



Design of Electrospun Hydrophobically Modified Polyacrylic acid Hydrogel Nanofibers and their Application for Removal of Ciprofloxacin

Gülmire İlyasoğlu¹ · Turdimuhammad Abdullah^{2,3,4,5} · Oguz Okay² · İsmail Koyuncu^{1,6}

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Abstract

The global water scarcity crisis has been exacerbated by the increasing demand for clean water and water pollution caused by the persistent release of pharmaceuticals such as ciprofloxacin, into water systems. The biodegradation and adsorption potential of CIP is crucial for its elimination in wastewater treatment systems. However conventional methods in wastewater treatment plant (WWTP) often struggle to efficiently eliminate of CIP from water due to its chemically stability and nonbiodegradability. Many researchers observed that CIP was not biodegraded even after 48 days in municipal WWTP, therefore no CIP removal occurred. The objective of this study was to investigate adsorption potential of CIP using a designed electrospun nanofiber. Within the scope of this purpose, we prepared electrospun acrylic acid (AAc)-based hydrogels modified with n-hexadecyl acrylate (C16A) for the first time and evaluated their efficacy in removing CIP from water. Our results show that the desired fiber size and surface smoothness can be obtained in the electrospun hydrogel containing 35 mol% of C16A. As a result, the AAc-based hydrogel nanofiber containing 35 mol% C16A exhibited superior adsorption properties. The adsorption efficiency of the hydrogel for CIP removal from aqueous medium was as high as 98% under equilibrium conditions. The adsorption process was found to follow the pseudo-second-order model, which suggests chemisorption as the dominant mechanism. Isothermal analysis showed that the adsorption fit well with the Langmuir model, suggesting single layer adsorption on a uniform surface. These results highlight the potential of AAc-based hydrogels for the sustainable removal of pharmaceuticals from wastewater, addressing a critical need in environmental contaminant management.

Gülmire İlyasoğlu and Turdimuhammad Abdullah have contributed equally.

✉ Gülmire İlyasoğlu
ilyasoglug@itu.edu.tr

✉ Oguz Okay
okay@itu.edu.tr

Turdimuhammad Abdullah
turdimuhammad.abdullah@izelkimya.com.tr

İsmail Koyuncu
koyuncu@itu.edu.tr

¹ National Research Center on Membrane Technologies, Istanbul Technical University, Istanbul 34467, Turkey

² Department of Chemistry, Istanbul Technical University, Istanbul 34467, Turkey

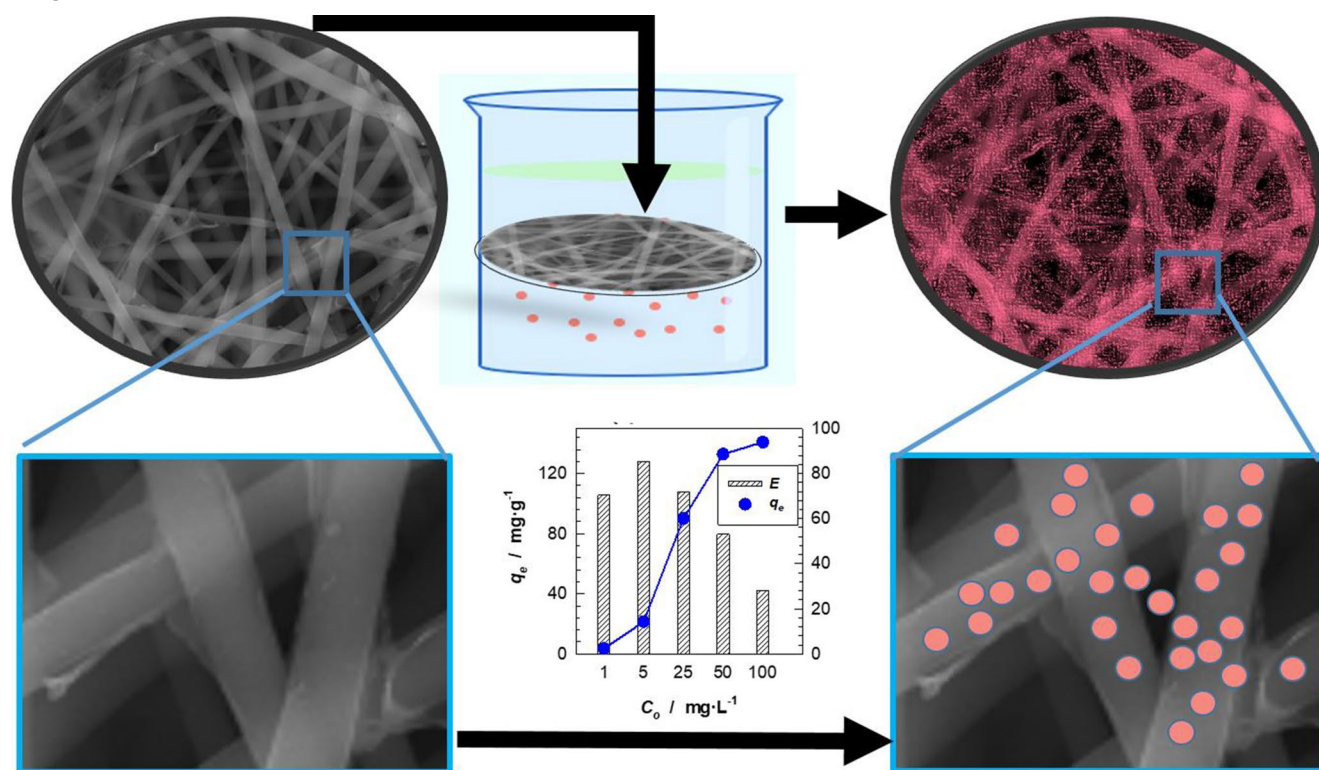
³ İzel Kimya Research and Development Center, Dilovası, Kocaeli, Turkey

⁴ Stem Cell and Tissue Engineering Application and Research Center, İstinye University, İstanbul, Turkey

⁵ Department of Metallurgical & Materials Engineering, Kocaeli University, Kocaeli, Turkey

⁶ Environmental Engineering Department, Istanbul Technical University, Istanbul 34469, Turkey

Graphical Abstract



Keywords Hydrogel · Nanofiber · Electrospinning · Adsorption · Emerging contaminant

Introduction

The demand for clean water is rapidly increasing worldwide, threatening to surpass the available 3% of fresh water as 97% of the Earth's water is salty seawater [1, 2]. The contamination of water resources by industrial activity has contributed to a global decline in freshwater availability. According to the forecast, more than 2/3 of the world's population may experience water scarcity by 2025^{2,3}. Consequently, research on sewage and wastewater treatment has become crucial due to contamination by a range of pollutants, including pathogens, heavy metals, and pharmaceuticals, which have recently emerged as a significant concern [4, 5]. Antibiotics, vital for treating bacterial infections, face challenges from their widespread use, particularly in densely populated urban areas [6, 7]. This contributes to environmental and healthcare concerns as a significant portion of these pharmaceuticals, ranging from 80 to 90%, is discharged back into the environment, impacting ecosystems and raising questions about long-term effects on human health [3, 6, 8]. Addressing these challenges requires a comprehensive approach emphasizing sustainable water

management and responsible antibiotic use to protect both public health and the environment [8, 9].

Various methods have been employed to remove antibiotics from different water systems, including bio-filtration, flocculation, photocatalytic degradation, chlorination, osmosis, and adsorption [4, 5, 10]. Among these techniques, adsorption stands out as a simple and cost-effective process [4, 10]. Numerous adsorbents such as graphene, carbon nanotubes (CNTs), cationic surfactants, and biomaterials like water-soluble proteins have been utilized for these pharmaceuticals [1, 11–13]. Notably, nanomaterials offer significant advantages over traditional adsorbents because of their unique structural and surface properties [4, 12]. Polymeric nanofibrous membranes, produced through electrospinning, have gained attention in wastewater treatment owing to their high surface area to volume ratio, high pore interconnectivity, high porosity, tunable morphology, and enhanced filtration efficiency [4, 11]. Particularly in water treatment, these membranes serve as effective adsorbents for the removal of pharmaceuticals. They can be functionalized with various active surface groups to increase their drug selectivity and adsorption ability [4]. Their adaptable structure and design enable them to target a wide range of pollutants, such as hormones, antibiotics, and painkillers, which are challenging

for traditional water treatment techniques to eradicate [4]. Furthermore, electrospinning is a low-cost, simple, proficient and versatile technology, scalable from portable to large-scale industrial production [14, 15]. Many studies confirm the significant potential of nanofibers in adsorbing diverse pharmaceutical groups from water systems, showcasing their promising role in wastewater treatment applications [16, 17]. For instance, Das et al. (2020) prepared a montmorillonite-impregnated cellulose acetate nanofiber membrane using the electrospinning method, which was then employed for the removal of ciprofloxacin (CIP) from water. They found that CIP adsorption was significantly influenced by pH, with the optimal range of 6–7.5, and the adsorbent demonstrated good reusability after desorption [18]. In another study, carbon nanofiber (CNF) prepared from electrospun polyacrylonitrile (PAN) with subsequent thermal treatment was investigated for effective removal of three environmental contaminants, namely CIP, bisphenol A, and 2-chlorophenol from water 19.

Acrylic acid (AAc) is the key monomer in producing polyacrylic acid (PAAc)-based superabsorbent hydrogels. Its distinctive carboxylic acid (COOH) groups are essential for shaping the water retention and swelling properties of these hydrogels [20–22]. The versatility of AAc is evident as it can easily be incorporated into hydrogel network structures through grafts, copolymers, and interpenetrating polymer networks (IPNs) [22, 23]. This diverse integration influences various aspects of the hydrogels, including molecular chain aggregation, charges, hydrophilicity, rheological behavior, and the range of applications [22, 23]. The presence of COOH groups in PAAc not only allows complex formation with a variety of chemical compounds but also facilitates the establishment of hydrogen bonds between PAAc chains, water molecules, and dyes due to the carbonyl and carboxyl groups [22, 24, 25]. Moreover, PAAc-based hydrogels are characterized by low toxicity, biocompatibility, and biosafety, rendering them well-suited for use in medical and environmental applications [26, 27]. They are environmentally benign and do not contribute to pollution, thereby ensuring safe usage across a range of fields, including effective removal of environmental contaminants [28]. For example, guar gum/acrylic acid and

guar gum/itaconic acid hydrogels demonstrated remarkable dye removal rates, reaching 70% and 87%, respectively [23]. Dil and Sadeghi (2018) developed gelatin/acrylic acid (PGE-AcA) hydrogels, which exhibited copper ion adsorption rates of 65.8% and 78.7% [29]. Mohamed et al. (2017) synthesized N-quaternized chitosan/PAA hydrogels, which demonstrated broad-spectrum heavy metal removal (44.0–93.3%) [30].

However, the conventional approach of preparing AAc-based hydrogels using chemical cross-linking is not suitable for processing using electrospinning technology due to their covalently cross-linked network structure preventing them from dissolving in solutions [31–33]. To address this processing obstacle, we recently developed physically cross-linked AAc-based hydrogels by incorporating n-hexadecyl acrylate (C16A) into the polymer structure (denoted as P(AAc-co-C16A) hydrogels), thus establishing hydrophobic interactions between the polymer chains. It is noteworthy that the omission of chemical cross-linkers and surfactants during the preparation of the present hydrogels is a distinguishing feature of our method [34]. The resulting hydrogels exhibited a remarkable characteristic of easy solubility in organic solvents, thereby opening up extended possibilities for their application.

In this study, we employed an electrospinning technique to fabricate novel nanofibrous P(AAc-co-C16A) hydrogels for the first time. The efficacy of these hydrogels in the removal of antibiotics was then assessed, thereby providing evidence for their potential applications in the remediation of environmental contaminants and the management of pharmaceutical waste. Ciprofloxacin (CIP) was selected as a model drug for the purposes of this study, with the objective of investigating the impact of the C16A mole fraction on the adsorption and desorption efficacy of the electrospun hydrogels. The rationale for the selection of CIP was based on the fact that it is an amphoteric quinolone antibiotic, with pKa values of 6.2 and 8.8, and an isoelectric point of 7.4 [35, 36]. This makes it challenging to remove from water due to its complex chemical nature and stability (Fig. 1). In addition, we investigated the adsorption characteristics of the hydrogel nanofibers using both reaction-based kinetic and isotherm models, as well as the mechanisms of ciprofloxacin

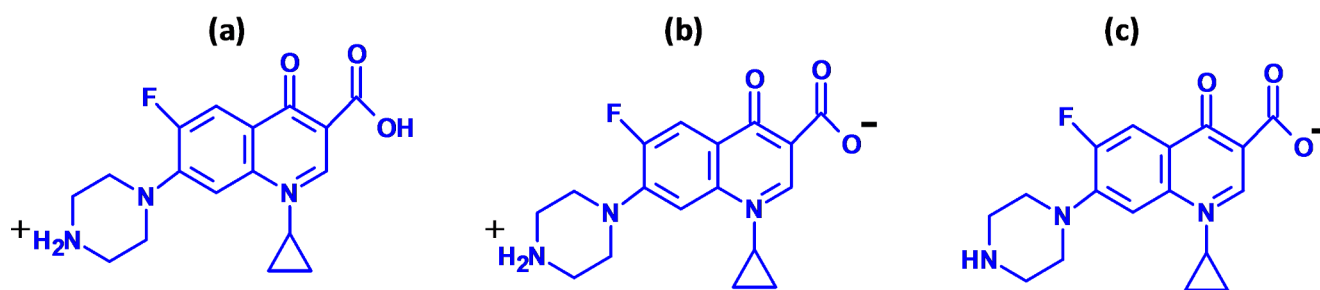


Fig. 1 Cationic (a), zwitterionic (b), and anionic forms (c) of CIP in aqueous solutions at low, neutral, and high pH values, respectively

(CIP) adsorption in the electrospun hydrogels. The findings collectively highlight a promising potential for these materials to contribute to the remediation of environmental challenges and the management of pharmaceutical waste.

Materials and Methods

Chemicals

Acrylic acid (AAc, Merck) was filtered through an alumina column to remove any inhibitor present, prior to its use. n-Hexadecyl acrylate (C16A) was purchased from TCI Chemicals. 2-Hydroxy-4'-(2-hydroxyethoxy)-2-methylpropiophenone (Irgacure 2959), chloroform, ethanol and alumina column were obtained from Sigma-Aldrich. Ciprofloxacin (CIP) (purity > 99%) was supplied from Menarini Türkiye Production Facility.

Synthesis of Poly(Acrylic acid-co-hexadecyl Acrylate) Hydrogels

In order to prepare a homogeneous AAc-C16A monomer solution containing 20–50 mol% C16A, the latter was first

melted at 35 °C on a magnetic stirrer. Thereafter, ethanol and AAc were added sequentially. Subsequently, Irgacure 2959, a photoinitiator at a concentration of 0.2 w/v%, was added to the solution. This solution was then placed in a 10 mL syringe and exposed to UV light at a wavelength of 365 nm for 24 h to fully polymerize the monomer solution. The hydrogel was then immersed in an excess of water to exchange ethanol with water (Fig. 2). We maintained the total monomer concentration at 3 M, while the mole fraction of C16A was varied between 25 and 50 mol % (Table 1). To illustrate the synthetic procedure, we present details for the preparation of P(AAc-co-C16A) hydrogels containing 35 mol % C16A. C16A (3.10 g) was first dissolved in 5.1 mL of ethanol at 35 °C. Following that, AAc (1.34 mL) and Irgacure 2959 (14.3 mg) were successively introduced to this solution while stirring with a magnetic stirrer. Subsequently, the solution was loaded into a 10 ml syringe and exposed to UV light with a wavelength of 360 nm for one day. Finally, the resulting hydrogel was soaked in an excess of water for 48 h. During this process, ethanol within the hydrogel network was replaced by water, leading to the creation of strong hydrophobic interactions among the hexadecyl (C16) side chains of C16A units due to increasing

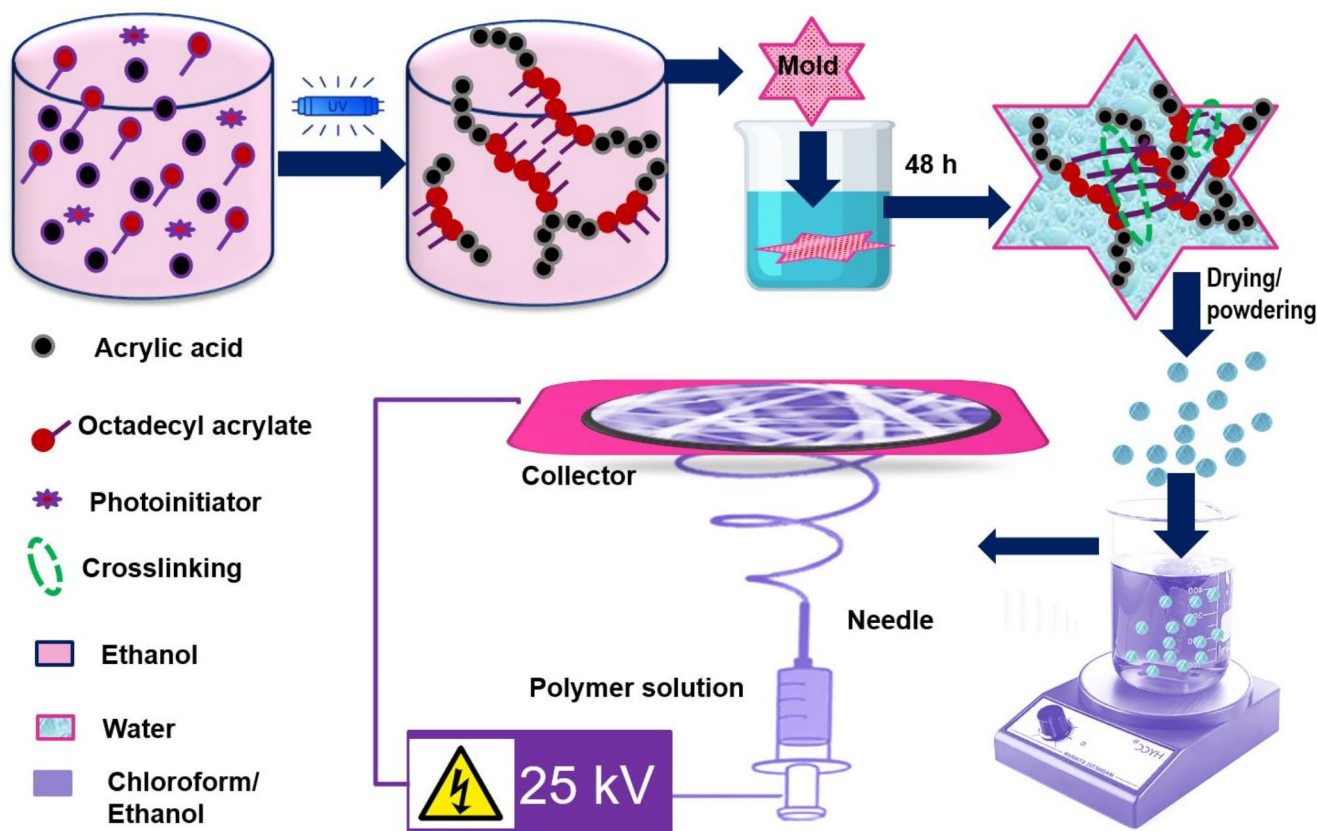


Fig. 2 Diagram showing the synthesis of PAA hydrogels with C16A units by the organosolv method, followed by bottom-to-top electrospinning to produce a nano-fiber network

Table 1 Preparation conditions of P(AAc-co-C16A) hydrogels

C16A mol%	C16A (g)	AAc (mL)	Ethanol (mL)	Irga-cure 2959 (mg)
25	2.22	1.54	5.91	14.3
30	2.66	1.44	5.50	14.3
35	3.10	1.34	5.10	14.3
40	3.55	1.23	4.69	14.3
45	3.99	1.13	4.28	14.3
50	4.43	1.03	3.87	14.3

flexibility of the polymer backbone and hence facilitating the formation of a supramolecular hydrogel [37].

Fabrication of Electrospun Nanofiber

The electrospinning procedure commenced with a preliminary step in which the hydrogels were dried at 37 °C inside a vacuum oven for a duration of four days. The dried hydrogels were then dissolved in a solution consisting of chloroform and ethanol, with a volume ratio of 1:2, using magnetic stirring for one hour [34]. The solution maintained a constant polymer concentration of 20 wt%. Subsequently, the prepared solution was then loaded into a 50 mL syringe and delivered through a Teflon tube to a multi-needle metal syringe consisting of four individual needles at a feed rate of 4 mL·h⁻¹. To initiate the electrospinning process, a voltage of 25 kV was applied across a 15 cm gap between the needle and a rotating aluminum drum. This electrospinning setup was arranged vertically, allowing for precise and controlled deposition of the polymer solution onto the surface of the rotating drum.

Characterization

The surface morphology of the nanofibrous membrane was examined using an FEI Quanta FEG 250 scanning electron microscope (SEM). Prior to examination, the membranes underwent a gold coating process to enhance imaging clarity and resolution. PerkinElmer Spectrum 100 Fourier transform infrared (FTIR) spectrometer was used to highlight the structural modifications of nanofibers and to examine the chemical groups of the manufactured nanofibers. The spectra were taken within the region of 650–4000 cm⁻¹. The zeta surface charges of nanofiber were determined as streaming potential with an electrokinetic analyzer (SurPAS, Anton Paar GmbH). For this purpose, the nanofiber was pre-washed with deionized water. Using 0.1 M NaOH and HCl solutions, the pH value of the solution was ranged from 4 to 9 in order to assess the pH impact during the analysis. Then, 1.0 mM KCl solution was used to circulate inside the adjustable gap cell containing nanofiber. X-Ray Diffraction

(XRD) measurement was conducted utilizing the Panalytical Empyrean instrument, equipped with Cu-K α radiating sources. The data were collected in the 2 θ range of 1–50° at a scan rate of 1°·min⁻¹. The swelling ratio (SR) of the electrospun hydrogels was ascertained by maintaining the hydrogel in water for a duration of 24 h, thereby acquiring the swelling weight (Ms). Subsequent to this, the hydrogel underwent complete drying, resulting in the determination of the dried weight (Md). The SR was then calculated as follows: SR = Ms/Md. The water contact angle (WCA) was evaluated by using the KSV Attension Theta contact angle meter. A water droplet was placed on the straight membrane surface from the micro-syringe with a needle and the contact angle was measured. Measurements were done at least ten different points of each membrane to minimize the experimental errors. The values were averaged and the WCA of membranes was determined. To determine the mechanical performance of the electrospun hydrogels, a fibrous membrane measuring 50 mm in length, 5 mm in width, and 0.1 mm in thickness was subjected to a tensile test at an elongation speed of 5 mm·min⁻¹ using a universal testing machine (Zwick Roell Z0.5 TH).

Adsorption and Desorption Method

The adsorption and desorption experiments of CIP by the nanofibers were conducted in a batch mode. The tests were conducted at 23 ± 2 °C using 250 mL flasks in an orbital shaker set to shake at 150 rpm. CIP solutions, each 100 mL in volume, at an initial concentration (C_0) of 5 mg·L⁻¹ and at pH = 6.4 were exposed to 5 mg of nanofiber containing 25, 35, 40, and 55 mol % C16A at various contact times (0–72 h) to find out the equilibrium time and ideal mole ratio of C16A to nanofiber in adsorption study. At specific time points (i.e. 0, 5, 10, 30, 60, 90, 120, 180, 240, and 360 min), 2 mL of the solution was withdrawn by 10 mL plastic syringe, filtered by 0.45 mm syringe filter, and stored in refrigerator for CIP analysis.

Freundlich and Langmuir isotherm models were used to analyze CIP adsorption on nanofibers at different initial CIP concentrations (1, 5, 25, 50, 75, 100 mg·L⁻¹). Additionally, pseudo-first-order and pseudo-second-order kinetic models were also studied. Table 2 provides the formulae for the isotherm and kinetic models. The amount of CIP absorbed at equilibrium q_e (in mg·g⁻¹) was estimated by,

$$q_e = (C_o - C_e) \frac{V}{W} \quad (1)$$

where C_o and C_e are the initial and equilibrium concentrations of CIP in the solution, respectively (mg·L⁻¹), V is the

Table 2 Mathematical equations for the isotherm and kinetic models used in this study. q_e and q_t (both in $\text{mg}\cdot\text{g}^{-1}$) are the amounts of CIP adsorbed at equilibrium and at time t , respectively, K_f ($\text{L}\cdot\text{g}^{-1}$) and $1/n$ are Freundlich characteristic constants relating to the capacity and intensity of the adsorption process, respectively, b is the Langmuir adsorption equilibrium constant ($\text{L}\cdot\text{mg}^{-1}$), Q^0 is the maximum adsorption capacity at monolayer coverage ($\text{mg}\cdot\text{g}^{-1}$), R_L is the dimensionless constant separation factor, k_1 (h^{-1}) and k_2 ($\text{g}\cdot\text{mg}^{-1}\text{h}^{-1}$) are the rate constants of the pseudo-first-order and second-order models, respectively [39]

Model	Equations	Linear expressions
Freundlich isotherm model	$q_e = K_f \cdot C_e^{\frac{1}{n}}$	$\log q_e = \log K_f + \frac{1}{n} \log C_e$
Langmuir isotherm model	$q_e = \frac{Q^0 \cdot b \cdot C_e}{1 + b \cdot C_e}$ $R_L = \frac{1}{1 + bC_0}$	$\frac{C_e}{q_e} = \frac{C_e}{Q^0} + \frac{1}{b \cdot Q^0}$
Pseudo-first order kinetic model	$\frac{dq_t}{dt} = k_1 (q_e - q_t)$	$\log \frac{(q_e - q_t)}{q_e} = -\frac{k_1 t}{2.303}$
Pseudo-second order kinetic model	$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2$ $h = k_2 \cdot q_e^2$	$\frac{t}{q_t} = \left[\frac{1}{k_2 q_e^2} \right] + \frac{1}{q_e} t$

volume of the CIP solution (L), and W is the weight of the nanofiber (g) [38].

In the desorption study, a surplus amount of CIP solution ($100 \text{ mg}\cdot\text{L}^{-1}$) was first added to a hydrogel nanofiber specimen containing 35 mol% C16A (20 mg), which was then stirred until equilibrium was established. The CIP-loaded nanofiber was then withdrawn from the liquid phase and mixed with distilled water. After 72 h, the concentration of CIP in the supernatant was measured. The desorption process continued by exchanging the supernatant to distilled water until the supernatant's concentration was below the estimation limit ($<0.1 \text{ mg}\cdot\text{L}^{-1}$). Cumulative desorption efficiency is calculated by the equation,

$$\text{Desorption efficiency (\%)} = (M_{des}/M_{ads}) 10^2 \quad (2)$$

where M_{des} is the cumulative amount of CIP desorbed from nanofiber and M_{ads} is the total amount of adsorbed CIP onto nanofiber [39].

Methods of Analysis

The C_e were measured using both on a UV-vis spectrophotometer (SmartSpec 3000, Bio-Rad Corp.) at the wavelength of 272 nm, and a SHIMADZU high performance liquid chromatography-mass spectrometry (HPLC-MS/MS). Six standards in a concentration range of $0.1\text{--}5 \text{ mg}\cdot\text{L}^{-1}$ were used to create a calibration curve with a coefficient of regression R [2] greater than 0.999 for UV-vis spectrophotometer. The SPD-M20A model UV-vis detector and Inertsil ODS-3 V model column (particle size $5 \mu\text{m}$, $4.6 \times 250 \text{ mm}$) were used at a flow rate of $1 \text{ mL}\cdot\text{min}^{-1}$, and $20 \mu\text{L}$ samples were injected to determine the pharmaceutical. 0.02 M phosphate buffer and acetonitrile (v/v) in a ratio of 77:23 was used for the mobile phase. Phosphate buffer was prepared daily using disodium hydrogen phosphate and orthophosphoric acid. Pharmaceuticals's peaks were determined at the wavelength of 272 nm for CIP and detention time was determined as 4.6 min on a DAD detector. The column

temperature was set to 40°C . The removal efficiency E was determined by the equation,

$$E \% = \left(1 - \frac{C_p}{C_f} \right) 10^2 \quad (3)$$

where, C_f and C_p are the concentrations in membrane feed streams and permeate, respectively [38].

Results and Discussion

Physico-chemical Properties of P(AAc-co-C16A) Hydrogels

Figure 3 displays FTIR spectra of the hydrogels with 35 mol % C16A after (a) and before the electrospinning process (b), along with the individual components, namely C16A (c), and AAc (d). Several distinct absorption peaks are observed shedding light on the chemical composition and structure of the copolymer hydrogel. The hydrogels both before and after the electrospinning step exhibit characteristic absorption peaks at 2850 and 2918 cm^{-1} (C-H stretching of alkyl groups), 1705 cm^{-1} (C=O stretching), 1451 cm^{-1} (CH-CH bending), and 1162 cm^{-1} (C-O stretching). These characteristic peaks also appear in the spectra of AAc and C16A providing a clear evidence of the presence and chemical identity of the components within the hydrogel. Following the electrospinning process, alterations in the intensity ratios of the bands at 1550 cm^{-1} and 1705 cm^{-1} , as well as in the region between 1000 cm^{-1} and 1400 cm^{-1} , were observed. These differences can be attributed to minor alterations in the intermolecular interactions within the polymer matrix, such as modifications in hydrogen bonding or conformational arrangements, which might occur during the electrospinning process [40, 41]. However, no significant shifts in the absorption bands are identified for the copolymer hydrogels after electrospinning. This lack of significant shifts implies that the electrospinning process did not

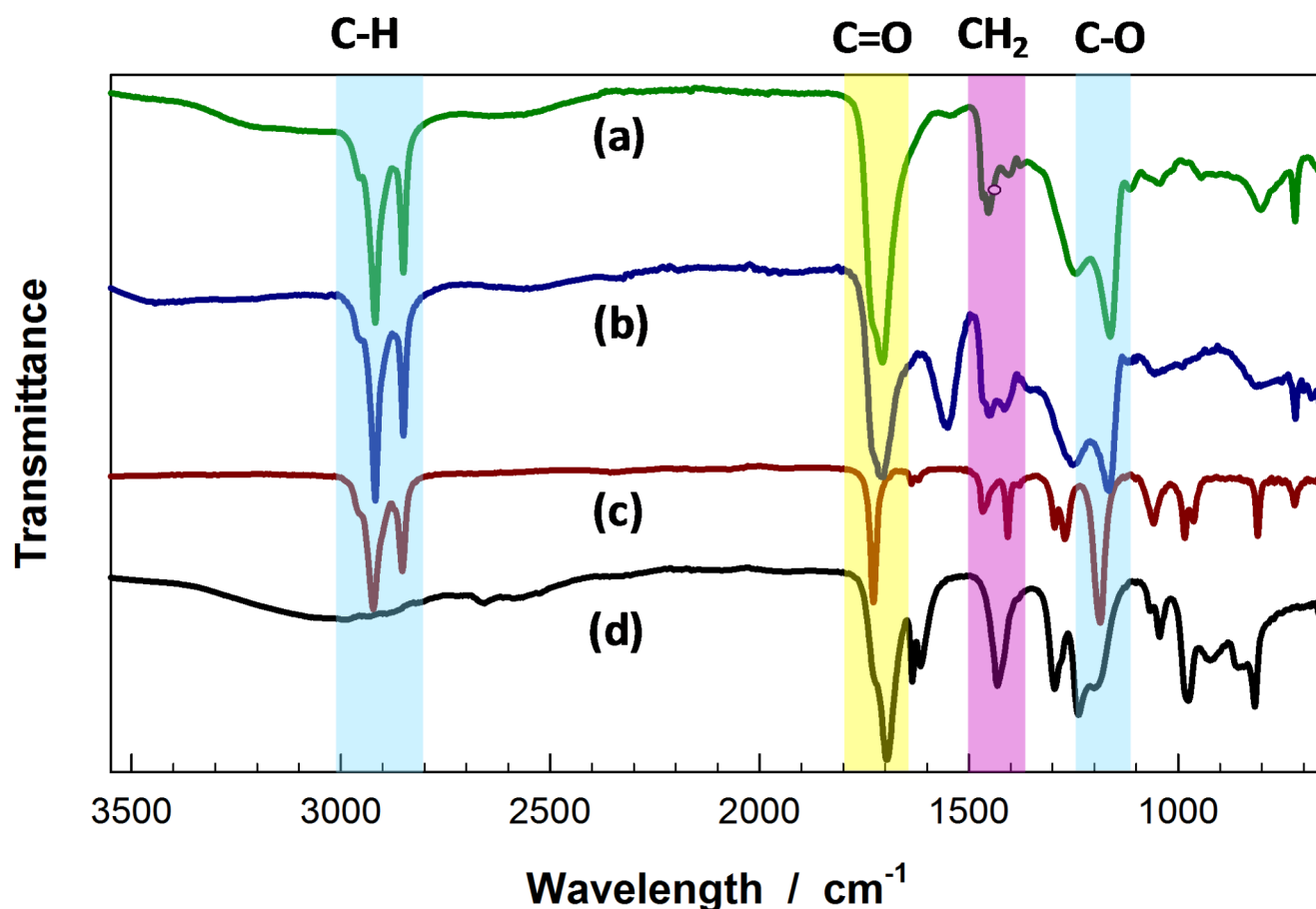


Fig. 3 FTIR spectra of P(AAc-co-C16A) hydrogels containing 35 mol % of C16A after (a) and before electrospinning (b), together with C16A (c) and AAc (d)

induce any discernible alterations in the chemical structure of the hydrogels. This is an important finding, as it suggests that the electrospinning method employed in this study did not adversely affect the chemical integrity of the hydrogel, ensuring the preservation of its desired properties and functionality (Fig. 3).

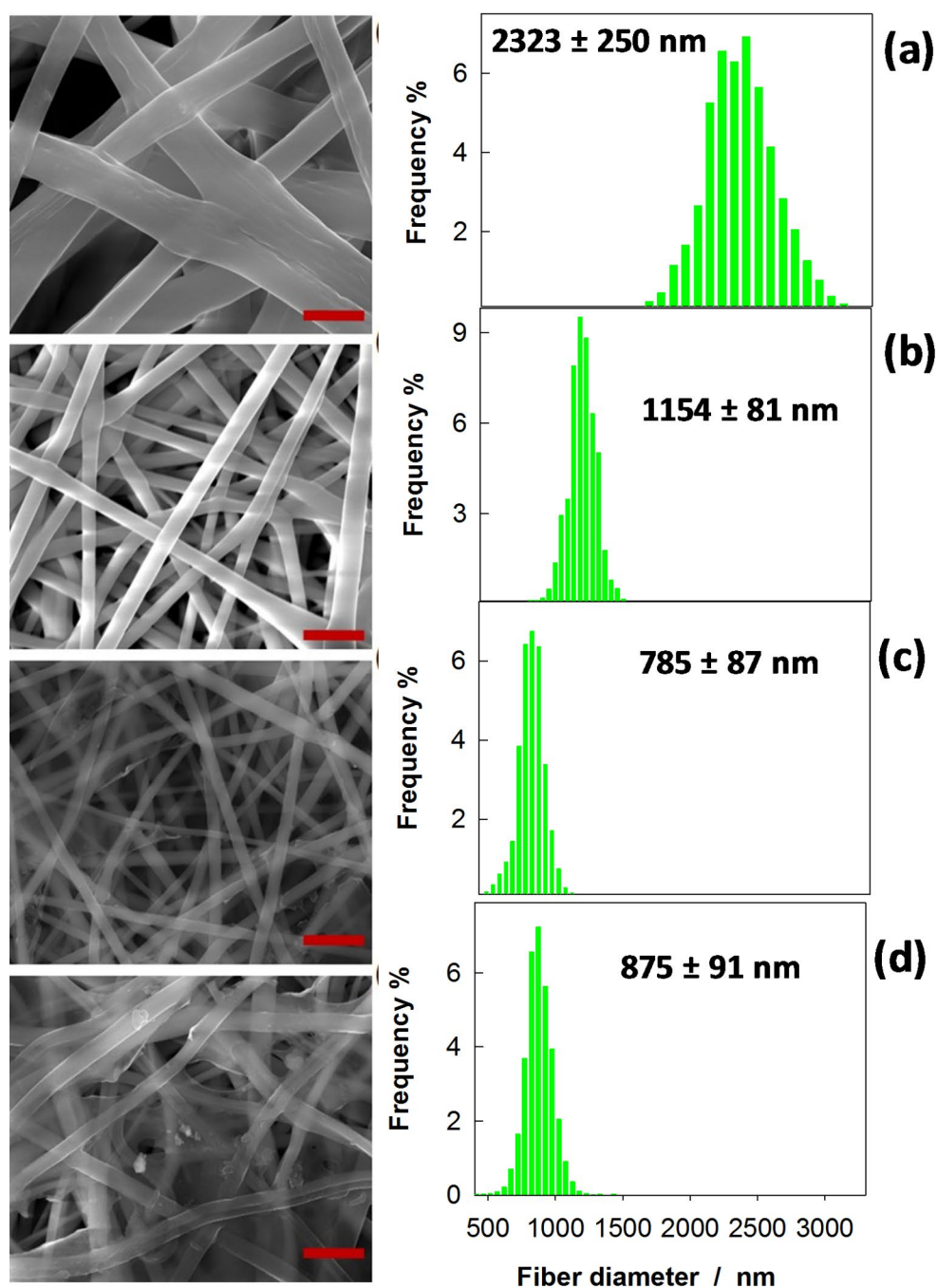
Characterization of Electrospun Hydrogel Nanofibers

Figure 4 shows SEM images and fiber size distributions of electrospun hydrogel nanofibers containing 50 (a), 35 (b), 30 (c), and 25 mol % C16A (d). An increase in the molar fraction of C16A in the hydrogels leads to the production of significantly larger fiber sizes and simultaneously, they exhibit a smooth surface without the presence of any beads. For instance, as the C16A content is increased from 25 to 50 mol %, the fiber size increases from 875 nm to 2223 nm. This phenomenon can be attributed to the heightened inter-chain interactions among copolymers due to the presence of a greater quantity of long alkyl side-chains of C16A segments [34]. Namely, the increased C16A mole

ratio introduces longer hydrophobic alkyl chains into the polymer matrix, increasing van der Waals forces and chain entanglement. These stronger interactions between the chains increase the overall viscosity of the polymer solution, which makes it more difficult for the jet to stretch under the electric field during the electrospinning process [42]. This results in larger fibers being formed due to reduced jet thinning. Moreover, decreasing the molar fraction of C16A below 30 mol % results in the formation of sheet-like structures within the nanofibrous network, which can be ascribed to the high surface tension caused by inadequate viscosity. Ultimately, the optimal morphology characterized by both the desired fiber size and surface smoothness was observed in the electrospun hydrogel containing 35 mol % C16A. This observation underscores the delicate balance required to achieve the desired morphology in P(AAc-co-C16A) hydrogels, with implications for their potential applications in various fields. Further investigations and fine-tuning of the molar fraction of C16A may offer opportunities for tailored hydrogel properties and applications.

XRD analysis was performed to ascertain the crystalline structure of electrospun PAAc-C16A hydrogels (Fig. 5a).

Fig. 4 SEM images and fiber size distributions of electrospun P(AAc-co-C16A) hydrogel nanofibers containing 50 (a), 35 (b), 30 (c), and 25 mol % C16A (d). The scale bars in the SEM images = 5 μ m



The results demonstrated the presence of a single, sharp peak at a 2θ angle of 21.7° , which was observed for all hydrogels with varying mole fractions of C16A. The observation of this peak is indicative of the hexagonal arrangement of the side alkyl chains of C16A units [43]. The swelling ratio (SR) of the electrospun hydrogels was found to decrease with increasing molar fraction of C16A (Fig. 5b). Meanwhile, the water contact angle (WCA) of nanofibrous P(AAc-co-C16A) hydrogels exhibits a notable dependence on the molar fraction of C16A integrated into the polymer network. At a molar fraction of C16A of 25%,

the water contact angle (WCA) is as low as 70° , indicating a predominantly hydrophilic surface. It can be attributed to an equilibrium between the hydrophilic acrylic acid units and the C16A segments, the latter of which are hydrophobic [43]. At this concentration, the hydrophilic units are the primary determinant of the surface properties. Nevertheless, when the C16A content reaches 30–50 mol%, there is a sharp increase in the WCA, reaching values between 110° and 120° . This rise in WCA reflects the enhanced hydrophobic character introduced by the long alkyl chains of C16A, which form a more hydrophobic surface layer [43, 44]. The

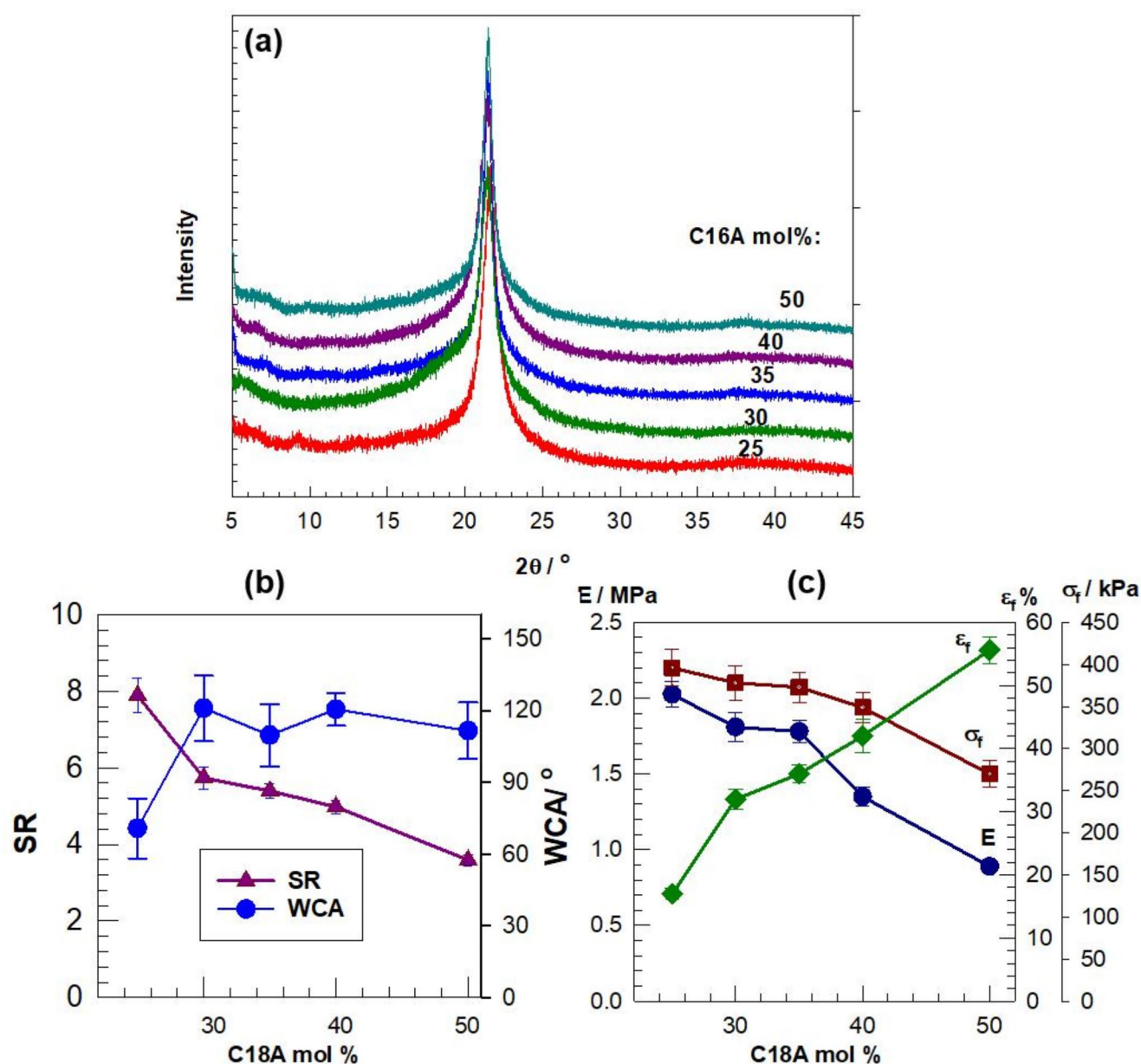


Fig. 5 (a) XRD spectra, (b) swelling ratio (SR) and water contact angle (WCA), and (c) Young's modulus E , tensile strength σ_f , and strain at failure ϵ_f of electrospun P(AAc-co-C16A) hydrogel nanofibers containing various mole fractions of C16A

transition from hydrophilic to hydrophobic behavior demonstrates how the controlled incorporation of C16A can be used to tune the surface properties of P(AAc-co-C16A) hydrogels, which is crucial for applications where specific surface wettability is desired. These include selective adsorption, controlled drug release, and hydrophobic filtration membranes [45, 46]. Figure 5c provides a summary of the Young's modulus, tensile strength, and strain at failure of the electrospun P(AAc-co-C16A) hydrogels with different C16A molar fractions. It is evident from the figure that there is an inverse relationship between the C16A molar fraction and both the elastic modulus and tensile strength.

Concurrently, the changes in the strain at failure are proportional to the C16A molar fraction. The observed trends in the mechanical properties of the hydrogels can be attributed to the morphology of the resulting fibrous networks, as evidenced by previous research [47, 48]. Specifically, smaller fibrous networks are characterized by a higher surface area-to-volume ratio, leading to increased stiffness. In contrast, larger fibrous networks exhibit a lower surface area-to-volume ratio, resulting in enhanced flexibility.

Optimization of Nanofiber Composition for CIP Removal

In the next step, we investigated the effects of the C16A mole fraction on the adsorption and desorption efficiencies of CIP in the electrospun hydrogels. CIP, which is a commonly used antibiotic, poses a significant challenge to water pollution due to its persistence and low biodegradability. This persistence in drinking water can lead to the emergence of antibiotic-resistant bacteria, making infections more difficult to treat, and can be toxic to aquatic life, disrupting ecosystems. In addition, long-term exposure can present health risks to humans, and the persistent nature of

the substance in the environment increases overall exposure risks. In addition, it is resistant to the traditional biological treatment processes used in wastewater treatment plants, making it difficult to remove effectively from wastewater [49–51].

CIP equilibrium conditions on nanofibers containing various amounts of C16A between 25 and 50 mol % were established with experiments conducted over various time periods. Figure 6a presents the impact of the contact time on CIP adsorption characteristics of a nanofiber with 35 mol % C16A. Here, CIP concentration in the solution C_t (circles), the amount of CIP adsorbed by the nanofiber q_t (triangles), and the E (bars) are shown as a function of the contact time t .

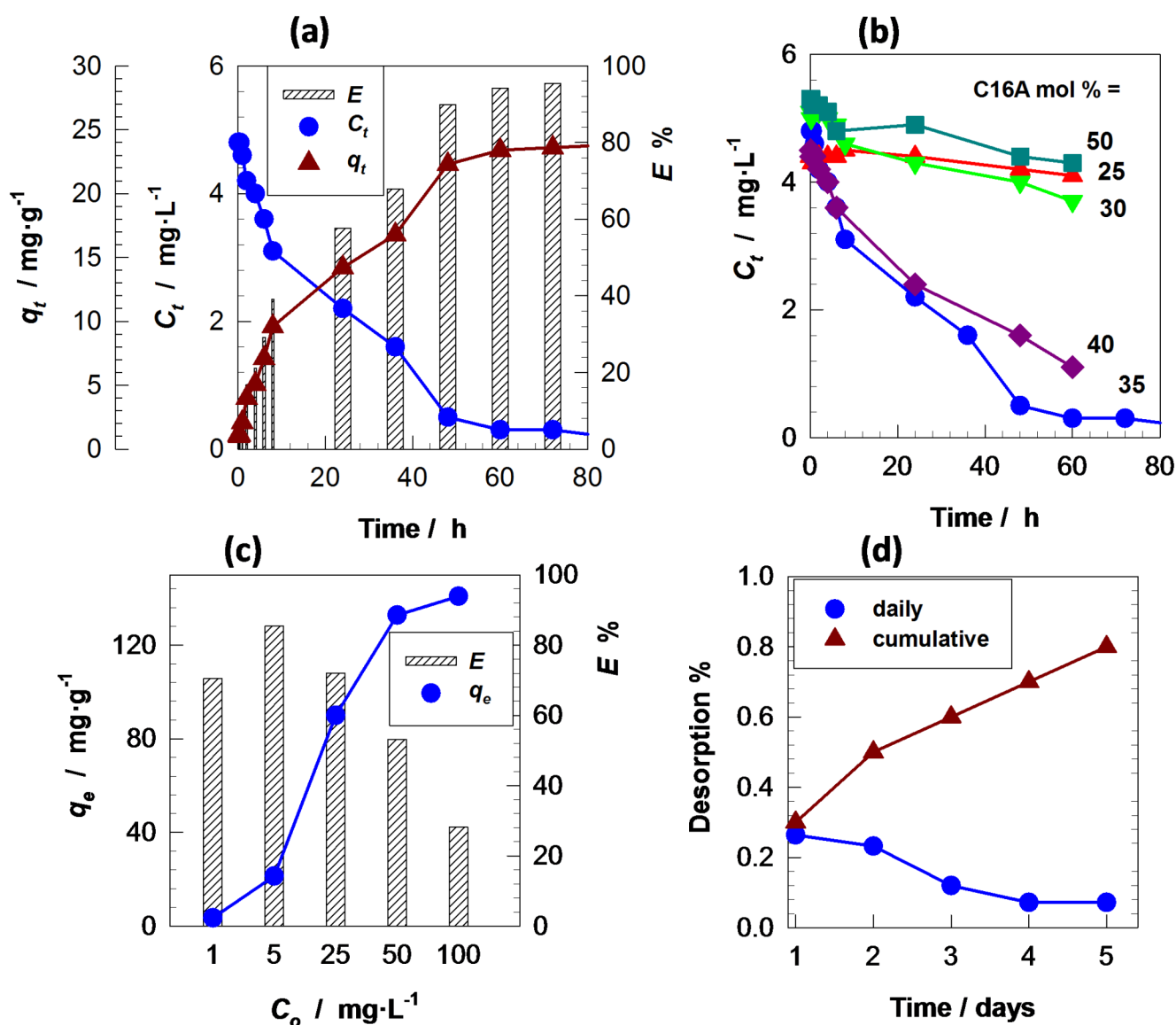


Fig. 6 Adsorption profiles of CIP by nanofibers. (a) CIP concentration in the solution C_t (circles), the amount of CIP adsorbed by the nanofiber q_t (triangles), and CIP E (bars) all plotted against the contact time. C16A = 35 mol %. (b) Effect of C16A content of nanofibers on the C_t

vs time plot. C16A contents are indicated. (c) Effect of C_o on q_e and E . (d) Desorption efficiency of a CIP-loaded nanofiber with 35 mol % C16A

CIP concentration initially decreases rapidly and 50% of the CIP in the solution could be removed within 8 h. The results also show that the adsorption reaches to equilibrium within 60 h. Figure 6b comparing the contact time dependence of C_t for nanofibers with various C16A contents reveals that the highest CIP removal performance of 96% corresponding to an adsorption capacity of $24 \text{ mg} \cdot \text{g}^{-1}$ was achieved at 35 mol % C16A within an equilibrium time of around 60 h.

C_o strongly influences the amount of CIP adsorbed onto nanofiber (Fig. 5c). With an increase in C_o from 1 to $100 \text{ mg} \cdot \text{L}^{-1}$, the q_e increases from 3.5 to $141 \text{ mg} \cdot \text{g}^{-1}$, and the optimum CIP E was attained when the C_o of CIP was $5 \text{ mg} \cdot \text{L}^{-1}$. Figure 6c also shows that the efficiency E initially increases but then decreases as C_o is increased. The reduction in the CIP adsorption efficiency at high C_o could potentially be attributed to limited binding sites on nanofiber for the adsorption process [52]. One possible explanation for q_e raising is that the greater concentration supplies a greater driving force, such as the adsorption gradient, which helps to overcome the obstacle of CIP transferring between the liquid and solid phases. Li et al. obtained a similar finding concerning the impact of CIP concentration on the adsorption of CIP by biochar derived from waste tea leaves [52]. Adsorption experiments conducted at various amounts of nanofiber showed that the C_e of CIP in the solution decreases while the E slightly increases with increasing amount of the nanofiber (Figure S1). For instance, increasing the amount of nanofiber from 10 to 100 mg leads to an increase in E from 88 to 96%.

In the desorption study, CIP-loaded nanofibers containing 35 mol % C16A were immersed in an excess of water, and the desorption of CIP from the nanofiber was monitored for 5 days after which the equilibrium was achieved. The loaded amount of CIP was 140.8 mg per gram of nanofiber, which is significantly higher than other nanofibers developed in many previous studies. For example, 13.8 mg per gram was reported as the maximum adsorption capacity of CIP for electrospun montmorillonite-impregnated cellulose acetate nanofiber membranes [18]. Similarly, the maximum adsorption capacity for carbon nanofibers was reported as $10.36 \text{ mg} \cdot \text{g}^{-1}$ [53]. Figure 6d shows the daily and cumulative desorption efficiency of CIP-loaded nanofiber plotted against the desorption time. The cumulative desorption efficiency was estimated to be 0.8% without pH adjustment indicating that 99.2% of CIP is adsorbed irreversibly by the nanofiber. Generally, desorption efficiency varies depending on both the environmental conditions such as pH and temperature and the adsorption mechanism between the substance and the adsorbent. Ji et al. (2021) discovered a desorption efficiency of 16.9% following the adsorption of CIP when the desorption phase was deionized water with a pH 7 [54]. However, based on the desorption results

presented in the study [55], NaOH (CIP 98.3%), NaCl (CIP 83.3%), and $\text{C}_2\text{H}_6\text{O}$ (CIP 64.7%) demonstrated better desorption performance compared to the other eluents used. Effective desorption performance may be aided by NaOH's ability to control the pH of the solution, which would prevent CIP's electrostatic attraction. In the meantime, CIP was successfully desorbed from the adsorbent due to the decomplexation impact of the OH^- in NaOH (Fig. 6).

Adsorption Isotherms and Kinetics

We can determine which model the real adsorption isotherm uses via comparison of the linear correlation coefficient or the degree of closeness between the experimental sites and the model.

Hence, a precise mathematical explanation of the adsorption isotherm is crucial for studying the removal process between CIP and adsorbents as well as for helping us understand the adsorption capacity of CIP and the characteristics of adsorbents [56]. Freundlich model is an experimental equation that assumes unequal adsorptive energies on the adsorbent surface. The Langmuir model is based on the foundation of equal adsorptive energies and uniform adsorption sites [56]. According to the Langmuir isotherm, which is an empirical model, the adsorbed layer's thickness is a single molecule (monolayer adsorption), and the adsorption process takes place at equivalent and identical specific localized spots. Even on nearby sites, there shouldn't be any lateral contact or steric barrier among the adsorbed molecules. In contrast to the Langmuir isotherm approach, the Freundlich model can be applied to multilayer adsorption because it is not limited to monolayer formation. It is not necessary for adsorption affinities and heat to be uniformly spread over the heterogeneous surface in this isotherm model [57].

The adsorption properties of hydrogel nanofibers were examined using both reaction-based kinetic and isotherm models. In order to understand adsorption mechanisms, the kinetic model fits the data from the adsorption process, which is mostly used to describe the adsorption rate of CIP by the adsorbent. Furthermore, we can determine the equilibrium period needed for adsorption to optimum efficiency by using adsorption kinetics. This is one of the most important details in adsorption data [58]. Figure 7 shows the plots of Lagergren pseudo-first order (a), and pseudo-second-order models (b) for the adsorption of CIP onto nanofiber with 35 mol % C16A. The symbols and solid lines represent the experimental and calculated data, respectively. Comparing the two reaction-based kinetic models, the value R [2] for the pseudo-second-order model is much closer to 1 than that of the pseudo-first-order model (0.98 vs. 0.92) indicating that the former model fits well the adsorption data

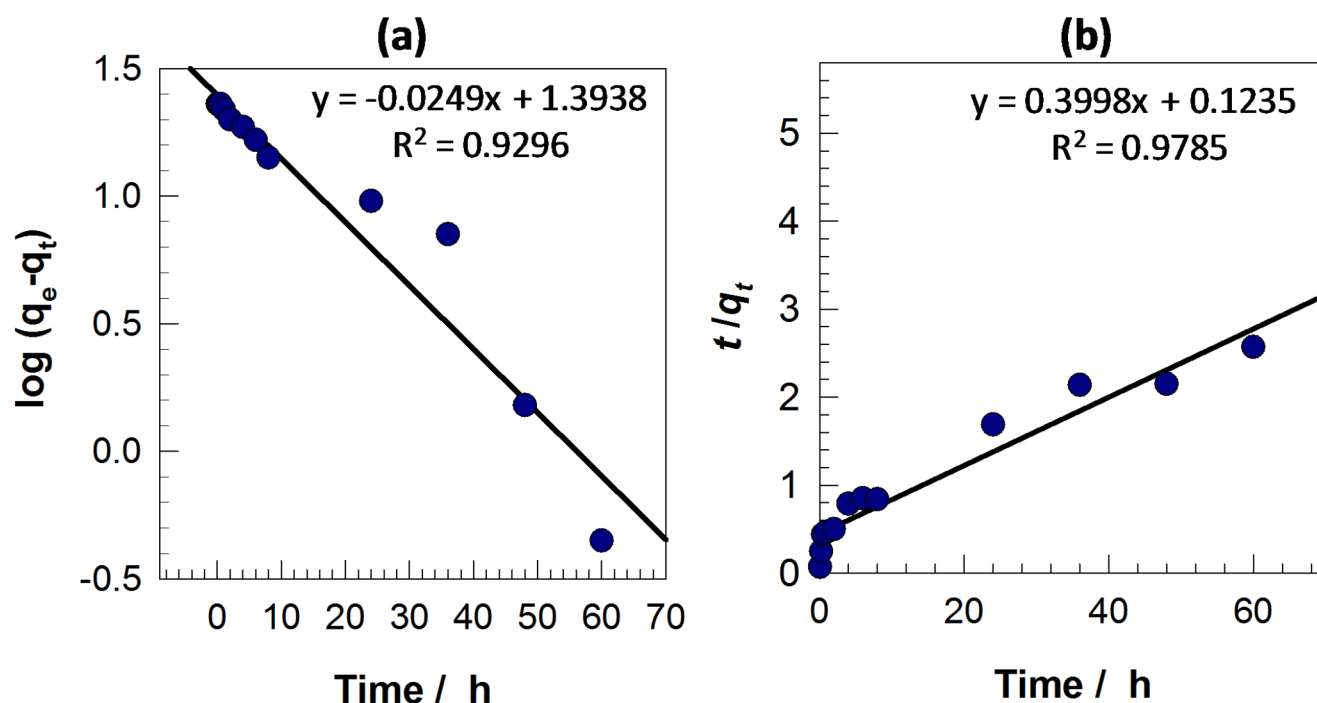


Fig. 7 Reaction base kinetics model for the adsorption of CIP onto nanofiber with 35 mol % C16A. The symbols and solid lines represent the experimental and calculated data, respectively, using Lagergren pseudo-first order (a) and pseudo-second-order models (b)

Table 3 Estimated pseudo-first order and pseudo-second order kinetic model parameters of adsorption

Kinetic model	Parameters	Value	R^2
Pseudo first-order	q_{exp} ($\text{mg} \cdot \text{g}^{-1}$)	23.8	0.9296
	q_{calc} ($\text{mg} \cdot \text{g}^{-1}$)	1	
	k_1 ($\text{L} \cdot \text{min}^{-1}$)	-3.2	
Pseudo second-order	q_{exp} ($\text{mg} \cdot \text{g}^{-1}$)	23.8	0.9785
	q_{calc} ($\text{mg} \cdot \text{g}^{-1}$)	8.1	
	k_2 ($\text{g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$)	1.3	
	h ($\text{mg} \cdot \text{g}^{-1} \cdot \text{min}^{-1}$)	85	

onto nanofiber (Table 3). This also reveals that the chemical adsorption is engaged as the rate-limiting stage in adsorption process and the adsorption capacity is directly correlated with the amount of active sites [19, 38]. This finding

is in accord with a desorption efficiency of less than 1% of the nanofiber, as shown in Fig. 5d, and also indicates formation of a chemical bonding among the adsorbent and the adsorbate. It can be said that CIP adsorption proceeds more quickly at the start of the adsorption process since the pseudo-second-order rate constant, k_2 , is computed as $1.3 \text{ g} \cdot \text{mg}^{-1} \cdot \text{min}^{-1}$, which is less than the starting adsorption rate constant (h) of $85 \text{ mg} \cdot \text{g}^{-1} \cdot \text{min}^{-1}$ (Table 3).

The Freundlich and Langmuir isotherm models were also used to examine the adsorption properties of hydrogel nanofibers. Figure 8 shows the plots of the adsorption isotherm data. Table 4 lists the predicted Freundlich and Langmuir isotherm parameters regarding CIP adsorption. In comparing the outcomes of the isotherm models, the Langmuir

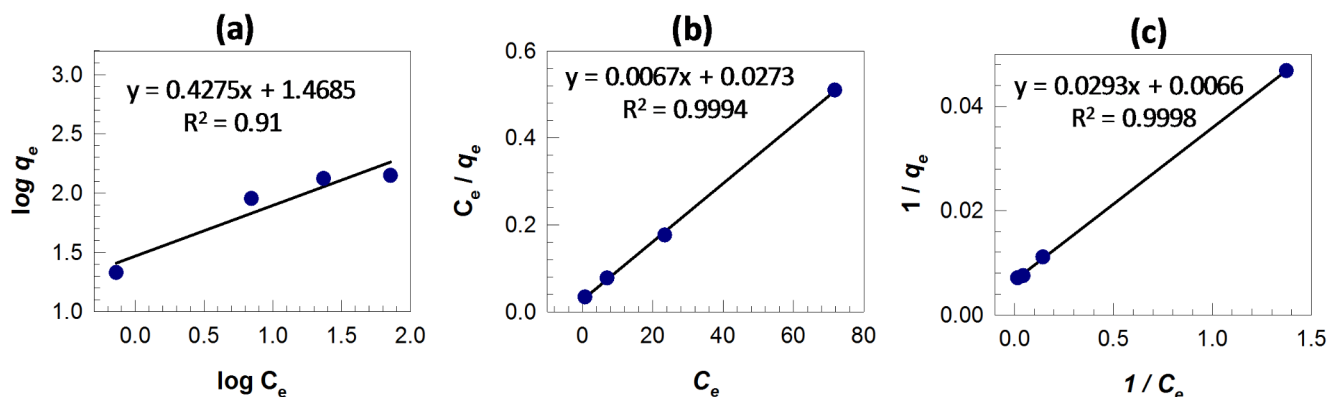


Fig. 8 Freundlich (a), Langmuir type 1 (b), and Langmuir type 2 isotherms (c) for adsorption of CIP onto nanofiber with 35 mol % C16A

Table 4 Estimated isotherm parameters of adsorption

Isotherm model	Parameters	Value	R^2
Freundlich	$K_f (\text{L} \cdot \text{g}^{-1})$	2.7	0.91
	n	0.7	
Langmuir Type 1	$Q^0 (\text{mg} \cdot \text{g}^{-1})$	36.6	0.9994
	$b (\text{L} \cdot \text{mg}^{-1})$	4.1	
	R_L	0.01–0.5	
Langmuir Type 2	$Q^0 (\text{mg} \cdot \text{g}^{-1})$	87.72	0.9998
	$b (\text{L} \cdot \text{mg}^{-1})$	4.56	
	R_L	6.5×10^{-5} – 6.5×10^{-3}	

Type 2 isotherm which represents the monolayer adsorption of CIP onto nanofiber had the highest R [2] value of 0.9998. It is possible to conclude that the adsorption of CIP on nanofiber is favorable since the R_L is between 0 and 1. Langmuir model assumes monolayer coverage, non-interaction among the sorbed species, homogeneous adsorption site in macroscopic view, and the adsorption energy is constant. According to Langmuir model, the rate of adsorption is proportional to the concentration of the liquid and the coated surface of the solid, and this deduction was also consistent with the experimental results [59, 60].

Adsorption Mechanism Analysis

The main mechanisms for organic chemical adsorption onto carbon materials mainly include van der Waals forces (permanent dipole-induced dipole forces and London dispersion forces), π - π interaction, electrostatic interaction, hydrophobic interaction, and hydrogen bonding [53]. The K_{ow} constant identifies a compound's hydrophobicity. Positive log K_{ow} values imply hydrophobic nature, with bigger values indicating greater hydrophobicity. In adsorption applications, molecules with $\log K_{ow} < 2.5$ have low sorption capability, $2.5 < \log K_{ow} < 4$ have moderate sorption capability, and $\log K_{ow} > 4$ have strong sorption capability [61]. Because CIP is a hydrophilic antibiotic with a low log K_{ow} value (0.038), hydrophobic interactions between CIP and nanofiber cannot be considered as the dominant removal mechanism for CIP from aqueous solutions. Pharmaceutical adsorption onto the heterogeneous catalyst surface was examined by Erdem H. and Erdem M. (2024), who discovered that adsorption ratios increased as log K_{ow} values increased [61]. The lowest removal efficiencies were 38% for CIP, which had the lowest log K_{ow} values. However, results presented in this study show that the hydrophilic CIP can adsorb on the nanofiber via electrostatic and π - π interactions as the dominant removal mechanisms. The π - π electron-donor-acceptor (EDA) interaction is considered one of the key driving forces due to the aromatic rings in CIP. At neutral pH, the benzene ring of CIP acts as a π electron-acceptor due to the strong electron-withdrawing effect of the fluorine group, while the nanofiber surface functions

as the electron-donor group [53]. It is suggested that electron-withdrawing groups strengthen the π - π interactions by lowering the electron density of the π electrons, thereby reducing the repulsive electrostatic interactions between the aromatic rings [56].

The adsorption mechanisms are affected by aqueous phase pH and closely related to CIP species and nanofiber zeta potential discharge. The zeta potential is an essential feature associated with the nanofiber surface charge that is closely linked to CIP elimination. Figure S2 showing pH-dependence of the zeta potential (ζ) of the nanofiber with 35 mol % C16A indicates that the nanofiber surface is negatively charged at all pHs between 4 and 10. The pH spectrum is critical for CIP removal using nanofibers via electrostatic interactions. At neutral pHs, the nanofiber has a ζ value of -32mV and the formal charge of CIP is zwitterionic form (Fig. 1) so that the drug elimination is mainly based on the electrostatic mechanism [52]. It is possible to deduce that CIP adsorption is facilitated by -OH groups, C=C bonds and C-H groups in aromatic rings, and phenolic C-O bonds. These chemical bonds in nanofiber were identified using FTIR. This shows that nanofiber is primarily composed of aromatics, and π - π interactions could happen within the adsorption process. According to [62] organic contaminants are mostly adsorbed by aromatics and aromatic π systems, which are the main components of pyrochars generated at 450 and 600 °C. Due to the presence of electron-withdrawing functional groups, aromatic π systems function as π acceptors, while hydroxyl groups in adsorbents act as electron donors [62, 63].

A CIP molecule has one benzene ring containing a fluorine group and two aromatic heterocyclic groups. The fluorine group linked to the benzene ring exhibits a significant electron-withdrawing capability, causing the aromatic ring become electron-deficient and functions as a π -acceptor [4]. Furthermore, the $-\text{NH}_2$ in aromatic heterocyclic groups may function as an electron donor. As a result, the π - π interactions among donor-donor, donor-acceptor, or acceptor-acceptor combinations may partially contribute to nanofiber's physical sorption capability for CIP.

Table 5 showed a comparison of the various nanofiber membrane by the recently published studies, which aimed at the removal of emerging pollutants. The literature presents methods for employing electrospun nanofiber membrane designs to retain emerging contaminants. The emerging contaminants are adsorbed by designed nanofibers. According to the majority of research in the literature, nanofibers serve as carriers for reactive nanoparticles with strong degradation capabilities. Once these nanoparticles are immobilized on the nanofiber surface, emerging pollutants can be effectively degraded through advanced oxidation, electrochemical or catalytic degradation processes. The adsorption

Table 5 The comparison of the fabricated polyacrylic acid hydrogel nanofibers with the recent works of literature regarding the removal of emerging pollutants

Nanofibers type	Emerging pollutants removal	Adsorption mechanisms	Ref.
Electrospun hydrophobically modified polyacrylic acid hydrogel nanofibers	Ciproloxacin	Electrostatic and π - π interactions	This study
Hypercrosslinked hydroxyl-rich porous organic polymers (HPOP)s	Diclofenac sodium, sulfamethoxazole, and acetaminophen	Hydrogen bonding, hydrophobic–hydrophobic, and π - π interaction	Ravi S. et al. (2022) [65]
Electrospun lignin nanofibers	Fluoxetine	Hydrogen bonding, van der Waals interactions, and π - π stacking.	Camiré A. et al. (2020) [11]
Electrospun polyacrylonitrile (PAN) nanofiber β -cyclodextrin (β -CD)	Bromophenol blue and atrazine	Hydrogen bonding,	Chabalala MB. et al. (2021) [66]
Porous β -cyclodextrin modified cellulose nano-fiber	BPA/BPF/BPS	Synergism of hydrophobic effects, hydrogen-bonding interactions and π - π stacking interactions	Lv Y. et al. (2021) [64]
Glutaraldehyde-crosslinked electrospun nanofibers	Tetracycline (TC)	-	Abdolmaleki AY. et al. (2018) [67]
Hydrophobic and polyporous electrospun fibrous membranes (EFMs)	Triclosan	Hydrogen bonding and pep bonding interactions	Xu J. et al. (2015) [68]

capacity and removal efficiency, as shown in the table, may be influenced by factors such as the materials and manufacturing process used in nanofiber synthesis, the conditions of adsorption, and the properties of the target pollutants. The study conducted by Lv Y. et al. (2021), revealed that the porous β -cyclodextrin modified cellulose nanofiber membrane (CA-P-CDP) had a lot of functional groups. The maximum adsorption capacities of bisphenol pollutants (BPA, BPS, and BPF) over CA-P-CDP were 50.37, 48.52, and 47.25 mgg⁻¹, respectively, at 25 °C [64]. In Camiré A. et al. (2020)'s study, the authors tested various ratios of lignin and PVA in electrospinning to achieve the optimal adsorption of fluoxetine residues from aqueous solutions. The best adsorption was achieved with a 50:50 ratio of alkali lignin

to the co-polymer poly(vinyl alcohol), resulting in a maximum adsorption capacity of 29 mgg⁻¹, which corresponds to approximately 38% of the contaminants being adsorbed by the nanofibers [11]. It is obvious that the novel electrospun hydrophobically modified polyacrylic acid hydrogel nanofiber membranes produced in this study showed good CIP removal. Furthermore, incorporating n-hexadecyl acrylate (C16A) into the polymer structure induce important modification in the nanofiber membrane properties.

Conclusion

In this study, fabrication of highly-interconnected nanofibrous membranes and its adsorption properties in the removal of CIP antibiotic were investigated. Electrospinning has been successfully applied in nanofiber fabrication. No significant shifts in the absorption bands are identified for the copolymer hydrogels after electrospinning, as indicated by FTIR results. According to SEM analysis, the optimal morphology characterized by both the desired fiber size and surface smoothness was observed in the electrospun hydrogel containing 35 mol % of C16A. This observation underscores the delicate balance required to achieve the desired morphology in P(AAc-co-C16A) hydrogels, with implications for their potential applications in various fields. Further investigations and fine-tuning of the molar fraction of C16A may offer opportunities for tailored hydrogel properties and applications. Zeta potential test shows that the nanofiber containing 35 mol % of C16A is negatively charged at all pHs. High CIP removal efficiencies of 98% were found using hydrogel nanofibers containing 35 mol % C16A. According to isotherm and kinetic model results, chemisorption predominated during the adsorption of CIP onto nanofiber. Owing to this, even though CIP serves as a hydrophilic antibiotic, it was not able to desorb from nanofiber with a high rate of success, indicating that the adsorption process is irreversible.

Overall, the developed hydrogel has the potential to yield promising results in the field of wastewater treatment. Moreover, the hydrogel nanofiber system offers a number of advantages, including low toxicity, biosafety, high adsorption capacity, and customizable properties for targeted adsorption, which makes it an effective option for specific contaminants. However, it is important to note that the system does have certain limitations. One such limitation is irreversible adsorption and potential fouling in complex matrices, which are inherent challenges associated with traditional bulk adsorbents. Future studies will concentrate on enhancing the reusability of these hydrogels, potentially through structural modifications or the incorporation of functional groups that facilitate desorption. Furthermore, it

is essential to assess the performance of these nanofibers in complex real wastewater matrices to determine their practical applicability. This could entail evaluating their effectiveness against diverse contaminants while addressing challenges such as fouling and chemical resistance. Finally, while extant literature provides evidence in support of the biocompatibility of analogous materials [69], it remains imperative that their biocompatibility be evaluated through experimental means. By optimizing these factors, it may be possible to harness the unique properties of hydrogel nanofibers to develop efficient, sustainable solutions for wastewater treatment, which could ultimately contribute to improved water quality and environmental sustainability.

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Data Availability Data is provided within the manuscript and supplementary information.

Declarations

Competing Interests The authors declare no competing interests.

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