RESEARCH ARTICLE

Removal of Pharmaceutical by Adsorption Process; Study Effect of Various Parameters and Determination of Pharmaceutical by Oxidative Coupling Reaction

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ABSTRACT

Pharmaceutical pollutants are among the most dangerous water pollutants, so a simple and expensive method was used to remove these pollutants, which is the adsorption method that relies on inexpensive and environmentally friendly surfaces. We studied different parameters like the effect of the weight of AC effect of concentration of the drug and adsorption isotherms. It was found that they are fitting Frendlich model, where the residual concentration after the adsorption process was measured using the coupling oxidation method to obtain the colored product with a wavelength 550 nm, where several factors were studied, including the influence of the volume of the reagent and the influence of the volume of the acid.The linearity range for Amoxlline was (1–10 mg/L) while the (LoD = 0.004) and (LoQ = 0.03 µg/mL).

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INTRODUCTION

Pharmaceuticals are among the most dangerous pollutants and are considered organic chemicals and a source of great concern due to their low biodegradability and high stability.[1-5] Pharmaceutical preparations comprise a large group of medical, veterinary, and human compounds and can be used worldwide. The flow of pharmaceutical preparations is in drinking water and groundwater.[6-11] There are several ways to treat these contaminants, such as photo-oxidation, biodegradation, ozone, and adsorption. Where adsorption is considered the most uncomplicated, inexpensive, and utmost versatile method for removing these contaminants, whereby highly efficient surfaces are used to remove pollutants, which are environmentally friendly and inexpensive, such as activated carbon, nanotube carbon, hydrogel, clay, graphite oxide, and others.[12-14] The research aims to remove pharmaceutical pollutants using the oxidative coupling method, where the surface was very efficient and environmentally friendly. The effect of weight, concentration, and adsorption isotherms were studied, and several techniques were used, such as fourier-transform infrared spectroscopy (FTIR), field emission scanning electron microscope (FESEM), energy dispersive x-ray analysis (EDX).

EXPERIMENTAL PART

Adsorption Studies

Study the optimum concentration, contact time, mass of AC, initial concentration of drug to give best removal percentage%, using 0.05 gm of AC in different conical flasks, to each flasks 100 mL from freshly prepared drug solution of initial concentration (100) mg/ mL. At 6000 rpm for 10 min. used the following equation to estimation adsorption capacity at equilibrium:

$$qe = \frac{Vsol.(Co - Ce)}{m} \quad (1)$$

The adsorption capacity and (E%) removal of the drug on the adsorbents were estimation in equations :

$$\%E = \frac{(Co - Ce)}{Co} \times 100 \quad (2)$$

RESULTS AND DISCUSSION

Effect of Acid Volume

Study the influence of several volumes of HCl on the absorbance product, utilizing HCl 1N by series several volumes (0.1 to 1 mL).found when using 0.6 mL from acid give the best absorbance and higher sensitivity while when the volume of acid increase the absorbance decrease and give low sensitivity.[2,11] The results are shown in Figure 1.

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Effect of Volume Sodium Nitrite

We studied the effect of several volumes of Sodium Nitrite from 0.1N at 0.2–0.8 mL and found that the good sensitivity and best absorbance reach at 0.5 mL whereas the top time (3 minutes) that enough the colored intenseness have been for diazotization complete of AMX.[15,16] The data is mentioned in Figure 2.

Effect of Reagent (CZP)

Study effect of several volumes of the CZP reagent (0.2–0.8 mL) to give higher stability of the color and best absorbance, sensitivity, and the optimum volume found 0.5 mL but when increase the volume reagent above 0.5 mL decrease the stability of the color and absorbance.[17,18] The data is mentioned in Figure 3.

Effect of NH3 Volume

Study the different volume of base NH3 (0.1–1.0 mL) found when the volume of the NH3 increase the stability of the color increase and the optimum volume 0.5 mL give best absorbance and sensitivity while when the volume of the NH_3 increase the absorbance, not effect or very low decrease,[19] the data as appears in Figure 4

Characterization

FT-IR

FTIR spectrum was Show for AC that indicated several surface functional groups. The broad band at around 3450 cm^{-1} is typically attributed to aquatic OH groups stretching. Figure 5 shows there is no real shift in our peaks absorption. This is indicated as physical adsorption.[20]

FESEM

The images (FESEM) show a sample surface via raster scanning over it by a great-energy beam of electrons. The electrons interact with the atoms comprising the sample to produce signals that have information about surface morphology.[21] Figure 6 shows a FESEM of the activated

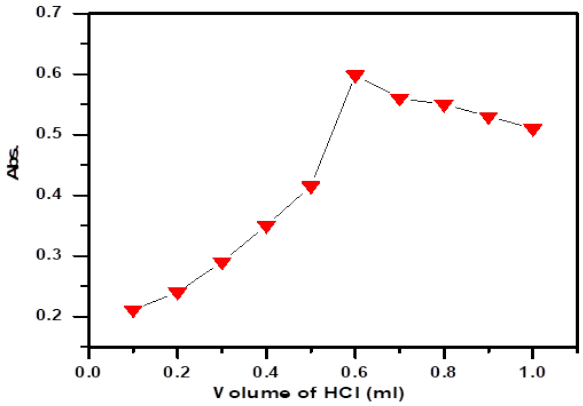

Figure 1: Influence of HCl Volume

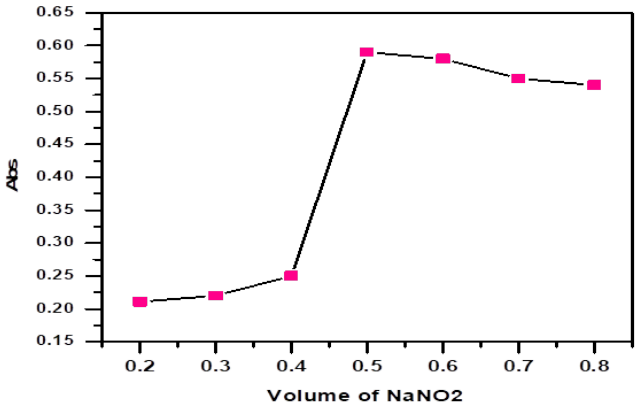

Figure 2: Influence of $NaNO_2$ volume

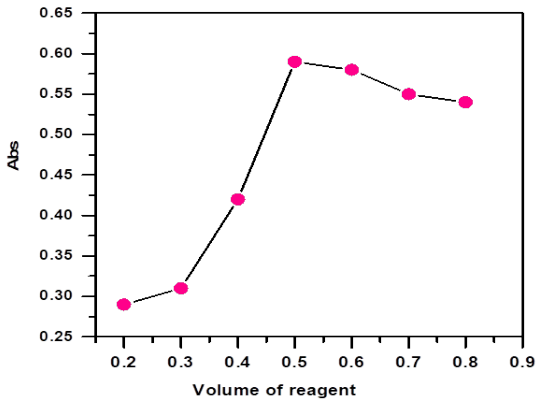

Figure 3: Influence of volume reagent

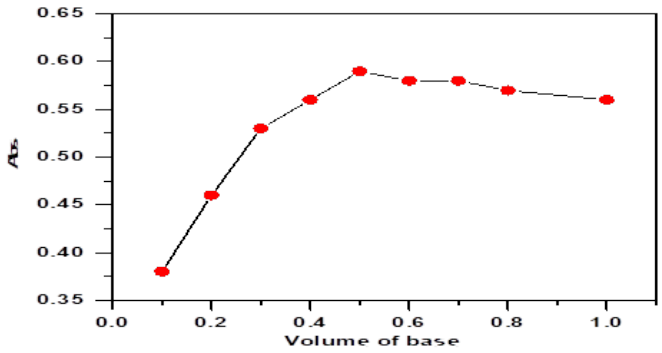

Figure 4: Influence of NH_3 volume

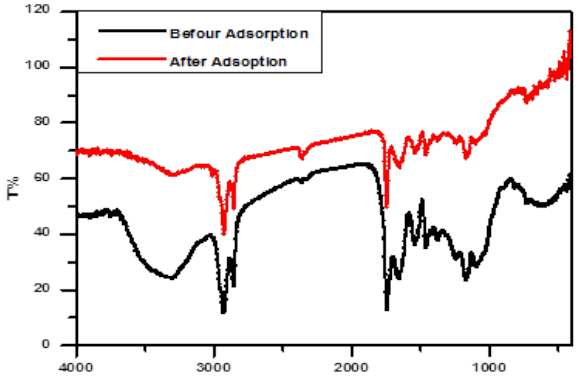

Figure 5: FT-IR spectra of AC before adsorption, after adsorption of AMX drug.

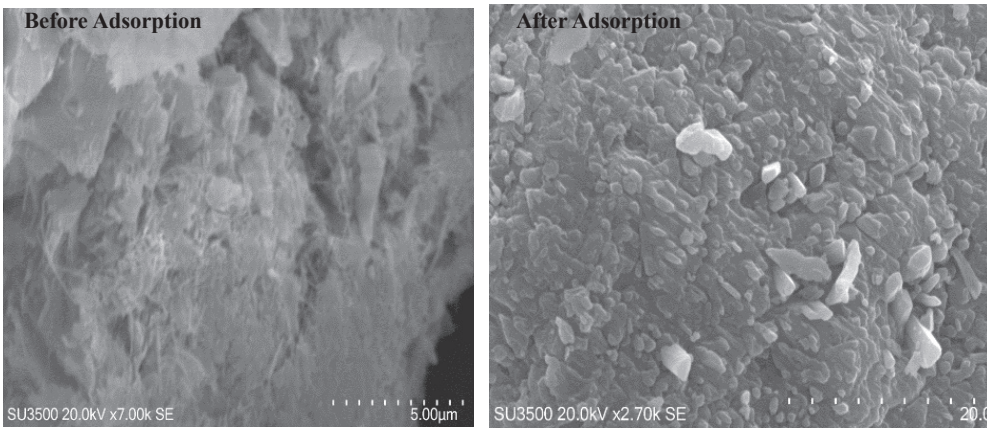

Figure 6: FESEM of Activated Carbone before and after adsorption

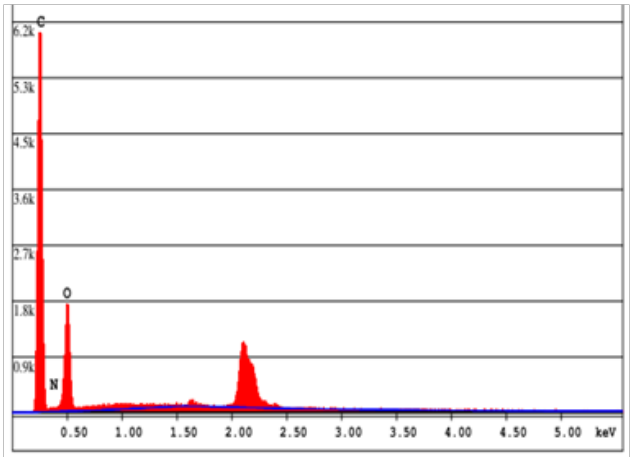

Figure 7 : Energy Dispersion x-ray: of AC

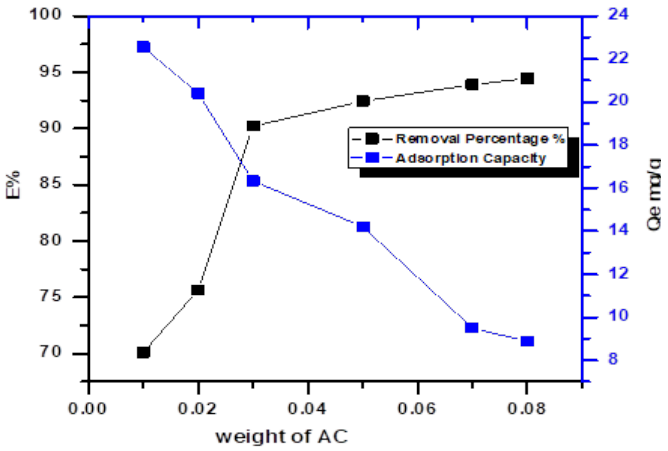

Figure 8: Influence of weight of AC on the E % of AMX drug onto AC.

carbon before and after the adsorption process. It reveals that the surface, after adsorption, appears in agglomerations, and the fullness of all active sites of surface are evidence of loading and occurrence of the adsorption process.[22]

Energy Dispersive X-Ray Analysis (EDX)

EDX is a versatile technique utilized for semi-quantitative and qualitative analysis, Figure 7 show the EDX patterns of AC, which verified the existence of AC and C in O.[23,24]

Effect of Mass of AC

Variation of adsorbent dosage appears that increasing the mass of AC in aqueous solution can data to increased removal AMX. The plot of removal % of AMX drug adsorption against the mass of AC. From Figure 8, it is observed that the removal E% of adsorption is risen by mass of AC increase. This can be attributed to the surface area increase of the AC, which in turn increases the binding sites. At greater mass, very fast adsorption onto the surface adsorbent leads to enhanced uptake of the drug.[25,26]

Effect of pH

The pH solution has a very active role in the adsorption method, especially in removing pollutants, where we notice the best percentage of removal in the basic medium. the influence of pH solution on the extraction of the target is studied in the pH

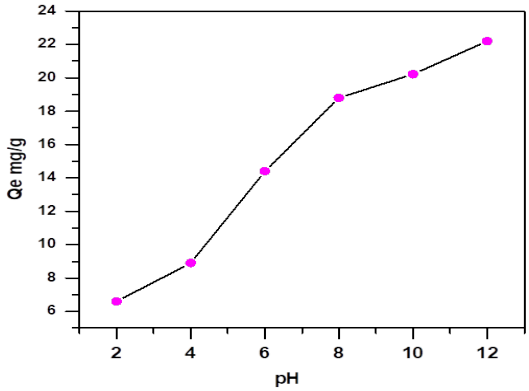

Figure 9: Effect of pH solution on adsorption AMX drug onto AC.

series of 2–12.when the pH increase from 2 to 12, the removal percentage % increase and Found the best adsorption capacity at pH12 and give 22.55 mg/g (Fugure 9).[27,28]

Adsorption Isotherms

Freundlich Isotherm

The isotherm Freundlich model is defined through the following equation 3.[29]

$$q_e = K_f C_e^{1/n_3}$$

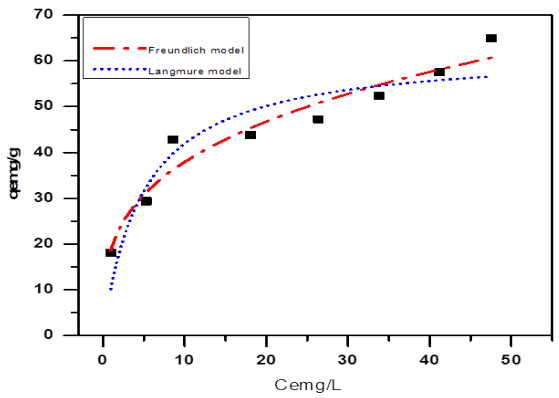

Figure 10: Plot of Qe vs. Ce

Table 1: Several factors Isotherm for the adsorption study of AMX onto AC

Temperature/°C		*10°C*
Freundlich	Kf	18.961 ± 2.3706
	1/n	0.3013 ± 0.0375
	R2	0.9351
Langmuir	qm (mg/g)	62.395 ± 5.199
	KL (L/mg)	0.205 ± 0.0788
	R2	0.8397

Isotherm Langmuir model

The Langmuir model has mainly been utilized for pollutants adsorption from liquid solutions. The nature of the adsorption process was derived by Langmuir alternative equation 4.[30]

The coefficients of estimation(R2) and model factors from the non-linear regressive way were listed in Table 1. A comparison of non-linear fitted curves from experimental data and two Isotherm models at 20°C appears in Figures 10.

A plot of qe vs. Ce (Figure 10) where the values of 1/n and K_F are obtained from the slope and intercept of the linear regressions (Table 1).

CONCLUSION

- A simple and sensitive method was used for spectrophotometric determination, which is the oxidative coupling method. This method gives high color stability within an hour.
- It was found that by increasing the surface weight, the removal percentage increases and the adsorption efficiency decreases and that the best pH is in the basic medium.
- The surface used is highly efficient in pharmaceutical pollutants.

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