Journal of Advanced Scientific Research

Available online through http://www.sciensage.info

ISSN
0976-9595
Research Article

EQUILIBRIUM ADSORPTION ISOTHERMS FOR METHYLENE BLUE DYE ONTO CSNP-SiO_2 NANOCOMPOSITE

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ABSTRACT
Today, environmental pollutants pose a threat to human societies and all living organisms, which is why they have attracted the attention of environmental researchers. In this study, dye adsorption from aqueous media with CSNP-SiO_2 Nanocomposite was investigated. The effects of different variables such as adsorbent dosage, pH, and temperature were studied. The results obtained showed that the adsorption of Methylene blue is dependent on adsorbent dosage, pH, and temperature. It was noted that the adsorption of all the dyes on CSNP-SiO_2 Nanocomposite decreases with an increase in the dosages, pH, and temperature. The adsorption isotherms at different temperatures were found to be of L-type. The optimum adsorbent dosage, pH, and temperature were found to be 3g/L, pH 6.4, and 60ºC, respectively. Adsorption equilibrium data of aqueous solutions were analyzed using Langmuir and Freundlich isotherms. The isotherm data could be well described by the Freundlich and Langmuir equations in the concentration range of 20-100 mg/l. An alkaline pH was favorable for the adsorption of dyes. Based on the adsorption capacity, it was shown that CSNP-SiO_2 Nanocomposite was a more effective removal dye solution.

Keywords: CSNP-SiO_2 Nanocomposite, Methylene blue, Langmuir and Freundlich Isotherm.

1. INTRODUCTION
Industrial, agricultural and domestic wastes, due to the rapid development in technology, are discharged in several receivers. Generally, this discharge is directed to the nearest water sources such as rivers, lakes, and seas. The textile dyeing process is an important source of contamination responsible for the continuous pollution of the environment. Several industries such as textiles, printing, and painting represent the main sources of contaminated water with dyes [1]. The dyes have been widely used in a variety of products such as textiles, color papers, foodstuffs, artificial fibers, and leathers [2]. Among the treatment options, adsorption has become one of the most effective and comparable low-cost methods for the decolorization of textile wastewater [3-4]. Different adsorbents have been used for the removal from aqueous solutions of various materials, such as dyes, metal ions, and other organic materials include perlite [5-10], Bentonite [11], silica gels [12], fly ash [13-14], lignite [15], peat [16], silica [17], etc. Natural polysaccharide chitosan; a derivative of chitin, is of great interest as an organic component in the composites developed for water treatment because of the high quantity of amino and hydroxyl groups [18-20], which is very important for sorption processes [21]. Recently, a natural polymer, such as Chitosan and its derivatives has significantly attracted interest [22]. This is because Chitosan is a low-cost and effective adsorbent compared with other adsorbents used in the adsorption of organic or inorganic pollutants [23]. Furthermore, due to the unique structure of Chitosan, it has proven to have outstanding removal capacities for dyes [24]. In culture media, chitosan has an antimicrobial effect on bacteria such as *Staphylococcus aureus, Enterobacter, Pseudomonas, Escherichia coli,* and *Bacillus Subtilis* [25-26]. SiO_2 is among the well-known inorganic adsorbents. The surface properties of amorphous silica, which is considered to be an oxide adsorbent, in many cases depend on the presence of silanol (OH) groups [27]. The OH groups act as the centers of molecular adsorption during their specific interaction with adsorbates, which is capable to form a hydrogen bond with the OH groups, or more generally, undergoing donor-acceptor interaction [28]. Among the inorganic oxides, SiO_2 is an interesting choice

for the formulation of an organic-inorganic composite carrier. Over the past years, the sol-gel process was widely used to create novel hybrid nanoscale materials based on organic and inorganic components [29]. This approach can produce particles at low temperatures and pressure. The advanced utility of CSNP-SiO_2 Nanocomposite for the development of drug carriers can be accomplished by controlling its morphology. The method proposed to obtain particles with a controlled morphology is spray drying, in particular, ultrasonic spray drying; one of the aerosol-assisted spray methods employing ultrasonic nebulizer [30]. By using ultrasonic nebulizer fine droplets in the range of 1-10 μm, particles can be produced which is smaller than particles attained by a nozzle or rotary atomizer [31]. In this work, CSNP-SiO_2 Nanocomposite cross-linked with TPP was synthesized by the ionotropic sol-gel method using water glass (Na_2SiO_3) as the silica precursor. The controlled morphology of particles was achieved by ultrasonic spray drying apparatus. The presence of functional groups, the morphology of the particles, and the size distribution of the obtained particles were investigated. In addition, the mechanism of the inotropic-sol gel method was also proposed. Methylene Blue (MB), a cationic dye, is widely used for colouring paper, printing cotton, dyeing leather, and indicating oxidation-reduction in analytical chemistry and also used as an antiseptic [32]. In this paper, a CSNP-SiO_2 Nanocomposite was prepared using precipitation method. The CSNP-SiO_2 Nanocomposite is tested as adsorbent for Methylene blue dye removal. It was found that they exhibited excellent adsorptive properties and stability toward Methylene blue dye. The overall objective in this work is to prepare CSNP-SiO_2 Nanocomposite along with dye adsorption studies. It is concluded that the CSNP-SiO_2 Nano-composite is a potential recyclable adsorbent for water treatment.

2. MATERIAL AND METHODS

The following chemicals were of analytical grades such as silicon dioxide (SiO_2), Chitosan, sodium tripoly-phosphate (STPP), sodium hydroxide, acetic acid, hydrochloric acid. Methylene blue dye was supplied by CDH Chemicals New Delhi and used as received. Methylene blue dye has a chemical formula of $C_{16}H_{18}ClN_3S$, a molecular weight of 319.85 g/mol, and λmax= 663 nm. Stock solutions were prepared by dissolving accurately weighed samples of dye in deionized water.

2.1. Preparation of CSNP-SiO_2 Nanocomposite

The sol-gel method was used for the synthesis of CSNP-SiO_2 Nanocomposite in this work. In a typical method, the solution contained 1.0 g of chitosan powder dissolved in 100 ml of 2% volume of acetic acid in a 500 ml Beaker with constant stirring by using a magnetic stirrer. This mixture was subsequently stirred for 2 hours after all of the chitosan had dissolved completely in the diluted acetic acid solution. The synthesis of chitosan nanoparticles was achieved by adding 2 ml hydrogen peroxide and 20 ml sodium tripolyphosphate (10% w/v) followed by a constant string with a magnetic stirrer at 140 rpm for 10 min. The Chitosan nanoparticle was mixed with Silicon dioxide (1.0g), then the reactant mixture was stirred with a magnetic stirrer at 140 rpm for 24 hrs. The mixture was dried in an oven at 50°C in a hot air Oven. After the mixture was dried, the calcination process was done at 600°C for 4 hours in a muffle furnace. After calcination, the powder obtained was crushed by using ball milling to get CSNP-SiO_2 Nanocomposite.

2.2. Batch equilibrium and kinetic studies

Adsorption experiments were carried out by adding a fixed amount of Chitosan Nanoparticle with Silica gel Nanocomposite (0.1g) to a series of 100mL conical flasks filled with 100mL diluted solutions (20-100mg/L). The conical flasks were then sealed and placed in a water-bath shaker and shaken at 100rpm with a required different Dosages, pH, and Temperatures. The flasks were then removed from the shaker (after 24hrs), and the final concentration of dye in the solution was measured at maximum wavelengths of Methylene blue dye (663 nm) using a double beam UV/vis spectrophotometer. The adsorption isotherms and kinetics were analyzed by determining the adsorption capacities at various parameter conditions. Mass balance equation was used, to find the amount of dye adsorbed in each flask which was determined by the equation,

$$q_e = \frac{C_I - C_F}{m} \times v$$

Where qe = Adsorbed amount (mg/g); C_I- initial concentration of (mg/L); C_F- Final concentration of (mg/L); V- Volume of adsorbate; m- Mass of used.

2.2.1. Effect of Different Dosages

A 0.1g sample of CSNP-SiO_2 Nanocomposite was added to each 100mL volume of Methylene blue dye solution. The dosages of dye solution tested were 0.1, 0.2, and

0.3mg/L, and the experiments were carried out at 30°C for 24 hrs.

2.2.2. Effect of temperature

A 0.1g sample of CSNP-SiO_2 Nanocomposite was added to each 100mL volume of Methylene blue dye aqueous solution. The experiments were carried out at 30°C, 45°C, and 60°C for 24hrs.

2.2.3. Effect of solution pH

Effect of solution pH was investigated at pH 5.4, 6.4 and 7.4, and 1.0g/L sample of CSNP-SiO_2 Nanocomposite was added to each 100mL volume of Methylene blue dye aqueous solution having an initial concentration of 40mg/L for a constant adsorption time of for 24hrs.

3. RESULTS AND DISCUSSION

3.1. Adsorption of Methylene blue on the effect of dosage, temperature, and pH

The Langmuir isotherm has found successful application for many other real sorption processes and it can be used to explain the sorption of dyes into CSNP-SiO_2 Nanocomposite. A basic assumption of the Langmuir theory is that the sorption takes place at specific sites within the adsorbent [33-42]. The data obtained from the adsorption experiment conducted in the present investigation was fitted in different sorbent dosages, pH, and temperature in isotherm equation as shown in fig. 1-3, which had shown the adsorption of dye at different adsorbent dosage, temperature, and pH by using CSNP-SiO_2 Nanocomposite. The adsorbent rate variation may be due to the number of positive charges on the sorbent surface which leads to no rejection of negatively charged dye molecule and thereby increasing the adsorption. The adsorption of dye increased with the increase in adsorbent dosage (fig. 1). The maximum percentage removal of about 80.0 g/L was obtained for an adsorbent dosage of 3.0 g/L for Methylene blue dye at 100 mg/L concentration. The increase in adsorption of dye with adsorbent dosage was due to the availability of more surface area of the adsorbent for adsorption. The result is similar to the observation of Namasivayam et al. [43].

The dye uptakes are much higher in acidic solutions than those in neutral and alkaline conditions. This explanation is a conflict with our data on the pH effect (fig. 2). It can be seen that the pH of the aqueous solution plays an important role in the adsorption of Methylene blue dye onto CSNP-SiO_2 Nanocomposite. The present results conflict with the results of Namasivayam et al. [43] and parallel to the results of Habib et al. [44]. The maximum percentage removal of 56.0 g/L was obtained in pH of 6.4 for Methylene blue dye at 140 mg/L concentration.

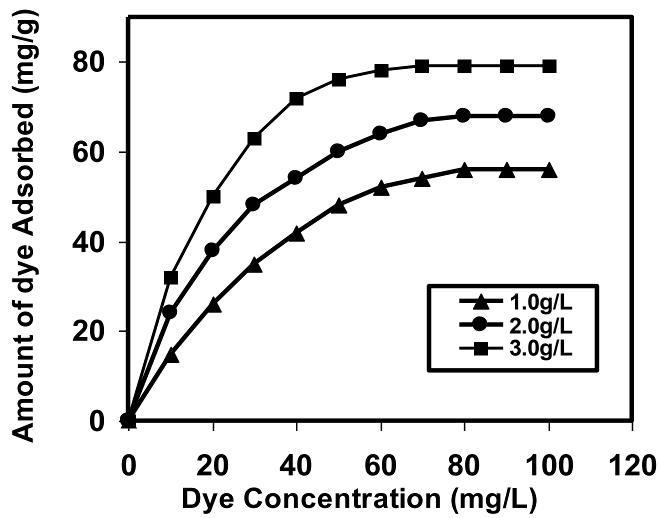

Fig. 1: Effect of Methylene blue dye uptake at different adsorbent dosages

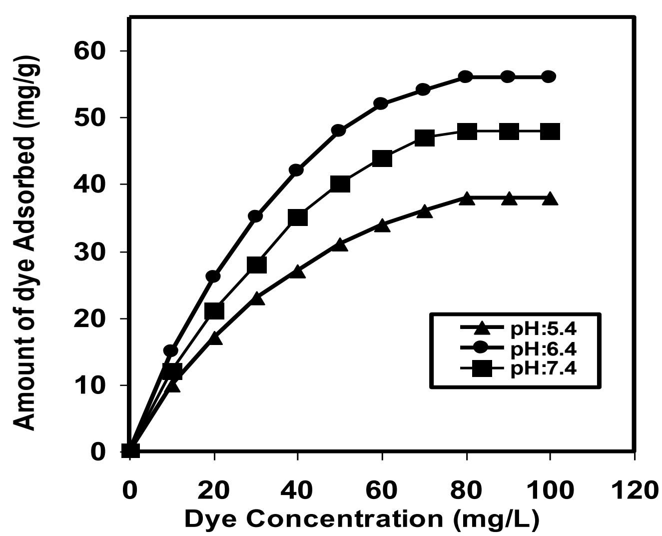

Fig. 2: Effect of Methylene blue dye uptake at different pH

Temperature is an important parameter for the adsorption process. A plot of the Methylene blue dye uptake as a function of temperature 30, 40, and 60°C is shown in fig. 3. The maximum percentage removal of 96.0 g/L was obtained for the temperature of 60°C for Methylene blue dye at 40 mg/L concentration. The adsorption of dye at a higher temperature was found to be greater compared to that at a lower temperature. The curves indicate the strong tendency of the process for monolayer formation [45]. The increase in temperature would increase the mobility of the large dye ion and also produces a swelling effect within the internal structure of the CSNP-SiO_2 Nanocomposite, thus enabling the large dye molecule to penetrate further [46-48]. Therefore, the adsorption capacity should largely depend on the chemical interaction between the functional groups on the adsorbent surface and the adsorbate and should increase with a temperature rising. The adsorption of dye at higher temperatures was found in the present investigation is similar to the results reported earlier [48].

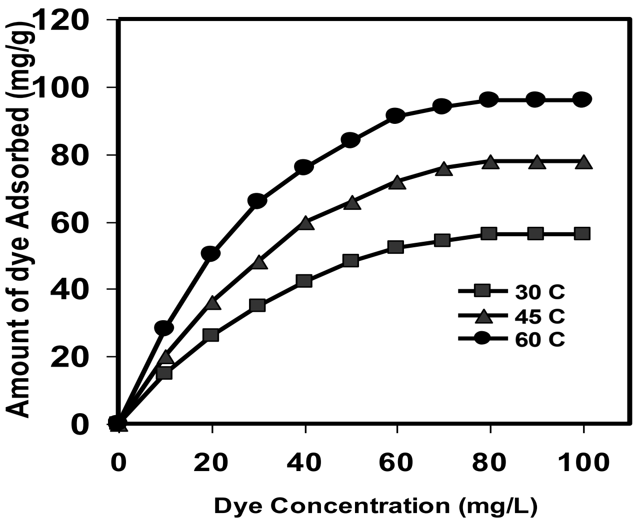

Fig. 3: Effect of Methylene blue dye uptake at different temperatures

3.2. Langmuir isotherm

The equilibrium adsorption isotherm is of fundamental importance in the design of adsorption systems. The isotherm expresses the relation between the mass of dye adsorbed at a particular dosage, temperature, and pH and the liquid phase of dye concentration. For any adsorption investigation, one of the most important parameters required to understand the behaviour of the adsorption process in the adsorption isotherm. The shape of an isotherm not only provides information about the affinity of the dye molecule for adsorption but also reflects the possible mode of adsorbing the dye molecule. The most common way of obtaining an adsorption isotherm is to determine the concentration of dye solution before and after the adsorption experiments, although several attempts have been made to find the adsorbed amount. A basic assumption of the Langmuir theory [49] is that sorption takes place at specific sites within the adsorbent [50]. The data obtained from the adsorption experiment conducted in the present investigation was fitted in different adsorbent dosage, pH, and temperature in the isotherm equation as shown in fig. 4-6. The saturation monolayer can be represented by the expression.

$$q_e = \frac{KbC_e}{(1+bC_e)}$$

$$\frac{1}{q_e} = \frac{1}{K} + \frac{1}{KbC_e}$$

A plot of ($1/q_e$ vs $1/C_e$) resulted in a linear graphical relation indicating the applicability of the above model as shown in fig. 4-6.

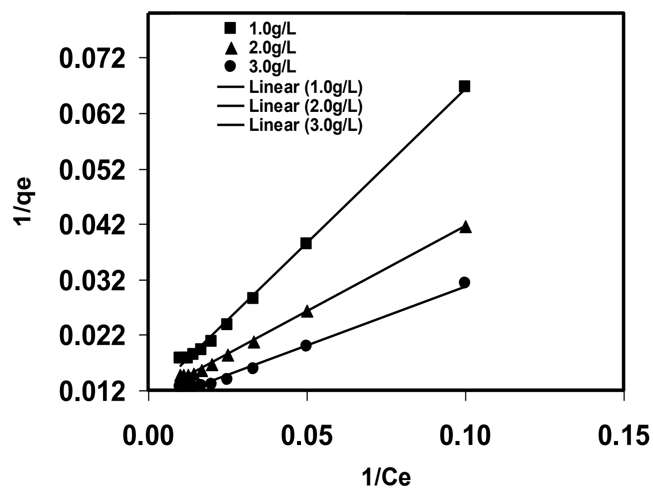

Fig. 4: Langmuir isotherm for the adsorption of Methylene blue dye using CSNP-SiO_2 Nano-composite at different adsorbent dosages

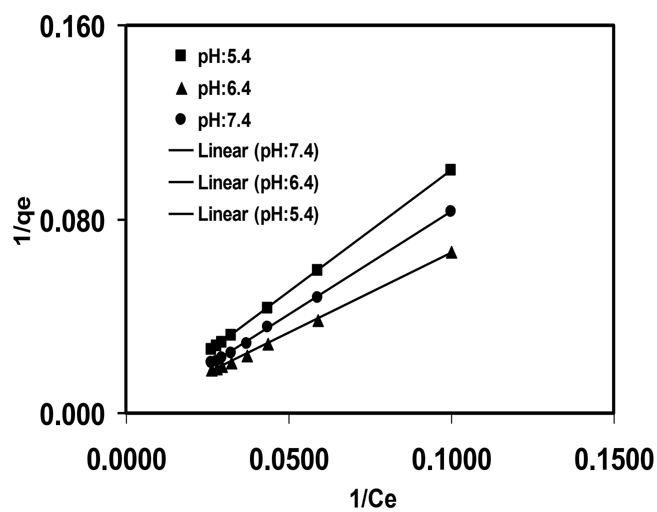

Fig. 5: Langmuir isotherm for the adsorption of Methylene blue dye using CSNP-SiO_2 Nano-composite at different pH

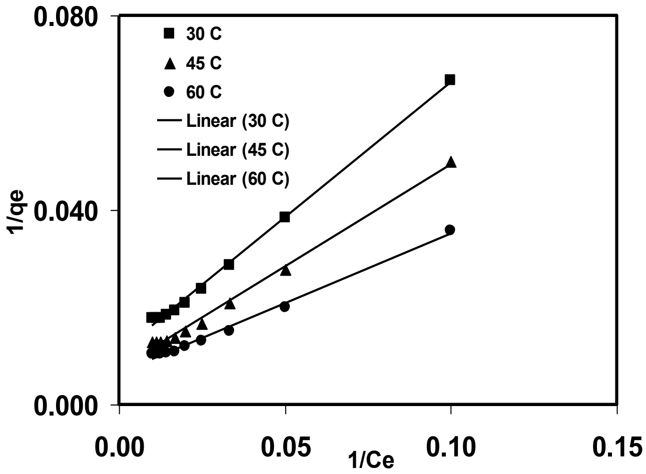

Fig. 6: Langmuir isotherm for the adsorption of Methylene blue dye using CSNP-SiO_2 Nano-composite at different Temperatures

The values are calculated from the slope and intercept of the different straight line representing the different sorbent dosages, pH and temperature (b) energy of adsorption and (k) adsorption capacity and Q_0 is represented by (K). The Langmuir isotherm constant (Q_0) in the above equation is a measure of the amount of dye adsorbed when the monolayer is completed. Monolayer capacity (Q_0) of the adsorbent for the dye is comparable as obtained from the adsorption isotherm. The observed statistically significant (at the 95% confidence level) linear relationship as evidence of these by the R^2 values (close to unity) indicate the applicability of the isotherm (Langmuir isotherm) and surface. The Langmuir isotherm constants along with correction coefficients are reported in table 1. It is also clear from the shape of the adsorption isotherm, that it belongs to the L_2 category of isotherm, which indicates the normal (or) Langmuir type of adsorption. Such isotherms are often encountered when the adsorbate has a strong intermolecular attraction for the surface of the adsorbent [51].

The L_2 shape of the isotherm observed in the present case implies that malachite green dye molecules must have been strongly attached to the CSNP-SiO_2 Nanocomposite.

3.3. Freundlich isotherm

Freundlich isotherm [52] is used for heterogeneous surface energies system. The sorption isotherm is the most convenient form of representing the experimental data at different adsorbent dosage, temperature and pH as shown in fig. 7-9. Moreover, the figs. show the batch isothermal data fitted to the linear form of the Freundlich isotherm [53].

$$q_e = K_F C_e^{1/n}$$

$$\ln q_e = \ln K_F /(1/n) \ln C_e$$

The various constants, associated with the isotherm are the intercept, which is roughly an indicator of sorption capacity (k_f) and the slope (1/n) sorption intensity values are recorded in fig.7-9.

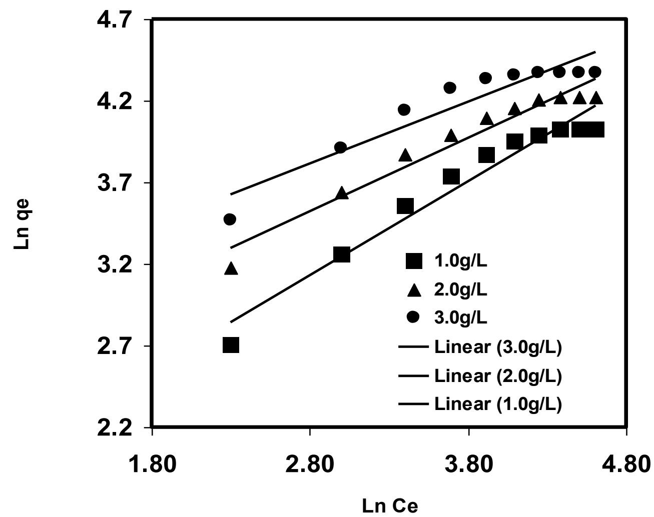

Fig. 7: Freundlich isotherm for the adsorption of Methylene blue dye using CSNP-SiO_2 Nano-composite at different adsorbent dosages

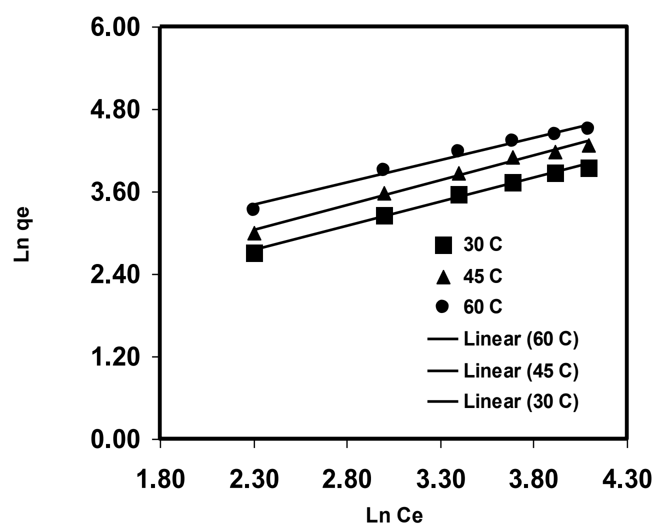

Fig. 8: Freundlich isotherm for the adsorption of Methylene blue dye using CSNP-SiO_2 Nano-composite at different pH

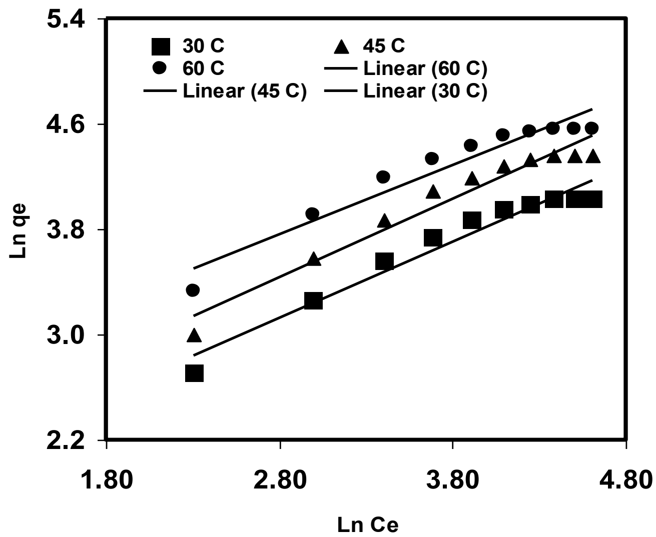

Fig. 9: Freundlich isotherm for the adsorption of Methylene blue dye using CSNP-SiO_2 Nano-composite at different temperatures

Freundlich isotherm has been illustrated to be a special case of heterogeneous surface energies and it can be easily extended to this case. It has been stated [54] that the magnitude of the exponent 1/n indicates the favourability and capacity of the adsorbent system. The values n>1 represent favorable adsorption conditions. In most of the cases the exponent between 1<n<10 shows the beneficial adsorption as shown in table 1.

The adsorption of malachite green dye from an aqueous solution using CSNP-SiO_2 Nanocomposite has been investigated under different reaction conditions in batch and equilibrium mode. The fitness of the Langmuir model in the present system shows the formation of monolayer coverage of the adsorbate at the outer space of the adsorbent. Freundlich model isotherm was analyzed. The monolayer adsorption capacity determined was reasonably high (g/L) at adsorbent dosage 58.79 (g/L), Temperature 57.70 (g/L), and pH 18.53 (g/L) for adsorption of (MB) dye respectively. The monolayer adsorption capacity was determined to be 22.86 to 53.82 mg/g. The values of dimensionless equilibrium parameter like separation factor (R_L) at different dosages, pH and temperature indicates the favorability of the process described in the present study.

Table 1: Langmuir and Freundlich isotherm constants at different Adsorbent dosages, pH and Temperatures (Methylene blue dye)

Adsorbent Dosage (g/L)	**Langmuir Isotherm -model**	**Freundlich Isotherm -model**
1.0	K= 518.79; b= 1.80; R^2= 0.9971	K_F=2.848; n= 0.350; R^2= 0.8469
2.0	K=28.14; b= 3.26; R^2= 0.9981	K_F= 2.256; n= 0.44;R^2= 0.8585
3.0	K= 22.33; b=4.71;R^2= 0.9874	K_F=1.5116; n= 0.66; R^2= 0.8754
pH	Langmuir Isotherm-model parameters	Freundlich Isotherm -model parameters
5.4	K= 18.53; b= 1.40; R^2= 0.9915	K_F=1.818; n= 0.53; R^2= 0.9814
6.4	K= 132.72; b=1.51 R^2= 0.9991	K_F=1.379; n= 0.73; R^2= 0.9901
7.4	K= 405.67.; b= 1.17;R^2= 0.9921	K_F= 1.1308;n= 0.88; R^2= 0.9939
Temperature (°C)	Langmuir Isotherm -model parameters	Freundlich Isotherm -model parameters
30	K= 51.88; b= 1.80; R^2= 0.9901	K_F=2.291; n= 0.444; R^2= 0.9328
45	K= 57.70; b= 2.37; R^2= 0.9957	K_F = 1.778; n= 0.562; R^2= 0.9492
60	K= 43.82.0;b= 3.51; R^2= 0.9929	K_F = 1.5116; n= 0.662; R^2= 0.9523

4. CONCLUSION

The CSNP-SiO_2 nanocomposite was used for the degradation of methylene blue. The low molecular weight of CS was regarded as toxic comparing the high molecular weight. The effect of various parameters for the adsorption of dye was also studied in this research. The results depicted that the nanocomposite can be used as a good adsorbent for the removal of industrial dyes. The adsorption process is dependent on CSNP-SiO_2 nanocomposite dosages, pH, and temperature. The adsorption parameters for Freundlich and Langmuir isotherms were determined. The result was very well described by Langmuir and Freundlich isotherm models. Finally, it is concluded that the adsorbent CSNP-SiO_2 nanocomposite could be a good alternative for the removal of methylene blue from an aqueous solution very effectively and economically.

5. ACKNOWLEDGEMENTS

Amutha Eswaran (Register No: 19214542052003) acknowledges the research center Sri Paramakalyani Centre for Excellence in Environmental Science, Manonmaniam Sundaranar University, Alwarkurichi for providing the support for this research work.

Conflict of Interest

The Authors did not declare any conflict of Interest

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