

RESEARCH ARTICLE

Enhanced Removal of Phenylephrine Hydrochloride using Eco-friendly Surface: Optimization, Isotherm, Kinetics, and Regeneration Studies

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ABSTRACT

Phenolic compounds are highly toxic pollutants. They are widely used in tanning, dye, chemical, cosmetic and pharmaceutical industries. They come from different sources, like industrial wastewater, gasworks, paper mill, chemical plants, coking factories, pharmaceutical industry, and solid castoff of coal tar. This study used a phenolic Phenylephrine hydrochloride PHCL drug, characterized by a very high toxicity in water. Therefore, a new surface with very high efficiency was prepared to remove this drug from water. The hydrogel surface properties such as fourier transform infrared spectroscopy (FTIR), field emission scanning electron microscope (FESEM) were studied. Two types of Frenelich and Langmuir isotherms were also studied. Through the results, it was found that obeys the Freundlich model. Three models of Kinetic first-order, second-order, and Elcovich models were found through the data to obey a false first-order model.

Keywords: Adsorption, Drug, Hydrogel, Isotherm, Kinetic model, Phenylephrine hydrochloride.

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INTRODUCTION

Pollution is the most widespread major problem that has caused a defect in the ecosystem and a dangerous problem that threatens human life. Therefore, it is difficult to obtain clean water in the existence of large amounts of wastewater. It is found that in all industrial countries the ratio of water pollution that affects the human system is increased.¹⁻⁵

Pharmaceutical products are used for human health promotion, animal care, and agriculture applications. Antibiotics are commonly used drugs for preventing or treating microbial infections.⁶⁻⁸ They are released into the water through wastewater treatment and pharmaceuticals plants. Pharmaceuticals are represented as bio-accumulative contaminant compounds in the environment of aquatic or terrestrial ecosystems.⁹ Oxidation processes are costly and difficult for the complete elimination of antibiotics. Adsorption is a physical technique is the most suitable and efficient treatment option for removing organic compounds for its high efficiency, simplicity, and economical in nature.¹⁰⁻¹³ Phenylephrine hydrochloride (PHE) (Nec-synephrine) is (R)-1-(3-hydroxyphenyl)-2-methyl-aminoethanol hydrochloride as shown in Figure 1. It is closely related to epinephrine¹⁴⁻¹⁶ is a crystalline white or pale-yellow salt fairly soluble in

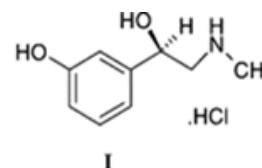


Figure 1: Chemical Structure Phenylephrine hydrochloride.¹⁷

water. It acts as a vasoconstrictor with negligible effect on the myocardium or the central nervous system. It is used as nose drops or subcutaneous injections to prevent hypotension during spinal anesthesia and treat hypertension, rhinitis, and sinusitis. Some selected physicochemical properties of PHE as shown in Table 1.

Preparation of Calibration Curve Phenylephrine Hydrochloride (PHCL)

Aliquots of a stock solution having several concentration (10–100 mg/L) of (PHCL) drug was prepared via simple dilution by DW of the standard solution (1000 mg/L). The aqueous solutions were prepared freshly. The absorbance was an estimation spectrophotometric ally utilizing UV-visible at λ_{\max} 277 nm. Estimation the accuracy and precision of the way, solutions having five several conc (Figure 2). The measured limit of detection LOD, Quantitation's, LOQ,

relative standard deviations percent (RSD%) and standard deviation (S.D.) (Table 2).²⁰

The (LOD) for the projected way was estimation utilizing equation (1). The S/N equal 3.^{21,22}

$$\text{LOD} = \frac{3 \text{ SD}}{\text{slope}} \quad (1)$$

The (L.O.Q) was estimation utilizing equation (2):^{21,23}

$$\text{LOQ} = \frac{10 \text{ SD}}{\text{slope}} \quad (2)$$

RESULT AND DISSECTION

FESEM

The study of the morphological properties of the surface of a hydrogel was studied by (FESEM) for a surface before and after the adsorption process, where it was noticed from the figure that the surface of the hydrogel formed white clumps and clumps similar to a cloud (Figure 3). However, after the adsorption process the surface became less rough as a result of the filling of most of the active sites of the surface and the surface became blackish evidence of the occurrence of the process adsorption.^{24,25}

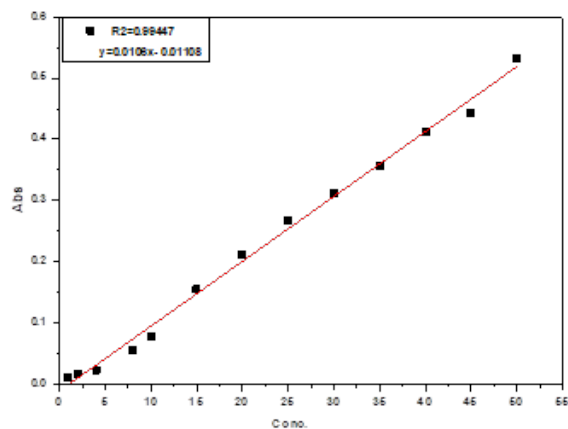


Figure 2: Calibration curve of PHCL.

FTIR

The hydrogel was characterized through spectroscopy FTIR from 4000–400 cm^{-1} with a resolution of 1 cm^{-1} . The IR spectra of hydrogel before and after PHCL adsorption are look in Figure 4, it is observed from the figure after the adsorption process that no new pick appears, only there is a slight change in the intensity of adsorption, and this is evidence of the occurrence of the adsorption method and the adsorption process is of physic sorption.^{26,27}

Table 1: Physicochemical properties of phenylephrine hydrochloride¹⁷⁻¹⁹

Physicochemical properties	Phenylephrine HCl
Molecular Formula	$\text{C}_9\text{H}_{14}\text{ClNO}_2$
Molecular weight (g/mole)	203.67
Solubility in water (mg/L)	100
Melting point	143–145 °C
Boiling point	700.2 °C at 760 mm Hg
Acid dissociation constant (pKa)	pK1 8.77; pK2 9.84

Table 2: Statistical of calibration curve (Figure 2) at several conc. of PHCL

Parameters	Proposed method PHE
λ_{max} (nm)	277
Beer's law limit ($\mu\text{g/mL}$)	1–100
Molar absorptivity (L/mol.cm)	2227.38
Sandal's sensitivity ($\mu\text{g/cm}$)	$1.035 * 10^{-7}$
Regression equation	$(Y = m X + C)$ $Y = 0.0106X + 0.01105$
Slope (m)	0.0106
Intercept (C)	0.01105
Correlation coefficient (r^2)	0.99447
% Relative Standard deviation (RSD%)	0.344
Standard deviation (SD)	0.211
Color	Color less
Detection limit LOD ($\mu\text{g/mL}$)	$1.79 * 10^{-3}$
Limit of Quantitation LOQ ($\mu\text{g/mL}$)	$1.9 * 10^{-3}$

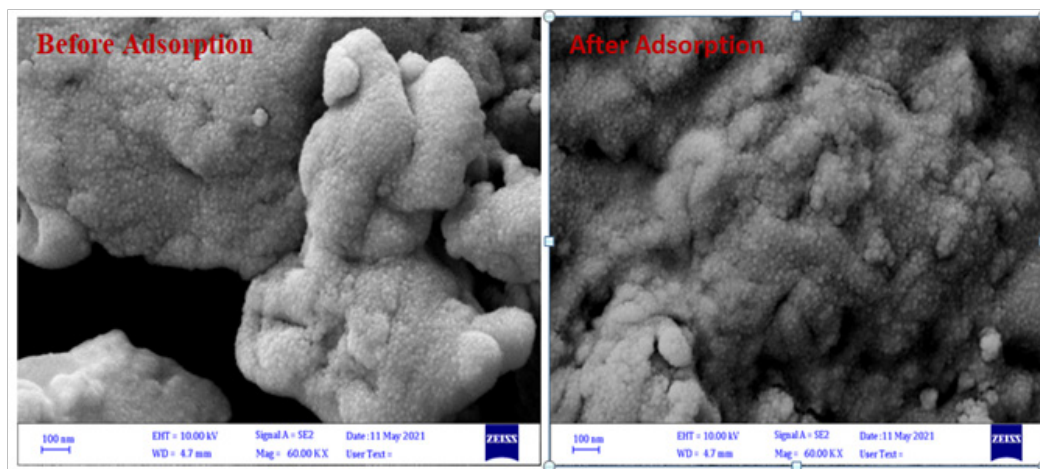


Figure 3: FESEM of hydrogel before and after adsorption

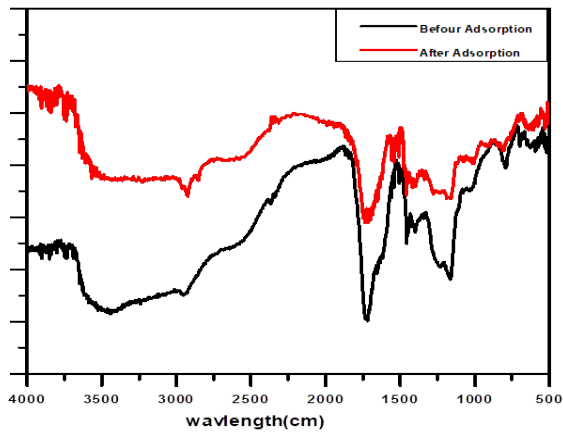


Figure 4: FTIR spectrum of hydrogel before and After adsorption

Effect of Concentration of Drug

Through Figure 5, several concentrations from (10–100 mg/L) of a PHCL drug were studied within the optimum condition: weight 0.05 gm, temperature 25°C, and pH 7.7. Where it was observed that with increasing PHCL drug concentration, the adsorption efficiency increased from (20–177 mg/g) in contrast to the removal percentage E%, it was observed with increasing PHCL drug concentration that it decreases from (87%–98%) and that depends on the number of active sites present on the surface when the concentration increase active sites decrease.^{28,29}

Surface Revitalization Experiments

The surface was reactivated using three solutions (distilled water, acid and base), where it was found that the best results were when using distilled water, as it is preferable to reactivate the surface and reuse it again, but the more surface activation processes, the lower the surface efficiency,³⁰ as shown in Table 3.

Adsorption Isotherms

Two model equations exist to study the adsorption equilibrium factor, and the supreme public being is the Freundlich, Langmuir model. Langmuir model (Figure 6) built on the theory that there is a fixed amount of active sites that are orderly dispersed through the surface of hydrogel, active sites have the identical ability for adsorption of a monolayer no interaction among molecules adsorbed. The Freundlich model (Figure 6) is viable for hetero-generous adsorption of the surface. This isotherm assumes the relationship is positive among adsorbate conc. adsorbent quantities on the surface. Estimating R2 via obeying the data equilibrium result for the hydrogel method utilizing Langmuir and Freundlich models. Figure 6 appear the best (R² = 0.9444) off to the Freundlich isotherm.^{31,32}

Adsorption Kinetics

The adsorption kinetics model gives information about adsorption mechanics. In this study, three models of kinetics adsorption were utilized, first order, second order, and Elcovich model. The first order as appearing in equation (5)

$$qt = qe [1 - \exp(-kf t)] \tag{5}$$

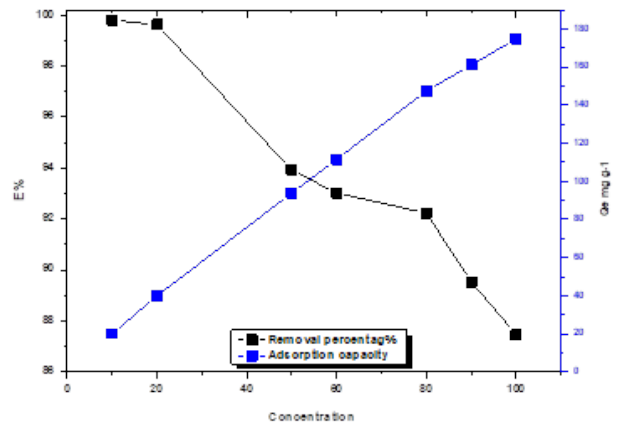


Figure 5: Effect of concentration of PHCL drug

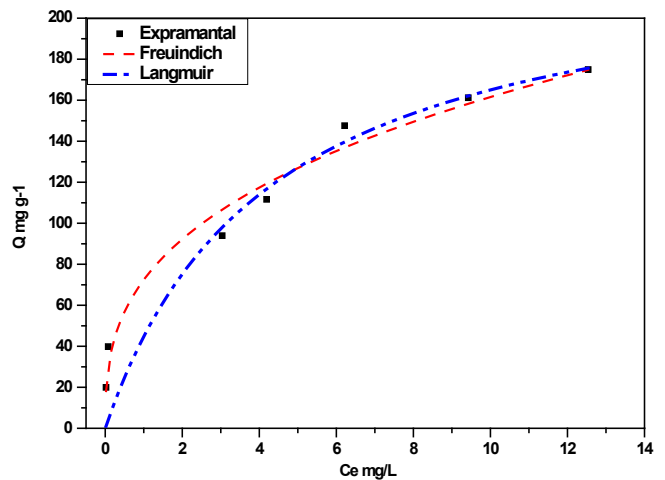


Figure 6: Different absorption isotherm patterns non-linear fit for absorbing the PHCL drug on hydrogel, primary conc. = 100 mg.L⁻¹, Temp. = 25°C, the adsorbent = 0.05 g

Table 3: The Freundlech, Langmuair, the model factors for PHCL drug absorbed onto hydrogel at 25°C.

Isotherm models	Parameters	PHCL
Langmauir	qm (mg.g ⁻¹)	235.16 ± 47.091
	K _L (L.mg ⁻¹)	0.235 ± 0.121
	R ²	0.9026
Freundlech	K _F	72.43 ± 6.111
	1/n	0.347 ± 0.033
	R ²	0.9745

The model kinetics of the adsorption process might too be called in the second equation.³³ The non-linear form of t equation is expressed as:

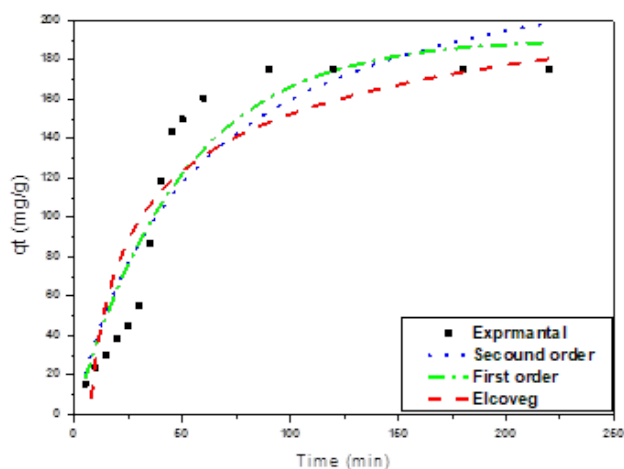
$$qt = K2qe2t/(1+K2qet) \tag{6}$$

The structure non-linear of the Elovich model (Chemi-sorption kinetic model) [49] model equation is commonly written as:

$$qt = 1/\beta [1/\ln(\alpha\beta)] + 1/\beta \ln t \tag{7}$$

Table 4: Kinetic model first model, second model, and Elcovich model correlation coefficients for PHCL drug adsorption on the hydrogel.

Model	Equation	Parameters	Value
First model	$qt = q_e [1 - \exp(-kt)]$	Kt (min^{-1})	0.21 ± 0.0122
		q_e (calc) (mg.g^{-1})	190.59 ± 4.122
		R2	0.8833
Second model	$qt = K_2 q_e^2 t / (1 + K_2 q_e t)$	K_2 (g/mg/min)	0.3103 ± 0.0410
		q_e (calc) (mg.g^{-1})	249.90 ± 2.563
		R2	0.8477
Elcovich model	$qt = 1/\beta \ln(\alpha\beta + 1)$	α ($\text{mg.g}^{-1} \text{min}^{-1}$)	0.433 ± 0.022
		β (g.min^{-1})	177.03 ± 1.44
		R2	0.777

**Figure 7:** First-order reaction kinetics, second-order and Elcovich model of PHCL drug adsorption on the hydrogel.

Three kinetics models of the adsorption method, which define the experimental result, elected for adsorption of PHCL drug on the hydrogel as appear in Figure 7. The resulting study was done utilizing the model first and second order. Table 4 data show that adsorption of PHCL drug via hydrogel is an achievable method because the value (R2) is best of model second-order compared to model first-order.^{13,34,35}

CONCLUSION

- In this study, a highly efficient and effective hydrogel surface was prepared to remove the pollutants present in the water.
- With an increase in the concentration of phenylephrine, the removal percentage decreases; on the contrary, the efficiency of adsorption increases
- The results obey the Freundlich model depending on the value of ($R_2=0.974$)
- Three Kinetic models were studied, and it was found that it obeys a model First model.
- Several techniques were measured for the prepared surface like FESEM and FTIR.

REFERENCES

1. Radhi IM, Abdulrazzak FH, Himdan TA. Influence of water in size of Synthesized Carbon Black Nanoparticles from Kerosene

by Flame Method. IOP Conf. Series: Materials Science and Engineering. 2019;571.

2. Walter K, Dodds M. Chapter 16 - Responses to Stress, Toxic Chemicals, and Other Pollutants in Aquatic Ecosystems, in Aquatic Ecology. 2020; 453-502.
3. Nupur N, Kriti D, Abhinav S, Rekha S. Supercritical Extraction of Valued Components From Animals Parts. Food Science. 2021;1:doi.org/10.1016/B978-0-08-100596-5.22673-5.
4. Romero F, Sabater S, Font C, Balczar J L, Acua V . Desiccation events change the microbial response to gradients of wastewater effluent pollution. Water Research. 2019;151:371-380.
5. Basam WM, Nader DR, Layth SJ, Hayder OJ. Synthesis and characterization of polyacrylamide hydrogel for the controlled release of aspirin. J. Pharm. Sci. & Res. 2018;10(11): 2850-2854.
6. Ali M. Jassm, Joud B A, Abdulrazzak FH, Alkaim AF, Hussein FH. Synthesis and Characterization of Carbon Nanotubes by Modified Flame Fragments Deposition Method. Asian journal of chemistry. 2017;29(12):2804-2808.
7. Aljeboree AM, Abbas NA. Colorimetric determination of Amoxicillin using 4-Aminoantipyrine and the effects of different parameters. Journal of Physics: Conference Series. 2019;12(5): 052067.
8. Danner MC, Robertson A, Behrends V, Reiss J. Antibiotic pollution in surface fresh waters: Occurrence and effects. Science of The Total Environment. 2019;664:793-804.
9. Quesada HB, Baptista A, Takaoka A, Cusioli L, Fernando S, Daiana D, Oliveira B, Charleston B. Surface water pollution by pharmaceuticals and an alternative of removal by low-cost adsorbents: A review. Chemosphere. 2019;222:766-780.
10. Abdulrazzak FH .Enhance photocatalytic Activity of TiO2 by Carbon Nanotubes. International Journal of ChemTech Research. 2016;9(3):431-443
11. Kumar R, Sarmah AK, Padhye LP. Fate of pharmaceuticals and personal care products in a wastewater treatment plant with parallel secondary wastewater treatment train. Journal of Environmental Management. 2019;233:649-659.
12. Turk S. Surface functionalised adsorbent for emerging pharmaceutical removal: Adsorption performance and mechanisms. Process Safety and Environmental Protection. 2019; 125:50-63.
13. Liqaa HA, Abbas R, Aljeboree AM, Abdulrazzak FH, Hussein FH, Alkaim AF. Role of Semiconductors (Zinc Oxide as a Model) for Removal of Pharmaceutical Tetracycline (TCs) from Aqueous Solutions in the Presence of Selective Light. International Journal of Recent Technology and Engineering (IJRTE). 2019;8(2S3): DOI : 10.35940/ijrte.B1270.0782S319.

14. Aljeboree AM, Alshirifi AN. Oxidative coupling of Amoxicillin using 4-Aminoantipyrine: Stability and higher sensitivity. *Journal of Physics: Conference Series*. 2019;1294(5): 052001.
15. Salem Y. Application of derivative emission fluorescence spectroscopy for determination of ibuprofen and phenylephrine simultaneously in tablets and biological fluids. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*. 2019;210:387-397.
16. Águila-Carrasco A. Effect of phenylephrine on static and dynamic accommodation. *Journal of Optometry*. 2019;12(1): 30-37.
17. Zhang Z, Zhang Y, Gerk PM. Preparation of phenylephrine 3-O-sulfate as the major in vivo metabolite of phenylephrine to facilitate its pharmacokinetic and metabolism studies. *Journal of Pharmacological and Toxicological Methods*. 2019;95:66-69.
18. Jasim LS, Radhy ND, Jamel HO. Synthesis and characterization of poly (acryl amide-Maleic acid) hydrogel: Adsorption kinetics of a malachite green from aqueous solutions. *Eurasian Journal of Analytical Chemistry*. 2018;13(1): 10.29333/ejac/101789
19. Maximino MD. Synergy in the interaction of amoxicillin and methylene blue with dipalmitoyl phosphatidyl choline (DPPC) monolayers. *Applied Surface Science*. 2019; 476:493-500.
20. Yaqo EA, Anae RA, Abdulmajeed MH, Tomi IHR, Kadhim MM. Electrochemical, morphological and theoretical studies of an oxadiazole derivative as an anti-corrosive agent for kerosene reservoirs in Iraqi refineries ;*Chemical Papers*. 2020; 74 (6):1739-1757.
21. Al-Jbouri AMS, Abood FM, Hindi NKK, Alkaim AF. Evaluation of antimicrobial activity of the aquatic extract against bacterial isolates from periodontitis in Babylon Province, Iraq; *Biochemical and Cellular Archives*. 2018;18:1345-1350.
22. Al-Duhaidahawi DL, Jabir MS, Al-Amiery AA, Moneim AEA, Alkaim AF, Kadhum AAH. Novel antioxidants compounds derived from isatine *Biochemical and Cellular Archives*. 2018;18 (1):709-713.
23. Abdoon FM, Yahyaa SY. Validated spectrophotometric approach for determination of salbutamol sulfate in pure and pharmaceutical dosage forms using oxidative coupling reaction. *Journal of King Saud University-Science*, 2018. 1: <https://doi.org/10.1016/j.jksus.2018.11.002>.
24. Aljeboree AM, Alkaim FA. Comparative Removal Of Three Textile Dyes From Aqueous Solutions By Adsorption: As A Model (Corn-Cob Source Waste) Of Plants Role In Environmental Enhancement. *Plant Archives*. 2019;19(1):1613-1620.
25. Bader AT, Zaied AM, Aljeboree AM, Alkaim AF. Removal of Methyl Violet (MV) From Aqueous Solutions By Adsorption Using Activated Carbon From Pine Husks (Plant Waste Sources). *Plant Archives*. 2019;19(2): 898-901.
26. Alkaim AF, Alqaragully MB. Adsorption of Basic Yellow Dye From Aqueous Solutions By Activated Carbon Derived From Waste Apricot Stones (ASAC): Equilibrium, And Thermodynamic Aspects *Int. J. Chem. Sci.* 2013; 11(2): 797-814.
27. Aljeboree AM, Abbas AS. Removal of Pharmaceutical (Paracetamol) by using CNT/ TiO₂ Nanoparticles. *Journal of Global Pharma Technology*. 2019; 11(01): 199-205.
28. Mhammed A, Atyaa AI, Radhy ND, Jasim LS. A new adsorption material based GO/PVP/AAc composite hydrogel characterization, study kinetic and thermodynamic to removal Atenolol drug from wast water. *IOP Conference Series: Materials Science and Engineering*. 2020;928(6):062023.
29. Layth SJ, Aljeboree MA. Removal of Heavy Metals by Using Chitosan/Poly (Acryl Amide-Acrylic Acid) Hydrogels: Characterization and Kinetic Study. *Neuro Quantology*. 2021;19(2): 31-37.
30. Alidadi MD, Mojtaba D, Fateme B, Farideh J, Ahmad H. Enhanced removal of tetracycline using modified sawdust: Optimization, isotherm, kinetics, and regeneration studies *Hosein Process Safety and Environmental Protection*. 2018;117: 51-60.
31. Abbas NK, Layth SJ. Synthesis and Characterization of Poly (CH/AA-co-AM) Composite: Adsorption and Thermodynamic Studies of Benzocaine on from Aqueous Solutions *International Journal of Drug Delivery Technology*. 2019;9(4):558-562.
32. Aljeboree AM. Spectrophotometric and Colorimetric Determination of Pharmaceutical by Oxidative Coupling Reaction: A Review. *Sys Rev Pharm*. 2020;11(4): 609-615.
33. Ho YS, McKay G. Orption of dye from aqueous solution by peat. *S Chem. Eng. J*, 1998;70:115.
34. Waleed KA, Safaa HG, Makarim AM, Layth SJ. Adsorptive Removal of Doxycycline From Aqueous Solution Using Graphene Oxide/Hydrogel Composite. *International Journal of Applied Pharmaceutics*. 2020;12(6): 100-106.
35. Waleed KA, Safaa HG, Nadher DR, Layth SJ. New Approach for Sulfadiazine Toxicity Management using Carboxymethyl Cellulose Grafted Acrylamide Hydrogel. *International Journal of Drug Delivery Technology*. 2020;10(2): 259-264.