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Original Research Article

Gel Beads (Orbeez balls) as an Adsorbent for Removal of Direct Yellow Dye from Aqueous Solutions

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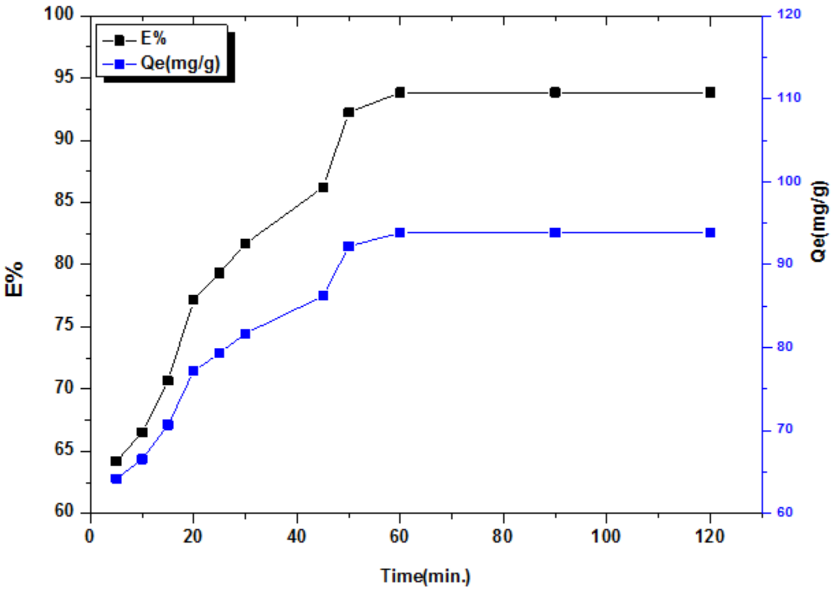
ABSTRACT

The study presents of Synthetic gel beads (Orbeez balls), specifically targeting their application in the adsorption of Direct yellow (DY) dye. Advanced characterization techniques like FTIR, FESEM, and TEM were employed, revealing significant findings. FTIR analysis showed changes in band intensities after dye adsorption, indicating gel beads -dye interactions. FESEM analysis revealed a more porous surface post after adsorption, enhancing adsorption efficiency. TEM corroborated the uniform distribution of gel beads. The study also explored the impact of gel beads on dye adsorption, identifying an optimal concentration for maximum capacity. An inverse relationship between adsorbent surface weight and dye adsorption per unit weight was observed, underlining the importance of surface weight in dye removal efficiency. The equilibrium time for DY dye adsorption was found to be around 2 hr, with a rapid initial adsorption rate that slowed over time. The study also found that adsorption efficiency decreases with increasing pH. The maximum adsorption capacity (Qe mg/g) determined for gel beads was determined to be 110.56 mg/g. increase adsorbent dosage increase removal percentage (E %) of 64.76% to 95.9 %, but decrease adsorption capacity (650.76 mg/g to 95.66 mg/g), for gel beads of DY dye. Adsorption isotherms indicate a multilayer process on a heterogeneous surface.

GRAPHICAL ABSTRACT



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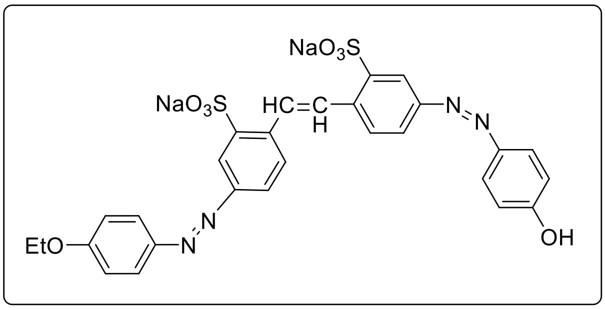
Introduction

In the context of escalating water pollution, primarily driven by population growth and industrialization, a critical challenge is identified: the contamination of water by organic dyes, notably prevalent in various industries such as textiles, paper, and leather. The focus is on Direct yellow (DY) dye, a commonly used industrial dye, with diverse applications in dyeing fabrics like cotton, wool, silk, and nylon, as well as in producing paper printing inks. However, DY dye is associated with significant drawbacks. DY dye in water causes digestive and respiratory irritation, nausea, vomiting, and skin ulcers, endangering environmental and human health [1-3]. Therefore, the most important methods for simultaneous disposal and removal of toxic substances are reverse osmosis, sedimentation, filtration, co-filtration, solvent extraction, flocculation, ion exchange, and oxidation, to remove harmful effects and industrial dyes from wastewater. However, these techniques and methods have inherent disadvantages of being economically infeasible, time consuming, expensive, ineffective removal, etc., leading to secondary pollution [4-8]. Therefore, the adsorption process is considered the most attractive and desirable method for removing industrial dyes from aqueous solutions because it is simple, inexpensive, effective, economical and versatile. Different adsorbents like activated carbons, clays, metal oxides, and gel beads s have been reported for adsorption of DY dye. Since gel beads s are newer entrants with multifunctional groups, great surface area, improved swelling, and best adsorption efficiency, that makes them excellent adsorbents for waste water [9-13].

Direct yellow (DY 12), is an Azo dye (diazo) usually utilized for cotton dyeing and also in leather and paper industries, due to its stability towards both alkali and acidic solutions being an ethylated product. Chemical formula: $C_{30}H_{26}N_4Na_2O_8S_2$, and the chemical structure is shown in Scheme 1 [14].

In the work, Synthetic gel beads (Orbeez balls) utilized as an effective adsorbent for the adsorption of basic dye alike DY dye. The properties of structural gel beads have been estimated by several analytical instrumentation techniques like FESEM, FTIR, XRD. The effects of equilibrium time, pH, adsorbent dose, and concentration of MB dye have been studied and Thermodynamics parameter has been analyzed. The adsorption efficiency has been estimation via the adsorption isotherm like Freundlich and Langmuir isotherm models.



Scheme 1. Chemical structure of DY 12 [15].

Experimental

Instruments and chemicals

UV-Visible spectrophotometer, FTIR, and Thermo gravimetric Analyzer were used in this work. TEM and FESEM were also used. Similarly, electric shakers were used in the investigation. High-purity chemicals included Hydrochloric Acid, Sodium Hydroxide, and Direct yellow. All these compounds came from Sigma Aldrich.

Synthetic gel beads (Orbeez balls)

Synthetic gel beads (Decorative beads), or so-called (Orbeez balls), were obtained from local markets in Hilla-Iraq, of Chinese origin. Each box contains 100 small balls.

Calibration curves of dY dye and determination of λ_{max}

DY dyes were tested for the maximum wavelength (λ_{max}) utilizing standard solutions concentration 1000 mg/L. A UV-Visible spectrophotometer captured 200-800 nm wavelengths utilizing a 1.0 cm quartz cell. DY dye absorption peaked at 430 nm. At this wavelength, DY dye has the maximum absorption in the UV-Vis spectrum (λ_{max}). This wavelength must be correct for accurate spectrophotometric analysis of this dye in diverse solutions. For the DY dye calibration curve, solutions with concentrations from 10-100 mg/L were generated. These solutions' absorbance was measured at the dye's maximum wavelength (λ_{max} = 430 nm) using a UV-Visible spectrophotometer. Figure 1 displyas the DY dye calibration curve, calculated via graphing absorbance vs. concentration.

Effect amount of gel beads

To study the effect of several the mass of the adsorbent in the adsorption method, 100 mL of DY dye solutions 100 mg/L, were included in several mass of the gel beads (varying from 0.01-0.15 g). These blends were then placed in a shaker at 25 °C for 1 hr at a rate of 130 rpm to achieve equilibrium. The absorbance was determined utilizing a UV-Visible spectrophotometer for the DY dye.

Equilibrium time for adsorption method

To check out the time it takes for the gel beads and adsorbate to get to equilibrium, a mass of gel beads 0.1 g, of 100 mg/L, and 100 mL for solution of DY dye. The time aspect was the only variable in this study. These solutions were placed in a shaker for stirring durations ranging from 5-120 min. The absorbance was then measured utilizing a UV-Visible spectrophotometer.

Effect of pH

A 0.1 g of the gel beads was added to a 100 mg/L dye solution at varied acidity function values (2-11) at 25 °C for 2 hr to study the influence of acidity function on adsorption adjusting the acidity function with 0.1 M HCl and NaOH solutions. To determine its effect on adsorption, the amount of adsorbed material was plotted against the acidity function value.

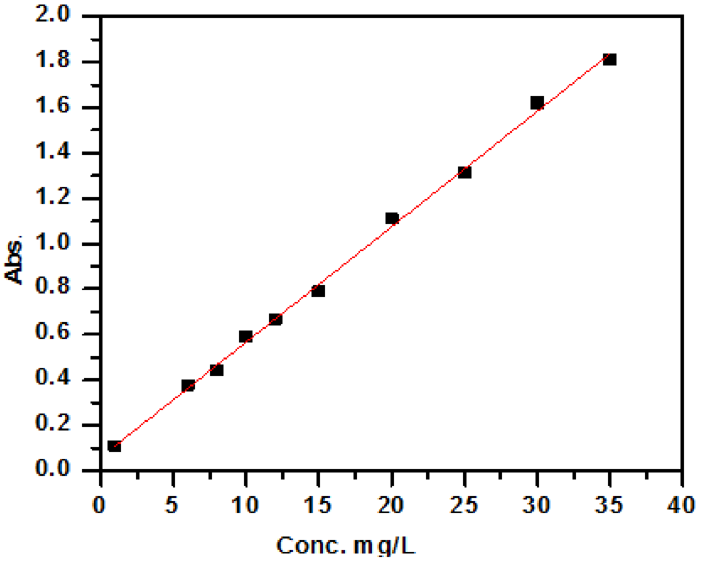


Figure 1. Calibration curve of DY dye in aqueous solution.

Results and Discussion

The gel beads prepared before and after adsorption of the DY dye were analyzed utilizing FT-IR spectrum to identify the positions of the bands in the spectrum of each of the prepared materials. The infrared spectrum showed the presence of stretchy bands and curvature within a range between (500-4000 cm^{-1}) [16].

The infrared spectra of gel beads, as shown in Figure 2, revealed variations in the intensity, width and shift of the bands including the disappearance and emergence of new bands. In the FTIR spectrum of gel beads, the characteristic hydroxyl group appears at 3440 cm^{-1}, overlapping with the NH group. For the gel beads, the band at 1728 cm^{-1} corresponds to the C=O bending vibration in hydrogel, while the bands at 1550 cm^{-1} and 1396 cm^{-1} are attributed to the asymmetric and symmetric stretching of COO^- groups, respectively. In addition, the band at 2962 cm^{-1} aligns with the carboxyl group associated with gel beads. Through the FT-IR spectrum after the adsorption of the DY dye were observed. These changes included variations in the intensity of bands, the emergence and disappearance of some bands, or their shift [17,18].

The Field Emission Scanning Electron Microscopy (FE-SEM) technique is used to study the gel beads characteristics of the before and after adsorption. This technique provides insights into the particle shape, their aggregation nature, crystalline structure, and surface area. It also helps in determining the surface's porosity or smoothness and the homogeneity of the gel beads distribution on the surface.

The FE-SEM images, as shown in Figures 3a and 3b, reveal that the surface of the gel beads is smooth, clear, and flaky. It also possesses a porous structure resembling a sponge and a network of tightly packed layers. The surface also contains many irregularly clustered wrinkles. However, after adsorption the morphological appearance of the sample surface changes significantly. There is an increase in the surface roughness and porosity with an irregular structure, Van der Waals forces binding the gel beads layers together [19-21].

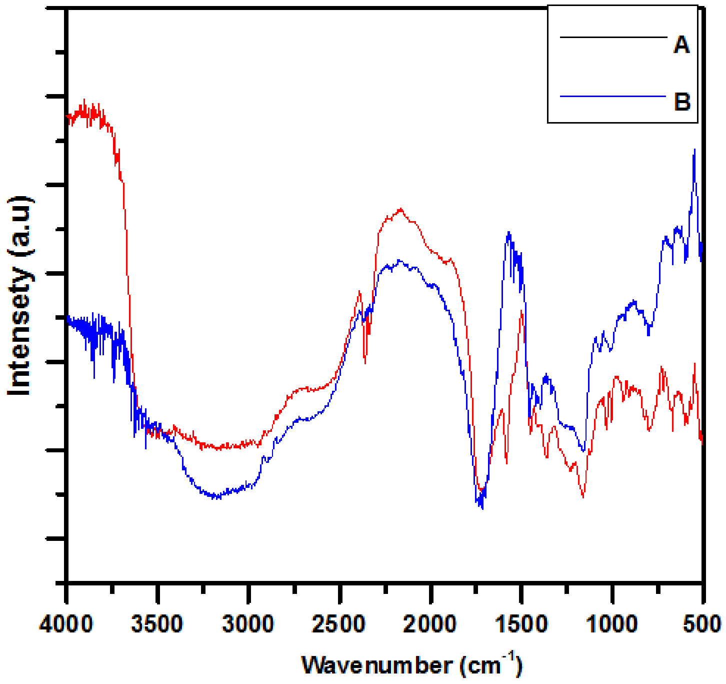


Figure 2. FTIR Spectrum of A) gel beads and B) after DY dye adsorption.

TEM

TEM images of gel beads showed formations of clusters with a uniform distribution and are arranged regularly, with the presence of some agglomerations compatible with the polymeric matrices due to the availability of hydrophilic functional groups, which enhance strong H-bonding interactions (Figure 4) [22].

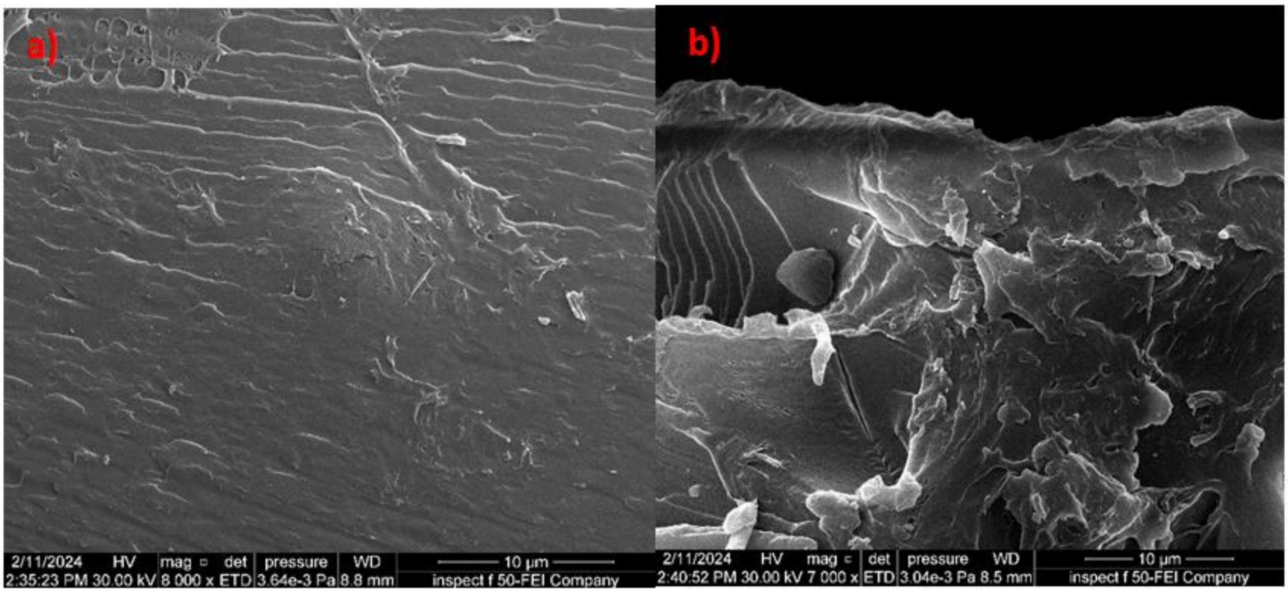


Figure 3. FESEM images gel beads (a) before; (b) after adsorption of DY dye.

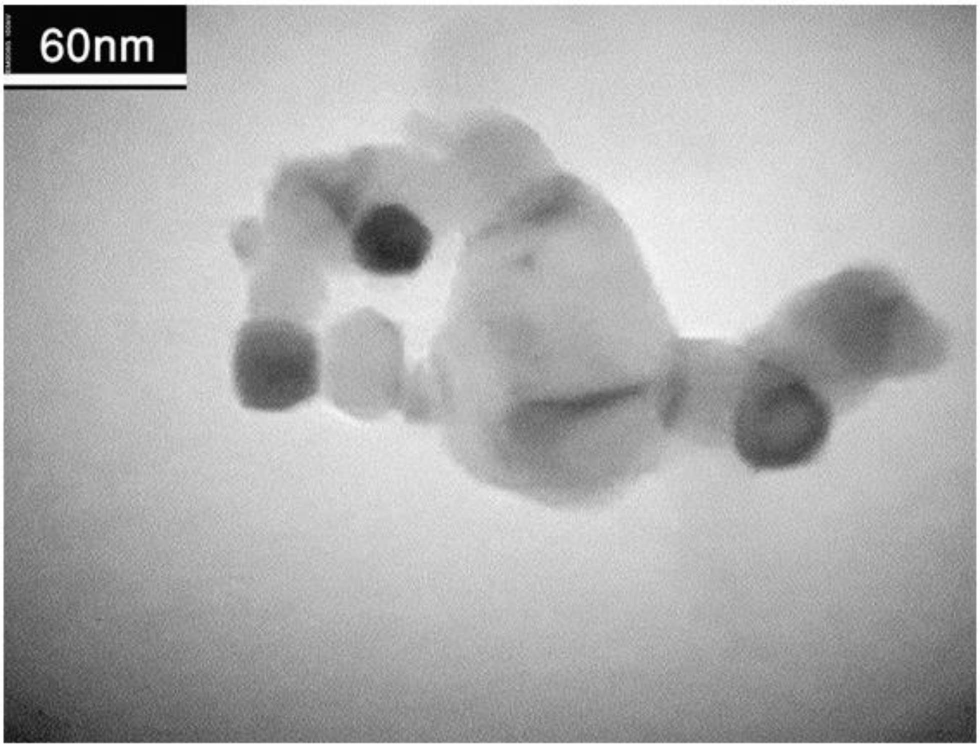


Figure 4. TEM images of gel beads.

Equilibrium time

The time required to reach equilibrium for DY dye at a concentration of 100 mg/L was studied at different time intervals ranging from 1 to 240 minutes, at 25 °C, pH=7.0, weight of gel beads 0.1 g. Figure 5 shows that the time required to reach equilibrium for DY dye using gel beads as an adsorbent surface is 1 hr [23]. The adsorption process increases with increasing time, starting rapidly at the beginning of the reaction and gradually reaching equilibrium. This is attributed to the active sites on the adsorbent surface being initially available for adsorption until they become occupied by dye molecules, and then the adsorption process slows down and becomes more challenging because the dye molecules have occupied all the active sites on the surface. The percentage of dye removal reached its highest level at the recorded equilibrium time, reaching a value of 95.86% [24-26].

Effect of weight of gel beads

The results shown in Figure 6 indicate that the removal efficiency E% of the adsorbent increases with an increase in its weight. This increase in efficiency is attributed to the enlarged surface area and, consequently, a greater number of active sites available for adsorption. However, it's important to note that while the removal efficiency increases, the adsorption capacity of substance adsorbed per unit weight of the adsorbent decreases [27]. This decrease in adsorption capacity with weight is due to the presence of unsaturated adsorption sites, meaning there are still available sites for adsorption. This reduction in surface area leads to a decrease in the number of adsorbate molecules per surface area when the mass of the adsorbent is increased. The preferred weight for adsorption in these experiments is identified as 0.1 g, which seems to provide an optimal balance between the surface area and the availability of active sites [28].

Effect of concentration of DY dye

The gel beads was used to removal initial DY dye concentration. The amount of adsorption required to remove DY dye initial concentration and the number of sites available on the adsorbent surface. Concerning the effect of removal concentration DY dye by gel beads, Figure 7 displays the removal efficiency versus of DY dye initial concentration.

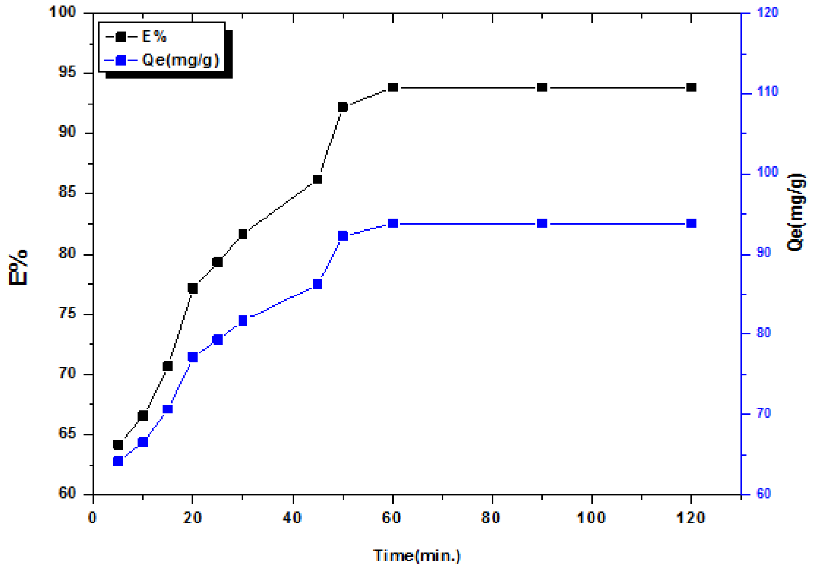


Figure 5. Effect of equilibrium time on the adsorbent amount and percentage of dye removal from gel beads.

The reduction in adsorption caused by a shortage of accessible active sites causes the drug clearance percentage to decline as concentration of DY dye rises. As drug uptake resistance reduces as DY dye concentration rises, the adsorption capacity ((Qe) mg/g) is proportional to initial dye concentration. Due to an increase in driving force, the adsorption rate likewise rises as the dye's starting concentration does [29].

Effect of pH

The study effect of pH on the adsorption of DY dye onto gel beads at a concentration of 100 mg/L was conducted over a pH (2-11), at 25 °C. Figure 8 demonstrates that the amount of dye adsorbed decreases with an increase solution pH. This decrease in adsorption at higher pH levels can be attributed to the surface charge characteristics of the adsorbent [30].

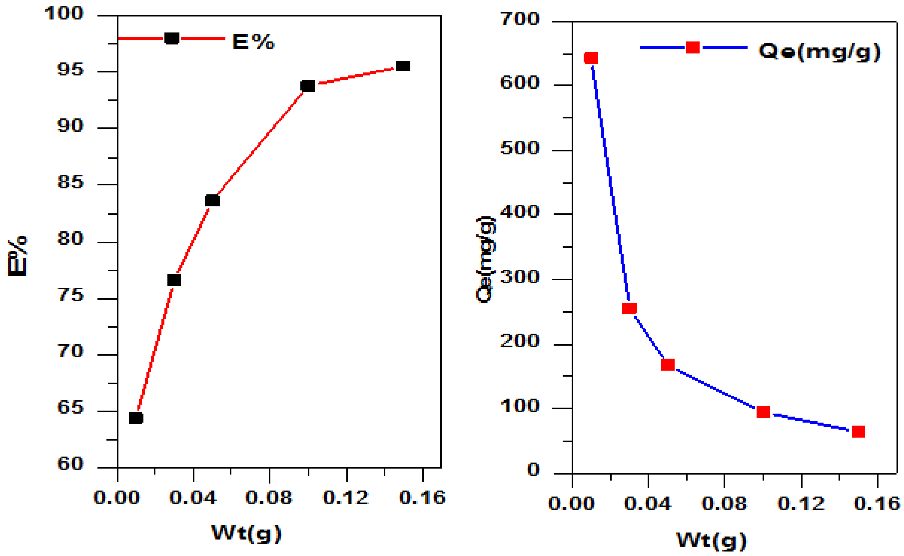


Figure 6. Effect of weight gel beads on the adsorption of DY dye

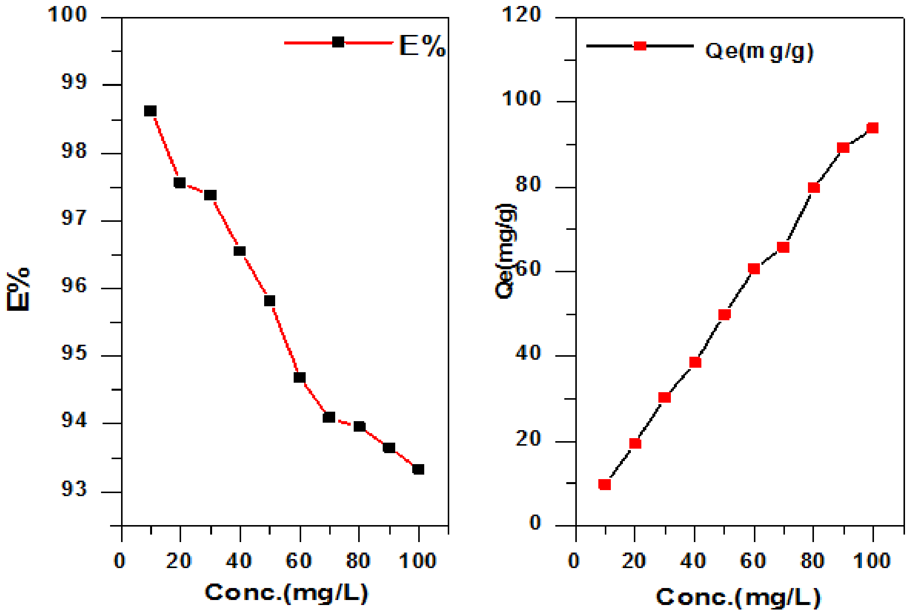


Figure 7. Effect of initial dye concentration DY dye onto gel beads.

As the pH increases, the active sites on the surface of the gel beads become negatively charged. This change in charge leads to electrostatic repulsion between the hydroxide ions (OH^-) present in the solution and the dye molecules at the adsorbent's active sites. The increase in pH results in a higher concentration of OH- ions, which compete with the negatively charged dye molecules for adsorption sites. This competition and the repulsion between similarly charged entities reduce the overall adsorption efficiency [28,31-32].

Adsorption isotherm study

In sorption equilibrium studies, two isotherm models are utilized, notably the Langmuir, Freundlich, models, as described by **Equations** 3 and 4.

$$q_e = \frac{q_m K_L C_e}{1+K_L C_e} \tag{3}$$

$$q_e = k_f \, C_e^{\frac{1}{n}} \tag{4}$$

Where, q_e is a quantity adsorbed per unit mass of adsorbent at equilibrium [mg/g], C_e is equilibrium concentration of adsorbate in solution after adsorption [mg/L], K_f: Empirical capacity factor or Freundlich constant [L/mg] or the dye amount adsorbed for unit equilibrium concentration, $1/n$: Freundlich exponent, if the value of n is equal to unity, the adsorption is linear; if below to unity, then adsorption process is chemical and if the value is above unity, then adsorption is a physical process.

The Langmuir isotherm, as shown in Figure 10, assumes a finite number of uniform active sites on the adsorbent surface, leading to the formation of a monomolecular layer with minimal intermolecular interactions. Conversely, the Freundlich isotherm, also presented in Figure 9, is applicable to heterogeneous surface adsorption [31]. It implies a direct proportionality between adsorbate concentration and adsorbent quantity on the surface, coupled with a decrease in sorption energy upon saturation of the adsorbent's sorption centers. To assess the dye-nanocomposite system, the coefficient of determination (R^2) was computed by fitting the experimental data to these isotherms. Figure 9 and Table 1 illustrate that the Langmuir isotherm provides a more robust correlation, evidenced by the highest R^2 value of 0.93833 [32].

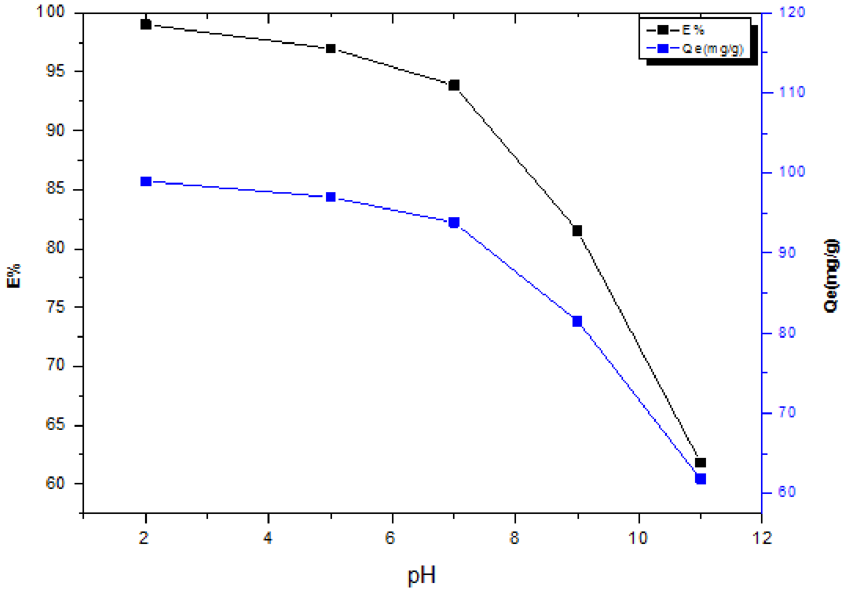


Figure 8. Effect of pH solution onto DY dye by using gel beads.

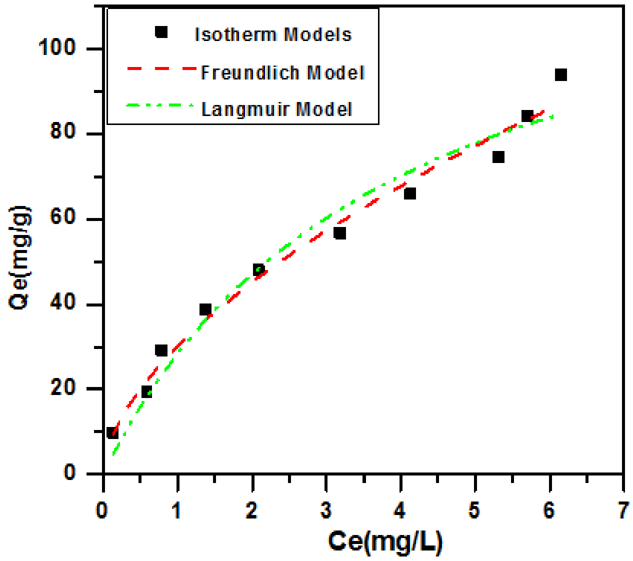


Figure 9. Non-linear fit of different adsorption isotherm models for adsorption of DY on nanocomposite, conc. = 100 mg/L, Temp. = 25 °C, contact time 1 h, and mass of adsorbent 0.1 g/L).

Table 3. Several factor isotherm for the adsorption study of DY dye onto gel beads.

Gel beads			
Freundlich	**K_f**	30.098	1.7533
	1/n	0.5866	0.0378
	R^2	0.9888	
Langmuir	**q_m (mg/g)**	137.836	17.9356
	K_L (L/mg)	0.2598	0.06966
	R^2	0.9615	

Conclusion

Removal of pollutant DY dye from aqueous solutions by adsorption with gel beads surfaces has been experimentally determined. The best results have been found at 25 °C, and adsorbent dosage 0.1 g of gel beads for both studying adsorption efficiency Q_e (mg/g) and percentage removal E%. The percentage removal E% and adsorption capacity as well as the percentage of DY dye increase with increasing contact time, surface area. However, adsorption efficiency Q_e (mg/g) has decreased with the increase of adsorbent dosage. The optimum contact time for equilibrium to be achieved is found to be 1 hr. The study also found that adsorption efficiency decreases with increasing pH. The maximum adsorption capacity (Q_e mg/g) determined for gel beads was determined as 110.56 mg/g.

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