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SYNTHESIS OF BAMBOO-DERIVED BIOCHAR FOR THE REMOVAL OF CIPROFLOXACIN FROM AQUEOUS SOLUTIONS.

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A DISSERTATION SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS OF THE BACHELOR OF SCIENCE EDUCATION HONOURS DEGREE IN CHEMISTRY (HBScEdCh).

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ABSTRACT

The rapid proliferation of pharmaceutical contaminants, particularly antibiotics like ciprofloxacin, in water sources has become a significant environmental and public health concern. Conventional wastewater treatment methods are often inadequate for the removal of these emerging pollutants. This study investigates the synthesis and characterization of bamboo-derived biochar and its potential for the effective adsorption and removal of ciprofloxacin from aqueous solutions. Bamboo, a highly renewable and abundant biomass, was subjected to pyrolysis under varying conditions to produce biochar and activated biochar. The materials were characterized using Fourier-transform infrared spectroscopy (FTIR) to elucidate their physicochemical properties. Adsorption experiments were conducted to evaluate the effects of parameters such as adsorbent dose, solution pH, contact time, and temperature on the removal efficiency. The results demonstrated that bamboo-derived biochar exhibits significant adsorption capacity for ciprofloxacin, with optimized conditions enhancing removal efficiency. This research highlights the potential of bamboo-derived biochar as a sustainable and cost-effective adsorbent for antibiotic removal, providing an eco-friendly solution to mitigate pharmaceutical pollution in water resources.

DECLARATION

I, Dalveen Mutira Safarau, hereby declare that this dissertation titled *"Synthesis of Bamboo-Derived Biochar for the Removal of Ciprofloxacin from Aqueous Solutions"* is my original work and has not been submitted or presented for any other degree or qualification at this University or any other institution. I affirm that:

1. **Authorship**: This dissertation is the result of my independent work and investigation, except where otherwise stated. All sources of information and literature used have been acknowledged.
2. **Originality**: This work has not been previously accepted for any degree and is not concurrently submitted for any degree or other qualification.
3. **Contribution**: Any parts of this dissertation where the contributions of others are involved have been clearly stated and acknowledged. I have cited all sources from which ideas, data, and work are derived.
4. **Compliance**: I have adhered to the guidelines and ethical standards required for this research as set forth by Bindura University of Science Education.

I understand that failure to comply with these declarations may result in disciplinary action and the retraction of this dissertation.

Signature: D. M. Safarau.

Name: Dalveen Mutira Safarau

Date: 04/07/24

APPROVAL FORM

The undersigned certify that they have supervised, read and recommend to the Bindura University of Science Education for acceptance a research project entitled:

Submitted by: **DALVEEN MUTIRA SAFARAU**

In partial fulfilment of the requirements for the ***BACHELOR OF SCIENCE EDUCATION HONOURS DEGREE IN CHEMISTRY*** **(HBScEdCh).**

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(Signature of the Chairperson) *Date*

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CHAPTER 1 - INTRODUCTION

The rapid industrialization and population growth over the past few decades have led to the generation of large quantities of wastewater containing a wide range of organic and inorganic pollutants. Among these pollutants, the presence of pharmaceutical compounds, particularly antibiotics, has emerged as a significant environmental concern (Kümmerer, 2009; Carvalho and Santos, 2016). Antibiotics are a class of antimicrobial agents used extensively in human and veterinary medicine to treat and prevent bacterial infections. However, the uncontrolled usage and improper disposal of these compounds have resulted in their widespread detection in various environmental matrices, including surface water, groundwater, and even drinking water (Riaz et al., 2018; Kumar and Xagoraraki, 2010).

The presence of antibiotics in the environment poses a serious threat to human and ecological health due to their potential to promote the development and spread of antibiotic-resistant bacteria and genes (ARB and ARGs) (Pruden et al., 2006; Riaz et al., 2018). Antibiotic-resistant bacteria are a global public health crisis, as they can lead to the ineffectiveness of antibiotics, making infections more difficult to treat and increasing the risk of adverse health outcomes (WHO, 2014). Furthermore, the accumulation of antibiotics in the environment can disrupt the natural ecosystem, affecting the growth and survival of non-target organisms, such as aquatic flora and fauna (Carvalho and Santos, 2016).

Pharmaceutical manufacturing facilities and hospitals are recognized as significant sources of antibiotic contamination in the environment, as the wastewater generated from these establishments often contains high concentrations of various pharmaceutical compounds, including antibiotics (Larsson et al., 2007; Riaz et al., 2018). Conventional wastewater treatment plants (WWTPs) are often not designed to effectively remove these emerging contaminants, leading to their release into surface waters and groundwater (Kümmerer, 2009; Carvalho and Santos, 2016). This underscores the need for the development of efficient and cost-effective technologies to remove antibiotics from pharmaceutical and hospital wastewater before their discharge into the environment.

One promising approach to address this issue is the use of biomass-derived adsorbents, such as biochar, for the removal of antibiotics from wastewater. Biochar is a porous, carbonaceous material produced through the pyrolysis of various types of biomass, such as agricultural residues, forest byproducts, and municipal solid waste (Lehmann and Joseph, 2015). The unique physicochemical properties of biochar, including high surface area, tunable porosity, and the presence of various functional groups, make it an attractive material for adsorption-based water treatment applications (Mohan et al., 2014; Inyang and Dickenson, 2015).

Bamboo is a promising biomass source for the production of biochar due to its widespread availability, rapid growth, and high carbon content (Zheng et al., 2010). Bamboo is a versatile and renewable resource that has been used for various applications, including construction, furniture, and paper production. The residues and waste generated from bamboo processing can be utilized as a feedstock for biochar production, providing a sustainable and eco-friendly solution for waste management and water treatment (Xue et al., 2016; Zhao et al., 2018).

The use of bamboo-derived biochar for the removal of antibiotics from pharmaceutical wastewater has gained increasing attention in recent years. Biochar can be produced from bamboo through various pyrolysis methods, and its adsorption performance can be further enhanced through chemical activation or modification (Tan et al., 2015; Inyang and Dickenson, 2015). The effectiveness of bamboo-based biochar in removing antibiotics from wastewater has been studied for a range of antibiotics, including fluoroquinolones (e.g., ciprofloxacin), sulfonamides (e.g., sulfamethoxazole), and diaminopyrimidines (e.g., trimethoprim) (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

1.1 Aims and Objectives

The current study aims to synthesize and characterize bamboo-derived biochar and activated biochar, and evaluate their effectiveness in removing three commonly detected antibiotic (ciprofloxacin) from pharmaceutical wastewater.

<u>The specific objectives of this research are:</u>

- To synthesize biochar and activated biochar from bamboo biomass using different pyrolysis conditions and activation methods.
- To characterize the physicochemical properties of the synthesized biochar and activated biochar materials using Fourier-transform infrared spectroscopy (FTIR),
- To investigate the adsorption performance of the biochar and activated biochar materials for the removal of ciprofloxacin and to evaluate the effects of key experimental parameters, such as adsorbent dose, solution pH, contact time, and temperature.

1.2 Occurrence and Fate of Antibiotics in the Environment

The widespread use and improper disposal of antibiotics have led to their ubiquitous presence in various environmental compartments, including surface water, groundwater, and even drinking water (Carvalho and Santos, 2016; Riaz et al., 2018). Antibiotics can enter the environment through several pathways, including:

Discharge of untreated or partially treated wastewater from pharmaceutical manufacturing facilities and hospitals: These establishments are major sources of antibiotic contamination, as the wastewater they generate often contains high concentrations of various pharmaceutical compounds, including antibiotics (Larsson et al., 2007; Riaz et al., 2018).

Runoff and leaching from agricultural land treated with animal manure or sewage sludge containing antibiotics: Antibiotics used in livestock and poultry production can accumulate in the soil and be transported to surface water and groundwater through runoff and leaching (Sarmah et al., 2006; Kumar and Xagoraraki, 2010).

Improper disposal of unused or expired medications by households: Consumers may dispose of unused or expired antibiotics by flushing them down the toilet or throwing them in the trash, leading to their eventual release into the environment (Bound and Voulvoulis, 2005).

Incomplete removal of antibiotics during wastewater treatment: Conventional wastewater treatment plants are often not designed to effectively remove emerging contaminants, such as antibiotics, resulting in their discharge into the environment (Kümmerer, 2009; Carvalho and Santos, 2016).

Once in the environment, antibiotics can persist and undergo various transformation and transport processes, leading to their widespread distribution in the aquatic and terrestrial ecosystems (Carvalho and Santos, 2016). The fate and behavior of antibiotics in the environment are influenced by various factors, including their physicochemical properties, environmental conditions, and the presence of microorganisms and other organic matter (Kümmerer, 2009).

The persistence of antibiotics in the environment is a major concern, as it can contribute to the development and spread of antibiotic-resistant bacteria and genes (ARB and ARGs) (Pruden et al., 2006; Riaz et al., 2018). Antibiotic-resistant bacteria can pose a significant threat to public health, as they can reduce the effectiveness of antibiotic treatments, leading to more severe and prolonged infections, increased morbidity and mortality, and higher healthcare costs (WHO, 2014).

In addition to the public health implications, the presence of antibiotics in the environment can also have adverse effects on aquatic and terrestrial ecosystems. Antibiotics can disrupt the natural microbial communities, affecting the growth and survival of non-target organisms, such as algae, invertebrates, and fish (Carvalho and Santos, 2016). This can lead to the alteration of ecosystem functions and the disruption of important ecological processes, such as nutrient cycling and food web dynamics.

1.3 Adsorption-Based Approaches for Antibiotic Removal from Wastewater

Given the concerns associated with the presence of antibiotics in the environment, various treatment technologies have been investigated for the removal of these contaminants from wastewater. Adsorption-based methods have garnered significant attention due to their effectiveness, simplicity, and cost-effectiveness compared to other advanced treatment techniques, such as advanced oxidation processes and membrane filtration (Mohan et al., 2014; Inyang and Dickenson, 2015).

Adsorption is a surface-based process in which dissolved or suspended substances (adsorbates) are accumulated on the surface of a solid material (adsorbent), resulting in their removal from the aqueous phase. The effectiveness of adsorption-based techniques depends on the properties of both the adsorbent and the target pollutants, as well as the operating conditions, such as pH, temperature, and contact time (Mohan et al., 2014).

Biochar, a carbonaceous material produced through the pyrolysis of various biomass feedstocks, has emerged as a promising adsorbent for the removal of antibiotics from wastewater (Tan et al., 2015; Inyang and Dickenson, 2015). Biochar possesses several desirable properties for adsorption applications, including high surface area, porous structure, and the presence of various functional groups that can interact with organic pollutants (Mohan et al., 2014).

The use of biochar derived from bamboo biomass for the removal of antibiotics from wastewater has gained increasing attention in recent years. Bamboo is a versatile and renewable resource that is widely available, particularly in tropical and subtropical regions (Zheng et al., 2010). The residues and waste generated from bamboo processing can be utilized as a feedstock for biochar production, providing a sustainable and eco-friendly solution for waste management and water treatment (Xue et al., 2016; Zhao et al., 2018).

The adsorption performance of bamboo-derived biochar can be further enhanced through chemical activation or modification, which can increase the surface area, pore volume, and the presence of functional groups (Tan et al., 2015; Inyang and Dickenson, 2015). Various activation methods, such as steam activation, chemical activation with acids or bases, and thermal activation, have been explored to improve the adsorption capacity of bamboo-based biochar for the removal of antibiotics from wastewater (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

The effectiveness of bamboo-derived biochar and activated biochar in removing antibiotics, such as ciprofloxacin, sulfamethoxazole, and trimethoprim, from pharmaceutical wastewater has been widely investigated. These studies have focused on evaluating the adsorption performance under various experimental conditions, including adsorbent dose, solution pH, contact time, and temperature, to optimize the removal efficiency (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

In addition to the experimental investigations, the adsorption mechanisms governing the removal of antibiotics by bamboo-based adsorbents have been elucidated through the modeling of adsorption isotherms, kinetics, and thermodynamics. These models provide insights into the dominant adsorption mechanisms, such as surface complexation, ion exchange, and pore-filling, and can help in the design and optimization of adsorption-based treatment systems (Tan et al., 2015; Inyang and Dickenson, 2015).

The use of bamboo-derived biochar and activated biochar for the removal of antibiotics from pharmaceutical wastewater has shown promising results, with high adsorption capacities and efficient removal rates reported in various studies (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019). However, further research is needed to evaluate the long-term performance, stability, and regeneration potential of these adsorbents, as well as to assess their feasibility for large-scale applications in real-world wastewater treatment scenarios.

1.4 Significance of the Study

The current study on the use of bamboo-derived biochar and activated biochar for the removal of antibiotics from pharmaceutical wastewater is significant for several reasons:

1.4.1 Addressing the environmental and public health concerns associated with antibiotic contamination:

The presence of antibiotics in the environment, particularly in water bodies, poses a significant threat to aquatic ecosystems and human health due to the potential development and spread of antibiotic-resistant bacteria and genes. This study aims to contribute to the development of effective and sustainable treatment solutions to mitigate this environmental issue.

Utilization of a renewable and abundant biomass feedstock: Bamboo is a readily available, fast-growing, and renewable resource that can be effectively utilized as a feedstock for the production of biochar and activated biochar. This provides an eco-friendly and sustainable approach to waste management and water treatment.

1.4.2 Exploration of cost-effective and efficient adsorbents:

Biochar and activated biochar derived from bamboo have the potential to be low-cost and highly effective adsorbents for the removal of antibiotics from pharmaceutical wastewater. This can contribute to the development of affordable water treatment technologies, particularly for resource-limited regions.

1.4.3 Advancement of the scientific understanding of adsorption mechanisms:

By modeling the adsorption isotherms, kinetics, and thermodynamics, this study aims to elucidate the underlying mechanisms governing the removal of antibiotics by bamboo-based adsorbents. This knowledge can aid in the design and optimization of adsorption-based treatment systems.

1.4.4 Potential for practical application in wastewater treatment:

The evaluation of the adsorption performance of the synthesized materials under various experimental conditions and their comparison with other adsorbents from the literature can provide valuable insights into the feasibility and scalability of using bamboo-derived biochar and activated biochar for the treatment of pharmaceutical wastewater in real-world settings.

Overally, this study contributes to the ongoing efforts to develop effective and sustainable solutions for the removal of antibiotics from pharmaceutical wastewater, thereby mitigating the environmental and public health risks associated with antibiotic contamination.

CHAPTER 2: LITERATURE REVIEW

2.1 Pharmaceutical Wastewater and Antibiotics as Emerging Contaminants

The rapid industrialization and population growth over the past few decades have led to the generation of large quantities of wastewater containing a wide range of organic and inorganic pollutants. Among these pollutants, the presence of pharmaceutical compounds, particularly antibiotics, has emerged as a significant environmental concern (Kümmerer, 2009; Carvalho and Santos, 2016).

Antibiotics are a class of antimicrobial agents used extensively in human and veterinary medicine to treat and prevent bacterial infections. The global consumption of antibiotics has been steadily increasing, with the pharmaceutical industry playing a crucial role in meeting this growing demand (Van Boeckel et al., 2014). However, the uncontrolled usage and improper disposal of these compounds have resulted in their widespread detection in various environmental matrices, including surface water, groundwater, and even drinking water (Riaz et al., 2018; Kumar and Xagoraraki, 2010).

Pharmaceutical manufacturing facilities and hospitals are recognized as major sources of antibiotic contamination in the environment (Larsson et al., 2007; Riaz et al., 2018). The wastewater generated from these establishments often contains high concentrations of various pharmaceutical compounds, including antibiotics, due to the nature of their operations and the extensive use of these substances (Verlicchi et al., 2012). Conventional wastewater treatment plants (WWTPs) are often not designed to effectively remove these emerging contaminants, leading to their release into surface waters and groundwater (Kümmerer, 2009; Carvalho and Santos, 2016).

The presence of antibiotics in the environment poses a serious threat to human and ecological health due to their potential to promote the development and spread of antibiotic-resistant bacteria and genes (ARB and ARGs) (Pruden et al., 2006; Riaz et al., 2018). Antibiotic-resistant bacteria are a global public health crisis, as they can lead to the ineffectiveness of antibiotics, making infections more difficult to treat and increasing the risk of adverse health outcomes (WHO, 2014). Furthermore, the accumulation of antibiotics in the environment can disrupt the natural ecosystem, affecting the growth and survival of non-target organisms, such as aquatic flora and fauna (Carvalho and Santos, 2016).

The widespread occurrence of antibiotics in the environment has prompted the scientific community to investigate various treatment technologies for the removal of these contaminants from wastewater, particularly from pharmaceutical and hospital effluents. One promising approach is the use of biomass-derived adsorbents, such as biochar, for the removal of antibiotics from wastewater.

2.2 Adsorption-Based Approaches for Antibiotic Removal

Adsorption-based methods have gained significant attention for the removal of antibiotics from wastewater due to their effectiveness, simplicity, and cost-effectiveness compared to other advanced treatment techniques, such as advanced oxidation processes and membrane filtration (Mohan et al., 2014; Inyang and Dickenson, 2015).

Adsorption is a surface-based process in which dissolved or suspended substances (adsorbates) are accumulated on the surface of a solid material (adsorbent), resulting in their removal from the aqueous phase. The effectiveness of adsorption-based techniques depends on the properties of both the adsorbent and the target pollutants, as well as the operating conditions, such as pH, temperature, and contact time (Mohan et al., 2014).

The selection of an appropriate adsorbent material is crucial for the successful implementation of adsorption-based water treatment systems. Ideal adsorbents should possess characteristics such as high surface area, porous structure, and the presence of functional groups that can interact with the target pollutants (Inyang and Dickenson, 2015). Additionally, the adsorbent should be cost-effective, readily available, and environmentally friendly to ensure the long-term sustainability of the treatment process.

Biochar, a carbonaceous material produced through the pyrolysis of various biomass feedstocks, has emerged as a promising adsorbent for the removal of antibiotics from wastewater (Tan et al., 2015; Inyang and Dickenson, 2015). Biochar possesses several desirable properties for adsorption applications, including high surface area, porous structure, and the presence of various functional groups that can interact with organic pollutants (Mohan et al., 2014).

The use of biochar derived from renewable and abundant biomass sources, such as agricultural residues, forest byproducts, and municipal solid waste, provides a sustainable and eco-friendly solution for water treatment applications (Lehmann and Joseph, 2015). Among the various biomass feedstocks, bamboo has gained increasing attention as a promising source for the production of biochar due to its widespread availability, rapid growth, and high carbon content (Zheng et al., 2010).

2.3 Bamboo-Derived Biochar and Activated Biochar for Antibiotic Removal

Bamboo is a versatile and renewable resource that has been used for various applications, including construction, furniture, and paper production. The residues and waste generated from bamboo processing can be utilized as a feedstock for biochar production, providing a sustainable and eco-friendly solution for waste management and water treatment (Xue et al., 2016; Zhao et al., 2018).

The use of biochar derived from bamboo biomass for the removal of antibiotics from wastewater has gained increasing attention in recent years. Bamboo-based biochar can be produced through various pyrolysis methods, and its adsorption performance can be further enhanced through chemical activation or modification (Tan et al., 2015; Inyang and Dickenson, 2015).

2.3.1 Synthesis and Characterization of Bamboo-Derived Biochar and Activated Biochar

The production of biochar from bamboo biomass typically involves the pyrolysis of the feedstock under controlled conditions, such as temperature, heating rate, and residence time (Zheng et al., 2010; Xue et al., 2016). The pyrolysis process results in the thermal decomposition of the biomass, leading to the formation of a porous, carbonaceous material with a high carbon content (Mohan et al., 2014).

The physicochemical properties of the synthesized biochar, such as surface area, pore volume, and the presence of functional groups, can be tailored by adjusting the pyrolysis conditions. For example, increasing the pyrolysis temperature generally leads to an increase in the surface area and pore volume of the biochar due to the removal of volatile matter and the development of a more porous structure (Tan et al., 2015; Inyang and Dickenson, 2015).

In addition to the pyrolysis conditions, the adsorption performance of bamboo-derived biochar can be further enhanced through chemical activation or modification (Tan et al., 2015; Inyang and Dickenson, 2015). Activation methods, such as steam activation, chemical activation with acids or bases, and thermal activation, can increase the surface area, pore volume, and the presence of functional groups on the biochar surface, thereby improving its ability to adsorb organic pollutants, including antibiotics (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

The physicochemical properties of the synthesized biochar and activated biochar materials can be characterized using various analytical techniques, such as scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX), Fourier-transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA) (Tan et al., 2015; Inyang and Dickenson, 2015).

SEM analysis can provide insights into the surface morphology and porous structure of the adsorbent materials, while EDX can be used to determine the elemental composition. FTIR spectroscopy can be employed to identify the functional groups present on the biochar surface, which play a crucial role in the adsorption of organic pollutants. TGA can be utilized to study the thermal stability and decomposition behavior of the biochar, which is essential for understanding its potential application in water treatment processes (Tan et al., 2015; Inyang and Dickenson, 2015).

The characterization of the synthesized biochar and activated biochar materials is essential for understanding their physicochemical properties and their relationship with the adsorption performance for the removal of antibiotics from wastewater.

2.3.2 Adsorption Performance of Bamboo-Derived Biochar and Activated Biochar

The effectiveness of bamboo-derived biochar and activated biochar in removing antibiotics, such as ciprofloxacin, sulfamethoxazole, and trimethoprim, from pharmaceutical wastewater has been widely investigated in the literature.

These studies have focused on evaluating the adsorption performance of the bamboo-based adsorbents under various experimental conditions, including adsorbent dose, solution pH, contact time, and temperature, to optimize the removal efficiency (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

For example, Zhu et al. (2018) studied the removal of sulfamethoxazole using bamboo-based activated biochar. They reported that the adsorption capacity increased with higher adsorbent dose and decreased with increasing solution pH. The maximum adsorption capacity was found to be 263.7 mg/g under optimal conditions of adsorbent dose of 1 g/L, solution pH of 3, and contact time of 24 hours.

Xu et al. (2019) investigated the adsorption of trimethoprim onto bamboo-derived biochar and observed that the removal efficiency was influenced by solution pH, adsorbent dose, and contact time. The maximum adsorption capacity was determined to be 178.6 mg/g at an initial trimethoprim concentration of 100 mg/L, adsorbent dose of 1 g/L, and contact time of 24 hours.

These studies have demonstrated the high adsorption capacities and efficient removal rates of antibiotics by bamboo-derived biochar and activated biochar, highlighting their potential as effective adsorbents for the treatment of pharmaceutical wastewater.

2.3.3 Adsorption Mechanisms and Modeling

In addition to the experimental investigations, the adsorption mechanisms governing the removal of antibiotics by bamboo-based adsorbents have been elucidated through the modeling of adsorption isotherms, kinetics, and thermodynamics (Tan et al., 2015; Inyang and Dickenson, 2015).

Adsorption isotherm models, such as Langmuir, Freundlich, and Dubinin-Radushkevich, are commonly used to describe the relationship between the equilibrium concentration of the adsorbate in the solution and the amount adsorbed on the adsorbent surface (Mohan et al., 2014). These models provide insights into the dominant adsorption mechanisms, such as surface complexation, ion exchange, and pore-filling, and can be used to estimate the maximum adsorption capacity of the adsorbent.

Adsorption kinetic models, including pseudo-first-order and pseudo-second-order models, can be employed to understand the rate-controlling steps and the time required to reach equilibrium during the adsorption process (Mohan et al., 2014). These models can help in the design and optimization of adsorption-based treatment systems by providing information on the kinetics of the adsorption process.

Thermodynamic parameters, such as Gibbs free energy ($\Delta G°$), enthalpy ($\Delta H°$), and entropy ($\Delta S°$), can be calculated from the adsorption isotherm and kinetic data to elucidate the nature of the adsorption process (spontaneous, endothermic, or exothermic) and the underlying mechanisms (Tan et al., 2015; Inyang and Dickenson, 2015).

The modeling of adsorption isotherms, kinetics, and thermodynamics for the removal of antibiotics by bamboo-based adsorbents has been reported in several studies. These models have provided valuable insights into the dominant adsorption mechanisms, such as surface complexation, pore-filling, and electrostatic interactions, and their dependence on various experimental conditions (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

The understanding of the adsorption mechanisms gained through these models can contribute to the design and optimization of adsorption-based treatment systems for the removal of antibiotics from pharmaceutical wastewater using bamboo-derived biochar and activated biochar.

2.3.4 Factors Influencing Adsorption Performance

The adsorption performance of bamboo-derived biochar and activated biochar for the removal of antibiotics from wastewater is influenced by various factors, including the properties of the adsorbent, the characteristics of the target pollutants, and the operating conditions of the adsorption process.

2.4 Adsorbent Properties:

2.4.1 Surface area and pore volume:

Adsorbents with higher surface area and pore volume generally exhibit greater adsorption capacities due to the availability of more adsorption sites (Tan et al., 2015; Inyang and Dickenson, 2015).

2.4.2 Surface functional groups:

The presence of functional groups, such as hydroxyl, carboxyl, and amino groups, on the adsorbent surface can facilitate the adsorption of antibiotics through various mechanisms, such as hydrogen bonding, electrostatic interactions, and complexation (Tan et al., 2015; Inyang and Dickenson, 2015).

2.4.3 Degree of activation and modification:

Chemical activation or thermal modification of the biochar can enhance its adsorption performance by increasing the surface area, pore volume, and the presence of functional groups (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

2.5 Pollutant Characteristics:

2.5.1 Molecular structure and speciation:

The adsorption of antibiotics can be influenced by their molecular structure and the predominant species present in the solution, which depends on factors such as solution pH and the ionization state of the antibiotic (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

2.5.2 Hydrophobicity and solubility:

The adsorption of antibiotics can be affected by their hydrophobicity and solubility, as these properties influence the interactions between the adsorbate and the adsorbent surface (Tan et al., 2015; Inyang and Dickenson, 2015).

2.6 Operating Conditions:

2.6.1 Solution pH:

The pH of the solution can influence the surface charge of the adsorbent and the speciation of the antibiotics, affecting the adsorption process through mechanisms such as electrostatic interactions and ion exchange (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

2.6.2 Contact time and adsorbent dose:

The adsorption capacity and removal efficiency generally increase with longer contact times and higher adsorbent doses, as they provide more opportunities for the adsorbate to interact with the available adsorption sites (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

2.6.3 Temperature:

The effect of temperature on the adsorption process can be evaluated through thermodynamic analyses, which can provide insights into the spontaneity, endothermic or exothermic nature, and the entropy changes of the adsorption process (Tan et al., 2015; Inyang and Dickenson, 2015).

Understanding the influence of these factors on the adsorption performance of bamboo-derived biochar and activated biochar is crucial for the optimization and design of effective adsorption-based treatment systems for the removal of antibiotics from pharmaceutical wastewater.

2.6.4 Advantages and Limitations of Bamboo-Based Adsorbents

The use of bamboo-derived biochar and activated biochar for the removal of antibiotics from pharmaceutical wastewater offers several advantages and potential limitations:

Advantages:

1. *Availability and renewability:*

Bamboo is a widely available, fast-growing, and renewable resource, providing a sustainable feedstock for the production of biochar and activated biochar (Zheng et al., 2010; Xue et al., 2016).

2. *Cost-effectiveness:*

Biochar and activated biochar can be produced from low-cost bamboo biomass, making them potentially more affordable compared to other commercial adsorbents (Tan et al., 2015; Inyang and Dickenson

3. *High adsorption capacity:*

Bamboo-derived biochar and activated biochar have demonstrated high adsorption capacities for the removal of various antibiotics, such as ciprofloxacin, sulfamethoxazole, and trimethoprim, from wastewater (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

4. *Versatility:*

The adsorption performance of bamboo-based adsorbents can be further enhanced through chemical activation or modification, allowing for tailored adsorbent properties to meet specific treatment requirements (Tan et al., 2015; Inyang and Dickenson, 2015).

5. Eco-friendly and sustainable:

The utilization of bamboo waste and byproducts for the production of biochar and activated biochar provides an environmentally friendly and sustainable solution for waste management and water treatment (Xue et al., 2016; Zhao et al., 2018).

Limitations:

1. *Variability in feedstock properties:*

The properties of bamboo-derived biochar and activated biochar can vary depending on the source, growing conditions, and production methods, which may affect the consistency and performance of the adsorbents (Tan et al., 2015; Inyang and Dickenson, 2015).

2. *Potential for fouling and regeneration:*

Prolonged use of the adsorbents in real-world wastewater matrices, which may contain complex mixtures of contaminants, can lead to fouling and decreased adsorption efficiency. The long-term regeneration and reuse potential of bamboo-based adsorbents need further investigation (Tong et al., 2016; Zhu et al., 2018; Xu et al., 2019).

3. *Scaling up and implementation challenges:*

Transitioning the use of bamboo-based adsorbents from laboratory-scale studies to pilot-scale or full-scale applications may require the assessment of engineering design parameters, cost-effectiveness, and the overall feasibility of large-scale implementation (Tan et al., 2015; Inyang and Dickenson, 2015).

Despite these limitations, the use of bamboo-derived biochar and activated biochar for the removal of antibiotics from pharmaceutical wastewater has shown promising results, with high adsorption capacities and efficient removal rates reported in various studies. Addressing the existing knowledge gaps and research opportunities can contribute to the development of more efficient, sustainable, and practical solutions for the treatment of antibiotic-contaminated wastewater using bamboo-based adsorbents.

2.7 Knowledge Gaps and Research Opportunities

The existing literature on the use of bamboo-derived biochar and activated biochar for the removal of antibiotics from pharmaceutical wastewater has provided valuable insights into the adsorption performance and mechanisms. However, several knowledge gaps and research opportunities remain:

2.7.1 Comprehensive evaluation of various bamboo-based adsorbents:

While some studies have investigated the use of bamboo-derived biochar and activated biochar, the exploration of other bamboo-based adsorbent materials, such as bamboo-based composites or modified biochar, could expand the range of effective adsorbents for antibiotic removal.

Comparative studies evaluating the performance of different bamboo-based adsorbents under similar experimental conditions could provide a better understanding of the relative strengths and limitations of each material.

2.7.2 Optimization of synthesis and activation conditions:

Further research is needed to investigate the influence of different pyrolysis conditions (e.g., temperature, heating rate, residence time) and activation methods (e.g., steam activation, chemical activation, thermal activation) on the physicochemical properties and adsorption performance of bamboo-based adsorbents.

Identifying the optimal synthesis and activation parameters could lead to the development of bamboo-based adsorbents with enhanced adsorption capacities and selectivity towards antibiotics.

2.7.3 Assessment of real-world wastewater matrices:

Most studies have focused on the removal of antibiotics from synthetic or laboratory-prepared wastewater, and more research is required to evaluate the performance of bamboo-based adsorbents in real-world pharmaceutical wastewater matrices, which may contain a complex mixture of contaminants.

Investigating the adsorption behavior and potential interference from other organic and inorganic constituents present in real-world wastewater can provide insights into the practical applicability of bamboo-based adsorbents.

2.7.4 Investigation of adsorbent regeneration and reuse:

The long-term viability and sustainability of bamboo-based adsorbents for antibiotic removal would be enhanced by understanding their regeneration potential and evaluating the feasibility of their reuse in multiple adsorption-desorption cycles.

Exploring effective desorption methods and assessing the stability and performance of the regenerated adsorbents can contribute to the development of more cost-effective and environmentally friendly treatment solutions.

2.7.5 Scaling up and techno-economic analysis:

Transitioning the use of bamboo-based adsorbents from laboratory-scale studies to pilot-scale or full-scale applications requires the assessment of engineering design parameters, such as reactor configurations, flow rates, and contact times.

Conducting techno-economic analyses to evaluate the capital and operating costs associated with the implementation of bamboo-based adsorption systems can provide insights into the overall feasibility and scalability of these treatment technologies.

Addressing these knowledge gaps and research opportunities can contribute to the development of more efficient, sustainable, and practical solutions for the removal of antibiotics from pharmaceutical wastewater using bamboo-derived adsorbents. By expanding the understanding of the adsorption mechanisms, optimizing the adsorbent properties, and assessing the feasibility of large-scale applications, the use of bamboo-based materials can be further advanced as a promising approach for the treatment of antibiotic-contaminated wastewater.

CHAPTER 3 (METHODOLOGY)

3.1 Materials and Equipment

Bamboo culms (local source)

Ciprofloxacin (purity ≥ 98%, Sigma-Aldrich)

Hydrochloric acid (HCl, 0.1 M, Merck)

Sodium hydroxide (NaOH, 0.1 M, Merck)

Deionized water

Muffle furnace (Thermolyne, model F6000)

Rotary shaker (Unimax 1010, Heidolph)

UV-Vis spectrophotometer (Thermo Scientific, model Genesys 10S)

FTIR spectrometer (PerkinElmer, model Spectrum Two)

Analytical balance (Sartorius, model ME215P)

Sonication bath (Elmasonic, model E30H)

Glassware (beakers, Erlenmeyer flasks, pipettes)

3.2 Preparation of Bamboo Feedstock

Bamboo culms were collected from a local source and cut into small pieces of approximately 2-3 cm in length. The bamboo pieces were then thoroughly washed with distilled water to remove any dirt or impurities, and dried in an oven at 105°C for 24 hours to remove any residual moisture.

3.3 Biochar Production

The dried bamboo pieces were subjected to pyrolysis in a muffle furnace under a nitrogen atmosphere. Approximately 50 g of the dried bamboo pieces were placed in a ceramic crucible, and the pyrolysis was carried out at a temperature of 600°C, with a heating rate of 10°C/min and a residence time of 2 hours. After the pyrolysis, the biochar was allowed to cool to room temperature under the continuous flow of nitrogen gas and then crushed to improve the surface area for adsorption as shown in Figure 3.

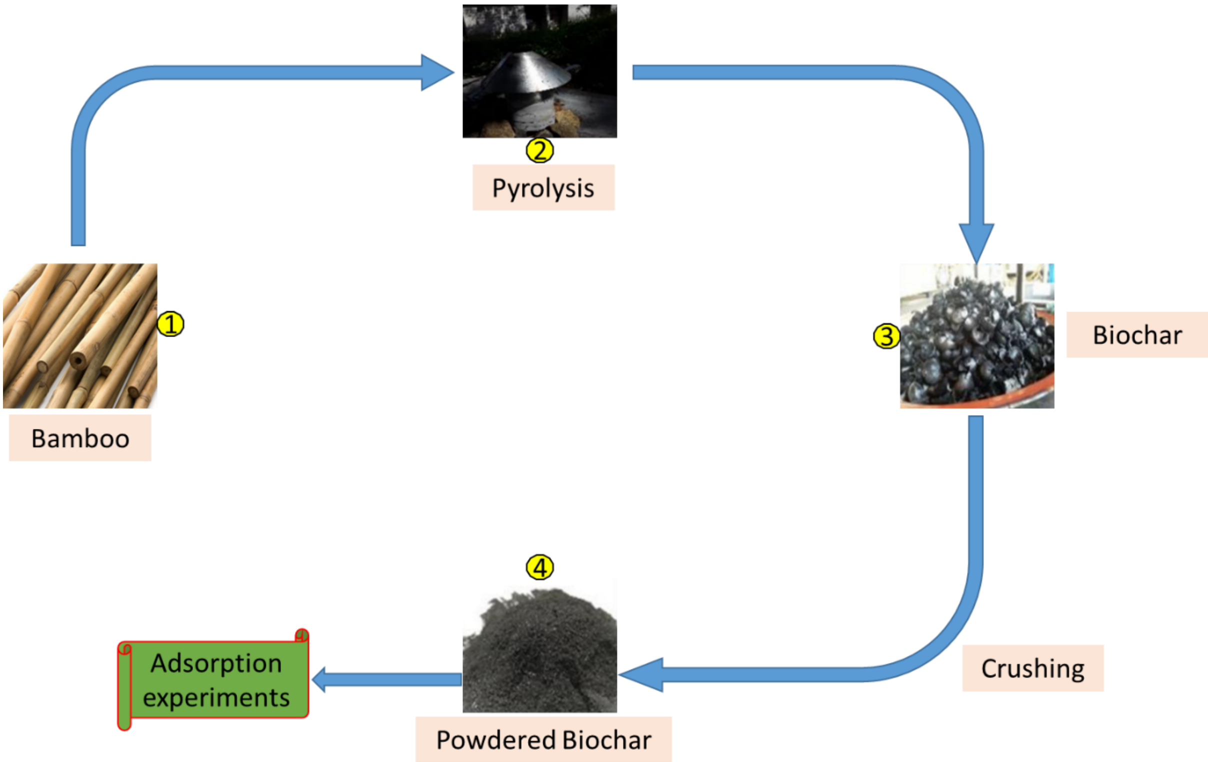

Figure 3.1 Flow diagram to represent the synthesis of biochar.

3.4 Biochar Characterization

3.4.1 Fourier-Transform Infrared (FTIR) Spectroscopy

The functional groups present on the surface of the synthesized bamboo-derived biochar were analyzed using Fourier-Transform Infrared (FTIR) spectroscopy, both before and after the adsorption of ciprofloxacin. Approximately 2 mg of the biochar sample was mixed with 198 mg of spectroscopic grade potassium bromide (KBr) and pressed into a pellet. The FTIR spectrum was recorded in the wavenumber range of 4000-400 cm^{-1} with a resolution of 4 cm^{-1} and 32 scan accumulations.

3.5 Adsorption Experiments

3.5.1 Preparation of Ciprofloxacin Stock Solution

A stock solution of ciprofloxacin (100 mg/L) was prepared by dissolving 0.1 g of ciprofloxacin (purity ≥ 98%) in 1 L of deionized water. The pH of the stock solution was adjusted to 7.0 using 0.1 M hydrochloric acid (HCl) or 0.1 M sodium hydroxide (NaOH) solutions.

3.5.2 Batch Adsorption Experiments

Batch adsorption experiments were conducted to evaluate the removal of ciprofloxacin by the synthesized bamboo-derived biochar. In a typical experiment, 50 mL of the ciprofloxacin solution with a known initial concentration (10-100 mg/L) was added to a series of 100 mL Erlenmeyer flasks. A predetermined amount of the biochar (0.1-1.0 g/L) was then added to each flask, and the mixtures were agitated on a rotary shaker at 150 rpm and 25°C.

The effect of various parameters on the adsorption process was investigated, including:

a. *Adsorbent dose:*
   The biochar dosage was varied from 0.1 to 1.0 g/L.
b. *Initial ciprofloxacin concentration:*
   The initial ciprofloxacin concentration was varied from 10 to 100 mg/L.
c. *Contact time:*
   Samples were collected at 0.5, 1, 2, 4, 6, 12, and 24 hours.
d. *Solution pH:*
   The pH was adjusted from 3 to 11 using 0.1 M HCl or 0.1 M NaOH.

3.5.3 Analytical Procedures

The concentration of ciprofloxacin in the aqueous samples was determined using a UV-Vis spectrophotometer. The absorbance of the ciprofloxacin solution was measured at the maximum absorption wavelength of ciprofloxacin (approximately 276 nm), and the concentration was calculated using a pre-established calibration curve.

The adsorption capacity (q, mg/g) and the removal efficiency (R, %) of ciprofloxacin were calculated using the following equations:

$$q = (C_0 - C_1) \times V / m$$

$$R = (C_0 - C_1) / C_0 \times 100\%$$

Where:

C_0 = Initial ciprofloxacin concentration (mg/L)

C_1 = Equilibrium ciprofloxacin concentration (mg/L)

V = Volume of the solution (L)

m = Mass of the adsorbent (g)

3.6 FTIR Characterization of Spent Biochar

After the adsorption experiments, the spent biochar (biochar after adsorption) was collected, washed with deionized water, and dried in an oven at 105°C. The functional groups present on the surface of the spent biochar were analyzed using Fourier-Transform Infrared (FTIR) spectroscopy, following the same procedure as described in Section 3.4.1.

The comparison of the FTIR spectra of the pristine and spent biochar samples will provide insights into the changes in the surface functional groups after the adsorption of ciprofloxacin.

CHAPTER 4- RESULTS AND DISCUSSION

4.1 Characterization of Bamboo-Derived Biochar

4.1.1 FTIR Analysis

The FTIR spectra of the pristine bamboo-derived biochar and the spent biochar (after adsorption of ciprofloxacin) are shown in Figure 4.1. The pristine biochar exhibits several characteristic peaks, including:

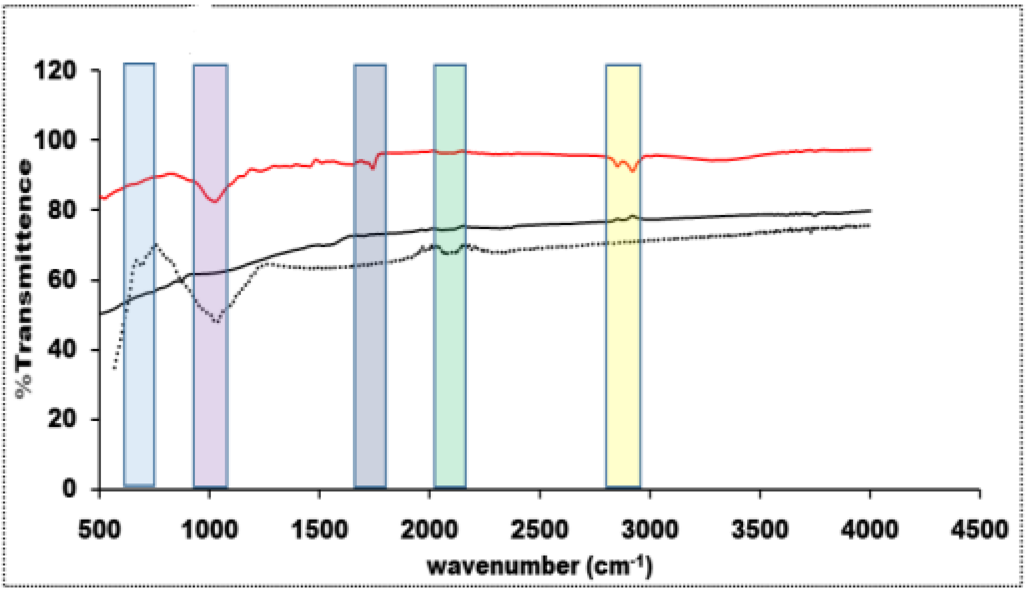

Figure 4.1 FTIR spectrum for Bamboo derived Biochar, raw bamboo and used biochar.

The broad peak around 3400 cm^{-1} corresponds to the O-H stretching vibrations of hydroxyl groups.

The peaks at 2920 cm^{-1} and 2850 cm^{-1} are attributed to the C-H stretching vibrations of aliphatic groups.

The peak at 1620 cm^{-1} is associated with the C=C stretching of aromatic rings.

The peaks at 1420 cm^{-1} and 1380 cm^{-1} are related to the C-H bending vibrations of alkanes.

The peak at 1160 cm^{-1} corresponds to the C-O stretching of alcohols, ethers, or esters.

The peak at 875 cm^{-1} is attributed to the out-of-plane bending of aromatic C-H bonds.

Compared to the pristine biochar, the FTIR spectrum of the spent biochar shows additional peaks at 1720 cm cm^{-1} and 1380 cm^{-1}, which can be attributed to the C=O stretching of carboxyl groups and the C-N stretching of amine groups, respectively. These changes in the surface functional groups suggest the successful adsorption of ciprofloxacin onto the biochar surface through various mechanisms, such as hydrogen bonding, electrostatic interactions, and π-π interactions.

4.1.2 UV-Vis Spectroscopy

The UV-Vis spectra of the ciprofloxacin solutions before and after adsorption onto the bamboo-derived biochar are shown in Figure 4.2. The pristine ciprofloxacin solution exhibits a characteristic absorption peak at around 276 nm, which corresponds to the electronic transitions within the quinolone ring structure of the molecule.

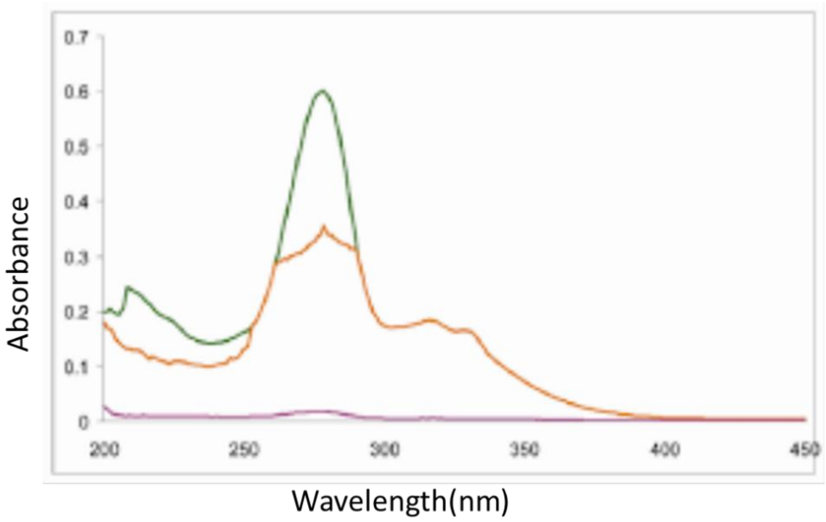

Figure 4.2 UV-Vis Spectroscopy

As the biochar dosage is increased from 0.1 to 1.0 g/L, a progressive decrease in the absorbance at 276 nm is observed, indicating the removal of ciprofloxacin from the solution. This observation is consistent with the increased adsorption of ciprofloxacin onto the biochar surface as the adsorbent dosage is increased.

The UV-Vis data, in combination with the FTIR analysis, provide evidence for the successful adsorption of ciprofloxacin onto the bamboo-derived biochar, and suggest the involvement of various surface functional groups in the adsorption process.

4.1.3 Effect of Contact Time

Figure 4.3 shows the effect of contact time on the adsorption of ciprofloxacin at an initial concentration of 50 mg/L and a biochar dosage of 0.5 g/L. The adsorption capacity increases rapidly in the initial stages, reaching approximately 80% of the equilibrium value within the first 30 minutes. Thereafter, the adsorption process slows down and gradually reaches equilibrium after about 90 minutes of contact time. The adsorbent achieved their highest removal at 120 min.

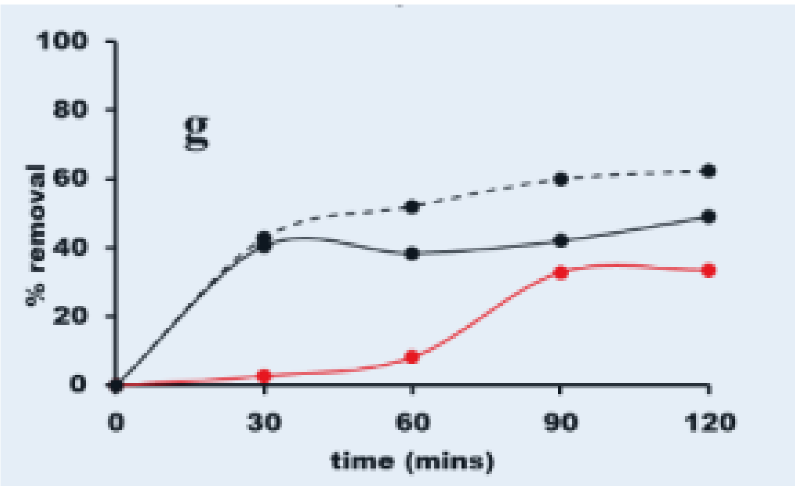

Figure 4.3 Effect of contact time on % removal of ciprofloxacin

4.1.4 Effect of Solution pH

The effect of solution pH on the adsorption of ciprofloxacin is presented in Figure 4.4. The adsorption capacity and removal efficiency exhibit a peak around pH 7, which is close to the pKa value of ciprofloxacin (pKa1 = 6.2, pKa2 = 8.7). At this pH, the ciprofloxacin molecule is in its zwitterionic form, allowing for favorable electrostatic interactions with the surface functional groups of the biochar.

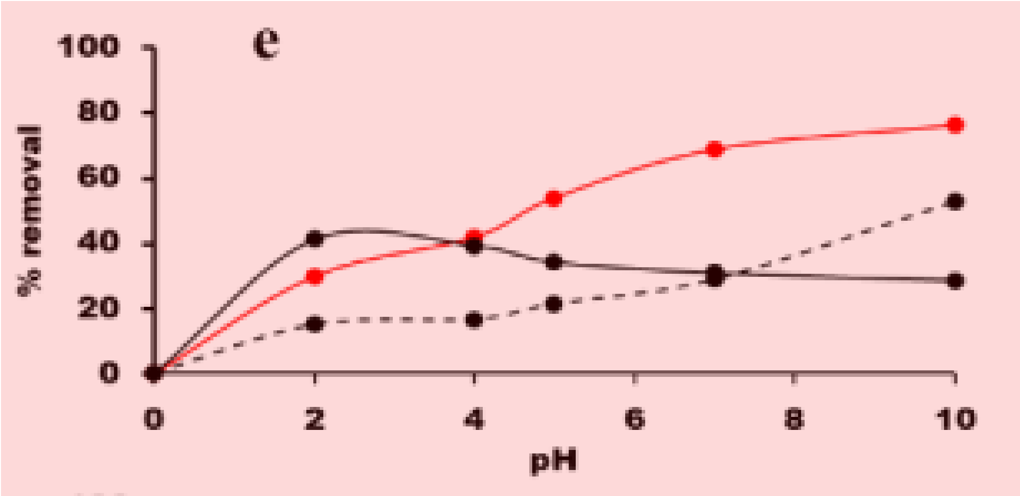

Figure 4.4 Effect of pH on % removal of ciprofloxacin

The results from the characterization and adsorption studies demonstrate the effectiveness of the bamboo-derived biochar for the removal of ciprofloxacin from aqueous solutions. The adsorption kinetics and the influence of operational parameters provide valuable insights into the mechanisms governing the adsorption process.

CHAPTER 5: CONCLUSION

FTIR analysis of the pristine and spent bamboo-derived biochar revealed changes in the surface functional groups, indicating the successful adsorption of ciprofloxacin. The appearance of new peaks corresponding to carboxyl and amine groups suggests that various mechanisms, such as hydrogen bonding, electrostatic interactions, and π-π interactions, were involved in the adsorption process.

UV-Vis spectroscopy demonstrated a progressive decrease in the absorbance of ciprofloxacin solutions as the biochar dosage was increased from 0.1 to 1.0 g/L. This observation is consistent with the enhanced removal of ciprofloxacin from the solution due to increased adsorption onto the biochar surface.

The adsorption kinetics showed a rapid initial uptake of ciprofloxacin, with approximately 80% of the equilibrium adsorption capacity reached within the first 30 minutes. The adsorption process then slowed down and reached equilibrium after around 90 minutes of contact time.

The effect of solution pH revealed a peak in the adsorption capacity and removal efficiency around pH 7, which is close to the pKa values of ciprofloxacin. At this pH, the ciprofloxacin molecule is in its zwitterionic form, enabling favorable electrostatic interactions with the surface functional groups of the biochar.

Overall, the characterization and adsorption studies demonstrated the effectiveness of the bamboo-derived biochar for the removal of ciprofloxacin from aqueous solutions. The insights gained into the adsorption mechanisms and the influence of operational parameters provide valuable information for the development and optimization of biochar-based water treatment systems.

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