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ADSORPTION OF CIPROFLOXACIN FROM AQUEOUS SOLUTION ONTO FE$_3$O$_4$/ GRAPHENE OXIDE NANOCOMPOSITE

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ABSTRACT: In this research, ciprofloxacin adsorption (CIP) onto graphene oxide synthesized with Fe$_3$O$_4$ (Fe$_3$O$_4$-GO) was applied *via* various adsorbent dose, contact time, temperature and initial CIP concentration. The results showed that the percentage removal of CIP decreases from 98.1 to %77.4, as the CIP concentration increases from 25 to 200 mg/L. Also, the amount of CIP adsorbed per unit mass of adsorbent increased from 49.25 to 309.6 mg/g with increasing in CIP concentration from 25 to 200 mg/L. The results showed that the adsorption was accurately represented by the pseudo-second-order model. Under different temperature, the CIP maximum adsorption on Fe$_3$O$_4$-GO calculated by pseudo-second-order kinetic model and were 197 mg/g (273 K), 191.6 mg/g (288 K), 182.2 mg/g (303 K) and 172.6 mg/g (318 K). Also, the results showed the existence of three steps during the adsorption process. The first stage was the transport of CIP molecule from the adsorbents external surface to the pores of the adsorbent internal structure. The second stage is the transport of CIP molecule from the adsorbent external surface to the pores of the adsorbent internal structure. The third stage is the adsorption of CIP molecule on the interior surface of the adsorbent. The results suggest that Fe$_3$O$_4$-GO could be employed as an effective material for the removal of CIP from aqueous solutions.

INTRODUCTION: Contamination of surface and even groundwater is becoming an increasingly serious problem worldwide because of the growing population and widespread use of chemicals, including pharmaceuticals [1-3]. The pharmaceuticals constitute an important and rapidly emerging class of pollutants worldwide [4, 5].

Pharmaceutical compounds have been recognized as a hazardous class of organic pollutants due to their extensive use and long term effects towards aquatic environment [6, 7]. Antibiotics constitute a group of pharmaceuticals that are widely used to treat several infectious diseases in both human and animals [8]. About 30-90% of the given antibiotic dose can remain undegradable in the human or animal body and is largely excreted as an active compound [9, 10].

Most antibiotics are incompletely metabolized; thus, their residues and degradation products are excreted and can enter water environments in various ways [11, 12]. Exposure to the residues and

metabolites of antibiotics may cause a variety of adverse effects in the environment, such as antibiotics resistance of microorganisms and chronic and acute toxicity for organisms [13, 14]. Among the antibiotics used, Ciprofloxacin (CIP) antibiotics are belonging to the Fluoroquinolones (FQs) family, are hydrolytically stable, difficult to degrade in water and widely detected in wastewater, soil and sediments [15, 16]. Environmental risks associated with SIP including antibiotic resistance of bacteria have been reported [17]. Pharmaceuticals are emerging contaminants and their effective removal from water resources is very important [18]. The efficiency of antibiotics removal from water in a traditional system is low because of their low biodegradability, high solubility in water, and complex molecular structures [19, 20, 21].

Therefore, various methods such as ozonation, oxidation, reduction, gamma-ray irradiation, photolysis, and adsorption have been suggested as potential and competitive methods to remove antibiotics from water [22, 23, 24]. Of these processes, it has been demonstrated that adsorption is a simple, effective, and economical method to remove pollutants from waters [25, 26]. Adsorption is the widely used method for removal of a broad range of antibiotics pollutants due to its simple design, easy operation, and relatively simple regeneration [27]. Currently, nanomaterials have been used by researchers to advance the removal and treatment of industrial wastewaters [28].
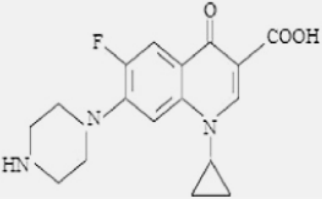
These nanomaterials are divided into two main categories such as nanometal and none-nano metal materials such as Fe_3O_4-graphene oxide (Fe_3O_4-GO) and graphene oxide or nano-carbon families, which are powerful and remarkable nanomaterials that could be used as a strong absorbent via their great and enormous specific surface area and minor internal diffusion resistance [29].

In present work, Fe_3O_4-GO was prepared to create a strong adsorbent with low prices and simple synthesis procedure to adsorb and separate the ciprofloxacin antibiotics from aqueous solution. The adsorption manner of CIP onto Fe_3O_4-GO nanocomposite was explored and the main adsorption mechanisms were surveyed in different initial CIP concentration, contact time, adsorbent dose and temperatures.

MATERIALS AND METHODS:
Materials: All chemical materials used were of analytical reagent grade and used without further purification. Analytical grade CIP was obtained from Sigma Aldrich Co., and used as received; its characteristics are given in **Table 1**. Graphene oxide nanoparticles were manufactured by improved Hummers method [29].

TABLE 1: CHARACTERISTICS AND STRUCTURE OF CIPROFLOXACIN

Weight	331.35 g/mol
Formula	$C_{17}H_{18}FN_3O_3$
Structure	

Solubility at water	150–6190 mg/L
λ_{max}	270 nm

Preparation of Graphene Oxide: Graphene oxide was prepared from graphite powder using a modified Hummer's method. Firstly, a 9:1 mixture of concentrated H_2SO_4: H_3PO_4 was added to a mixture of graphite flax (3 g, 1 wt equivalent) and $KMnO_4$ (18 g, 6 wt), producing a slight exothermal to 35-40 °C. Then the reaction was washed and the residues were centrifuged (4000 rpm for 4 h) and the supernatant decanted away. The remaining material after the multiple-wash process was mixed with 200 mL of diethyl ether. The obtained solid on the filter was vacuum-dried overnight at room temperature.

Preparation of Fe_3O_4-GO Nanocomposite: Firstly 15 mL of graphene oxide (2.72 mg/mL) was dispersed into 40 mL ethanol with stirring. Then 0.9522 g of $FeCl_3 \cdot 6H_2O$ (3.5228 mmol) and 1.0520 g of $FeSO_4 \cdot 7H_2O$ (3.7842 mmol) were dissolved in 10 mL of distilled water under sonication and solution was injected dropwise into the graphene oxide suspension and stirred for 0.5 h. The resulting mixture was heated to 68 °C before the ammonia solution was added to adjust the pH to 10. The mixture was stirred at 68 °C for 2 h and cooled to room temperature. The Fe_3O_4-GO composite was separated from the mixture using a permanent magnet and rinsed two times with ethanol and distilled water, respectively before being dried at 65 °C for 12 h.

Adsorption Study: Batch adsorption experiments were carried out using a shaker. The effects of contact time, CIP concentration, adsorbent dose and temperature on adsorption were investigated. For each experimental run, 0.5 g adsorbent and 100 mL of CIP solution of known concentration were transferred into a flask and agitated by shaker at a constant speed of 180 rpm with a required adsorption time. At predetermined time intervals, the solutions were centrifuged at 3600 rpm for 10 min. The adsorption kinetics was determined by analyzing adsorption capacity from the aqueous solution at different time intervals. Kinetics solution of different concentrations in the range of 273-318 K was agitated until the equilibrium was achieved.

Concentrations of CIP were determined by finding out the absorbance characteristic wavelength using UV-spectrophotometer. A standard solution of the CIP was taken and the absorbance was determined at different wavelengths to obtain a plot of absorbance versus wavelength. The wavelength corresponding to maximum absorbance (λ_{max}) was determined from this plot. The $\lambda_{max}$ for CIP was found to be 270 nm. Calibration curves were plotted between absorbance and concentration of the CIP solution. The adsorbed amount (qe) of CIP was calculated by the following equation: [30]

$$qe = ((C_o - C_e) \times V) / M$$

Where, C_0 and C_e are the initial and equilibrium concentrations of CIP (mg/L), m is the mass of sorbent (g), and V is the volume of solution (L).
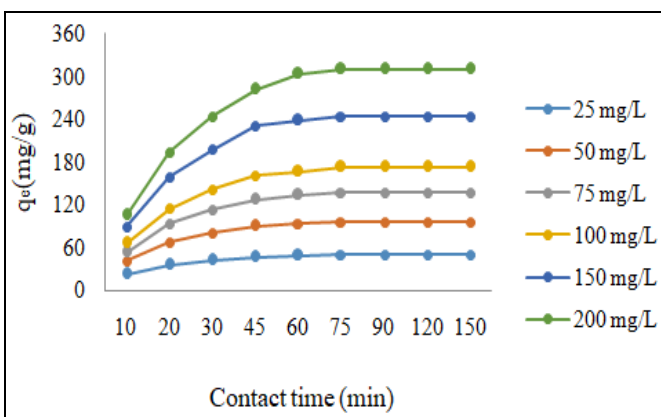
RESULTS AND DISCUSSION:
Effect of Initial CIP Concentration and Contact Time: The initial concentration of CIP in solution provides an important driving force in overcoming mass transfer resistance between the aqueous and the solid phases [24]. Equilibrium adsorption studies have been performed to determine the capacity of the adsorbent, and the equilibrium is established when the concentration of adsorbate in the bulk solution is in dynamic balance with that on the surface [30]. The effect of initial CIP concentration is shown in **Fig. 1**. For 0.6 g/L of adsorbent, the percentage removal of CIP decreases from 98.1 to 77.4% as the CIP concentration increases from 25 to 200 mg/L. But, the amount of CIP adsorbed per unit mass of adsorbent increased with increasing in CIP concentration **Fig 2**.
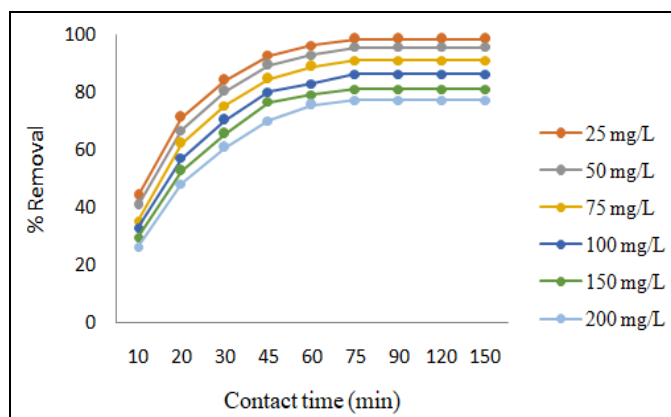
FIG. 1: EFFECT OF INITIAL CIP CONCENTRATION AND CONTACT TIME
(Dose= 0.6 g/L, pH = 7, and Temp= 25 ℃)

FIG. 2: EFFECT OF INITIAL CIP CONCENTRATION AND CONTACT TIME ON ADSORPTION CAPACITY
(Dose= 0.6 g/L, pH = 7, and Temp= 25 ℃)

This may be due to all CIP there in solution could intermingle with the binding sites at lower concentration and thus the percentage adsorption was higher than those at higher initial CIP concentration [31, 32]. At higher concentrations, lower adsorption yield is due to the saturation of adsorption sites [33]. At low concentrations, sorption sites took up the available CIP molecule more rapidly while at higher concentrations the rate of diffusion became slow [33, 34]. The effect of contact time on the removal of CIP onto Fe_3O_4-GO for different concentration of CIP is shown in **Fig. 2**. The results revealed that the rate of CIP removal is higher at the beginning, which was due to the large available surface area [35].

As the surface adsorption sites become exhausted, the uptake rate is controlled by the rate at which the adsorbate is transported from the exterior to interior sites of the adsorbent. Almost 70% of the total CIP

molecule was adsorbed in the initial 10 min period and remained almost unchanged after 75 min, indicating that the adsorption process has reached equilibrium.

Effect of Adsorbent Dosage: The effect of changing the adsorbent dosage on the removal of CIP was studied by varying the adsorbent dosage from 0.1 to 0.9 g/L while keeping the other experimental conditions as constant **Fig. 3**.

An increase in percentage removal of CIP with increasing adsorbent dosage was observed whereas the adsorption capacity for the adsorbents decreased. This is due to the larger surface area and availability of more surface functional groups at higher concentration of adsorbent [36, 37]. The decrease in adsorption capacity can be explained with the reduction in effective surface area of the adsorbent [38].
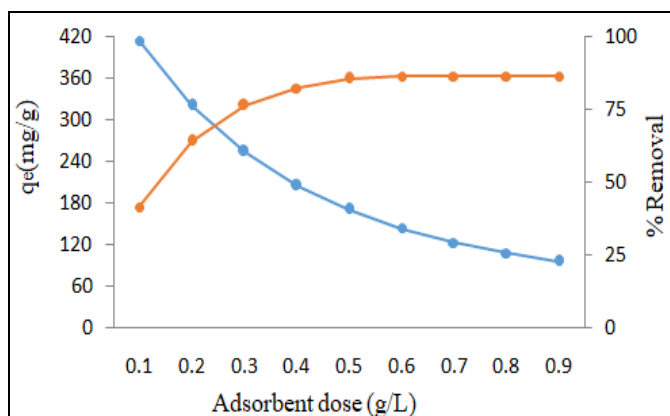
FIG. 3: EFFECT OF ADSORBENT DOSE (C_0= 100 mg/L, pH = 7, Contact time 75 min and Temp= 25 °C)

Adsorption Kinetics: Adsorption kinetics experiments were to study the impact of contact time and assess the dynamic characteristics. The amount of Fe_3O_4-GO was added to 200 mL CIP solution, adsorption amount at time t is calculated by the following formula: [39]

$$q_t= (C_0-C_t)V/ m$$

Where, q_t is the adsorption capacity of Fe_3O_4-GO at time t (mg/g), V is the volume of the solution (L), C0 is the initial concentration of CIP (mg/L) Ct is the concentration of the sample at time t (mg/L). Four kinetic equations: pseudo-first-order kinetic, pseudo-second-order kinetic, film diffusion model and intraparticle diffusion model were examined to fit the experimental data. They are listed below:

Pseudo-first order model: [40]

$$Ln (q_t-q_e) = Ln (q_e) − k_1t$$

Pseudo-second order model: [41]

$$t/qt = 1/(k_2.q_2)+t/q_e$$

Intra-particle diffusion model: [42]

$$q_t= K_3 \ t^{0.5} + C$$

Film diffusion model: [43]

$$Ln (1−q_t/q_e) = −K_4 \ t + A$$

Where, q_t is the adsorption capacity of Fe_3O_4-GO at time t (mg/g), q_e is the equilibrium adsorption capacity of Fe^3O_4-GO (mg/g), K_1 is the adsorption rate constant (h^{-1}), K_2 is the rate constant of second-order equation (g/mg. h), K_3 is the intraparticle diffusion rate constant (mg/gmin1/2), and C is a constant that gives an idea about the thickness of the boundary layer (mg/g). K4 (h^{-1}) and A are liquid film diffusion constants. Whether the dynamic model was suitable for describing the adsorption of CIP by Fe_3O_4-GO, normalized standard deviation $\Delta q(\%)$ was used for further authentication. $\Delta q (\%)$, given as: [44, 45]

$$\Delta q = 100 \sqrt{\frac{\sum [(q_{exp} - q_{cal})/ q_{exp}]^2}{N-1}}$$

Where N is the number of data, q_{exp} is the experimental adsorption capacity (mg/g) q_{cal} is the calculated adsorption capacity (mg/g).

The analysis results obtained by linear regression were summarized in **Table 2**. The Lower R^2 and large Δq (%) showed that the pseudo-first-order kinetics model was not suitable for adsorption of CIP. The pseudo-second-order kinetic model can be fitted to the data, which the linear correlation coefficient is higher than 0.995 at all temperature **Fig. 4** and calculating the adsorption capacity and adsorption experiments relatively consistent.

Therefore the pseudo-second-order kinetic model was suitable to describe the adsorption process of CIP on Fe_3O_4-GO. The maximum adsorption quantity of CIP calculated by pseudo-second-order kinetic model and was 197 mg/g (273 K), 191.6 mg/g (288 K), 182.2 mg/g (303 K) and 172.6 mg/g (318 K). According to the secondary rate constant

(K_2) and initial adsorption rate (h), the adsorption rate slowed down with the temperature increasing. It indicated that the high temperature is not conducive to the adsorption. The adsorption process is usually controlled by either or both of them. In order to determine which one is the rate-controlling step, the intraparticle diffusion model and film diffusion model were used to fit to the results of adsorption dates. If the linear plot of qt versus t½ is a straight line through the origin, indicating that the intraparticle diffusion is the only rate-controlling step. However, from the multi-linear curve obtained in the study, indicated that two or more steps occurred in the adsorption process [44].

TABLE 2: KINETIC PARAMETERS FOR THE ADSORPTION OF CIP ONTO Fe₃O₄-GO

| T (K)	$q_{e\ exp}$ (mg/g)	Pseudo-first order				Pseudo-second order			
		K_1	$q_{e\ (cal)}$	R^2	$\Delta q(\%)$	$q_{e\ (cal)}$	R^2	K_2	$\Delta q(\%)$
273	194.5	0.049	145.2	0.795	9.325	197	0.997	0.067	1.452
288	186.4	0.032	137.9	0.814	10.84	191.6	0.999	0.054	2.641
303	179.7	0.017	129.8	0.808	12.46	182.2	0.998	0.046	1.563
318	167.8	0.012	118.4	0.829	9.896	172.6	0.997	0.022	1.072
		Intra-particle diffusion model				Liquid film diffusion			
		K_3	C	R^2	$\Delta q(\%)$	K_4	A	R^2	$\Delta q(\%)$
	273	14.12	69.45	0.941	4.45	0.244	0.096	0.924	3.45
	288	16.44	62.75	0.952	3.89	0.228	0.117	0.913	2.24
	303	17.25	58.94	0.938	5.11	0.212	0.145	0.889	2.88
	318	19.16	53.12	0.942	3.27	0.195	0.168	0.927	1.93
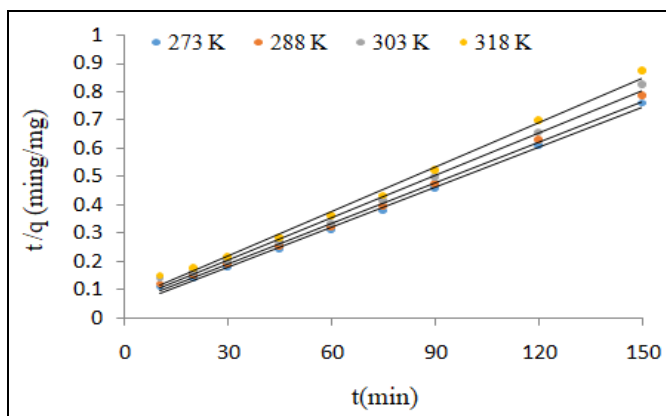
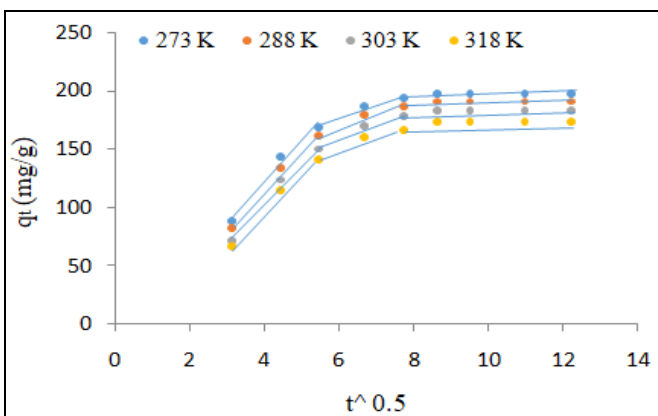**FIG. 4: PSEUDO-SECOND ORDER KINETICS MODELS FOR THE ADSORPTION OF CIP ADSORPTION**

FIG. 5: INTRAPARTICLE DIFFUSION KINETICS MODELS FOR THE ADSORPTION OF CIP ADSORPTION

As seen in **Fig. 5**, the initial slope was big, which mean the adsorption rate was fast. That might be the surface adsorption or transient absorption. The second part was gradually adsorbed phase, which the liquid film diffusion was the main controlling step. The third step is the equilibrium period, as the concentration of adsorbate in solution was very low; the intra-particle diffusion was not the controlling step anymore.

The values of K_3 and C in intra-particle diffusion were obtained by the second curve. C was used to describe the thickness of the boundary layer. The increasing value of C showed that the thickness of the boundary layer increased. As seen from the result, the thickness of the boundary layer increased with the increasing temperature. In liquid film diffusion model, with the increasing of temperature, the value of K_4 decreased. That indicated that the high temperature was adverse to liquid film diffusion. As temperatures went up, liquid film diffusion would become the limit factor.

CONCLUSION: Environmental pollution caused by industrial effluents is an important issue. Biosorption of CIP from aqueous solution using Fe₃O₄-GO was investigated in a batch mode. The results showed that increasing the dose of the adsorbent and the contact time increased the CIP removal. So with the increase in the absorbent dosage from 0.1 to 0.9 g/L, the percentage of removal increases from 41.4 to 86.8% at initial CIP

concentration 100 mg/L, whereas adsorption capacity decreases from 414 to 86.3 mg/g.

On the other hand, kinetic parameters have been investigated with pseudo-first-order, pseudo-second-order, intra-particle diffusion model and liquid film diffusion. The result of experimental data indicates that the pseudo second-order equation fit better than the other kinetics.

CONFLICTS OF INTEREST: Authors have declared that no competing interests exist.

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