RESEARCH ARTICLE

Synthesis and Characterization of Sodium Alginate-g-polyacrylic Acid Hydrogel and its Application for Crystal Violet Dye Adsorption

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ABSTRACT

The sodium alginate-g-polyacrylic acid (SA-g- pAAc) hydrogel synthesis from sodium alginate and acrylic acid by free radical polymerization using N is used in this work N'-Methylene-bis-acrylamide MBA as a cross-linked agent, and the hydrogel was prepared as a super absorption active ingredient for organic pollutants. (SA-g-pAAc) the hydrogel was used to remove crystal violet (CV) dye from its aqueous solutions. Characterization of hydrogel was studied by (FTIR - XRD - FE-SEM - AFM – BET and BJH). The isotherm freundlich model can describe the adsorption process and influence of different conditions of pH, temperature, and ionic salt effect; the prepared hydrogel has demonstrated high adsorption and efficiency in removing the CV dye.

Keywords: hydrogel, acrylic acid, sodium alginate, crystal violet, adsorption.

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INTRODUCTION

Water is an essential resource, so water availability is necessary for the survival of living things on earth. However, enormous amounts of pollutants are introduced directly or indirectly into the water, making it unsafe for daily use.[1,2] Where dyes are increasingly present in water bodies, and these dyes do not suffer from natural decomposition, and as a result, the continuous color will hinder the passage of light to water and thus spoil the ecosystem, and this is one of the main problems involved in water coloring.[3,4] 10–15% of the unused dyes directly enter the wastewater in the staining or pollution process, but some reactive dyes are lost in the dyeing process, up to 50%.[5] Also, many industrial dyes are toxic and carcinogenic.[6,7] Therefore, disposal of liquid waste from various industries is a common problem that many countries face for a long time, as liquid waste contains several pollutants, including acids, bases, compounds, and toxic colors. Therefore, the effluent containing the dye must be treated and removed economically, up to the specified concentration, before being discharged into the water.[8] Many techniques are used to remove the dye, but the most feasible is the use of hydrogels to adsorption the dye from wastewater.[9] This is because the hydrogel is more beneficial as it is non-toxic and has the potential of a polymeric network, and can be reused well, cheap, and even chemically and physically stable.[10] A polymer is a network that supports the swelling property of hydrogels, enabling it to withstand a large amount of water without dissolving.[11] Sodium alginate (SA) is a natural anion polymer that is not biologically degradable and sensitive to pH, and non-toxic.[12,13] Therefore, the sodium gene-based adsorbents in water treatment have shown a clear form due to their biocompatibility and non-toxic nature.[14,15] In this research, (SA-g-pAAc) hydrogel was prepared; this work has shown that the hydrogels have effectiveness and rapid ability to adsorption crystal violet dye, we can expect that the (SA-g-pAAc) hydrogel, with its mechanical properties, can play a more important role in the adsorption field.

MATERIALS AND METHOD

Chemicals and Materials

Sodium alginate powder (SA), acrylic acid (AAc) HCl, N, N'-Methylene-bis-acrylamide (MBA), potassium persulfate (KPS), crystal violet (CV), and sodium hydroxide were sourced from Kemiou Chemical Reagent Co, Ltd, China. All chemicals were of pure analytical degree and used with purification. All solutions were prepared with deionized water, 0.1N NaOH, and 0.1N HCl adjusted the pH of the solution.

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Synthesis (SA-g-pAAc) Hydrogel

(SA-g-pAAc) the cross-linked hydrogel was prepared by the polymerization of free radicals in the aqueous solution, which included dissolving (0.131 g) of sodium alginate in (5 mL) of distilled water. It was placed in a three-barrel circular beaker connected to a condensate and nitrogen separation suppressor and gas inside the water bath while stirring until completely dissolved, then 3 g of acrylic acid and 0.05 g of soluble MBA and KPS soluble agent (1 mL) of distilled water, respectively, are added by suppressing the separation to the reaction mixture with continuous stirring, in the presence of nitrogen gas and at 70ºC temperature for three hours. At this point, the synthetic hydrogel is formed and washed with distilled water with continuous stirring for 6h for the sake of purity.

Hydrogel Properties

Prepare the Adsorbent Surface

The hydrogel was washed more than once with distilled water to get rid of dust and soluble materials, and then the surface was dried at a temperature of 50ºC for a period to be then ground and sifted with a different sieve (100, 400) µm, and 100 µm particles were used and kept in tight containers and used when conduct experiments for this study.

Hydrogel Analysis

FTIR Infrared Spectra Analysis

Fourier transformed Infrared spectroscopy (FTIR) was used to identify functional groups in the prepared hydrogel. FTIR spectroscopy data for the prepared surface were obtained within the wavelength range (4000–400) cm^{-1} using potassium bromide (KBr).

X-ray Diffraction Analysis

To study the crystalline properties of the prepared hydrogel, an XRD technique was used using a single wavelength light (1.5104) angstrom from a CuKα source.

Emission Scanning Electron Microscopy Technique FE-SEM

To study the properties of the external surface of the prepared surface, and FE-SEM was used as the image was taken after being coated with a thin layer of gold and low pressure.

Atomic Force Microscope

This technique was used to study the properties of the external structure of the prepared hydrogel, thickness, and grain size as well as obtaining three-dimensional images of the hydrogel.

Surface Area and Porosity Analysis of BET-BJH

To study the surface properties of the prepared hydrogel such as the surface area and pore diameters distribution, the BET isotherms method (adsorption-desorption) and (BJH) method were used to distribute the pore diameters.

Effect of Contact Time

The equilibrium time between the hydrogel surface and the CV dye was determined with all conditions confirmed. With the time factor changed, the solution of C.V dye concentration (250 ppm) was added, and the hydrogel was added in a ratio of (0.05 g) to 10 mL of the dye. Then the solutions were placed in the centrifuge at different periods, and then the residue from the dye concentration was determined.

Adsorption Isotherms

To determine the adsorption isotherms of the dye, different concentrations of the C.V attended their concentrations within the range (100–1000 ppm), 10 mL of these concentrations were added to (0.05 gm) of the hydrogel at pH 7 and temperature of 25ºC and after 120 minutes passed centrifuge solutions at 6000 rpm for 15 minutes, and then the spectrophotometric dye concentration was measured.

Effect of Temperature on Adsorption

The effect of temperature on adsorption isotherms has been studied at different temperatures (10–30)ºC for the dye on the hydrogel surface, (0.05 g) of hydrogel has been added to different concentrations of dye solution, in the same way as an experiment of adsorption.

Effect of pH

The effect of pH on the adsorption process was studied. A weight of (0.05 g) of the hydrogel was taken to add to it a solution of CV dye with a concentration (250 ppm), at different pH values (2–12) where the pH was organized using a solution of HCl, and NaOH value pH was measured using a pH meter.

Effect of Ionic Strength

The effect of ionic strength was studied by taking different weights (0.001–0.051 g) from salts (NaCl, KCl and $CaCO_3$), a CV dye solution (250 ppm) was added to volumetric flasks containing (0.05 g) of the prepared hydrogel with the same method of the experiment of adsorption conducted.

RESULTS AND DISCUSSION

FTIR Analysis

The FT-IR infrared spectrum of (SA-g-pAAc) hydrogel, as shown in Figure (3a), at range (3500–3200 cm^{-1}) absorption band instructed to the overlapping that occurred between the N-H and O-H bands, and the absorption bands at the frequency (2615 cm^{-1}), the C-H bands found in aliphatic compounds within the hydrogel composition represent the symmetric and asymmetric vibration of the CH_2 groups present in the hydrogel. Also, a band was found at (1735 cm^{-1}) belonging

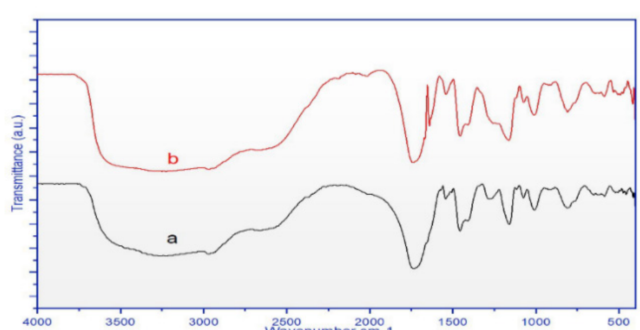

Figure 3: FTIR spectrum of (a) hydrogel (b) hydrogel after adsorption CV

to the carbonyl group C=O present in the carboxylic acid, as two groups COO^- symmetric and asymmetric appeared at the two frequencies (1543–1410 cm^{-1}) respectively due to sodium alginate. No band of (Na-O) appeared. This indicates the interference of acrylic acid and sodium alginate,[16-18] after adsorption of CV, we observe, as shown in Figure (3b), the shifts of the bands towards lower wavenumber as in the carbonyl group C = O, have shifted from 1735 to 1685. The absorption bands within the range 1600–1600 refer to the C = C group for the aromatic benzene ring.[19,20]

X-ray Diffraction

The XRD spectra are shown in Figure 4 showed the presence of a broad peak within the angular range 2θ (15–30)º, which indicates the amorphous in nature of the chemical composition of the hydrogel, as it was observed in the XRD spectrum of the hydrogel a wide beam at 2θ = 20.52º.[21]

Field Emission -Scanning Electron Microscopes (FE-SEM)

(SA-g-pAAc) hydrogel has demonstrated that its surface is rough and porous and is a nanocomposite that has a sponge-like structure and a mesh with compact layers. It contains many irregularly grouped wrinkles,[22,23] as shown in Figure (5, a). Upon adsorption of the dye on the hydrogel surface, the FE-SEM image shown in Figure (5, b) showed that the surface is smoother and more coherent. Thus, the hydrogel surface has become completely covered with the dye molecules, confirming the adsorption process.[24,25]

Atomic Force Microscopy (AFM)

AFM images showed that the surface is porous and that the average roughness is high, and that the surface contains more declines than the tops, as well as it is bumpy[26,27] nature, and as shown in Figure 6.

Surface Area Analysis and Porous Surface Nature

(SA-g-pAAc) hydrogel isotherm is prepared classified by Figure (7 a,b) adsorbent isotherm-desorption of nitrogen N_2 to the prepared hydrogel is of the fourth class (IV), and this means that the multi-layer adsorption and this isotherm are hysteresis loops of H_3 and this indicates that the surface pores are not regular in porous distribution.[28,29]

Effect of Contact Time

The time required to reach the equilibrium state of the CV dye was studied at a concentration of 250 ppm and different periods (1–240 minutes) and a temperature of 25ºC and a constant weight of hydrogel 0.05 g and at pH 7.0 for the CV dye where it was found that the time required to reach the equilibrium state is (120 minutes), and as shown in Figure 8, it has been observed that the adsorption process increases with increasing time, so the adsorption is very rapid at the beginning (20 minutes), after which the increase is gradual until reaching the equilibrium time.[30]

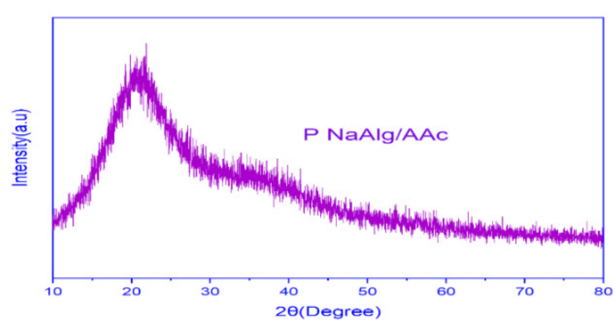

Figure 4: XRD spectrum of the hydrogel

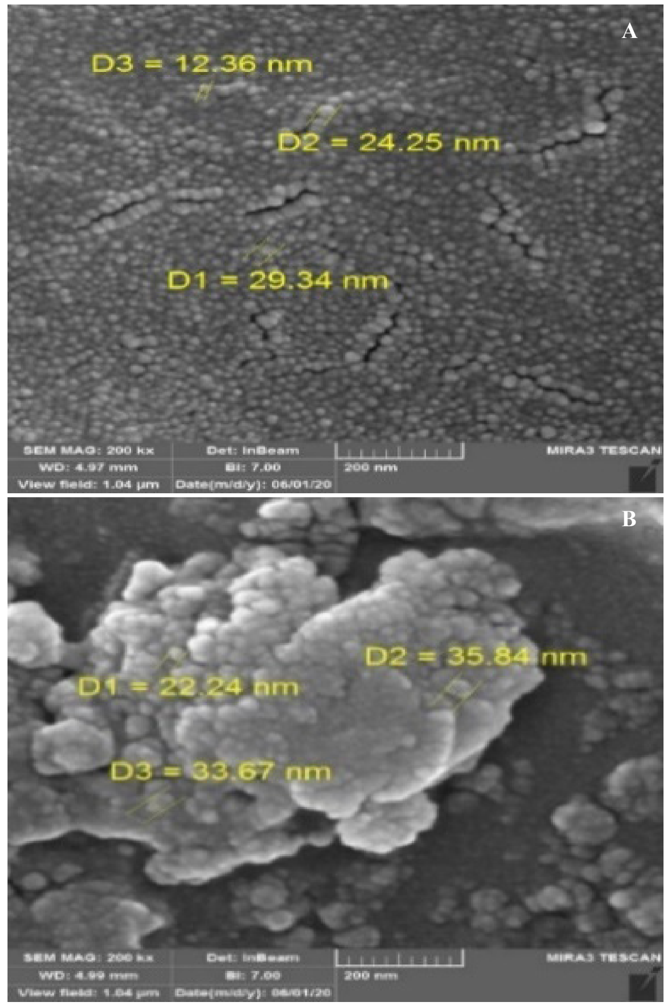

Figure 5: a: FE-SEM image of (SA-g-pAAc) hydrogel, b: after adsorption CV dye on the hydrogel

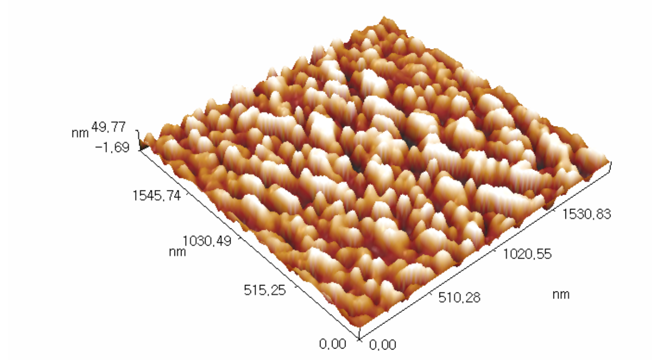

Figure 6: AFM image three dimensional of the hydrogel

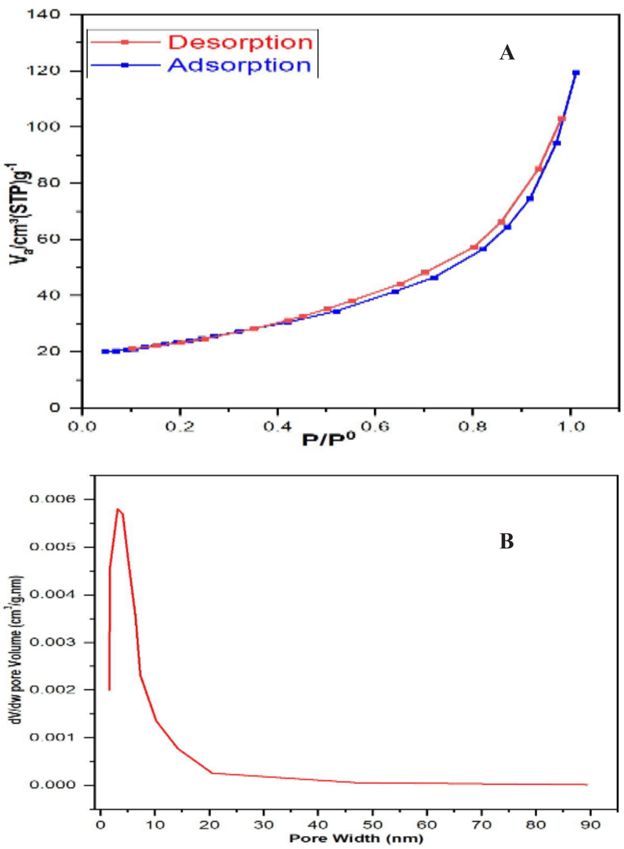

Figure 7: A: adsorbent isotherm - desorption of nitrogen N_2 to BET, B: adsorbent isotherm - desorption of nitrogen N_2 to BJH

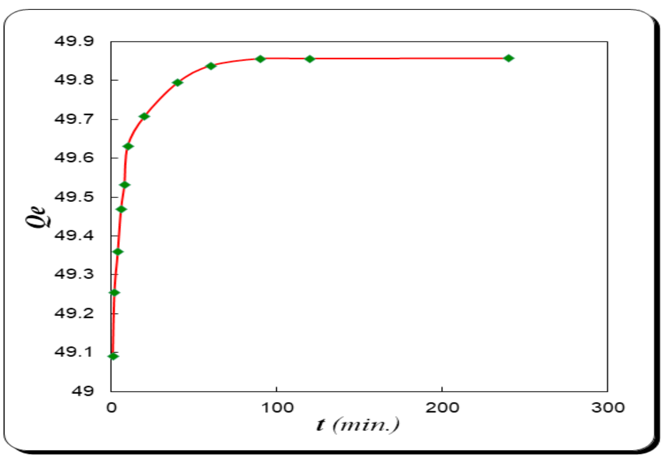

Figure 8: Effect of contact time on CV adsorption

Adsorption Isotherms

Adsorption isotherms were calculated for the CV dye on the adsorbent surface, and it was observed that the adsorption process corresponds to class (L) according to the Giles classification. As this class shows, the dye particles adsorbed on the adsorbent surface will be oriented horizontally, and the adsorption will be multi-layer. It has also been observed that the application of isotherm- Freundlich with the adsorption process data at equilibrium to adsorb the CV dye (Figure 8), and this indicates the heterogeneous nature of the surface or surfaces that support sites of different affinity,[31,32] and as shown in Figures 9 (a,b), and Table 1.

Table 1: Isotherm parameters for Langmuir and Freundlich models

Langmuir equation			*Freundlich equation*		
K_L	q_m	R^2	K_F	n	R^2
0.1327	333.333	0.7271	56.9770	1.96155	0.9733

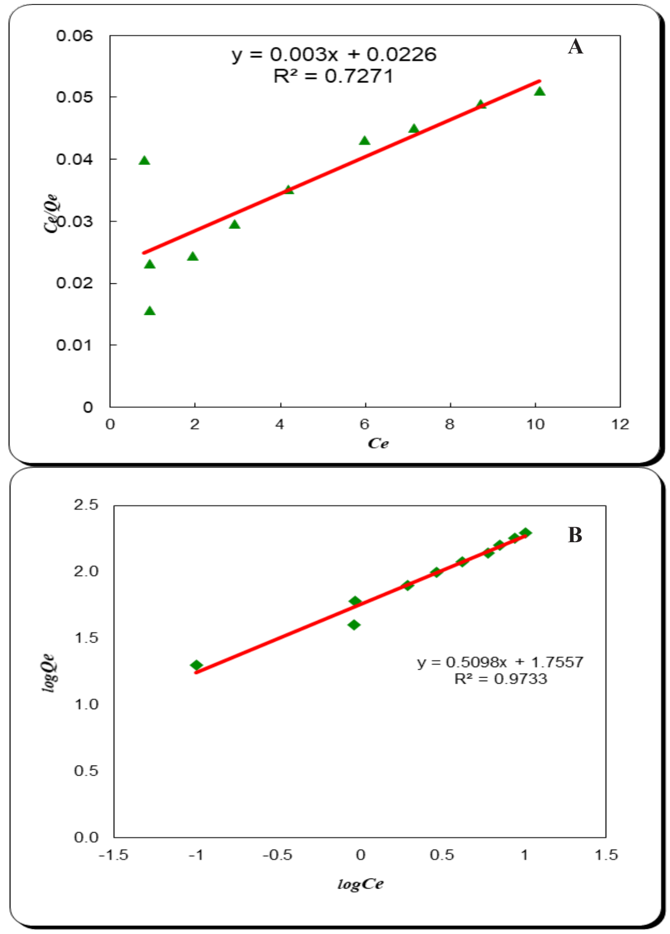

Figure 9: A: The linearized Langmuir isotherm for CV adsorption at 25ºC, B: The linearized Freundlich isotherm for CV adsorption at 25 ºC

Effect of Temperature and Calculation of Thermodynamic Parameters

CV dye adsorption has been studied on the surface of the prepared hydrogel at different temperatures (10,15,20,25, and 30)ºC. The adsorption process of the CV dye on the hydrogel has decreased when the temperature rises, this means that the adsorption process is exothermic, as it is shown in Figure 10, and the reason for this is that the increase in temperature will increase the solubility of the minutes of the solute in the solvent, thereby reducing the adsorption affinity of the solute in the solution towards the adsorbent surface.[33,34]

The values of thermodynamic parameters have been calculated, and the results are shown in Table 2 have shown that the value of enthalpy ΔH is negative, and this indicates that the process of adsorption is exothermic, It was also observed that free energy change was of negative value, meaning that

Table 2: Thermodynamic parameters of adsorption CV on the hydrogel surface

ΔH (kJ.mol^{-1})	*ΔG (kJ.mol^{-1})*	*ΔS (J.mol^{-1}.k^{-1})*	*Equilibrium Constant (K)*
6.427.55-	14609.0-	27.92	100.56

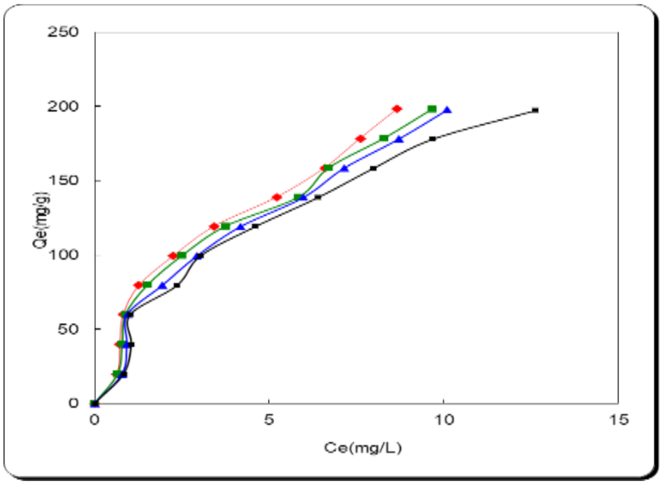

Figure 10: Effect of temperature on the adsorption capacity of C.V dye

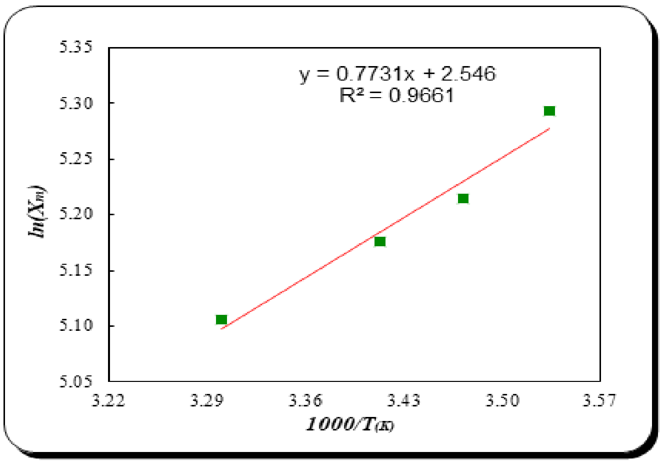

Figure 11: Plot of ln Xm against reciprocal absolute temperature for adsorption crystal violet on the hydrogel surface

the adsorption process was spontaneous. As for the value of the change in entropy (ΔS), it is positive, and this indicates that the adsorbed particles are in continuous motion on the surface more than in solution[35,36] as shown in Figure 11 and Table 2.

Effect of pH

The effect of pH on the adsorption process of the CV dye on the adsorbent surface studies with a concentration of (250 ppm) at the values of the different pH (2–12) and fixing the other conditions. As shown in the Figure 12, the adsorption capacity is increased with the increase of pH, that when the pH is low, the concentration of H^+ ions is very high in the solution. Then competition will occur between it and the dye molecules with a positive charge on the active sites of the adsorbent surface, which leads to reduced adsorption,[37,38] but in the case of high pH, the adsorption capacity increases when the pH rises.[39]

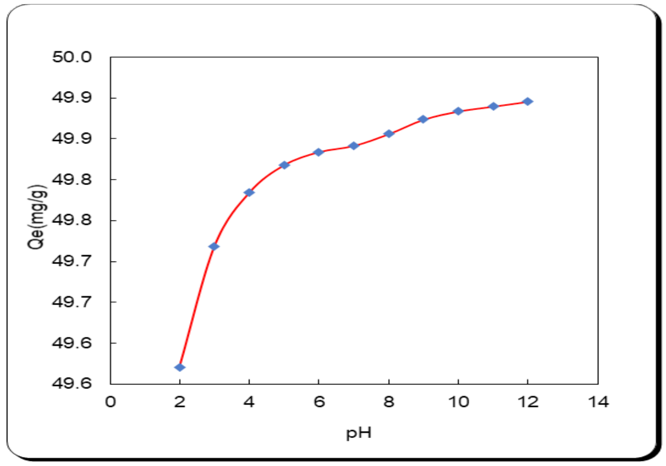

Figure 12: Effect of pH values on CV adsorption on the hydrogel

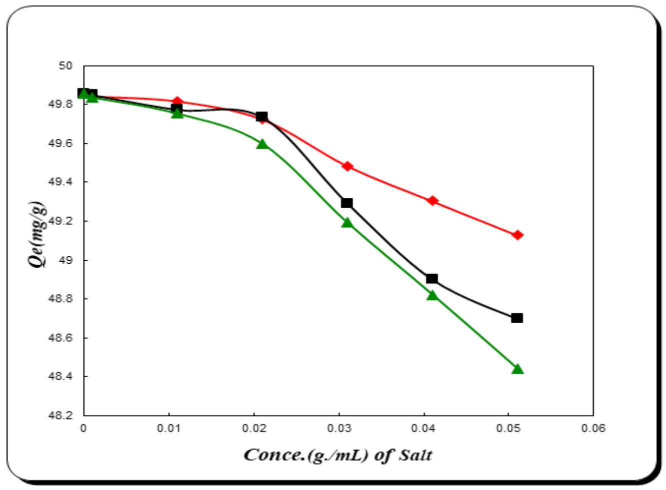

Figure 13: Effect of ionic strength on CV adsorption of the hydrogel

Effect of Ionic Strength

The effect of ionic strength in the adsorption process has been studied. The results have shown, as in Figure 13, that the adsorption capacity of the CV dye on the hydrogel decreases when the salt concentration increases, and this is due to the effect of competition between the positive ions of salts and the positive dye molecules on the active sites of the adsorbent surface.[40]

CONCLUSIONS

(SA-g-pAAc) hydrogel has been used as adsorption to remove the CV dye from its aqueous solutions. Temperature, equilibrium time, ionic strength, and the pH solution play a large role in the adsorption capacity of the hydrogel. The prepared hydrogel showed effective adsorption of the CV stain, and the isotherm described the adsorption process- freundlich model and the adsorption process was spontaneous, and this was explained by the negative value of the free energy change.

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