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Synthesis And Characterization Of NTADCIP/P(AA-Co-AM) Composite: Adsorption And Removal Studies Of Drug From Aqueous Solutions

Safaa H. Ganduh[1], Aseel M Aljeboree*[2], Makarim A. Mahdi[3], Layth S. Jasim[4]

[1]Department of Chemistry Pharmaceutical, College of Pharmacy, University of Al-Qadisiyah, Iraq
[2]Department of Chemistry, College of Sciences for Girls, University of Babylon, Hilla, Iraq
[3,4]Department of Chemistry, College of Education, University of Al-Qadisiyah, Diwaniya, Iraq
Corresponding Authour: **annenayad@gmail.com**

ABSTRACT

Background: Amoxicillin (AMX) is a β-lactam semi-synthetic antibiotic pertinence of penicillins group . it is partially (about 70–95%) excreted as unchanged chemically via urine and faeces in to the domestic sewage and thence dis charged to the wastewater treatment plants. Method :The effect of several experimental parameters for example primary concentration of Amoxicillin drug (2-40 mg L-1),contact time , adsorbent dose (0.01 -0.1 g) on the adsorption of Amoxicillin drug were investigated. Result : appear that the adsorption of AMX drug find to rise with increase in primary concentration drug, and contact time but decreases of the adsorbent mass It was found that AMX drug adsorption That percentage removal E% (27.5641%) , (52.654%) and (76.7094%) when the temperature increase (17-25 oC) .study three isotherm Freundlech, Langmuir and Temkin find the best isotherm Langmuir the highest correlation coefficients (R^2 = 0.9809) .The drug concentration was measured before and after adsorption through utilizing UV-Visible spectrophotometer at 278 nm .

Keywords: Pharmaceuticals, Antibiotics, Amoxicillin, Adsorption ,Isotherm,Ultrasound .

Correspondence:
Aseel M Aljeboree
Department of Chemistry, College of Sciences for Girls, University of Babylon, Hilla, Iraq

*Corresponding author: Aseel M Aljeboree email-address: annenayad@gmail.com

INTRODUCTION

personal care products (PPCPs) and Pharmaceuticals are widely used in human and animal life and it is acknowledged It has recently been one of the most dangerous pollutants in the aquatic environment [1]. As a data of recurrent utilize, large quantities of P.P.C.Ps have been freed in to water environments [2, 3], and the pollution of surface and ground water has just as a significant issue in late years [3]. Amoxicillin is a semi-synthetic β-lactam antibiotic belonging to the group of penicillin's. The chemical structure of amoxicillin contains of d-4-hydroxy phenyl glycine side chain attached to 6-amino penicillanic acid (6-APA) moiety Because of its broad-spectrum amino penicillin extensively utilized in veterinary medicine of the treatment of bacterial infections caused via Gram-positive Gram-negative organisms. The chemical structures of AMX [4-7]are shown in Fig. 1. AMX . and Some selected physio- chemical properties of AMX are appear in Table 1Adsorption method is in general utilized for removal of drug , dye oil, odor, organic contaminants and colors , chiefly from a phase liquid system , in order to it is considered an active path to remove drug from wastewater, since it is not devastating and simple to stratify [8] . The method cost for removal drug by adsorption lies fundamentally on the cost of the regeneration of adsorbent and adsorbent . NTADCIP/P(AA-co-AM)composite are very good replace for costly mercantile charcoal (activated carbon) utilized in adsorption processes [9, 10] In opinion of this study, we will describe and locate key method parameters: adsorbent mass and primary drug concentration,. The experimental data will be appraised by Langmuir , Freundlich and Timken adsorption isotherms.

Table 1. Physicochemical properties of drug Amoxicillin

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Molecular weight	365.3
Solubility(mg/L)	3431
Log KOW	0.88
Molecular Formula	$C_{16}H_{19}N_3O_5S$
Melting point	195 °C
Acid dissociation constant (pKa)	7.4(amine) ,2.4(carboxyl) and 9.6(phenol)
Flash point	402.2 °C
Boiling point	761 mm Hg at742.3 °C

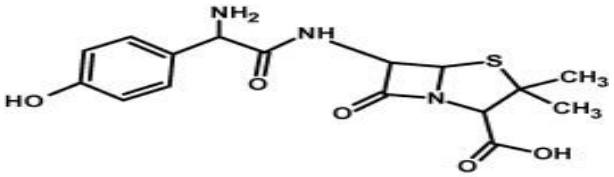
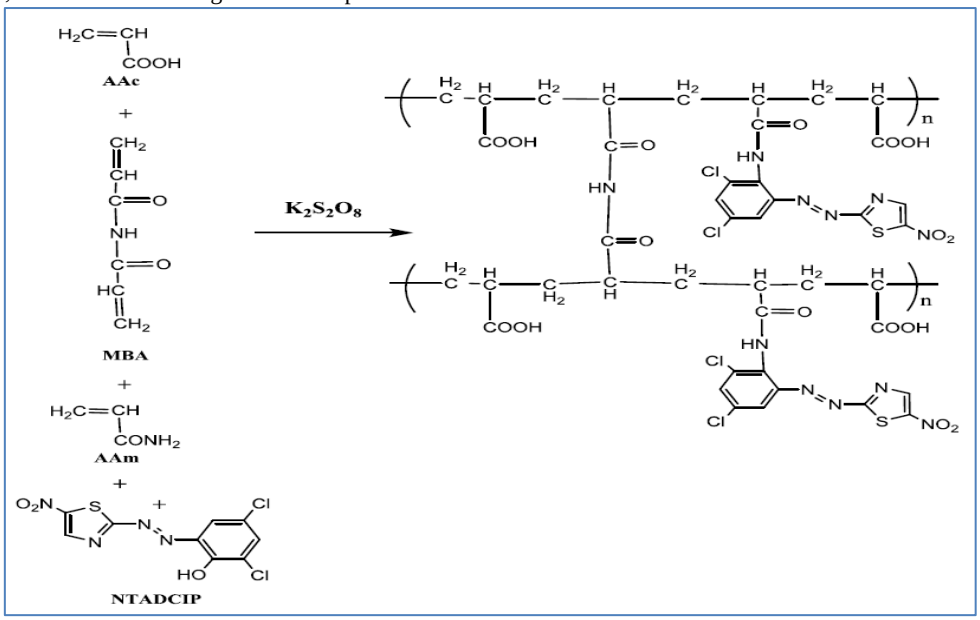


Figure 1: The amoxicillin ((2S,5R,6R)-6-{[(2R)-2-amino-2-(4-hydroxyphenyl)-acetyl]amino}-3,3-dimethyl-7-oxo-thia-1-azabicyclo[3.2.0]heptane-2-carboxylic acid) structure.[11]

PREPARE OF (NTADCLP/P(A.A-CO-AM) COMPOSITES

The compound was prepared by free radical method . The method included dissolving 0.1 g of polymer In 2 ml of absolute ethanol, then add 2 ml of acrylic acid in the presence of nitrogen gas for a period And the record from dissolving 0.02 (MBA) for 60 seconds, then adding the clamping agent solution, such as acrylic amide KPS (grams) in 2 ml of D.W, and then the starting solution of potassium persulfate is added. By taking 0.02 grams and dissolving it in 2 milliliters of distilled water gradually to the reaction mixture and transfer this mixture To a water bath at a temperature of 55 ° C for 30 minutes to complete the polymerization process, then the polymer is extracted And cut into small pieces and then wash with distilled water with constant stirring for 6 hours, and replace the water each Half an hour until the non-reactive materials are disposed of, then it is dried in the oven at 55 ° C and grinded to become .Ready to conduct experiments and the following diagram shows the method of preparation .



Scheme 1: preparation of (NTADClP/P(AA-co-AM) composites

PREPARATION OF CALIBRATION CURVE

Stock solution having several concentrations (2–40 mgL^{-1}) of (100 $mg.L^{-1}$) solution. The following aqueous solution was prepared daily fresh solution . The maximum wavelength absorbance (λ max) was recorded for (drug Amoxicillin AMX) dissolved in aqueous media and find 278nm . Absorbance was measured at the λmax for dye and plotted against the concentration values of AMX as shown in Fig.2 .

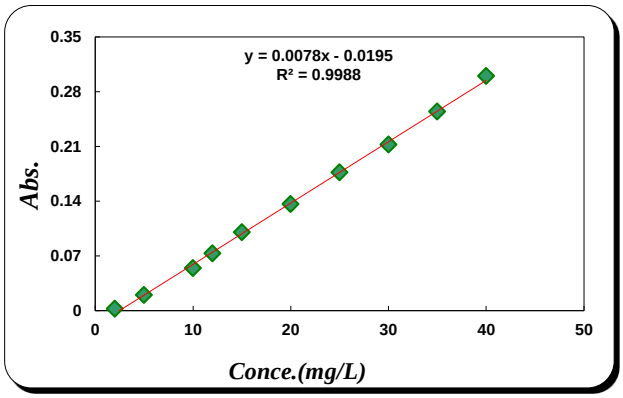


Figure 2: Calibration curve for the Amoxicillin

EFFECT OF SEVERAL FACTORS OF ADSORPTION METHODS OF AMX DRUG ONTO NTADCIP/P(AA-CO-AM)COMPOSITE

EFFECT OF DRUG CONCENTRATION [2-40PPM]

About 0.05 gm from NTADCIP/P(AA-co-AM)composite added to (2,5,10,12,15 ,20, 25,30, 35 and 40 ppm)from drug putted in 50ml conical flask and using ultrasound at 25°C by constant stirrer for 1 hr.

EFFECT OF TEMPERATURE SOLUTION [12-25 ^{0}C]

study affect of the temperature solution via agitating of variants temperatures (12, 17 and 25°C) , quantity dosage 0.05 g of NTADCIP/P(AA-co-AM)composite and 10ml concentration (AMX) drug (12ppm) utilizing ultrasound. Studying the experimentation at normal pH=6.1.and the equilibrium time was reached at one hour .

EFFECT THE MASS OF NTADCIP/P(AA-CO-AM)COMPOSITE [0.01-0.1]

The influence of quantity amount was studied through agitating of several weight (0.01 ,0.025 , 0.05, 0.075 and 0.1)g , in 10 mL of (AMX) drug concentration (12ppm) using ultrasound at 25 °C. Studying the experimentation at normal pH=6.1.and the equilibrium time was reached at one hour .

RESULTS AND DISCUSSION

ANALYSIS OF THE SEM

The surface of adsorbent was too characterized via (S.E.M) before and after the adsorption experements utilizing drug AMX. images S.E.M onto NTADCIP/P(AA-co-AM)composite (Fig. 3(a)) appear the color bright dark onto surface. After AMX drug adsorption onto NTADCIP/P(AA-co-AM)composite as the surface was turned to light color (Fig.3(b)). This might be due to AMX drug adsorption on the surface of the NTADCIP/P(AA-co-AM)composite[12] .

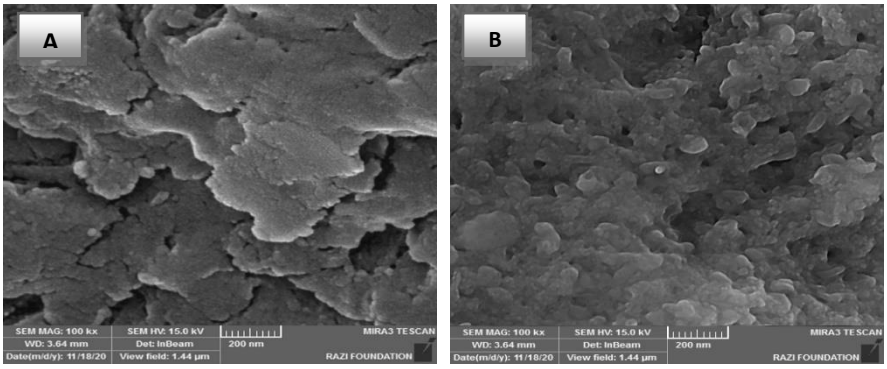


Fig. 3: SEM image of NTADCIP/P(AA-co-AM)composite. (A) before and (B) after adsorption of AMX drug

FT-IR CHARACTERIZATION FOR ADSORBENT/ADSORBATE

The NTADCIP/P(A.A-co-AM)composite was characterized via F.T-IR spectroscopy. spectra F.T-IR was collected in the mid -IR range from 4000 to 400 cm^{-1} with a resolution of 1 cm^{-1}. The spectra F.T-IR of NTADCIP/P(AA-co-AM)composite before and after AMX drug adsorption are appear in Fig. 4 The F.T-IR pattern appear reduced in an intensity of bands next the adsorption, too there is a difference real among NTADCIP/P(AA-co-AM)composite before and after interaction by AMX drug that have been suggested a phy-sorption phenomenon happens as a data of attractive forces among the NTADCIP/P(AA-co-AM)composite surface and AMX drug under investigation [13, 14].

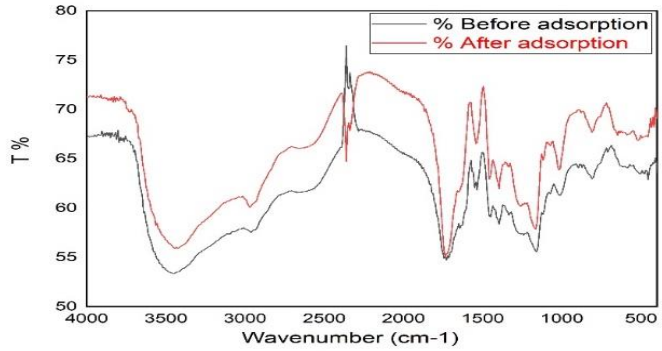


Fig. 4: FT-IR spectra of NTADCIP/P(AA-co-AM)composite (a) before, and (b) after adsorption of AMX drug

ATOMIC FORCE MICROSCOPY (AFM)

AFM was used to determine the surface topography of the hydrogel, fig 5 shows a three-dimensional image of surface composite NTADCIP and NTADCIP/P(AA-co-AM) , Table 2 shows that the mean roughness (Ra) and the root mean square roughness (Rq) of the surface The composite is greater than the coarse coefficients of the gel NTADCIP .What is confirmed that the added ketchup may be added)It clearly affected the topography of the surface as the reagents' particles spread and homogeneous along the surface of the hyaline. Which caused great roughness of the surface. [15]

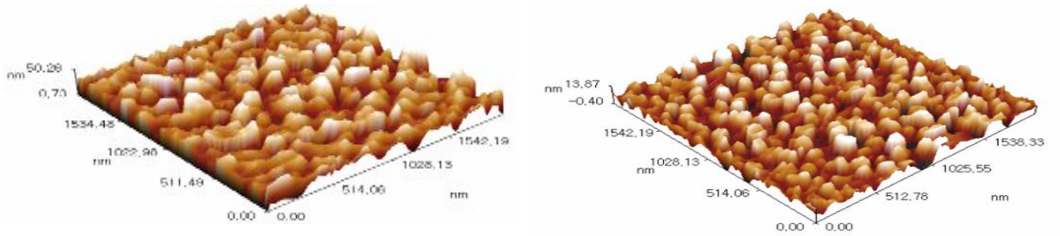


Figure 5: 3D image of AFM NTADCIP (A), and NTADCIP/P(A.A-co-AM) composite(B)

Table (2): the statistical roughness coefficients of the samples prepare

Amplitude Factors	NTADCIP	NTADCIP/P(AA-co-AM)
Mean roughness (Ra)	**2.32**	**7.99**
Root mean square roughness (Rq)	**11.10**	**9.9**
surface Skewness (Rsk)	**-0.16**	**-0.0492**
surface Kurtosis (Rku)	**1.98**	**2.68**

EFFECT WEIGHT OF NTADCIP/P(A.A-CO-AM)COMPOSITE

The results for the AMX drug uptake via different amounts (0.01–0.1g) of the NTADCIP/ P(A.A-co-AM)composite in Fig. 6. The equilibrium of AMX drug uptake removal percentage rise as the quantity of the adsorbent rise because of the increased surface area of NTADCIP/P(AA-co-AM)composite that gives more active sites for AMX drug adsorption. In the present experiment, the 0.05 grams of NTADCIP/P(A.A-co-AM)composite chosen as the best adsorbent dosage because, at this quantity, the equilibrium (saturation) achieved. 0.05 g of NTADCIP/P(AA-co-AM)composite selected for subsequent experiments [9, 16, 17].

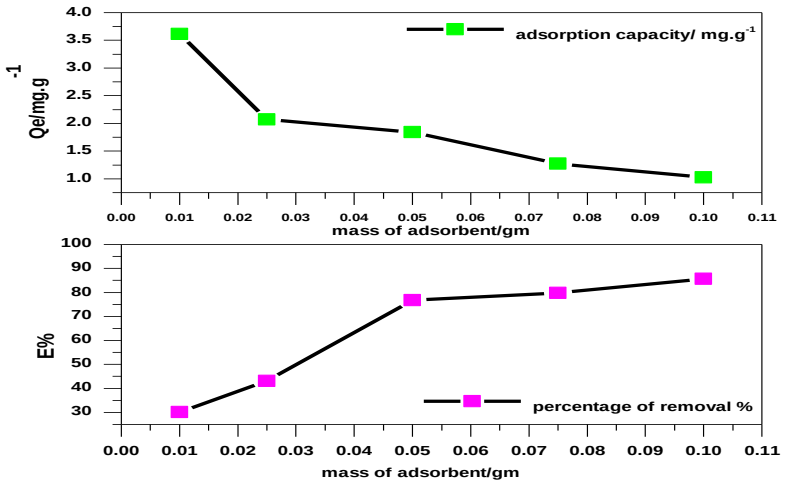


Figure 6: The effect the mass of NTADCIP/P(AA-co-AM)composite concentration on AMX drug. Exp. conditions: AMX conc. 12 mg.L^{-1} pH 6, contact time 1 h, Temp 25.

EFFECT OF PRIMARY CONCENTRATION OF AMX DRUG

The quantity of adsorption of drug percentage removal is highly reliant on the primary concentration drug. The effect of primary conc. drug depends on the immediate relation among the drug concentration and the available sites on surface of the adsorbent [18]. The effect of primary concentration of drug on the removal of drug by NTADCIP/P(AA-co-AM)composite is Figure (7) presents the removal efficiency versus drug concentration. The drug removal percentage decreases with drug concentration increase due to reduction in adsorption for the lack of available active sites. The adsorption capacity ((qe) mg/g) is proportional with initial drug concentration as the drug uptake resistance decreases with the rise in concentration drug. The adsorption rate too rises by the increase in the drug initial concentration due to driving force increase [19-21].

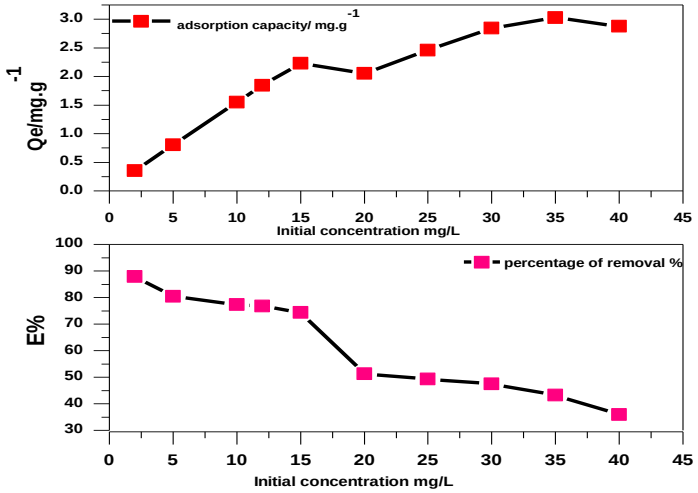


Figure 7: Effect of primary conc. on the removal percent and amount of adsorbed AMX drug onto (NTADCIP/P(AA-co-AM)composite Temp. = 25°C, and adsorbent mass 0.05g).

EFFECT OF SOLUTION TEMPERATURE

study affect of the temperature solution via agitating of variants (12, 17 and 25°C) temperatures weight of adsorbent 0.05 g of NTADCIP/P(AA-co-AM)composite and 10 mL (AMX) drug concentration (12 ppm) utilizing Ultrasound at 25°C as appear in fig. 8 .studying the experimentation at normal pH=6.1. reach contact time 1hr at a string speed as constant. Temperature rise is joined via adsorption efficiency raise of the NTADCIP/P(A.A-co-AM)composite (the higher the temperature give the best adsorption). Information on strength bond , spontaneity and randomity can be acquired via treatment of the thermodynamic of adsorption method. It is significant to note that an fundamental prerequisite of adsorption studies at several temp. solution is taking in to consideration the changes density of the solution and solubility of substance by temp. [22-24].

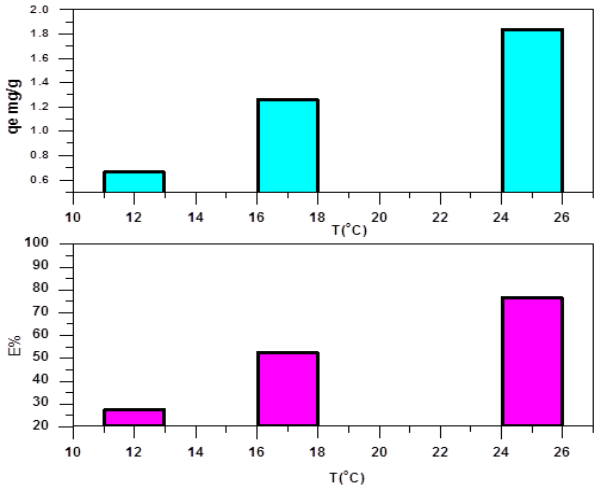


Figure 8: Influence of the temperature solution on the E% , quantity of adsorbed AMX drug on to (NTADCIP/P(AA-Co-AM)composite) contact time 60min. , Temp. = 25°C, and adsorbent quantity 0.05g)

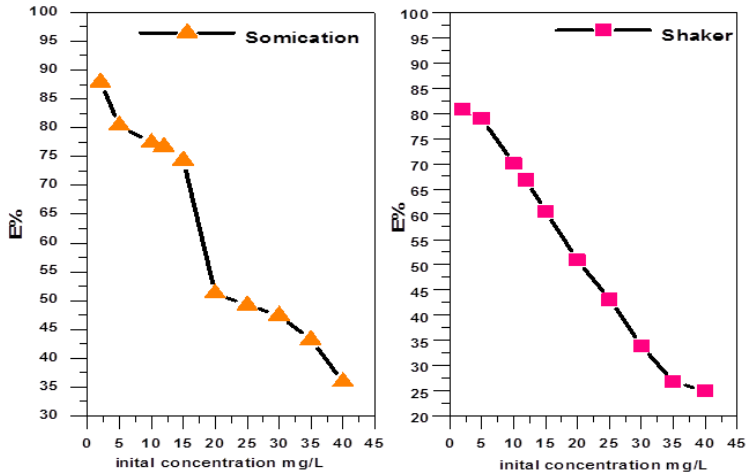


Figure (9): Comparative between Sonication and Shaker by use (NTADCIP/P(AA-co-AM)composite)

A COMPARATIVE ADSORPTION BETWEEN SONICATION AND WATER BATH SHAKER TO REMOVAL PHARMACEUTICALS POLLUTANT .

A comparative between sonication and water bath Shaker by using (NTADCIP/ P(AA-Co-AM)composite) show in fig. (9). The good results of the percentage of removal (E%) of sonication 87.8846% to 35,529% and Shaker 80.833 % to 24.967% for drug (Amoxicillin AMX) [24-26].

INFLUENCE OF CONTACT TIME AND ADSORPTION KINETICS

The required time to reach the equilibrium case of AMX drug adsorption onto (NTADCIP/P(AA-Co-AM)composite) was calculated as a function of the time equilibrium for a constant drug concentration at 25 °C at several time intervals (1-220min), a constant mass of (NTADCIP/P(AA-co-AM)composite)0.05g and pH 6.0 the data are appear in Fig. 10, the adsorption efficiency of AMX drug was augmented as the time elongated until reaching maximum value (saturation state); after that,

the adsorption efficiency decreases through rising time due to a desorption method. The models of kinetics of the adsorption method, that define the experimental data, elected for adsorption of AMX drug on to (NTADCIP/P(AA-co-AM)composite) presented in Fig. 10. Experimental result study was done utilizing the pseudo-second-order and the pseudo-first model appear in Table 3 data display adsorption of AMX drug via the (NTADCIP/P(AA-co-AM)composite) is achievable method because the value (R^2) is great of the pseudo-First order model compared to the pseudo second –order [9, 27, 28].

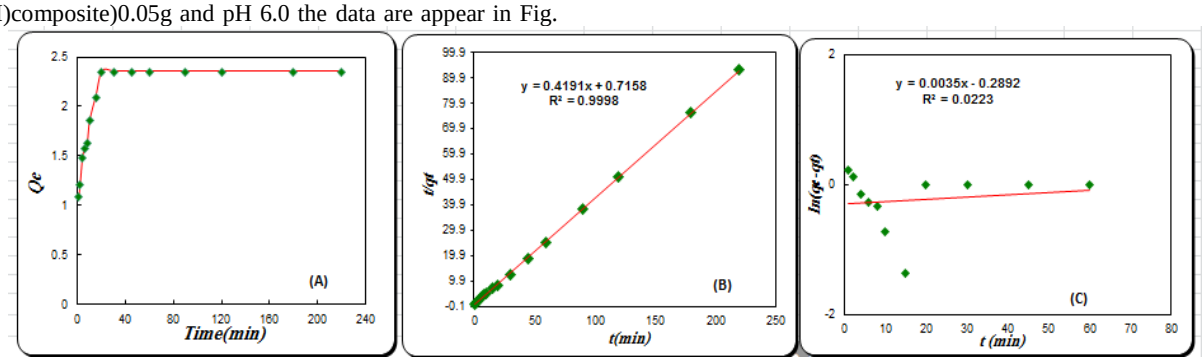


Figure 10: Influence of reaction time (A), pseudo first order (B), and the pseudo second-order (C) on AMX drug adsorption

Table 3: Adsorption kinetics factors the adsorption of AMX drug

First-order					
Slope 0.4191	**Intercept** 0.7158	**k1 (min)$^{-1}$** -0.4191	**qe (mg/g)** 2.045823	**R^2** 0.9998	
Second-order					
Slope 0.0035	**Intercept** 0.2892	**qe (min)$^{-1}$** 285.7143	**k2 (g.mg.min^{-1})** -4.2E-05	**H** -3.45781	**R^2** 0.0223

ADSORPTION ISOTHERMS

Numerous equations isotherm are existing of investigating sorption equilibrium factors, the most public being is the Freundlech, Langmuir and Temkin model. The isotherm of Langmuir isotherm (Fig. 11A) built on the theory that there is a fixed quantity of active sites, which regularly dispersed over the surface of adsorbent, these sites have identical desirability of adsorption of a mono molecular layer and no interaction among adsorbed molecules[9, 23, 28]The Freundlech model (Fig. 11B) applicable of heterogeneous surface adsorption. This isotherm supposes a positive relationship among adsorbate conc. adsorbent amounts on the surface. Also, the energy sorption proportionally declines at the end of the sorption centers of the adsorbent. Timken isotherm (Fig. 11C) comprises an element that taking in to the account of interactions of adsorbent-adsorbate. By disregarded the meager and important value of conc., the isotherm supposes the adsorption heat of wholly molecules in the layer should linearly reduction more willingly than logarithmic by coverage. Calculation of correlation coefficients done via fitting the experimental equilibrium data of the NTADCIP/P(AA-co-AM)composite method

utilizing Langmuir, Freundlech, isotherm. Figure 6 appear the highest correlation coefficients (R^2 = 0.9809) related to the Langmuir isotherm; these findings. The values of Langmuir constants as well as the correlation coefficient are presented in Table (4)

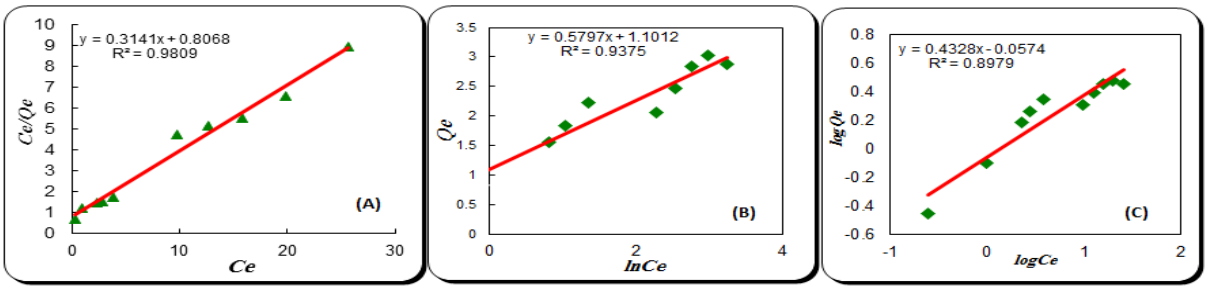


Figure 11: Langmuir (A), Freundlech (B), and Temkin (C)isotherms of AMX drug adsorption on NTADCIP/P(AA co-AM)composite .

Table 4:The correlation coefficients and constants of model of Langmuir, Freundlich and Timken of adsorption AMX drug adsorbed on to surface of NTADCIP/P(AA-co AM)composite at 25 °C.

Langmuir equation			Freundlich equation			Timken equation		
K_L	qm	R^2	K_F	N	R^2	K_T	b	R^2
0.38844	3.18369	0.9809	12.62409	1.72503	0.9375	0.87579	0.4328	0.8979

These application could using as a model for new synthesized organic compounds beside the applications of adsorption we can use in photo catalytic degradation [29-32]

CONCLUSION

The adsorption of the drug AMX on the NTADCIP/P(A.A-co-AM)composite was studied in a different parameters like premier drug AMX concentration, solution of temperature, and mass adsorbent. The data appeared that adsorption AMX drug raise with increased in premier concentrations AMX drug, temperature whereas it decreased with excess in weight of adsorbent. The data find of the investigational refer to that the adsorption time equilibrium at 1hr and the adsorption method is find to fit pseudo first order kinetics.

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