Removal of Metformin hydrochloride from Aqueous Solutions by using Carboxymethyl cellulose-g-poly(acrylic acid-co-acrylamide) Hydrogel: Adsorption and Thermodynamic Studies

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Abstract

The Sodium Carboxymethyl Cellulose-grafting-Poly(Acryl Amide-Acrylic Acid) hydrogel was synthesized through free radical polymerization method of CMC, AAm, and AAc. Potassium persulfate(KPS) and N, N methylene bis-acrylamide (MBA) were used as initiator and crosslinking agent, respectively. FTIR, XRD, FESEM, and TGA were used to characterize the synthesized hydrogel. The hydrogel was used to remove metformin hydrochloride drug from aqueous solutions. The analysis clarifies the effect of several parameters on the quantity of adsorbate, including pH at a range of (1.2-12), the temperature at a range of (15, 20, 25 and 30°C), equilibrium time (1-240 min), and adsorbate weight at a range of (0.001-0.1 g). It is discovered that as temperature increases and pH decreases, the quantity of adsorbate present decreases. Equilibrium time 120 min. The adsorption isotherm seems to indicate that the adsorption mechanism followed Freundlich and Temkin models. Thermodynamic functions were measured, and it was discovered that the adsorption mechanism is exothermic and spontaneous.

Keywords: Sodium Carboxymethyl Cellulose, Hydrogel, Metformin hydrochloride, adsorption.

Introduction

Hydrogels are three-dimensional (3D) polymeric materials that can hold high concentrations of water or biological fluids without dissolving in them. Its ability to absorb water is due to the presence of hydrophilic functional groups such as (OH, CONH, CONH₂, and SO₃H), and their insolubility results from the cross-linking of the polymer chains with each other chemically or physically, which prevents soluble and maintains the three-dimensional network in the event of swelling [1-3]. Because of their high water content, they have a degree of flexibility very similar to that of biological tissues, which makes them an ideal material for use in many applications. one of the main applications of hydrogels has been the removal of contaminants from aqueous solutions [4]. Along with the swollen hydrogels, contaminants are often adsorbed above the external surface [5].

Sodium Carboxymethyl Cellulose (CMC) has numerous applications due to its unique chemical, physical, and biological properties, especially its biocompatibility. It has good sensitivity to pH change because of the presence of NH₂ and COOH groups. It is a polymer dissolved in water,
swelling more in the base than in the acidic medium. Therefore it can be used in controlled drug delivery systems, where the drug loaded on it is well released in the intestine[6].

Metformin hydrochloride (MH) is useful for individuals with type 2 diabetes mellitus. It has a short half-life, so repeated dosing is required to keep the molecules concentration constant in the body of humans. Therefore, researchers have begun to attempt to lower the dosages of metformin hydrochloride by loading on polymers such as hydrogel[7, 8]. This study showed that the [CMC-g-P(AAm-AAc)] hydrogel demonstrated exceptional quick adsorption of drugs.

Experimental
Chemicals and Materials

Sodium Carboxymethyl cellulose (CMC), N,N'-Methylene- bis-acrylamide (MBA), and Acrylamide (AAm) were purchased from Himedia. Potassium persulfate (KPS) was purchased from Fluka. Acrylic Acid (AAc) was purchased from Thomas maker. Hydrochloric Acid was purchased from BDH. Sodium hydroxide was purchased from Alpha Chemika. Metformin hydrochloride (MH) was purchased from Samarra company.

Preparation of the [CMC-g-P(AAm-AAc)] hydrogel

A series of processes were used to prepare the [CMC-g-P(AAm-AAc)] hydrogel as shown in scheme (1) by the method of free radical polymerization. 1g of CMC was dissolved in 20mL of deionized water with heating to 60°C until homogeneous of the solution. The initiator KPS solution (0.1g/2mL of deionized water) was added at 60°C to generate free radicals. After cooling the mixture, the AAm solution (1g/1mL of deionized water), 4mL of AAc, and the MBA solution (0.05g/1mL of deionized water) were added continuously stirring. All previous additions were done with the presence of nitrogen gas. Finally, the mixture was placed in test tubes and left in the water bath at a temperature of (70°C) for 2 hr to allow the hydrogel formation. It will then cut, washed, dried, and ground as in Fig.1[9-11].

Scheme(1): Preparation pathway of the [CMC-g-P(AAm-AAc)] hydrogel[11].
Characterization of hydrogel

Infrared spectra analysis (FTIR)

FTIR was used to characterize the functional groups in the [CMC-g-P (AAm-AAc)] hydrogel and to determine the extent of displacement after adsorption of MH drug. Within the wavelength range (400-4000) cm\(^{-1}\) after pressing with KBr.

X-ray diffraction (XRD)

To identify the crystalline properties of the synthesized [CMC-g-P (AAm-AAc)] hydrogel, X-ray diffraction technique was used. The X-ray diffraction was studied using a Siemens-D500 diffractometer at 35 kV, with Cu-K\(\alpha\) radiation, at wavelength 1.5104 Å, and with a scan angle of 2θ at range (10-80).

Field Emission Scanning Electron Microscopy (FESEM)

To identify the morphology of the [CMC-g-P (AAm-AAc)] hydrogel, before and after the adsorption of MH, a (FESEM) was used with different magnification powers and under 8000 KV voltage.

Thermal Gravimetric Analysis (TGA)

To identify the physical and chemical properties of the [CMC-g-P (AAm-AAc)] hydrogel, a TGA technique was used by following mass change resulting from slow and continuous heating from 50 to 800 °C with N\(_2\) gas atmosphere and at the rate of 5°C/min.

Determination of the maximum wavelength (\(\lambda_{\text{max}}\)) of MH

To determine the (\(\lambda_{\text{max}}\)) of MH, a solution of the drug with a concentration (10 ppm) was synthesized. The absorbance spectrum of the drug was recorded by UV-Vis spectroscopy within the wavelengths (200-800) nm. It was found that the (\(\lambda_{\text{max}}\)) = 232 nm, as shown in Fig.2 (a).

Calibration curve of MH

It was determined by preparing solutions of different concentrations (1-30)ppm, and the absorbance was measured for each solution at \(\lambda_{\text{max}} = 232\) nm by using UV-Visible Spectroscopy. Then, the relationship between absorbance and concentration was plotting, as shown in Fig.2 (b).
The effect of surface weight on the adsorption of MH was studied by adding 10 mL of 80 ppm from drug solution to different weights (0.005-0.1) g of the [CMC-g-P (AAm-AAc)] hydrogel. Samples were shaken at 25°C for 120 min, and then the solutions were separated and centrifuged. The absorbance of solutions was measured at $\lambda_{\text{max}} = 232$ nm.

Equilibrium Time

It was established that the equilibrium time between the hydrogel and the drug was fixed to all conditions. With the time factor changed, the concentration of the drug (80 ppm) was applied, and the solution was composited to a ratio of (0.05 g) to 10 mL of it. Then the solutions were put in the centrifuge and the residual sample left in there for (1-240) minutes, and then the results were found.

Adsorption isotherms

To determine adsorption isotherms, 0.05 g of the [CMC-g-P (AAm-AAc)] hydrogel was shaking for 120 min with 10 ml from different concentrations (5-200) ppm of the drug solution at 15°C. The adsorbed drug quantity was calculated by the equation:

$$Q_e = \frac{V(C_0 - C_e)}{m} \quad \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldots \ldOTS
The effect of temperature on MH adsorption isotherms was studied at different temperatures (15, 20, 25, and 30° C). Different concentrations of the drug solution were synthesized (5-200) ppm, and (0.05 g) was added from the hydrogel to 10 mL of each solution. The solutions were shaken for 120 min, and then they were measured absorbance of each solution at (λmax = 232 nm).

Effect of pH

It was studied by preparing different pH solutions (1.2-12) and adding (0.008 g) of the drug and (0.05 g) from the surface to each of them. Then the absorbance of solutions was measured at (λmax = 232 nm).

Result and discussion

FTIR analysis

FTIR of the [CMC-g-P(AA-co-AAm)] hydrogel was shown in Figure (3a). The broad peak at (3200-3500) cm⁻¹ is attributed to the overlap of NH and OH groups. The peaks at (2900-2960) cm⁻¹ are attributed to the CH aliphatic group. The peak at 1745 cm⁻¹ is due to the C=O group of Acrylic acid. The small peaks at (1600-1700) cm⁻¹ are attributed to the C=O group acrylamide. The peaks at (1542.95 and 1396.37) cm⁻¹ are attributed to the asymmetric and the symmetric stretching of the C=O group in the carboxylate anion, respectively. The peaks at (1010.63 and 1204.92) cm⁻¹ are attributed to the EC–O–C and C–N stretching, respectively. A shifting of some peaks after drug adsorption (Fig. 3b) refers to the interaction between the functional group of the hydrogel and the drug. The occurring of shifting at the peaks (3440.77-3417.63; 3209.33-3232.47; 2592.15-2584.44; 2314.42-2329.85; 1612.38-1615.52; 1542.95-1558.38) cm⁻¹ proves the drug adsorption[2, 10, 11].
Fig. 3: FTIR of (a) the [CMC-g-P(AA-co-AAm)] hydrogel (b) the [CMC-g-P(AA-co-AAm)] / MH.

X-ray diffraction analysis

The XRD of the hydrogel (Fig. 4) is shown a main broad peak at position ($2\theta=21.194^\circ$) and d-spacing of (d=4.397\text{Å}); this refers to the non-crystalline nature of the hydrogel\cite{13}.

Fig. 4: XRD of the [CMC-g-P(AA-co-AAm)] hydrogel.

FESEM analysis

FESEM images of the hydrogel (Fig. 5 a,b) showed that its surface was rough, porous, and had wrinkles due to irregular agglomerations, which results from its containment of carboxyl, amide, and hydroxyl groups\cite{14,15}.
While, FESEM images of the hydrogel after adsorption of the drug (Fig.5 c,d) showed that its surface was smoother and less porous or nonporous as a result of filling the surface pores with drug particles, and a layer of them is formed on the surface, and thus the surface becomes covered with drug particles, and this proves that adsorption has occurred\cite{16}.

**Thermal Gravimetric Analysis TGA**

The TGA curve for the hydrogel (Fig.6) showed four stages of a gradual decrease in weight with an increase in temperature from 30 to 800°C. The first stage happens at (40-165)°C because of the loss of water molecules or moisture. The second happens stage at (165-260)°C, because of decarboxylation and deamination of the hydrogel chain as a CO$_2$ and NH$_3$ gas, respectively. The third stage happens at (260-345)°C because of the decomposition of polysaccharides. The fourth stage happens at (345-549)°C, because of degradation of the hydrogel chain\cite{17,18}. 

**Fig.5: FESEM images of (a,b) the [CMC-g-P(AA-co-AAm)] hydrogel, (c,d) after loading MH.**
Fig.6: TGA of the [CMC-g-P(AA-co-AAm)] hydrogel.

Effect of Adsorbent Weight

It was observed that the adsorption increased with the increase in the weight of the hydrogel due to the increase in the active groups. Upon reaching a weight (0.05 g), it was observed that the adsorption did not increase with the increase in the weight of the hydrogel approximately because the solution was saturated with the hydrogel [19, 20].

Equilibrium time

It was discovered that the time required to reach the equilibrium state of the MH is 120 min, as shown in (Fig.8). It has been found that the adsorption mechanism accelerates with time until it reaches equilibrium at 120 minutes. This condition can be explained by the adsorbate's initial occupying of the active sites, after which the rise is incremental until it reaches the equilibrium time[21-23].

Adsorption isotherms

The adsorption isotherms of MH were calculated on the hydrogel surface. The adsorption was found to be identical to class (S) according to the classification of Giles(Fig.10a). The adsorbent molecules
are oriented vertically or diagonally on the hydrogel surface, and the adsorption is multi-layer. Additionally, it was observed that the Langmuir, Freundlich, and Temkin isotherms were used in conjunction with the equilibrium adsorption process data to adsorb the MH drug[24], indicating the heterogeneous existence of the surface. As shown in (Figures 11, 12, 13) and table 1.

![Fig. 9: adsorption isotherm of MH at 15°C.](image1)

![Fig. 10: The linear Langmuir isotherm of MH adsorption at 15°C.](image2)

![Fig. 11: The linear Freundlich isotherm of MH adsorption at 15°C.](image3)

![Fig. 12: The linear Temkin isotherm of MH adsorption at 15°C.](image4)

| Table 1: Isotherm parameters for Langmuir, Freundlich, and Temkin models |
|---|
| **Langmuir equation** | **Freundlich equation** | **Temkin equation** |
| $k_L$ | $q_m$ | $R^2$ | $k_T$ | $n$ | $R^2$ | $k_T$ | $B$ | $R^2$ |
| 0.160 | 1.077 | 0.975 | 0.007 | 0.225 | 0.9781 | 91722 | 0.314 | 36 | 0.8 |
| 38355 | 23796 | 73 | 1 | 4 | 28 | 0.225 | 9273 | 26 | 8 |
| 2 | 7 | 0.225 | 0.225 | 91722 | 98 | 0.314 | 36 | 0 | 0.8 |
| 8 | 3 | 0.225 | 0.225 | 91722 | 98 | 0.314 | 36 | 0 | 0.8 |
3-1. Effect of temperature

As shown in Fig. 14, the adsorption of MH has decreased as the temperature rises. The reason for this is that as the temperature rises, the solubility of the solute in the solvent increases, lowering the adsorption of the solute in the solution \[15, 25\]. The thermodynamic parameter values have been determined. The results are shown in Table (2) have shown that $\Delta H$ and $\Delta G$ are negative, indicating that the adsorption process is exothermic and spontaneous, respectively. The change in entropy $\Delta S$ is negative, indicating that the adsorbed MH molecules are more likely to be in continuous motion on the surface than in solution \[26, 27\], as illustrated in Fig (15).

![Fig.14: Effect of temperature on MH adsorption.](image)

![Fig.15: Plot of ln Xm against reciprocal absolut Temperature for adsorption of MH.](image)

| $\Delta H$ (kJ.mol\(^{-1}\)) | $\Delta G$ (kJ.mol\(^{-1}\)) | $\Delta S$ (J.mol\(^{-1}\). k\(^{-1}\)) | Equilibrium Constant (K) |
|-----------------------------|-----------------------------|-----------------------------|-----------------------------|
| -53.8190162                 | -4.004209115                | -170.0164064                | 2.06984127                  |

**Effect of pH**

As shown in Fig. 11, at low pH, the concentration of hydrogen ions $H^+$ increases, which increases competition with drug molecules to occupy the active sites on the adsorbent surface; thus, the amount of adsorbed drug decreases. And the opposite occurs when the pH value increases\[28, 29\].
Fig. 16: Effect of pH on adsorption of MH.

Conclusion

The [CMC-g-P(AA-co-AAm)] hydrogel was used to remove MH from aqueous solutions by adsorption technique. The equilibrium time, temperature, and pH of the solution all had a significant effect on the adsorption capability. Freundlich and Temkin isotherm equations model were found to be superior to the Langmuir model in describing the adsorption of MH on the hydrogel. The adsorption process of MH is exothermic and spontaneous.

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