

# Column Study on Adsorption-Desorption Behavior of Pharmaceutical Pollutants from Synthetic Water by Immobilized *Chlorella Sorokiniana* Algae

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Article Info	ABSTRACT
<p><b>Article history:</b>  Received Sept., 22, 2025  Revised Nov. 20, 2025  Accepted Dec., 10, 2025</p> <p><b>Keywords:</b>  Antibiotic removal  Bioremediation  Chlorella  Column Study</p>	<p>The release of pharmaceutical contaminants, especially antibiotics, into the ecosystem is a major environmental concern. This study evaluates the effectiveness of the adsorption and desorption process for ciprofloxacin and rifampicin, two common antibiotics due to their known adverse environmental effects. The adsorption process was conducted using a moving column filled with immobilized <i>Chlorella Sorokiniana</i> algae. The study aimed to understand the effect of flow rate on column efficiency and the formation of the breakthrough curve under different operating conditions. The column was set up with a diameter of 4 cm and a length of 90 cm, and the drug solution was pumped at various flow rates (1, 5, and 10 ml/min). Samples were collected from the column outlet at specific time intervals, and the drug concentration was analyzed using a UV-Vis device. The results showed that increasing the flow rate decreased the time required to reach the drug's breakthrough point, with a steeper curve and faster column saturation. After adsorption was complete, the drug removal process was performed using a suitable solvent (5% nitric acid) to ensure drug recovery and analyze the process performance. The results showed an increase in the removal rate during the first 60 minutes, followed by a decrease with time. Mathematical models (Yoon-Nelson, Adams-Bohart, and Thomas), were applied to simulate the adsorption curves and determine the column capacity and removal efficiency. They also helped estimate the effect of flow rate on column dynamics. The models indicated that the column can remove a high percentage of the drug at low flow rates. This study provides a qualitative improvement in the design of moving columns for use in treating wastewater containing pharmaceuticals. The study provides realistic operational data from which precise, detailed adsorption processes can be designed by determining optimal flow rates and other operating conditions to achieve high efficiency. The results can be generalized to other pharmaceuticals. Therefore, it can be considered a practical framework for applying adsorption and dissolution processes in industrial and laboratory settings, leading to the development of management strategies for pharmaceutical-contaminated water to minimize its environmental impacts.</p>

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## 1.Introduction

Water pollution is one of the most significant and pressing environmental challenges of the modern era. Emerging and persistent pollutants, particularly pharmaceuticals, including synthetic and biosynthetic organic compounds, threaten human health and ecosystems alike [1]. Among these

organic pollutants, pharmaceuticals, particularly antibiotics, stand out as emerging and worrisome contaminants, whose presence in aquatic environments around the world is increasing due to untreated discharges from homes, hospitals, and pharmaceutical production plants [2]. Ciprofloxacin (CIP), a broad-spectrum fluoroquinolone antibiotic, and rifampicin (RIF), a key drug for the treatment of tuberculosis, are of particular interest due to their high environmental persistence and low biodegradability. Both pollutants have potential toxic effects on non-target microorganisms in aquatic systems.[3] Their presence, even at low concentrations, poses a significant risk of accelerating the emergence and transmission of antimicrobial resistance (AMR) genes, threatening the effectiveness of anti-infective treatments and posing a global threat to public and animal health [2], [3]. Conventional water treatment technologies (such as chemical precipitation and chlorination) are associated with inefficiency in completely removing these pollutants or lead to the formation of more toxic or resistant products than the original target compounds [4]. Although some advanced technologies such as advanced oxidation process (AOPs) or reverse osmosis (RO) demonstrate higher efficiency, their widespread application is limited by high operating costs, control difficulties, and high energy consumption, which stimulates the search for sustainable and cost-effective alternatives[4], [5]. This has led to a shift in research toward less expensive biomaterials, including agricultural and industrial wastes and natural biomass which is cost effective and environmentally friendly treatment technologies. Among these techniques, bioremediation has received significant attention due to its high efficiency, simple design, and widespread application. Bioremediation, which uses living organisms or their products to mitigate or remove pollutants, avoiding the formation of harmful by-products and in line with the principles of the circular economy.[6] In this context of talking about bioremediation, green algae stand out as an excellent medium for adsorption and removal of organic pollutants due to their natural availability, the ability to cultivate and grow at rapid and controllable growth rates, and their innate ability to remove a wide range of pollutants via multiple mechanisms, including surface adsorption, intracellular accumulation, and/or biodegradation [7], [8]. Algal cell walls are rich in natural polymers such as cellulose, pectin, and proteins, which provide functional groups (e.g., carboxyl, amine, hydroxyl, and phosphate) that serve as effective binding sites for charged molecules such as antibiotics via various interactions (e.g., electrostatic, hydrogen bonding) [5, 6]. Furthermore, algal biomass can be easily harvested, dried, and prepared as biomaterials for use in continuous treatment systems. Although there are studies that have explored the potential of algae in column systems [8], their performance in removing of high-risk antibiotics such as ciprofloxacin and rifampicin remains fully unexplored. such as fixed-bed adsorption columns, potentially providing an economical and environmentally friendly solution [9], [10]. This study aims to evaluate the performance of *Chlorella Sorokiniana* algae immobilized in an adsorption column for the removal of ciprofloxacin and rifampicin from contaminated aqueous solutions. The algae were prepared and grown, and the dynamics of the adsorption process in the column system were studied. The effects of flow rate, the most important one on the shape of the breakthrough curve and overall column performance will be investigated. This study contributes to bridging the gap between batch laboratory research and real-world engineering applications through continuous column systems, paving the way for the use of these renewable raw materials in industrial wastewater treatment.

## 2. Mathematical modeling of antibiotic adsorption in fixed columns

Interpretation of breakthrough curves produced by adsorption of air pollutants in fixed columns is done by using several mathematical models to estimate the kinetic constants and adsorption capacity. The most important of these models are the Thomas model, the Adams-Bohart model, and the Yoon-Nelson model.

### 2.1 Thomas Model

The Thomas model (1944) is one of the models applied in analyzing the practical data of adsorption columns. The application of the Thomas model is based on the basic assumption that the adsorption process follows second-order kinetics similar to the Langmuir model, and that diffusion within the granule is so small that there is no internal resistance. It is applied for its simplicity and accuracy for such systems [11] .

$$\frac{C_t}{C_o} = \frac{1}{1 + \exp\left(\frac{K_{Th} q_o M}{Q} - K_{Th} \cdot C_o \cdot t\right)} \text{----- (1)}$$

And of linearized form as:

$$K_{Th} \cdot C_o \cdot t - \frac{q_o M}{Q} K_{Th} = \ln\left(\frac{C_o}{C_t} - 1\right) \text{----- (2)}$$

Where

$C_t$  is concentration of the drug at the exit of the column over time  $t$ , mg/L.

$C_o$  is initial concentration of the drug, mg/L.

$K_{Th}$  is Thomas constant, L/g.min.

$q_o$  is maximum adsorption capacity, mg/g.

$M$  is mass of the adsorbent in the column, g.

$Q$  is volumetric flowrate, L/min.

$t$  is time, hr

## 2.2 Yoon-Nelson Model

The Yoon-Nelson model is theoretically simpler and does not require details about the physical properties of the adsorbent, such as flow rate or mass of the adsorbent used. However, it assumes that the decrease in adsorption rate of a particular molecule is proportional to the probability of that molecule being adsorbed to the surface at that moment, as well as the probability of it not being adsorbed and passing through the column. This means that the active sites may be half saturated and the other half free, and this corresponds to the moment in which ( $C_t/C_o$ ) is 50%. [12], [13]

$$\frac{C_t}{C_o} = \frac{1}{1 + \exp(K_{YN} \cdot (\tau - t))} \text{----- (3)}$$

And of linearized form as:

$$K_{YN} \cdot (\tau - t) = \ln\left(\frac{C_t}{C_o - C_t}\right) \text{----- (4)}$$

Were

$K_{YN}$  is Yoon-Nelson constant, 1/min.

$\tau$  is time necessary for adsorbed 50% of  $C_t/C_o$ .

## 2.3 Adams-Bohart Model

The Adams-Bohart model focuses on the initial description of breakthrough curves, i.e., before the breakthrough point is reached. It assumes that the adsorption rate is proportional to all remaining concentrations in the solution and the remaining capacity of active sites in the adsorbent. Therefore, this model is less accurate at the full saturation stage[14].

$$\frac{C_t}{C_o} = \exp\left(K_{AB} \cdot C_o \cdot t - K_{AB} \cdot N_o \frac{Z}{U_o}\right) \text{----- (5)}$$

And of linearized form as:

$$K_{AB} \cdot N_o \frac{Z}{U_o} - K_{AB} \cdot C_o \cdot t = \ln \left( \frac{C_t}{C_o} \right) \text{----- (6)}$$

Were

$K_{AB}$  is Adams-Bohart constant, L/mg.min.

$N_o$  Volumetric adsorption capacity or the maximum concentration of pharmaceutical contaminants that the adsorbent can tolerate, mg/L.

$Z$  height of adsorbent layer, Cm.

$U_o$  superficial velocity, Q/A, Cm/min.

$A$  cross sectional area of column,  $\text{Cm}^2$ .

#### 2.4 Mass Transfer Zone MTZ

This region represents the part of the column in which the actual change in drug concentration from its initial value to its final value occurs. It is known as the region where the possibility of the adsorption process occurring is very high, and the length of this region is an important indicator of the column's performance efficiency, as the shorter of MTZ, the greater the efficiency of the adsorbent material [15].

$$L_{MTZ} = Z * \left( 1 - \frac{t_b}{t_s} \right) \text{----- (7)}$$

Were

$L_{MTZ}$  is length of mass transfer region, Cm.

$Z$  is total height of adsorption layer, Cm.

$t_b$  is breakthrough time or 0.05 of  $C_t/C_o$ .

$t_s$  is saturation time or 0.95 of  $C_t/C_o$ .

#### 2.5 Empty Bed Contact Time (EBCT):

This is the theoretical time a solution spends within the space occupied by the absorbent medium at a given flow rate. It is calculated as follows:

$$EBCT = \frac{V_{bed}}{Q} \text{----- (8)}$$

Where:

$V_{bed}$  is the volume of the medium, and

$Q$  is the flow rate.

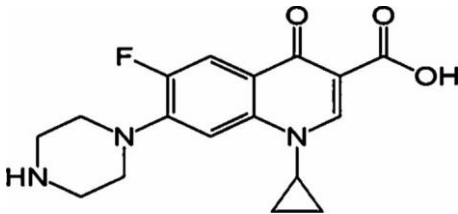
EBCT is used as a criterion for designing adsorption systems; the higher the EBCT, the greater the removal efficiency because the solution has more time to interact with the medium.[16], [17]

### 3. Materials and Methods

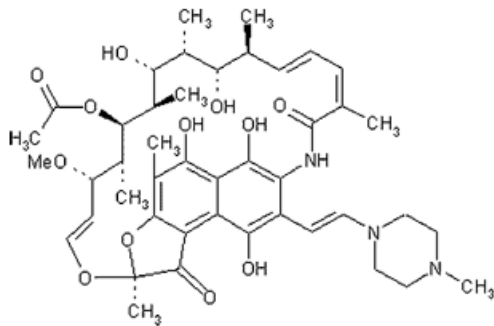
#### 3.1. Antibiotics

Ciprofloxacin  $C_{17}H_{18}FN_3O_3$  and rifampicin used in this research was obtained from Samarra Pharmaceutical and Chemical Industries Company (Samarra, Iraq). Table 1,2 shows the chemical structure, molecular weight and pKa values of ciprofloxacin and rifampicin [18], [19]. Both of them were prepared at a concentration of 50 mg/L.

**Table1. Physicochemical properties and molecular structure of CIP**

Molecular structure	
Molecular formula	$C_{17}H_{18}FN_3O_3$
Molecular weight (g/mol)	331.3
PKa of carboxylic acid group	$0.15 \pm 5.9$
PKa of basic N-moiety	$8.89 \pm 0.11$

**Table 2. Physicochemical properties and molecular structure of RIF**

Molecular structure	
Molecular formula	$C_{43}H_{58}N_4O_{12}$
Molecular weight (g/mol)	822.953

Log K <sub>ow</sub>	4.24
pK <sub>a</sub>	1.70- 7.90
Solubility (H <sub>2</sub> O, mg/mL)	1.4

### 3.2. Culture medium

The culture medium used, BG-11 (HiMedia, M1958), was prepared by dissolving 1.627 g of it in 1 liter of distilled water. 0.1 NM sodium hydroxide or hydrochloric acid was used to achieve a pH of 7. Sterilization of the medium is an essential step to eliminate any unwanted microorganisms using a heat sterilizer at 121°C/2 bar for 15 minutes.

### 3.3 Culturing the Microalgae species

*Chlorella sorokiniana* sp.MH 923013, isolated from the Tigris River and registered in the GenBank database (accession number MH 923013) [20], was used. It was cultured in a pre-prepared culture medium until harvest, prior to use in experimental work.

### 3.4 Biomass Preparation and Immobilization

Due to mechanical instability and to prevent biomass drift and maintain a high surface area, *Chlorella sorokiniana* algae was immobilized using a polymer matrix composed of sodium alginate (NaAlg) and polyvinyl alcohol (PVA), both of which are biocompatible, non-toxic, and widely used in biosorption applications [21], [22]. The biomass was mixed with a 3% (w/v) sodium alginate solution and a 10% (w/v) PVA solution under gentle stirring to form a homogeneous gelatinous suspension. The mixture was then syringed into a 2% (w/v) calcium chloride (CaCl<sub>2</sub>) solution to promote bonding and pellet formation. These pellets were annealed for 24 hours at 4°C and then rinsed with distilled water to remove excess calcium ions. The immobilized pellets were stored aseptically and used within one week for column packing.

### 3.5 Column Adsorption- Regeneration and Desorption Studies

Column experiments were conducted to evaluate the dynamic removal performance of *Chlorella sorokiniana* for ciprofloxacin and rifampicin under continuous flow conditions. a fixed glass column with an internal diameter of 5.4 cm and a height of 100 cm was used. The column was evenly packed with 120 gm dried dead algae (*Chlorella sorokiniana*) biomass immobilized with sodium alginate and PVA at a ratio of 60% by volume. The column was supported by a filter layer at the bottom to prevent loss of the adsorbent, as well as a filter at the top. The bed height was approximately 55 cm. Figure 1 shows a schematic diagram of the experimentally packed column. Prior to each run, the algal column was gently fed with distilled water to stabilize the bottom and remove any loose particles.

Antibiotic solution with a fixed initial concentration of 50 ppm was continuously introduced into the column in upper flow mode using a peristaltic pump to precisely control the inlet water flow rate. To study the effect of hydraulic loading on adsorption dynamics, three different flow rates were used: 1.0 mL/min, 5.0 mL/min, and 10.0 mL/min. Each flow rate was tested in a separate experiment under identical experimental conditions, maintaining a constant ambient temperature (25 ± 2 °C).

Outlet solutions were collected at regular intervals (every 60 minutes during the initial stages and at extended intervals near the point of penetration) until the column was fully saturated (i.e., when the outlet solution concentration approached that of the inlet). Samples were manually collected into sterile tubes, labeled, and immediately analyzed or stored at 4°C until further treatment. The concentrations of ciprofloxacin and rifampicin in the collected samples were determined using X-ray-visible spectroscopy at  $\lambda$  max = 440 nm for ciprofloxacin after adding the iron (III) reagent, and  $\lambda$  max = 475 nm for rifampicin.

The volume of each collected sample and the corresponding time were accurately recorded to generate breakthrough curves (Ct/C<sub>0</sub>) versus time or volume treated) for each flow rate. These curves

were used to evaluate the adsorption capacity, breakthrough time, and mass transfer performance of the column system.

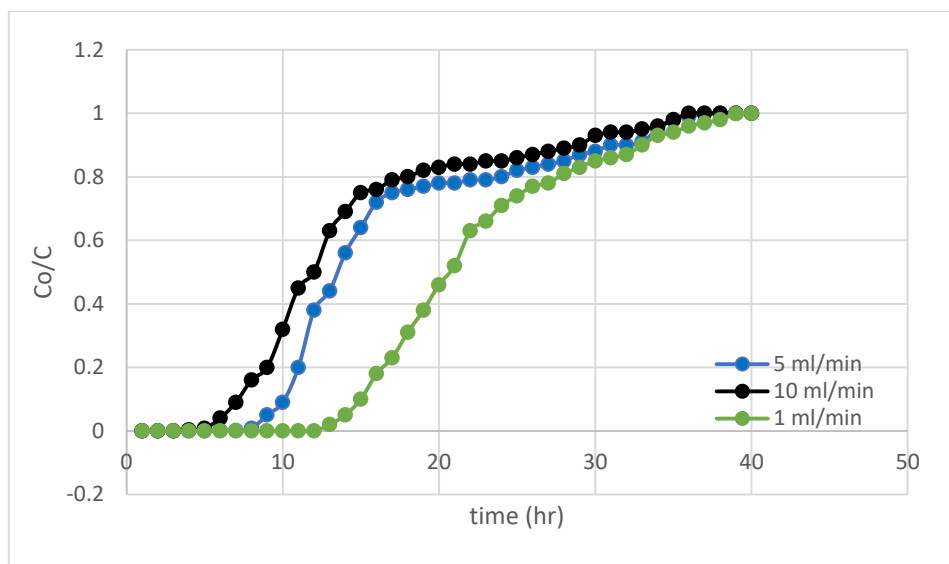


**Figure 1. Experimental Column**

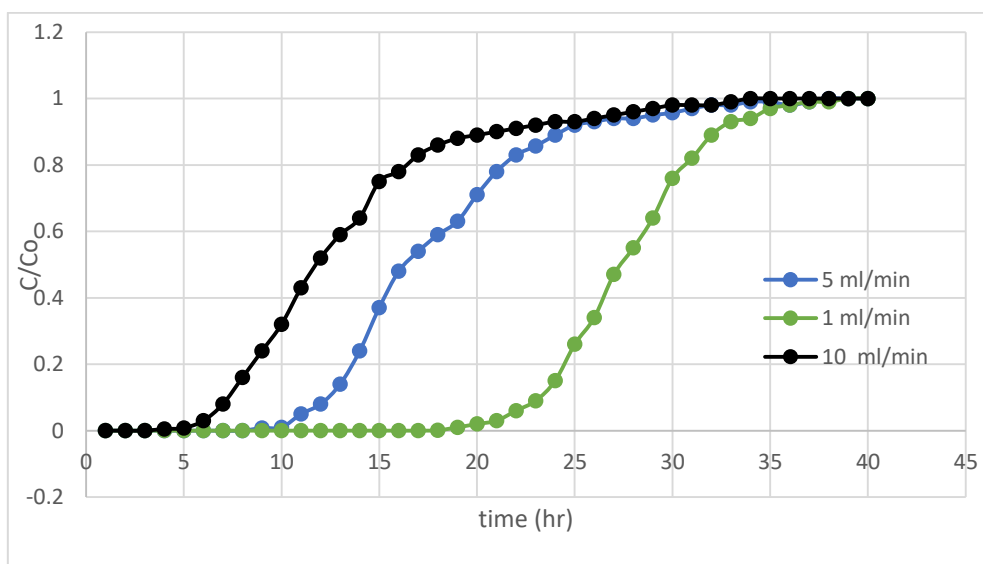
## **4. Results and discussion**

### **4.1 Effect of Flow Rate**

Flow rate is a critical operational parameter that influences the performance of adsorption systems, as it determines the contact time between the adsorbent and the biosorbent within the column. In this study, the effects of different flow rates (1.0, 5.0, and 10.0 mL/min) on the removal efficiency of ciprofloxacin and rifampicin by immobilized *Chlorella sorokiniana* were systematically investigated under identical conditions. Figures 2, 3 show the breakthrough curves for rifampicin and ciprofloxacin. At the lowest flow rate (1.0 mL/min), a longer residence time was achieved, allowing the antibiotic molecules greater opportunity to interact with the functional groups on the algae surface. As a result, the penetration time was significantly delayed, and the removal efficiency was enhanced. When flowrate decrease, longer breakthrough time and a significant delay in reaching total column saturation. This is attributed to the increased contact time between the drug solution and the algae, which improves mass transfer and enables better utilization of adsorption sites. The current data are consistent with recent literature, where Alishiri, M [23] demonstrated that reducing the flow rate in adsorption column studies improves the total adsorption capacity, while Vieira, R. A. L [24] indicated that lowering the flow rate leads to increased removal efficiency of pharmaceutical contaminants. Meina, L [25] also showed that as flowrate increase, time of breakthrough decrease. Same results wer postulated by Dhiman, N [26] and Peng [27].



**Figure 2. Rifampicin Breakthrough curve at different flowrates.**



**Figure 3. Ciprofloxacin Breakthrough curve at different flowrates.**

The breakthrough curve showed a gradual slope, indicating extended adsorption and a high mass transfer zone within the column. This suggests that at slower flow rates, equilibrium conditions were more closely approached. Therefore, optimizing the flow rate is essential to achieve a balance between run time and removal efficiency, as lower flow rates generally result in higher adsorption capacities due to longer mass transfer and diffusion times. MTZ for rifampicin region which represents the part of the column in which the actual change in drug concentration from its initial value to its final value occurs was 55.88, 64, 81.8 cm, for 1, 5, and 10 ml/min. respectively. While for ciprofloxacin, their values were 36.36, 62.06, and 72.22 cm for same flowrates.[28]

The calculated empty bed contact times (EBCTs) of 38, 7.6, and 3.8 hours at flow rates of 1, 5, and 10 mL/min, respectively, showed a clear inverse relationship between flow rate and contact time. Residence time decreases with increasing flow rate, reducing the likelihood of contact with active aggregates. Consequently, the adsorption efficiency decreases.[29].



## 4.2 Desorption -Regeneration study

The desorption of antibiotics from algae is an important factor affecting their mobility and persistence in ecosystems. Figures 4 and 5 shows a two-phase kinetic pattern. The first represents an initial rapid release of adsorbed ions during the first hour, followed by a slower phase leading to a pseudo-steady state. In the first phase, desorbing occurs from the surface sites where the adsorption occurred, where the driving force is high and diffusion barriers are small or non-existent. The second phase reflects desorbing from interfacial sites or functional groups that possess stronger bonds, where diffusion is more impeded. This phenomenon demonstrates the heterogeneous nature of uptake sites and the different affinities between antibiotics and sorbents. The findings of [30] .were give same behavior, they suggest that pharmaceutical contaminants adsorbed at surface sites are easily accessible because their binding is due to van der Waals forces or electrostatic attractions, while the slower phase indicates that contaminants are bound to internal sites where interactions are stronger and diffusion is restricted. While [31], addressed that to effect of molecular stracture and the nature of interaction.

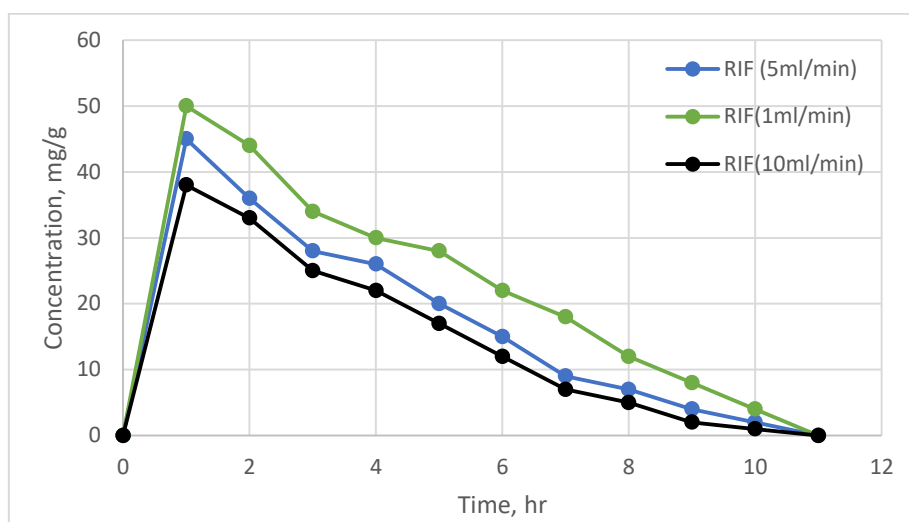


Figure 4. Rifampicin Desorption

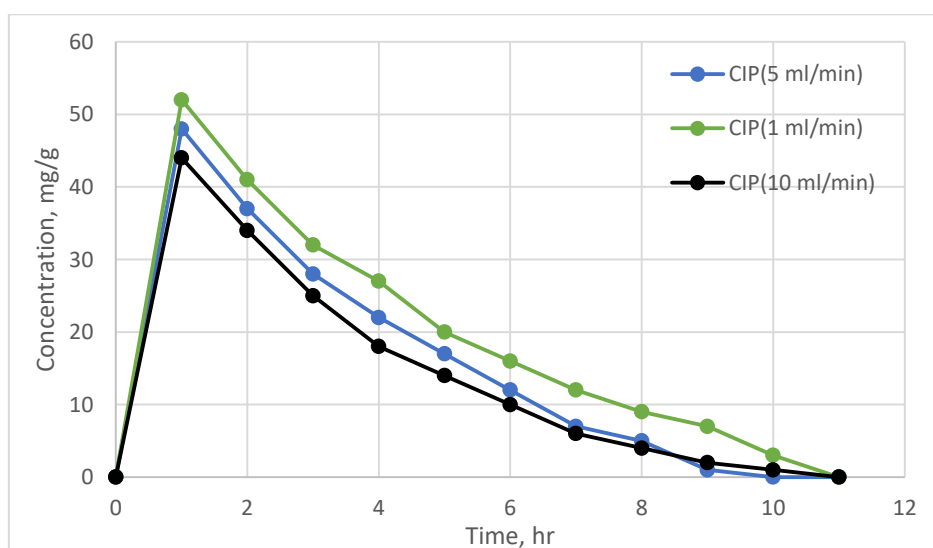
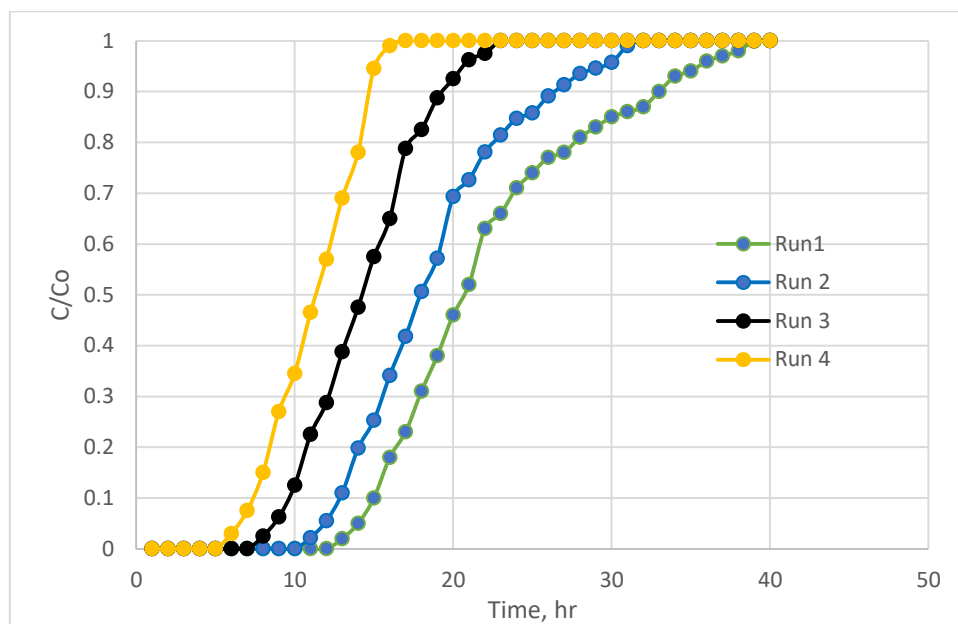


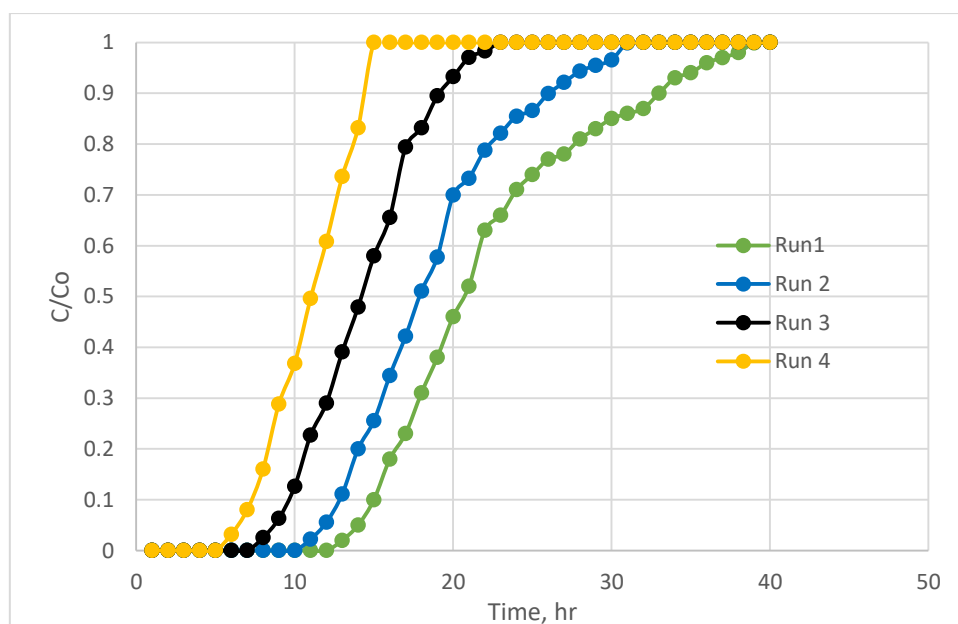
Figure 5. Ciprofloxacin Desorption

While Regeneration experiments as in Figure 6, and 7 showed a gradual decrease in adsorption efficiency over the four consecutive cycles, with penetration time decreasing from 11 hours in the initial cycle to 5 hours in the fourth cycle. This decrease in adsorption capacity can be explained by the loss of some active sites or by toxicity, unlike the synthetic materials used for adsorption. Furthermore, continuous exposure to high concentrations of antibiotics causes a marked disruption in

the algae structure and can lead to the loss of active groups on the cell walls, such as carboxyl and amine, which are responsible for removal, thus reducing the overall adsorption capacity[32]. Pore clogging and physical and morphological changes can occur, reducing adsorption efficiency with each cycle. The retention of some contaminants within the pores may be strongly associated with the low acid concentrations that cannot remove them. Pore clogging reduces the available surface area and thus prevents the diffusion of new contaminants in successive cycles[33]. In addition, repeated chemical and hydraulic treatments can alter the physical structure of the column and its contents, potentially causing compaction or the formation of preferential channels, reducing the efficiency of contact between the two phases. Organic components specifically responsible for adsorption, such as lipids and polysaccharides, can also be eliminated [34]



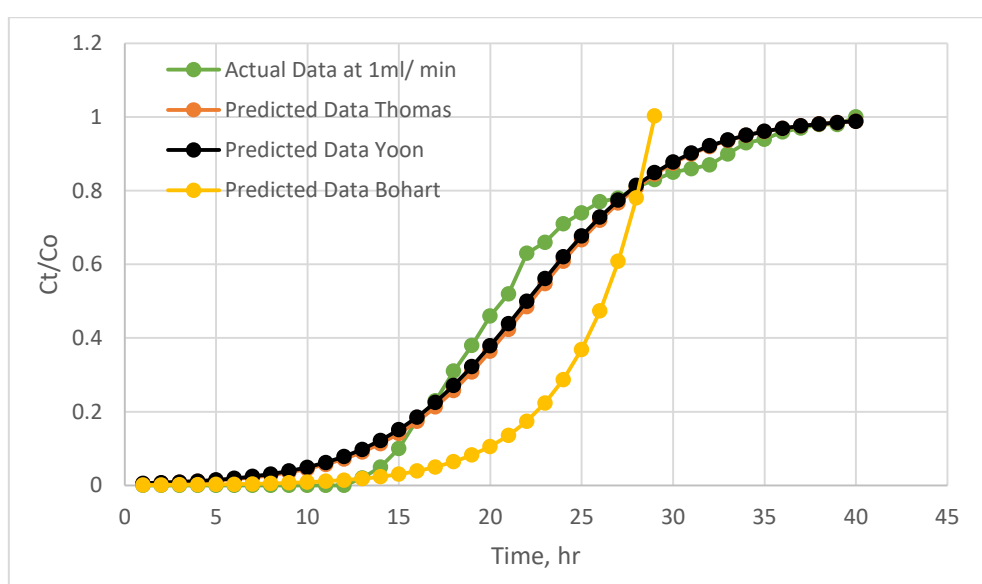
**Figure 6. Rifampicin Breakthrough curve After regeneration for four cycles**



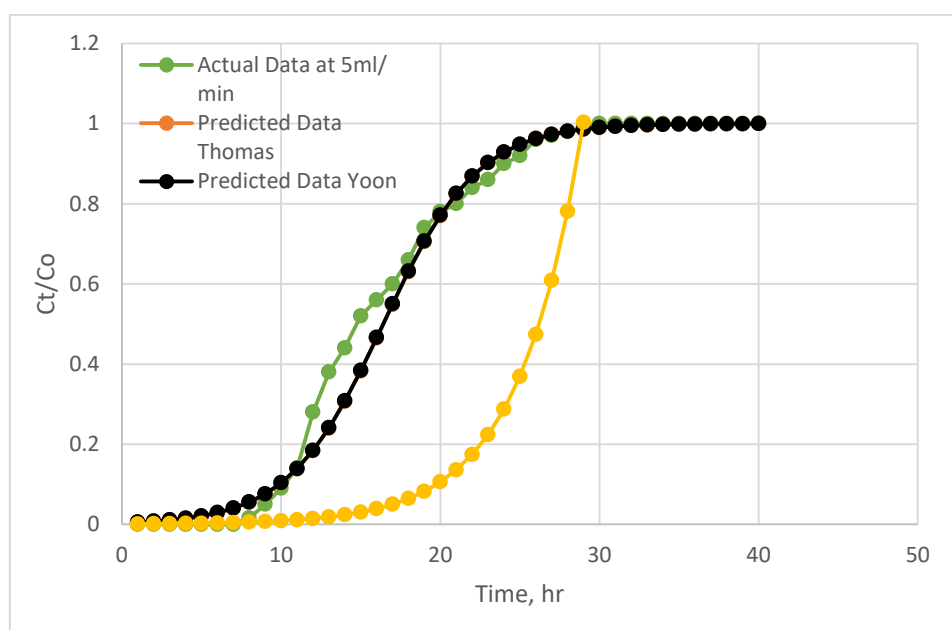
**Figure 7. Ciprofloxacin Breakthrough curve After regeneration for four cycles**

### 4.3 Behavior of modeling of antibiotic adsorption in fixed columns

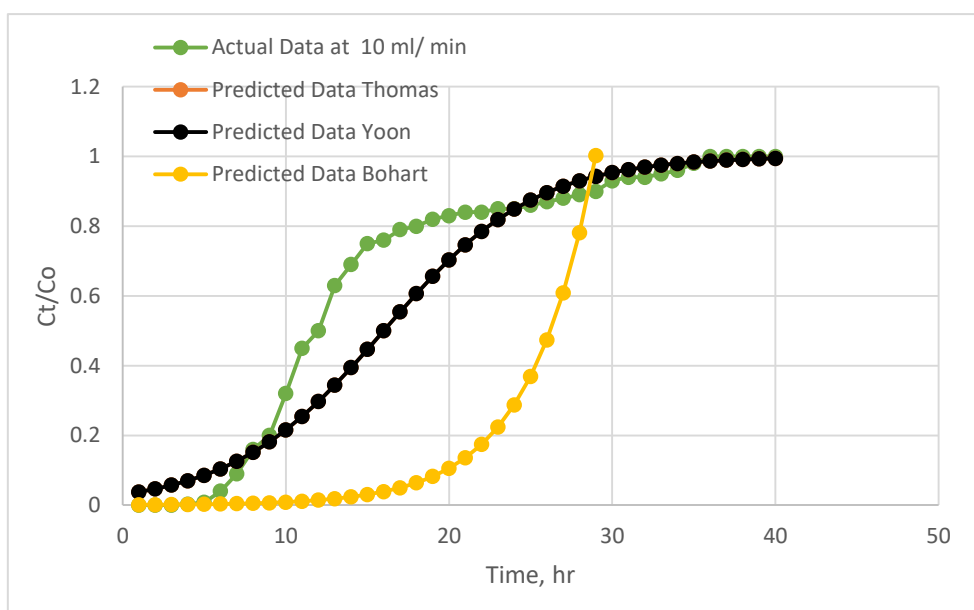
Figure 8-13 illustrate the adsorption behavior in a fixed column at a flow rate of 1,5, and 10 ml/min and compare it with the predicted values using the Thomas, Yoon–Nelson, and Bohart–Adams models. It is clear that the actual data curves fall close to the predictions of both Thomas and Yoon, indicating that these two models are able to simulate the adsorption dynamics well, especially in the middle part of the breakthrough curve. However, the Bohart model gave a larger deviation from the experimental values at long times because the model primarily assumes the adsorption rate depends on the initial concentration and column capacity, ignoring other parameters. These results are consistent with previous studies, where Pal, D [35] demonstrated that the Thomas model is suitable for predicting the performance of adsorption columns for various pharmaceutical contaminants due to its reliance on the Langmuir equilibrium, while Feizi, F [36] demonstrated that the Yoon–Nelson model is simple and accurate in predicting the breakthrough midpoint ( $C_t/C_o = 0.5$ ). In contrast, the Bohart–Adams model often exhibits bias when used over long periods due to its simplifying assumptions. Same findings were illustrated by Ahmad, A. A [37] and García-Mateos [38] and Rafati, L [39].



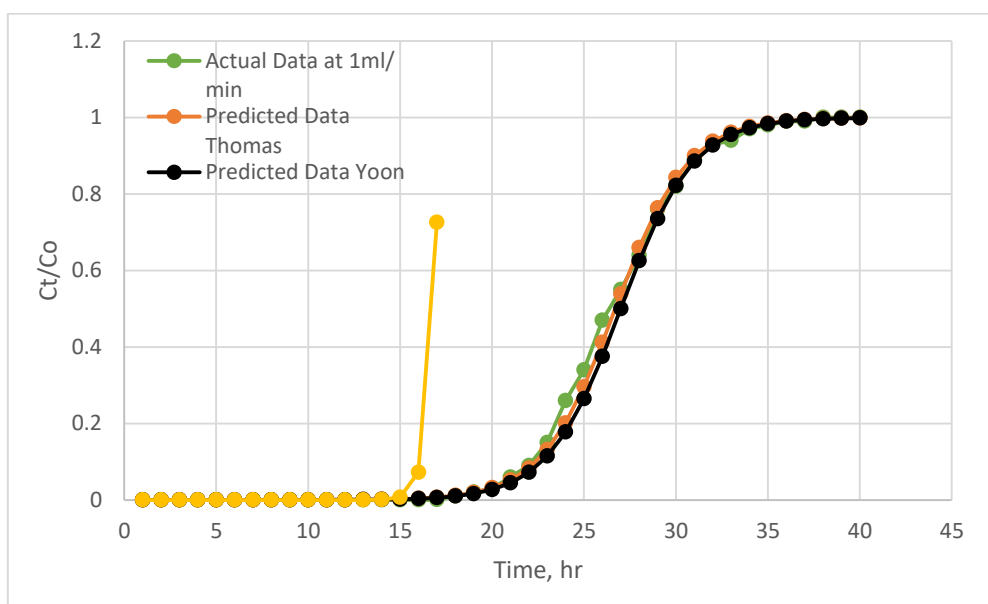
**Figure 8. Modeling of adsorption Thomas, Yoon-Nelson, and Bohart at 1 ml/min.**



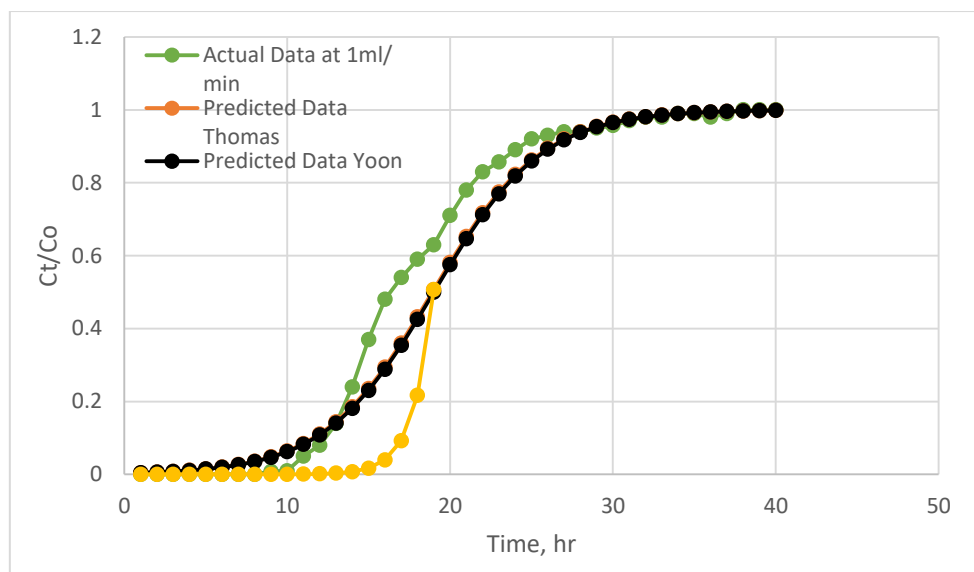
**Figure 9. Modeling of adsorption Thomas, Yoon-Nelson, and Bohart at 5 ml/min.**



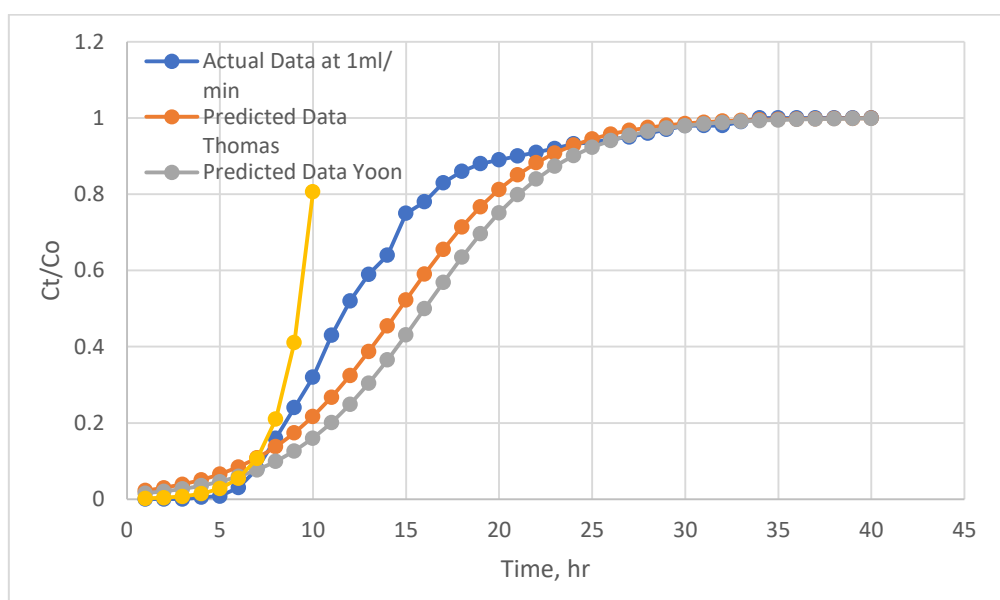
**Figure 10. Modeling of adsorption Thomas, Yoon-Nelson, and Bohart at 10 ml/min.**



**Figure 11. Ciprofloxacin adsorption Thomas, Yoon-Nelson, and Bohart at 1 ml/min.**



**Figure 12. Ciprofloxacin adsorption Thomas, Yoon-Nelson, and Bohart at 5 ml/min.**



**Figure 13. Ciprofloxacin adsorption Thomas, Yoon-Nelson, and Bohart at 10 ml/min.**

The three models give the same behavior. However, increasing the flow rate accelerated the breakpoint and caused a decrease in the total adsorption time.

## 5. Conclusion

The study demonstrated the success of the *Chlorella*-bound algae adsorption column technology in removing the pharmaceutical contaminants rifampicin and ciprofloxacin from aqueous media. The removal rate reached 100% for rifampicin over 12 hours at a flow rate of 1 ml per minute, and 10 ml per minute for 5 hours before the breakthrough point occurred. The removal rate for ciprofloxacin was also 100% for 20 hours, 10 hours, and 5 hours at flow rates of 1 ml per minute, 10 ml per minute, and 10 ml per minute. The flow rate had the most significant impact on the column's dynamic performance, as the results showed an inverse relationship between the breakthrough time and the total adsorption capacity of the column. The decrease in contact time between the liquid and solid phases at high flow rates reduces the efficiency of mass transfer and the contaminant's access to the actual adsorption sites. The results demonstrated the superiority of the Thomas and Nelson models in accurately describing the entire breakthrough curve, while the Bohart model had a large deviation, which limited its usability.

The mass transfer zone values showed significant changes in the dynamics of the mass transfer zone. The length of the zone ( $L_{MTZ}$ ) decreased significantly from 81.8 to 64 then to 55.88 for rifampicin and from 72.36 to 62.06 to 36.36 cm with decreasing flow rate, indicating efficient use of the adsorbent layer and improved column performance. The calculated empty bed contact times (EBCTs) of 38, 7.6, and 3.8 hours at flow rates of 1, 5, and 10 mL/min, respectively indicates decreasing in adsorption process.

## Acknowledgment

The authors gratefully acknowledge the technical support and laboratory facilities provided by the Chemical Engineering and Civil Engineering departments at University of Diyala /College of Engineering.

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