



Biowaste-derived magnetite biochar for adsorption of norfloxacin from water samples

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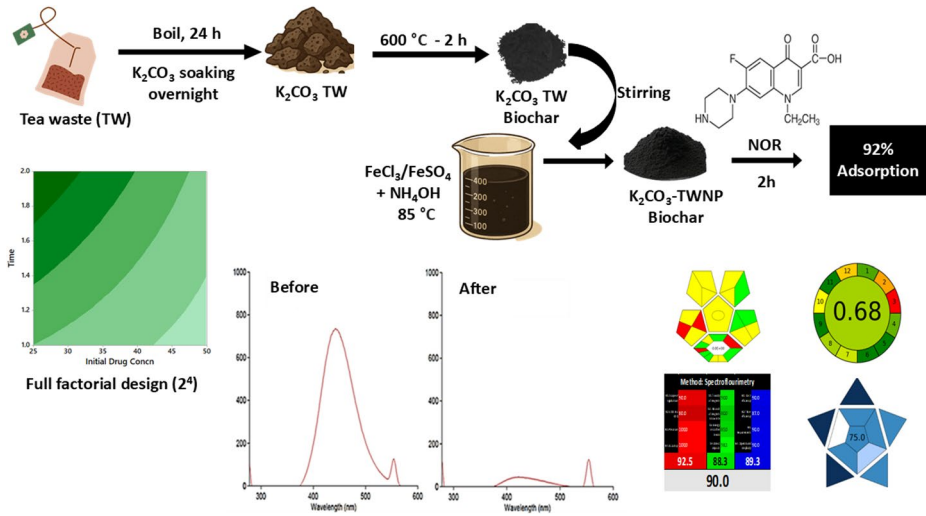
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Abstract

Pharmaceutical residues in water resources pose serious risks to human health and aquatic ecosystems. Herein, an environmentally friendly magnetite-loaded biochar was synthesized from K_2CO_3 activated tea waste and evaluated for the adsorptive removal of norfloxacin, a widely used fluoroquinolone antibiotic, from water samples. This study combines novel K_2CO_3 activated tea waste with the facile separation of magnetite adsorbent. The synthesized nanoparticles were characterized using transmission electron microscopy, Fourier transform infrared spectroscopy, X-ray diffraction, Brunauer-Emmett-Teller and dynamic light scattering. A validated spectrofluorimetric method has been developed for monitoring norfloxacin concentration throughout the treatment protocol. The effects of solution pH, adsorbent dosage, initial norfloxacin concentration, and contact time on adsorption efficiency were systematically investigated using a 2^4 full factorial experimental design. Initial drug concentration was identified as the most significant factor, followed by contact time. Under optimized conditions, a removal efficiency of $92 \pm 0.41\%$ was achieved for $25 \mu\text{g mL}^{-1}$ norfloxacin within 2 h at pH 5. Adsorption kinetics and isotherm models were subsequently evaluated to elucidate the adsorption behavior. The adsorbent exhibited a maximum adsorption capacity of 136.99 mg g^{-1} . The reusability of these nanoparticles has been investigated for six treatment cycles, demonstrating the efficacy of the adsorbent after several uses. Furthermore, the applicability of the developed method was validated by screening and analyzing real water samples, including Nile River and tap water. The greenness and whiteness levels of the proposed method were assessed using Complex GAPI, AGREE, BAGI, and WAC tools. These findings demonstrate the potential of converting agricultural waste into a reusable adsorbent for facile and cost-effective treatment of antibiotic-contaminated water.

Extended author information available on the last page of the article

Graphical abstract



Keywords K_2CO_3 -activated biochar · Tea waste · Norfloxacin removal · Biochar-based adsorbents · Magnetite nanoparticles · Greenness assessment · Pharmaceutical wastewater treatment

1 Introduction

Analytical techniques have detected different organic pollutants in water resources that pose harmful concerns to aquatic systems and humans (Rodríguez-Serin et al., 2022). The detection of pharmaceutical compounds in aquatic environments is attributed to industrial effluents as well as hospital and domestic discharges. Such contamination, especially with antibiotics, raises the need for effective water treatment to guard against their possible toxic effect (Massima Mouele et al., 2021). Antibiotics in wastewater are partially removed through processes such as adsorption onto sludge, transformation during treatment, and biodegradation. Nevertheless, conventional wastewater treatment methods remain insufficient for the complete elimination of these compounds, resulting in the persistence of antibiotics in treated effluents and their accumulation in sludge. Furthermore, some transformation products retain biological activity, emphasizing the critical role of wastewater treatment systems in controlling both the removal efficiency and redistribution pathways of antibiotic contaminants (Sabri et al., 2020). The discharge of antibiotics into the environment may lead to antibiotic residues in water, which can disturb the normal flora in the human body and result in antibiotic-resistant bacteria (Lyu et al., 2017). Antibiotic resistance arises from genetic adaptations that enable bacteria to survive exposure to antimicrobial agents through the acquisition and expression of resistance genes. These genes facilitate several resistance mechanisms, including target-site modification, efflux pump activation, and enzymatic drug inactivation, thereby reducing antibiotic effective-

ness. The rapid dissemination of resistance genes has become a major environmental and public health concern (Elbaiony et al., 2025). Among the pharmaceutical compounds, Norfloxacin (NOR) is considered one of the most frequent compounds detected in water. NOR, chemically designated as 1-ethyl-6-fluoro-1,4-dihydro-4-oxo-7-(1-piperazinyl)-3-quinolinecarboxylic acid, is a first-generation fluoroquinolone antibiotic that has emerged as an environmental contaminant of concern due to its widespread use, persistence, and potential ecological impacts (Yiruhan et al., 2010). It is commonly used to treat prostatitis, urinary tract infections, and sexually transmitted diseases, and is also used as prophylaxis against cirrhosis and ascites. Several analytical techniques were reported for the analysis of fluoroquinolones in aqueous solutions and pharmaceutical preparations, such as spectrophotometry (Seifrtová et al., 2010), HPLC (Baeza et al., 2022), voltammetry (Raghavendra et al., 2010; Solangi et al., 2005), capillary electrophoresis (Springer et al., 2015) and spectrofluorometry (Yao et al., 2018).

Adsorption is considered an efficient method for treatment of wastewater from different pharmaceutical contaminants (Mansouri et al. 2021). Natural adsorbents such as clays, charcoal, green or black leaves, tea waste, fruit waste, rice husk, chitosan, and red mud are considered economic, eco-friendly, easily available, recyclable, and rich in chemical composition with a high surface area (Dehghani et al., 2023; El-Baz et al. 2020; Jianle Li et al. 2024). Tea waste is mainly obtained from the commercially available tea leaves and is used in several drug adsorption protocols (Patil et al. 2019). One of the most efficient green adsorbents is biochar or activated carbon in its form. Biochar is obtained from pyrolysis at a temperature range of 200–900°C (B. Li et al. 2022). Biochar has attracted considerable attention as a sustainable, cost-effective, and environmentally friendly adsorbent owing to its high efficiency in removing pharmaceutical pollutants from wastewater (Keerthan et al. 2020). Advanced biochar composites have emerged as highly efficient materials for environmental remediation owing to their remarkable physicochemical properties. The integration of biochar with metal oxides, nanomaterials, polymers, and organic coating enhances its surface area, functional groups, and surface reactivity, resulting in superior adsorption performance. These modifications enable the effective removal of various contaminants, including heavy metals and antibiotics, while also improving material stability and reusability. Consequently, engineered biochar composites represent a promising approach for wastewater treatment applications (Y. Yang et al. 2025). Literature reviews revealed different examples of biochar, such as K_2CO_3 biochar, NaOH biochar, paper sludge biochar, sugar cane biochar, nut shells biochar, and tree bark biochar (Mylavarapu et al. 2013). Tea waste was reported for the removal of different types of drugs from wastewater, such as phenylephrine hydrochloride (Omran et al. 2024), zidovudine (Mashoene et al. 2023), aspirin (Nordin et al. 2023), different metals, such as copper and nickel ions (Jean Claude et al. 2022), and iron and lead (Ahluwalia and Goyal 2005). Several investigations have been conducted examining the decrease of NOR from wastewater matrices utilizing a range of biochar-based adsorbent materials. According to the reported systems, different examples include biochar synthesized from waste disposable bamboo chopsticks (Ming Zhang et al. 2023a, b, c), as well as clay biochar materials fabricated from potato biomass (Y. Li et al. 2017), biochar-based sludge adsorbent (Medeiros et al. 2023), engineered biochar using physical or chemical activation to improve the properties of biochar (Mohammadian et al. 2023), magnetic biochar adsorbents derived from sewage sludge and woodchips

(Santhosh et al. 2020), biochar derived from the luffa sponge (Feng et al. 2018), amorphous TiO₂-modified biochar (Hu et al. 2024a), ethanol-combined ball milling biochar (S. Yang et al. 2024), sulfidated nanoscale zerovalent iron loaded nitrogen-doped biochar (Xu et al. 2024) and biochar from *Atropa belladonna* residue (Miaomiao Zhang et al. 2024a). Despite their promising adsorption performances, many of these biochar-based materials rely on precursors with limited availability or involve complex, time-consuming, and multi-step synthesis and modification procedures, which may hinder large scale implementation and sustainability. Biochar has been explored for the removal of pharmaceutical contaminants, however, studies employing K₂CO₃-activated tea waste derived biochar nanoparticles for NOR removal are still limited. Also, detailed investigations of the adsorption mechanism, supported by kinetic and isotherm modeling, have not been fully investigated. The research aimed to evaluate the adsorption performance of magnetite-activated tea waste biochar as a sustainable adsorbent for NOR removal from aqueous media. A full factorial experimental design was carried out to optimize the adsorption process and maximize removal efficiency. In addition, adsorption kinetics and isotherm models were investigated. The environmental sustainability of the proposed methodology was evaluated using multiple green analytical assessment tools, including the Analytical GREENness Metric Approach (AGREE), White Analytical Chemistry (WAC), Complex Green Analytical Procedure Index (Complex GAPI), and Blue Applicability Grade Index (BAGI).

2 Experimental methodology

2.1 Chemicals and reagents

NOR standard with purity of 100.10% ± 0.24 was supplied from EIPICO (Egypt). Phosphoric acid, methanol, and disodium hydrogen phosphate were purchased from Merck (Germany). All the mentioned reagents, including hydrochloric acid, ammonium hydroxide, potassium carbonate, ferric chloride, ferrous sulphate were obtained from El Nasr Pharmaceutical Chemicals (Egypt). The commercial tea used in this study was sourced from the local market (Lipton®). Double distilled water was utilized in all experimental procedures.

2.2 Instruments

A Cary Eclipse fluorescence spectrophotometer (Agilent Technologies, USA) was used. Bruker IFS 66 V (Germany) was utilized for Fourier transform infrared (FTIR) spectroscopy. A high-resolution transmission electron microscope (TEM 2100; JEOL, Japan) was used to perform a TEM study of the nano-adsorbent. Crystallographic characterization was applied via X-ray diffraction (XRD) employing a Bruker D8-Advance diffractometer (USA). The textural properties, encompassing pore structure characteristics and specific surface area, were proposed using a Quantachrome NOVA Touch 4LX analyzer (USA), with data interpretation through Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) models. Zetasizer Malvern Nano ZS-ZEN 3600 (UK) was used for zeta potential measurement. To analyze the experimental results, Minitab (version 16.1.1) was used.

2.3 Preparation of adsorbent

2.3.1 Standard solutions

Stock NOR solution was prepared at a concentration of $25 \mu\text{g mL}^{-1}$ in 0.02 M phosphate buffer at pH 5. Buffer solution was prepared by dissolving 2.72 g of potassium dihydrogen phosphate in 800 mL of distilled water, followed by adjustment of the pH to 5 through dropwise addition of 0.1 M sodium hydroxide solution, and diluting to a final volume of 1000 mL with distilled water (Hulanicki & Głab, 2004). Appropriate aliquots of NOR stock solution were subsequently diluted with the same buffer to obtain a working solution of $2.5 \mu\text{g mL}^{-1}$.

2.3.2 Tea waste (TW)

Tea waste was prepared according to reported literature (Fan & Zhang, 2021; Kumbhar et al., 2022) with minor modifications. Briefly, 100 g of tea leaves were boiled in 500 mL water for 24 h, instead of being directly washed with distilled water. After boiling, the material was thoroughly washed with distilled water for at least three cycles, then dried in oven at 100°C for 24 h. The dried tea waste was ground into a fine powder for further use.

2.3.3 K_2CO_3 modified tea waste (K_2CO_3 -TWBC)

A quantity of 40 g of the previously prepared tea waste was immersed in a 10%w/v potassium carbonate (K_2CO_3) solution for 24 h. Then, it was rinsed with distilled water, followed by three successive washing cycles to remove any residual chemical particles, followed by oven drying at 100°C for a duration of 24 h. The dried particles were added to a high furnace oven at 600°C for 2 h to obtain activated carbon or biochar. The obtained particles were cooled before use.

2.3.4 K_2CO_3 biochar modified tea waste activated with magnetite nanoparticles (K_2CO_3 -TWBC-NP)

Five grams of ferrous sulfate and twelve grams of ferric chloride were dissolved in 200 mL of deionized water under heating at 85°C and vigorous stirring. Ammonium hydroxide was then added dropwise with stirring for 30 min until complete precipitation was observed. Subsequently, 5 g of K_2CO_3 activated tea waste biochar was added, and stirring was continued for an additional 2 h to allow effective incorporation of magnetite nanoparticles. The resulting particles were collected by centrifugation and washed thoroughly with distilled water, followed by washing with methanol for three consecutive cycles. Finally, the product was dried using vacuum at 60°C for 1 h.

2.4 Characterization of adsorbent (K_2CO_3 -TWBC-NP)

The dimension and morphology of the K_2CO_3 biochar TWNP were presented by Transmission Electron Microscopy (TEM) analysis. The sample was added to a copper grid, dried, and then investigated at 200 kV. Zeta potential of the adsorbent was analyzed at different

pH media (from 3 to 10), where the prepared particles were analyzed using Dynamic Light Scattering (DLS) at fixed angle of 173° at 25°C . IR spectra of the adsorbent were examined in the range $500\text{--}4000\text{ cm}^{-1}$ using Fourier transform infrared spectroscopy. (Fahmy et al., 2024). The crystallographic structure of the synthesized material was determined by means of X-ray diffraction (XRD) analysis, and the surface characteristics were proposed through nitrogen adsorption–desorption isotherm measurements, and interpretation was done by utilizing the Brunauer–Emmett–Teller (BET) and Barrett–Joyner–Halenda (BJH) models.

2.5 Spectrofluorometric method development

Monitoring of NOR concentration during treatment was performed using a spectrofluorometric method with excitation wavelength at 277 nm and emission wavelength at 443 nm. For the construction of a calibration curve, seven different NOR concentrations in phosphate buffer pH 5, from 0.1 to $2.5\ \mu\text{g mL}^{-1}$, were measured. Regression equation was calculated and used to monitor residual NOR concentrations. Validation of the spectrofluorometric method was carried out according to the ICH guidelines by calculating the following parameters: linearity, accuracy, interday precision and intraday precision, limit of detection, and limit of quantification (ICH guideline, 2023).

2.6 Preparation of treated samples

NOR ($25\ \mu\text{g mL}^{-1}$) was added into a beaker, followed by 0.5 g of $\text{K}_2\text{CO}_3\text{-TWBC-NP}$, and the solution was stirred for 2 h, then filtered to remove traces of adsorbent. Different aliquots from the resulting solution were quantitatively transferred into a series of 10-mL volumetric flasks so that the final concentration of NOR ranges from 0.1 to $2.5\ \mu\text{g mL}^{-1}$. Finally, the adsorption percentage was determined by the developed spectrofluorometric method. All experiments were carried out in triplicate, and the results were presented as mean \pm standard deviation.

2.7 Experimental design

The influence of operational parameters, such as pH, initial NOR concentration, contact time, and biochar loading, was systematically investigated. As follows in Table 1, two levels were applied to each of the four mentioned factors for a full factorial design (2^4), designated as high (+1) and low (-1). Accordingly, as illustrated in Table 2, a total of sixteen experimental runs were applied. All experimental procedures were performed at ambient room temperature.

Table 1 Actual factors and the levels used in a two-level full factorial design experiment

Factor Name	Factor Code	Low level	High level
pH	A	5	7
Time (h)	B	1	2
Adsorbent amount (g)	C	0.5	1
Initial drug concentration ($\mu\text{g mL}^{-1}$)	D	25	50

Table 2 Two-level full factorial design experiment

Run No.	Factor code (A)*	Factor code (B)*	Factor code (C)*	Factor code (D)*	% adsorption
1	+1	-1	+1	+1	65.00±1.24
2	-1	+1	+1	-1	87.00±0.87
3	+1	-1	-1	+1	68.00±1.56
4	-1	-1	-1	+1	68.59±0.45
5	-1	+1	+1	+1	77.20±1.78
6	+1	+1	+1	-1	86.06±0.92
7	+1	+1	-1	+1	61.37±1.63
8	-1	-1	+1	-1	77.89±0.38
9	-1	+1	-1	+1	74.22±1.45
10	+1	-1	+1	-1	70.08±0.71
11	+1	-1	-1	-1	74.18±1.82
12	+1	+1	-1	-1	88.48±0.54
13	+1	+1	+1	+1	64.14±1.33
14	-1	-1	+1	+1	63.37±1.67
15	-1	-1	-1	-1	75.79±0.98
16	-1	+1	-1	-1	92.21±0.41

* A=pH, B=Time, C= initial drug concentration and D=adsorbent amount

2.8 Preparation of real water samples from the Nile River and tap water

Tap water samples were collected from various locations across Cairo, then pooled and filtered using 0.45 μm membrane filter. After confirming that NOR was not present, samples were spiked with NOR 25 $\mu\text{g mL}^{-1}$ at pH 5. To this solution, 0.5 g of the adsorbent was added, and the mixture was stirred for 2 h at pH 5, under optimum conditions. The solution was filtered and measured. Surface water samples were collected from different locations in Cairo along the Nile River using clean plastic bottles and gloves, following water sampling protocols (Gad et al., 2022). The uncapped bottle was held upside down near the water's edge, filled, then sealed and labeled with the collection date, time, and location. To prevent contamination, the samples were stored in a cooler until received in the laboratory, where they were processed using the same procedure previously described for tap water samples. All experiments were carried out in triplicate, and the results were presented as mean±standard deviation.

2.9 Kinetics studies

An initial concentration of 25 $\mu\text{g mL}^{-1}$ of NOR was applied in contact with a fixed quantity of adsorbent (0.5 g) over a series of different time intervals, specifically at 0, 20, 40, 60, 80, 100, and 120 min, to evaluate the effect of contact time on adsorption efficiency. The resulting solutions were filtered and diluted with phosphate buffer to obtain a final concentration of 2.5 $\mu\text{g mL}^{-1}$. The decrease in the remaining NOR concentrations over time was monitored by the developed spectrofluorometric method.

2.10 Adsorption isotherms

Aliquots of (25, 50, 75, 100, and 125 $\mu\text{g mL}^{-1}$ at pH 5.0) were maintained in contact with a constant amount of adsorbent (0.5 g) in a series of beakers for 2 h. The solutions were

filtered and diluted with phosphate buffer to obtain final concentrations of 0.1 to 2.5 $\mu\text{g mL}^{-1}$. Finally, the final concentrations were analyzed by the developed spectrofluorometric method.

3 Results and discussion

Green adsorbents are of low cost, wide available, can be easily of regenerated, and have high adsorption capacity. Among these, tea waste was selected due to its economic and sustainable nature, large-scale availability, and proven effectiveness as an adsorbent (Muzammal et al., 2024). The use of activated tea-waste-derived biochar was favored over directly activated tea waste due to the fundamental physicochemical advantages imparted by the carbonization process. Conversion of tea waste into biochar produces a stable aromatic carbon matrix with enhanced structural integrity, controlled porosity, and improved surface homogeneity, which are essential for efficient and reproducible adsorption processes. In this study, tea-waste-derived biochar was modified with potassium carbonate and magnetite nanoparticles to enhance its adsorption properties for the removal of NOR from water sources. The proposed modification approach proved to be time and resource-efficient, yielding an adsorbent capable of achieving significant NOR removal under optimized conditions.

3.1 Preparation of adsorbent

The boiling step of tea waste was essential, as it promoted the removal of tannins, polyphenols, and other soluble organic constituents. This treatment reduced coloration and eliminated potential interfering species, thereby improving adsorption efficiency and minimizing interference in the spectrofluorimetric analysis. Biochar is considered a porous granular solid material having a large surface and rich in surface functional groups. By comparing conventional tea waste with chemically modified biochar, the activated carbon source generally has higher adsorption capacity, with functional groups variation. Carbonization also removed soluble organic components such as sugars, proteins, and polyphenols, thereby minimizing leaching and secondary contamination during water treatment. In addition, the carbon-rich surface of biochar provided higher π -electron density and well-defined oxygen-containing functional groups, promoting stronger interactions with organic pollutants, including fluoroquinolone antibiotics. These characteristics further enabled effective anchoring and dispersion of magnetite nanoparticles, enhancing adsorbent stability and reusability. In contrast, directly activated tea waste lacks sufficient mechanical and chemical stability, exhibits higher variability, and shows limited reusability, making tea waste derived biochar a more suitable and sustainable adsorbent platform.

Biochar has different adsorption mechanisms such as electrostatic interactions, ion exchange, surface adsorption, precipitation, and pore filling. At 600 °C, K_2CO_3 could promote the carbonation reaction and accelerate the dehydrogenation process, yielding a porous biochar. K_2CO_3 activation enhances surface functional groups, such as hydroxyl, carboxyl, and carbonyl groups. These groups can engage in various interactions with adsorbates, including π - π electron donor-acceptor interactions, hydrogen bonding, electrostatic attraction. In addition, the aromatic and graphitic structures within the biochar matrix contribute to hydrophobic interactions, aiding in the adsorption of organic molecules (Ganguly et al.,

2020). The selection of 600 °C for a relatively short duration of 2 h represents a balanced approach between achieving desirable material properties and limiting energy consumption. The use of high temperature has a significant enhancement in physicochemical properties, including surface area, pore development, and availability of active sites, all of which are critical for efficient pollutant removal. Despite the associated economic and environmental costs, the use of tea waste as a low-cost and abundant waste reduces overall material expenses. Moreover, the adsorption efficiency and reusability of the produced biochar contribute to lowering the total treatment cost compared to conventional methods.

3.2 Characterization of the adsorbent

In general, the tea biochar surface has a variety of functional groups, allowing its activity as an adsorbent (Aboulsoud, 2024). FT-IR analysis of tea waste revealed a broad absorption band around 3438 cm^{-1} , which points to the existence of hydroxyl groups. Signals found in the $2925\text{--}2853\text{ cm}^{-1}$ range were likely due to the stretching of aliphatic C–H bonds. A set of peaks between 1620 and 1640 cm^{-1} suggested the presence of carbonyl groups, typically found in amide structures. Meanwhile, peaks at 1549 and 1525 cm^{-1} were associated with secondary amines and additional C=O vibrations. The band at 1448 cm^{-1} corresponded to aromatic C–H bending, while the one at 1370 cm^{-1} was linked to bending of methyl groups. Other notable features included a peak at 1238 cm^{-1} , which was attributed to SO_3 group stretching, one at 1146 cm^{-1} associated with C–O–C bonds in polysaccharides, and another at 1037 cm^{-1} indicating C–O–H stretching. When comparing the spectra, it was clear that K_2CO_3 increased the probability of hydrogen bond formation with nanoparticles. Additionally, a peak at 600 cm^{-1} was observed and interpreted as the Fe–O band (Fig. 1). FTIR spectra were also obtained before and after NOR adsorption. The O–H/N–H stretching band shifted from 3402 cm^{-1} to 3412 cm^{-1} with noticeable broadening after adsorption, indicating hydrogen bond formation between surface hydroxyl groups and the carbonyl oxygen of NOR carboxylate group (Fig. 1). A pronounced shift of the C=C/C=N aromatic stretching band from 1584 to 1626 cm^{-1} , accompanied by newly appearing aromatic C–H bending peaks at 882 and 794 cm^{-1} , provided evidence of $\pi\text{--}\pi$ stacking interactions between the electron-rich biochar surface and the electron-deficient quinolone ring of NOR. At the optimum adsorption pH of 5, NOR exists predominantly in its cationic form due to protonation of the piperazine nitrogen ($\text{pK}_{\text{a}1}=6.2$), while the $\text{K}_2\text{CO}_3\text{-TWBC-NP}$ surface carries a net negative charge as confirmed by zeta potential measurements, favoring electrostatic attraction. This is supported by the broadening of the C–O/C–N bending band at 1400 cm^{-1} and the appearance of N–H stretching peak at 3227 cm^{-1} attributable to the protonated piperazine --NH^+ of adsorbed NOR. Additionally, the downshift of the C=O stretching to 1694 cm^{-1} in the post adsorption spectrum indicated possible carboxylate coordination with iron sites on the magnetite nanoparticle surface. Also, the appearance of C–F stretching vibrations at $1057\text{--}1129\text{ cm}^{-1}$ indicated NOR retention on the adsorbent surface. Collectively, these spectral changes confirmed that NOR adsorption onto $\text{K}_2\text{CO}_3\text{-TWBC-NP}$ proceeded through multi-mechanism pathway involving hydrogen bonding, $\pi\text{--}\pi$ stacking, electrostatic attraction, and surface coordination with iron centers.

To investigate the surface morphology of ($\text{K}_2\text{CO}_3\text{-TWBC-NP}$) as an adsorbent, TEM pictures were taken (Fig. 2). The TEM images revealed a heterogeneous carbonaceous matrix decorated with Fe_3O_4 nanoparticles. The observed spherical composite nanopar-

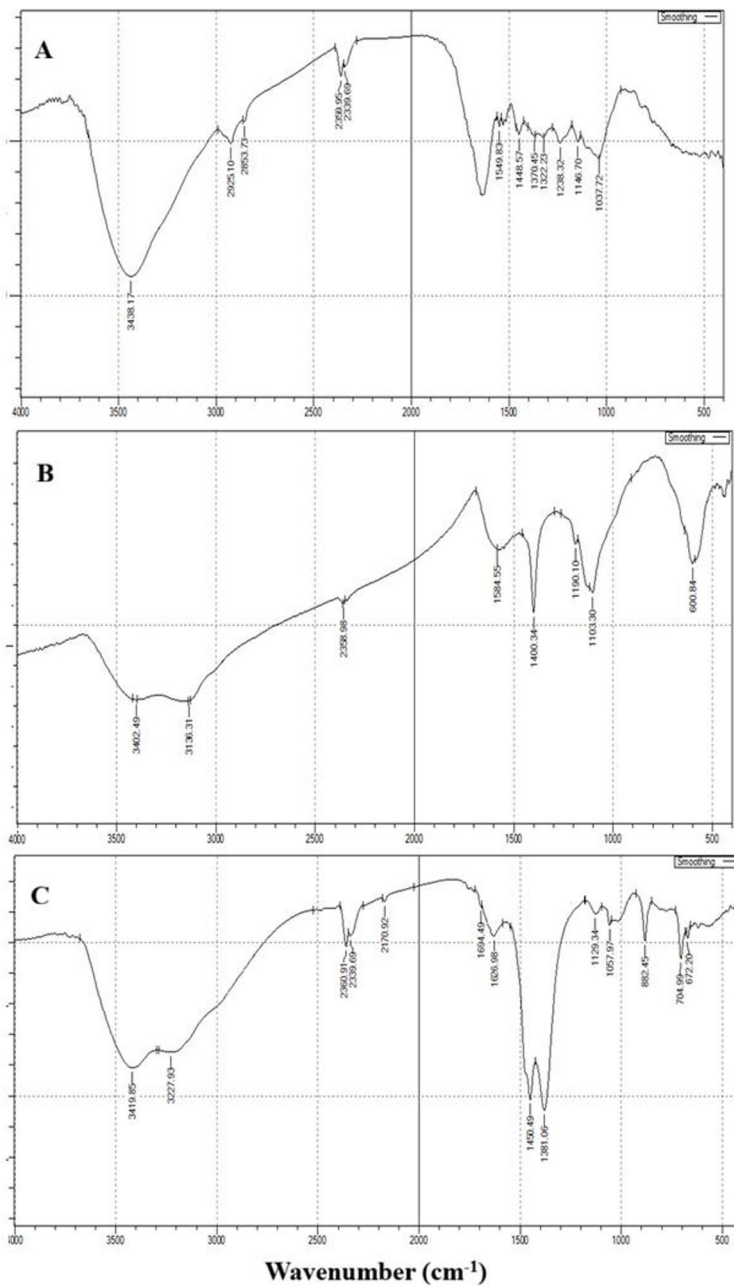


Fig. 1 IR spectra of (A) unmodified tea waste particles, (B) K₂CO₃ biochar modified tea waste activated with magnetite nanoparticles (K₂CO₃-TWBC-NP), (C) K₂CO₃-TWBC-NP after norfloxacin adsorption

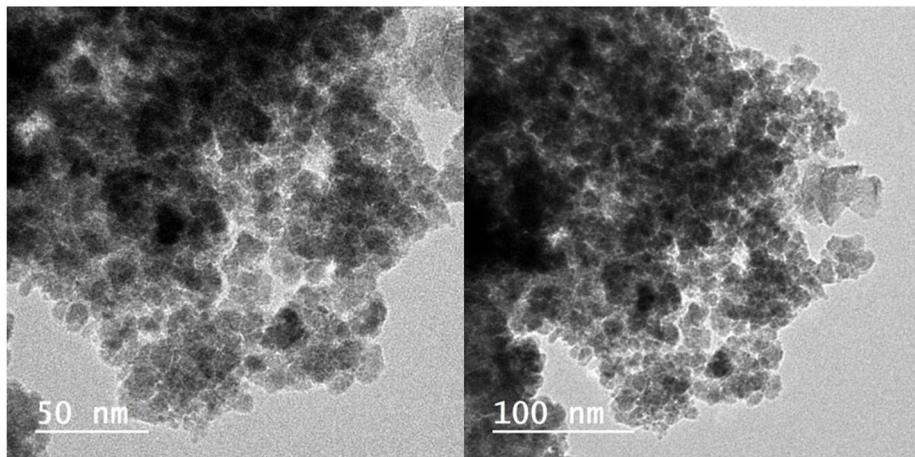
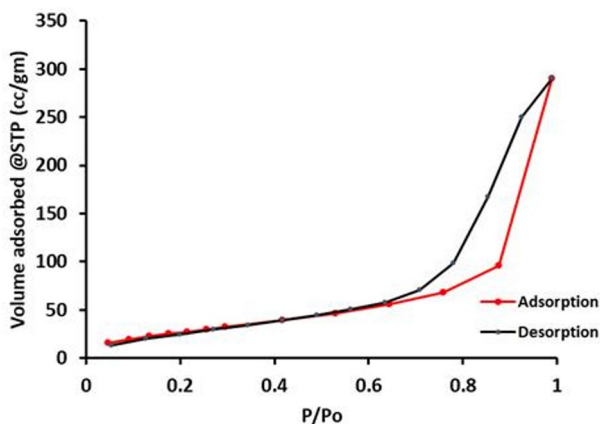


Fig. 2 TEM images of K_2CO_3 biochar modified tea waste activated with magnetite nanoparticles (K_2CO_3 -TWBC-NP)

Fig. 3 The obtained nitrogen adsorption/desorption isotherms for K_2CO_3 -TWBC-NP



ticles exhibited an average particle size of approximately 50 nm, with slight agglomeration attributed to magnetic interactions between magnetite particles. In addition, the loosely packed structure and lighter contrast regions suggest successful pore development. Nitrogen adsorption–desorption analysis revealed a type IV isotherm with an H3 hysteresis loop (Fig. 3), confirming the mesoporous nature of the adsorbent and the presence of slit-shaped pores typical of activated carbonaceous materials. The K_2CO_3 -modified biochar exhibited a specific surface area of $105.382 \text{ m}^2 \text{ g}^{-1}$, a pore radius of 1.921 nm, and a total pore volume of 0.425 cc g^{-1} . These textural properties were favorable for NOR adsorption because the mesoporous structure facilitated molecular diffusion and provided accessible adsorption sites. Considering that the molecular dimensions of NOR are substantially smaller than the measured pore diameter, the generated pores were sufficiently large to permit NOR transport into the internal pore network, thereby enhancing adsorption efficiency through both surface adsorption and pore-filling mechanisms. The observed porosity can be attributed to

K_2CO_3 activation, which promotes the development of pore structures through the thermal decomposition of the activating agent and the removal of volatile components during pyrolysis. This process increased surface accessibility and generated additional adsorption sites. Following Fe_3O_4 incorporation, some pore blockage might be expected due to nanoparticle deposition within the biochar matrix. However, the retention of a relatively high pore volume and mesoporous structure suggested that pore accessibility was largely preserved after modification. Therefore, the adsorption performance was likely governed by the combined effects of accessible porosity, surface functional groups, and Fe_3O_4 -associated active sites. This observation indicates that adsorption is not solely dependent on surface area but also on the synergistic contribution of pore structure, surface chemistry, and specific interactions between NOR molecules and the modified biochar surface.

The XRD pattern (Fig. 4) revealed a broad diffraction feature at $2\theta \sim 18.14^\circ$, characteristic of amorphous carbon, alongside distinct peaks at 30.20° , 35.57° , 43.19° , 57.14° , and 62.69° , corresponding to the (220), (311), (400), (511), and (440) planes of magnetite, respectively, confirming the successful incorporation of crystalline Fe_3O_4 nanoparticles within the biochar matrix (N. Zhang et al. 2023a, b, c). At pH 5, a zeta potential of -19.4 ± 0.46 was measured. Potential slightly increased to -19.4 ± 1.17 and -22.3 ± 0.8 when the pH was raised to 7 and 9, respectively. Although FTIR, BET, zeta potential, TEM, XRD provided evidence regarding the structural properties, surface functionality, porosity, and adsorption behavior of the developed adsorbent, these methods cannot directly identify changes in surface chemical states or elemental distributions after adsorption. Consequently, the proposed adsorption mechanisms involving electrostatic attraction, hydrogen bonding, π - π interactions, and pore-filling effects are inferred from indirect but complementary experimental evidence.

3.3 Spectrofluorometric analysis

Method validation and regression equation were presented in Table 3. Signal intensities measured across seven different concentrations exhibited good linearity, and a low limit of detection (LOD) was achieved ($0.05 \mu\text{g mL}^{-1}$). Figure 5 illustrated the comparison between the fluorometric analyses of standard and sample solutions following drug adsorption using tea waste biochar magnetite nanoparticles (K_2CO_3 -TWBC-NP). Additionally, Fig. S1 displayed an overlay of standard NOR concentrations employed for the construction of the cal-

Fig. 4 XRD pattern of K_2CO_3 -TWBC-NP

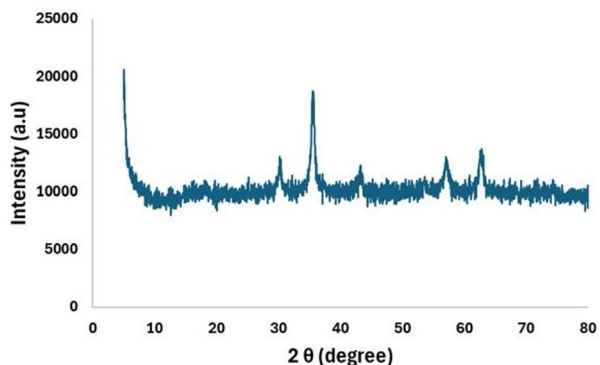


Table 3 Validation parameters of the proposed spectrofluorometric method

Validation parameters	Norfloxacin
Linearity ($\mu\text{g mL}^{-1}$)	0.1–2.5 $\mu\text{g mL}^{-1}$
Regression equation	$y=80.47x+96.447$ $r=0.9992$
Accuracy (mean \pm RSD) ¹	98.91 \pm 1.16
Inter-day precision (%RSD) ²	0.166
Intraday precision (%RSD) ³	0.205
LOD ($\mu\text{g mL}^{-1}$) ⁴	0.030
LOQ ($\mu\text{g mL}^{-1}$) ⁵	0.092

Average percentage recovery of nine determinations over three concentration levels

² The RSD of three different concentrations, 0.5, 1.0 and 2.0 $\mu\text{g mL}^{-1}$ of norfloxacin, determined in triplicate on different days

³ The RSD of three different concentrations: 0.5, 1.0 and 2.0 $\mu\text{g mL}^{-1}$ of norfloxacin determined in triplicate on the same day

⁴ Determined via calculations, 3.3 (SD of the response/slope)

⁵ Determined via calculations, 10 (SD of the response/slope)

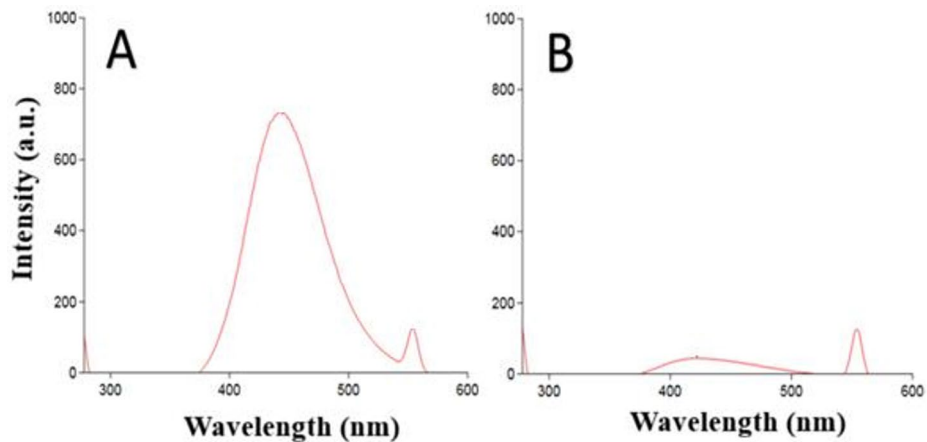


Fig. 5 Emission spectra of standard NOR (A) and (B) treated NOR sample (25 $\mu\text{g mL}^{-1}$) after adsorption using 0.5 g K_2CO_3 biochar modified tea waste activated with magnetite nanoparticles (K_2CO_3 -TWBC-NP) at pH 5 (excitation at 277 nm and emission at 443 nm)

ibration curve. The spectrofluorometric determination of NOR offered several advantages over HPLC, particularly in terms of sensitivity, cost, and environmental impact. Owing to the native fluorescence of NOR, spectrofluorometry provided high sensitivity, enabling detection at low concentrations without complex sample preparation. The method required minimal solvent consumption, in contrast to HPLC, which relies heavily on organic solvents, thereby enhancing its greenness and reducing environmental burden. Additionally, spectrofluorometric analysis involved lower operational and instrument costs and significantly shorter analysis time, making it more suitable for routine and high-throughput applications.

3.4 Experimental design studies

All adsorption experiments were conducted in triplicate under identical experimental conditions. The reported results were expressed as mean \pm standard deviation. The standard deviation was calculated from the replicate measurements and used to assess the repeatability of the experimental procedure. The relatively low standard deviation values obtained throughout the study indicate satisfactory experimental precision and reproducibility. Statistical analyses were performed using the average values obtained from the replicate experiments. The levels studied for the amount of adsorbent, contact time, pH, and initial concentration were used according to the previous literature. (Madikizela & Pakade, 2023). A full factorial design was employed to evaluate the individual and interactive effects of the selected factors and to identify the optimal experimental conditions for NOR adsorption, as shown in Table 1. Samples were analyzed by the developed spectrofluorometric assay, with the percentage removal values mentioned in Table 2. Full factorial design analysis was applied at a 95% confidence interval ($p < 0.05$), with NOR adsorption percentage as the designated response variable. A Pareto chart was constructed and utilized to quantitatively assess the relative significance and magnitude of influence of each factor as well as their interactive effects on the adsorption response. The direction and significance of these effects were illustrated using a normal probability plot of standardized effects. Among the variables studied, initial concentration of NOR showed the most significant impact, exhibiting a negative effect, indicating that higher adsorption efficiency was achieved at lower NOR concentrations (Cheikh et al., 2023). Contact time was identified as a statistically significant parameter exerting a positive effect on the adsorption response ($p < 0.05$), showing that a marked improvement in NOR removal efficiency at elevated levels of these variables, as shown in Fig. 6. The Pareto chart revealed that initial concentration and contact time were the only statistically significant factors influencing NOR adsorption, whereas the interaction terms did not exceed the significance threshold. Contact time represented the second most influential factor, reflecting the importance of mass transfer and the progressive occupation of active adsorption sites until equilibrium is achieved. In contrast, the interaction terms were statistically insignificant, indicating that the effects of contact time, and adsorbent dosage were largely independent within the experimental range investigated. From an environmental perspective, these findings imply that optimization of NOR removal can be effectively

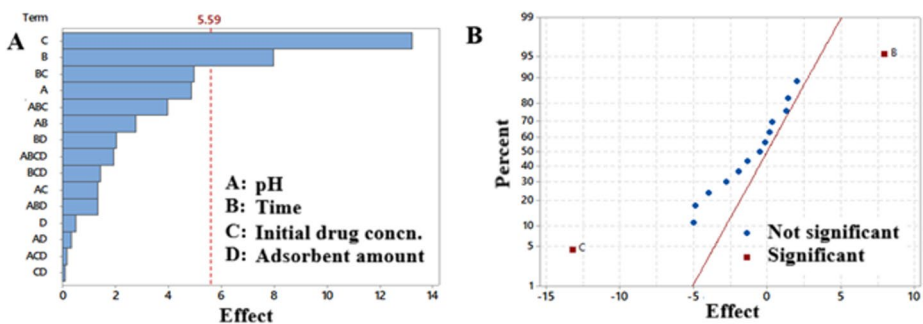


Fig. 6 (A) Pareto chart of the standardized effects of single and interaction factors on NOR adsorption. (B) Normal plot of the standardized effects of single and interaction factors on NOR adsorption

achieved by controlling NOR concentrations and ensuring sufficient contact time, while the remaining variables exert comparatively smaller effects under the studied conditions.

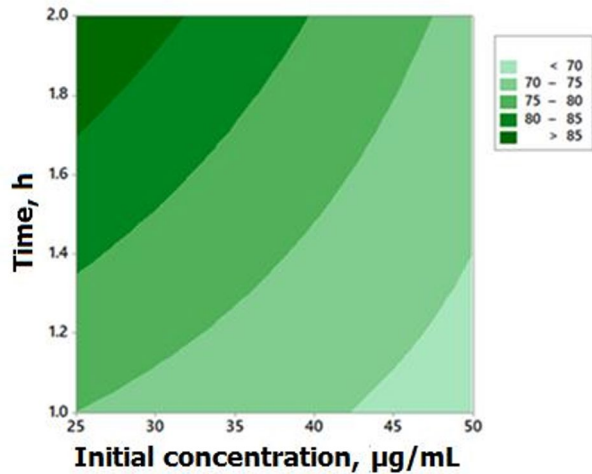
The mathematical regression equation applied from the experimental design to make a summary and describe the relationship between the parameters investigated and the adsorption response was expressed as follows:

$$Y = -59.41 + 18.19 A + 103.0 B + 2.365 C + 271.6 D - 12.20 AB - 0.2936 AC - 43.03 AD - 1.620 BC - 179.7 BD - 6.434 CD + 0.1456 ABC + 28.41 ABD + 0.9536 ACD + 4.165 BCD - 0.6168 ABCD$$

where $Y = \% \text{ Adsorption}$, $A = \text{pH}$, $B = \text{Time}$, $C = \text{initial drug concentration}$ and $D = \text{adsorbent amount}$. ANOVA was carried out, and the results were calculated as shown in Table S1. A lack of fit value of 0.521 was obtained, showing that it was not significant and that the model demonstrated acceptable predictive ability. Residual analysis was evaluated to verify the adequacy of the factorial design model (Fig. S2). The normal probability plot showed that the residuals closely followed a straight line, indicating approximate normality of the error distribution. Furthermore, the residuals versus fitted values plot exhibited a random scatter around zero with no distinctive trend. These findings demonstrate that the developed model can be used for interpretation and optimization purposes. In addition, increasing adsorbent loading to 1 mg mL^{-1} was found to be non-significant ($p > 0.05$). This could suggest a plateau effect for the biochar over a range of $0.5\text{--}1 \text{ mg mL}^{-1}$. Maximum adsorption efficiency ($92\% \pm 0.41$) was obtained at pH 5 in the presence of $25 \text{ } \mu\text{g mL}^{-1}$ NOR and 0.5 g of adsorbent in 2 h.

The adsorption behavior of NOR was strongly influenced by the combined effects of NOR ionization and the surface charge characteristics of the adsorbent. NOR possesses two dissociation constants ($\text{pK}_{a1} = 6.3$ and $\text{pK}_{a2} = 8.7$) (Fig. S3), resulting in different ionic forms depending on solution pH. At pH values below 6.3, NOR predominantly exists in its cationic form (NOR^+), whereas between pH 6.3 and 8.7 it mainly occurs as a zwitterionic species (NOR^\pm), and above pH 8.7 it is converted to its anionic form (NOR^-). Zeta potential measurements showed that the K_2CO_3 -modified tea waste biochar surface remained negatively charged throughout the investigated pH range, with values of -19.4 ± 0.46 , -19.4 ± 1.17 , and -22.3 ± 0.80 at pH 5, 7, and 9, respectively. Consequently, at pH 5 the electrostatic attraction between the positively charged NOR species and the negatively charged biochar surface favored adsorption, resulting in the highest removal efficiency ($92 \pm 0.41\%$). At pH 7, where NOR predominantly exists as a zwitterionic species, adsorption remained relatively high ($88 \pm 1.78\%$) due to the coexistence of electrostatic interactions, hydrogen bonding, π - π interactions, and pore-filling effects. These findings indicate that NOR adsorption is governed by the interplay between solution speciation and adsorbent surface charge, while non-electrostatic interactions such as hydrogen bonding, π - π electron donor-acceptor interactions, and pore-filling mechanisms further contribute to adsorption over the entire pH range studied. A contour plot was also generated to identify the optimal experimental conditions for NOR adsorption (Fig. 7). The highest removal efficiency was predicted at pH 5, with extended contact time beyond 1.5 h and lower initial concentrations of NOR. It should be noted that the usage of biological wastewater treatment, such as the anammox, which is the anaerobic ammonium oxidation process, could complement the adsorption system in treatment schemes. Anammox enables energy efficient nitrogen removal under anaerobic conditions, while the adsorption technique can remove the pharmaceutical contaminants. The elimination of antibiotics by the adsorption technique may reduce their inhibitory effects on anammox bacteria, resulting in a good enhancement of

Fig. 7 Contour plot for NOR initial concentrations versus time (hold values: pH 5, adsorbent loading of 0.5 mg mL^{-1})



performance. This integration shows a promising and sustainable approach for wastewater treatment (Fu et al., 2025).

3.5 Kinetics studies

The adsorption kinetic data were analyzed using the two most widely employed kinetic models, namely pseudo first order and pseudo second-order models. The pseudo-first-order kinetic model is expressed as follows: $\ln(Q_e - Q_t) = \ln Q_e - K_L t$, as Q_t is considered the quantity of NOR adsorbed per unit mass of adsorbent at a given contact time t (min), K_L is the rate constant (min^{-1}), and Q_e is the quantity of adsorbate (NOR) adsorbed per unit mass of adsorbent (biochar) at equilibrium (mg g^{-1}). The second model pseudo-second-order equation can be demonstrated as: $t / Q_t = 1 / K_2 Q_e^2 + t / Q_e = 1 / V_0 + t / Q_e$, where k_2 ($\text{g mg}^{-1} \text{min}^{-1}$) is the rate constant, and V_0 ($\text{mg g}^{-1} \text{min}^{-1}$) is the initial adsorption rate. The adsorption kinetic data were fitted to the pseudo first order and pseudo second order models to investigate the adsorption mechanism. The adsorption of NOR occurred rapidly at the beginning of the kinetics study due to the presence of several binding sites in biochar at the initial stage, and then the process of adsorption reached a plateau by increasing the adsorption time to 140 min. The suitability of the adsorption kinetic models was evaluated based on the correlation coefficient (R^2), as shown in Fig. 8. The pseudo second order model exhibited an R^2 value greater than 0.99, indicating an excellent agreement between the experimental and predicted data. This result suggested that the pseudo second order model provided a more accurate and reliable description of the adsorption process than the pseudo first order model. The superior fitting performance of the pseudo second order model indicated that it was the most appropriate model for describing the adsorption kinetics under the experimental conditions. Moreover, the calculated adsorption capacity (Q_e) from the pseudo second order model closely aligned with the experimental values (Q_{exp}), further confirming the model's suitability for describing NOR adsorption behavior as shown in Table 4. These findings indicated that the adsorption process may be largely influenced by chemisorption mechanisms, involving valence-force interactions arising from electron sharing or exchange between NOR molecules and functional groups present on the adsorbent surface (Zhang et

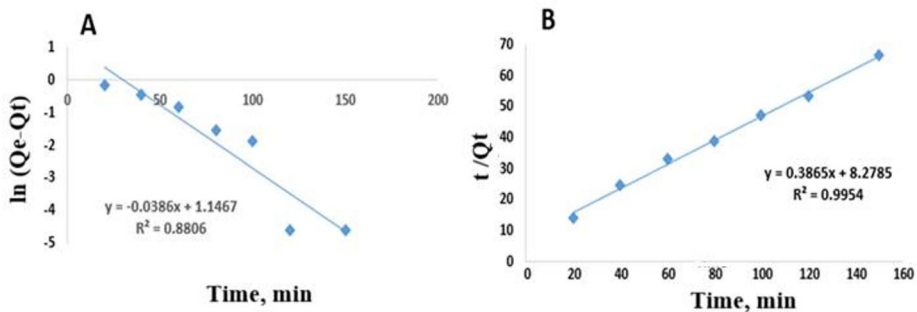


Fig. 8 NOR kinetics model (A) First order kinetics and (B) Second order kinetics

Table 4 Parameters for kinetic models of norfloxacin adsorption

Concentration ($\mu\text{g mL}^{-1}$)	Exp. Data	Pseudo First kinetics			Pseudo-second kinetics		
		Q_e	K_1	Q_e	R^2	K_2	Q_e
2.5	2.265	0.01675	3.1477	0.8806	0.50432	0.12079	0.9954

al. 2023a, b, c). However, fitting to this model does not confirm the exact nature of these interactions, as some physical adsorption processes can also follow pseudo second order kinetics (Rahman et al. 2004).

3.6 Adsorption isotherms

The adsorption isotherms study gives data about the maximum adsorption capacity of biochar and the molecular adsorption performance (Jie Li et al., 2018). Percentage removal does not fully reflect the performance of an adsorbent due to its highly dependence on the experimental parameters such as the initial concentration, the dose of adsorbent. However, the adsorption capacity (q_e and q_m) provides a consistent measurement of adsorbent efficiency. The most applied isotherm equations are Langmuir and Freundlich models. To determine the most suitable model for NOR adsorption process for modified K_2CO_3 -TWBC-NP, the following linear forms of these equilibrium isotherms equations should be measured: $C_e q_e = 1 q_m K_L + C_e q_m$ (1), $\text{Log } q_e = \text{Log } K_F + 1/n \text{ Log } C_e$ (2) where q_e (mg/ g) is the quantity of drug adsorbed, C_e (mg/L) is the equilibrium drug concentration, q_m (mg/g) represents the maximum adsorption capacity, K_F and n are the Freundlich constants and K_L (L/mg) is the Langmuir constant. Models were modified to best fit the parameters. (Ishaq et al., 2024). The adsorption data for NOR on the surface of TWBC nanoparticles were analyzed using the linear forms of both Langmuir and Freundlich isotherm models at ambient temperature. The regression coefficients (R^2) obtained were 0.9519 for the Langmuir model and 0.9923 for the Freundlich model (Fig. 9). These values indicated that Freundlich isotherm provided a better fit for the experimental data. The high R^2 value, approaching unity, suggested a strong interaction between NOR and the surface of the TWBC nanoparticles, reflecting an effective adsorption process. The maximum adsorption capacity (q_m) of NOR onto K_2CO_3 -activated biochar reached 136.66 mg g^{-1} . The Freundlich constant K_F was found to be 103.15 L mg^{-1} , which indicated a high adsorption capacity of the adsorbent used with norfloxacin. The adsorption factor n was 2.36, which was in the favorable range, as the number of n

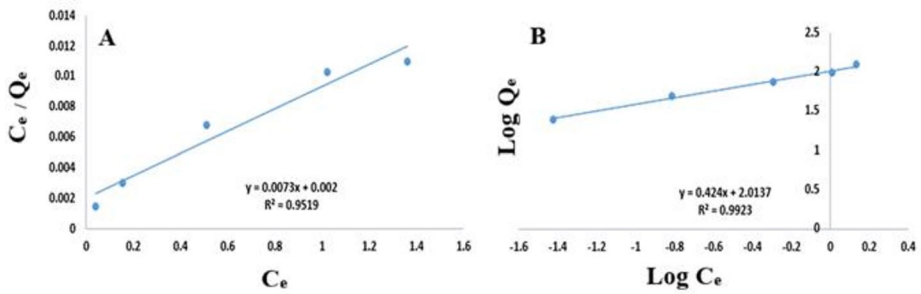


Fig. 9 Adsorption isotherm of NOR (A) Langmuir model (B) Freundlich model

Table 5 Comparison of recently reported literature for biochar adsorbents

No	Adsorbent	q_m (mg g^{-1})	Contact time	pH range	Regenera- tion cycles	Comment	Ref.
1	K_2CO_3 -TWBC-NP	136.9	120 min	5–7	6 cycles	facile preparation	This work
2	K_2CO_3 -activated lignin-based biochar	666.2	120 min	5–7	4–5 cycles	complex preparation	Miaomiao Zhang et al. 2024b
3	KOH sludge biochar	243	24 h	2–5	3 cycles	complex preparation	Ding et al., 2024)
4	Humic acid modified magnetic biochar	81.08	180 min	2–12	3 cycles	complex multi-step preparation	Zhao et al., 2019)
5	Coffee ground biochar	69.8	180 min	6	3 cycles	facile preparation	Nguyen et al., 2022
6	Coco peat biochar	95	90 min	7	3 cycles	facile preparation	Poddar et al., 2024
7	Amorphous TiO_2 - modified biochar	78.14	240 min	6	Not mentioned	sol-gel impregnation	Hu et al. 2024b)
8	Chemically aged bio- chars ($\text{H}_2\text{O}_2/\text{KMnO}_4$)	16–49	168 h	3–7	Not mentioned	simple aging, reduced efficiency in real water	Lu et al., 2024
9	Straw-derived biochar	30–40	180 min	5–9	2–3 cycles	Low capacity - weak reuse	X. Li et al. 2023
10	Biochar from waste disposable bamboo chopsticks	76.17	240 min	6	Not mentioned	moderate complexity involving washing waste chopsticks	Ming Zhang et al. 2023a, b, c
11	Biochar derived agri- cultural biomass (rice straw)	58.48	180 min	7–8	5 cycles	facile preparation - pyrolysis at 500 °C	(Phuong & Loc, 2022)

should be more than one and less than 10. This means a favorable adsorption on a heterogeneous surface. The correlation coefficient R^2 was 0.9923, which showed excellent fitting with experimental data to Freundlich model. Comparison with literature-reported methods demonstrated that the adsorption capacity obtained in this study was competitive among low-cost biochar-based adsorbents. As shown in Table 5, the developed adsorbent exhibited an adsorption capacity of 136.99 mg g^{-1} toward NOR, which is competitive with several previously reported biochar-based adsorbents. Although higher adsorption capacities have been reported for certain chemically engineered biochar, such materials often require more

complex synthesis procedures, extensive chemical modification, or higher preparation costs. In contrast, the present adsorbent was produced from abundant agricultural waste through a relatively straightforward preparation route and demonstrated efficient NOR removal within a moderate contact time (120 min), good performance within an environmentally relevant pH range (5–7). Therefore, the significance of the developed material lies in its balanced combination of adsorption performance, sustainability, ease of preparation, and practical applicability.

3.7 Reusability of K_2CO_3 -TWBC-NP

After a 2 h adsorption process using K_2CO_3 -TWBC-NP, the spent adsorbent was recovered and regenerated for subsequent reuse. The collected material was initially rinsed with distilled water (one cycle) to remove loosely bound residues, followed by three consecutive methanol washing cycles as a desorption step to eliminate retained NOR molecules. The regenerated adsorbent was then dried in an oven at 100 °C until complete dryness. The reusability of the adsorbent was assessed through consecutive adsorption–desorption cycles conducted under identical conditions. The fresh adsorbent initially achieved $92 \pm 0.41\%$ removal efficiency for $25 \mu\text{g mL}^{-1}$ NOR at pH 5 using 0.5 g adsorbent within 2 h under magnetic stirring. After adsorption, the suspension was filtered, and the residual NOR concentration was determined spectrofluorometrically. A slight reduction in the recovered amount of K_2CO_3 -TWBC-NP adsorbent was observed during successive regeneration cycles, with an estimated loss of approximately 2–5% per cycle over six adsorption–desorption runs due to particle loss during washing, filtration, and handling. The relatively low percentage loss indicates acceptable mechanical stability and structural integrity of the prepared magnetic biochar.

The adsorption efficiency decreased from 92% to $78 \pm 0.51\%$ after three regeneration cycles and further to $68 \pm 1.26\%$ after six cycles, corresponding to performance losses of approximately 14% and 24%, respectively. Although the adsorbent retained a considerable proportion of its initial adsorption capacity after repeated reuse, the decline indicates progressive deterioration of adsorption performance during successive regeneration cycles. This reduction may be attributed to gradual blockage or deactivation of active adsorption sites, minor structural alterations of the biochar surface, and unavoidable material loss during washing and recovery procedures. Therefore, the adsorbent still has an acceptable adsorption capacity after all these previous cycles, which shows a moderate reusability and a good potential for application under the optimized regeneration conditions (Fig S4). Although reusability experiments demonstrated that the adsorbent retained adsorption capability over adsorption–desorption cycles and exhibited relatively low material loss during regeneration, direct assessment of structural and chemical stability after repeated use was beyond the scope of this work. Consequently, potential changes in surface functional groups, crystallinity, pore structure, and iron release were not specifically evaluated.

3.8 Screening and application of water samples

3.8.1 Screening of water samples

Water samples collected from the Nile River and tap water were screened for the presence of NOR. Results showed no detectable peak at excitation 277 nm and emission 443 nm, indicating the absence of NOR in both samples (Fig. S5).

3.8.2 Application of real water sample and tap water sample

The treated Nile River and tap water samples were evaluated against a standard NOR solution. Before analysis, both water samples were pre-filtered to remove suspended solids. The applicability of the prepared adsorbent for water treatment was investigated using samples spiked with NOR at an initial concentration of $25 \mu\text{g mL}^{-1}$. Following adsorption, NOR removal concentrations reached $23.25 \pm 0.33 \mu\text{g mL}^{-1}$ for Nile River water and $22.75 \pm 0.39 \mu\text{g mL}^{-1}$ for tap water, corresponding to residual concentrations of $1.75 \pm 0.33 \mu\text{g mL}^{-1}$ and $2.25 \pm 0.39 \mu\text{g mL}^{-1}$, respectively. The obtained results demonstrated high adsorption efficiencies of $93 \pm 1.32\%$ and $91 \pm 1.57\%$ for Nile River (Fig. S6) and tap water samples (Fig. S7), respectively, confirming the effectiveness of the synthesized adsorbent for practical water treatment applications. The satisfactory adsorption observed in Nile River and tap water samples suggested that the prepared adsorbent maintained its affinity toward NOR in the presence of naturally occurring matrix constituents. Natural waters commonly contain dissolved inorganic ions (e.g., Ca^{2+} , Mg^{2+} , Na^+ , Cl^- , SO_4^{2-}), as well as dissolved organic matter, which may compete for adsorption sites or alter adsorbent surface properties. Despite these potential interferences, removal efficiencies exceeding 90% were achieved, indicating limited matrix effects under the investigated conditions. Nevertheless, the concentration and composition of dissolved ions and organic matter were not specifically characterized in this study; therefore, their individual contributions to adsorption behavior could not be quantified.

It should be noted that NOR concentrations employed in the present study were higher than those typically reported in environmental waters, where concentrations were commonly found in the ng L^{-1} to $\mu\text{g L}^{-1}$ range. These elevated concentrations represented a worst-case contamination scenario, allowing evaluation of adsorbent performance under demanding conditions. The ability of the prepared adsorbent to achieve high removal efficiencies at such elevated NOR levels suggests that the lower concentrations commonly encountered in natural waters are covered within the investigated adsorption range and would be expected to be removed effectively. However, adsorption behavior at environmentally relevant concentrations may differ due to reduced concentration gradients and adsorption-driving forces.

3.9 Assessment of the analytical method greenness profile

Green techniques show a framework for analyzing how materials and methods deal with eco-friendly practices, including waste reduction, safety, and energy use. In water treatment research, this evaluation helps to prevent contaminants from being introduced into the environment (Yin et al., 2024). This work uses green assessment to examine the sustainability of biochar-based nanomaterials employed in the removal of NOR from water samples. Thus, while greenness metrics suggest an environmentally friendly method, energy and solvent consumption must be considered when comparing with alternative adsorbents or scaling up the process. Greenness scores indicate moderate environmental impact, but performance remains the primary evaluation criterion. Regeneration solvents can have different environmental impacts due to their unwanted effects, such as corrosiveness, toxicity, and volatility. Also, organic solvents can lead to risks to air quality and aquatic life. The regeneration can lead to the generation of a second stream of waste that contains both the used solvent and desorbed pollutants. The use of greener and less hazardous regeneration solvents, together with optimization of regeneration conditions to minimize solvent consumption, is essential

for improving the overall sustainability of the adsorption process. Although the proposed method demonstrated favorable performance in the applied greenness assessment tools, the regeneration procedure required methanol as a desorption solvent, which introduces additional environmental and safety considerations. Therefore, the sustainability of the overall process should be assessed. On one hand, the adsorbent was prepared from low-cost tea waste and showed reusability over multiple adsorption–desorption cycles. On the other hand, solvent consumption during regeneration contributes to the environmental organic waste of the process. Consequently, the proposed system should be regarded as a promising and relatively sustainable approach rather than a completely green solution.

3.9.1 Green Analytical Procedure Index (GAPI)

GAPI uses five pentagrams to evaluate each step of the applied analytical method. The colors of pentagrams are used as a color code: green, yellow, and red. This color is defined as having low, medium, and high environmental impact. The GAPI tool was applied to assess the safety, health, and environmental impact of the analytical method, and the corresponding pentagrams summarize these green metrics. In the sample preparation and analysis, there were 7 green, 6 yellow, and 2 red. The red zone was for the transportation of the sample to the analysis site and the offline collection of the sample (Płotka-Wasyłka & Wojnowski, 2021). Figure 10A presented GAPI assessment, illustrating the safety, health, and environmental impacts of the analytical steps. The color-coded sections highlight variations in greenness, identifying both higher and lower impact stages, and thereby provide an overall environmental performance of the method. In the pre-analysis process, there are 5 green, 3 yellow, and 2 red. The red areas reflect the usage of heat and the appearance of vapor in the whole environment (Abd El-Fatah et al., 2024). GAPI results indicated generally acceptable environmental performance; however, some procedural steps exhibited moderate environmental impacts associated with operational requirements rather than the adsorbent synthesis process itself.

3.9.2 Analytical Greenness Metric Approach (AGREE)

AGREE is a visual and quantitative metric used to show whether the method is considered friendly to the environment or not, based on the twelve principles of Green Analytical Chemistry (Pena-Pereira et al., 2020). The greenness profile of the proposed method was assessed using the AGREE metric, yielding a final score of 0.68, which indicates reasonable and moderate adherence to the principles of Green Analytical Chemistry (Fig. 10B). This study showed good greenness scores according to standard metrics; it required furnace heating at 600 °C and multiple washing steps, which were energy and solvent intensive. These steps improved the adsorption performance of the biochar, but they reduced the overall sustainability of the method. The use of organic solvents partially reduced the overall sustainability of the method according to these limitations.

3.9.3 Blue applicability grade index (BAGI)

The pictogram available indicates dark blue color, which means excellent results; blue color indicates medium effects; light blue indicates poor method application, and white color indi-

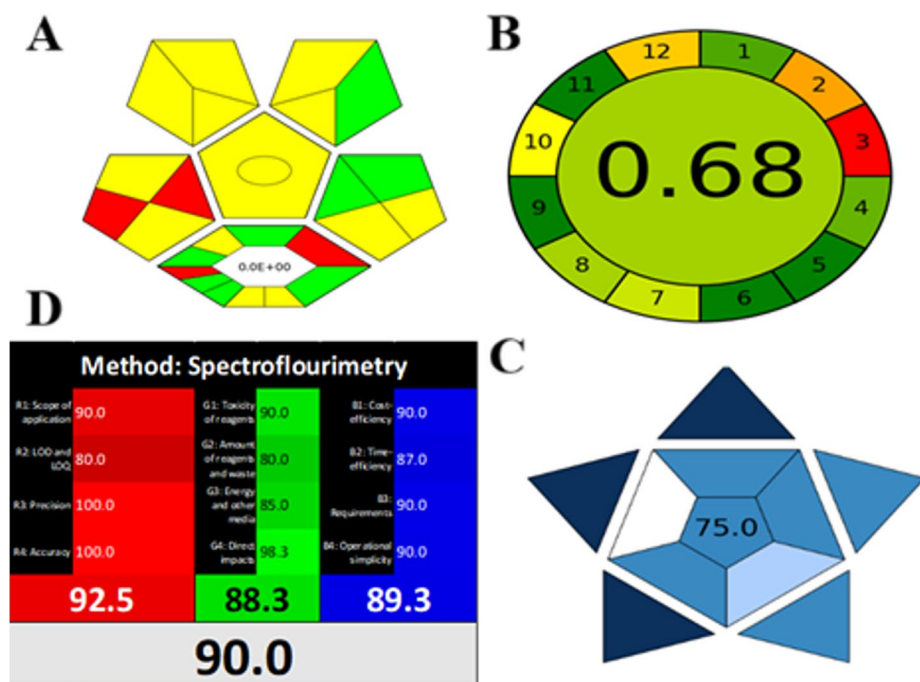


Fig. 10 Greenness and whiteness evaluation results of the developed method by (A) GAPI (B) AGREE (C) BAGI index and (D) WAC tool

cates no effect at all (Gałuszka et al., 2023). The number that is presented in the middle indicates the overall score of the applied analytical method. The score is out of 100 (Manousi et al., 2024). The developed method was more than 60, which indicated acceptable application of the used method (Fig. 10C).

3.9.4 White Analytical Chemistry (WAC)

The analytical method was evaluated using WAC, a technique that balances analytical performance, environmental impact, and practical applicability (Nowak et al., 2021). The method achieved a whiteness score of 90.0, indicating a good level of sustainability. The red section scored 92.5, reflecting a wide range of applicability of the proposed method with high accuracy and precision. The green section scored 88.3, which assures the alignment of the proposed method with the green chemistry principles. Finally, the blue section scored 89.3, reflecting the strong feasibility of the method application with good cost-effectiveness, time-efficiency, simple operation, and minimal instrument or facility requirements. Finally, the spectrofluorimetric method demonstrates a well-balanced technique across all three WAC dimensions (Fig. 10D). The WAC assessment highlighted the environmental and operational advantages of the proposed method, particularly the use of waste biomass as a precursor and its economical implementation. However, certain environmental concern remained, mainly arising from solvent consumption and the energy demand associated with thermal treatment.

4 Conclusion

A facile, and cost-effective K_2CO_3 -activated tea waste biochar incorporated with magnetite nanoparticles was synthesized, characterized, and used for the adsorptive removal of NOR from aqueous media. Tea waste biochar represented an alternative to conventionally activated carbon contributing to waste usage and reduced environmental pollution. The effects of experimental factors including pH, adsorbent dosage, contact time, and initial NOR concentration, were systematically optimized using a full factorial design. Adsorption behavior was described by kinetic and isotherm models, indicating favorable interactions between NOR molecules and the adsorbent surface, with a maximum adsorption capacity of 136.66 mg g^{-1} . Although the maximum adsorption capacity obtained in this study was lower than that reported for some highly engineered biochar adsorbents, the developed material offered several practical advantages, including the utilization of low-cost tea waste, a relatively simple synthesis procedure and good reusability. These characteristics make the proposed adsorbent a promising and sustainable candidate for the treatment of antibiotic contaminated water under practical operating conditions. The developed adsorbent demonstrated satisfactory regeneration capability over six regeneration cycles. Although a gradual decline in performance was observed, the material remained effective after repeated reuse, indicating practical potential for wastewater treatment applications. In addition, the greenness of the developed approach was assessed using Complex GAPI, AGREE, BAGI, and WAC tools, which revealed minimal environmental impact. Overall, the obtained results demonstrate that K_2CO_3 -TWBC-NP was an efficient, reusable, and environmentally sustainable adsorbent for pharmaceutical wastewater treatment. Future studies should investigate its large-scale application, long-term stability and alternative regeneration strategies, including lower toxicity solvents or solvent minimization, to further improve overall sustainability.

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Author contributions Lamis M. Fahmy: Writing – original draft, Methodology, Formal analysis, Data curation. Dalia Mohamed: Writing – review & editing, Visualization, Supervision, Conceptualization. Marianne Nebsen: Writing – review & editing, Visualization, Supervision, Conceptualization. Ahmed H. Nadim: Writing – review & editing, Validation, Investigation, Data curation.

Data availability The authors declare that the data supporting the findings of this study are available within the paper and its Supplementary Information files. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request.

Declarations

Ethical approval (Not applicable).

Conflict of interest The authors have no conflict of interest.

Competing interests The authors state that they have no known competing financial interests or personal ties that could have influenced the research presented in this study. There is no conflict of interest to declare.

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