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Original Article

Preparation of Sodium Alginate/ Acrylamide/ 2-Hydroxyethyl Methacrylate Based Hydrogel by Gamma Radiation and its Application for the Removal of Dye from Waste Water

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Abstract - *Three-dimensional networks of hydrophilic polymers that have a high capacity to absorb water are called hydrogels. Investigation on the removal of monovalent cationic dyes, such as Methylene Blue, from aqueous solutions using sodium alginate/acrylamide/2-hydroxyethyl methacrylate hydrogel was undertaken in order to find a solution for the environmental waste water problem. NaAlg/AAm/HEMA blend hydrogels are made without the use of an external cross-linker by applying gamma radiation from a Co-60 source. The effects of factors like HEMA concentration and radiation dose on the produced hydrogels' (NAH hydrogel's) gel content and swelling behaviour were carefully examined. 25 kGy was selected as the optimal dose, and 2% HEMA was selected as the optimal HEMA content based on the gel fraction and swelling ratio. FTIR was used to characterize the hydrogel that was produced. This hydrogel was used to investigate the kinetics and isotherms of methylene blue dye clearance. With maximum dye adsorption of 219.65 mg/g, the adsorption behavior of NaAlg/AAm/HEMA hydrogel was pseudo-second-order and closely matched the Freundlich isotherm model. For MB adsorption, thermodynamics and pH analyses were performed. At high pH (9) and at high temperature (333 K), the adsorption was greater. This hydrogel shows promise in treating wastewater that contains MB dye solution.*

Keywords - *Hydrogel, Methylene blue, Gamma radiation, Swelling ratio, Dye Adsorption.*

1. Introduction

Large amounts of colored effluents are released into wastewater by the textile, dyeing, printing, cosmetics, food coloring, and papermaking industries [1]. Approximately 7×105 tons of different dyes are produced annually, and more than 10,000 dyes are commercially accessible [2]. An estimated 12% of the dyes produced annually are released as effluents by textiles, industrial processes, and associated industries [3]. Aquatic life and the food chain can suffer greatly from even minute levels of dyes discharged into streams. The dye can induce allergic reactions, dermatitis, and skin irritation. In humans and aquatic life, some dyes can potentially cause cancer and mutagenesis [4]. Nonetheless, most dyes are resistant to both photo-degradation and bio-degradation [5-7]. As a result, wastewater containing dyes must be cleaned. Numerous treatment methods, such as coagulation and flocculation [8], membrane separation [9], oxidation or ozonation [10, 11], electro-coagulation [12], and adsorption, have been used to date to eradicate dyes. Among these, adsorption has been found to be a somewhat simple, affordable, efficient, and potentially useful method for getting rid of dyes and other contaminants [13–14]. A wide range of adsorbents has been developed and applied to eliminate color from aqueous effluents. Many adsorbents have two main drawbacks: low dye adsorption capability and secondary contamination from the adsorbing components. [15]

Modern technologies that are not only affordable but also highly efficient in removing impurities must be used to develop wastewater purification methods. Recently, it has been shown that hydrogels can absorb pollutants from contaminated water [16]. Hydrogels are a special kind of super absorbent that can absorb water hundreds of times its dry weight. They consist of networks of cross-linked polymeric chains in three dimensions that are connected by physical or covalent bonds. They have garnered a lot of attention recently because of their many uses [17–18]. Hydrogel has numerous uses, including drug delivery systems, tissue engineering, self-healing materials, hemostasis bandages, artificial organ construction, biosensors, sorbents, and the removal of dyes and heavy metals from wastewater. Hydrogel's high swelling ratio, hydrophilicity, and biocompatibility set it apart from other smart materials. Hydrogel is currently the subject of numerous investigations, with a focus on its use across various areas [19–22]. A recent study examined the use of a hydrogel nanocomposite to remove methylene blue from wastewater [23]. Super-adsorbent hydrogels were prepared and used in another investigation to remove methylene blue from aqueous solution [24]. Removal of Methylene Blue Dye Using Biodegradable Superabsorbent Hydrogel Polymer Composite Incorporated with Activated Charcoal [25] is another example of a study on the removal of methylene blue. The design of composite hydrogels for the treatment of wastewater and water contaminated with dyes is the subject of another review [26]. The current work describes the creation of a previously unreported new adsorbent from Sodium alginate/Acrylamide/2-Hydroxyethyl methacrylate. It is also a good idea to make hydrogel utilizing gamma radiation rather than a hazardous cross-linker.

In this work, gamma radiation from a Co-60 source was used to create Sodium alginate/Acrylamide/2-Hydroxyethyl methacrylate mix hydrogels without the use of an external cross-linker. The effects of factors like HEMA concentration and radiation dose on the produced hydrogels' (NAH hydrogel's) gel content and swelling behaviour were carefully examined. 25 kGy was selected as the optimal dose, and 2% HEMA was selected as the optimal HEMA content based on the gel fraction and swelling ratio. FTIR was used to characterize the hydrogel that was produced. This hydrogel was used to investigate the kinetics and isotherms of methylene blue dye clearance. With maximum dye adsorption of 219.65 mg/g, the adsorption behavior of NaAlg/AAm/HEMA hydrogel was pseudo-second-order and closely matched the Freundlich isotherm model. For MB adsorption, thermodynamics and pH analyses were performed. At high pH (9) and at high temperature (333 K), the adsorption was greater. This hydrogel shows promise in treating wastewater that contains MB dye solution.

2. Experimental

2.1. Preparation of Sodium alginate (NaAlg)/ Acrylamide (AAm)/ 2-hydroxyethylmethacrylate (2-HEMA) hydrogels

The radiation cross-linking approach was used to prepare the hydrogel. The necessary amount of sodium alginate was dissolved at 80 ± 5°C for 45 minutes while being constantly stirred at 200 rpm in order to create an aqueous solution of 2% sodium alginate (w/w). Once the NaAlg solution had cooled to room temperature, it was placed in a beaker and combined with 2% HEMA aqueous solution and 8% AAm aqueous solution. AAm (8%) and NaAlg (2%) concentrations were set before varying percentages of HEMA (2%, 4%, 6%, and 8%) were added to the solutions in order to assess the impact of HEMA on the network characteristics. The combinations were then put in glass tubes after being poured into polyethylene plastic bags and sealed. The samples were then exposed to gamma rays from a ^{60}Co gamma source (a 90 kCi Batch Type Panoramic Irradiator purchased from BRIT, India) at radiation dosages of 10–30 kGy in the air at room temperature. The dosage rate was 6.8 kGy/h, and the irradiator activity was 68.63 kCi. The resulting cylindrical hydrogels were then divided into tiny pieces, allowed to air dry, and then heated at 45°C until their weight remained constant. Radiation crosslinking is beneficial because the chemical approach of hydrogel preparation necessitates the use of a hazardous crosslinking agent.

2.2. FTIR Analysis

Using a Fourier Transform Infrared Spectrometer (model: Shimadzu FT-IR-8400S, Japan), FTIR spectra of NaAlg/AAm/HEMA hydrogels were captured. To create clear disks, the dried samples were ground with KBr and then compressed under high pressure. The disks were then put in the sample holder to record the spectrum. 4000-800 cm^{-1} was the range of wave numbers.

2.3. Determination of gel fraction

To extract the soluble fraction, the constant-weight dried gel samples were stored at room temperature for 24 hours in a beaker filled with water. The gel samples were then dried in an oven set to 45°C until they reached a consistent weight. The samples' gel fractions were computed as follows.

$$\text{Gel fraction (\%)} = (W_1/W_0)*100 \tag{1}$$

Where W_1 is the weight of the dried gel after extraction and W_0 is the initial weight of the dried gel.

2.4. Determination of Swelling Properties

At room temperature, the dried gels were submerged in distilled water and let to swell. After using filter paper to remove extra surface water, the swelling gels were periodically removed and weighed. Until there was no more weight gain, the process was repeated. The following formula was used to determine the swelling ratio (SR):

$$\text{Swelling ratio \%} = (W_t - W_d)/W_d * 100 \tag{2}$$

Where W_d is the weight of dried gel before swelling and W_t is the weight of the swollen gel at time t.

2.5. Determination of Dye Adsorption

The dye was dissolved in distilled water to create a water-soluble dye (Methylene blue) solution. Hydrogels were introduced to dye solutions. Periodically, the amount of dye that was left in the aqueous solution was measured using UV-visible spectroscopy (UV- 1800 Series, Shimadzu, Japan). The following formula yields the equilibrium adsorption capacity, qe (mg/g).

$$q_e = \frac{(C_0 - C_e)}{W}\, v \qquad (3)$$

Where C_o and C_e are the concentration of dye in the initial solution and aqueous phase after treatment of a certain period of time, respectively (mg/L), V is the volume of solution (L), and W is the weight of the hydrogel (g).

3. Results and Discussion

3.1. Synthesis of Hydrogel

NaAlg, AAm, and HEMA were used in the gamma radiation approach to create hydrogels without the use of an initiator or cross-linker.

The detailed procedure of preparation of PSD blend hydrogel is shown in the following (Figure 1).

3.2. Optimization of Sodium Alginate / Acrylamide/ HEMA Hydrogel Composition

With the NaAlg and AAm contents set at 2% and 8%, respectively, the HEMA composition was adjusted between 2 and 8% to improve the hydrogel composition (Table 1). Hydrogels were created with a dosage of radiation (30kGy). Next, the effect of HEMA concentration on the gel fraction and swelling ratio were measured.

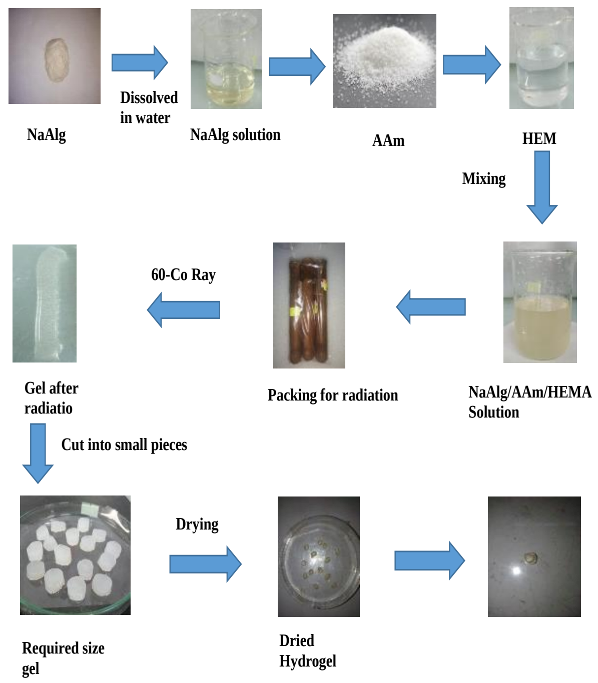

Fig. 1 Preparation of NaAlg/AAm/HEMA hydrogels by gamma radiation

Table 1. Feed composition at a radiation dose of 30 kGy for the prepared hydrogels

Sample	NaAlg %	AAm %	HEMA %
NAH2	2	8	2
NAH4	2	8	4
NAH6	2	8	6
NAH8	2	8	8

3.2.1. Effect of HEMA on Swelling Ratio

The hydrophilicity of the polymer chains determines the hydrogel's swelling ratio. Prepared hydrogels exposed to 30 kGy radiation were examined for swelling behaviour. Over time, the swelling ratio increased. The produced hydrogel first inflated quickly in water before eventually reaching equilibrium values after about 26 hours. The impact of HEMA concentration on swelling ratio is depicted in Figure 2. The swelling ratio of the NAH blend hydrogel was shown to rise as the amount of HEMA increased from 2% to 8%.

3.2.2. Effect of HEMA Concentration on gel Fraction

The portion of a polymer that has not dissolved is called the gel fraction. It is evident from Figure 3 that when the concentration of HEMA increases, the gelation exhibits a little decreasing trend. The gel percentage dropped to 78.75% from 84.46%.

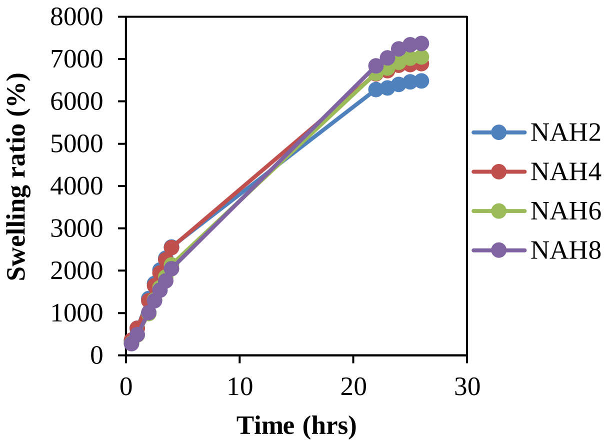

Fig. 2 Effect of HEMA concentration on a swelling ratio of NAH hydrogels

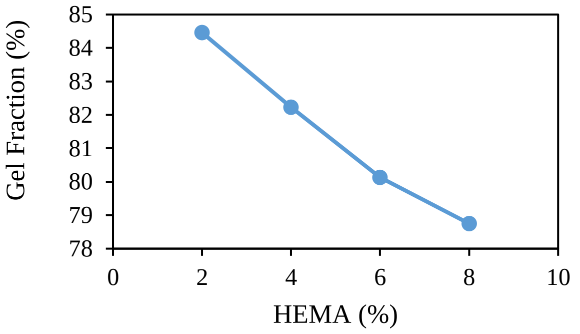

Fig. 3 Effect of HEMA content on gel fraction of NAH hydrogels

NAH2 hydrogel was selected as the ideal composition because, despite the fact that adding more HEMA enhanced the swelling ratio, the gel became extremely soft, thick, and challenging to work with. Thus, NAH2 hydrogel was chosen for additional research.

3.3. Effect of Radiation Dose on Properties of NAH2 Hydrogel

The effect of radiation dose on the swelling ratio and gel fraction was studied.

3.3.1. Effect of Radiation Dose on Swelling Ratio of NAH2 Hydrogel

Different radiation doses, such as 20 kGy, 25 kGy, and 30 kGy, were used to create NAH2 hydrogels. Figure 4 illustrates how the swelling ratio falls as the radiation exposure rises.

3.3.2. Effect of Radiation Dose on gel Fraction of NAH2 Hydrogel

The hydrogel (NAH2) gel fractions rose as radiation doses increased, as seen in Figure 5, reaching their maximum gel fraction of 88.78% at 30 kGy. Figure 5 illustrates the gel fraction's increasing trend from 20 to 30 kGy. The concentration of free radicals in the monomer/polymer system rises as the radiation exposure does. Since cross-linking is known to be caused by free radicals, an increase in radiation dose causes more crosslinking between polymer chains, which raises the gel fraction.

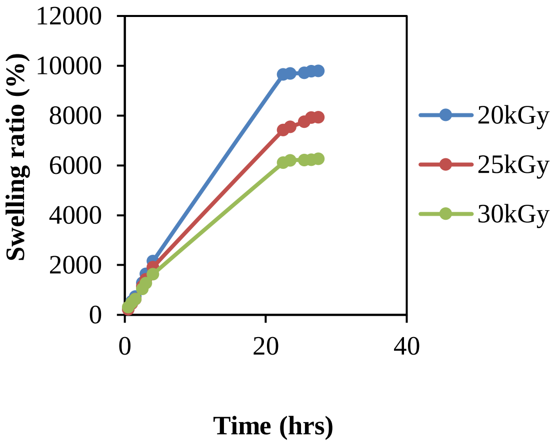

Fig. 4 Effect of radiation dose on swelling ratio of NAH2 hydrogel

3.4. FTIR Analysis

The IR spectra of the NaAlg/AAm/HEMA hydrogel are displayed in Figure 6. The asymmetric and symmetrical vibrations of the carbonyl group (COO−) at 1660 cm^{-1} and 1450 cm^{-1}, respectively, were linked to the bands of alginate. Strong peaks at 3199 and 3365 cm^{-1} in the spectra are attributed to the −NH2 group of AAm. The OH group of HEMA and the band of the −NH2 group overlap at 3365 cm^{-1}.

3.5. Adsorption Study

3.5.1. Effect of Adsorbent Dosage

Using varying doses of adsorbents (0.01−0.06 g, dry mass of NAH hydrogels), the effect of adsorbent dosage on the adsorption of MB (at a concentration of 25 ppm) was investigated. Plots of adsorption capacity (mg/g) at various adsorbent dosages are displayed in Figure 7. Figure 7 illustrates how the adsorbed amount per unit mass of adsorbent decreases as the adsorbent dosage increases. The number of active sites on the adsorbent's surface increases with adsorbent dosage, which results in a decrease in adsorption capacity. Therefore, 0.02 g adsorbents were used for further adsorption studies.

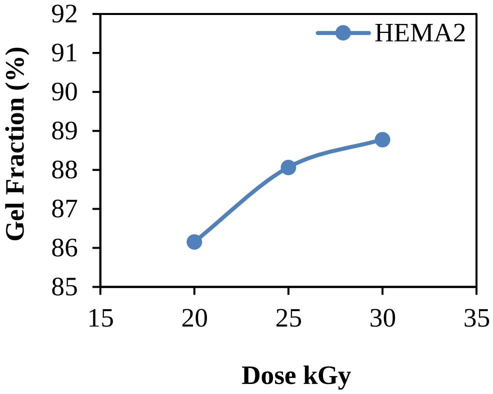

Fig. 5 Effect of radiation doses on gel fraction of NAH2 hydrogels

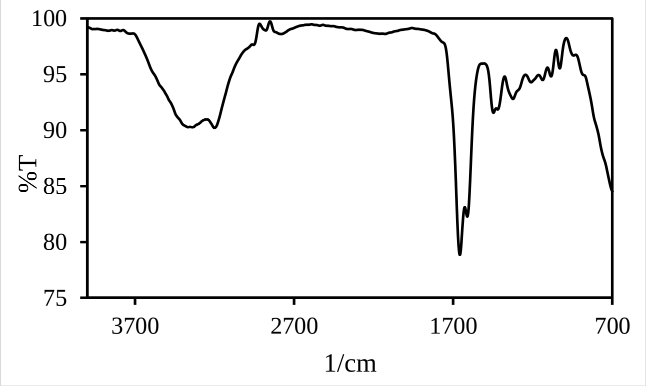

Fig. 6 FTIR spectra of NaAlg/AAm/HEMA hydrogel

3.5.2. Adsorption Kinetics and Mechanism

The effect of contact time on MB elimination was examined. Within six hours of contact time, the equilibrium was achieved, and it was discovered that the elimination of MB rose with contact time.

The absorption of metal ions by hydrogel was fitted using pseudo-first and pseudo-second-order kinetic models. The models are expressed using the following equations [27–28]:

$$\log(Q_e - Q_t) = \log Q_e - \frac{k_1}{2.303t} \qquad (4)$$

$$\frac{t}{Q_t} = \frac{1}{Qe^2 k_2} + \frac{t}{Qe} \tag{5}$$

Here,

Q_t= Adsorption of ions of metal during random time (mg/g)

Q_e= Adsorption of ions of metal during equilibrium (mg/g)

k_1 = Rate constant (1/h) for adsorption (1st order)

k_2 = Rate constant (g /h.mg) for second-order adsorption (2nd order)

Plotting log (Q_e - Q_t) vs. t, rate constants of the pseudo 1st order have been measured, and Figure 8 shows it

On the other hand, when plotting t/Qt against t, rate constants of the pseudo 2nd order were calculated, and Figure 9 shows it.

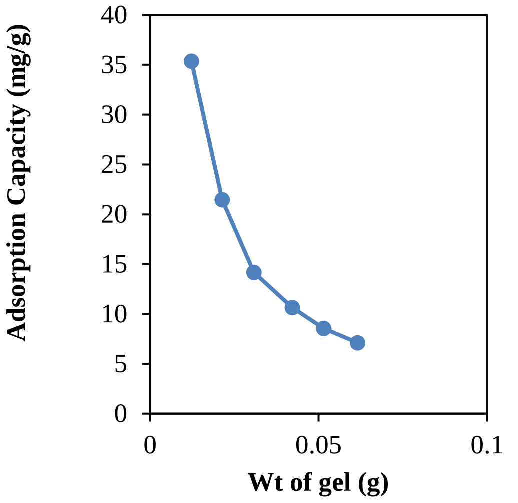

Fig. 7 Effect of NAH2 adsorbent doses on adsorption capacity (mg/g)

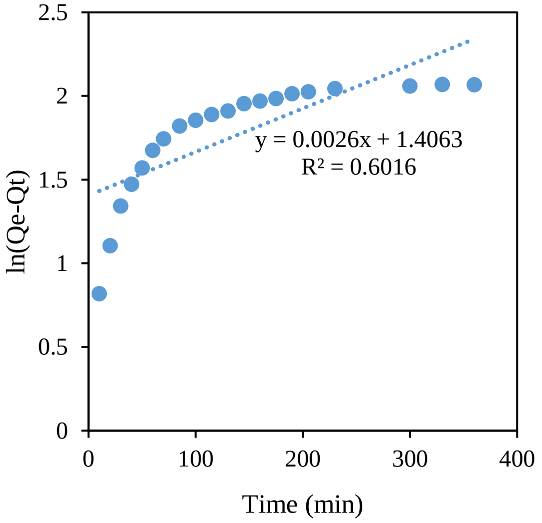

Fig. 8 Pseudo 1st order kinetics of MB adsorption

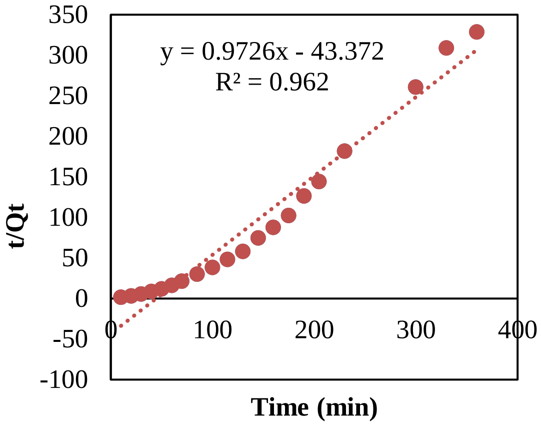

Fig. 9 Pseudo 2nd order kinetics of MB adsorption

R^2 value of pseudo 1st order is 0.6016, which is lower than R^2 value of pseudo 2nd order 0.962. So, the pseudo-2nd-order kinetic model fits with the experimental kinetic data. R-squared (R^2) measures the matching with linearity (when its value is close to 1, it indicates a good match with linearity), indicating how well the model fits the data.

3.5.3. Adsorption Isotherm of MB Dye

The Freundlich and Langmuir isotherms are most frequently employed to characterize the adsorption properties of an adsorbent. A set quantity of hydrogel (0.02 gm) was submerged in 25 ml dye solutions with concentrations ranging from 50, 100, 200, 300, 400, and 500 ppm in the equilibrium adsorption isotherm studies.

Langmuir Isotherm

The assumptions on which the Langmuir isotherm is based are- monolayer adsorption, identical adsorption sites, and any molecule to be adsorbed are independent of its neighbouring sites occupancy [29]. Eqn (6) is the equational expression of Langmuir Model.

$$C_e/Q_e = 1/Q_m K_L + C_e/Qm \tag{6}$$

Where C_e is the equilibrium concentration (mg/L), Q_m is the monolayer saturation adsorption capacity of the adsorbent (mg/g), Q_e is the equilibrium adsorption capacity, and K_L is the Langmuir adsorption constant (L/mg). Figure 10 shows the Langmuir isotherm of MB adsorption. A further analysis of the Langmuir equation was made on the basis of a dimensionless equilibrium parameter, R_L, also known as the separation factor given by equation 7.

$$R_L = \frac{1}{1 + K_L C_0} \tag{7}$$

Where K_L is the Langmuir adsorption constant (L/mg), and C_0 is the initial concentration (mg/L) of the Cr (III) ion.

The value of R_L lies between 0 and 1 for favorable adsorption, while $R_L > 1$ represents unfavourable adsorption, and $R_L = 1$ represents linear adsorption, while the adsorption process is irreversible if $R_L = 0$.[30]. K_L was 0.01196, and the separation factor (R_L) was found to be 0.24. The obtained R_L values were in the range of 0–1, which indicates the favourable adsorption of MB onto the NAH-based hydrogel

Freundlich Isotherm

Freundlich isotherm is applicable to adsorption processes that occur on heterogonous surfaces [31]. This isotherm gives an expression [eq^n (8)], which defines the surface heterogeneity and the exponential distribution of active sites and their energies [32].

$$\log Q_e = \log K_r + 1/n \log C_e \tag{8}$$

Where C_e is the equilibrium concentration (mg/L), Q_e is the equilibrium adsorption capacity, K_F is the sorption capacity (mg/g), and n is an empirical parameter.

The plot of log Qe vs log Ce shown in Figure 11 was pictured from the experimental data. Thus, a plot of log Qe vs log Ce should be a straight line with a slope of 1/n and an intercept of log K_F. This model deals with the multilayer adsorption of the substance on the adsorbent surface.

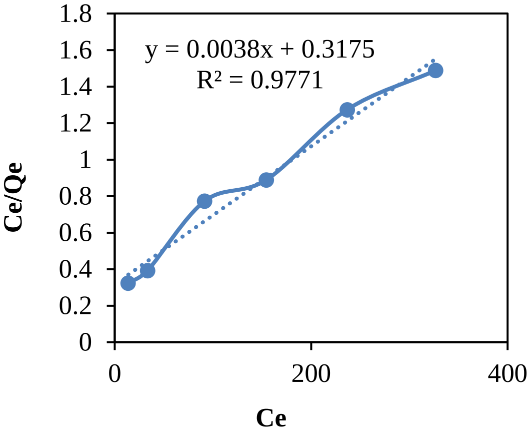

Fig. 10 Langmuir isotherm of MB adsorption

In Freundlich data, if n=1, then adsorption is linear. If n<1, then adsorption is a chemical process; if n>1, then adsorption is a physical process. The value of n ranges from 1-10, representing a good adsorption process [33] in the present study n=2.006.

It can be seen that the (R^2) value of the Freundlich isotherm model is almost similar to that of the Langmuir isotherm model. The Freundlich model is suitable for describing the adsorption of adsorbent with an uneven surface. The adsorbent can be adsorbed on multiple layers for high-concentration adsorption. Therefore, it can be presumed that the adsorption of MB on NAH-based hydrogel follows the Freundlich adsorption model. A picture of the dye after MB adsorption is shown in Figure 12.

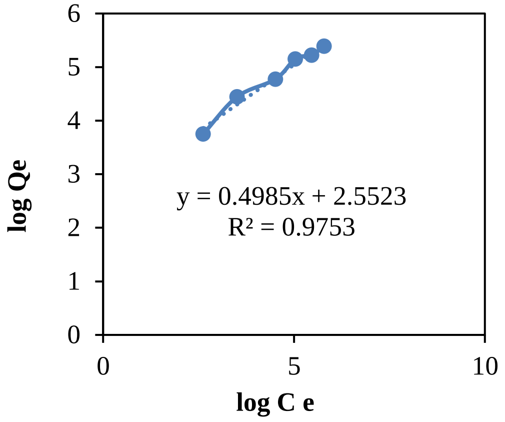

Fig. 11 Freundlich isotherm of MB adsorption

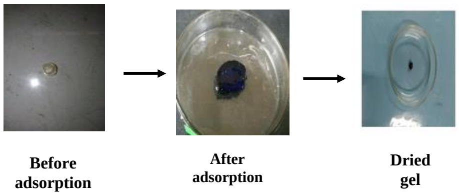

Fig. 12 MB dye adsorption

3.5.4. Thermodynamics Studies

The thermodynamic parameters such as free energy change (ΔG), enthalpy change (ΔH), and entropy change (ΔS) were obtained from the van't Hoff equation [34-35]:

$$\mathrm{Log}\frac{Qe}{Ce} = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \tag{9}$$

$$\Delta G = \Delta H - T\,\Delta S \tag{10}$$

Where ΔG is the free energy change (kJ mol^{-1}), R is the universal gas constant (8.314 J mol^{-1} K^{-1}), ΔH is the enthalpy change (KJ mol^{-1}), ΔS is the entropy change ($\mathrm{JK}^{-1}\mathrm{mol}^{-1}$), and T is the absolute temperature (K). The values of ΔH and ΔS were calculated from the slope and intercept of the van't Hoff plots of log(qe/Ce) versus 1/T, as shown in Figure 12.

For the temperature study, 0.0201 g NAH2 gel was placed in 25 ml of 100 ppm MB dye solution and observed after 5 hours. The effect of increasing temperature on the adsorption capacity was studied in the range of 276K, 292K and 333K under constant parameters at equilibrium conditions. The result is shown in Figure 13.

The positive value of ΔH (4.96404 KJ mol−1) indicated that the adsorption of MB dye molecules onto NAH gel was endothermic. Regarding the value of ΔS, it was found to be positive (17.3396J K^{-1} mol^{-1}), which indicates the increase in disorder at the solid-liquid interface during the adsorption process. The calculated ΔG values showed a decrease with increasing the temperature from 276 K to 333 K, indicating that the adsorption of MB dye molecules onto NAH gel is more feasible and spontaneous at high temperatures. Thermodynamics parameters are shown in Table 2.

3.6. pH study on MB Adsorption

For 24 hours, 0.021 g of NAH2 gels were submerged in 25 ml of a 100 ppm MB dye solution. The adsorbent surface becomes more negatively charged due to deprotonation when the pH of the solution is raised from 3 to 9, which increases the number of fixed ionized -OH groups. This produces two kinds of electrostatic forces: 1) an electrostatic interaction between the negatively charged hydrogel network and the positively charged dye, and 2) electrostatic repulsion forces between the neighbouring ionized groups of polymer networks, which cause the polymer chains inside the hydrogel structure to expand. Therefore, an ionic combination between the MB dye molecules and the hydrogel networks has formed as the pH has increased, increasing the dye adsorption (mg/g). In Figure 14, the impact of pH on MB adsorption is displayed.

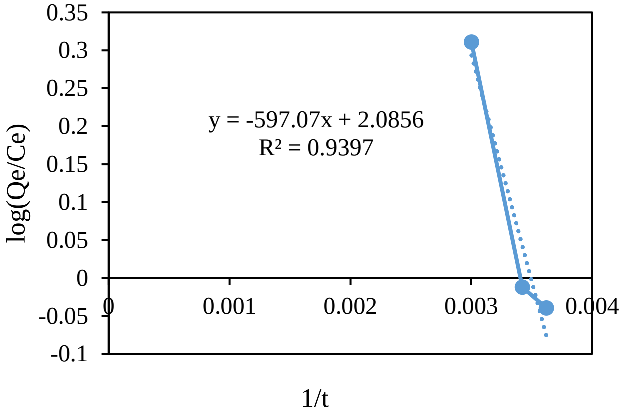

Fig. 13 Thermodynamics study of MB adsorption

3.7. Desorption study

The adsorbents were exposed to desorption using a 2M HCl solution for 24 hours following the MB dye's adsorption on the NAH hydrogel. The ratio of desorption was enough.

Table 2. Thermodynamics parameters

Dye	**ΔH(KJ mol^{-1})**	**ΔS(J K−1 mol^{-1})**	**ΔG(KJ mol^{-1})**		
			276K	**292K**	**333K**
MB	4.96404	17.3396	-4.781	-5.059	-5.770

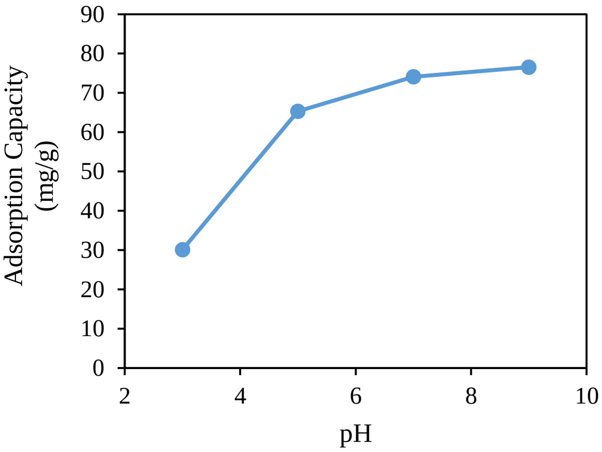

Fig. 14 Effect of pH of MB adsorption

4. Conclusion

Without the need for an external cross-linker, NaAlg/AAm/HEMA mix hydrogels were created by applying gamma radiation from a Co-60 source. The effects of factors like irradiation dose and HEMA concentration on the characteristics of the produced hydrogels (NAH hydrogel) were carefully examined. FTIR was used to characterize the hydrogel that was produced. Using the developed adsorbent to remove methylene blue dye from an aqueous solution produced encouraging results.

The isotherm and kinetics of MB adsorption were examined. With maximum dye adsorption of 219.65 mg/g, the adsorption behavior of NaAlg/AAm/HEMA hydrogel fit pseudo-second-order and was in good agreement with the Freundlich isotherm model. For MB adsorption, thermodynamics and pH analyses were performed. At high pH (9) and at high temperature (333 K), the adsorption was greater. This hydrogel shows promise in treating wastewater that contains MB dye solution. However, a clearer picture of the issue can be obtained by examining the MB dye adsorption in the presence of other coexisting dyes from actual waste water. This kind of research will be conducted in the future.

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