar gum powder at 34 °C

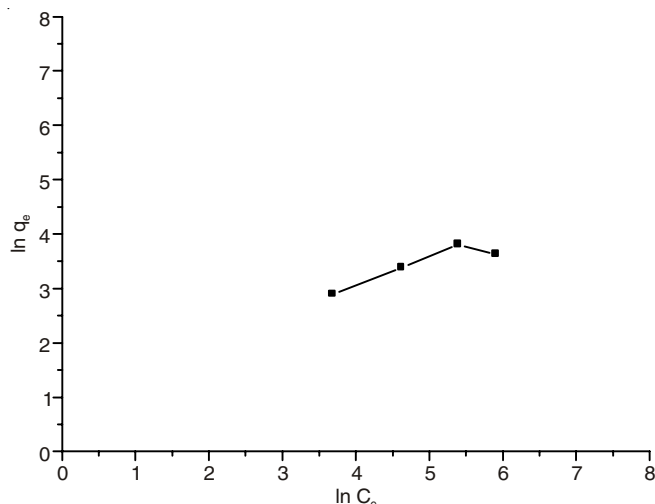


Fig. 9. Freundlich model curve for methyl orange sorption onto guar gum powder at 34 °C

intercept of the plot. The essential characteristic of the Langmuir isotherm can be expressed in term of a dimensionless constant separation factor R_L which is given by the following eqn. 5:

$$R_L = \frac{1}{(1 + bC_0)} \quad (5)$$

where C_0 is highest initial concentration of adsorbate (mg/L or ppm) and b (L/mg) is Langmuir constant. The value of R_L indicates the shape of the isotherms to be either unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 < R_L < 1$), or irreversible ($R_L = 0$). The R_L values between 0 and 1 indicates favourable adsorption. The value of R_L in the present investigation was found to be 0.52 at 34 °C indicating that the adsorption of methyl orange on guar gum powder is favourable. The value of correlation coefficient (R^2) comes out to be 0.96. Similar results were reported by Mogaddasi *et al.* [30].

Another isotherm studied was the Freundlich isotherm [31] which describes heterogeneous system and is given by eqn. 6:

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

where K_F and n are Freundlich constant with K_F (mg/g (L/mg)^{1/n}) is the adsorption capacity of the sorbent and n giving an indication of favourable adsorption process. Values of $n > 1$ represent favourable adsorption condition [30]. The linear form of the equation is used to produce a graph of $\ln(q_e)$ against $\ln(C_e)$ to determine the constants K_F and n (Fig. 9). Value of K_F and n are calculated from the intercept and slope of the plot, from the eqn. 7:

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

The value of R^2 according to Freundlich isotherm comes out to be 0.837. The values of other constants are given in Table-2.

Another adsorption isotherm model studied was Temkin isotherm. The Temkin isotherm [32] has been generally applied in the following form:

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

TABLE-2 LANGMUIR, FREUNDLICH AND TEMKIN ISOTHERM MODEL CONSTANTS AND CORRELATION COEFFICIENT FOR ADSORPTION OF METHYL ORANGE BY GUAR GUM POWDER	
Isotherm	Isotherm parameters
Langmuir isotherm	
Q_0 (mg/g)	45.45
b (L/mg)	0.039
R^2	0.959
R_L	0.52 at 34 °C
Freundlich isotherm	
K_F	1.818
N	2.35
R^2	0.837
Temkin isotherm	
A	1.80
B	22.0
R^2	0.864

The above equation can be linearized as eqn. 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 constant A and B determined by plotting q_e against $\ln C_e$ (Fig. 10). According to Temkin, some indirect sorbate/adsorbate interaction occurs during adsorption and because of these interactions, the heat of adsorption of all molecules in the layer would decrease linearly with coverage.

Table-2 indicates that the Langmuir isotherm fitted well with the experimental data as correlation coefficient R^2 is 0.96. In case of Freundlich and Temkin isotherm, the correlation coefficient R^2 is less than 0.96, which indicate poor agreement with experimental data. The monolayer adsorption capacity according to Langmuir model was 45.45 mg/g. The Langmuir isotherm fit well because of homogeneous distribution of active sites onto guar gum powder surface. This finding was similar to other studies on the sorption of methyl orange dye on different sorbents.

Adsorption kinetics: Adsorption kinetics is done to determine the rate of adsorption according to pseudo-first-order

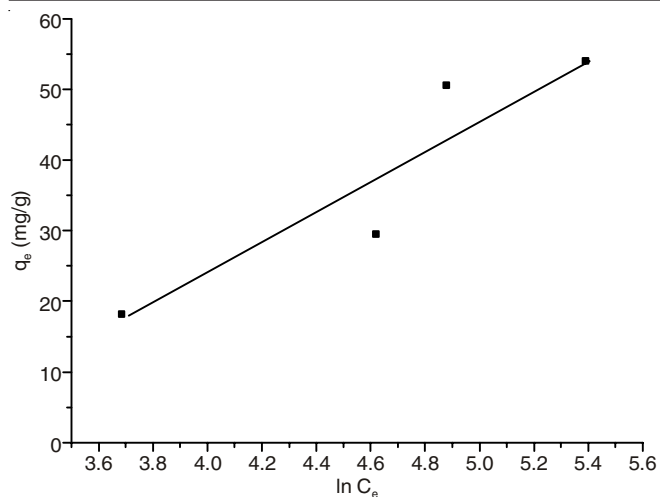


Fig. 10. Temkin isotherm for methyl orange sorption onto guar gum powder at 34 °C

model and pseudo-second-order model. Lagergren [33] explained a linear form of pseudo-first-order model in a form of eqn. 10:

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

Pseudo first order rate constant k_1 was determined by plotting a linear graph between $\log (q_e - q_t)$ against time (Fig. 11). If the plot was found to be linear then the adsorption process is a pseudo first order process. 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

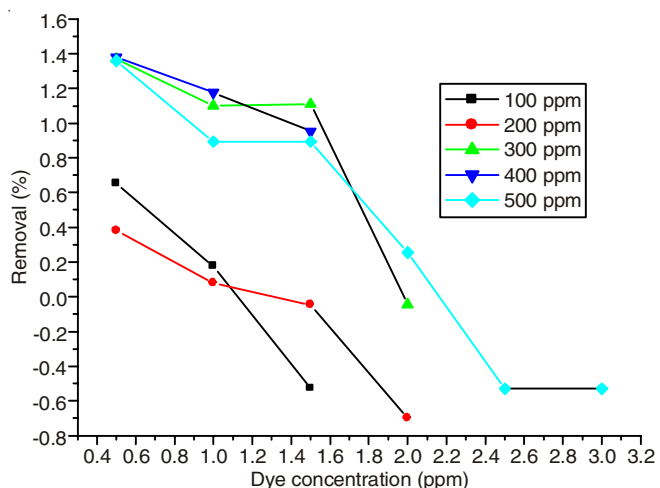


Fig. 11. Pseudo-first-order adsorption kinetics of methyl orange onto guar gum powder

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 do not follow first-order kinetics.

The pseudo-second-order kinetics [34] may be expressed in a linear form as eqn. 11:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e t} \tag{11}$$

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 (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 $R^2 > 0.99$. Mogaddasi *et al.* [30] worked upon the kinetic result of methyl orange on camel thorn plant and concludes that the sorption of methyl orange on adsorbent follows the pseudo-second-order kinetic model.

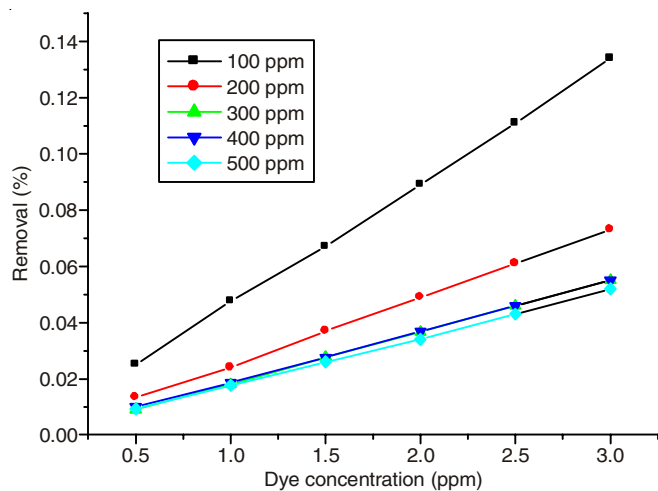


Fig. 12. Pseudo-second-order adsorption kinetics of methyl orange onto guar gum powder

Therefore it was concluded that in present investigation, the pseudo-second-order model better represented the adsorption kinetics as compared to pseudo first order kinetic model.

Conclusion

In this study, the adsorption ability of guar gum powder sorbent to remove methyl orange from aqueous solution was investigated. Since guar gum powder is abundantly and locally available, the resulting sorbent is expected to be economically

TABLE-3
COMPARISON OF PSEUDO-FIRST-ORDER, PSEUDO-SECOND-ORDER ADSORPTION RATE CONSTANTS AND CALCULATED AND EXPERIMENTAL q_e VALUES OBTAINED AT DIFFERENT INITIAL METHYL ORANGE CONCENTRATIONS

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	18.0	2.72	19.12	0.99	0.31	19.31	0.99
200	29.4	1.88	5.88	0.68	0.55	30.04	0.99
300	50.4	1.95	87.77	0.75	0.02	64.36	0.98
400	54.0	0.98	39.47	0.99	0.02	67.57	0.99
500	37.8	2.39	70.93	0.93	0.01	59.00	0.88

viable for removal of one of the azo dyes from aqueous solutions. The experimental results were calculated by three models, Langmuir, Freundlich and Temkin isotherms. The adsorption studies fitted well with Langmuir isotherm as compared to others and it gives monolayer adsorption capacity of 45.45 mg/g. Data for kinetic studies are in well agreement for pseudo-second-order kinetic equation which further help us to conclude that guar gum powder could be used as one of the low cost biosorbent for removal of methyl orange from aqueous solutions.

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