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Review

ADSORBENTS FOR CIPROFLOXACIN REMOVAL FROM AQUEOUS ENVIRONMENTS

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Abstract: Ciprofloxacin (CFX), a fluoroquinolone antibiotic, is widely used in aquaculture for fish growth and treatment of bacterial infections in humans and animals. Its persistence in the environment may induce high resistance to bacteria and potential threat to aquatic ecosystem and human health. In this mini-review we summarized the properties of CFX and its occurrence in water, while highlighting the effectiveness of recently reported adsorbents for its removal as documented in literature. The adsorption mechanisms and future directions in this research domain were also discussed to shed insight into viable applications in wastewater treatment.

***Keywords*:** Adsorbent; Adsorption; Ciprofloxacin; Wastewater treatment

Ciprofloxacin: An Overview

The occurrence of emerging contaminant particularly pharmaceuticals in water sources has recently gained major concerns as it can pose potential hazards to environment and negative effects to human

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health such as reproductive interference, neurotoxicity, and carcinogenicity. The extensive and continuous discharge of pharmaceutical residues into the surface water contributed to the rise in concentration coming from various routes such as municipal, hospital, agricultural, industrial effluent and body excretion.

Ciprofloxacin (CFX), a well-known fluoroquinolone antibiotic, has been widely employed worldwide for the treatment and prevention of bacterial infections in both humans and animals. Specifically, in Malaysia, CFX was included in the list of commonly used antibacterial agents in 2015 and 2016, effectively utilized for the treatment of upper respiratory tract infection (URTI), urinary tract infection (UTI), sinus and skin infections.[1,2] In addition, it has been applied in the agriculture and aquaculture sectors as prophylactic agent and growth promoters.[3]

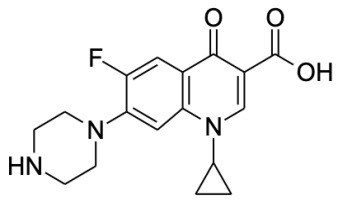
Nowadays, the presence of pharmaceutical contaminants, including CFX, in surface and drinking water has become a subject of global concern, rendering risks to the environment and human health. The presence of CFX has been reported in surface water, with concentrations ranging from ng L^{-1} to mg L^{-1}. In Malaysia, it has been detected in Gombak River[4], while in other countries such as Yangtze River in China[5], and Musi River in India.[6] In a recent study, Praveena et al.[7] reported a 0.32 ng L^{-1} CFX in tap water in Putrajaya, Malaysia.

The presence of pharmaceutical traces in water bodies arises from human or animal excreta due to incomplete metabolism, improper disposal of expired or unused medicines and agricultural runoffs.[8] It is also due to the incomplete removal of pharmaceutical residues in sewage treatment plants. Yacob et al.[9] highlighted that the sewage treatment plants exhibit poor removal efficiency for pharmaceuticals, ranging from 20 to 90%, resulting in the pharmaceutical residues into the river water. Conventional physical, chemical and biological methods of effluent treatment are generally unable

to effectively degrade these emerging contaminants, leading to their entry into the ecosystem.[4,7]

CFX is relatively persistent and stable in the natural environment, and so not easily degradable[10] because of its complex chemical structure and properties as shown in Table 1.[11] Despite the low amounts, the accumulation of CFX brings serious concerns due to possible risks of biological and genetic damage.[12] A prolonged exposure to CFX, even at low concentrations may contribute to the development of antibiotic-resistance bacteria as it reduces the effectiveness of antibiotic in treating infections, endocrine disruption as well as acute and chronic toxicity towards human and aquatic organisms.[13] Thus, there is an urgency to find effective ways to remove CFX from water sources.

Table 1. Chemical and physicochemical properties of ciprofloxacin.[11]

Properties	Ciprofloxacin
IUPAC ID	1-cyclopropyl-6-fluoro-4-oxo-7-(piperazin-1-yl)-1,4-dihydroquinoline-3-carboxylic acid
Molecular formula	$C_{17}H_{18}FN_3O_3$
Molecular structure	
Molecular weight (g mol^{-1})	331.35
Chemical class	Fluoroquinolone antibiotic
Water solubility (mg L^{-1})	650 (pH = 7)
Ionization	Acidic, basic, zwitterion
Maximum wavelength, λ_{max}	277 nm
Dissociation constant (pKa)	pKa1 = 6.1, pKa2 = 8.7
The molecular dimensions size (Å)	13.2 Å × 8.2 Å × 2.5 Å

Coagulation,[14] advanced oxidation,[15] ozonation,[16] and photocatalysis[17] have been reported for the removal of antibiotics from water. It should be noted that these methods often suffered from high energy consumption, inefficient removal of antibiotics, expensive equipment requirements, and complex operating procedures.[18] Among others, adsorption with wide selection of solid adsorbents, is a preferred treatment method for CFX. Nonetheless, there is a quest for efficient and cost-effective adsorbents to remove CFX from water.

CFX adsorbents in action

Adsorption is a recognized treatment method for water pollutants. It is defined as a process in which target molecules (adsorbate) from a solution accumulate on the surface of a solid material (adsorbent). Generally, the adsorption process involves several sequential mass-transfer steps. It begins with the transfer of adsorbate molecules from the bulk solution towards the adsorbent surface (bulk diffusion). Next, the molecules diffuse across the stagnant liquid boundary layer surrounding the adsorbent particle (film diffusion). After overcoming the film resistance, the adsorbate molecules migrate through the liquid-filled pores of the adsorbent toward the internal absorption sites (intraparticle diffusion). Finally, the adsorbate molecules are retained on the active sites through various adsorbate-adsorbent interactions.[18]

The uptake mechanism of pharmaceutical contaminants by porous materials is predominantly governed by the physicochemical interactions established between the pollutant molecules and the surface functionalities of the porous matrix. These interactions can occur through several pathways depending on the characteristics of both the contaminant and the material. Previous literature has identified several types of interactions involved in the adsorption of pharmaceuticals, such as electrostatic attraction, hydrogen

bonding, hydrophobic interaction, π-π staking (dispersive), electron–donor–acceptor interaction, complexation and pore filling.[19] Notably, these interactions may not occur simultaneously in every adsorption of antibiotics. The dominance in interactions typically lies upon the chemical structures and physicochemical properties of adsorbates.[20] Also worth mentioning is the textural and surface chemistry of adsorbent, including the specific area, pore size distribution and functional groups, that play a crucial role in the removal of antibiotics from water. Additionally, the operating conditions, like pH, temperature, ionic strength, adsorbate concentration, residence time, etc. can as well be manipulated to achieve better interactions and adsorption capacity. Therefore, the need for a comprehensive understanding of the mechanism governing the adsorption of pharmaceuticals, specifically CFX, is imperative to enhance the effectiveness of the adsorption process.

Inherent properties of CFX adsorbents

Table 2 presents an overview of recent studies on the performance of various CFX adsorbents. In general, the maximum adsorption capacity (q_{max}) is influenced by a range of factors, including the adsorbent preparation, properties of adsorbents (e.g., surface area, porosity, etc.), surface functionalities, and process conditions (e.g., adsorbent dose, initial CFX concentration, pH, contact time, and temperature), as documented in literature. Generally, adsorbent featuring a greater BET surface area (S_{BET}) tends to exhibit higher CFX adsorption. This correlation is attributed to the fact that a higher specific surface area provides an increased number of active sites available for CFX molecules to bind. For instance, Zhang et al.[3] achieved a remarkable CFX adsorption capacity of 462 mg g^{-1} using desilicated rice husk activated carbon with S_{BET} of 1020 m^2 g^{-1}. In contrast, Patel et al.[21] reported a lower CFX adsorption capacity of 25 mg g^{-1} using banana peel biochar, which featured a smaller S_{BET} of 590 m^2 g^{-1}.

Table 2. Performance of ciprofloxacin adsorbents as reported in literature.

Adsorbent	S_{BET} ($m^2\ g^{-1}$)	q_{max} (mg g^{-1})
Desilicated rice husk residue activated carbon[3]	1020	461.9
Nanostructured pomegranate peel activated biochar[10]	1150	142.9
Amine-functionalized magnetic bamboo activated carbon[11]	982	245.6
Azolla filiculoides marine algae activated carbon[13]	716	35.1
Banana peel biochar[21]	587	23.3
H_2SO_4-modified date palm leaflets carbon (wet)[22]	24	133.3
Mangosteen peel activated carbon[23]	420	29.8
Biochar from used-tea leaf[24]	8	238.1
Jerivá endocarp activated carbon[25]	1435	335.8
Salix psammophila waste activated carbon[26]	1328	279.7
Manganese ferrite modified biochar from vinasse waste[27]	95	357
Sesame straw biochar[28]	935	197.8
Magnetic biochar of camphor leaf[29]	915	449.4
Peanut shell activated carbon[30]	629	87.7
Corncob activated carbon[30]	1006	175.0
Cotton activated carbon[31]	571	61.3
Cherry stalk activated carbon[31]	949	410.1
Rice husk activated carbon[32]	1539	398
Acidified red mud[33]	17	7
Hydrochar activated carbon from cellulose cotton textile waste[34]	972	192

Conversely, activated carbons derived from peanut shell, corncob and cotton demonstrated more effective CFX removal owing to their rich hierarchical porous structures.[30] The formation of specific pore structures was found to be a direct outcome of the process conditions employed during the preparation or modification of adsorbent. In short, the pore structure indicates the accessibility of CFX towards the adsorbent and directly influences its adsorption capacity.

Li et al.[24] reported a considerably high CFX adsorption capacity at 238 mg g^{-1} even with a relatively low S_{BET} of 8 $m^2\ g^{-1}$ of tea leaf biochar. Similarly, Xiang et al.[27] reported the removal of CFX at 357 mg g^{-1} by manganese ferrite-modified biochar derived from vinasse waste with S_{BET} of 95 $m^2\ g^{-1}$. It suggests the interplay of functional groups and aromaticity in

determining the adsorption behavior of the adsorbent. It is noteworthy that the functional groups present on the adsorbent establish various types of interactions with the adsorbate, contributing to the high adsorption capacity. For example, Ouyang et al.[30] recognized the roles of oxygen- and nitrogen-containing functional groups of biochar in the formation of hydrogen bonds with both the acceptor and donor sites in CFX molecules, resulting in the effective removal of CFX. Hence, the specific area and pore distribution are not the sole factors dominating the effective CFX adsorption. Some researchers concluded that the maximum capacity of CFX is greatly affected by the introduction of ammonium nitrogen, hydroxyl and carbonyl groups on the surface of adsorbents which easily interacted with CFX molecules via coordination, electrostatic and H-bonding interactions.[3,11] Figure 1[35] illustrates the possible interactions between CFX molecules and adsorbent surface.

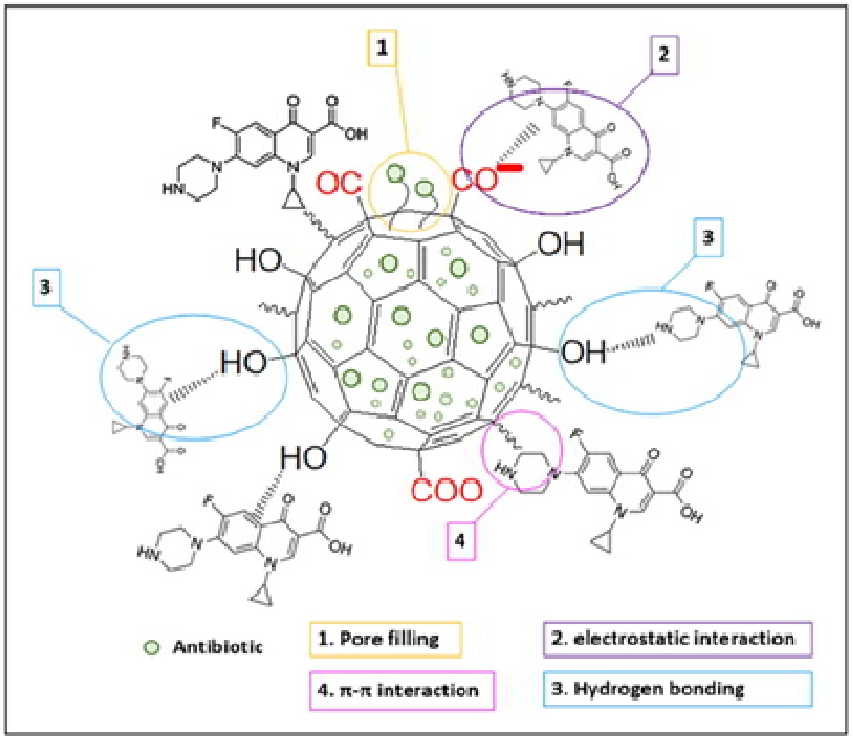


Figure 1. Mechanisms of CFX adsorption.[35]

Roles of operating conditions on adsorption

A critical factor influencing the adsorption of CFX is the adsorbent dosage. Generally, increasing the adsorbent dosage tends to enhance the adsorption efficiency. This is associated with the increased availability surface area and pore volume, leading to the enrichment of surface functional groups and adsorption sites. For instance, an increase in adsorbent dosage of activated carbon obtained from *Azolla filiculoides* marine algae, from 0.25 to 2.5 g L^{-1}, significantly improved the CFX adsorption from 48.5 to 81.95 %.[13] Tran et al.[23] recognized a positive correlation between the adsorbent dosage and CFX removal by mangosteen peel activated carbon that is attributed to the enhancement of surface area and availability of active sites. However, it is noteworthy that further increases in adsorbent dosage may eventually lead to a reduction in CFX uptake. This phenomenon can be explained by several possible factors, including particle agglomeration, solute availability and electrostatic interactions, all of which reduce the effective surface area of the adsorbent.[13,23] Furthermore, if the adsorbent is less effective for CFX, increasing the dosage will substantially decrease the adsorption capacity because CFX adsorbed does not increase proportionally with dosage. Therefore, it becomes imperative to determine appropriate dosage to achieve optimal adsorption capacity.

Typically, the adsorption capacity and removal performance of CFX increased with increasing concentration. Hamadeen and Elkhatib[10] observed an increase in CFX adsorption onto nanostructured pomegranate peel activated biochar as the concentration increases from 10 – 120 μg mL^{-1}. Similarly, Li et al.[24] reported an increase in CFX removal with a maximum efficiency of 50 % at 200 mg L^{-1}. It can be generalized as the concentration-based driving force that enhances the mass transfer of CFX molecules to the

accessible surface sites.[10,24] However, as the initial concentration is further increased from 200 to 500 mg L^{-1}, the efficiency experienced a slight decline from 50 to 35 %, arising from the limited availability of binding sites on the adsorbent to accommodate more CFX molecules.[24]

The pH of solution exerts an influence on the adsorption process as it controls the adsorbent surface charge and degree of ionization of the adsorbate. In addition, the pH at the point of zero charge (pHpzc) of the adsorbent and the acid dissociation constant (pKa) of CFX can have an interplay in the adsorption process. CFX is known to exist in various states, including cations (CFX+), zwitterions (CFX±) and anions (CFX-) and it possesses two pKa values of 6.1 (pKa1) and 8.7 (pKa2)[31], as depicted in Figure 2. Typically, when the solution pH < pKa1, CFX exists in the cationic form due to the protonation of amine groups.[36] On the other hand, when the pH of the solution is higher than pKa2, CFX is predominantly in its anionic form due to the loss of a proton from the carboxylic group. Accordingly, when pKa1 < pH < pKa2, CFX will be in its zwitterionic state. From the viewpoint of pHpzc, the surface an adsorbent tends to be positively charged when the solution pH is below its pHpzc and becomes negatively charged when the pH exceeds its pHpzc. Generally, due to the positively charged nature of CFX adsorbent, its surface is acidic when solution pH < pHpzc, thus it exhibits greater affinity for negatively charged ions. Conversely, when the surface becomes negatively charged at solution pH > pHpzc, it becomes more favorable for positively charged ions.[36]

Several research works have been carried out to understand the effect of pH on CFX adsorption using various adsorbent and relatively consistent optimum pH was reported in acidic condition. According to Patel et al.[21], the highest CFX adsorption was observed between pH 4 – 8, because, at this condition, many cationic, neutral, and zwitterionic species

becomes predominant in the solution, leading to strong electrostatic and H-bonding interactions. It is evident, given that the pHpzc of banana peel biochar is 9.8. However, as pH exceeding 8.64, there is a decline in adsorption due to repulsive interaction between the negatively charged sites and CFX anions. Similar results were recorded for CFX adsorption by sour cherry stalk activated carbon.[31] An increase in adsorption with increasing solution pH from 4 to 6.3 shows the significance of both electrostatic and hydrophobic interactions in CFX adsorption. The pH of the solution affects the surface charge properties of adsorbents and the chemical speciation, solubility and ionization of CFX to some degree.

Thermodynamic parameters like Gibbs free energy (ΔG°), enthalpy (ΔH°), and entropy (ΔS°) are frequently used to describe the adsorption of CFX at different solution temperatures. The range of solution temperature is between 288–333 K. In most studies, the ΔG° increases with temperature, and so the CFX adsorption becomes intensified and more spontaneous. It occurs due to a reduction in solution viscosity, which enhances the diffusion of CFX molecules through the external boundary layer and facilitates its entry into the internal pores.[11] Conversely, the decrease in CFX capacity with increasing temperature is attributed to pore expansion, which allows CFX molecules to escape from the solid phase. Also, the high temperature encourages intense collisions among molecules, further reducing the adsorption capacity.[10,37] From literature, the CFX adsorption has been observed to be both physically and chemically driven. The adsorption is mostly described as endothermic (positive ΔH°), wherein the uptake increased with increasing solution temperature. The CFX adsorption is predominantly physical with $\Delta H^\circ < 40$ kJ/mol. Typically, the positive ΔS° reflects favorable adsorption due to an increase in randomness at the solid-liquid interface.[21,30]

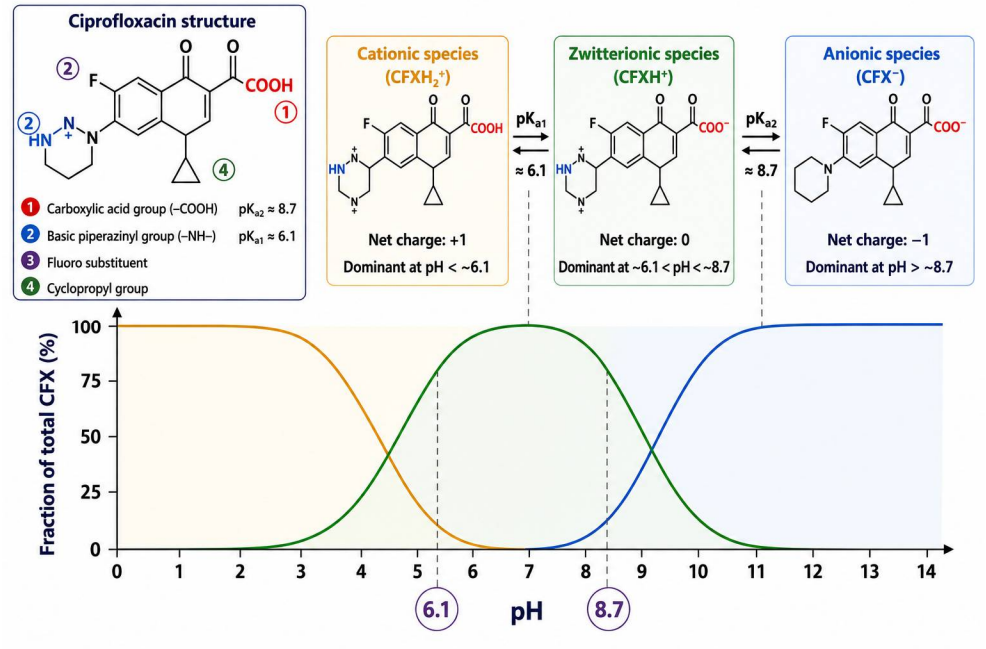


Figure 2. Speciation behavior of ciprofloxacin (CFX) as a function of pH.

Interpretation of adsorption models

Kinetic equations are generally used to describe the adsorption behavior of adsorbed molecules with respect to the reaction time and in response to various factors affecting the reaction rate. In many adsorption studies, pseudo-first-order, pseudo-second-order, and intra-particle diffusion model are among the common adsorption models used to analyze the kinetics of the process. Research findings consistently reveal that the adsorption data of CFX align more closely to pseudo-second-order model signifying a dominant chemosorption process.[28,29] A study by de Oliveira Carvalho et al.[25], on the other hand, reported that the kinetics data fitted well with Avrami model.

The pattern of CFX adsorption typically demonstrates a sharp increase in the initial stage with time due to the plentiful active sites on adsorbent surface. As time progresses and the active sites become gradually occupied, so stabilizing the adsorption process to reach a point of

equilibrium. It is attributed to the diminishing number of adsorption sites and desorption of CFX.[29] Several studies reported the applicability of intra-particle diffusion model to describe the diffusion mechanisms and the rate limiting step in the adsorption process. The linear curve of q_t against $t^{0.5}$ that deviates from origin implies that the adsorption is solely driven or controlled by particle diffusion. [26,28]

The isotherm models are widely used to describe the equilibrium data, mechanisms and surface properties of the adsorbents. The CFX adsorption data in literature are best fitted into Langmuir[11,13,23] and Freundlich[26,27], Sips[28], Liu[25], and a hybrid of Langmuir and Koble-Corrigan[3] models. In most cases, the Langmuir model suggests that the adsorbent surface is homogeneous and that the adsorption is monolayer, where each site is occupied by only one CFX molecule. Meanwhile, Freundlich model implies a multilayer CFX accumulation onto heterogeneous adsorbent surface.

What's next for ciprofloxacin adsorbents?

While the literature on CFX adsorbents is growing[38,39,40], there are still gaps to unlock the true potential for industrial applications as summarized in Table 3. The future of CFX adsorption is to make significant strides to align with environmental sustainability during the preparation and modification of adsorbent materials. It should be noted that the existing production methods contribute to the release of chemicals, thereby jeopardizing the environmental ecosystem. Thus, a future direction must consider the sustainable production of CFX adsorbents, emphasizing to renewable feedstocks and the recovery of chemicals or reagents utilized in both the preparation and regeneration of spent adsorbents.

The applicability of CFX adsorption lies in the need to handle real matrices. The actual river or effluent often contains a mixture of various contaminants and organic matter that can interfere the adsorption. Fast forward, the adsorbents must be tailored to selectively capture CFX from the

complex matrices. Later, the pre-adsorbed CFX can be recovered through solvent regeneration and purified for reuse. The frequency and effectiveness of regeneration cycles could also provide insights into optimizing the lifetime of adsorbents and reducing the overall operational cost of the adsorption process. This overall strategy closes the loop in the adsorption process, aligning with zero-waste principles and the circular economy by promoting resource recovery and minimizing environmental impact.

The present dynamic models are used to explain the column behavior but are inadequate to accurately represent the initial breakthrough phase. Ideally, the effluent concentration ratio (C_t/C_o) is zero at time $t = 0$, implying that no adsorbate appears in the effluent initially. The discrepancy in mathematical assumptions arises due to factors such as axial dispersion, mass transfer resistance, and non-instantaneous adsorption kinetics, which influence the movement of CFX molecules through the column. As a result, existing models may overestimate the early-stage adsorption behavior, leading to inaccurate predictions of breakthrough and overall column performance.[41] Addressing this limitation requires the refinement of dynamic models to incorporate realistic initial conditions and improve their predictive capability for practical applications.

The mass transfer resistance determines the efficiency of CFX adsorption in column system. Often, this is not widely documented in much of the published studies. The advanced simulation tools, such as Aspen Adsorption is useful to model the column operation. Through dynamic simulation, mass transfer with axial dispersion can be estimated, providing deeper insights into adsorbent behavior and enabling the optimization of column design for improved efficiency. This approach allows precise prediction of breakthrough that is pivotal for designing large-scale continuous adsorption system.

Overall, although considerable progress has been made in the development of adsorbents for CFX removal, several challenges remain before their large-scale implementation can be realized. Future research should prioritize the sustainable production of adsorbents from renewable feedstocks while minimizing chemical consumption and promoting reagent recovery to reduce environmental impacts. Greater emphasis is also needed on evaluating adsorption performance under realistic water matrices, improving adsorbent selectivity, regeneration efficiency, and long-term stability to support resource recovery and circular economy principles. In addition, advances in dynamic adsorption modelling are required to better account for mass transfer phenomena, axial dispersion, and breakthrough behavior, thereby enhancing the accuracy of column performance prediction and facilitating the design and optimization of continuous adsorption systems for practical water treatment applications.

Table 3. Key research gaps and future perspectives for ciprofloxacin adsorbents in water treatment.

Research aspect	Current limitation	Future research direction
Sustainable adsorbent production	High chemical consumption and waste generation	Develop renewable feedstocks, greener synthesis, and reagent recovery
Real wastewater application	Most studies use synthesis solutions	Evaluate performance in real wastewater containing competing ions and natural organic matter
Selectivity	Poor selectivity in complex matrices	Design adsorbents with higher affinity for CFX
Regeneration	Limited regeneration cycles reported	Improve regeneration efficiency and long-term stability
Scale-up	Predominantly batch studies	Pilot-scale and continuous fixed-bed investigations
Dynamic modelling	Existing models poorly predict early breakthrough	Develop models incorporating mass transfer and axial dispersion

Concluding remark

The presence of ciprofloxacin (CFX) in water bodies poses potential toxicity and antibiotic resistance towards the microorganisms. Various adsorbents have demonstrated promising removal performance. However, adsorption efficiency lies upon the interplay of inherent chemistry of CFX, physicochemical properties of adsorbent and operational conditions. Despite these advances, several challenges remain before adsorption can be widely implemented for practical water treatment. Future research should focus on developing sustainable and low-cost adsorbents, evaluating their performance under realistic wastewater conditions, and improving their selectivity, regeneration efficiency, and long-term stability. Furthermore, more robust dynamic adsorption models that accurately account for mass transfer and breakthrough behavior are needed to optimize continuous adsorption systems and facilitate large-scale applications.

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Conflicts of interest

The authors declare no conflict of interest.

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