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# Kinetic and Equilibrium Studies of Salicylic acid Adsorption from Contaminated Water by (Alginate/Chitosan/Cobalt ferrite) Nanocomposites.

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## ABSTRACT/RESUME

***Abstract:*** *The main objective of this study is the synthesis of the (Alginate / Chitosan/ Cobalt ferrite) nanocomposites. The asdsorbent was characterized via Fourrier Transform Infrared Spectroscopy (FTIR), X-ray diffraction (DRX) and zeta potential. The adsorption tests of salicylic acid on the (Alginate / chitosan / cobalt ferrite) adsorbent were carried out at a temperature of 25°C, pH equal to 4, a mass of 10 mg and a concentration of 10 mg /L. Under these conditions, the adsorption capacity was found to be of the order of 10 mg /g, which corresponds to an equilibrium time of 90 min. The kinetics of adsorption is correctly written by the kinetic model of the pseudo second order. The adsorption data were fitted using the Langmuir and Freundlich isotherms, and the obtained modeling equilibrium adsorption data suggested that the (Alginate / Chitosan/ Cobalt ferrite) nanocomposites sample contained homogeneous adsorption sites that fit the Langmuir adsorption model well.*

## I. Introduction

Emerging pollutants include chemical, pharmaceutical, biological or micropollutant contaminants without clearly defined regulatory status. These are substances, not necessarily new, but newly identified, for which data on their presence, fate in the environment and their potential impacts on health or the environment are fragmentary [1]. Among these emerging pollutants are particularly concerned drugs subject or not to medical prescriptions (antibiotics, pharmaceuticals, hormones) for human or veterinary use, products of daily use (detergents, disinfectants, antioxidants ...) and products of industrial origin. The number of molecules involved is constantly changing both in terms of the parent products and their degradation products (natural or from treatment). The identification and study of the fate of emerging pollutants constitute major challenges for the sustainable management of water resources. Adsorption is currently one of the most widely used separation techniques for decontaminating industrial water [2,3]. Indeed, the performance and the efficiency of this technique depend preponderantly on the nature of the support used as adsorbent. Cobalt ferrite is a soft ferrite with good magnetic properties and high electrical resistivity [4]. The use of biopolymers as adsorbents is an emerging technique for the treatment of industrial effluent containing toxic metals, and has been of great interest to researchers [5].Chitosan exhibits reasonable solubility in acidic aqueous media, chemical stability, biodegradability, and biocompatibility. Chitosan contains a large number of chemical groups, such as amine and hydroxyl groups, and other functionalities may also be added to the polymer chain, resulting in strong interactions and adsorption properties with molecules and ions [6]. Alginates are receiving increasing attention due to their extraordinary affinity to heavy metal ions and several functional groups. Particularly in gel particle form. In addition, the materials are abundant, biocompatible and environmentally friendly, making them potential biosorbents for the removal of pollutants from wastewater [7].

The objective of this study is to test the new (Alginate/ Chitosan / Cobalt ferrite) nanocomposites adsorbent for the removal of salicylic acid from contaminated water.

## II. Materials and Methods

### II.1.Material

Alginate, Sodium hydroxide NaOH (98%), salicylic acid were purchased from Aldrich. Cobalt chloride ($CoCl_2$ .6 $H_2O$), calcium chloride ($CaCl_2$ 0,1M) were supplied by Biochem. Ferric chloride ($FeCl_2$, $4H_2O$ (97%)) was supplied by de Riedel-deHaen. Chitosan is extracted from shrimp shell according to the protocol mentioned in [8].

### II.2.Synthesis of Cobalt Ferrite

Cobalt ferrite has been synthesized according to the following protocol [9]:
A 12.9 g of ($CoCl_2$) and 25.34g of ($FeCl_2$, $4H_2O$) were solubilized in 100 mL of distilled water, the pH was adjusted using a solution of NaOH (9M) to maintain the pH equal to 9. The solution thus obtained was hydrolyzed by refluxing for 2 hours. The precipitate was recovered by centrifugation and then rinsed with distilled water several times. Finally, the precipitate was dried in an oven at 60 °C under air. After drying, the product was milled.

### II.3. Synthesis of the (Alginate /Chitosan/ Cobalt ferrite) nanocomposites

For the preparation of the (Alginate /Chitosan /Cobalt ferrite) nanocomposites, a 0.25 g of chitosan is introduced into 12 mL of distilled water. The dissolution reaction of chitosan was carried out by the addition of 0.4 mL of acetic acid. The mixture thus obtained is stirred for 24 hours, then 0.25 g of alginate was solubilized in 12 mL of distilled water using stirring for 30 min, then, the two solutions were mixed and a mass about 0.5 g of cobalt ferrite was dispersed in a volume of 4 mL of distilled water which is kept stirring during 15min, then the three solutions were mixed and the product obtained was introduced into a syringe and added dropwise in a bath of a solution of calcium chloride (0.1M $CaCl_2$). The mixture is left to rest for a maturation period of two days. The beads are recovered, filtered and then washed with distilled water several times and dried in an oven at 60 ℃ under air.

## III. Characterization

The (Alginate /Chitosan / Cobalt ferrite) nanocomposites was characterized by transform infrared spectroscopy of the JASCO 4200. The IR spectra were measured in the 400–4000 $cm^{-1}$ region. The XRD patterns were obtained with a Perkin Elmer diffractometer with Cu Kα a radiation (α=1.54059 Å). The experiments were carried out directly on the samples studied in the range of 2θ which varies from 5 ° to 75°. The effect of pH on the variation of the zeta potential (ξ) makes it possible to determine the isoelectric point. This is defined as the pH value for which the zeta potential is zero. The zeta potential measurements were carried out using a device (Horiba Scientific Nano Particle Analyzer SZ-100) as a function of the pH, using volumes of 10 mL of solution. The measurements were made for different pH values: 2; 4; 6; 8 and 10.

## IV. Adsorption studies

In a thermostated cell a 10mg of adsorbent was added to 10mL of 10mg/L of salicylic acid aqueous solution, which was previously adjusted to a desired pH (with 0.1N HCl and 0.1N NaOH). Sampling of the supernatant liquid was realized at specified time intervals. The determination of the concentrations of the emerging pollutant was carried After magnetic filtration and analyzed directly by UV-visible spectrometric of the type "Shimadzu Uv Spectrophotometer, Uv-1800", at the wavelength equal to ʎ = 293nm. The adsorption capacity Q was computed from the following equation (1):

$$Qe = \frac{(Co - Ce) * V}{w} \qquad (1)$$

With
Qe is the adsorption capacity ($mg.g^{-1}$), $C_0$ and Ce are the initial and equilibrium concentrations (mg $L^{-1}$) respectively, V is the volume of the solution (L) and w is the weight of the adsorbent in g.

## V. Results and Discussion

### V.1. Characterization

In order to highlight the magnetic properties of the nanocomposite, several tests were carried out; a test showed that a mass of the nanocomposite was attracted by the magnet, which confirms the magnetic properties.
The FTIR spectrum of adsorbent is shown in Figure 1. The peak observed at 3396 $cm^{-1}$ was assigned to the vibration of O-H bonds. The peaks attributed to the vibration of C-H were obvious at 1628 $cm^{-1}$. The wavelengths located at 1628, 1375, 1026 and 536 $cm^{-1}$ correspond to the groups $NH_2$, C-O-H, C-O-C and the groups C-N respectively. The peaks recorded at 490 $cm^{-1}$ and 680 $cm^{-1}$ indicate the tetrahedral and octahedral Fe-O bonds respectively. The results are consistent with the work [10]. X-ray diffraction analysis of the dried and crushed (Alginate / Chitosan /Cobalt ferrite) nanocomposites is shown in Figure 2., The diffractogram obtained has been indexed. The peaks, located at 2θ equal to 17; 27; 29.6; 31.5; 33.5; and 36 were assigned respectively to the reticular planes (1), (2), (3), (4), (5), and (6),The recording of these peaks indicates the spinel structure of cobalt ferrite [11]. The peaks located at 2θ equal to 49 and 50 were assigned to the reticular planes (7) and (8) is the typical peak of alginate [12]. The peaks located at 2θ equal to 59 and 60 were assigned to the reticular planes (9) and (10) is the typical peak of chitosan [13]. The point of zero loads $pH_{PZC}$ is the parameter corresponding to the pH for which the surface of the solid has a zero charge. The isoelectric point named PZC was determined at pH = 4. The surface is positively charged when the pH is below 4 and negatively charged when the pH is above 4 [14].
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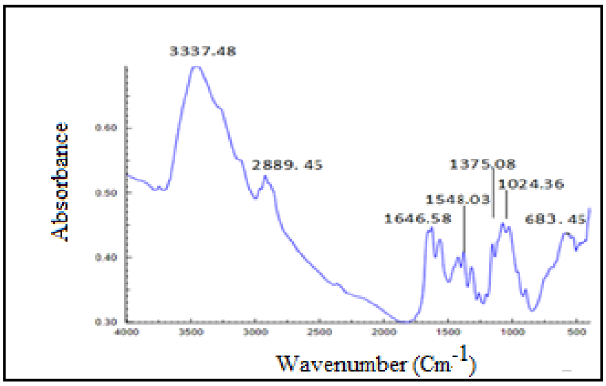


***Figure 1.*** *FTIR spectrum of (Alginate/Chitosan/Cobalt ferrite) nanocomposites.*

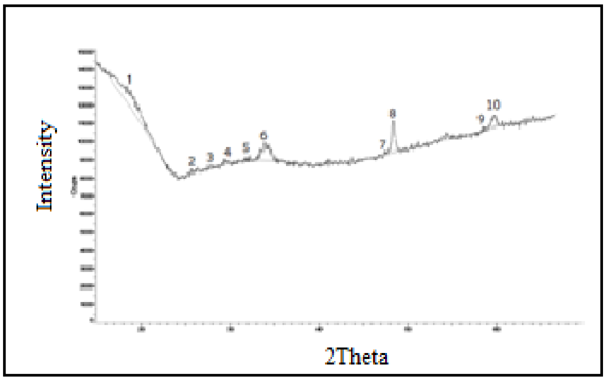


***Figure 2.*** *Diffractograms of (Alginate/Chitosan/Cobalt ferrite) nanocomposites.*

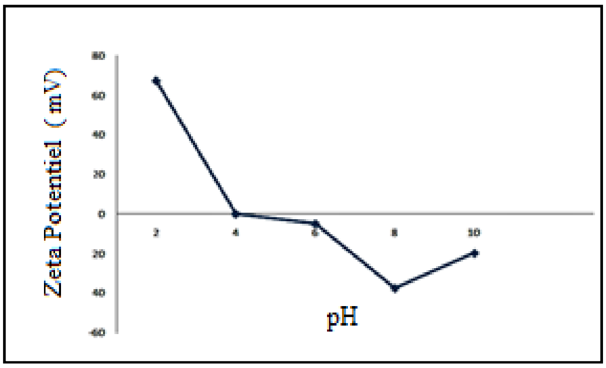


***Figure 3.*** *Determination of the pHpzc point of (Alginate / Chitosan / Cobalt ferrite) nanocomposites.*

**V.2. Study of the adsorption of salicylic acid on the synthesized (Alginate / Chitosan / Cobalt ferrite) nanocomposites**

The determination of the equilibrium time was carried out at a temperature of 25 °C, using a shaker. We have prepared several solutions in flask of 10 mL capacity, by varying the time from 10 minutes to 24 hours. The figure 4 shows the adsorption capacity of salicylic acid on (Alginate / Chitosan / Cobalt ferrite) nanocomposites as a function of time. The curve obtained after these experiments showed that the adsorption process has two distinct parts. During the first part (90 min) the adsorption capacity increases rapidly until reaching a value equal to 10 mg / g. This result is in perfect agreement with the results found by Moulay et al [15].

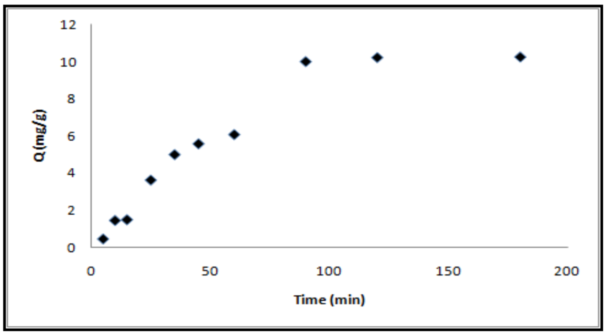


***Figure 4.*** *Adsorption capacity of salicylic acid on (Alginate / Chitosan / Cobalt ferrite) nanocomposites as a function of time.*

In this study several amounts of the adsorbent have been varied from 5mg to 80 mg. Figure 5 shows the variation of the adsorption capacity as a function of the weight of the (Alginate / Chitosan / Cobalt ferrite) adsorbent, it has been found that the adsorption capacity is maximum at a value of weight of 5 mg. This adsorption capacity decreases as the mass increases. This phenomenon may be explained by the bulk and unsaturation of the adsorption sites. This same trend was reported by D. Imessaoudene [16] in the study of biosorption treatment of aqueous effluents from a nuclear facility.

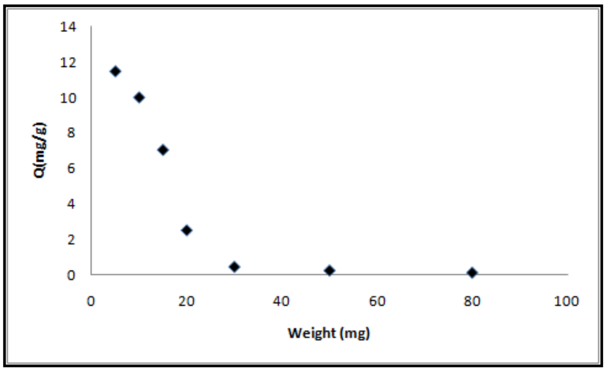


***Figure 5.*** *Adsorption capacity of salicylic acid on the (Alginate/Chitosan/Cobalt ferrite) nanocomposites as a fonction of adsorbent weight .*

The concentration of salicylic acid was varied from 10 to 60 mg/L. From Figure 6 which shows the variation of the adsorption capacity of (Alginate / Chitosan / Cobalt ferrite) nanocomposites as a function of the increase of the initial concentration, the figure indicates that the adsorption process comprises a distinct step. The adsorption capacity rapidly increased to an initial concentration value of 40 mg / L and a large amount of salicylic acid was removed from the 60 mg / L solution. This phenomenon has been observed also by the work of Bensacia et al [17, 18].

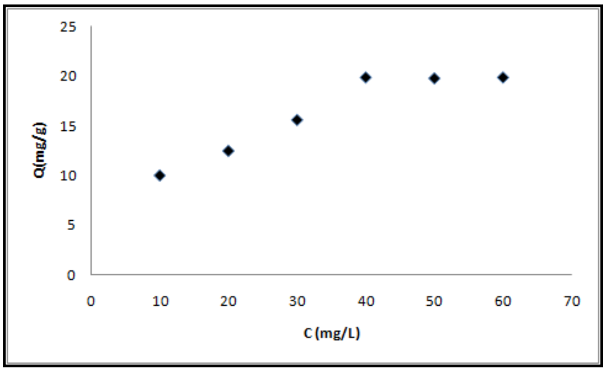


***Figure 6:*** *Adsorption capacity of salicylic acid on the (Alginate/Chitosan/Cobalt ferrite) nanocomposites as a fonction initial concentration.*

Figure7 shows the variation of the adsorption capacity of (Alginate / Chitosan / Cobalt ferrite) nanocomposites as a function of the pH, analyzing the figure, there is an increase in the adsorption capacity of salicylic acid when the pH increases up to the value of pH equal to 4 with an adsorption capacity of 10 mg/g beyond this value a decrease of the adsorption capacity is noticed that can be explained by the fact that when the pH is lower than the isoelectric point of the adsorbent (equal to 4), The surface of the nanocomposites is positively charged and the acid salicylic is in the form of anions (pka = 3), The increase in the pace is probably due to the attractive interaction forces between the adsorbent and the adsorbate, then a decrease in the adsorption capacity was observed, starting at pH equal to 4, This may be due to the repulsive interaction forces between the negatively charged (Alginate/chitosan/cobalt ferrite) nanocomposites and the salicylic acid anions. This result is consistent with that of Seffah et al [10].

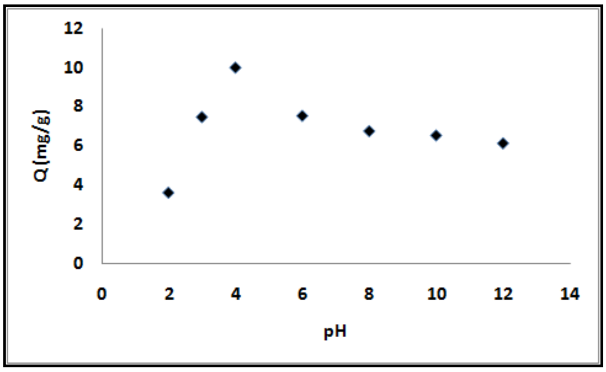


***Figure 7:*** *Adsorption capacity of salicylic acid on the (Alginate/chitosan/cobalt ferrite) nanocomposites as a fonction of pH.*

### V.3. Adsorption Kinetics

Adsorption kinetics is essential for wastewater treatment because it provides the necessary information on the reaction pathway and adsorption dynamics.

#### V.3.1. Kinetic model of the pseudo first order

The kinetic model of the pseudo first order [19] is represented by the following equation:

$$\ln(Q_e - Q_t) = \ln Q_e - k_1 \, t/2.303 \qquad (2)$$

Where:
Qe: amount adsorbed at equilibrium ($mg.g^{-1}$),
Qt: quantity adsorbed at time t ($mg.g^{-1}$),
$K_1$: pseudo first order rate constant ($min^{-1}$).

#### V.3.2. Kinetic Model of the pseudo second order

The pseudo second model according to previous work of Ho and McKay [20] is described by equation (3) as follows:

$$\frac{t}{Q_t} = \frac{1}{Q_e} t + \frac{1}{k_2 Q_e^{\,2}} \qquad (3)$$

Where:

Qe: amount adsorbed at equilibrium ($mg.\ g^{-1}$).
Qt: quantity adsorbed at time ($mg.\ g^{-1}$).
$K_2$: pseudo second order rate constant ($g.mg^{-1}.min^{-1}$).

The pseudo-first-order and pseudo-second-order sorption kinetics parameters are shown in Table 1.

***Table 1****: Parameters of pseudo first order and second order models*

| Pseudo first order | Pseudo second order |
|---|---|
| $K_1$=0,49 $min^{-1}$ | $K_2$ =0,039 $g.mg^{-1}.min^{-1}$ |
| Qe = 13,69 $mg.g.^{-1}$ | Qe= 10.23 $mg.g.^{-1}$ |
| Qe exp= 10.02 $mg.g.^{-1}$ | Qe exp= 10.02 $mg.g.^{-1}$ |
| $R^2$= 0.96 | $R^2$= 0.98 |

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The correlation coefficients for the pseudo-second-order model are much higher than those for the pseudo-first-order model. Therefore, the experimental values of Qe do not agree well with the calculated theoretical values. Thus, the sorption mechanism of the salycilic acid onto (Alginate /Chitosan/Cobalt ferrite) nanocomposites does not follow the pseudo-first-order kinetics model. The adsorbent system is described well by the pseudo-second-order kinetics model, which implies that the adsorption of the emerging pollutant onto (Alginate / Chitosan/ Cobalt ferrite) nanocomposites may occur via a chemical process involving valence forces that share or exchange electrons.

### V.3.3. Adsorption isotherms

The adsorption isotherm indicates how the adsorbed molecules are distributed between the liquid and solid phases when the adsorption process reaches equilibrium. Analyzing the isotherm data by fitting them to different models is important to determine which model is suitable for design purposes. To describe the adsorption of the salycilic acid studied, two models most frequently used are studied: Freundlich, and Langmuir. All the results were carried out at temperature equal to 25 °C, pH 4 and an equilibrium time of 90 minutes.

#### V.3.3.1. Freundlich Model

Freundlich isotherm theory says that the ratio of solute adsorbed onto a given mass of sorbent to the solute concentration in solution is not constant across different concentrations. This model is based on the relation between the adsorbed quantity and solute concentration at equilibrium (Ce). It describes non-ideal and reversible adsorption and is not restricted to a monolayer. The Freundlich equation [21] is expressed by the following relation:

$$\log Q_e = \log K_F + n \log C_e \qquad (4)$$

By plotting log (Qe) = f (log Ce), the results obtained are represented in Figure 8 from which the slope of the line is n and the ordinate at the origin is log $K_F$.

#### V.3.3.2. Langmuir Model

The Langmuir model assumes that a monomolecular layer forms when adsorption occurs without any interaction between the adsorbed molecules. All of the adsorption sites on the surface involved are assumed to be energetically identical, with no transmigration of the sorbate across the solid sorbent surface. Langmuir [22] proposes the following model:

$$\frac{C_e}{Q_e} = \frac{1}{Q_m}.C_e + \frac{1}{Q_m.b} \qquad (5)$$

By plotting (Ce /Qe) = f (Ce), the results obtained are shown in Figure 9, where the slope of the line is 1 /$Q_m$ and the ordinate at the origin is log 1 / $Q_m$.b.

The correlation coefficients and adsorption parameters extracted from the linear plots of the two Freundlich, and Langmuir models after adsorption of salycilic acid on the (Alginate/Chitosan/Cobalt ferrite) nanocomposites are summarized in the following Table 2.

***Table 2.*** *Parameters of the modeling relating to adsorption isotherms of salycilic acid*

| (Alginate /Chitosan /Cobalt ferrite) nanocomposites | |
|---|---|
| **Freundlich** | **Langmuir** |
| $K_F$= 1.11 | B(L.mg$^{-1}$)=0.81 |
| n=1.29 | Qm(mg/g)=7.028 |
| $R^2$= 0.83 | $R^2$= 0.98 |

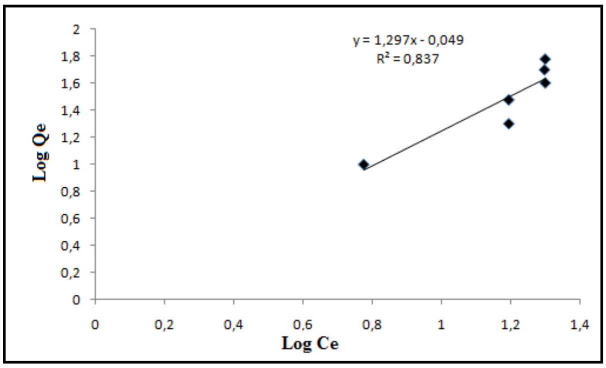


***Figure 7.*** *Modeling the adsorption of salicylic acid by Freundlich model.*

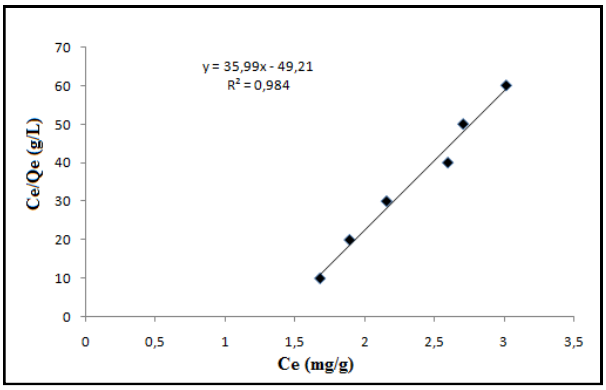


***Figure 8.*** *Modeling the adsorption of salicylic acid by Langmuir model.*

Several authors used the Freundlich and Langmuir isotherms for the removal of different pollutants from wastewater using several sorbents. Our results for the (Alginate / Chitosan/Cobalt ferrite) nanocomposites are presented in Table 2. The Langmuir and Freundlich isotherm parameters and correlation coefficients are summarized. The coefficient of determination ($R^2$) was found to be 0.98 for salicylic acid using the Langmuir model and 0.83 for the Freundlich model. These results indicate that the emerging pollutant adsorption to (Alginate /Chitosan/Cobalt ferrite) nanocomposites fitted the Langmuir model better than the Freundlich model, which may be due to the homogenous distribution of active sites on the nanocomposites surface because the Langmuir equation assumes a homogenous surface where all sites have equal adsorption energies.

## VI. Conclusion

(Alginate / Chitosan / Cobalt ferrite) nanocomposites, was used to remove emerging pollutant from contaminated water. The nanocomposite characterization was achieved by Fourrier Transform Infrared Spectroscopy (FTIR), X-ray diffraction and zeta potential. These techniques have shown the existence of biopolymers in its composition namely alginate and chitosan on the one hand and in the other hand the appearance of Fe-O molecules, which indicates the presence of $CoFe_2O_4$ (cobalt ferrite). The study of adsorption tests allowed us to demonstrate the effectiveness of the (Alginate / Chitosan / Cobalt ferrite) nanocomposites in the removal of salicylic acid, and we have evaluated the effectiveness adsorption capacity of 10 mg/g for an equilibrium time of 90 min, pH equal to 4, mass equal to 10 mg, an initial concentration of 10 mg./ L and a temperature of 25 °C. The high $R^2$ values indicate that the salicylic acid adsorption to the (Alginate /chitosan/cobalt ferrite) nanocomposites follows a pseudo-second-order kinetics model. The Langmuir and Freundlich isotherm models for salicylic acid adsorption to the (Alginate /chitosan/cobalt ferrite) adsorbent were studied, and the Langmuir model best fits the adsorption data. The (Alginate /Chitosan/Cobalt ferrite) nanocomposites can be a new material for removing emerging pollutants from wastewater with high efficiency.

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