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FULL PAPER

Peripherally modified poly (acrylic acid)/ ZnO nanocomposite hydrogel with selective superadsorption properties

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In this study, removal of three toxic chemical pollutants, such as crystal violet (CV) dye, tetracycline (TC) drug, and phenol (PH) using sodium alginate-*g*-poly (acrylicacid-co-sodium, 4-ethenylbenzenesulfonate, hydrate)/Zinc oxide, and SA-g-poly(AC-co-EBS)/ZnO hydrogel composite was prepared by co-polymerization method by addition of free radicals. The hydrogel composite was characterized by TEM, FESEM, and XRD. Removal of laboratory sample aqueous pollutants (dyes, drugs, and phenol) using hydrogel composite to give low absorbance (0.0001) utilizing UV-Vis spectrophotometer for at a chosen wavelength for 2 h. Comparative between ((SA-g-(PAAc-co-EBS)/ZnO, (SA-g-(PAAc-co-EBS), and ZnO NPs) surfaces as adsorbents. The best results of the percentage of removal (E%) of three pollutants arrange in order increasing (SA-g-(PAAC-co-EBS)/ZnO NPs > SA-g-(PAAC-co-EBS) > ZnO NPs), the good results of the percentage removal (E%) of hydrogel composite, (92, 451%, 87.56%, and 82.56%) for CV, TC, and PH, respectively. Likewise, comparative between the amount of Zinc oxide nanoparticles (ZnO NPs) decorated of (SA-g-(PAAC-co-EBS) using (0.05, 0.08, 0.1, and 0.15 g). The good results of the percentage of removal (E%) of three pollutants about 0.1 g ZnO NPs. Re-cyclability and desorption studies indicated the best re-cycling performance of the prepared composite. Based on the results, the prepared hydrogel composites can be useful as a promising, ecological, cost-effective, and efficient material for dyes decontamination. Studies was carry out using several desorption agents at concentration (0.01 N) like HNO_3, NaOH, H_3PO_4, H_2SO_4, HCl, and water. The hydrogel composite, was regeneration with 100% can be desorbed in diluted hydrochloric acid (0.01 N). The isotherm Freundlich and Langmuir models are also introduced, it has been found that all results follow the Freundlich model in the presence of three pollutants; this nonlinearity is higher when using the Freundlich model.

KEYWORDS

Removal; hydrogel; isotherm; crystal violet dye; tetracycline drug; phenol.

Introduction

Water has an important and fundamental role in human, animal, and plant life. Most of the world's population does not have access to clean drinking water of acceptable standards. The most important source of drinking water used in the world is groundwater [1-6]. One of the most important different traditional methods for removing dyes from their aqueous solution, it was found that adsorption is one of the utmost significant and easiest economic methods, ease of operation and design, and also has the ability to remove low concentrations of dyes and toxic substances [2,7-10].

Pharmaceuticals are chemicals commonly used in the treatment and diagnosis of diseases. Also, to include veterinary compounds applied to illegal drugs pharmaceuticals are produced in very large quantities and consume a variety of drugs used in the treatment of mankind, including synthetic hormones, statins, antibiotics, cytotoxins, and anti-inflammatories. Tetracycline (TC) has been most utilized in human treatment and husbandry animal, with the global yearly production of large than 20,000 tons, it has been one of the utmost repeatedly discover antibiotics in the waste water utilized to treat a number of infections, having acne, malaria, brucellosis, cholera, plague, and syphilis. Common side effects include vomiting, rash, diarrhea, and loss of appetite. Its formula is $C_{22}H_{24}N_2O_8$ and its molecular weight is 444.4 gm/mol. Phenol is also a common water pollutant, and it is considered as a carcinogen and a danger to human health, even at low concentrations. Therefore, the treatment of industrial water containing phenol has become necessary. Phenol (also called carbolic acid, phenylic acid hydroxybenzene, and phenic acid) is an aromatic organic compound with the molecular formula C_6H_5OH. It is a white crystalline solid that is volatile. The molecule consists of a phenyl group ($-C_6H_5$) bonded to a hydroxyl group (−OH) and it is mildly acidic. Its molecular weight is 49.113 g/mol, (λ_{max}) = 270 nm, and its IUPAC name is Benzenol. Phenol and its chemical derivatives are essential for production of polycarbonates, epoxies, Bakelite, nylon, detergents, and herbicides such as phenoxy herbicides and numerous pharmaceutical drugs [11,12].

Hydrogel is a three-dimensional polymer with a network structure. It has a high ability to swell in water, and does not have the ability to dissolve in water and retains a large amount of water. Hydrogels can be divided into Natural hydrogels and synthetic hydrogels by source material [13,14]. Natural hydrogels like fibrin, Cyclodextrin, collagen, chitosan, dextrin, explain good Biodegradable, and biocompatibility thus, always have poor mechanical properties. Preparation hydrogels, showing high mechanical strength, are cross-linked polymers prepared by polymerization in the presence of water [13,15-16].

In this study, SA-g-poly(AC-co-EBS)/ZnO hydrogel composite was prepared by means of co-polymerization method by initiators and cross-linking agents. Properties and characterization of hydrogel composites were studied using several techniques. The effect of ZnO NPs content on the adsorption was studied towards three pollutants. The influence of several factors was studied to determine optimum condition for adsorption of three pollutants.

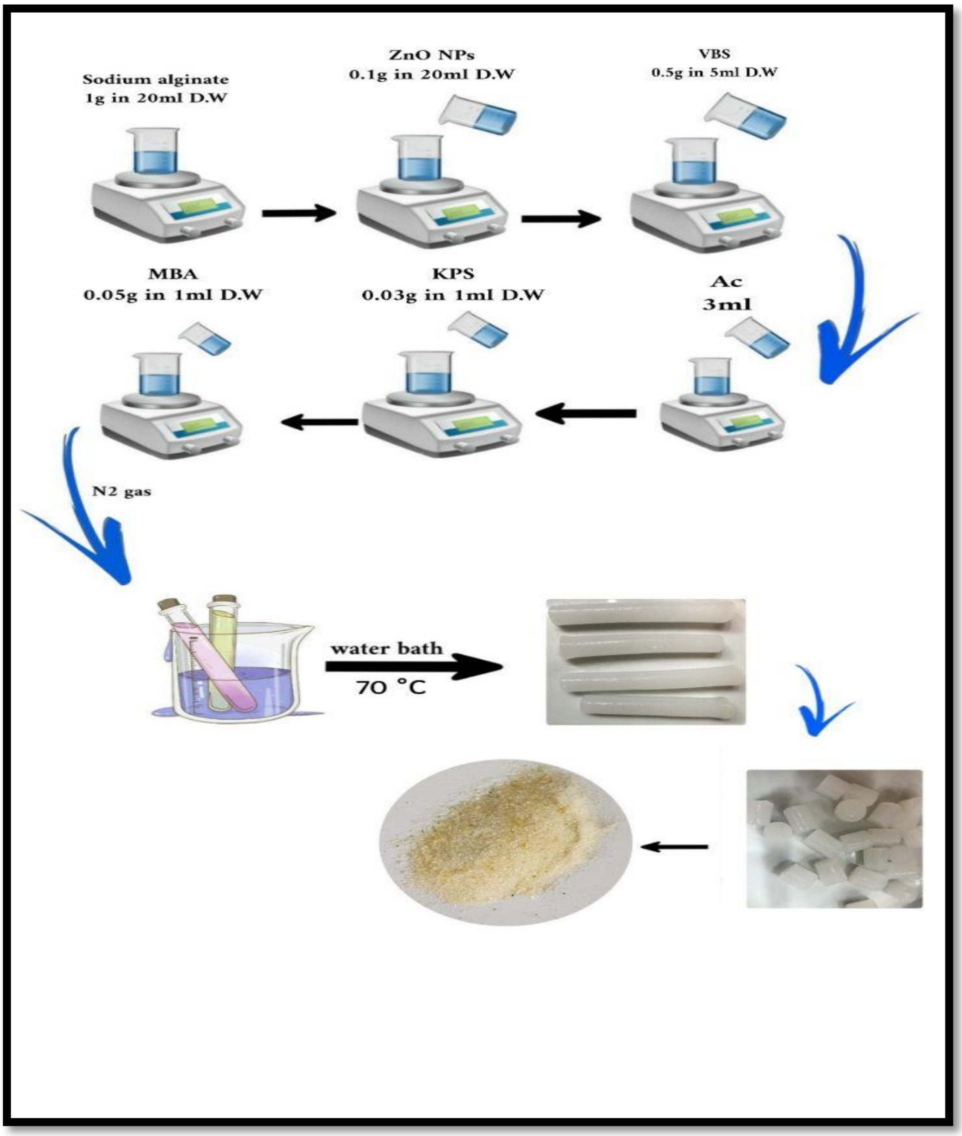
Experimental

Material

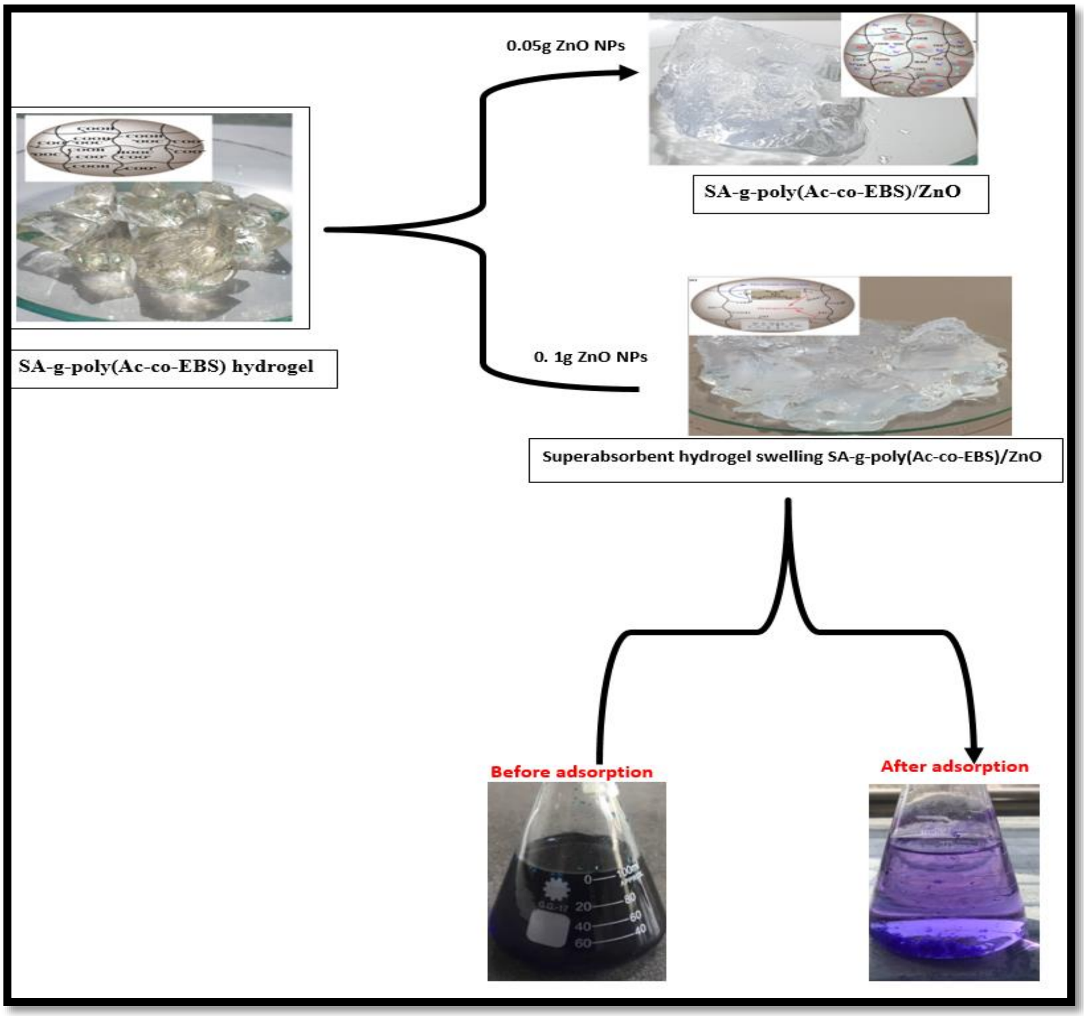
Sodium alginate (SA), Sodium-4-ethenylbenzenesulfonate, hydrate (EBS), Acrylic acid (AA), potassium per sulfate (KPS), N´N-dimethyl acrylamide (MBA), zinc oxide(ZnO), crystal violet (CV) dye, tetracycline (TC) drug, and phenol (PH) were all analytical grade and obtained from Sigma Aldrich.

Preparation and enhanced behavior swelling of superabsorbent hydrogels reinforced with ZnO NPs

Superabsorbent SA-g-poly(Ac-co-EBS)/ZnO hydrogel composite was prepared based on co-polymerization of free radical, and then based on sodium alginate (SA), of which 1 g was dissolved, using ZnO NPs. (0.1 g) in 20 mL of distilled water, after 2 h, added (0.5 g) sodium-4-ethylbenzene sulfonate (EBS) followed by acrylic acid (AC) 3 mL. After that, initiator potassium was added per sulfate KPS 0.03 g and cross-linked N, N'-methylene-bis-acrylamide (MBA) (0.05 g) in 5 mL distilled water. The mixture was placed in a water bath at 70 °C, It has been determined that the creation of the transparent, insoluble hydrogel known as SA-g-poly(Ac-co-EBS)/ZnO hydrogel composite. To obtain particles with the sizes of 180-425 μm, hydrogel was thoroughly washed with an ethanol/water mixture (4:1, v/v), dried in an oven, and sieved to complete the reaction and form the hydrogel, as shown in Scheme 1, and to improve swelling of hydrogel in water by varying weight of ZnO NPs from (0.05-0.1) g, the proposed adsorption process of dye, as depicted in Scheme 1.



SCHEME 1 Preparation of superabsorbent SA-g-poly(Ac-co-EBS)/ZnO hydrogel composite



SCHEME 2 Enhancement of the swelling behavior of superabsorbent hydrogels reinforced with ZnO NPs and adsorption process

Determination of optimum wavelengths (λ_{max}) and calibration curves of (Crystal violet (CV) dye, tetracycline(TC) drug, and phenol (PH))

A Crystal violet dye is a very well-known cationic dye that has a molecular formula ($C_{25}N_3H_{30}Cl$), and molecular weight (407.98 g/mol); the dye is odorless Violet powder applied for various purposes, also extensively used in textile dying and paper printing. A Tetracycline (TC) drug is a very well-known antibiotic that has a molecular formula ($C_{22}H_{24}N_2O_8$), and molecular weight (444.4 g/mol); the drug is yellow powder used for various purposes, also extensively used human and veterinary medicine for the treatment of bacterial. Phenol (PH) is a very well-known pollutant that has a molecular formula (C_6H_6OH), and molecular weight (49.11 g/mol); phenol is colorless utilized on large scales in chemical industry including the production of pesticides, pharmaceuticals, and dye.

A stander solution (1000 mg.L^{-1}) was prepared via (1.0 g) of pollution in (1000 mL) D.W. To estimate the maximum wavelength of three pollutants CV, TC, and PH, the UV-Visible absorption spectra of three pollutant solutions was recorded within wavelengths of 200-800 nm. Wavelength λ_{max} CV= 590 nm, λ_{max} TC= 350 nm, and λ_{max} PH= 270 nm in Figure 1.

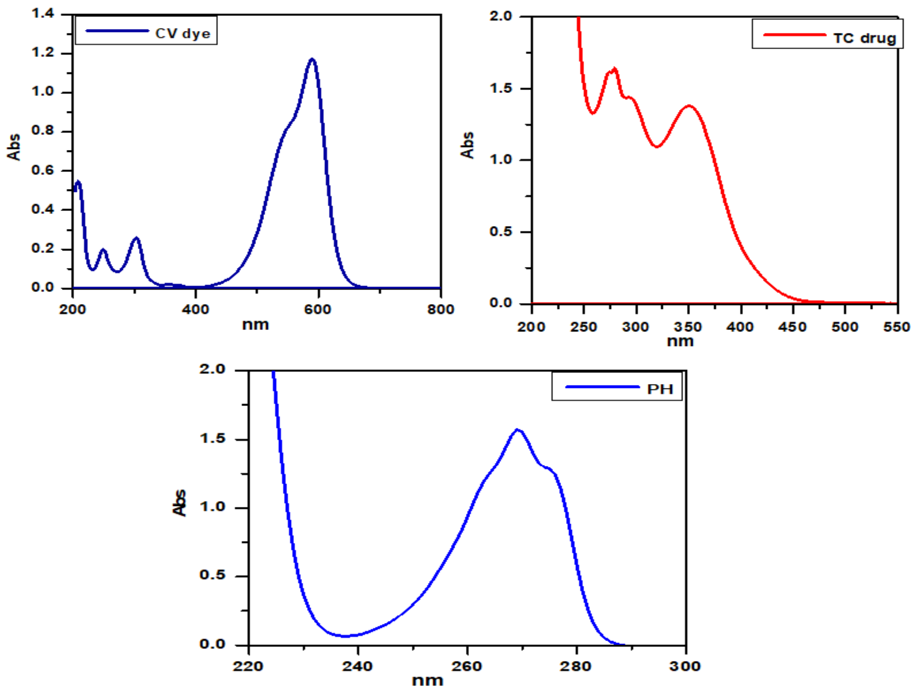


FIGURE 1 UV-Visible absorption spectra of crystal violet (CV) dye, tetracycline (TC) drug, and phenol (PH)

The calibration curve of several concentrations of three pollutants, such as crystal violet (CV) dye, tetracycline (TC) drug and phenol (PH), was prepared in serial dilutions (2-100 mg/L). Absorbance was measured at the λ_{max} of three pollutant and plotted against the concentration values of CV dye, TC drug, and PH in Figure 2.

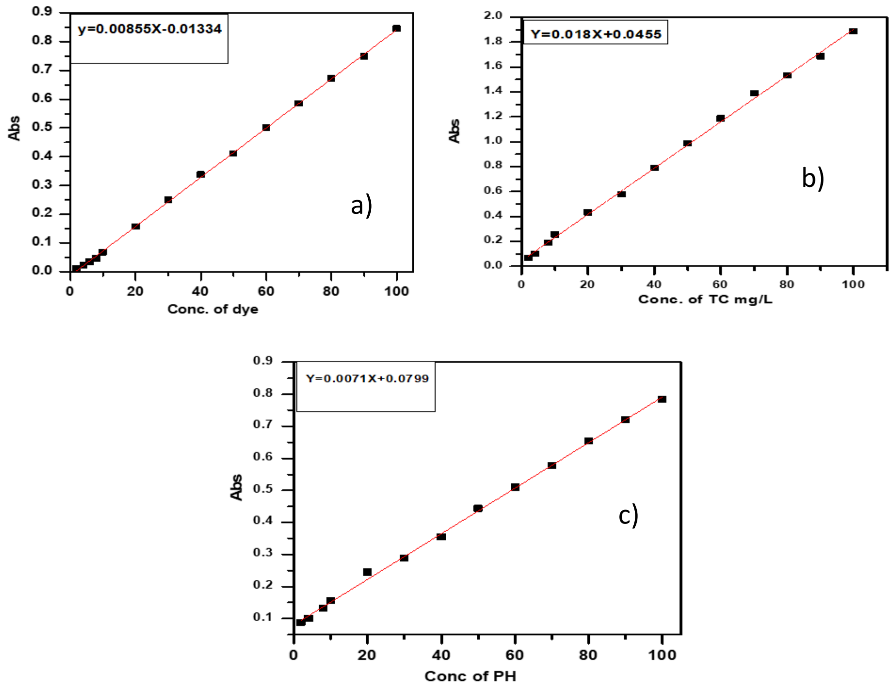


FIGURE 2 Calibration curve for a) crystal violet (CV) dye, b) tetracycline (TC) drug, and c) phenol (PH)

To determine the accuracy and precision of the process, solutions should have ten different concentrations. The measured detection limit LOD, limits of Quantitation's, LOQ, SD, and RSD% are provided in (Table 1) The limit of detection (LOD) and limits of Quantitation's (LOQ) was experimentally calculated using Equations (1 and 2) [17,18].

$$\mathrm{LOD} = \frac{3\ \mathrm{S.D}}{\mathrm{b}} \quad (1)$$

$$\mathrm{LOQ} = \frac{10\ \mathrm{S.D}}{\mathrm{b}} \quad (2)$$

Where, S.D is the standard deviation and *b* is the sensitivity, namely the slope of the calibration curve.

TABLE 1 Statistics result of calibration for several concentrations of CV dye, (TC) drug, and phenol (PH)

Parameters	Proposed Method CV	Proposed Method TC	Proposed Method PH
λ_{max} (nm)	595	335	272
Beer's law limit (µg/mL)	2-100	2–100	2–100
Regression equation	(Y= m X+C) Y=0.0086X- 0.033	(Y = m X + C) Y=0.0186X+0.0456)	(Y = m X + C) Y=0.0071+0.0799)
Slope (m)	0.0086	0.0186	0.0071
Intercept (C)	-0.0133	0.0456	0.0799
Correlation coefficient (r^2)	0.9997	0.9988	0.9987
Color	Violet	Yellow	colorless
Detection limit LOD (µg/mL)	1.028*10-4	1.012*10-4	1.033*10-4
limit of Quantitation LOQ (µg/mL)	3.42*10-4	3.43*10-4	1.87*10-4
% Relative Standard deviation (RSD%)	90.198	75.3314	63.988
standard deviation (SD)	0.2947	0.6381	0.2445
Molar absorptivity (L/mol.cm)	3.508*103	8.226*104	6.681*103
Sandal's sensitivity (µg/cm)	0.116*10-6	0.054*10-6	0.0734*10-6

Removal of pollutants (pharmaceutical, dyes, and chemical compound) using hydrogel nanocomposite.

A laboratory sample of 100 mL of pharmaceutical pollutants containing (Amoxicillin (AMX), phenylephrine hydrochloride (PHE), Tetracycline (TC), Paracetamol (PR), Vitamin B6 (pyridoxine), Riboflavin (RF), and day pollutant (Crystal Violet (CV), Brilliant Blue (BB), Methylene Blue (MB), Congo red (CR), Direct yellow (DY), Reactive blue (RB), and other sample by mixing several pollutants (Phenol (PH), 4-Clorophenole (CPH), and Amoxicillin (AMX), phenylephrine hydrochloride (PHE), Tetracycline (TC), paracetamol (PR), Vitamin B6 (pyridoxine), Riboflavin (RF), Brilliant Blue (BB), Crystal Violet (CV), Methylene Blue (MB), Congo red (CR), and Direct yellow (DY)) with a riffle concentration were used in this experiment, and then about 0.04 g of nanocomposite was added in 100 mL conical flask. After that, the mixture was put in Shaker water bath for 1 h.

Results and discussion

FESEM and TEM

FE-SEM has been utilized as a primary instrument to characterize the morphology surface and fundamental physical possessions of the adsorbent. The micro-graphs of hydrogel show that flat porous surface (Figure 3a), and Hydrogel/ZnO NPs not smooth porous surface and appear changes in the morphology of phase for new irregular bulky particles presence on the Hydrogel/ZnO NPs surface [19]. This leads to increased surface texture protuberance and coarseness. This increases the surfaces of the absorbents that facilitate water diffusion in to the absorbent (Figure 3b). FESEM of adsorbent material was taken before and after CV dye, TC drug, PH adsorption on Hydrogel/ZnO NPs surface (Figure 3c, 3d, and 3e), respectively [20,21]. Figure 3f) shows the TEM images of ZnO Hydrogel/ZnO NPs, where ZnO NPs was observed embedded inside the Hydrogel.

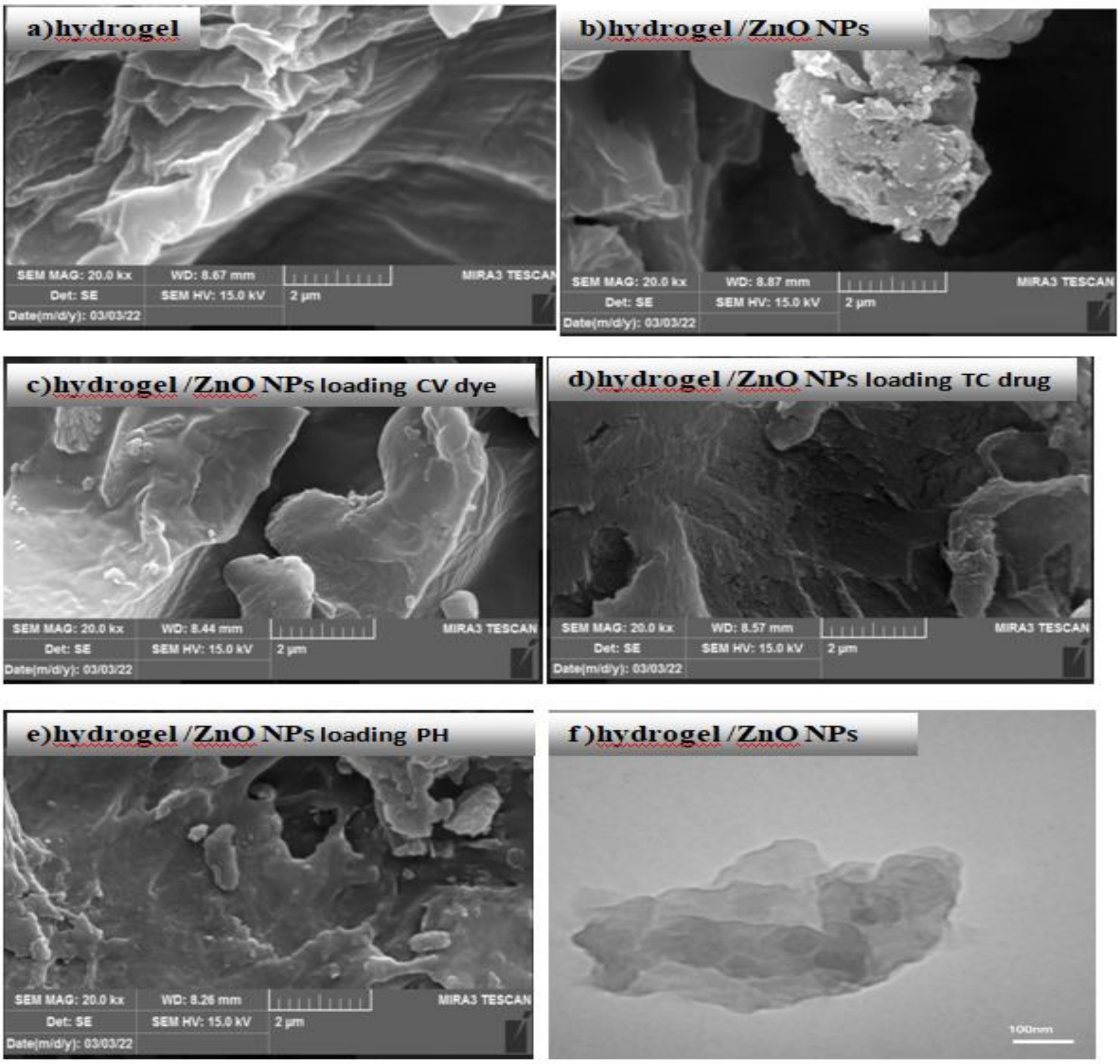


FIGURE 3 FESEM images of a) hydrogel, b) hydrogel/ZnO NPs, c) hydrogel/ZnO NPs loading CV dye, d) hydrogel/ZnO NPs loading TC drug, e) hydrogel/ZnO NPs loading PH, and f) TEM hydrogel/ZnO NPs

Removal of pollutants by Using SA-g-poly(Ac-co-EBS)/ZnO hydrogel composite

A laboratory sample of 100 mL of pollutants ((pharmaceutical, dyes, and chemical compound)) with a refry concentration were used in this experiment, and then about 0.04 g hydrogel/ZnO was added in 100 mL conical flask, and mixture was put in a shaker water bath for 2 h. After that, the supernatant was separated via centrifuge and the remaining concentration was measured using UV-Visible spectrophotometer [22,23]. The result was shown in Figures (4-6).

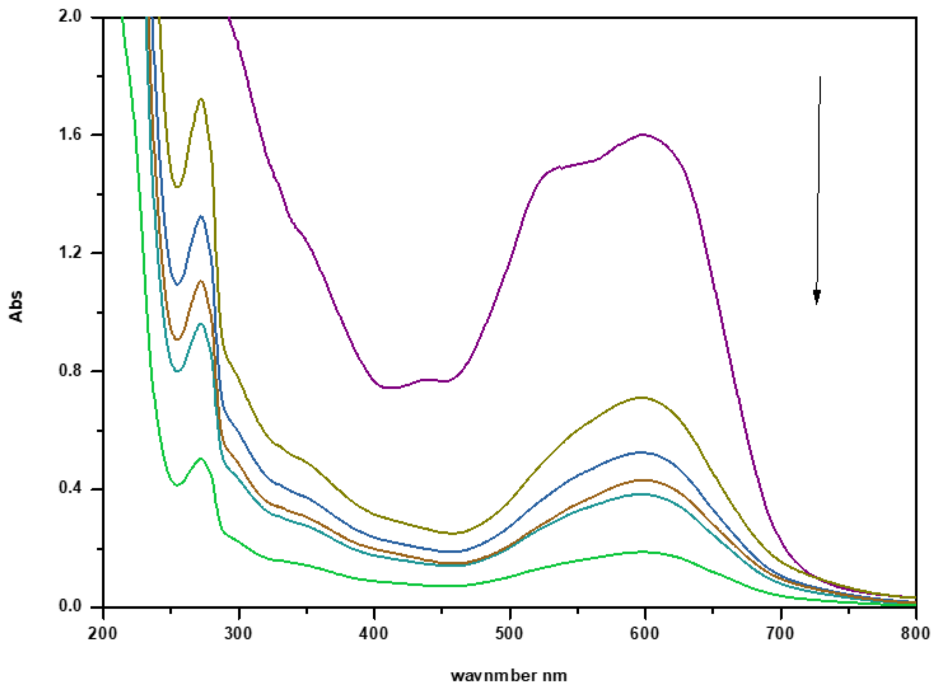


FIGURE 4 Spectrum of removal pollutants (dyes and drugs) using Hydrogel/ZnO NPs), Exp. Condition: contact time 2 h, weight absorbent 0.04 g, and Temp. = 30 °C)

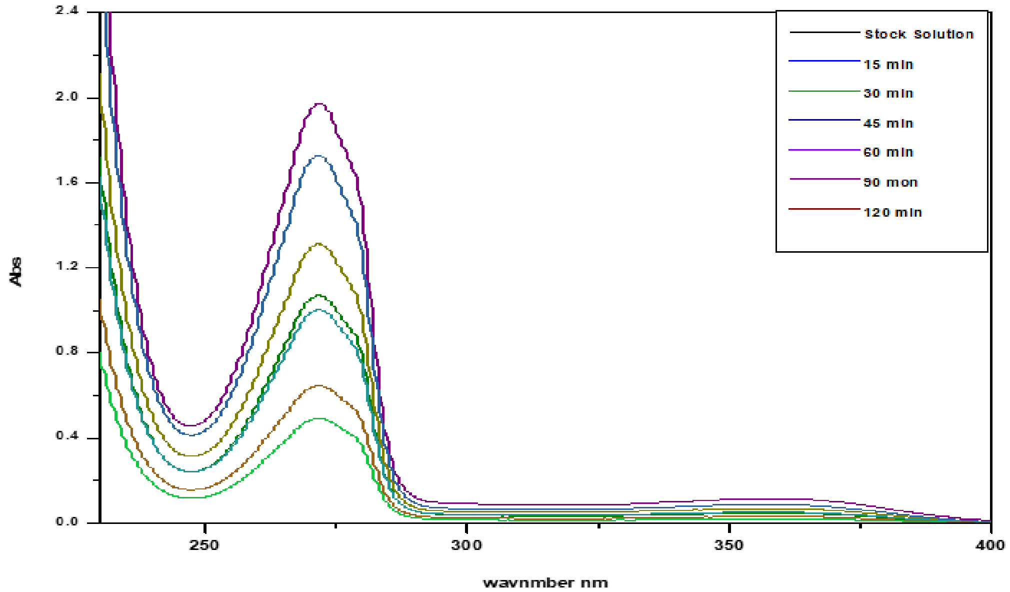


FIGURE 5 Spectrum of removal pollutants (drugs) using Hydrogel/ZnO NPs Exp. Condition: contact time 2 h, weight absorbent 0.04 g, and Temp. = 30 °C)

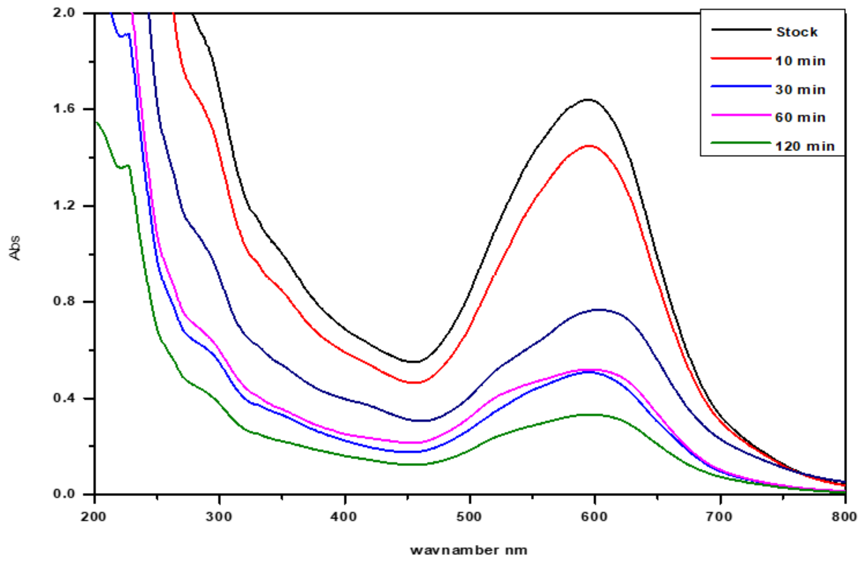


FIGURE 6 Spectrum of removal pollutants (dyes) by Hydrogel/ZnO NPs), (Exp. Condition: contact time 2 h, weight absorbent 0.04 g, and Temp. = 30 °C)

Comparative adsorption between different surfaces

A comparative study of (ZnO NPs, (SA-g-(PAAc-co-EBS) hydrogel, and (SA-g-(PAAc-co-EBS/ZnO) hydrogel composite) surfaces as adsorbents wear was carried out. A sample of 100 mL of three pollutant concentration (100 mg.L-1) are used in this study, and then added to a conical flask (Erlenmeyer) in the presence of 0.04 g from prepared (ZnO NPs, (SA-g-(PAAC-co-EBS) hydrogel, (SA-g-(PAAC-co-EBS/ZnO) hydrogel composite), and put in a shaker water bath for 2 h. After that, the supernatant was separated by centrifuge and measured the remaining concentration using UV-Visible spectrophotometer at the λ_{max} nm [22,23]. The best data of the removal percentage (E%) for three pollutants in order increasing: (SA-g-(PAAC-co-EBS)/ZnO) hydrogel composite > (SA-g-(PAAC-co-EBS) hydrogel > ZnO NPs is presented in in Figure 7.

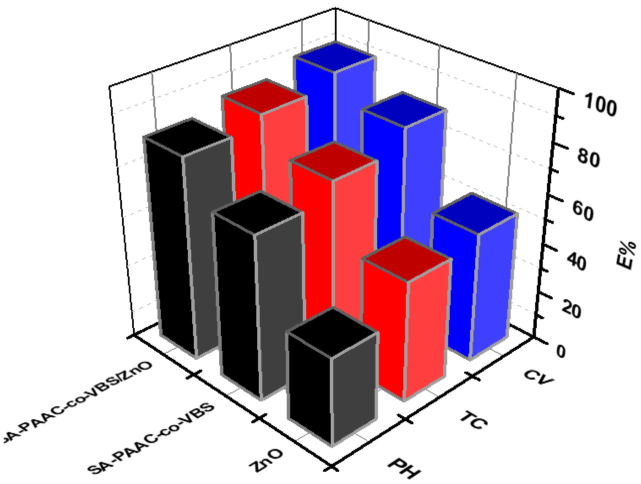


FIGURE 7 Comparative effect of adsorption between different surfaces

Also, the comparison between the amounts of Zinc oxide (ZnO NPs) decorated of (SA-g-(PAAC-co-EBS) hydrogel using (0.05, 0.08, 0.1, and 0.15 g) is shown in Figure 8. The best results of the removal percentage (E%) of three pollutants (CV, TC, and PH) about 0.1 g ZnO (92.451%, 88.55%, and 82.44%) are at the same order .

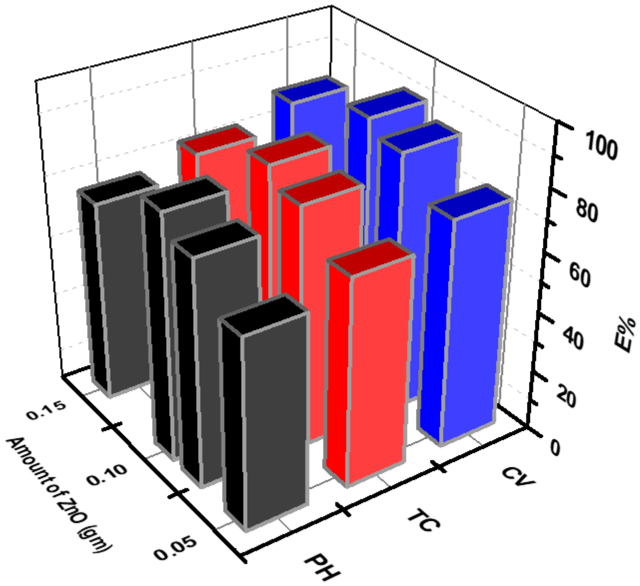


FIGURE 8 The effect comparative between the amounts of ZnO decorated SA-g-(PAAC-co-EBS) hydrogel

Regeneration and desorption of (SA-g-(PAAc-co-EBS)/ZnO) hydrogel composite

The regeneration of hydrogels, after sorption, is one of the important economic parameters for the treatment method. It helps elucidating the mechanism of removal three pollutants (CV, TC, and PH) from Pollutant -loaded adsorbent, re-generation mechanism, and re-cycling of spent adsorbents, which in turn may reduce operational cost and protect the environment from secondary pollution. Three pollutant (CV, TC, and PH) desorption studies were carried out using several desorption agents at concentration (0.01 N) like NaOH, H_2SO_4, HCl , H_3PO_4 , HNO_3, and water [13, 30-32]. The hydrogel composite was regenerated with 100% that can be desorbed in diluted hydrochloric acid solution because the –COO– is converted into –COOH in acidic solutions, and correspondingly, the electrostatic interactions between (SA-g-(PAAC-co-VBS)/ZnO) and three pollutant (CV, TC, and PH) are weakened. The performance and reuse of hydrogel composite using HCl solution in three pollutant adsorption processes was investigated up to 3 steps under the optimal conditions [21,24]. After 3 cycles of using (SA-g-(PAAc-co-EBS)/ZnO NPs), the efficiency is still significant (>80%) and this shows that (SA-g-(PAAc-co-EBS)/ZnO NPs) is probable renewable absorber, as shown in Figure 9.

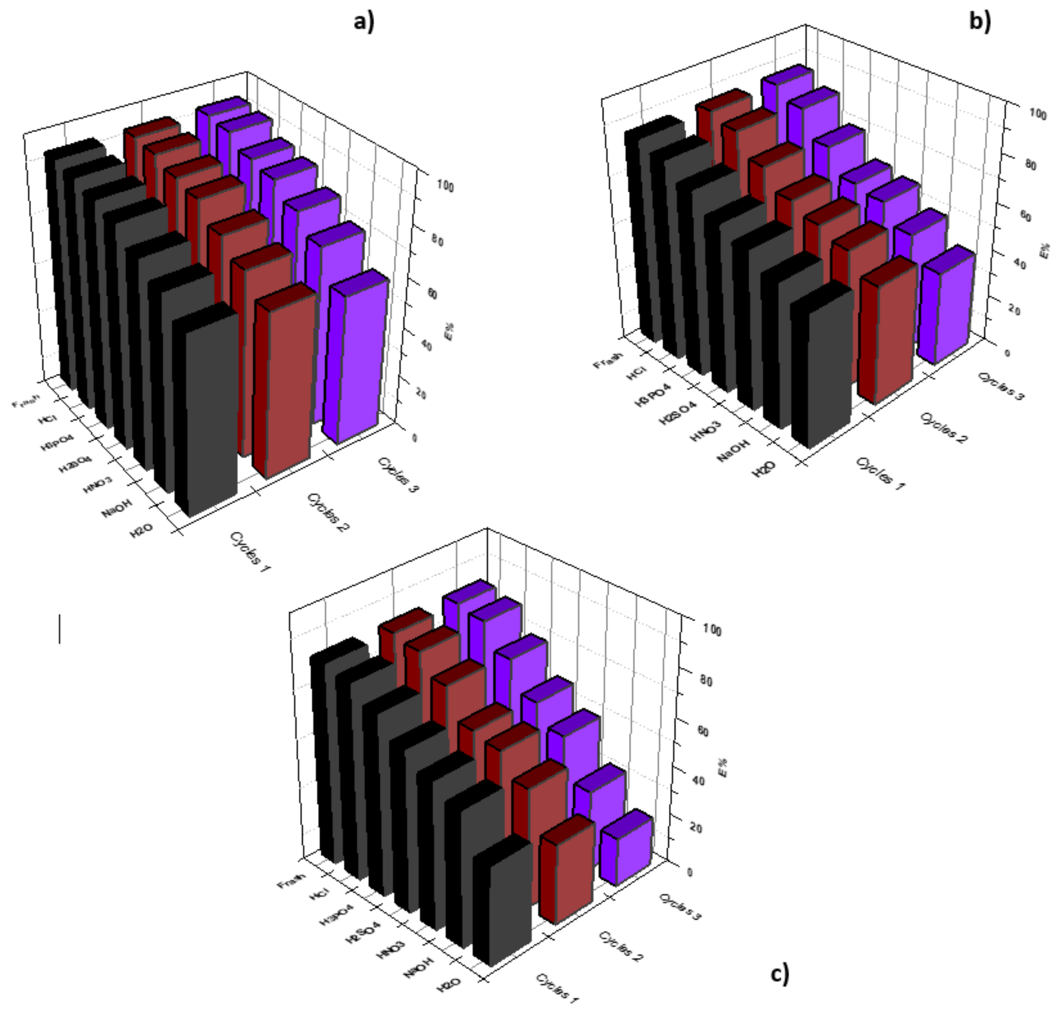


FIGURE 9 Comparison of a) CV dye, b) TC drug, and c) PH of percentage removal (E%) using fresh and three cycles regenerated hydrogel composite

Adsorption models

Freundlich model

The Freundlich model is defined through the following Equation (1) [25].

$$q_e = K_f C_e^{1/n} \quad (3)$$

Where, q_e is the adsorbed amount per unit mass of adsorbent at equilibrium (mg/g), (mol/g), C_e is the equilibrium concentration (mg.L^{-1}), (mol/L), K_f is the empirical Freundlich constant or capacity factor (L/mg), and 1/n is Freundlich exponent, if the value of n is equal to unity, the adsorption is linear; if below to unity, adsorption process is chemical and if the value is above unity, adsorption is a physical process [26,27].

Langmuir model

The Langmuir model is mostly utilized for three pollutants adsorption from liquid solutions. The nature of the adsorption method was derived via Langmuir alternative Equation (2) [28].

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \quad (4)$$

The coefficients of estimation (R^2) and isotherm factors from the nonlinear regressive process were listed in Table 2. A comparison of nonlinear fitted curves from experimental result and several models at 30 °C are illustrated in Figure 10. A plot of q_e vs. Ce, where the values of K_F and 1/n are obtained from the intercept and slope of the linear regressions (Table 2). The R^2 values for the isotherm Freundlich at 30 °C are (R^2=0.9969), (R^2=0.9899), and (R^2=0.9697) of CV, TC, and PH on to hydrogel nanocomposite, respectively [29].

TABLE 2 Several factors isotherm for the adsorption study of three pollutants onto hydrogel/ZnO nanocomposite

		Hydrogel/ ZnO nanocomposite		
Type of pollutant		**CV dye**	**TC drug**	**PH**
	K_f	152.0695 ± 8.3612	50.405± 2.964	60.9307 ± 5.638
Freundlich	1/n	0.3917 ± 0.0133	0.4675 ± 0.023	0.4444 ± 0.0408
	R^2	0.9969	0.9899	0.9697
	q_m (mg/g)	1056.623 ± 122.63	259.156 ± 33.456	282.599 ± 53.845
Langmuir	K_L (L/mg)	0.05124 ± 0.01907	0.1465 ± 0.0474	0.1581 ±0.0716
	R^2	0.9185	0.9309	0.8862

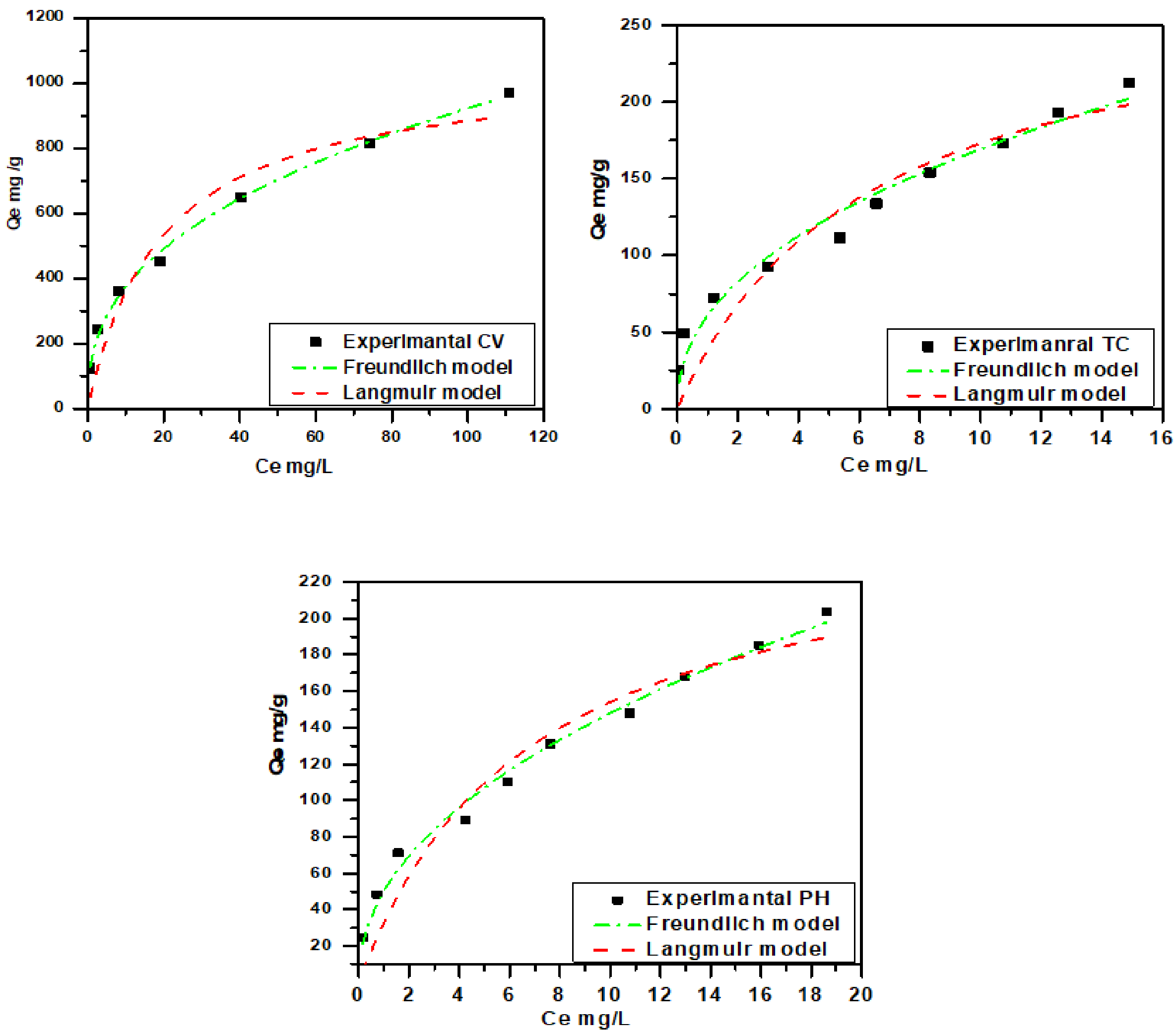


FIGURE 10 Several adsorptions models of nonlinear fit of adsorption CV dye, TC drug, and PH onto hydrogel/ZnO nanocomposite

Conclusion

One of important kinds of eco-friendly and low-cost organic or inorganic super-absorbent hydrogel composite was prepared by free radical co-polymerization of sodium alginate (SA), acrylic acid (AA), and Zinc oxide in aqueous solution. The best equilibrium time for equilibrium to be achieved is found to be 2 h. The interaction between dose and initial concentration showed a significant effect on the adsorption process. Adsorbent showed fits better to isotherm Freundlich which suggests that adsorption is heterogeneous. Removal of laboratory aqueous pollutants (dyes, drugs) of hydrogel composite was done to give low absorbance (0.0001) using UV-Visible spectrophotometer for at a chosen wavelength for 2 h. The best percentage of removal (E%) for three pollutants the order increasing: SA-g-poly(Ac-co-EBS)/ZnO hydrogel composite NPs > SA-g-poly(Ac-co-EBS) hydrogel > ZnO NPs. 10. The (SA-g-(PAAc-co-EBS)/ZnO NPs was regenerated with 100% can be desorbed in diluted hydrochloric acid solution in the three pollutants (CV, TC, and PH). The adsorption process was investigated up to 3 steps under the optimal conditions.

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Conflict of Interest

The authors declare that there is no conflict of interest.

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