

RESEARCH PAPER

Pharmaceutical Wastewater Treatment Using Nano/Micro-Structured Activated Carbon: Comparative Adsorption of Ampicillin and Phenylephrine and Reusability Assessment

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ABSTRACT

This study investigates the potential of low-cost activated carbon (AC) derived from fig (*Ficus carica*) leaves for the removal of two common pharmaceutical contaminants—Ampicillin (AMP) and Phenylephrine (PHN)—from aqueous solutions. The AC was synthesized via chemical activation using phosphoric acid (H_3PO_4) in a 1:1 weight ratio, followed by thermal treatment under nitrogen at 450 °C for 1 hour to enhance surface area and porosity. The resulting material was washed, dried, and characterized using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), and transmission electron microscopy (TEM), confirming a porous morphology with diverse surface functionalities and structural integrity. Batch adsorption experiments under varying conditions revealed significantly higher removal efficiency for AMP (92.8%) compared to PHN (71.3%), attributed to differences in molecular structure, polarity, and interaction mechanisms with AC. Adsorption was predominantly governed by hydrogen bonding with surface hydroxyl and phosphate groups introduced during activation. Reusability was evaluated over five adsorption–desorption cycles using ethanol as the regenerant. The AC retained over 85% efficiency for AMP and approximately 60% for PHN after five cycles, demonstrating good regeneration potential and structural stability. These results highlight fig leaf-derived activated carbon as a promising, sustainable, and selective adsorbent for pharmaceutical wastewater treatment applications.

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INTRODUCTION

Ampicillin is a β -lactam antibiotic of the aminopenicillin group, and it is one of the most frequently used drugs in human and veterinary medical practice because of the broad-spectrum activity against Gram-positive and some Gram-negative bacteria. It works by preventing bacteria from synthesizing cell walls, making this drug helpful in treating both respiratory and urinary infections, as well as GI and skin infections. Ampicillin is commonly used both orally and parenteral and is listed as a WHO Essential Medicine due to its clinical efficacy and widespread availability. Although it plays a vital role as a therapeutic agent, the widespread and non-selective use of ampicillin has generated serious environmental concerns [1, 2]. A large proportion of applied antibiotics, such as ampicillin, are excreted without metabolism and so are discharged into the municipal wastewater systems. Pharmaceuticals may not be removed effectively from water bodies by traditional wastewater treatment structures, and residual antibiotics are often introduced into surface water. This fuels the increasing crisis of antimicrobial resistance (AMR), representing a significant challenge for public health and the stability of ecosystems [3, 4].

Furthermore, ampicillin in aquatic environments can disturb macrobiotic, inhibit native biodegradation, and select for pathogenic bacteria, leading to the spread and evolution of resistance. As a result, the environmental stability of ampicillin as well as the potential for environmental impacts make it essential to develop reliable methods for detection, monitoring, and remediation. With increasing pharmaceutical pollution worldwide, and the nature of water as a medium for contaminant transportation, the study on the environmental behavior, fate, and remediation of ampicillin is a focus in environmental science and water treatment study. The purpose of this study is to fill this gap and also to consider new materials and techniques available for efficient removal of ampicillin from aqueous systems, which is very important to the environment and public health [5, 6].

Phenylephrine is a synthetic sympathomimetic drug primarily available as an oral tablet, nasal spray, or ophthalmic solution. It functions mainly as an α 1-adrenergic receptor agonist. It is used in some over-the-counter treatments for heart block,

orthostatic hypotension, and nasal congestion, as well as off-label for the treatment of hypotension in anaesthesia. As it is commonly present in over-the-counter and prescription drugs, phenylephrine can be commonly found in domestic wastewater and surface water systems [7]. The environmental occurrence of phenylephrine is becoming a more substantial concern for its possible ecotoxicological effects. As with most pharmaceutical ingredients, phenylephrine is only incompletely metabolised in the human body, and is excreted unchanged. Conventional WWTPs are not designed to remove pharmaceutical residues, thereby allowing the continuous release of phenylephrine and similar micropollutants into the aquatic environment. Such compounds are persistent in aquatic environments, interfere with aquatic microbial communities, cause alterations to hormonal and physiological processes in marine organisms and add to the problem of pharmaceutical pollution in general. Several techniques have been developed to solve this problem. Highly oxidizing technologies, such as Advanced Oxidation Processes (AOPs) (ozonation, Fenton reactions, photocatalysis), have been proven effective in degrading phenylephrine to less hazardous products. The cost-effective and efficient procedures that have been developed for the removal of phenylephrine from aqueous phases by adsorption, specifically on activated carbon and other porous materials, have also been studied. Furthermore, bio-treatments based on selected microbial strains or enzymatic systems can be potentially exploited to biodegrade phenylephrine in controlled contexts [8].

However, the removal of phenylephrine is still inadequate and inefficient, especially in large-scale or realistic wastewater treatment. Further studies on the environmental fate, behavior, and remediation of phenylephrine are imperative to mitigate its ecological effects and ensure water safety in the future.

Among them, carbon-based composites — exceptionally engineered and structured carbon materials, such as comb-shaped carbon or structured carbon — have received considerable attention for environmental remediation. Such materials include activated carbon, carbon nanotubes (CNTs), graphene and functional derivatives thereof, all of which are known to have high surface area, adjustable pore size, chemical stability and adjustable surface functional groups [9-11]. Unfortunately, the high cost and

low stability severely limit their applications in environmental remediation and use as a common adsorbent. In water treatment fields, SCMs are widely employed for the adsorption of organic dyes, heavy metals, pharmaceutical traces, pesticides, and emerging pollutants through processes such as physisorption, chemisorption, electrostatic interaction and π - π stacking. Their surface characteristics can be modified chemically or physically to enhance selectivity and adsorption capacity for specific pollutants. Furthermore, after being encapsulated in composite membranes or membrane systems, the removal efficiency and mechanical stability of these membranes can also be significantly enhanced [12]. In addition to adsorption, engineered carbon materials have been applied in AOPs, photocatalysis and electrocatalytic systems, serving as rheological spacers or active catalysts. Such multifunctionality not only captures pollutants but also degrades them, providing an environmentally friendly method for remediation. In view of the growing problem of persistent pollutants in natural environments, the fabrication and application of comb-like or ordered carbon materials offer a novel approach to addressing complex pollution issues [13-15]. This work is based on the synthesis, characterization, and environmental application of modified carbon material-based systems for removing Ampicillin and Phenylephrine. The chemical structures of these compounds shown in Fig. 1.

MATERIALS AND METHODS

Preparation of Activated Carbon from Fig Leaves Using Phosphoric Acid

Leaves of fresh fig (*Ficus carica*) are collected, washed with distilled water to free them from dust, dirt and extraneous material. The dust-free leaves were air-dried for 2–3 days and then dried at 105°C overnight in an oven to constant weight. The dried leaves are then ground into a fine powder using a mechanical grinder and sieved to ensure the particles are of uniform size, typically ≤ 1 mm. The dried biomass is impregnated with phosphoric acid (H_3PO_4 , usually 85%) in a 1/1 weight ratio (1 g of biomass per 1 mL of acid solution). The solution is stirred thoroughly and left at room temperature for 12–24 hours to ensure it thoroughly pervades and activates the precursor sufficiently. The impregnated biomass is loaded into a muffle furnace or tubular reactor and heated in a nitrogen (N_2) atmosphere to minimize oxidation of the material. The carbonization takes place at 350–400 °C for 1.5 to 2 h with a heating rate of 10°C/min. During pyrolysis, the H_3PO_4 participates in the pore generation through dehydration and cross-linking to generate a porous carbon matrix. The carbonized product is cooled and then washed with hot distilled water several times until the wash water pH is neutral (\sim pH 6.5–7) to remove unreacted acid and insoluble impurities. A further wash with a minimal amount of ethanol can be incorporated to eliminate organics and help with drying. The washed materials are subsequently

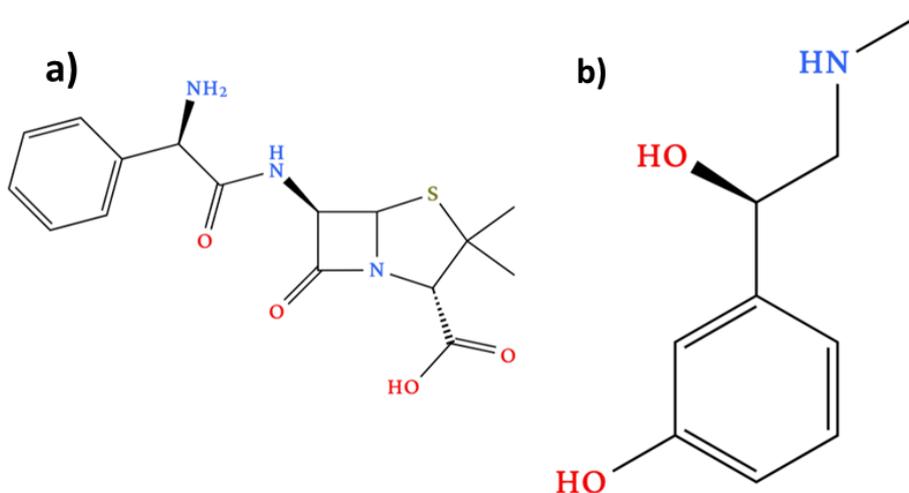


Fig. 1. Chemical structures in 2D view of a) Ampicillin and b) Phenylephrine compound.

dried in an oven at 105°C for 12 hours to obtain the final activated carbon. The end product is kept in sealed containers to prevent moisture absorption before characterization or use. as shown in Fig. 2.

RESULTS AND DISCUSSION

FESEM Analysis of Activated Carbon Pre and Post Adsorption

Fig leaves based activated carbon surface morphologies before and after adsorption of Ampicillin and Phenylephrine were analyzed using Field Emission Scanning Electron Microscopy (FESEM). The FESEM images of the sample before adsorption showed a very porous, irregular, and heterogeneous surface with numerous cavities and well-developed pore networks. This porous network is also inherently important for adsorption, resulting in a large surface area that facilitates the rapid diffusion and interaction of water contaminant molecules toward the active binding sites. Fig. 3a Surface morphology. The surface morphology of the samples before and after adsorption of Ampicillin were compared in FESEM images. Most of the pores were partly or entirely blocked, and the surface was smoother and denser, indicating that Ampicillin molecules had entered entirely the available sorption sites

on the goethite. The degree of surface coverage and morphological change also suggests that activated carbon has a strong interaction with Ampicillin, which may be resonating with the much higher removal efficiencies found during the adsorption studies. Fig. 4b. In contrast, the surface modifications were lower after Phenylephrine adsorption. Although some of the pores seemed occluded or distorted, the structuration was less destroyed in comparison with the Ampicillin-loaded sample. This implies a less strong binding or only surface interaction between Phenylephrine and the activated carbon. This reduced surface area coverage and open pore retention might be related to the lower adsorption efficiency of Phenylephrine [14, 16]. The differences in post-adsorption morphology can be interpreted in terms of the structural and chemical properties of the drug molecules. Ampicillin has greater polarity as well as more functional groups that can be hydrogen bonded and have electrostatic interaction, which resulted in stronger matrix formation on the carbon surface. In contrast, Phenylephrine probably interacted via weaker interactions with the surface, resulting in less functionalization. Fig. 3c. These FESEM results indicate that adsorption efficiency is closely



Fig. 2. Preparation of Nano/Micro-Structured Activated Carbon from Fig Leaves Using Phosphoric Acid.



related to the extent of surface interaction, which are visually demonstrated by the alterations of surface morphology of the adsorbed media before and after adsorption [17].

TEM Analysis of Acid-Activated Carbon: Surface Morphology

TEM analyzed the surface morphology of the acid-activated carbon. Nanostructure and surface

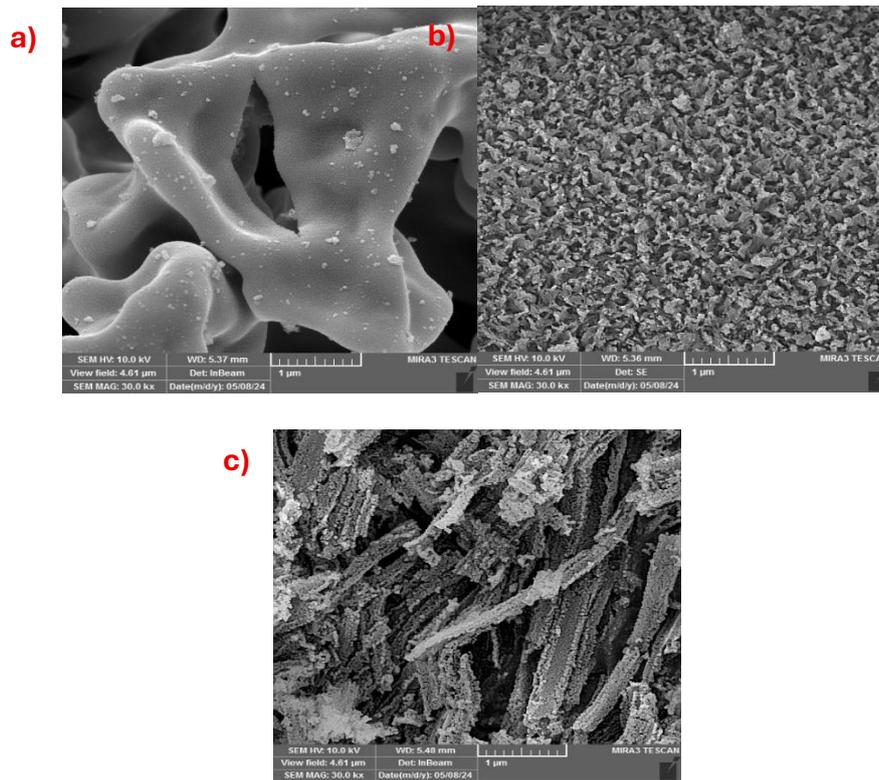


Fig. 3. a) Fig leaves-based Nano/Micro-Structured Activated Carbon, b) after adsorption of the Ampicillin drug, c) Phenylephrine drug.

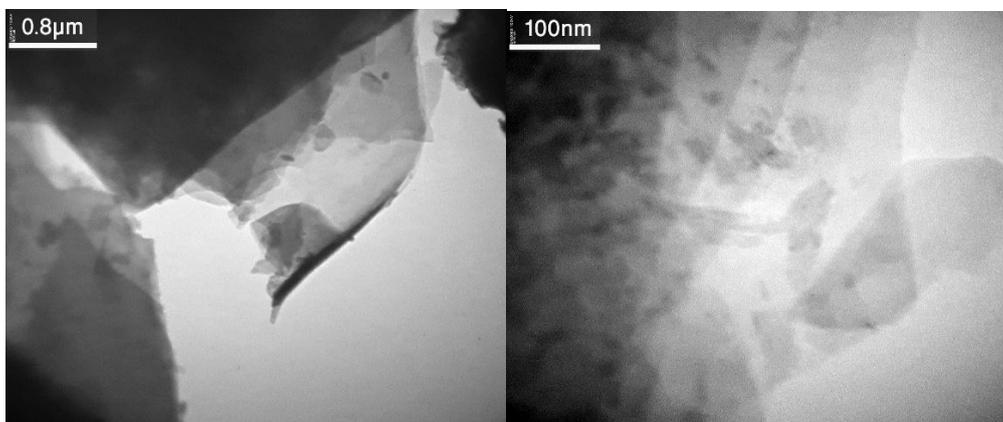


Fig. 4. TEM Analysis of Nano/Micro-Structured Activated Carbon.

morphology of activated carbon prepared from fig leaves by phosphoric acid activation were studied by Transmission Electron Microscopy (TEM). TEM showed a disordered, highly porous carbon texture typical of the chemical activation matrix of amorphous carbon structures. The acid-activated carbon featured an anatomic, sponge-like structure, with a honeycomb of mesoporous interconnections and voids at the nanometer level, identified. The particles were porous with pores from micro- to mesopores that allowed the generation of a high specific surface, which is necessary for adsorption-related phenomena [9]. The dark areas shown in the TEM micrographs are dense carbon areas, whereas the lighter regions match well with the developed pore channels, as shown in Fig. 4.

Furthermore, the significant contrast in both edges and pore walls may indicate that the activation protocol led to complete development of surface defects and function groups that eventually bring better adsorption properties. The lack of a crystalline fringe in the image is also consistent with amorphous material, which is characteristic of a chemically activated biochar. Overall, the TEM results further indicate that phosphoric acid activation can prepare a non-crystalline carbon structure with a large number of accessible adsorption sites, making it applicable

for removing pollutants from aqueous solutions [13, 18].

XRD Analysis of Acid-Activated Carbon

The X-ray diffraction (XRD) pattern of the acid-activated carbon derived from fig leaves reveals essential information about its structural properties and degree of crystallinity. The diffraction pattern is characterized by broad, low-intensity peaks, which are typical of amorphous carbonaceous materials. A broad diffraction band typically appears around $2\theta = 20^\circ\text{--}30^\circ$, corresponding to the (002) plane of disordered graphitic structures. This peak indicates the presence of randomly arranged aromatic carbon layers with poor long-range order [19]. A second, weaker and broader band may also appear near $2\theta = 40^\circ\text{--}45^\circ$, associated with the (100) plane, which represents in-plane structural disorder within the carbon matrix. Fig. 5

The absence of sharp and well-defined peaks in the XRD pattern confirms that chemical activation using phosphoric acid disrupts the crystalline domains, leading to the formation of an amorphous, highly porous structure. This disordered arrangement is desirable in adsorption applications, as it is typically associated with a high surface area, abundant active sites, and enhanced accessibility of adsorbate molecules. Overall, the

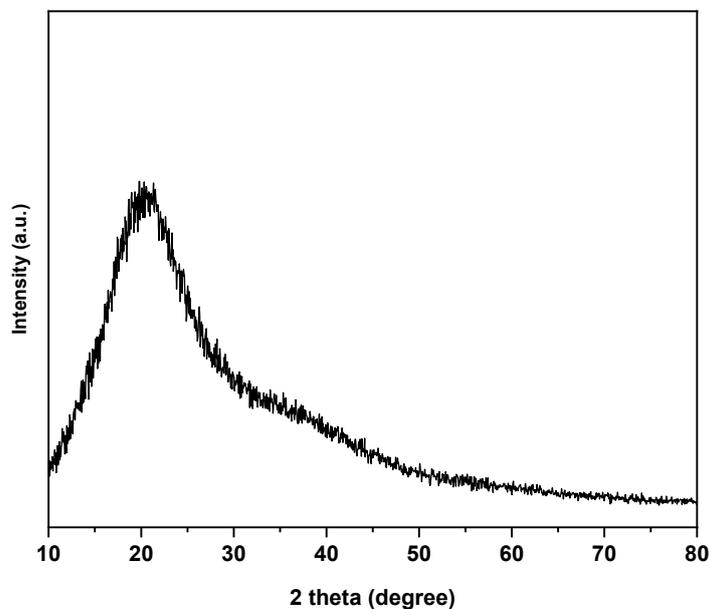


Fig. 5. XRD Analysis of Nano/Micro-Structured Activated Carbon.

XRD results demonstrate that the acid activation process successfully produced amorphous activated carbon with minimal graphitic ordering, which is consistent with the material's high adsorption potential for pollutants in aqueous solutions [20].

Regeneration and Reuse of the Nano/Micro-Structured Activated Carbon

Regeneration studies are essential for assessing the behavior and practical feasibility of adsorbent materials over the long term. On a commercial level, the reusability of the adsorbent with negligible loss in performance directly dictates the sustainability and profitability in operation. In this study, the reusability of activated carbon for Ampicillin removal was determined by five successive adsorption–desorption cycles with optimal experimental conditions (shown in Fig. 6).

After each adsorption cycle, the spent activated carbon was separated from the solution by filtration, washed well with ethanol to desorb the Ampicillin molecules, and dried before it was employed again. The regenerated activated carbon

was subsequently reused for the treatment of fresh Ampicillin solutions under the aforementioned optimum conditions (pH 7, 30° C, 0.05g dosage, 80 mg/L initial concentration, and 120 minutes of contact time). After repetitive usage, a slight decline in adsorption capacities in the following cycles was observed; however, above 90% of the removal ability of the adsorbent was retained after five adsorption–desorption cycles. This slight reduction can be attributed to partial pore plugging or irreversible binding sites; however, the overall performance remained strong. The results demonstrate that the activated carbon exhibits good regeneration ability and structural stability when used for treating pharmaceutical wastewater [18]. As well, the ability to regenerate and reuse activated carbon drastically decreases the necessity of periodic replacement so that material costs can be reduced and generation of secondary waste can be minimized. This makes activated carbon an effective and environmentally benign adsorbent, and therefore, a potential sorbent for use in industrial wastewater treatment systems aimed at treating pharmaceutical-based

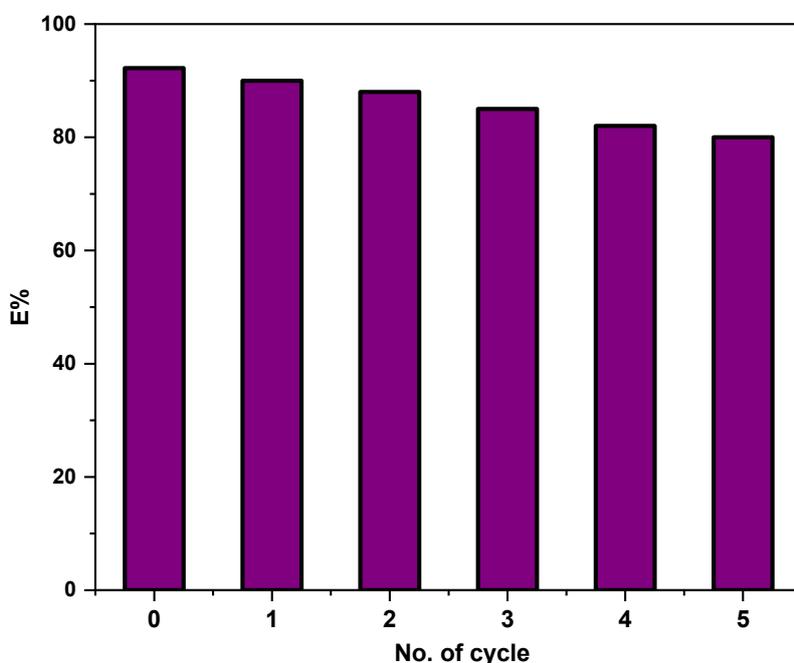


Fig. 6. Regeneration and Reuse of the Activated Carbon from the Ampicillin drug: optimum conditions (pH 7, 30°C, 0.05g dosage, 80 mg/L initial concentration, and 120 minutes of contact time).

pollutants, such as ampicillin [21].

Phenylephrine Adsorption by Regeneration of Nano/Micro-Structured Activated Carbon

The regeneration of CDAD after adsorption of phenylephrine was much less than the adsorption efficiency before reaching the maximum. The decrease in the reusability is originated from the strong and possibly irreversible adsorption of phenylephrine molecules onto the surface of the activated carbon that restricts their successful desorption during the regeneration processes [22].

As a typical drug, phenylephrine is a phenylethanolamine derivative, and its structure includes an aryl and polar group (hydroxyl, amine), which could produce strong hydrogen bonding and electrostatic attraction with the activated carbon surface containing oxygen functional groups (such as -OH, -COOH) and a π -conjugated system. It is possible that both scenarios, due to partial chemisorption or deep infiltration

into micropores, increase the difficulty of the desorption processes, even with conventional regenerates such as ethanol or water. Also, the molecular structure and moderate volatilisation of phenylephrine might result in its adsorbent remaining on the adsorbent. Steric hindrance from the functional groups can also hinder the easy access of desorption agents, especially in the micropore spaces. Over a series of regeneration doses, these factors accumulate, leading to a gradual decline in the number of active sites and, consequently, an improvement in the reusability of the material. The noted low regeneration efficiency suggests a potential disadvantage of using activated carbon in the elimination of phenylephrine, particularly in a reusable system [5, 18]. This discovery indicates that the carbon surface should be chemically modified for rapid desorption, and/or an alternative method of regeneration, such as thermal treatment or an advanced oxidation process, should be explored for the efficient reuse of the adsorbent in practical

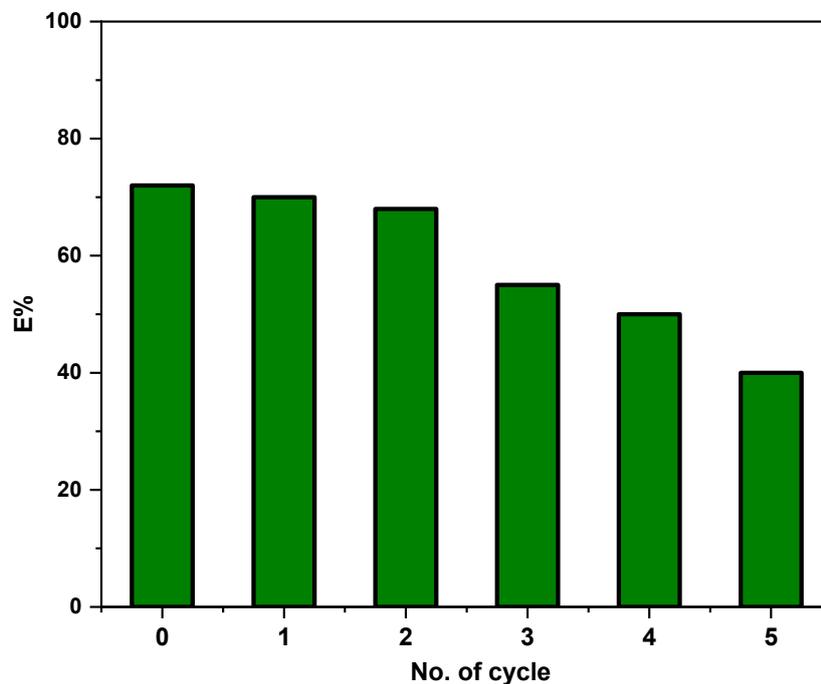


Fig. 7. Regeneration and Reuse of the Nano/Micro-Structured Activated Carbon from the Phenylephrine drug: optimum conditions (pH 7, 30° C, 0.05g dosage, 80 mg/L initial concentration, and 120 minutes of contact time).

applications, as shown in Fig. 7.

Study and Comparison of Ampicillin and Phenylephrine Adsorption on Nano/Micro-Structured Activated Carbon

The adsorption characteristics of two pharmaceuticals, Ampicillin and Phenylephrine, the adsorption on activated carbon under the same experimental conditions. There was a high removal efficiency of Ampicillin, with significantly lower adsorption ability and regeneration capacity of Phenylephrine. The higher adsorption of Ampicillin can be explained by the existence of representatives of several polar functional groups (e.g. carboxyl, amine and β -lactam ring) in its molecular structure, which readily enable strong electrostatic interactions, H-bonding and π - π stacking with the surface functionalities of AC. Furthermore, the moderate molecular size and hydrophilicity of Ampicillin favour its diffusion processes into the porous carbon, maximising surface accessibility to, and interaction with, active sites. By contrast, Phenylephrine exhibited a weak adsorption behaviour [11, 23].

. Even though it also has polar groups (like -OH, -NH₂), the (physical or chemical) nature or

distribution of the polar group can result in weaker carbon-structure interactions. Furthermore, the steric hindrance and depopulated electron cloud states (reduced aromaticity) in the molecular conformation of Phenylephrine, compared to Ampicillin, might further prevent its access to micropores and active binding sites. This makes the surface less attractive, leading to decreased uptake [24].

Another key parameter affecting this difference is the extent of the desorption in the regeneration cycles as shown in Fig. 8. Although Ampicillin was well desorbed and exhibited stable removal efficiency in consecutive cycles, Phenylephrine adsorption capacity dramatically decreased after regeneration. This indicates a more substantial or irreversible bonding of Phenylephrine to the activated carbon surface (possibly as a result of the deep penetration into the micropores or the partial chemisorption), and thus a partial lack of recyclability [3, 13].

In brief, the higher adsorption performance of Ampicillin on activated carbon compared to Phenylephrine is mainly attributed to the differences in molecular framework, polar nature, and pore accessibility, and the role of adsorbent-

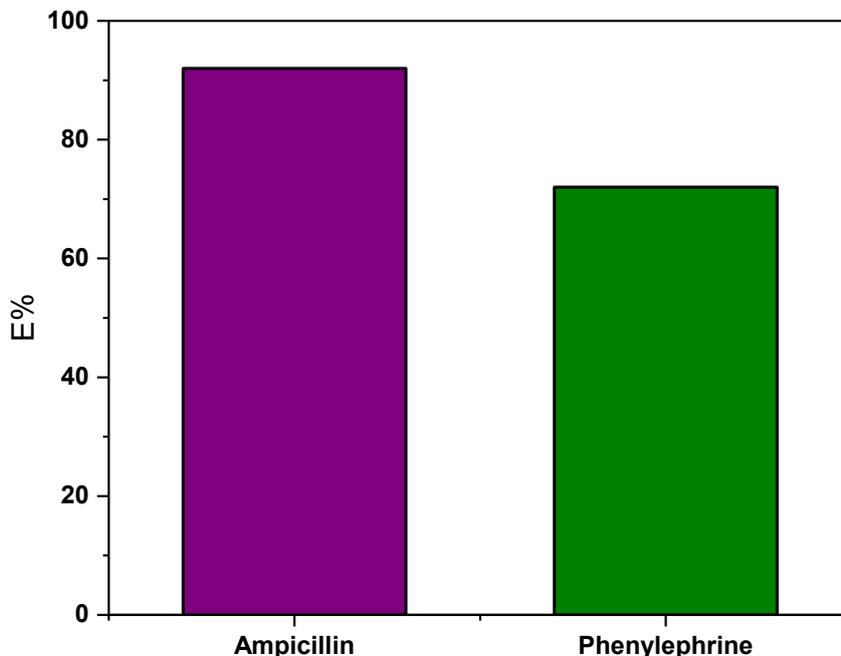


Fig. 8. Comparison of Ampicillin and Phenylephrine Adsorption on Nano/Micro-Structured Activated Carbon.

adsorbate interaction forces. These results emphasise the significance of molecular design in the suitability of A for the removal of selected pharmaceutical contaminants from water systems [21, 25].

CONCLUSION

This work showed that acid-activated carbon was successfully produced from fig plant (*Ficus carica*) leaves using phosphoric acid as an activating agent. The AC had a high degree of porosity and good surface chemistry, which made it a powerful ADS for the elimination of the PPCPs from aqueous solutions. The adsorption experiments illustrated that the material had a higher removal rate for Ampicillin than Phenylephrine. This variation is attributed to ampicillin's higher polarity and more diverse functional groups, which allow for better connections, hydrogen bonds, and electrostatic attraction with the functional groups on the surface of the activated carbon. However, Phenylephrine was found to be less or not adsorbed to SC with its much lower binding affinity to the carbon surface and a possible steric effect. Cyclic adsorption-desorption operation of Ampicillin established that the activated carbon possessed an excellent adsorption capacity, and the resultant efficiency was not significantly reduced over various adsorption-desorption cycles, implying the structural robustness and surface reusability. The regeneration efficiency for Phenylephrine was, however, substantially less, indicating stronger or less reversible binding or less desorption inertia. All in all, fig leaf cell-derived activated carbon is a low-cost, eco-friendly, and efficient material for the adsorption removal of specific pharmaceutical pollutants, especially Ampicillin in polluted water, which has considerable potential in the application of cyclic utilisation for water treatment.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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