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RESEARCH ARTICLE

Sustainable removal of amoxicillin and tetracycline from aqueous media using Pine-Leaf derived activated carbon: Adsorption performance and regeneration potential

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ABSTRACT

This study evaluates the feasibility of using low-cost pine (Pinus spp.)-originated PLAC prepared from leaves for the adsorption of two frequently reported pharmaceuticals, Amoxicillin (AMX) and Tetracycline (TC), from water. The activated carbon was prepared by chemical activation (H_3PO_4). Dried and powdered pine needles were impregnated with H_3PO_4 at a mass ratio of 1:1 to increase the porosity and surface reactivity and thermally treated in a nitrogen atmosphere at 450 °C for 2 hr. The material was washed and dried thoroughly to provide a high surface area adsorbent. The activated carbon was characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction (XRD), Thermogravimetric analysis (TGA), Field emission scanning electron microscopy (FESEM) and Transmission electron microscopy (TEM). These analyses confirmed the preparation of a stable, microporous carbon matrix with a rich surface functional group. Adsorption studies were conducted using a series of batch adsorption experiments, and several operating conditions were tested to determine the removal percentage of the pharmaceuticals. The adsorption capacity of AMX was larger than that of TC, and its corresponding optimum removal efficiency was 97.44% for AMX and 81.83% for TC, respectively. The higher proportion of AMX removal is attributed to its molecular polarity, functional group interactions, and higher adsorption affinity with the activated carbon material. The adsorption mechanism was suggested to involve hydrogen bonding with the hydroxyl and phosphate groups formed after activation, as well as electrostatic interactions between the charged drug molecules and the adsorbent surface. Studies regarding the regeneration of the material using water were carried out over five adsorption–desorption cycles. The adsorbent exhibited removal efficiencies exceeding 85% for AMX and approximately 60% for TC, demonstrating its reasonable structural stability and moderate recyclability. The degradation in TC removal may be attributed to its low adsorption and assembly, likely due to pore blockage by TC molecules. In summary, it is demonstrated that pine-leaf-derived activated carbon is a promising, environmentally friendly, and cost-effective adsorbent for the adsorption of pharmaceuticals from wastewater, exhibiting potential for regeneration and pollutant-specific performance.

Keywords: Amoxicillin (AMX); Tetracycline (TC); drugs, regenerations; removal, adsorption

1. Introduction

Pharmaceuticals are detected in environmental water with increasing frequency, and their presence has raised growing concerns due to their chronic effects, potential bioaccumulation, and adverse impact on both the aquatic ecosystem and human beings. Amoxicillin (AMX) and Tetracycline (TC), extracted from pharmaceutical manufacturing, hospital reservoirs, and domestic wastewaters, are considered one of the most commonly found antibiotics in wastewater[1, 2]. These pollutants are recalcitrant and cannot be removed entirely by ordinary water-treatment processes, leading to emerging research on the sustainable treatment of these contaminants. Adsorption is recognised as the most advanced and economically viable technique for removing pharmaceutical pollutants from water, due to its simplicity, low cost, and ease of operation[3, 4]. Following numerous adsorbents, activated carbon (AC) is one of the most used adsorbents, but mainly when it is obtained from cheap sources of primary, renewable origin material (biomass). In recent studies, research has focused on valorising agricultural remains for AC synthesis with favourable environmental aspects and reducing treatment costs[5]. Pine (Pinus spp.) was the first taxon of interest in this sense. Leaves as a rich and untapped lignocellulosic biomaterial for AC production. One of the most commonly characterised activated carbons is chemically modified with phosphoric acid (H_3PO_4) and increased porosity, surface area, and the diversity of functional groups, which is an essential factor for a high adsorption performance[6-8].

This work focuses on the performance of activated carbon derived from pine leaves for removing AMX and TC from aqueous solutions. The PLAC was prepared by chemical activation with phosphoric acid and was characterized by various physicochemical methods such as XRD, TGA, FTIR, TEM, and FESEM. Removal efficiencies were determined through batch adsorption studies conducted under multiple operating conditions. This adsorbent was also tested for its reusability over several cycles to evaluate its practical benefits for sustainable water treatment. The results will provide additional support for the use of inexpensive and environmentally friendly adsorbents for the reclamation of water contaminated with pharmaceuticals.

2. Experimental part

2.1. Preparation of pine leaves-based activated carbon through phosphoric acid

The PLAC was prepared from pine needles (Pinus spp.) and pine combs, as well as eucalyptus leaves (Eucalyptus spp.), through chemical activation using phosphoric acid (H_3PO_4) as the activating agent. The collected biomass was thoroughly washed with distilled water to remove surface impurities, then air-dried for 48 hours and oven-dried at 105 °C for 24 hours. The dried material was ground and sieved to a particle size of less than 1 mm. For activation, 5 g of the powdered biomass was impregnated with H_3PO_4 and stirred to ensure uniform saturation, followed by soaking at room temperature for 12 hours to facilitate chemical diffusion. The impregnated material was placed in a lidded ceramic crucible and carbonized in a muffle furnace under a nitrogen atmosphere. Carbonization was carried out at 450 °C for 2 hours with a heating rate of 10 °C/min. After cooling to room temperature, the carbonized product was washed with hot distilled water until the pH of the filtrate reached near neutrality (pH 6.5–7.0), ensuring the removal of residual acid and soluble impurities. Optional rinsing with ethanol was performed to eliminate remaining organic matter. Finally, the activated carbon was oven-dried at 105 °C for 12 hours, ground into fine powder, and stored in a vacuum desiccator for characterization and adsorption experiments. As shown in **Figure 1**

Figure 1. Preparation of pine leaves-based activated carbon by phosphoric acid

3. Result and Discussion

3.1. FESEM image analysis of PLAC before and after adsorption

The surface morphology of activated carbon from pine leaves was characterised using a Field Emission Scanning Electron Microscope (FESEM) before and after adsorption of AMX and TC. As seen in **Figure 2a**, the original surface was highly porous, uneven, and heterogeneous, consisting of various interconnected pores and cavities [9, 10]. This highly porous network is a crucial factor for the adsorbent, providing a high surface area to enhance the diffusion and attachment of pollutant molecules to active sites. After adsorption of AMX (**Figure 2b**), the order of surface morphology had been altered. The majority of the pores have been wholly or partially obstructed, and the surface has become visibly smoothed and densified. These differences indicate that Amp molecules were trapped and adsorbed onto carbon matrices. The amount of surface coverage suggests that the participation of the drug and the adsorbing agent is relatively high, which is in agreement with the high removal percentage obtained in batch adsorption experiments[11, 12].

On the other hand, samples loaded with TC (**Figure 2c**) showed only slight surface modifications. Although some pores were blocked and a few surfaces altered, the structure remained integrated compared to the AMX-treated sample[13]. This suggests a weaker and/or less surface-governed interaction with the activated carbon in Pronase compared to PLAC, as evidenced by its lower adsorption efficiency. This difference in the post-adsorption morphology can be attributed to the distinct chemical structures and functional groups of the two drugs. AMX exhibited stronger interactions with the adsorbent surface due to its higher polarity and the presence of more sites for hydrogen bonding and electrostatic interactions. In contrast, TC presumably adsorbs through weaker, non-specific interactions with the particle surface, leading to lower surface coverage and adsorption. In general, the FESEM results indicate that the adsorption capability is directly related to surface interaction, which can be observed from the morphological variations before and after the adsorption[14].

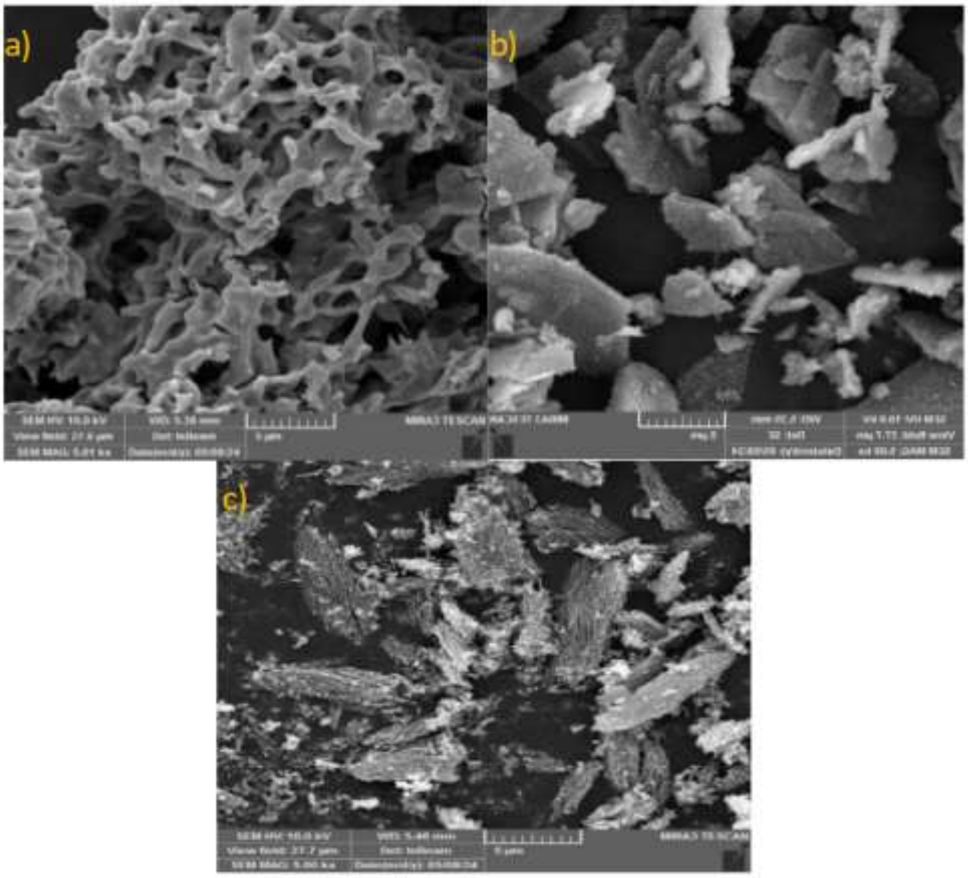

Figure 2. Field emission scanning electron microscope (FESEM) a) PLAC, b-c) PLAC before and after adsorption of AMX and TC, respectively

3.2. TEM of pine Leaf-Derived Acid-Activated carbon

The texture and nanostructure of the acid-activated carbon prepared from pine leaves by phosphoric acid activation were characterised in Transmission Electron Microscopy (TEM). **Figure 3**: TEM images indicate that the carbon structure of the as-synthesised PLAC is disordered and highly porous, similar to other amorphous carbon materials obtained by chemical activation [15]. The activated carbon exhibited a spongy structure characterised by interconnected micropores and mesopores. This structure, resembling a honeycomb, offers a superior specific surface area, the factor of primary importance for efficient absorption of pollutants from water. The dark regions in TEM images are attributed to carbon-dense areas. In contrast, the bright areas are due to well-developed pore channels, which further manifests the high porosity of the catalysts[16].

The high contrast of pore walls and edges indicates that phosphoric acid activation successfully generated structural defects and surface functional groups, contributing to the improvement in the adsorptive capacity of the adsorbent. Moreover, the absence of crystallite fringes in the micrographs indicates the non-crystalline (amorphous) nature of the carbon, as is typical of chemically activated biochar. In conclusion, TEM observations reveal that the structure of pine leaf activated carbon is highly porous and amorphous, with numerous surface features, making it a potential material for adsorbing various environmental pollutants from aqueous media[17, 18].

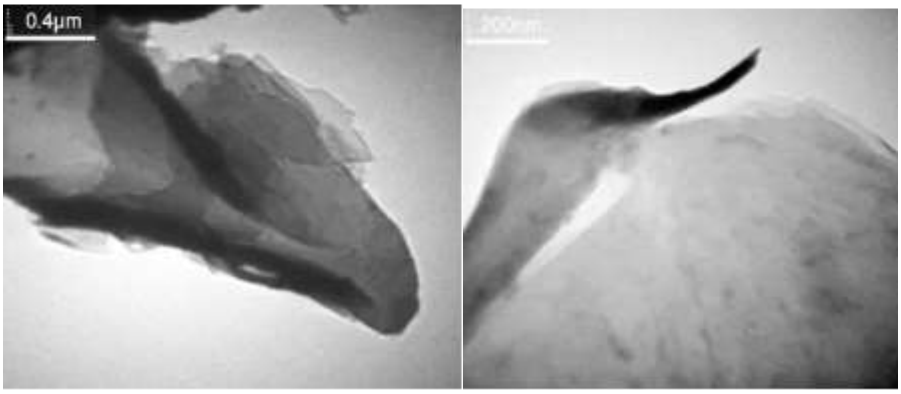

Figure 3. TEM image of PLAC surface

3.3. XRD Analysis of Pine Leaf-Derived Acid-Activated Carbon

Figure 4 shows the XRD pattern of acid-activated carbon prepared from pine leaves, which exhibits broad, low-intensity peaks indicative of an amorphous carbon structure. A prominent diffraction band around 2θ = 20°–30° corresponds to the (002) plane of disordered graphitic layers, while a weaker band near 2θ = 40°–45° suggests in-plane structural disorder. The absence of sharp crystalline peaks confirms that phosphoric acid activation disrupts graphitic domains, yielding a non-crystalline, porous carbon matrix. This disordered structure enhances surface area and active site accessibility, making the material highly suitable for the adsorption of aqueous pollutants [19, 20].

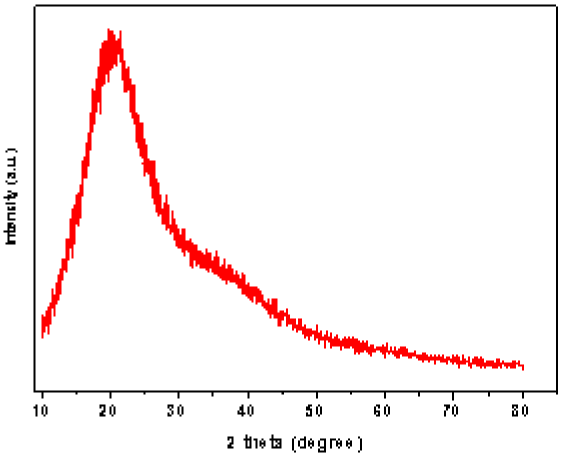

Figure 4. XRD analysis of PLAC surface

3.4. TGA curve for pine Leaf-Derived Acid-Activated carbon

The thermogravimetric analysis (TGA) curve of the phosphoric acid-activated carbon shows minimal weight loss across the temperature range from room temperature to 600°C, indicating high thermal stability (**Figure 5**). The initial slight decrease in weight (below 100 °C) can be attributed to the evaporation of physically adsorbed moisture or volatile components. As the temperature increases beyond 150°C, only a minor and gradual weight loss is observed, suggesting the absence of thermally unstable functional groups or residual organics. The total weight loss remains below approximately 10–15%, even at temperatures up to 600 °C, confirming that the prepared activated carbon possesses a highly stable and condensed carbonaceous structure. This thermal behavior is typical of materials that have undergone effective chemical activation, particularly with H_3PO_4, which promotes the formation of a cross-linked, aromatic carbon matrix that resists decomposition. The result supports the suitability of the prepared activated carbon for high-temperature applications, such as adsorption in thermal or harsh environments.

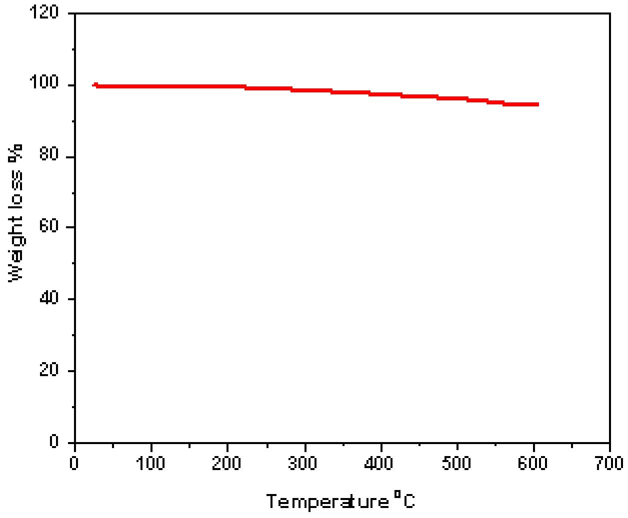

Figure 5. Thermogravimetric analysis (TGA) curve of PLAC

3.5. FTIR spectra of the pine leaf activated carbon (PLAC)

The FTIR spectra of the pine leaf activated carbon (PLAC) and its dye-loaded forms (PLAC-AMX and PLAC-TC) provide insight into the surface functional groups and their interaction with the adsorbed molecules **Figure 6**. PLAC (blue line): The spectrum shows characteristic peaks around 3400 cm^{-1}, attributed to O–H stretching vibrations from hydroxyl groups or adsorbed moisture. Peaks near 2920 cm^{-1} are typically assigned to C–H stretching in aliphatic chains. A broad band around 1650 cm^{-1} corresponds to C=O stretching, likely from carboxyl or carbonyl groups. Peaks in the 1000–1200 cm^{-1} region suggest C–O stretching vibrations associated with alcohols, ethers, or esters, which are common on the surface of activated carbon.

PLAC-AMX and PLAC-TC (red and black lines, respectively): After adsorption of (AMX) and (TC), notable changes in intensity and position of several bands are observed: The O–H and C=O bands exhibit shifts or reduced intensity, indicating possible hydrogen bonding or electrostatic interactions between the adsorbent surface and the dye molecules. New peaks or intensified bands may appear in the 1500–1600 cm^{-1} range, which can be attributed to the aromatic ring vibrations or amide groups present in AMX and TC. The changes in the fingerprint region (below 1200 cm^{-1}) further support the binding of functional groups from the dye molecules onto the carbon surface. These spectral changes confirm the successful adsorption of AMX and TC onto PLAC, likely through multiple mechanisms including hydrogen bonding, π–π interactions, and electrostatic attraction.

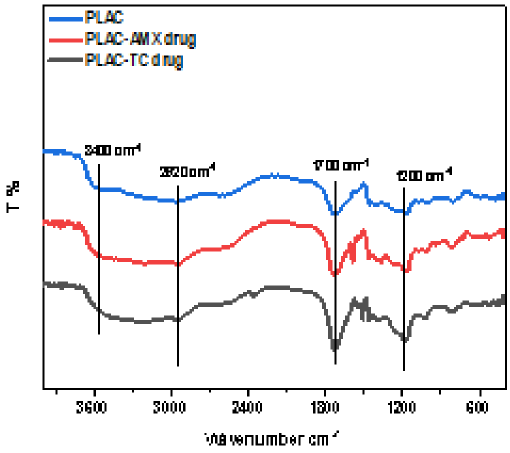

Figure 6. FTIR spectra of the pine leaf activated carbon (PLAC)

3.6. Regeneration and reusability of Pine Leaf-Derived Activated Carbon

The regeneration of activated carbon after adsorption is a critical factor for the economic and environmental sustainability of water treatment processes. Effective regeneration allows the recycling of spent adsorbents, minimizes operational costs, and helps prevent secondary pollution. In this study, desorption experiments were conducted to evaluate the reusability of pine leaf-derived activated carbon after adsorbing (AMX) and (TC). Various desorption agents were tested, including H_2SO_4, NaOH, H_3PO_4, HCl, HNO_3, ethanol, methanol, and distilled water, at three different concentrations (0.01, 0.05, and 0.1 N). The desorption performance was evaluated to understand both the adsorption–desorption mechanism and the chemical stability of the adsorbent. Among all tested agents, distilled water showed the highest regeneration efficiency, achieving complete desorption (close to 100%) for AMX and high recovery rates for TC, as summarized in **Tables 1 and 2**. These findings confirm that the activated carbon maintains its structural integrity and adsorption capacity over multiple cycles, demonstrating its practical applicability as a reusable and cost-effective adsorbent for pharmaceutical-contaminated water.

Table 1. Comparison of the desorption efficiency of several types of solutions for the AMX drug onto the surface of the PLAC

Adsorption/ Desorption (0.01N)	recovery percentage R%	Adsorption/ Desorption (0.05 N)	recovery percentage R%	Adsorption/ Desorption(0.1 N)	R%
fresh	97	Fresh	97	Fresh	97
water	96	water	96	water	96
Ethanol	90	Ethanol	86	Ethanol	82
Methanol	90	Methanol	85	Methanol	81
H_3PO_4	86	H_3PO_4	77	H_3PO_4	70
HCl	80	HCl	70	HCl	67
H_2SO_4	77	H_2SO_4	66	H_2SO_4	60
HNO_3	70	HNO_3	61	HNO_3	57
NaOH	44	NaOH	33	NaOH	30

Table 2. Comparison of the desorption efficiency of several types of solutions for the TC drug onto the surface of the PLAC

Adsorption/ Desorption (0.01N)	recovery percentage R%	Adsorption/ Desorption (0.05 N)	recovery percentage R%	Adsorption/ Desorption(0.1 N)	R%
fresh	81	Fresh	81	Fresh	81
water	80	water	80	water	80
Ethanol	77	Ethanol	70	Ethanol	67
Methanol	70	Methanol	63	Methanol	58
H_3PO_4	66	H_3PO_4	55	H_3PO_4	50
HCl	65	HCl	50	HCl	44
H_2SO_4	50	H_2SO_4	44	H_2SO_4	40
HNO_3	44	HNO_3	40	HNO_3	37
NaOH	30	NaOH	28	NaOH	28

3.7. Desorption behaviour and regeneration efficiency

In this study, a range of desorption agents—H_2SO_4, NaOH, H_3PO_4, HCl, HNO_3, ethanol, methanol, and distilled water—were evaluated for their ability to regenerate pine leaf-derived activated carbon after adsorption of (AMX) and (TC). Among these agents, distilled water demonstrated the highest regeneration efficiency for AMX, outperforming both acidic and basic eluents[21]. This superior performance is attributed to the reversible nature of the interaction between AMX and the carbon surface, primarily driven by hydrogen bonding and weak electrostatic forces. These interactions are more easily disrupted by water molecules, enabling the desorption of AMX without damaging the carbon structure. In contrast, TC showed lower regeneration efficiency, even with more aggressive desorption agents. This suggests that TC interacts more strongly or irreversibly with the adsorbent, possibly through multi-point binding, chelation, or steric entrapment within micropores, which hinders its release during regeneration. Overall, the results indicate that distilled water is a practical, eco-friendly, and non-destructive regenerant, particularly for pharmaceuticals like AMX that exhibit moderate surface interactions. The lower regeneration of TC highlights the influence of molecular structure and binding strength on desorption behavior as shown in **Figure 7**[22].

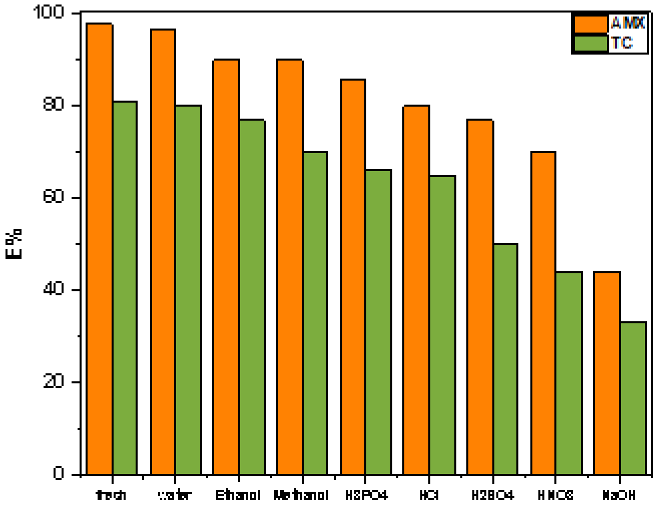

Figure 7. Reusability of PLAC surface in AMX and TC Adsorption with several reagents: (Exp. 100 mg/L, 0.05 g , pH 7 , and T 25 ° C)

3.8. Reusability of PLAC in AMX and TC adsorption

The reusability of acid-activated carbon derived from pine leaves was evaluated through six consecutive adsorption–desorption cycles using distilled water as the regenerating agent, under optimal adsorption conditions. As shown in **Figure 8**, the adsorbent retained over 97.88 % removal efficiency for AMX and above 81.23 % for TC after four cycles, indicating strong regeneration capacity and surface stability. However, a gradual decline in performance was observed after the fourth cycle, particularly in the removal of TC. This reduction in efficiency can be attributed to several factors: saturation or blockage of active sites, partial degradation of functional groups, and structural deterioration of the porous carbon matrix during repeated adsorption–desorption operations. These changes can reduce the number of accessible adsorption sites and alter the surface chemistry of the activated carbon, thereby limiting its interaction with drug molecules. Despite the observed decrease, the carbon remained effective through six cycles, demonstrating its renewable nature, mechanical resilience, and suitability for repeated use in pharmaceutical wastewater treatment[23-25]. These findings confirm that pine leaf-based activated carbon is a promising, low-cost, and sustainable adsorbent, particularly for antibiotics such as AMX and TC in aqueous environments.

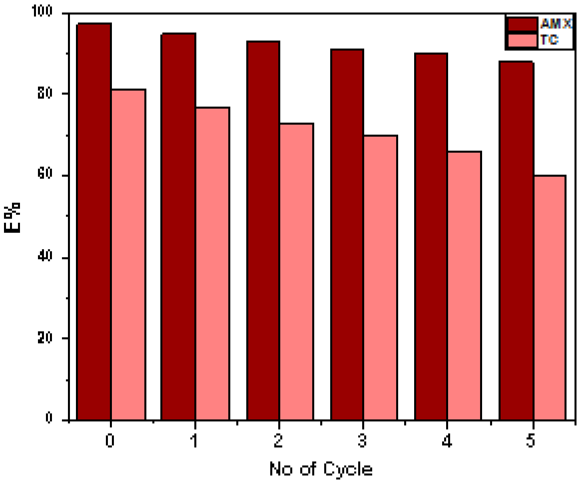

Figure 8. Reusability of PLAC in AMX and TC Adsorption (Exp. 100 mg/L, 0.05 g , pH 7 , and T 25 ° C)

3.9. Comparative adsorption performance: Amoxicillin vs. Tetracycline

The potential of pine leaf activated carbon (PLAC) for adsorbing two commonly used antibiotics, (AMX) and (TC), was examined. The PLAC was found to have notably higher removal performance for AMX than for TC in the comparison of the results under the same experimental conditions. This variation may be due to the respective physicochemical differences between the two drugs [26]. AMX is more polar and smaller in size, which facilitates its mobility and diffusion into the activated carbon's porous matrix. In addition, AMX is multifunctional, having amino, carboxyl and hydroxyl groups that can form strong electrostatic interactions and hydrogen bonding with the surface functional groups (e.g., –OH, –COOH, $–PO4^{3-}$) of the acid-activated carbon. On the other hand, TC has a much larger molecular size and structure than the AMX drug, and its large molecular size slightly minimises its diffusion into the micropores. Its adsorption behavior is further influenced by its zwitteric character and the multitude of pKa values that can result in different speciation of the molecule in aqueous solutions at various pH levels. This could lead to fewer interactions of them with the active sites of the adsorbent surface, explaining the poor efficiency of the removal. Generally, the higher adsorption of AMX is due to a stronger bond between the adsorbent and adsorbate, and a good molecular match of AMX with the surface of activated carbon, which improves the diffusion mechanism through the porous network[27]. The results demonstrate the significant effect of the molecular structure, polarity and functional group content on the adsorption behaviour of pharmaceuticals on biomass-oriented activated carbon. as shown in **Figure 9**.

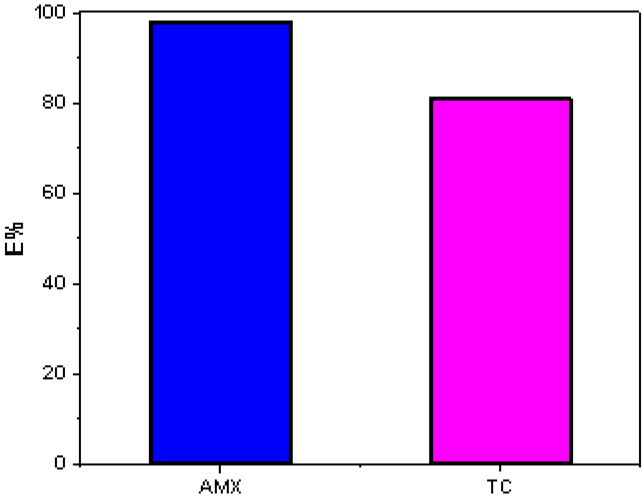

Figure 9. Comparative adsorption performance: AMX vs. TC (Exp. 100 mg/L, 0.05 g , pH 7 , and T 25 ° C)

3.10. Considerations and environmental impact

The activated carbon (PLAC) developed in this study demonstrates promising potential for scale-up due to its simple preparation steps and reliance on low-cost, locally available biomass waste (pine leaves). The chemical activation using phosphoric acid is widely recognized and can be adapted to industrial conditions with minor modifications. In terms of cost, the use of agricultural residues significantly reduces raw material expenses, making PLAC a potentially more affordable alternative to commercial activated carbon. From an environmental standpoint, the process aligns with sustainable practices by utilizing biomass waste and reducing environmental pollution. The washing steps after activation ensure the removal of excess activating agents, thereby minimizing any ecological risks. Moreover, the use of PLAC for dye removal from wastewater contributes to mitigating water pollution, highlighting its environmental benefit in real-world applications. As shown in **Table 3**

Table 3. Comparison of the adsorption capacities of several adsorbents

Biosorbents	E%	Reference
Coffee waste	77	[28]
Mango stone	87	[29]
Moringa oleifera	89	[30]
coconut shell	90	[31]
Apricot stone	86	[32]
Con Curb	66	[22]
Oaktree fruit waste	87	[33]
SSPC-AC	78	[34]
pencil peel (PP)	58	[35]
Lemon shell	97	[36]
tea dust	93	[37]

4. Conclusion

The present study successfully demonstrates the production of acid-activated carbon from pine (Pinus spp.) leaves, utilising phosphoric acid as the activating agent. The produced material had a very porous structure and possessed a chemically rich surface, which were both characteristics of a promising adsorbent for removing pharmaceutical pollutants from water. The batch adsorption test indicated that the activated carbon was more effective at removing AMX than TC. In addition, cyclic adsorption–desorption experiments demonstrated the reusability and structural robustness of the pine leaf-based activated carbon. The regeneration performance was best, with efficiency retention of over 85% of AMX, even after several cycles. The TC elution rate, as a percentage of the recovered level, however, was less than that of H_2O, indicating a relatively higher surface association or incomplete desorption. In summary, the activated carbon developed from pine leaves is considered a sustainable, low-cost, and practical material for selective extraction of AMX and other antibiotic drug pollutants from water, and may have great reuse potential for practical water treatment technologies.

Conflict of interest

The authors declare no conflict of interest.

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