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Preparation of highly efficient new poly sodium alginate (acrylic acid-co-acrylamide) grafted ZnO/CNT hydrogel nanocomposite: Application adsorption of drug, isotherm and thermodynamics

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ABSTRACT

The present study used a free radical copolymerization approach to synthesize hydrogel of new poly sodium alginate (acrylic acid-co-acrylamide) grafted ZnO/CNT hydrogel nanocomposite. SA-g-P(Ac-co-AM)/ZnO-CNT hydrogel nanocomposite was characterized via X-ray diffraction (XRD), Thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FT-IR), and Field emission scanning electron microscope (FE-SEM). In the adsorption experiments, Clonazepam (CLZ) drug was used. In this study absorption process was carried out in shaker water batch for the drug and an equilibrium system to investigate the adsorption of drug onto SA-g-P(Ac-co-AM)/ZnO-CNT hydrogel nanocomposite. The parameter influenced the adsorption capacities, like equilibrium time, initial of drug concentrations, pH solution temperature of solution and adsorbent dosage. The maximum adsorption capacities and removal percentage of CLZ were 95.14 mg g^{-1} (85.56%). The adsorption thermodynamics for drug were in accordance with isotherm Freundlich model. It is shown that adsorption of the drug is spontaneous and endothermic presses.

Keyword: Adsorption, Removal, Clonazepam (CLZ), Drug, Hydrogel, Isotherm, Thermodynamic.
Article type: Research Article.

INTRODUCTION

Adsorption is a surface phenomenon that includes the adhesion of molecules, ions, or atoms of a substance to the surface of another substance. The substance that gets deposited or adsorbed is termed an "adsorbate," whereas the substance on which deposition/adsorption occurs is termed an "adsorbent". The adsorbate may form a single layer on the surface of the adsorbent, the adsorption then called monolayer adsorption. When more than one layer is deposited, adsorption is referred to as multimolecular adsorption (Alqaragully *et al.* 2015; Alkaim & Ajobree 2020; Jasim & Aljeboree 2021; Isola *et al.* 2022; Alhattab *et al.* 2023). The process at which the absorbed substance released from the surface of adsorbent referred to as desorption. Absorption process categorized into two types according to the nature of binding to the absorbent surface. Physical Adsorption (Physisorption): This type of absorption occurs when the adsorbate substance adhered to the surface of absorbent by a weak physical force called van der Waal's force. Chemical adsorption (Chemisorption): This type of absorption occurs when the solid absorbent has electron unsaturated surface which covalently bind to the adsorbate substance (Mosaa *et al.* 2019; Mahmoud *et al.* 2020; Abid Alradaa & Kadam 2021). Hydrogels are a three-dimensional network that belong to the family of water-insoluble cross-link polymers with hydrophilic properties, which make these polymers swell seven to ten orders of magnitude larger than their original size as a result of absorbing a large amount of water (Bo Gao *et al.* 2021; Mohammadzadeh Pakdel *et al.* 2022). These polymers are affected by pH, temperature, ionic strength, and existence of some compounds. The cross-link structure of these polymers is held

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together as water-swollen gels by: polymer crystallites, hydrophobic interactions, bio-recognition interactions or affinity, hydrogen bonds, ionic forces, and primary covalent cross-links (Dijana Takić Miladinov *et al.* 2016; El Shafey *et al.* 2021; Radia *et al.* 2022).

MATERIALS AND METHODS

Experimental Part

Synthesis of Carbon decorated / ZnO Nanocomposite:

Nanocomposite (CNT/ ZnO) was synthesized using the hydrothermal process (Fig.1). Ten grams of zinc acetate together with 5 g of oxalic acid and 1.0 g of CNT were mixed with 100 mL distilled water in Erlenmeyer flask and shacked for 30 min. The obtained mixture was transferred into the stainless steel or Teflon-lined autoclave and heated at 160 °C for 24 h under autogenous pressure. The obtained mixture was filtered, washed with distilled water several times, sonicated for 10 min, and dried at 80ºC for 24 h to produce a dark-brown fine powder.

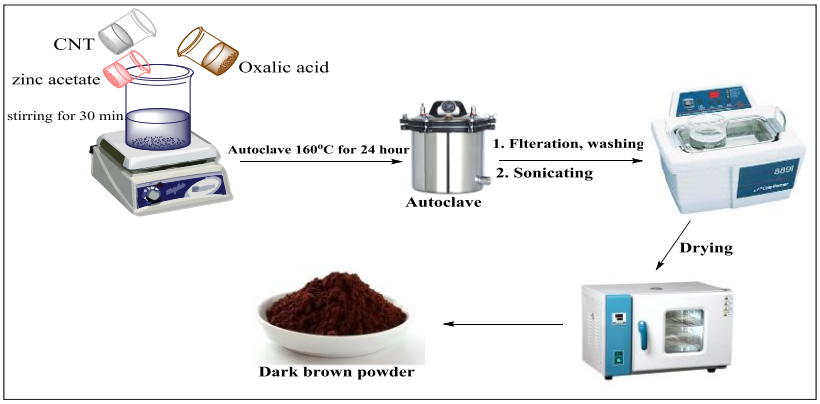

Fig. 1. Precreation of CNT/ ZnO Nanocomposite.

Synthesis of poly (acrylic acid)-based nanocomposite hydrogel

The nanocomposite was synthesized via free radical polymerization using N,N'-Methylene bis-acrylamide as cross-linking agent. A total of 0.2 g of ZnO/CNT was shacked with 20 mL distilled water for 5 h and sonicated for 2 h. The obtained mixture was added gradually to 40 mL 5% sodium alginate with stirring for 3 h at 25 ºC. After stirring turned the reaction mixture into a uniform, pasty-like gelatinous solution, 0.5 g acrylamide and 3 mL acrylic acid were added. Then, 0.05 g potassium persulfate (as a free-radical initiator for polymerization) together with 0.05 g N,N'-Methylenebisacrylamide (as acrylamide cross-linking agent) were added. Afterward, the reaction mixture was heated at 75 ºC in thermostatically controlled water bath to produce the poly (acrylic acid)-based nanocomposite hydrogel (SA-g-P(Ac-co-AM)/ZnO-CNT). The hydrogel obtained was washed several times with distilled water to remove unreacted species and dried in an oven at 60 ºC. Finally, the dried hydrogel was milled into a fine powder (Fig. 2).

Adsorption Isotherm

The batch adsorption method was used to investigate the adsorption behaviour of two drug onto super nonabsorbent hydrogel (SA-g-P(Ac-co-AM)/ZnO-CNT). A known quantity of hydrogel (0.1 g) in 100-mL solution containing 100 mg L^{-1} quantity of two drug derivatives in 100-mL Erlenmeyer flasks. The reaction mixture was shacked water bath for 60 min and then centrifuged for 10 min. The remaining pollutants concentration in the resultant supernatant was determined using the proposed method. The adsorption capacity (Qe) and percentage (E%) adsorption was estimated in Equations 1-2.

$$\mathrm{Qe}\left(\frac{\mathrm{mg}}{\mathrm{g}}\right) = \frac{(\mathrm{Co} - \mathrm{Ce})\mathrm{Vml}}{\mathrm{M\ gm}} \qquad (\mathbf{1})$$

$$\mathrm{E} = \frac{\mathrm{Co} - \mathrm{Ce}}{\mathrm{Co}} \times 100 \qquad (\mathbf{2})$$

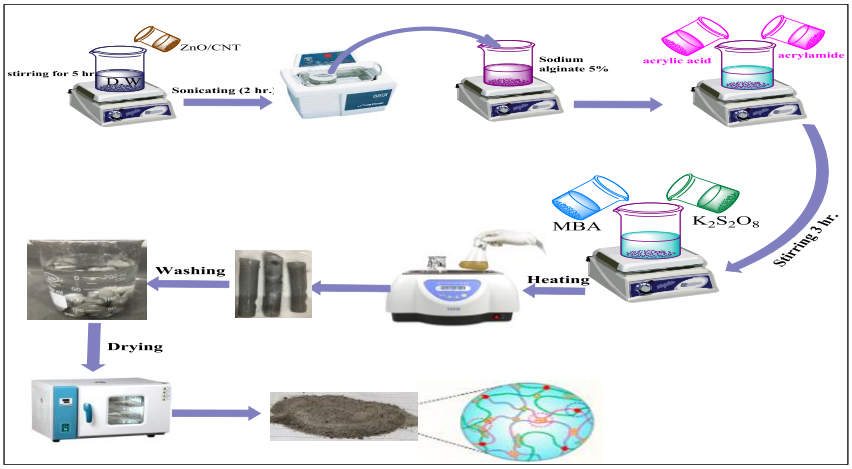

Fig. 2. Preparation (SA-g-P(Ac-co-AM)/ZnO-CNT) hydrogel nanocomposite.

RESULTS AND DISCUSSION

Characterization of adsorbent (SA-g-P(Ac-co-AM)/ZnO-CNT) hydrogel nanocomposite

FTIR spectra were used to characterize the SA-g-P(Ac-co-AM)/ZnO-CNT hydrogel nanocomposite before and after adsorption process onto CLZ drug (Fig. 3), leading to a pure peak and finding more than a few intensity of adsorption nanocomposite. The hydrogels before loading CLZ drug, clarify a clear decrease in FTIR spectra and did not appear new peak (Banerjee &Chattopadhyaya 2017; Saif 2020). In addition, hydrogel had the acidic group, leading to several change in absorption intensity.

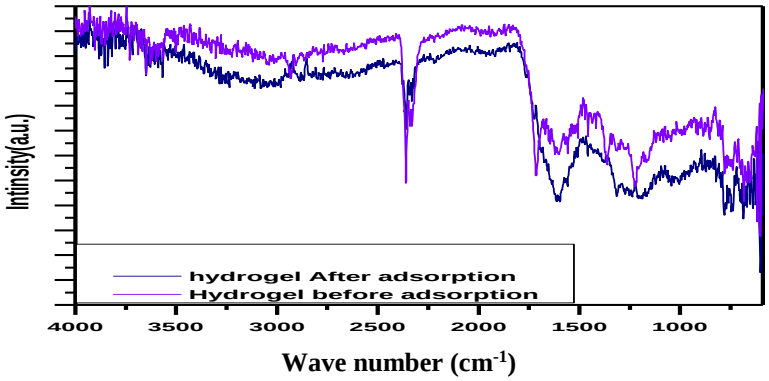

Fig. 3. FTIR spectra characterize the (SA-g-P(Ac-co-AM)/ZnO-CNT) hydrogel nanocomposite.

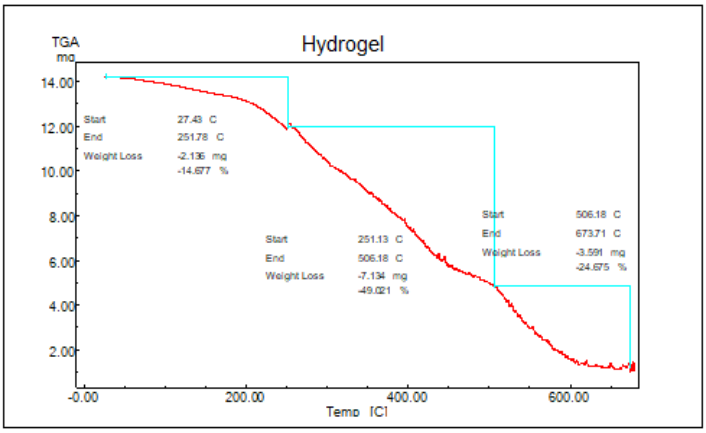

Fig. 4. TGA Analysis of (SA-g-P(Ac-co-AM)/ZnO-CNT) hydrogel nanocomposite.

The TGA analysis of SA-g-P(Ac-co-AM)/ZnO-CNT hydrogel nanocomposite, shown in Fig. 4, exhibited added confirmation that ZnO/CNT integrates in to hydrogel (Zhou *et al.* 2013). A loss weight at range temperature of

10-600 °C, of which 14.6% happened at temperatures less than 200 °C evaporation of the free water and 49.02% happened in the range temperature of 200-400 °C due to desorption water and volatility (Luo 2020; Yasin *et al.* 2021). The crystalline of SA-g-P(Ac-co-AM)/ZnO-CNT hydrogel nanocomposite was analysed via X-ray diffraction and the registered patterns are shown in Fig. 5. The SA-g-P(Ac-co-AM)/ZnO-CNT hydrogel nanocomposite X-ray diffraction type refers to the semi-crystalline of the nanocomposite with one broad peak at 2θ° =20.32 related indicating the dispersion of CNT into the polymeric network. Also, the appearance of crystalline peaks in 2θ° = 32.32, 2θ° =34.32 and 2θ° =36.33 indicates the loading of zinc oxide nanoparticles (Alradaa & Kadam 2021).

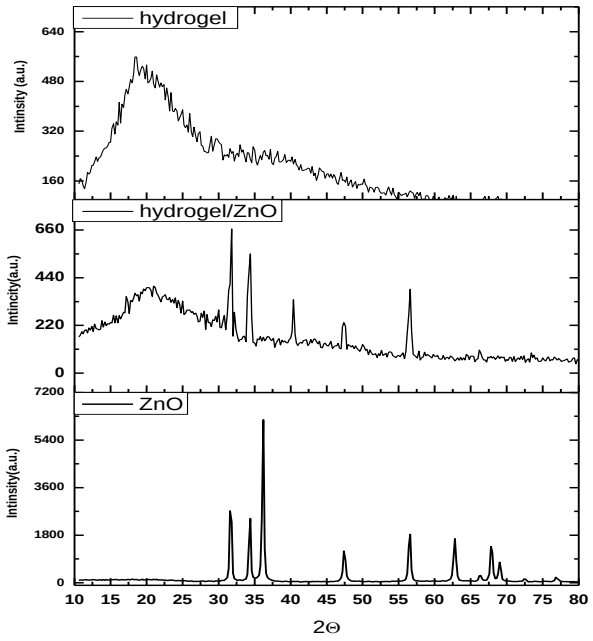

Fig. 5. X-ray diffraction: a) ZnO NPs; b) hydrogel; c) hydrogel nanocomposite.

The technique FESEM was utilized to study the properties of hydrogel after the addition of ZnO NPs and CNT and also after the loading CLZ drug (Fig. 6). Zinc oxide nanoparticles were observed in the Fig. 6a, containing small white globular clusters carefully crowded together; also (b) representing carbon nanoparticles at a distance of 200 nm. Fig. 6c exhibits the hydrogel, vast groups were observed, and contained many gaps, indicating the roughness of the hydrogel surface (Sourbh Thakur et al. 2022). However, after the loading of ZnO / CNT onto hydrogel, all gaps were filled and the surface become homogeneous containing many active sites, since by incorporation of ZnO /CNT on the hydrogel surface, it increased the surface area and a high porosity structure. Fig. 6e exhibits that FE-SEM of nanocomposite was before and after CLZ drug adsorption on SA-g-(PAAC-co-VBS)/ZnO surface, and Fig. 6f displays that EDX synthesized elements of SA-g-P(Ac-co-AM)/ZnO-CNT hydrogel nanocomposite C, O, and Zn, which indicates the presence of ZnO/CNT onto SA-g-P(Ac-co-AM)/ZnO-CNT hydrogel nanocomposite (El Shafey *et al.* 2021; Thakur S 2018).

Effect of different parameters

Effect of equilibrium time

Fig. 7 illustrates equilibrium time of CLZ drug on the nanocomposite to adsorption capacity which increased quickly for 20 min and then lightly increased until reaching its maximum contact time (1 h); fast mass transfer of CLZ drug molecules starts via attaching and separating toward the solid surface. Afterward, the particles of nanocomposite started to reach equilibrium time, and adsorption process reached equilibrium (Jawad *et al.* 2021)

Effect of adsorbent of Nanocomposite

By varying the adsorbent dosage of hydrogel nanocomposite (0.05-0.15 g), the estimate of effect of nanocomposite on the removal percentage (E%), and adsorption capacity of CLZ drug (as shown in Fig. 8), an improvement removal of CLZ drug in aqueous solution was detected as the nanocomposite weight was increased.

Thus, the improvement in CLZ drug removal rate (%) from 64.26 to 94.84% was occurred by elevating the weight of nanocomposite from 0.05-0.15 g. These results occurred in the enhancement number of active sites. Also, adsorption capacity was affected by the elevated weight of nanocomposite, leading to declining from 125.15 to 70.26 mg g^{-1}. This may be illustrated according to the collision of particles of adsorbent with each other (Aljeboree *et al.* 2019a).

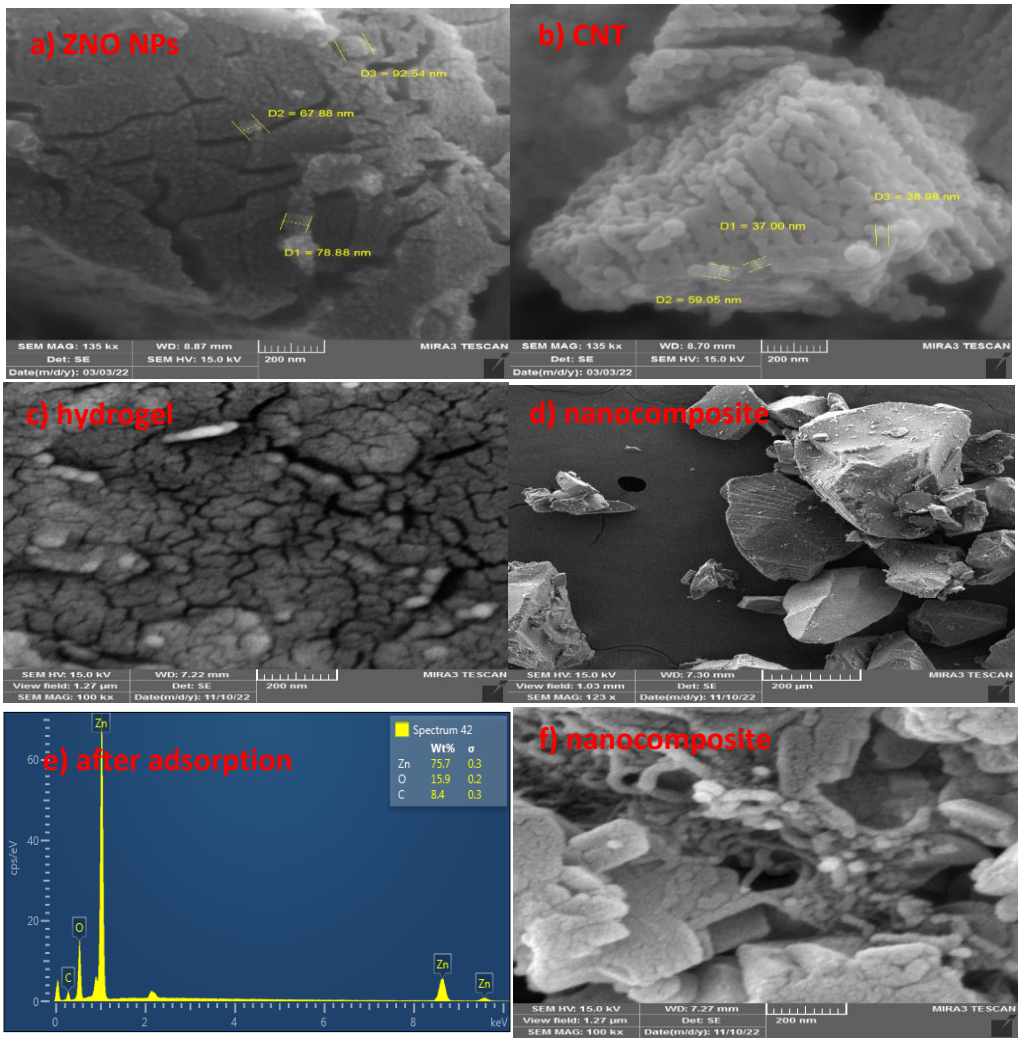

Fig. 6. FESEM image: a) ZnO NPs; b) CNT; C) hydrogel; d) nanocomposite; e) nanocomposite after adsorption; and f) EDX of nanocomposite.

Effect of solution pH

To study the effect of pH solution on removal rate (%) and adsorption efficiency of nanocomposite, experiments were conducted at concentration of CLZ drug 100 mg L^{-1}, 0.1 g nanocomposite and 25 ºC. Fig. 9 illustrates that the sorption of CLZ drug occurred at maximum pH 6 and raised with solution pH elevation at pH 10. The low solution pH of CLZ drug onto nanocomposite at pH 2, may be due to the charge of the surface which became positively charged. Thus making ions (H^+) competes effectively with (H^+) drug causing a declined adsorption capacity of drug (Aljeboree *et al.* 2019b).

Effect of CLZ drug concentration

The examination of the alterations in CLZ removal rate (%) via several solutions concentrations from 25 to 200 mg L^{-1} CLZ in 100 mL and 0.1 g nanocomposite at 25 ºC and also contact time of 1 h (shown in Fig. 10) shows that the elevated concentration of CLZ leads to drop in removal rate (%) from 98.98% to 76.88%, however, elevated adsorption capacity from 20 mg L^{-1} to 159 mg L^{-1} (Luo 2020; Yasin *et al.* 2021).

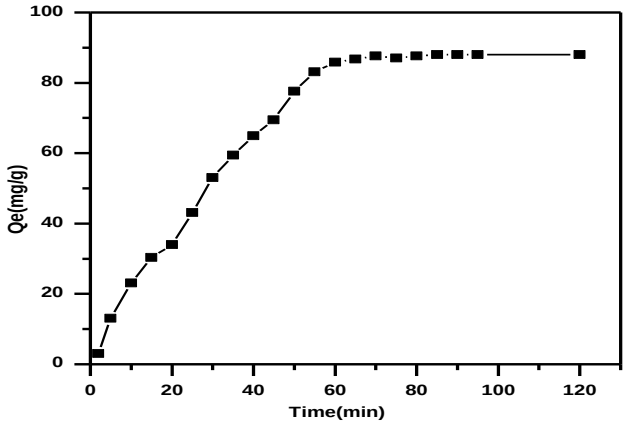

Fig. 7. Effect of equilibrium time of CLZ drug onto hydrogel nanocomposite.

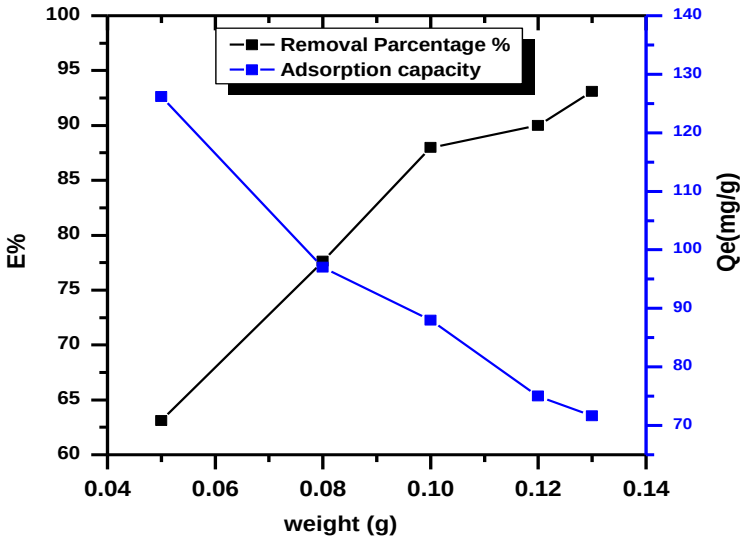

Fig. 8. Effect of weight of hydrogel nanocomposite.

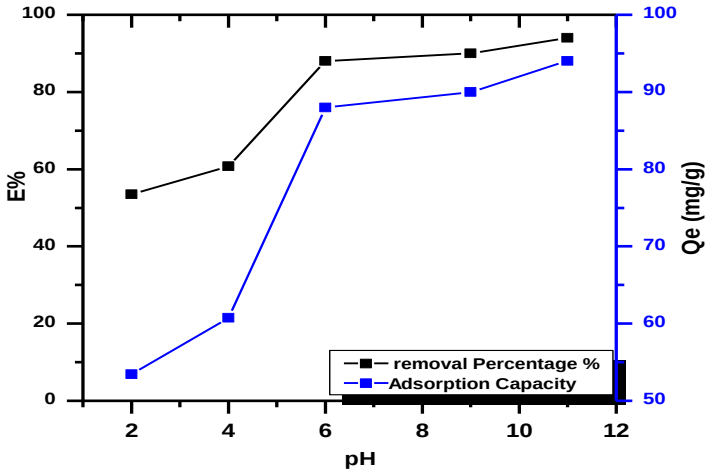

Fig. 9. Effect of pH solution of CLZ drug onto hydrogel nanocomposite.

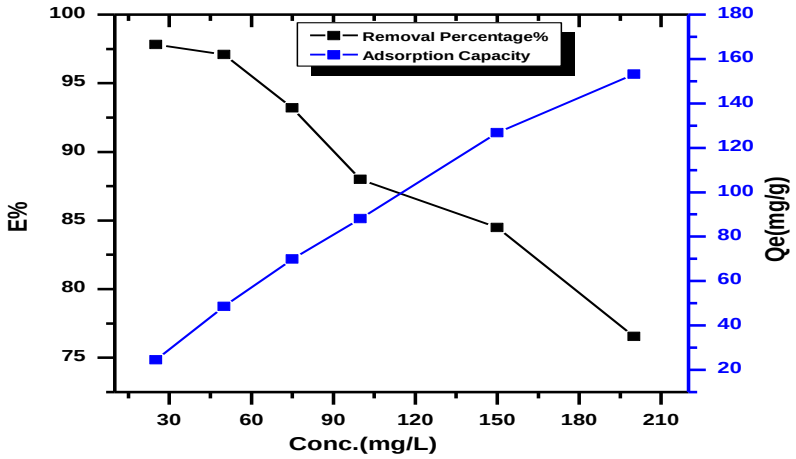

Fig. 10. Effect of drug concentration onto hydrogel nanocomposite.

Adsorption Isotherm

To give details the adsorption capacity of the nanocomposite, the equilibrium investigational data were studied to confirm compliance with the equations model of Langmuir and Freundlich isotherms (Powers 2003; Gamoudi & Srasra 2019). Freundlich Isotherm is an experiential equation based on adsorption on to heterogeneous surface as calculated in equation 3.

$$Q_e = k_f C_e^{\frac{1}{n}} \quad \textbf{(3)}$$

Langmuir Isotherm is used for the adsorption of a solute from solution as adsorption monolayer on a surface taking number finite of identical sites (del Mar Orta *et al.* 2019). The model is setup on different essential assumptions: (i) the sorption occurs at set sites homogeneous adsorbent; (ii) once a dye molecules of site occupies; (iii) the adsorbent (at equilibrium) has ability limited for the adsorbate; (iv) total of sites are congruous and energetically identical. The non-linear equation of isotherm Langmuir is obtained by equation 4.

$$Q_e = \frac{Q_m K_L C_e}{1 + K_L C_e} \quad (\mathbf{4})$$

The KF values and (R^2) are found from Freundlich models (Table 1). The adsorption of dye better fitted to Freundlich isotherm with the best R^2 =0.9877 comparison with Langmuir isotherm (Chayid & Ahmed 2015; Jawad *et al.* 2021; Aljeboree & Alkaim 2019). The Freundlich isotherm exhibits a better fitted to the adsorption data than the Langmuir isotherm (as shown in Fig. 11).

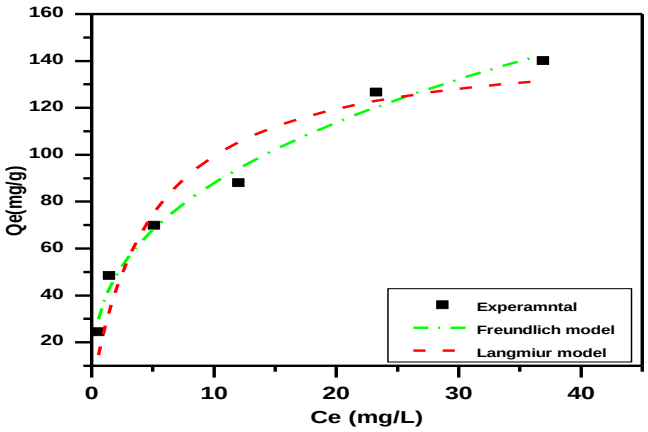

Fig. 11. Adsorption isotherm two model Freundlich and Langmuir Isotherm nanocomposite.

Effect of temperature and thermodynamic parameters

To estimate whether the ongoing adsorption method was exothermic or endothermic. The isotherms adsorption was estimated for several pollutants-adsorbent systems. The removal of CLZ was studied at several solution temperatures (15-35 ºC) in the presence of several concentrations of CLZ (10-100 mg L^{-1}) as shown in Fig. 12.

The data appeared that the equilibrium adsorption efficiency of CLZ was raised while increasing the temperature of solution for all CLZ concentrations (Chayid & Ahmed 2015; Jawad *et al.* 2021; Aljeboree & Alkaim 2019).

Table 1. several factor isotherms for the adsorption study of drug on to hydrogel nanocomposite

Hydrogel nanocomposite		Mean	± SD
Freundlich	K_f	37.419	± 3.348
	1/n	0.37	± 0.084
	R^2	0.98814	
Langmuir	q_m (mg g^{-1})	149.66	± 16.876
	K_L (L mg^{-1})	0.199	±0.0811
	R^2	0.9011	

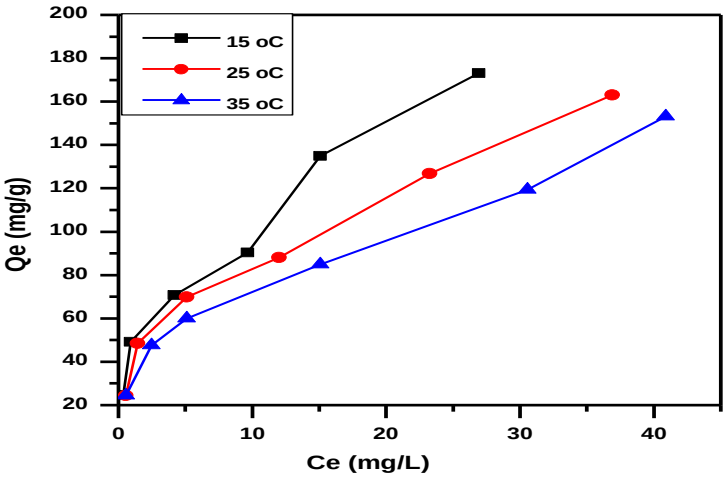

Fig. 12. Effect of solution temperature on the adsorption of CLZ drug onto nanocomposite.

The thermodynamic study useful in this work included change in free energy (ΔG), change in enthalpy (ΔH), and in change entropy (ΔS). Van't Hoff equation was utilized to estimate the thermos-dynamic factors as following in equations 5 to 7.

$$K_e = \frac{(Q_{max}) * Wt\ (0.1\ gm)}{(C_e) * V(0.1L)} \times 1000 \qquad (5)$$

$$\Delta G = -RT \ln K_e \qquad (6)$$

$$\ln X_m = -\frac{\Delta H}{RT} + Cons. \qquad (7)$$

The quantitative thermos-dynamic result of CLZ on the adsorbent nanocomposite as appeared in Table 2. This Table appears the values of ΔH for CLZ drug is positive indicating that the adsorption method is endo-thermic. All methods of adsorption considered spontaneous from the ΔG negative value, while, positive value of ΔS for CLZ (Osman *et al.* 2020).

Table 2. Thermodynamic parameter ΔS, ΔG and ΔH for CLZ drug adsorbed onto nanocomposite.

Thermodynamic Factors			
ΔH (kJ/mol)	ΔG (kJ/mol)	ΔS (kJ/K.mol)	Equilibrium Constant (Ke)
16.029	-4.311	94.11	5.47

CONCLUSION

In this study, preparation of new hydrogel nanocomposite via free radical polymerization process was utilized to adsorb CLZ-drug. The best mass of hydrogel nanocomposite was 0.1 g to give the best removal rate (%) and adsorption efficiency of Qe (mg g^{-1}). The elevated concentration of CLZ leads to decline in removal rate from

9898% to 76.88%. At the same time, increase in the adsorption capacity from 20 mg L^{-1} to 159 mg L^{-1} leads to the best time for equilibrium to be reached 1 h. the thermodynamic parameter study revealed spontaneous nature and endothermic of adsorption method utilize nanocomposite. Also, behaviour of the equilibrium of nanocomposite was fitted with Freundlich isotherm.

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