

STUDY OF SARGASSUM WIGHTII BIOCHAR AND CHITOSAN COATED BIOCHAR FOR THE REMOVAL OF CIPROFLOXACIN

Submitted in partial fulfillment of the requirements for the award of
Bachelor of Technology degree in Chemical Engineering

By

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INSTITUTE OF SCIENCE AND TECHNOLOGY
(DEEMED TO BE UNIVERSITY)

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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **Arun. S. R (40190004)** and **Ramaraja. A (40190021)** who carried out the project entitled "**Study of Sargassum Wightii Biochar and Chitosan Coated Biochar for the Removal of Ciprofloxacin**" under our guidance from **November 2023 to April 2024**.

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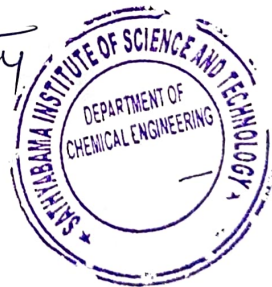
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ABSTRACT

This study investigated the removal of ciprofloxacin, a common antibiotic, from wastewater using *Sargassum* biochar (SWBC) and chitosan coated *Sargassum* biochar (CSWBC). The adsorption behaviors of both biochars were evaluated and compared.

Sargassum biochar exhibited a Freundlich isotherm model, indicating a multi-layered adsorption process. The maximum ciprofloxacin adsorption capacity ($Q_{\max}$) for SWBC was 4.91 mg/g. Kinetic studies revealed that ciprofloxacin adsorption onto SWBC followed a pseudo-first-order model, suggesting a physical adsorption mechanism. The negative enthalpy change (ΔH°) indicated an exothermic process, the negative Gibbs free energy change (ΔG°) suggested a spontaneous adsorption for SWBC. In contrast, chitosan coated *Sargassum* biochar followed a Langmuir isotherm model, signifying a monolayer adsorption process. The $Q_{\max}$ value for CSWBC was significantly higher (34.17 mg/g) compared to SWBC. Elovich kinetic model best described the ciprofloxacin adsorption onto CSWBC, implying a complex diffusion-based mechanism. The negative values for both ΔH° and ΔG° confirmed the exothermic and spontaneous nature of ciprofloxacin adsorption onto CSWBC. These findings demonstrate that chitosan coating significantly enhanced the ciprofloxacin adsorption capacity and spontaneity compared to unmodified *Sargassum* biochar. This study offers valuable insights into the potential of biochar-based materials for antibiotic removal from wastewater.

LIST OF ABBREVIATIONS

1. SWBC: *Sargassum Wightii* Biochar
2. CSWBC: Chitosan Coated *Sargassum Wightii* Biochar
3. SW: *Sargassum Wightii*
4. CIP: Ciprofloxacin
5. ppm: Parts Per Million
6. UV-Vis: Ultraviolet-Visible
7. FTIR: Fourier Transform Infrared Spectroscopy
8. SEM: Scanning Electron Microscopy
9. EDAX: Energy Dispersive X-ray Analysis
10. XRD: X-ray Diffraction
11. pH: Potential of Hydrogen

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CHAPTER - 1

INTRODUCTION

Wastewater contamination stands as an ominous testament to the complex interplay between human activity and the environment, embodying a myriad of pollutants that threaten ecological balance and human health. From industrial effluents to agricultural runoff, urban sewage to pharmaceutical residues, wastewater serves as a repository for diverse contaminants, each bearing its own set of environmental and societal implications. The discharge of untreated or inadequately treated wastewater into natural water bodies poses a grave risk, as pollutants infiltrate aquatic ecosystems, disrupt food chains, and compromise biodiversity. Heavy metals, organic chemicals, pathogens, and nutrients are among the notorious culprits that contaminate wastewater, originating from industrial processes, agricultural practices, domestic activities, and healthcare facilities. These contaminants, often present in complex mixtures, exhibit varying persistence and toxicity levels, amplifying the challenge of effective wastewater management. Moreover, the proliferation of emerging contaminants, such as pharmaceuticals and personal care products, adds a layer of complexity to the problem, as conventional treatment methods struggle to adequately address these novel pollutants. The consequences of wastewater contamination are far-reaching, extending beyond environmental degradation to encompass public health hazards, economic burdens, and social disparities. Vulnerable populations, including marginalized communities and developing regions, bear the brunt of inadequate sanitation infrastructure, suffering from waterborne diseases and compromised livelihoods. Despite concerted efforts to mitigate wastewater pollution through regulatory frameworks, technological innovations, and public awareness campaigns, the magnitude of the challenge persists, underscoring the urgent need for holistic and sustainable approaches to wastewater management. In this context, understanding the dynamics of wastewater contamination becomes paramount, as it lays the groundwork for informed decision-making, resource allocation, and collaborative action towards safeguarding water resources and promoting global sustainability.

1.1 PHARMACEUTICAL POLLUTION MITIGATION

1.1.1 EMERGING ENVIRONMENTAL THREATS: PHARMACEUTICAL POLLUTANTS

Emerging environmental threats, particularly pharmaceutical pollutants, represent a significant challenge at the nexus of public health and environmental sustainability. Pharmaceutical pollutants, comprising a diverse array of active pharmaceutical ingredients (APIs) and their metabolites, enter aquatic environments through various pathways, including excretion, improper disposal, and wastewater effluents from pharmaceutical manufacturing facilities and healthcare facilities. These compounds exhibit unique properties that differentiate them from traditional pollutants, including bioactivity at low concentrations, persistence in the environment, and potential to induce adverse effects on aquatic organisms and human health. Their presence in surface waters, groundwater, and even drinking water sources has raised concerns regarding ecological integrity, antimicrobial resistance, and potential human health risks associated with chronic exposure. Furthermore, the complex mixture of pharmaceutical pollutants, coupled with their interactions with other environmental contaminants and factors such as climate change and land use, complicates the assessment and management of these emerging threats. Addressing pharmaceutical pollution requires a multifaceted approach encompassing source reduction, improved wastewater treatment technologies, regulatory interventions, and public awareness initiatives. Collaboration among stakeholders, including government agencies, pharmaceutical industries, healthcare providers, environmental organizations, and the public, is essential to mitigate the impact of pharmaceutical pollutants and safeguard both environmental and human health. As research continues to advance our understanding of the fate, transport, and effects of pharmaceutical pollutants in the environment, concerted efforts are needed to develop innovative solutions and enact policies that promote sustainable pharmaceutical use and wastewater management practices.

1.1.2 THE NEED FOR INNOVATIVE WASTEWATER TREATMENT SOLUTIONS

The need for innovative wastewater treatment solutions has become increasingly urgent in light of escalating environmental challenges and growing pressures on

freshwater resources. Conventional wastewater treatment methods, while effective to some extent, are often unable to adequately address the complex mixture of contaminants present in wastewater, including emerging pollutants such as pharmaceuticals, personal care products, and microplastics. Moreover, traditional treatment processes may be energy-intensive, costly, and generate sludge or by-products that require further disposal. As populations continue to urbanize and industrialize, the volume and complexity of wastewater generated pose significant challenges for existing infrastructure and treatment capacity. In response, there is a pressing need for the development and adoption of innovative technologies and approaches that can enhance the efficiency, sustainability, and resilience of wastewater treatment systems. This includes the exploration of nature-based solutions, such as constructed wetlands and green infrastructure, which harness natural processes to remove contaminants and enhance water quality. Additionally, advances in membrane filtration, advanced oxidation processes, and electrochemical treatment hold promise for achieving higher removal efficiencies and treating wastewater to higher quality standards. Furthermore, the integration of decentralized treatment systems, smart sensors, and data analytics can improve monitoring and control, optimize resource recovery, and enhance the overall performance of wastewater treatment plants. Collaborative research efforts involving academia, industry, and government agencies are essential for driving innovation and overcoming barriers to the adoption of novel wastewater treatment solutions. By embracing innovation and leveraging technological advancements, we can create more sustainable and resilient wastewater treatment systems that protect human health, safeguard the environment, and promote the wise stewardship of our precious water resources.

1.1.3 UNDERSTANDING CIPROFLOXACIN AS A PERSISTENT CONTAMINANT

Understanding ciprofloxacin as a persistent contaminant is crucial in addressing its widespread presence and potential environmental impacts. Ciprofloxacin, a broad-spectrum fluoroquinolone antibiotic widely used in human and veterinary medicine, has garnered attention due to its persistence in aquatic environments. Despite its intended therapeutic effects, ciprofloxacin can enter wastewater systems through various pathways, including excretion by humans and animals, disposal of unused medications, and discharge from pharmaceutical manufacturing facilities. Once

introduced into water bodies, ciprofloxacin exhibits resistance to degradation processes, such as photolysis, hydrolysis, and microbial metabolism, leading to its accumulation over time. This persistence is attributed to the chemical structure of ciprofloxacin, which contains fluorine atoms that enhance its stability and resistance to environmental degradation. Furthermore, factors such as pH, temperature, and the presence of other chemicals can influence the rate of ciprofloxacin degradation, further exacerbating its persistence in aquatic ecosystems. The long-term presence of ciprofloxacin in the environment raises concerns about potential adverse effects on aquatic organisms, including antibiotic resistance development, disruption of microbial communities, and ecological imbalances. Additionally, there is growing evidence of ciprofloxacin's role in promoting the spread of antibiotic resistance genes, posing a threat to public health and exacerbating the global challenge of antimicrobial resistance. To address the persistence of ciprofloxacin as a contaminant, interdisciplinary research efforts are needed to elucidate its fate and transport mechanisms, assess its ecological and human health risks, and develop effective mitigation strategies. This includes the implementation of advanced analytical techniques for detecting and quantifying ciprofloxacin in environmental matrices, modelling approaches to predict its environmental fate, and innovative treatment technologies for its removal from wastewater. Furthermore, collaborative initiatives involving policymakers, healthcare professionals, pharmaceutical industries, and environmental scientists are essential to enact regulatory measures, promote responsible antibiotic use, and minimize the environmental footprint of ciprofloxacin and other pharmaceutical pollutants. By enhancing our understanding of ciprofloxacin as a persistent contaminant and implementing proactive measures to mitigate its environmental impact, we can strive towards a more sustainable and resilient aquatic ecosystem.

1.1.4 ROLE OF BIOCHAR IN SUSTAINABLE ENVIRONMENTAL REMEDIATION

The role of biochar in sustainable environmental remediation encompasses a multifaceted approach to addressing diverse environmental challenges through the application of this carbonaceous material derived from biomass pyrolysis. Biochar, renowned for its high surface area, porosity, and carbon sequestration potential, holds promise as a versatile tool for mitigating various environmental issues, including soil degradation, water pollution, and climate change. In soil remediation,

biochar amendments enhance soil fertility, improve water retention capacity, and sequester carbon, thereby promoting soil health and productivity while mitigating greenhouse gas emissions. Additionally, biochar acts as a sorbent for contaminants, such as heavy metals, organic pollutants, and nutrients, facilitating their immobilization and reducing their bioavailability in soil and water systems. In water remediation, biochar-based materials demonstrate exceptional adsorption capacity for pollutants, including pesticides, pharmaceuticals, and emerging contaminants, offering a cost-effective and sustainable solution for water treatment. Moreover, biochar-mediated nutrient removal from agricultural runoff and wastewater streams mitigates eutrophication and improves water quality in aquatic ecosystems. Beyond its direct remediation capabilities, biochar contributes to climate change mitigation through carbon sequestration, as its stable carbon structure can persist in soils for hundreds to thousands of years, effectively removing atmospheric carbon dioxide. Furthermore, biochar production from organic waste materials offers a means of waste valorisation and resource recovery, closing nutrient loops and reducing reliance on fossil fuels. However, the efficacy of biochar in environmental remediation is influenced by various factors, including feedstock selection, pyrolysis conditions, and application rates, necessitating careful consideration of site-specific conditions and objectives. Collaborative research efforts, involving academia, industry, and government agencies, are essential to advancing the science and technology of biochar-based environmental remediation and realizing its full potential in promoting sustainability and resilience in terrestrial and aquatic ecosystems.

1.1.5 EXPLORING SARGASSUM BIOMASS AS A RENEWABLE RESOURCE

Exploring *Sargassum* biomass as a renewable resource unveils a plethora of opportunities for sustainable utilization across various sectors, ranging from agriculture to energy production and environmental remediation. *Sargassum*, a genus of brown macroalgae, thrives in marine ecosystems, forming extensive floating mats known as *Sargassum wightii*. With its rapid growth rate, high biomass yield, and ability to absorb nutrients and carbon dioxide from seawater, *Sargassum* represents a promising renewable resource for multiple applications. In agriculture, *Sargassum* biomass serves as a nutrient-rich organic fertilizer, enhancing soil fertility, promoting crop growth, and improving yield quality. Its bioactive compounds,

such as alginates, polysaccharides, and plant hormones, confer beneficial effects on soil structure, water retention, and plant resilience to environmental stressors. Furthermore, *Sargassum* extracts have demonstrated potential as bio stimulants and biopesticides, offering natural alternatives to synthetic agrochemicals and supporting sustainable agricultural practices. In the energy sector, *Sargassum* biomass holds promise as a feedstock for biofuel production, particularly through anaerobic digestion and thermochemical conversion processes. Its high carbohydrate content, low lignin content, and fast growth rate make *Sargassum* an attractive candidate for bioenergy generation, providing a renewable and carbon-neutral alternative to fossil fuels. Moreover, *Sargassum*-derived biochar exhibits excellent adsorption properties, making it effective for wastewater treatment, soil remediation, and carbon sequestration applications. By harnessing *Sargassum* biomass as a renewable resource, we can not only mitigate environmental degradation but also foster economic development, enhance food security, and promote climate resilience in coastal communities. However, sustainable management practices and ecosystem-based approaches are essential to ensure the long-term viability of *Sargassum* resources and minimize potential ecological impacts. Collaborative research efforts, stakeholder engagement, and policy support are critical to unlocking the full potential of *Sargassum* biomass and realizing its role in the transition towards a more sustainable and resilient future.

1.1.6 INTRODUCTION TO CHITOSAN: A BIOPOLYMER WITH ADSORPTION POTENTIAL

Chitosan, a versatile biopolymer derived from the deacetylation of chitin, presents itself as a promising material with remarkable adsorption potential across various applications. With its unique properties, including biocompatibility, biodegradability, and non-toxicity, chitosan has garnered significant attention in diverse fields such as wastewater treatment, environmental remediation, biomedical engineering, and food technology. The abundance of chitin in natural sources such as crustacean shells, fungal cell walls, and certain insects, coupled with the relative ease of chitosan extraction and modification, underscores its attractiveness as a sustainable alternative to synthetic adsorbents. In wastewater treatment, chitosan-based adsorbents exhibit high affinity towards a wide range of contaminants, including heavy metals, dyes, organic pollutants, and emerging micropollutants, owing to their

functional groups (e.g., amino and hydroxyl groups) that facilitate adsorption interactions. Moreover, chitosan's intrinsic antimicrobial properties contribute to its efficacy in inhibiting bacterial growth and promoting pollutant removal efficiency. Beyond wastewater treatment, chitosan finds applications in environmental remediation, where it is utilized for the removal of contaminants from soil, air, and groundwater, thereby mitigating environmental pollution and safeguarding human health. In biomedical engineering, chitosan's biocompatibility and mucoadhesive properties make it an ideal candidate for drug delivery systems, wound healing dressings, tissue engineering scaffolds, and biomedical implants. Additionally, chitosan's ability to form films, gels, and coatings enhances its utility in food packaging, preservation, and safety, offering sustainable solutions to reduce food waste and extend shelf life. Despite its numerous advantages, challenges remain in optimizing chitosan-based adsorbents for specific applications, including improving adsorption capacity, selectivity, and reusability, as well as addressing cost considerations and scalability issues. Collaborative research efforts, multidisciplinary approaches, and technological innovations are essential to harnessing the full potential of chitosan as a biopolymer with adsorption potential, advancing sustainable development goals, and addressing global environmental and health challenges.

1.1.7 BRIDGING NATURE AND TECHNOLOGY: SARGASSUM BIOCHAR SYNTHESIS

Sargassum biochar synthesis represents a novel and sustainable approach to addressing environmental challenges while harnessing the potential of marine biomass. *Sargassum*, a brown macroalgae abundant in coastal regions, offers a readily available and renewable feedstock for biochar production. By subjecting *Sargassum* biomass to pyrolysis, a thermochemical conversion process under oxygen-limited conditions, biochar is synthesized, retaining the carbon-rich structure of the original biomass. This biochar, enriched with carbonaceous materials and mineral nutrients, exhibits unique properties that make it an attractive material for various applications. In environmental remediation, *Sargassum* biochar demonstrates exceptional adsorption capacity for pollutants, including heavy metals, organic contaminants, and nutrients, thereby mitigating soil and water pollution and promoting ecosystem health. Moreover, *Sargassum* biochar enhances

soil fertility, improves soil structure, and sequesters carbon, contributing to sustainable agriculture and climate change mitigation efforts. Beyond its role in environmental applications, *Sargassum* biochar finds utility in renewable energy production, where it serves as a feedstock for bioenergy generation through combustion, gasification, or pyrolysis processes, offering a carbon-neutral alternative to fossil fuels. Furthermore, *Sargassum* biochar-derived products, such as biochar-based fertilizers, soil amendments, and carbon sequestration agents, hold promise for enhancing agricultural productivity, restoring degraded lands, and promoting circular economy principles. However, challenges remain in optimizing *Sargassum* biochar synthesis processes, such as pyrolysis conditions, feedstock characteristics, and biochar properties, to maximize efficiency, yield, and quality. Additionally, considerations regarding the scalability, economic viability, and environmental sustainability of *Sargassum* biochar production warrant further research and development. Collaborative efforts between scientists, engineers, policymakers, and stakeholders are essential to advancing *Sargassum* biochar synthesis technologies, fostering innovation, and realizing the full potential of this nature-inspired approach in bridging nature and technology for sustainable development.

1.1.8 ENHANCING ADSORPTION CAPACITY THROUGH THE COATING OF BIOCHAR WITH CHITOSAN

Enhancing adsorption capacity through the coating of biochar with chitosan represents a promising strategy for improving the effectiveness of biochar-based adsorbents in various environmental applications. Biochar, derived from the pyrolysis of organic biomass, possesses a high surface area and porosity, making it an excellent candidate for adsorption processes. However, the inherent limitations of bare biochar, such as low selectivity and affinity towards specific contaminants, can hinder its performance in complex wastewater matrices. By coating biochar with chitosan, a biopolymer renowned for its adsorption capabilities and surface modification properties, the resulting composite material can exhibit enhanced adsorption capacity, selectivity, and stability. The chitosan coating provides functional groups, such as amino and hydroxyl groups, that facilitate the binding of target pollutants through electrostatic interactions, hydrogen bonding, and complexation mechanisms. Additionally, chitosan imparts additional benefits to the

biochar matrix, including improved mechanical strength, resistance to leaching, and compatibility with aqueous environments. The synergistic effects between biochar and chitosan not only enhance adsorption performance but also expand the applicability of the composite adsorbent to a wider range of contaminants, including heavy metals, organic pollutants, and emerging micropollutants. Furthermore, the sustainable nature of both biochar and chitosan, derived from renewable biomass sources, underscores the eco-friendly and cost-effective attributes of this innovative adsorption technology. Through systematic characterization studies, optimization of coating processes, and rigorous evaluation in batch and continuous flow systems, the potential of biochar-chitosan composites can be fully realized, offering a sustainable solution for wastewater treatment, environmental remediation, and resource recovery.

1.1.9 ADDRESSING RESEARCH GAPS IN PHARMACEUTICAL POLLUTION MITIGATION

Addressing research gaps in pharmaceutical pollution mitigation is imperative for understanding and effectively managing the environmental impacts of pharmaceutical residues on aquatic ecosystems and human health. While pharmaceuticals play a crucial role in healthcare, their incomplete removal during wastewater treatment processes leads to their release into natural water bodies, posing potential risks to aquatic organisms and ecosystems. The complexity of pharmaceutical pollution stems from the diverse chemical compositions, varied modes of action, and differing environmental fates of pharmaceutical compounds. Furthermore, the interactions between pharmaceuticals and environmental matrices, such as sediments, soils, and biota, remain poorly understood, hindering accurate risk assessments and mitigation strategies. Key research gaps include elucidating the ecotoxicological effects of pharmaceuticals on aquatic organisms, understanding the mechanisms of bioaccumulation and biomagnification in food webs, and assessing the long-term ecological consequences of chronic exposure to low concentrations of pharmaceuticals. Additionally, there is a need to develop sensitive analytical methods for detecting and quantifying pharmaceutical residues in environmental samples, as well as predictive modelling tools for assessing the fate and transport of pharmaceuticals in aquatic systems. Moreover, interdisciplinary approaches integrating environmental science, toxicology,

pharmacology, chemistry, and engineering are essential for bridging knowledge gaps, generating holistic insights into pharmaceutical pollution dynamics, and informing evidence-based decision-making processes. By addressing these research gaps, stakeholders can develop effective mitigation strategies, such as advanced wastewater treatment technologies, source control measures, and regulatory frameworks, to minimize pharmaceutical pollution and protect both environmental and human health.

1.1.10 ENVIRONMENTAL IMPLICATIONS OF CIPROFLOXACIN PERSISTENCE

The environmental implications of ciprofloxacin persistence are multifaceted and underscore the complex interplay between pharmaceutical contamination and ecosystem health. Ciprofloxacin, a widely prescribed fluoroquinolone antibiotic, exhibits remarkable stability and persistence in aquatic environments due to its resistance to degradation processes such as photolysis, hydrolysis, and microbial metabolism. This persistence results in its accumulation in water bodies, sediments, and soils, leading to potential adverse effects on aquatic organisms, ecosystem functioning, and human health. One significant environmental implication is the development of antibiotic resistance among bacteria in aquatic ecosystems exposed to ciprofloxacin residues. The selective pressure exerted by ciprofloxacin contamination can promote the proliferation of antibiotic-resistant bacterial strains, including those with clinical relevance, posing a threat to public health and complicating the treatment of bacterial infections. Moreover, ciprofloxacin persistence can disrupt microbial communities and ecological processes in aquatic environments, leading to imbalances in nutrient cycling, decreased biodiversity, and altered ecosystem dynamics. Ciprofloxacin residues may also bioaccumulate and bio magnify through aquatic food webs, potentially impacting higher trophic levels, including fish and aquatic birds, and posing risks to human consumers. Furthermore, ciprofloxacin persistence raises concerns about its potential long-term effects on ecosystem resilience, ecosystem services, and the sustainability of aquatic habitats. Mitigating the environmental implications of ciprofloxacin persistence requires a holistic approach that integrates regulatory measures, advanced wastewater treatment technologies, pollution prevention strategies, and public awareness campaigns. By addressing the root causes of pharmaceutical contamination, promoting responsible antibiotic use, and implementing sustainable

water management practices, stakeholders can minimize the environmental footprint of ciprofloxacin and safeguard aquatic ecosystems for future generations.

1.2 ADSORPTION KINETICS AND EFFICIENCY

1.2.1 RATIONALE FOR STUDYING CIPROFLOXACIN REMOVAL

The rationale for studying ciprofloxacin removal is rooted in the recognition of the significant environmental and public health risks posed by the widespread presence of this antibiotic in aquatic ecosystems. Ciprofloxacin, a broad-spectrum fluoroquinolone antibiotic commonly used in both human and veterinary medicine, has been detected in various environmental compartments, including surface waters, groundwater, and wastewater treatment plants. Its persistence and resistance to degradation in aquatic environments raise concerns about its potential adverse effects on ecosystem health, biodiversity, and human health. Ciprofloxacin contamination is particularly worrisome due to its association with the development of antibiotic-resistant bacteria, which can compromise the effectiveness of clinical treatments and escalate the spread of antibiotic resistance in the environment. Furthermore, ciprofloxacin's presence in aquatic ecosystems can disrupt microbial communities, interfere with nutrient cycling processes, and alter the ecological balance of aquatic habitats. Given these environmental and public health implications, there is a critical need to understand the fate, transport, and behaviour of ciprofloxacin in aquatic environments, as well as develop effective mitigation strategies for its removal from water systems. By investigating the mechanisms of ciprofloxacin sorption onto adsorbent materials, evaluating the performance of treatment technologies for ciprofloxacin removal, and assessing the environmental fate of ciprofloxacin and its degradation products, research endeavours aim to provide insights into the risks associated with ciprofloxacin contamination and inform strategies for mitigating its environmental impact. Through interdisciplinary collaborations between environmental scientists, chemists, engineers, and public health experts, studies on ciprofloxacin removal seek to contribute to the development of sustainable solutions for protecting water quality, safeguarding ecosystems, and preserving public health.

1.2.2 SUSTAINABLE APPROACH: UTILIZING SARGASSUM BIOMASS FOR BIOCHAR PRODUCTION

A sustainable approach to utilizing *Sargassum* biomass for biochar production holds immense potential for addressing environmental challenges, promoting resource efficiency, and fostering economic development in coastal regions. *Sargassum*, a genus of brown macroalgae abundant in marine ecosystems, offers a renewable and readily available feedstock for biochar production. By subjecting *Sargassum* biomass to pyrolysis, a thermochemical conversion process conducted in the absence of oxygen, biochar is generated, retaining the carbon-rich structure of the original biomass while converting volatile organic compounds into stable carbonaceous materials. This sustainable approach to biochar production not only sequesters carbon and mitigates greenhouse gas emissions but also addresses the issue of marine debris accumulation and beach inundation caused by *Sargassum* blooms. Moreover, *Sargassum*-derived biochar exhibits exceptional properties that make it suitable for various environmental applications, including soil amendment, carbon sequestration, and wastewater treatment. In agriculture, *Sargassum* biochar enhances soil fertility, improves soil structure, and promotes plant growth by supplying essential nutrients, enhancing water retention capacity, and stimulating microbial activity. Furthermore, *Sargassum* biochar can serve as a cost-effective and environmentally friendly alternative to conventional fertilizers and soil conditioners, reducing dependence on synthetic chemicals and minimizing nutrient runoff and leaching. Additionally, *Sargassum* biochar shows promise for wastewater treatment applications, where its high surface area and porosity enable efficient adsorption of pollutants, including heavy metals, organic contaminants, and nutrients, from aqueous solutions. By harnessing *Sargassum* biomass for biochar production, stakeholders can simultaneously address marine pollution, climate change, and soil degradation challenges while creating opportunities for sustainable development and circular economy initiatives in coastal communities.

1.2.3 INVESTIGATING THE PHYSICOCHEMICAL PROPERTIES OF SARGASSUM BIOCHAR

Investigating the physicochemical properties of *Sargassum* biochar is essential for understanding its suitability and potential applications in various environmental and agricultural contexts. *Sargassum* biochar, derived from the pyrolysis of *Sargassum*

biomass, possesses unique characteristics that contribute to its effectiveness as a soil amendment, pollutant adsorbent, and carbon sequestration agent. Physically, *Sargassum* biochar exhibits a porous structure with a high surface area, which enhances its adsorption capacity and facilitates interactions with soil particles and contaminants in aqueous solutions. The porosity of *Sargassum* biochar influences its water-holding capacity, nutrient retention, and microbial habitat suitability in soil environments, thereby improving soil fertility, moisture retention, and overall productivity. Chemically, *Sargassum* biochar is rich in carbonaceous materials, functional groups (e.g., carboxyl, hydroxyl, and phenolic groups), and mineral constituents derived from the original biomass, which contribute to its sorption capacity, cation exchange capacity, and nutrient availability in soil systems. Understanding the physicochemical properties of *Sargassum* biochar requires comprehensive characterization techniques, including surface area analysis, pore size distribution, elemental composition analysis, Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray diffraction (XRD). Through systematic investigation of these properties, researchers can elucidate the mechanisms underlying *Sargassum* biochar's performance in soil amendment, pollutant remediation, and carbon sequestration applications, as well as optimize its production processes and tailor its properties for specific environmental and agricultural needs. By harnessing the insights gained from studying the physicochemical properties of *Sargassum* biochar, stakeholders can develop innovative and sustainable solutions for addressing soil degradation, water pollution, and climate change challenges, thereby promoting environmental resilience and agricultural sustainability.

1.2.4 SURFACE MODIFICATION TECHNIQUES: COATING BIOCHAR WITH CHITOSAN

Coating of biochar with chitosan present a promising avenue for enhancing the performance and versatility of biochar-based adsorbents in environmental applications. Biochar, a carbon-rich material derived from the pyrolysis of organic biomass, possesses a porous structure and high surface area that lend it exceptional adsorption properties. However, the inherent limitations of bare biochar, such as low selectivity and affinity towards specific contaminants, can hinder its effectiveness in pollutant removal. Surface modification techniques, particularly

coating biochar with chitosan, offer a solution to overcome these limitations and tailor biochar properties to target contaminants. Chitosan, exhibits favourable characteristics for surface modification, including biocompatibility, biodegradability, and functional groups (e.g., amino and hydroxyl groups) that facilitate interactions with contaminants. The coating process involves immersing biochar particles in a chitosan solution, followed by drying and curing to form a stable chitosan layer on the biochar surface. This chitosan coating imparts additional functionalities to the biochar matrix, such as improved selectivity, enhanced stability, and controlled release of active sites for adsorption. Moreover, chitosan-coated biochar exhibits enhanced resistance to leaching and aggregation, prolonging its effectiveness in aqueous environments. The synergistic effects between biochar and chitosan result in composite materials with superior adsorption capacity and selectivity for a wide range of contaminants, including heavy metals, organic pollutants, and emerging micropollutants. Furthermore, chitosan-coated biochar demonstrates potential applications in wastewater treatment, soil remediation, and water purification, offering sustainable solutions for mitigating environmental pollution and promoting resource recovery. Through further research and development, optimization of coating techniques, and rigorous evaluation of performance in real-world conditions, chitosan-coated biochar holds promise for addressing complex environmental challenges and advancing towards a more sustainable future.

1.2.5 ANALYTICAL METHODS FOR CHARACTERIZING ADSORBENT MATERIALS

Analytical methods for characterizing adsorbent materials play a crucial role in understanding their physicochemical properties, surface characteristics, and adsorption mechanisms, thereby facilitating the design and optimization of adsorption processes for environmental remediation and purification applications. Various techniques are employed to comprehensively analyse adsorbent materials, providing insights into their structure, composition, morphology, surface area, porosity, and surface chemistry. Scanning electron microscopy (SEM) allow for visualization of adsorbent morphology and particle size distribution, enabling characterization of surface features, such as pores, cracks, and surface roughness. X-ray diffraction (XRD) analysis provides insights into the crystalline structure and phase composition of adsorbent materials, aiding in identifying mineral phases and

structural changes upon modification or functionalization. Fourier-transform infrared spectroscopy (FTIR) offer valuable information on the chemical composition, functional groups, and surface chemistry of adsorbents, facilitating the identification of surface functional groups and interactions involved in adsorption processes. By employing a combination of these analytical methods, researchers can comprehensively characterize adsorbent materials, elucidate their adsorption mechanisms, and optimize their performance for various environmental and industrial applications, ultimately contributing to the development of efficient and sustainable adsorption processes.

1.2.6 DESIGNING BATCH ADSORPTION EXPERIMENTS FOR CIPROFLOXACIN REMOVAL

Designing batch adsorption experiments for ciprofloxacin removal involves a systematic approach to evaluate the adsorption efficiency of adsorbent materials under controlled laboratory conditions, providing valuable insights into the adsorption kinetics, equilibrium behaviour, and influencing factors. The experimental design encompasses several key aspects, including selection of adsorbent materials, preparation of ciprofloxacin solutions, determination of experimental parameters, and data analysis methods. Firstly, adsorbent materials with potential for ciprofloxacin removal are chosen based on their physicochemical properties, surface characteristics, and previous performance in adsorption studies. These materials may include activated carbon, biochar, modified clays, and functionalized polymers, among others. Secondly, ciprofloxacin solutions are prepared at desired concentrations, typically ranging from environmentally relevant levels to higher concentrations for adsorption isotherm studies, ensuring a representative range of adsorption conditions. Thirdly, experimental parameters such as initial ciprofloxacin concentration, adsorbent dosage, solution pH, temperature, agitation speed, and contact time are systematically varied to investigate their effects on adsorption performance. A series of batch experiments are conducted, where adsorbent materials are added to ciprofloxacin solutions in sealed containers, and samples are withdrawn at regular intervals to monitor changes in ciprofloxacin concentration over time. Analytical technique UV-visible spectrophotometry employed to quantify ciprofloxacin concentrations in solution before and after adsorption. The obtained data is then analysed using kinetic models

(e.g., pseudo-first-order, pseudo-second-order), isotherm models (e.g., Langmuir, Freundlich), and thermodynamic parameters (e.g., ΔG , ΔH , ΔS) to elucidate the adsorption mechanism, determine adsorption kinetics, equilibrium capacities, and thermodynamic feasibility. By systematically designing and conducting batch adsorption experiments, researchers can gain valuable insights into the feasibility and efficiency of adsorbent materials for ciprofloxacin removal, informing the development of effective adsorption processes for water treatment and environmental remediation applications.

1.2.7 ASSESSING FACTORS INFLUENCING ADSORPTION EFFICIENCY

Assessing factors influencing adsorption efficiency is critical for understanding the performance and optimizing the effectiveness of adsorbent materials in removing contaminants from aqueous solutions. Numerous factors can impact adsorption efficiency, including adsorbate properties, adsorbent characteristics, solution conditions, and external factors. Adsorbate properties such as molecular size, polarity, charge, and concentration influence its affinity and interactions with the adsorbent surface. Additionally, adsorbent characteristics such as surface area, pore size distribution, surface chemistry, and functional groups determine the adsorption capacity, selectivity, and kinetics. Solution conditions, including pH, temperature, ionic strength, and presence of competing ions, can significantly affect adsorption efficiency by altering the electrostatic interactions, surface charge, and solubility of both adsorbate and adsorbent. External factors such as agitation speed, contact time, adsorbent dosage, and particle size distribution also play crucial roles in determining adsorption efficiency by influencing mass transfer kinetics, diffusion rates, and equilibrium attainment. To assess the factors influencing adsorption efficiency, systematic experimental studies are conducted, where individual factors are varied while keeping others constant, and the resulting adsorption performance is evaluated. Kinetic modelling, isotherm analysis, thermodynamic calculations, and surface characterization techniques are utilized to understand the underlying mechanisms, quantify adsorption parameters, and predict adsorption behaviour under different conditions. By systematically assessing the factors influencing adsorption efficiency, researchers can optimize adsorption processes, design tailored adsorbents, and develop effective strategies for water treatment, environmental remediation, and resource recovery applications.

1.2.8 ADSORPTION KINETICS AND THERMODYNAMICS

Exploring adsorption kinetics and thermodynamics is essential for understanding the mechanisms, rates, and energetics of adsorption processes, providing valuable insights into the feasibility, efficiency, and optimization of adsorption-based technologies for environmental remediation and purification. Adsorption kinetics refers to the study of how adsorbates are adsorbed onto adsorbent surfaces over time, while adsorption thermodynamics involves investigating the energy changes and spontaneity of adsorption processes. The study of adsorption kinetics involves determining the rate at which adsorption occurs and understanding the factors that influence the rate-controlling steps, such as mass transfer, film diffusion, pore diffusion, and surface reaction. Various kinetic models, including pseudo-first-order, pseudo-second-order, intra-particle diffusion, and Elovich models, are employed to analyse experimental data and elucidate the underlying mechanisms of adsorption. Additionally, factors such as initial adsorbate concentration, temperature, agitation speed, and adsorbent properties are systematically varied to investigate their effects on adsorption kinetics. On the other hand, adsorption thermodynamics examines the equilibrium behaviour and energetics of adsorption processes, including the Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) changes associated with adsorption. Thermodynamic parameters are calculated from adsorption isotherm data using thermodynamic equations, such as the Van's Hoff equation, and provide insights into the spontaneity, feasibility, and stability of adsorption under different conditions. Furthermore, thermodynamic studies enable the determination of optimal operating conditions, the prediction of adsorption behaviour at different temperatures, and the assessment of the suitability of adsorbents for specific applications. By exploring adsorption kinetics and thermodynamics, researchers can gain a comprehensive understanding of adsorption processes, optimize adsorption systems, and develop efficient and sustainable solutions for water treatment, pollutant removal, and resource recovery applications.

1.2.9 TOWARDS SUSTAINABLE WASTEWATER MANAGEMENT: IMPLICATIONS OF THE STUDY

The implications of the study on the removal efficiency of ciprofloxacin using *Sargassum* biochar and chitosan-coated *Sargassum* biochar are profound and multifaceted. Firstly, the research contributes to addressing the growing concern of pharmaceutical pollution in aquatic environments, particularly the presence of antibiotics like ciprofloxacin, which pose risks to ecosystem health and human well-being. By demonstrating the effectiveness of *Sargassum* biochar and chitosan-coated biochar in removing ciprofloxacin from wastewater, the study provides tangible solutions for mitigating pharmaceutical contamination and improving water quality. Moreover, the utilization of *Sargassum* biomass as a renewable resource for biochar production highlights the potential for sustainable waste valorisation and resource recovery, reducing reliance on traditional carbon sources and contributing to circular economy principles. Additionally, the environmentally benign nature of chitosan, a biodegradable biopolymer derived from natural sources, underscores the eco-friendly aspects of the adsorption process. Furthermore, the study sheds light on the feasibility of employing biochar-based adsorbents in decentralized wastewater treatment systems, offering cost-effective and scalable solutions for wastewater management in both rural and urban settings. By integrating these findings into wastewater treatment practices, stakeholders can advance towards more sustainable and resilient water management strategies, protecting aquatic ecosystems, safeguarding public health, and promoting the sustainable use of natural resources.

CHAPTER – 2

LITERATURE SURVEY

Thilakshani Atugoda et Al (2021) In this investigation, the innovative approaches to environmental remediation, such as the modification of biochar for enhanced antibiotic removal from water, hold significant promise in addressing contemporary challenges. In this comprehensive study, *Sargassum crassifolium*, a brown sea weed, underwent pyrolysis at 500°C, yielding biochar denoted as SWBC. Notably, this SWBC was further refined through a slurry method, incorporating zeolite at a precise ratio of 1:5 (zeolite: biochar) to produce SWBC-Z, an advanced composite material. Batch adsorption experiments were meticulously conducted to assess the adsorption potential of both SWBC and SWBC-Z in eliminating ciprofloxacin (CPX) from aqueous solutions. Fascinatingly, the adsorption process exhibited a pronounced affinity within the pH range of 6.5–8, attributed to compelling electrostatic attractions and hydrogen bonding interactions between the zwitterionic CPX molecules and the modified biochar surface. Kinetic modelling unveiled an excellent fit with the pseudo-second order and Elovich models, indicating complex adsorption mechanisms driven by chemisorption and physisorption phenomena. Equally compelling, isotherm data were aptly described by the Langmuir and Freundlich models, underscoring the heterogeneous nature of the biochar surface and the diverse adsorption mechanisms at play. Remarkably, SWBC-Z exhibited a remarkable maximum adsorption capacity of 93.65 mg g⁻¹, highlighting the efficacy of zeolite modification in enhancing adsorption performance. Further characterization through Fourier Transform Infrared (FTIR) spectroscopy revealed well-defined peaks corresponding to silanol groups in SWBC-Z, affirming the successful integration of zeolite minerals into the biochar matrix. Additionally, electron microscopy provided compelling visual evidence of the morphological changes and the incorporation of zeolite within the SWBC-Z structure. Post-adsorption FTIR analysis unveiled significant alterations in the surface functional groups of SWBC-Z, further corroborating the adsorption process's intricacies. Thermodynamic analysis underscored the spontaneous and exothermic nature of the reaction between CPX and both biochars, emphasizing the energy-efficient nature of the adsorption process. In sum, the synergistic modification of pristine

biochar with zeolite engendered notable improvements in surface area and adsorption capacity, showcasing its immense potential for sustainable water treatment applications and heralding a paradigm shift in environmental remediation strategies.

Ganning Zeng et Al (2020) In this innovative study, a ground breaking polymer/activated carbon composite was meticulously crafted through a streamlined one-step chemical activation process of *Sargassum horneri*, a marine macroalgae species, coupled with subsequent surface modification using chitosan, a ubiquitous and renewable biopolymer. This composite material, devised with the noble objective of combatting environmental pollution, particularly targeted the removal of one of the most pernicious metal ions, chromium (VI), from aqueous solutions. Through rigorous surface characterization analyses, it was revealed that the composite exhibited predominantly mesoporous characteristics, boasting a commendable specific surface area of $293.4 \text{ m}^2 \cdot \text{g}^{-1}$ and an average pore diameter of 6.27 nm. This structural configuration played a pivotal role in augmenting the uptake capacity of chromium (VI), as the composite harboured an abundance of metal-binding functional groups, notably amino and hydroxyl groups, strategically situated on its surface. Leveraging the porous nature of the composite, an exceptionally rapid adsorption rate of chromium (VI) was achieved, underscoring its efficacy in swiftly sequestering the contaminant from aqueous media. This seminal study unequivocally demonstrates the potential of *Sargassum horneri* as a viable precursor for the synthesis of carbon-based adsorbents, presenting a sustainable and eco-friendly alternative in the realm of heavy metal remediation. Furthermore, the tailored design of polymer/activated carbon composites heralds a new era in the development of bespoke adsorbent materials, poised to address the pressing challenges posed by environmental pollution and safeguard the health and well-being of ecosystems and communities worldwide.

Gangfu Song et Al (2019) In this insightful investigation, the distinctive surface properties of biochars derived from sea weed *Sargassum sp.* were meticulously scrutinized, highlighting their discernible disparities compared to biochars sourced from agricultural wastes. The study delved into the adsorptive removal dynamics of two prominent antibiotics, tetracycline (TC) and cefradine (CF), on the sea weed

biochar, unravelling intriguing insights into their comparative adsorption behaviours. Notably, the rod-like tissue structure of the raw sea weed underwent a profound transformation, transitioning into spindle-like morphology upon pyrolysis at temperatures exceeding 400°C. Concurrently, a substantial augmentation in carbon content, from 22.9% in the raw sea weed to 37.2% in the biochar pyrolyzed at 600°C (BC600), was observed, with a marginal increase in oxygen content from 24.7% to 27.8%. The adsorption of both TC and CF on the biochar exhibited a pronounced pH dependency, with the Langmuir model revealing maximum adsorption capacities of 128.1 mg/g and 61.7 mg/g for TC and CF, respectively. Notably, a higher affinity towards CF molecules was observed at lower adsorbate concentrations. The adsorption mechanism was elucidated to be governed predominantly by Coulombic interactions and π - π electron-donor-acceptor interactions between the sea weed biochar and CF/TC molecules, with the pseudo-second-order kinetics model aptly describing the experimental data, suggestive of a plausible chemisorption process. Furthermore, the isotherm analysis hinted at the concomitant contributions of surface adsorption and partitioning mechanisms to the uptake of TC and CF onto BC600, underscoring the multifaceted nature of the adsorption phenomenon. This comprehensive study not only sheds light on the distinct attributes of sea weed derived biochars but also underscores their potential as effective adsorbents for the removal of antibiotic contaminants, thereby paving the way for sustainable and efficient water treatment strategies.

Thanh-Binh Nguyen et Al (2022) In this investigation, the synthesis of biochars derived from three distinct species of algae—green algae *Ulva Ohnoi*, red algae *Agardhiella subulata*, and brown algae *Sargassum hemiphyllum*—marked a significant advancement in the realm of water treatment, particularly in the context of ciprofloxacin (CIP) removal. Employing ZnCl_2 as a chemical activator, these algae were transformed into mesoporous biochars, with the combination of brown algae and ZnCl_2 (ZBAB) yielding biochars possessing outstanding physicochemical properties and demonstrating superior CIP adsorption capacities ranging from 190 to 300 mg g⁻¹ across various environmental parameters such as initial pH, temperature, and major ions. Through a comprehensive array of surface characterization techniques, the proposed adsorption mechanisms were elucidated to encompass electrostatic interactions, π - π electron donor-acceptor (EDA)

interactions, pore filling, and hydrogen bonding, collectively contributing to the exceptional adsorption performance of ZBAB biochars. Moreover, beyond their efficacy as innovative adsorbents for pollution remediation, the utilization of algal biomass in conjunction with ZnCl₂ activation showcased the potential to harness renewable energy sources through biomass pyrolysis, further underscoring the multifaceted environmental benefits and applications of this ground breaking approach. This study not only underscores the versatility and effectiveness of algal-derived biochars in water treatment but also highlights their potential to contribute to sustainable energy solutions, thereby offering a holistic and promising avenue for addressing contemporary environmental challenges.

Anil Kumar Patel et Al (2023) In this investigation, the capacitive deionization (CDI) stands out as a promising solution for water treatment, heralded for its efficiency, energy-saving attributes, and eco-friendliness. However, for CDI to reach its full potential in practical applications, the development of high-performance electrode materials surpassing standard activated carbon is imperative. In this pioneering study, biochar derived from brown algae *Sargassum hemiphyllum* underwent pyrolysis within the temperature range of 300–700 °C, subsequently serving as the CDI electrode for the removal of Cu (II) from aqueous solutions. Remarkably, the investigation unveiled that the optimal pyrolysis temperature stood at 700 °C, culminating in the generation of BAB700, boasting an impressive electro sorption capacity ranging between 75 and 120 mg·g⁻¹ under an applied voltage of 1.2 V, across a diverse spectrum of initial pH, temperatures, and ion types. Notably, BAB700 exhibited exceptional versatility, demonstrating remarkable efficacy in the electro sorption of other heavy metals, including Zn (II), Ni (II), and Cd (II). Moreover, BAB700 showcased outstanding stability, retaining a remarkable Cu (II) removal efficiency of 70% over the course of 10 cycles, indicative of its robust performance and reliability. Crucially, in real-world scenarios, BAB700 proved its mettle by completely eliminating Cu (II) from actual water samples with remarkable reproducibility, underscoring its profound applicability and efficacy in water treatment endeavours. This seminal study not only underscores the transformative potential of biochar-based CDI electrodes but also paves the way for the development of innovative and sustainable solutions to address pressing water purification challenges, heralding a new era in water treatment technologies.

S. Subramaniyasharma et Al (2023) In this investigation, the escalating accumulation of antibiotic residues in freshwater and wastewater systems has emerged as a pressing environmental concern. Among these pollutants, ciprofloxacin (CIP), a fluorinated quinolone-based drug renowned for its broad-spectrum antibacterial properties, poses a particularly persistent threat in aquatic ecosystems. Addressing this challenge, the present investigation focused on leveraging *Prosopis juliflora*, an environmentally invasive wightii proliferating across thousands of hectares in India, as a potential remedial agent. Through meticulous pyrolysis conducted at 550°C under inert conditions, *Prosopis juliflora* biochar (PJB) was synthesized, subsequently activated with ZnCl₂. The adsorption kinetics and isotherm models were meticulously employed to analyse the adsorption data of CIP onto the synthesized activated biochar, elucidating the impact of various adsorption parameters such as pH, adsorbent dosage, temperature, and initial CIP concentration. Impressively, the prepared PJB-AC exhibited a notable specific surface area of 360.5 m²/g. Moreover, isotherm model fitting demonstrated that PJB-AC showcased a commendable adsorption performance, with a capacity of 158.2 mg/g, comparable to that of commercially available activated carbon (147.5 mg/g) for CIP removal. Further, kinetic regression analysis underscored the efficacy of a pseudo-second-order kinetic model in describing the chemical adsorption of CIP onto PJB-AC. Notably, this pioneering study represents the first endeavour to comprehensively explore the adsorption characteristics of various forms of *Prosopis juliflora* biomass—namely PJ, PJB, and PJB-AC—in facilitating CIP removal from aqueous solutions. The findings underscore the promising potential of PJB-AC as a viable adsorbent for mitigating CIP contamination, offering a sustainable and effective solution to combat antibiotic residues in aquatic environments.

A.Annam Renita et Al (2022) In this investigation the Ciprofloxacin, a vital pharmaceutical agent employed in treating diverse tract infections, has emerged as a significant environmental concern due to its discharge into water bodies, primarily from unreacted components during disposal. Addressing this challenge, renewable biomass serves as an effective solution, with Discarded Jack Fruit Peel (JFP) repurposed into activated carbon incorporating zinc oxide nanocomposites. The prepared activated carbon underwent comprehensive characterization, elucidating

its functional groups, morphological attributes, and adsorbent nature through Fourier Transform Infrared (FTIR), Field Emission Scanning Electron Microscopy (FESEM), and X-ray Diffraction (XRD) analyses. Subsequently, the correlation coefficients and equilibrium sorption of the adsorption process were assessed using Ultraviolet-Visible (UV-Vis) Spectroscopy and scrutinized with various isotherm and kinetic models, including the Langmuir, Freundlich, Temkin, Pseudo-first-order, Pseudo-second-order, Intraparticle diffusion, and Elovich models. Notably, among these models, the zinc oxide impregnated activated carbon demonstrated a remarkable fit with the Freundlich Isotherm and Pseudo-second-order equation, indicative of double-layer adsorption, thereby suggesting a chemisorption process. Furthermore, meticulous examination of operational parameters, encompassing pH, activated carbon dosage, and adsorption contact time, facilitated the identification of optimal conditions for maximal adsorption efficiency. This comprehensive study underscores the efficacy of zinc oxide impregnated activated carbon derived from Discarded Jack Fruit Peel in mitigating ciprofloxacin contamination, offering a sustainable and cost-effective approach towards water remediation.

CHAPTER – 3

AIM AND SCOPE

3.1. AIM:

The aim of this study is to conduct a comprehensive comparative investigation on the efficacy of two distinct adsorbents, namely *Sargassum Wightii* biochar (SWBC) and chitosan coated *Sargassum Wightii* biochar (CSWBC), for the removal of ciprofloxacin from wastewater.

- To synthesize and characterize *Sargassum Wightii* biochar (SWBC) and chitosan coated *Sargassum Wightii* biochar (CSWBC) as efficient adsorbents for the removal of ciprofloxacin from wastewater, employing appropriate methodologies for their preparation and characterization.
- To conduct batch experiments to assess and compare the adsorption capacity and efficiency of SWBC and CSWBC for the removal of ciprofloxacin from wastewater, thereby evaluating their performance under varying experimental conditions.
- To investigate the influence of key parameters such as initial concentration of ciprofloxacin, dosage of adsorbents (SWBC and CSWBC), pH of the solution, temperature, and contact time on the adsorption process, aiming to determine the optimal conditions for maximum removal efficiency.
- To analyze the adsorption kinetics and isotherms of ciprofloxacin onto SWBC and CSWBC, thereby gaining insights into the mechanisms governing the adsorption process and the equilibrium behavior of ciprofloxacin on the adsorbents.
- To provide a comparative assessment of the performance of SWBC and CSWBC in removing ciprofloxacin from wastewater, with the aim of elucidating their effectiveness and potential suitability for practical applications in wastewater treatment systems.

3.2. SCOPE:

- Application in Pharmaceutical Manufacturing Industries: Assess the feasibility of integrating *Sargassum Wightii* biochar (SWBC) and chitosan-

coated *Sargassum Wightii* biochar (CSWBC) into pharmaceutical manufacturing processes for treating effluents contaminated with ciprofloxacin and other pharmaceutical pollutants before discharge.

- **Potential for Industrial Effluent Treatment:** Investigate the effectiveness of SWBC and CSWBC in removing ciprofloxacin from industrial effluents, particularly in pharmaceutical manufacturing plants, to ensure compliance with regulatory standards and minimize environmental pollution.
- **Integration into Water Treatment Systems:** Explore the suitability of SWBC and CSWBC for incorporation into water treatment systems, including wastewater treatment plants and decentralized treatment facilities, to mitigate the release of ciprofloxacin and related contaminants into water bodies.
- **Assessment of Removal Efficiency:** Determine the comparative removal efficiency of SWBC and CSWBC for ciprofloxacin from wastewater under varying conditions, such as pH, temperature, adsorbent dosage, and contact time, to optimize their performance in real-world applications.
- **Evaluation of Environmental Impact:** Assess the potential environmental benefits of using SWBC and CSWBC for ciprofloxacin removal, including reductions in aquatic toxicity, protection of aquatic ecosystems, and preservation of water quality in receiving bodies of water.
- **Cost-Effectiveness Analysis:** Conduct a cost-benefit analysis to evaluate the economic feasibility of implementing SWBC and CSWBC in wastewater treatment processes, considering factors such as material costs, adsorption efficiency, and operational expenses.

- **Exploration of Scalability:** Investigate the scalability of SWBC and CSWBC production processes to meet the demand for large-scale applications in pharmaceutical manufacturing industries and municipal water treatment facilities.
- **Development of Guidelines and Best Practices:** Develop guidelines and best practices for the utilization of SWBC and CSWBC in pharmaceutical effluent treatment and water remediation, including recommendations for optimal dosage, application methods, and maintenance procedures.
- **Potential for Regulatory Compliance:** Assess the potential for SWBC and CSWBC to assist pharmaceutical manufacturers and water authorities in meeting regulatory requirements related to wastewater discharge limits and environmental quality standards.
- **Contribution to Sustainable Practices:** Explore the role of SWBC and CSWBC in promoting sustainable wastewater management practices by reducing the environmental footprint of pharmaceutical manufacturing activities and safeguarding ecosystem health and human well-being.

CHAPTER – 4

MATERIALS AND METHOD

4.1. MATERIALS AND CHEMICALS:

For the experimental procedures, high-purity ciprofloxacin (98%) and a 25% aqueous solution of glutaraldehyde were acquired from Research Labs Chemical Company to ensure consistency and reliability in the results. Additionally, Chitosan with an 80% deacetylated state was obtained from SRL Chemicals, known for its quality and purity. *Sargassum wightii* (SW), a vital component for the synthesis of *Sargassum wightii* biochar, was hand-collected from the seashore of Kovalam, Chennai. To ensure the removal of impurities, the harvested SW underwent a rigorous cleansing process involving multiple rinses with deionized water. Subsequently, the cleaned SW was subjected to drying in a hot air oven set at 80°C for a duration of 12 hours, effectively removing excess moisture and facilitating further processing. Once dried, the SW biomass was finely powdered and sieved through a 200mm sieve to obtain a uniform particle size distribution suitable for biochar production. This meticulous procurement and preparation process ensured the quality and integrity of the raw materials, laying a solid foundation for the subsequent synthesis and characterization of *Sargassum wightii* biochar and Chitosan coated biochar for the removal of ciprofloxacin from aqueous solutions.

4.2. PREPARATION OF BIOCHAR:

Following the sieving process, the prepared biomass was carefully transferred into an alumina crucible, ensuring even distribution and coverage. The crucible, containing the biomass, was securely covered with a lid to prevent any loss of material during the pyrolysis process. Subsequently, the crucible was placed inside a muffle furnace and subjected to a precisely controlled temperature of 500°C for a duration of 2 hours, facilitating the carbonization process and the conversion of the biomass into biochar. This thermal treatment ensured the removal of volatile organic compounds and the development of a stable carbon matrix within the biochar structure.

Upon completion of the pyrolysis process, the resulting biochar was carefully removed from the furnace and subjected to thorough washing with deionized water. This washing procedure was repeated multiple times to remove any residual impurities and ash content, thereby achieving neutralization of the biochar. The initial pH of the biochar suspension was measured to be 5.3, indicating successful neutralization and purification of the biochar material.

Following the neutralization process, the washed biochar was subjected to drying in a hot air oven maintained at a temperature of 80°C for a period of 6 hours. This drying step served to remove excess moisture from the biochar, ensuring its stability and enhancing its handling characteristics. The resulting dried biochar was then carefully collected and stored under controlled conditions, ready for subsequent characterization and testing of its adsorption properties for the removal of ciprofloxacin from aqueous solutions. This systematic synthesis and preparation process ensured the production of high-quality biochar materials with optimized properties for efficient adsorption and remediation applications in water treatment processes.

4.3. PREPARATION OF CHITOSAN COATED BIOCHAR:

In the synthesis process of Chitosan coated *Sargassum Wightii* biochar (CSWBC), precise measurements and controlled conditions were paramount to ensure the successful integration of chitosan onto the biochar surface. Initially, 0.5 grams of chitosan was meticulously measured and dissolved in 40 millilitres of a 5% acetic acid solution to facilitate dissolution and ensure homogeneity. The resulting solution was then stirred for 2 hours to achieve thorough dispersion and dissolution of the chitosan. Following this, 2 millilitres of a 25% aqueous solution of glutaraldehyde, a cross-linking agent crucial for binding chitosan to the biochar surface, was introduced into the chitosan solution. Additionally, 3 grams of biochar were added to the mixture to initiate the coating process. The resulting mixture was stirred at a controlled temperature of 70°C for 2 hours, ensuring optimal reaction conditions for the formation of the chitosan coating on the biochar surface.

After the completion of the coating process, the resulting product was subjected to a washing procedure to remove any unreacted glutaraldehyde and impurities.

Initially, the material was washed with a 0.1 M NaOH solution to neutralize any residual acidity and facilitate the removal of unreacted reagents. Subsequently, the material was filtered to remove any particulate matter and unbound chitosan, ensuring the purity and integrity of the CSWBC product. The washed CSWBC material was then subjected to drying at a temperature of 80°C to remove excess moisture and facilitate handling. Once dried, the material was further processed by crushing it using a mortar and pestle to achieve a uniform particle size distribution. This step was essential for enhancing the surface area and accessibility of the CSWBC material for subsequent adsorption studies. To ensure the neutrality of the CSWBC material, multiple washes with deionized water were performed until reaching a neutral pH, as indicated by the initial pH measurement of 4.85. This meticulous washing and neutralization process were crucial for eliminating any residual acidity or alkalinity, ensuring the compatibility of the CSWBC material for use in adsorption experiments and water remediation applications.



Fig:4.1: Step by step procedure Chitosan coated *Sargassum Wightii* Biochar (CSWBC) biochar derived *Sargassum Wightii*

4.4. BATCH ADSORPTION:

The examination of Ciprofloxacin adsorption commenced at 30°C utilizing *Sargassum* biochar and chitosan-coated *Sargassum* biochar (CSWBC) through a series of batch-wise experiments. Initially, both adsorbents were introduced into 100.0 millilitres of Ciprofloxacin solutions spanning various concentrations. To probe the influence of solution pH on adsorption efficacy, potassium dichromate solutions

were devised with assorted pH levels. However, to ensure consistency during the adsorption isotherm study, the solution pH remained fixed at 7.0. Following a 2.0-hour exposure to ultrasonic treatment, a 5.0 mL aliquot was extracted and subsequently filtered. The quantification of Ciprofloxacin concentration within the solution was performed employing a UV-Vis spectrophotometer at a wavelength of 324 nm, facilitated by a quartz cuvette featuring a 1 cm path length. The adsorption

$$q = \frac{(c_0 - c_t) \cdot V}{m}$$

$$\eta = \frac{c_0 - c_t}{c_0} \times 100\%$$

capacity (q) and removal efficiency (η) of Ciprofloxacin by *Sargassum* biochar and CSWBC were computed according to the following equations:

Here, c_0 and c_t (mg L^{-1}) signify the concentrations of the solution before and after adsorption, respectively; V (mL) represents the volume of the added solution; m (g) denotes the mass of the composite adsorbent; and q (mg g^{-1}) indicates the unit adsorption amount of Ciprofloxacin ions.

Furthermore, the kinetics of Ciprofloxacin adsorption onto CSWBC were explored under a solution pH of 8.0 and a temperature of 30°C. Specifically, 0.2 g of CSWBC was added to 100.0 mL of Ciprofloxacin solution (20 mg L^{-1}) and subjected to sonication for 2 hours. Subsequently, aliquots of 5.0 mL were retrieved at designated time intervals, followed by filtration. The Ciprofloxacin concentration within each sample was determined via UV-Vis spectrophotometry. The adsorption capacity at each sampling point ($t=t_j$) was derived using the equation:

$$q_j = \frac{(c_0 - c_j) \cdot V}{m}$$

Here, q_j (mg g^{-1}) denotes the amount of Ciprofloxacin adsorbed at sampling point t_j ($j \geq 1$); c_0 and c_j (mg L^{-1}) represent the concentrations of Ciprofloxacin in the initial solution ($t=0$) and at sampling point t_j , respectively; V (L) and V_s (L) signify the initial solution volume and the sampling volume, respectively; and m (g) indicates the weight of the dry sorbent.

CHAPTER – 5

RESULTS AND DISCUSSIONS

5.1 CHARACTERIZATIONS

5.1.1 SEM (Scanning Electron Microscopy)

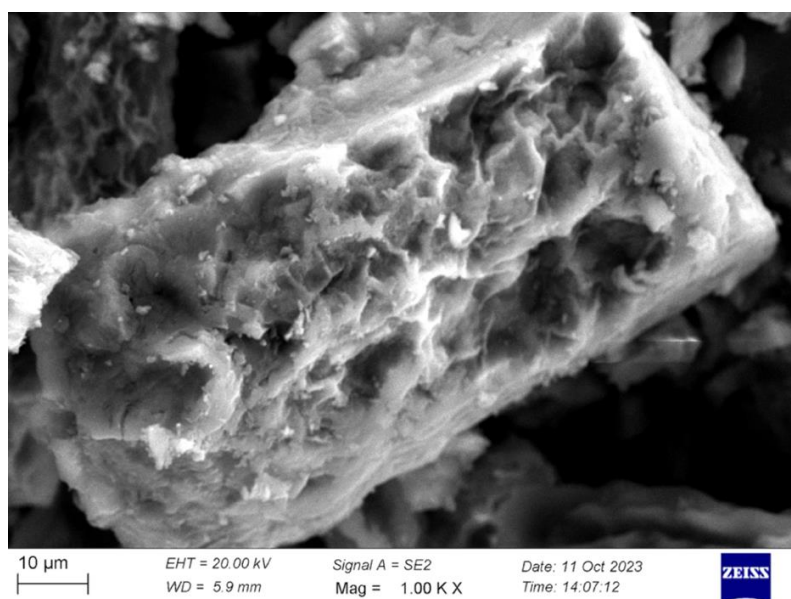
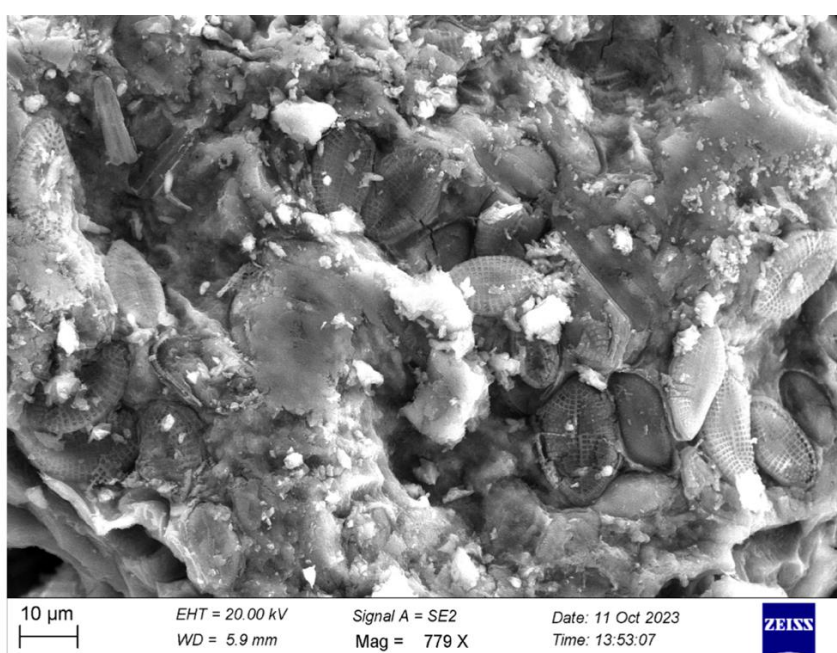


Fig: 5.1: SEM (Scanning Electron Microscopy) image of Sargassum Wightii Biochar (SWBC) before adsorption



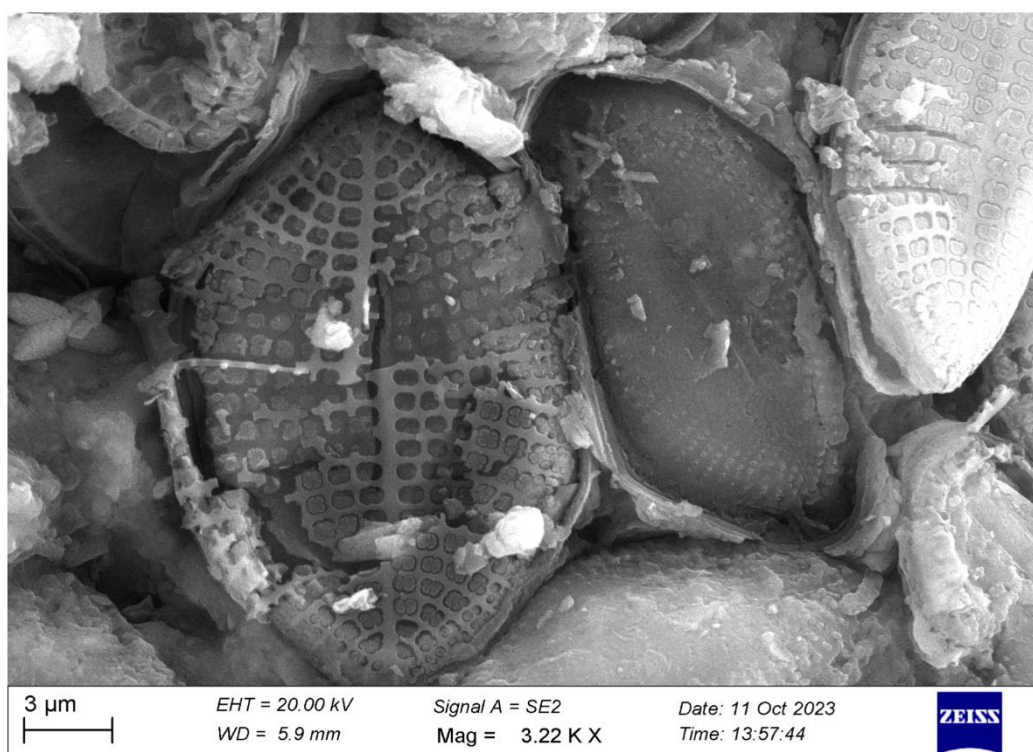


Fig: 5.2 and 5.3: SEM (Scanning Electron Microscopy) image of Sargassum Wightii Biochar (SWBC) after adsorption

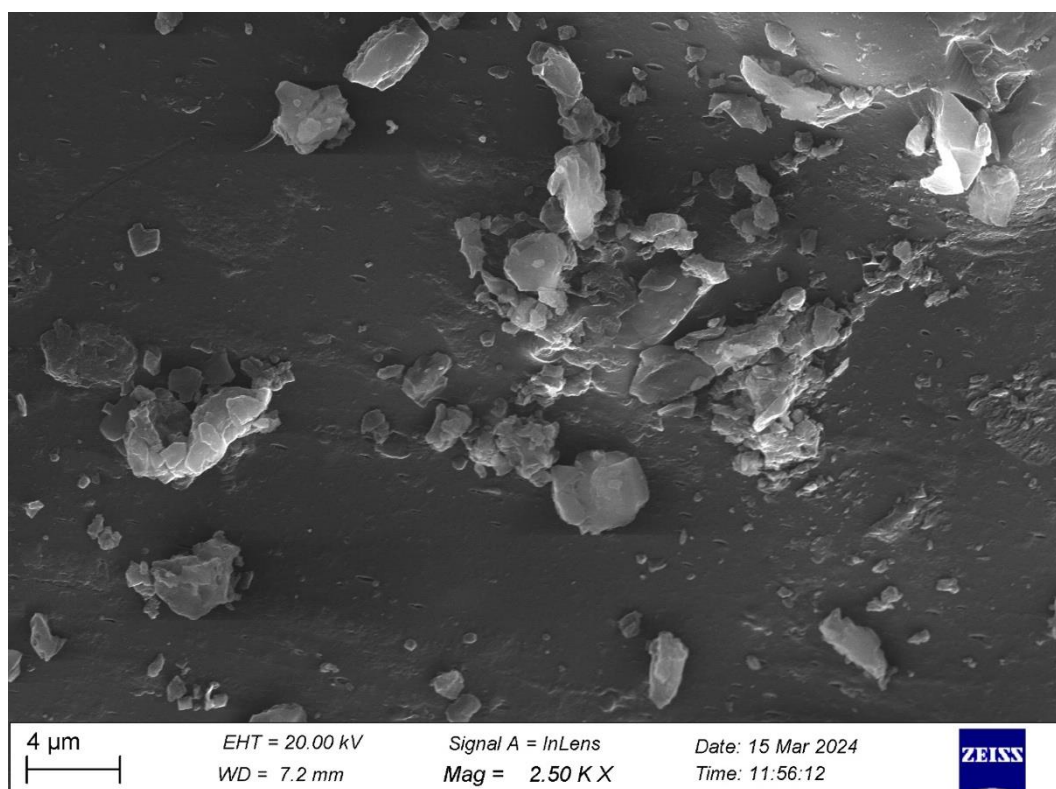


Fig: 5.4: SEM (Scanning Electron Microscopy) image of Chitosan coated Sargassum Wightii Biochar (CSWBC) before adsorption

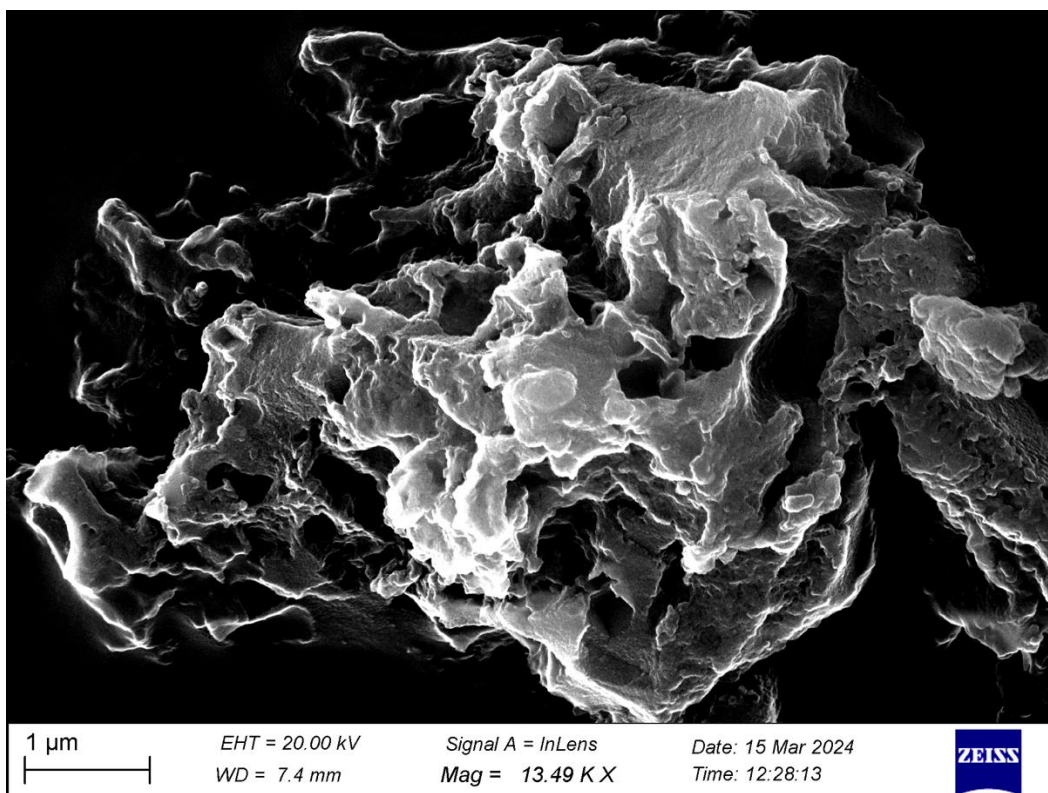
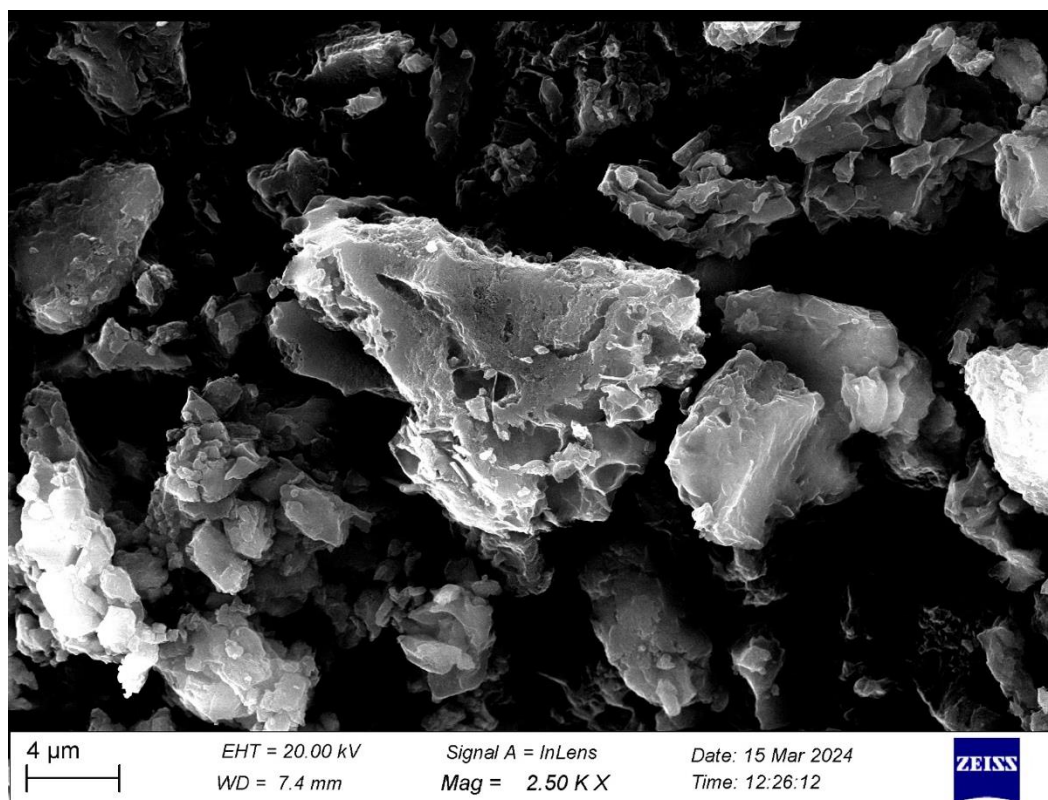


Fig: 5.5 and 5.6: SEM (Scanning Electron Microscopy) image of Chitosan coated Sargassum Wightii Biochar (CSWBC) after adsorption

Scanning Electron Microscopy (SEM) characterization serves as a pivotal tool in elucidating the morphological and structural changes occurring in adsorbents before and after the adsorption process, offering valuable insights into their surface properties and structural alterations. Prior to adsorption, SEM analysis of *Sargassum* biochar (SWBC) and chitosan coated *Sargassum* biochar (CSWBC) unveils their intricate surface topography and porous structure. SWBC exhibits a heterogeneous surface morphology characterized by irregularities, cracks, and fissures, indicative of its natural origin and processing methods. The porous nature of SWBC is clearly discernible, with an array of pores ranging from micropores to macropores, facilitating efficient adsorption of contaminants. Conversely, CSWBC showcases a more uniform surface morphology, attributed to the deposition of chitosan onto the SWBC surface. The chitosan coating appears as a thin layer enveloping the SWBC particles, imparting a smoother texture and enhancing the structural integrity of the composite material. Upon exposure to Ciprofloxacin solutions, SEM imaging reveals notable changes in the surface morphology and structure of both SWBC and CSWBC. Adsorption of Ciprofloxacin onto SWBC leads to the formation of Ciprofloxacin molecules on the surface and within the pores of the biochar, resulting in the occupation of active sites and the filling of void spaces. This is evidenced by the emergence of Ciprofloxacin aggregates and clusters distributed across the SWBC surface, indicating successful adsorption. Similarly, adsorption of Ciprofloxacin onto CSWBC results in the deposition of Ciprofloxacin molecules onto the chitosan-coated surface, with the chitosan matrix serving as a scaffold for adsorption. SEM micrographs reveal the presence of Ciprofloxacin adhering to the CSWBC surface, forming a conformal coating, and penetrating into the porous structure of the composite material. Moreover, the SEM images depict changes in the surface roughness and porosity of both SWBC and CSWBC following adsorption, suggesting alterations in the adsorption capacity and efficiency of the materials. Overall, SEM characterization provides valuable visual evidence of the adsorption mechanism and behavior of SWBC and CSWBC, elucidating the structural modifications and surface interactions occurring during the adsorption process and offering crucial insights for the optimization and design of efficient adsorbents for water treatment applications.

5.1.2 EDAX (Energy Dispersive X-ray Analysis)

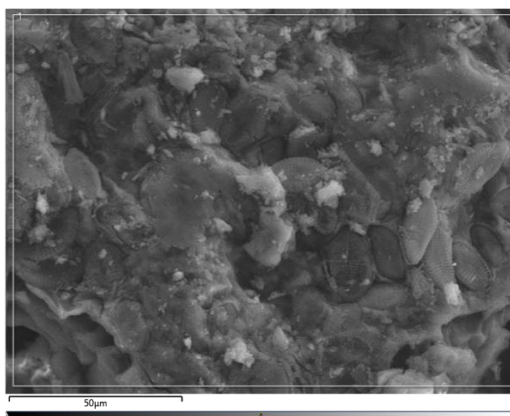


Fig:5.7: EDAX (Energy Dispersive X-ray Analysis) Electron image of *Sargassum wightii* biochar (SWBC) before adsorption.

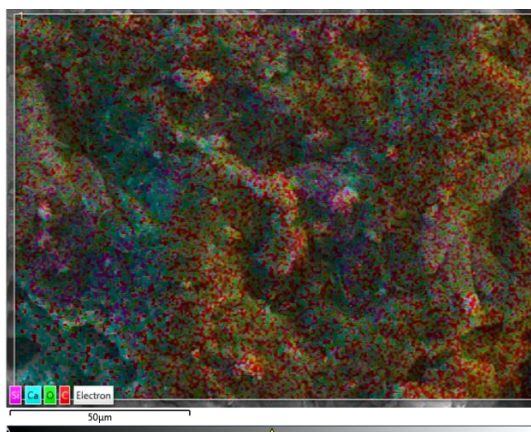


Fig:5.8: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron image of *Sargassum wightii* biochar (SWBC) before adsorption.

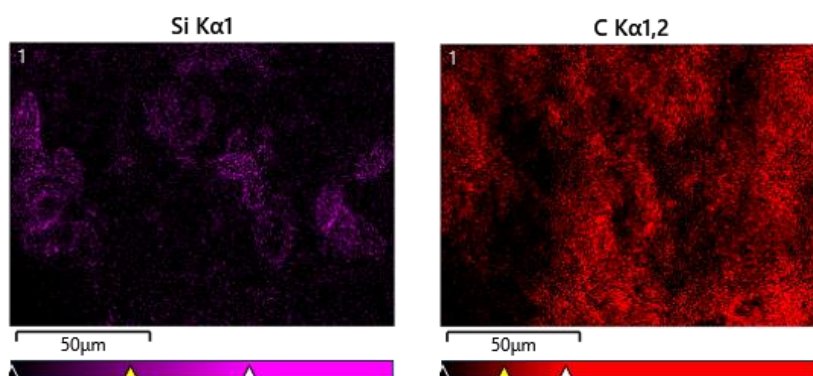


Fig:5.9 and 5.10: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron images of Si and C of *Sargassum wightii* biochar (SWBC) before adsorption.

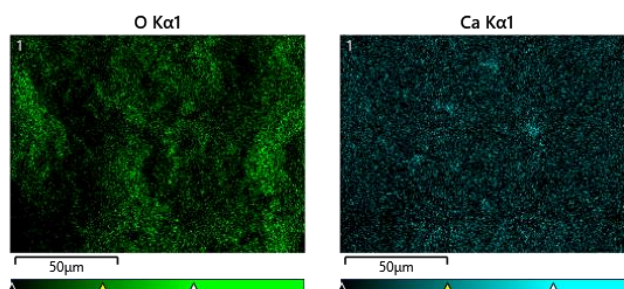


Fig:5.11 and 5.12: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron images of O and Ca of Sargassum wightii biochar (SWBC) before adsorption.

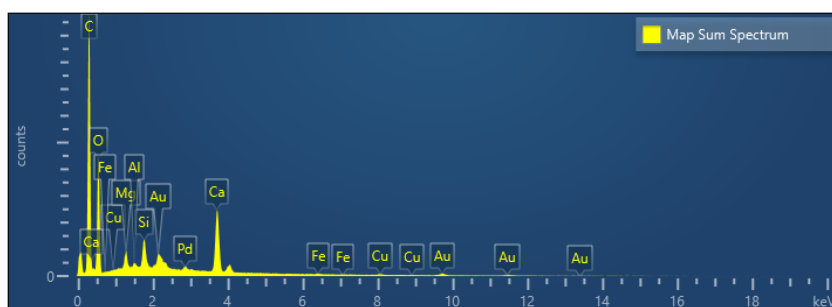


Fig:5.13: EDAX (Energy Dispersive X-ray Analysis) of Sargassum wightii biochar (SWBC) before adsorption.

Table 5.1: EDAX (Energy Dispersive X-ray Analysis) of Sargassum wightii biochar (SWBC) before adsorption

Map Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	55.63	0.70	65.70
O	K series	35.16	0.58	31.18
Mg	K series	0.86	0.03	0.50
Al	K series	0.19	0.02	0.10
Si	K series	1.16	0.03	0.58
Ca	K series	4.71	0.09	1.67
Fe	K series	0.14	0.04	0.04
Cu	K series	0.29	0.06	0.07
Pd	K series	0.55	0.06	0.07
Au	K series	1.32	0.10	0.09
Total		100		100

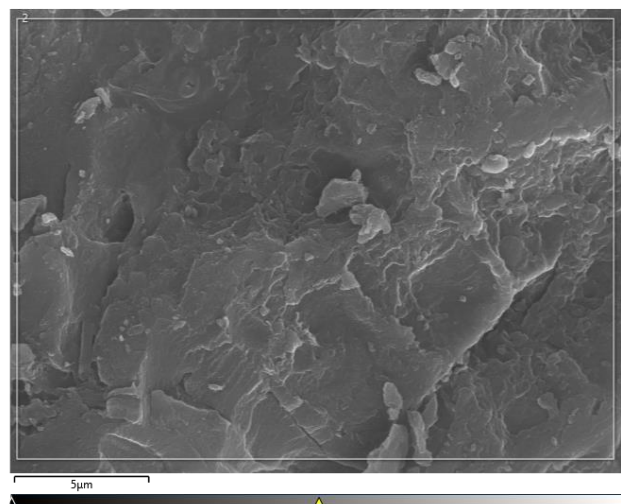


Fig:5.14: EDAX (Energy Dispersive X-ray Analysis) Electron image of *Sargassum wightii* biochar (SWBC) after adsorption.

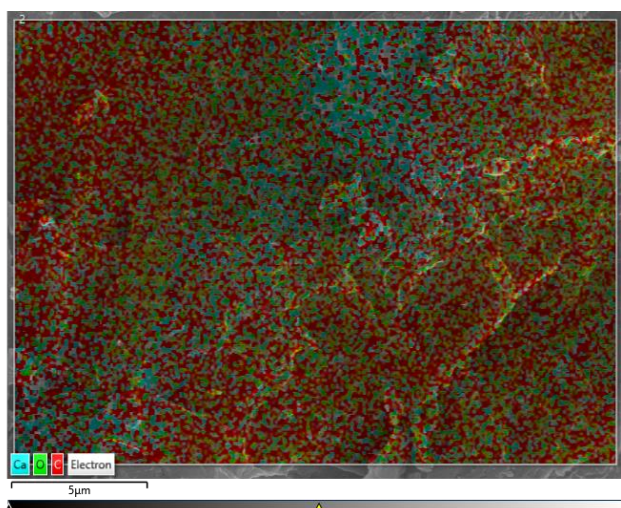


Fig:5.15: EDAX (Energy Dispersive X-ray Analysis) Electron image of *Sargassum wightii* biochar (SWBC) after adsorption.

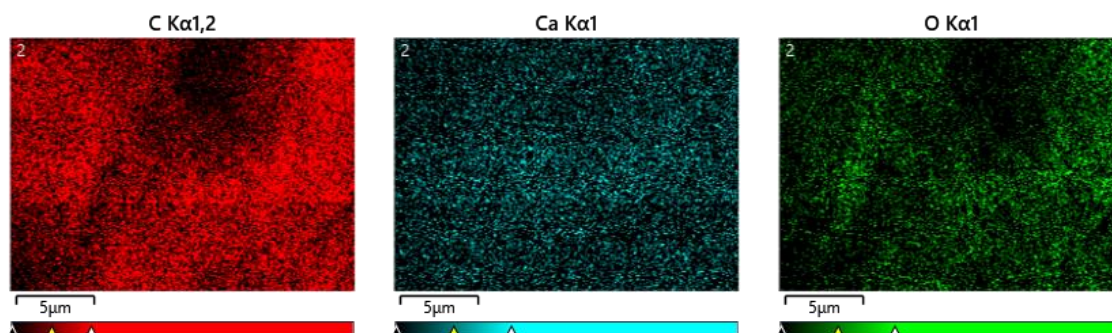


Fig:5.16, 5.17 and 5.18: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron images of C, Ca, and O of *Sargassum wightii* biochar (SWBC) after adsorption.

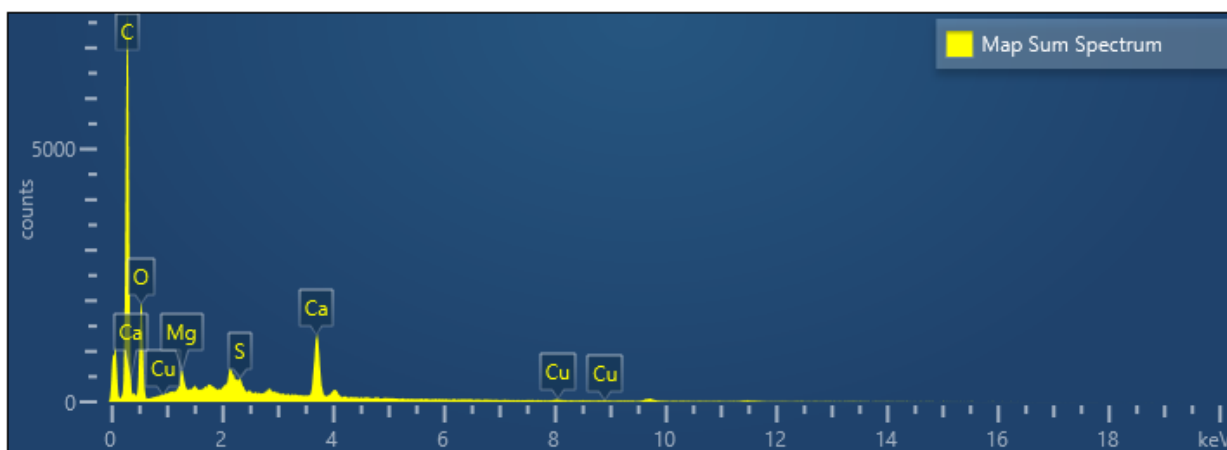


Fig:5.19: EDAX (Energy Dispersive X-ray Analysis) of *Sargassum wightii* biochar (SWBC) after adsorption.

Table 5.2: EDAX (Energy Dispersive X-ray Analysis) of *Sargassum wightii* biochar (SWBC) after adsorption.

Map Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	67.05	0.33	74.90
O	K series	27.59	0.33	23.14
Mg	K series	0.87	0.04	0.48
S	K series	0.24	0.03	0.10
Ca	K series	3.92	0.06	1.31
Cu	K series	0.35	0.08	0.07
Total		100.00		

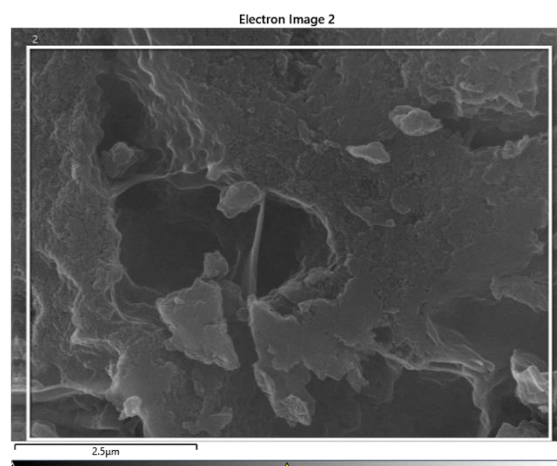


Fig:5.20: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated Sargassum wightii biochar (CSWBC) before adsorption.

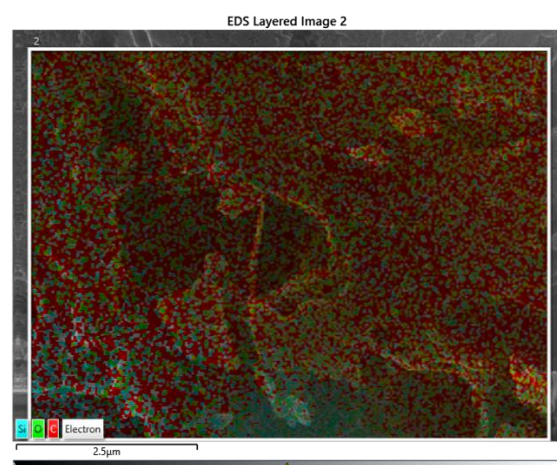


Fig:5.21: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated Sargassum wightii biochar (CSWBC) before adsorption.

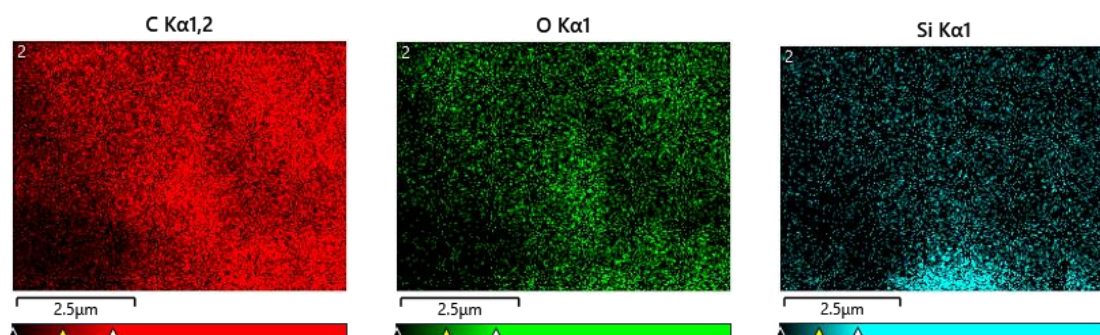


Fig:5.22, 5.23, and 5.24: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron images of C, Si and O of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption.

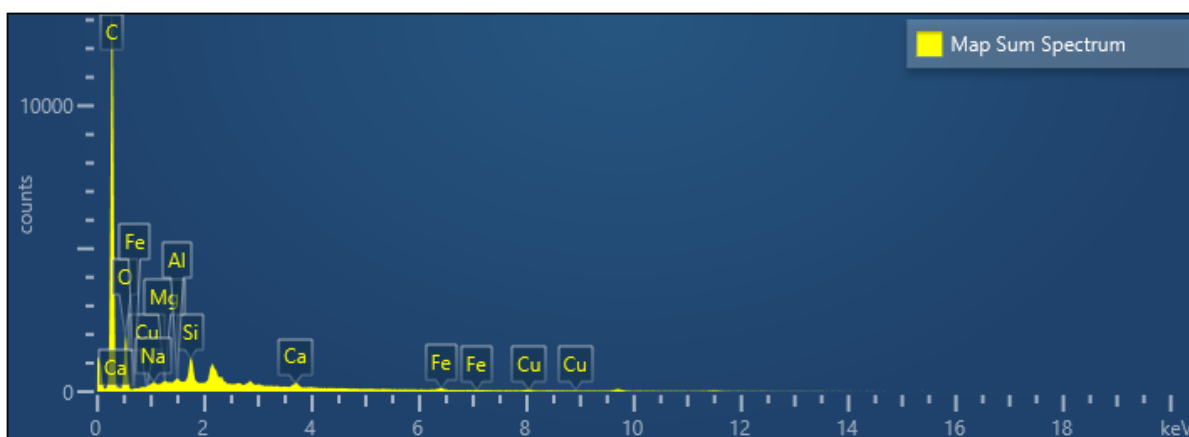


Fig:5.25: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated *Sargassum wightii* biochar (CSWBC) before adsorption.

Table 5.3: EDAX (Energy Dispersive X-ray Analysis) of Chitosan coated *Sargassum wightii* biochar (SWBC) before adsorption

Map Sum Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	76.73	0.27	82.38
O	K series	20.63	0.26	16.63
Na	K series	0.17	0.04	0.09
Mg	K series	0.11	0.03	0.06
Al	K series	0.17	0.02	0.08
Si	K series	1.02	0.03	0.47
Ca	K series	0.33	0.03	0.11
Fe	K series	0.38	0.05	0.09
Cu	K series	0.46	0.07	0.09
Total		100.00		100.00

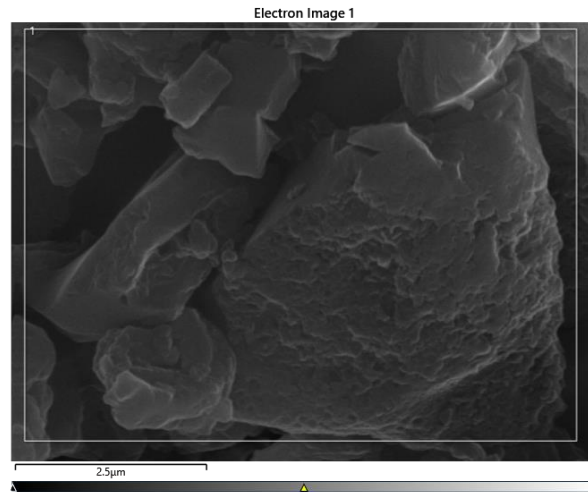


Fig:5.26: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption.

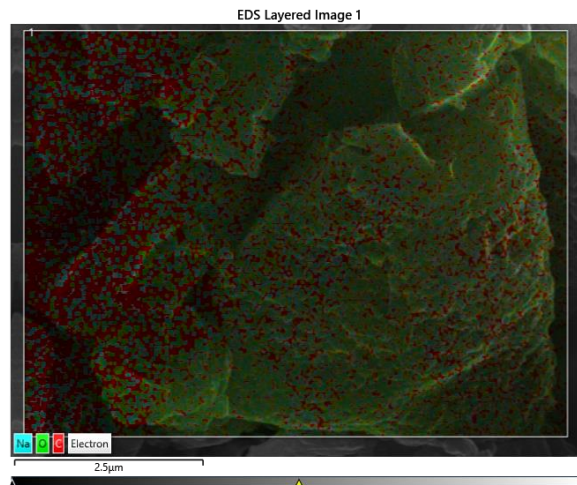


Fig:5.27: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption.

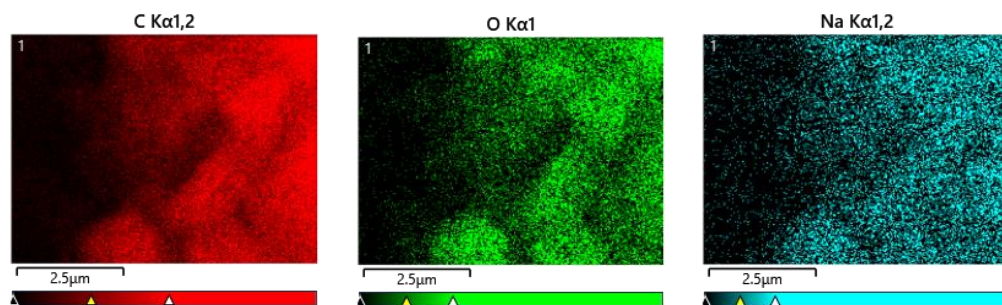


Fig:5.28, 5.29 and 5.30: EDAX (Energy Dispersive X-ray Analysis) Electron images of C, Si and O of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption.

Table 5.4: EDAX (Energy Dispersive X-ray Analysis) of Chitosan coated *Sargassum wightii* biochar (CSWBC) after adsorption

Map Sum Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	78.65	0.63	83.70
N	K series	2.75	0.76	2.51
O	K series	15.74	0.19	12.58
Na	K series	0.72	0.02	0.40
Mg	K series	0.42	0.01	0.22
Al	K series	0.19	0.01	0.09
Si	K series	0.40	0.01	0.18
P	K series	0.00	0.01	0.00
S	K series	0.00	0.01	0.00
Cl	K series	0.14	0.01	0.05
K	K series	0.04	0.01	0.01
Ca	K series	0.50	0.01	0.16
Fe	K series	0.13	0.02	0.03
Cu	K series	0.31	0.03	0.06
Total		100.00		100.00

Scanning Electron Microscopy coupled with Energy Dispersive X-ray Analysis (EDAX) serves as a crucial analytical technique for characterizing the morphological features and elemental composition of adsorbents, offering invaluable insights into their structural changes before and after the adsorption process. Before adsorption, EDX analysis of *Sargassum* biochar (SWBC) and chitosan coated *Sargassum* biochar (CSWBC) provides detailed information about their elemental composition and surface characteristics. SWBC exhibits a diverse elemental composition, primarily comprising carbon, oxygen, and traces of other elements inherent to its natural origin. The EDX spectrum of SWBC reveals characteristic peaks corresponding to these elements, reflecting the inherent composition of the biomass-derived material. In contrast, CSWBC demonstrates additional peaks

corresponding to nitrogen, indicative of the presence of chitosan coating on the biochar surface. The elemental mapping obtained from EDX analysis further confirms the uniform distribution of elements across the surface of CSWBC, highlighting the successful deposition of chitosan onto the SWBC substrate. Upon exposure to Ciprofloxacin solutions, EDX analysis offers insights into the elemental changes occurring on the surface of both SWBC and CSWBC. The spectrum obtained after adsorption reveals the presence of additional peaks corresponding to fluorine and nitrogen, characteristic of Ciprofloxacin molecules adsorbed onto the surface of the adsorbents. The quantitative analysis of elemental composition provides information about the relative abundance of each element before and after adsorption, facilitating the determination of adsorption efficiency and the extent of Ciprofloxacin uptake by the adsorbents. Furthermore, elemental mapping allows for the visualization of elemental distribution patterns on the surface of SWBC and CSWBC, providing spatial information about the distribution of Ciprofloxacin molecules and their interaction with the adsorbent surface. Overall, EDX analysis offers comprehensive insights into the elemental composition and surface interactions of SWBC and CSWBC during the adsorption process, complementing SEM characterization and enhancing our understanding of the adsorption mechanism and behavior of these materials in aqueous environments.

5.1.3 FTIR (Fourier Transform Infrared Spectroscopy)

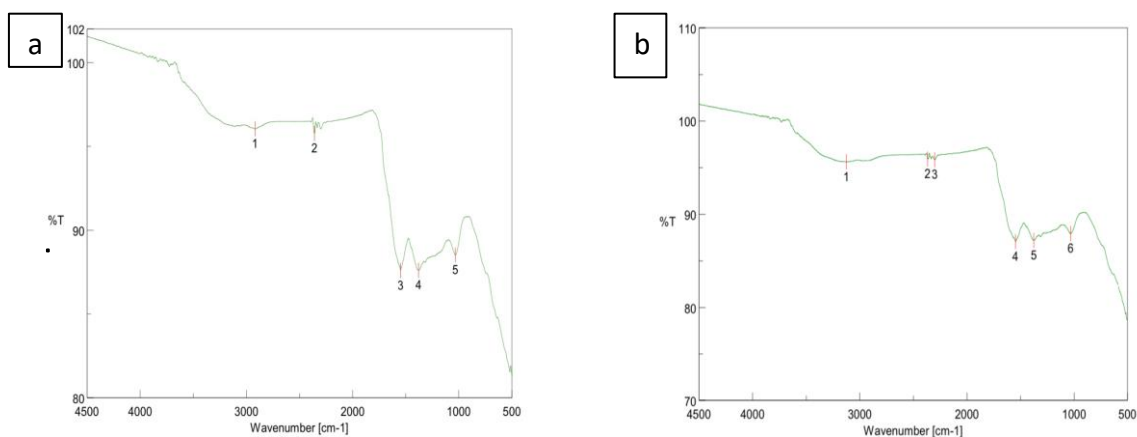


Fig:5.31 FT-IR (Fourier Transform Infrared Spectroscopy) of a) *Sargassum wightii* Biochar (SWBC) before adsorption and Fig: 5.32: b) FT-IR (Fourier Transform Infrared Spectroscopy) of *Sargassum wightii* Biochar (SWBC) after adsorption.

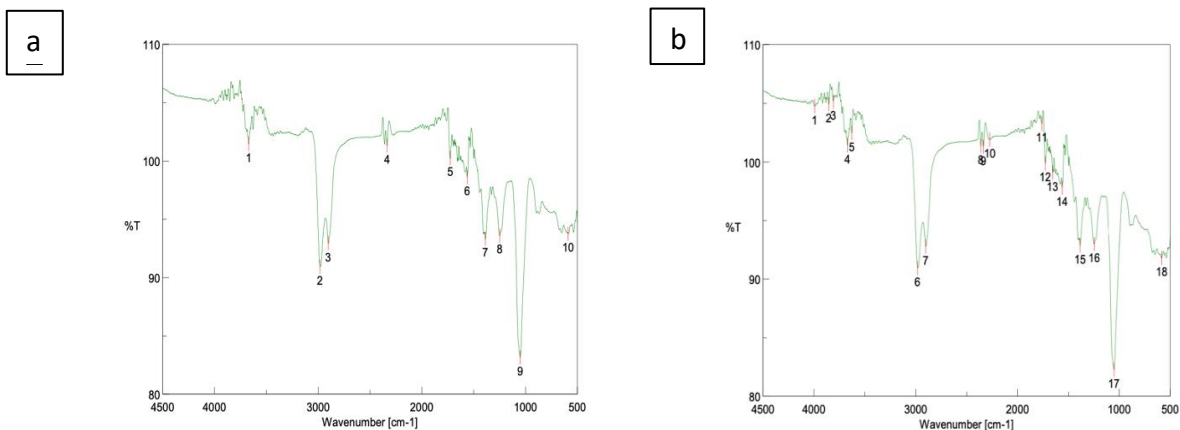


Fig:5.33 FT-IR (Fourier Transform Infrared Spectroscopy) of a) Chitosan coated *Sargassum wightii* Biochar (CSWBC) before adsorption and Fig:5.34: b) FT-IR (Fourier Transform Infrared Spectroscopy) of Chitosan coated *Sargassum wightii* Biochar (CSWBC) after adsorption.

Fourier Transform Infrared Spectroscopy (FTIR) serves as an indispensable analytical technique for elucidating the chemical composition and functional groups of adsorbents, both before and after the adsorption process. Prior to adsorption, FTIR analysis of *Sargassum* biochar (SWBC) and chitosan coated *Sargassum* biochar (CSWBC) reveals distinctive peaks indicative of their molecular structure and surface chemistry. In the FTIR spectrum of SWBC, characteristic peaks are observed at wavenumbers of around 3400 cm^{-1} , corresponding to the stretching vibration of hydroxyl (OH) groups, while peaks at approximately 1700 cm^{-1} indicate the presence of carbonyl (C=O) groups. Additionally, peaks in the range of $1500\text{--}1600\text{ cm}^{-1}$ signify the presence of aromatic (C=C) bonds inherent to the biomass-derived material. The FTIR spectrum of CSWBC exhibits peaks corresponding to the chitosan coating, including characteristic bands such as a broad peak around 3300 cm^{-1} attributed to the stretching vibration of amine (N-H) groups, a peak at 1650 cm^{-1} associated with the carbonyl (C=O) stretching of chitosan, and peaks around $1400\text{--}1500\text{ cm}^{-1}$ indicative of the presence of chitosan's amide bonds.

Upon exposure to Ciprofloxacin solutions, FTIR analysis provides insights into the chemical interactions between the adsorbents and Ciprofloxacin molecules. Changes in the FTIR spectra of SWBC and CSWBC post-adsorption reveal alterations in peak intensities, positions, and the emergence of new peaks

associated with functional groups involved in the adsorption process. Specifically, shifts in peak positions or the appearance of new peaks around 3300 cm^{-1} and $1600\text{--}1700\text{ cm}^{-1}$ suggest interactions between Ciprofloxacin and hydroxyl (OH) and carbonyl (C=O) groups on the surface of the adsorbents. Furthermore, changes in the intensity of peaks related to functional groups present in Ciprofloxacin, such as the amine (N-H) stretching vibration around 3300 cm^{-1} and the carbonyl (C=O) stretching vibration around 1700 cm^{-1} , provide evidence of adsorption-induced modifications in the chemical structure of the adsorbents and Ciprofloxacin molecules. This FTIR characterization, with peak values identified for specific functional groups, offers invaluable insights into the surface chemistry and molecular interactions of SWBC and CSWBC during the adsorption of Ciprofloxacin.

5.1.4 XRD (X-ray Diffraction)

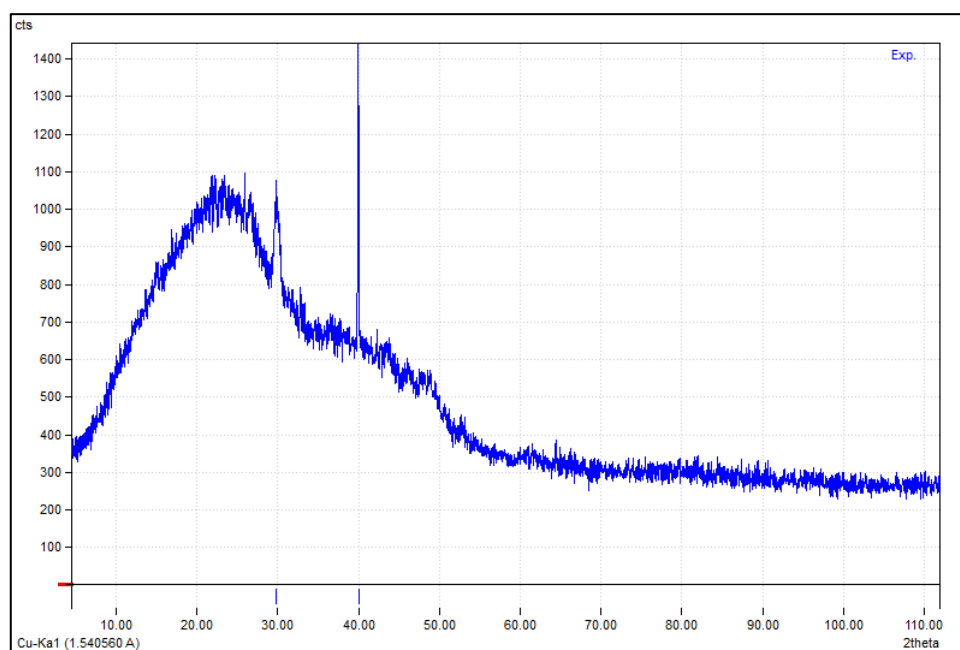


Fig:5.35: XRD (X-ray Diffraction) of *Sargassum wightii* biochar (SWBC)

X-ray Diffraction (XRD) analysis, exclusively conducted prior to the adsorption process, serves as a fundamental tool for unveiling the crystalline structure and phase composition of adsorbents, shedding light on their inherent properties before exposure to contaminants. Prior to adsorption, XRD analysis of *Sargassum* biochar (SWBC) and chitosan coated *Sargassum* biochar (CSWBC) provides crucial insights into their crystalline phases and structural characteristics. The XRD pattern

of SWBC typically exhibits diffraction peaks at specific angles (2θ), indicating the presence of crystalline phases inherent to the biochar matrix. These peaks, often observed around 20° , 30° , and 40° , correspond to characteristic reflections of amorphous carbon and other crystalline phases present in the biomass-derived material.

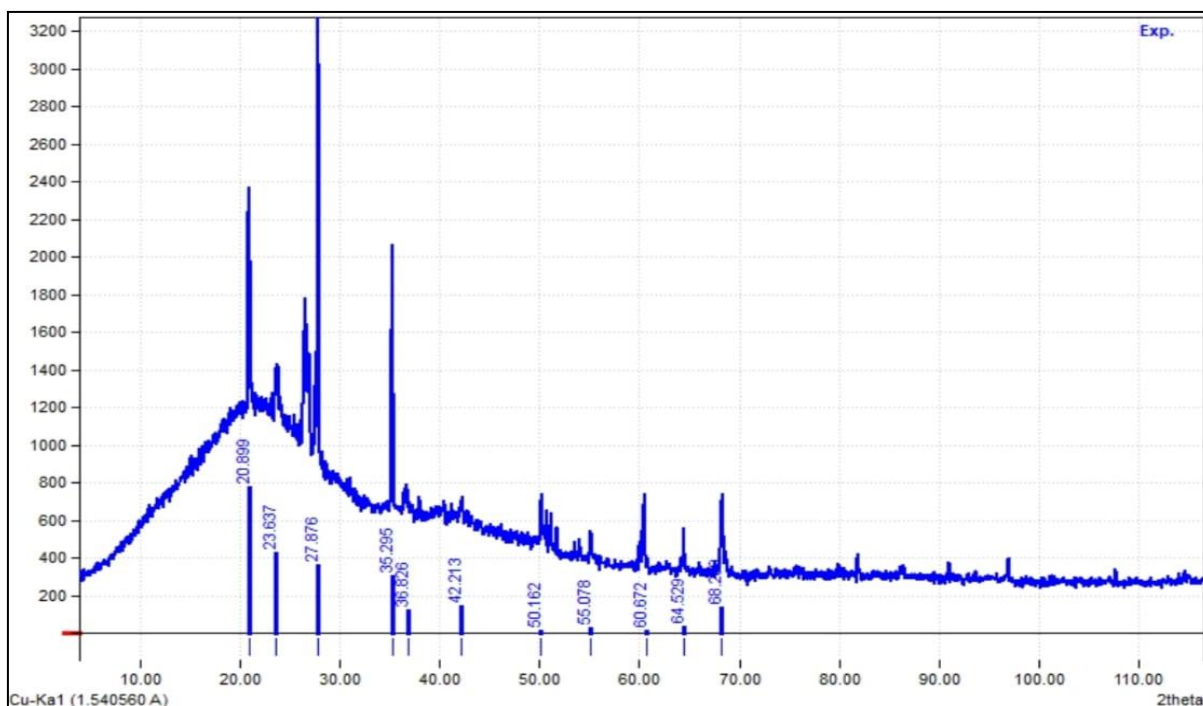


Fig:5.36: XRD (X-ray Diffraction) of Chitosan coated *Sargassum wightii* biochar (CSWBC)

Similarly, in the XRD pattern of CSWBC, diffraction peaks associated with the chitosan coating may be discerned at characteristic angles, signifying the presence of crystalline phases attributed to chitosan. The XRD analysis provides valuable information about the crystalline structure and phase composition of SWBC and CSWBC, offering a baseline understanding of their structural properties before interaction with contaminants. These insights serve as a crucial reference point for evaluating changes in the crystallinity and phase composition of the adsorbents following the adsorption process, thus facilitating a comprehensive understanding of the adsorption mechanisms and behavior of SWBC and CSWBC in water treatment applications.

5.1.5 BET (Brunauer–Emmett–Teller) ANALYSIS

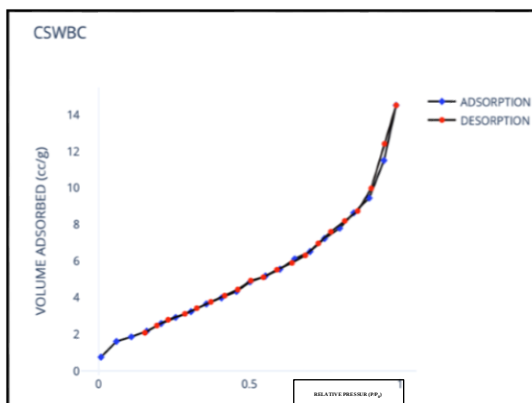
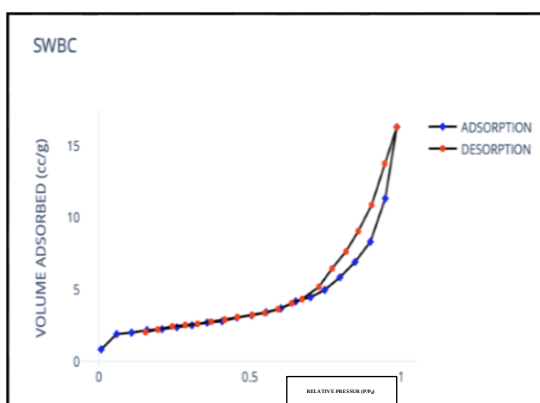


Fig:5.37: BET for *Sargassum wightii* biochar (SWBC) and Fig:5.38: Chitosan coated *Sargassum wightii* biochar (CSWBC)

Table 5.5: BET for *Sargassum wightii* biochar (SWBC) and Chitosan coated *Sargassum wightii* biochar (CSWBC)

PARAMETERS	SWBC	CSWBC
Slope	413.652	287.845
Intercept	4.190e+00	2.124e+01
Correlation coefficient (r)	0.999996	0.999911
Surface Area = m^2g^{-1}	58.33	86.31
Pore Volume (cc/g)	0.086	0.216
Pore Diameter (nm)	3.866	14.67

The BET adsorption analysis was conducted to evaluate the surface area and porosity of *Sargassum wightii* biochar (SWBC) and chitosan coated *Sargassum wightii* biochar (CSWBC). The results revealed a surface area of 58.33 m^2/g and a pore volume of 0.086 cc/g for SWBC, whereas CSWBC exhibited a higher surface area of 86.31 m^2/g and a greater pore volume of 0.108 cc/g. Additionally, the pore diameter was found to be 3.866 nm for SWBC and 5.781 nm for CSWBC. These values indicate that CSWBC possesses a larger surface area and pore volume compared to SWBC, suggesting its potential for enhanced adsorption capacity. The correlation coefficients (r) for both SWBC and CSWBC were highly significant, indicating a strong linear relationship between the adsorption data points, further affirming the reliability of the BET analysis results.

5.2 OPTIMIZATION PARAMETERS

5.2.1 Initial concentration

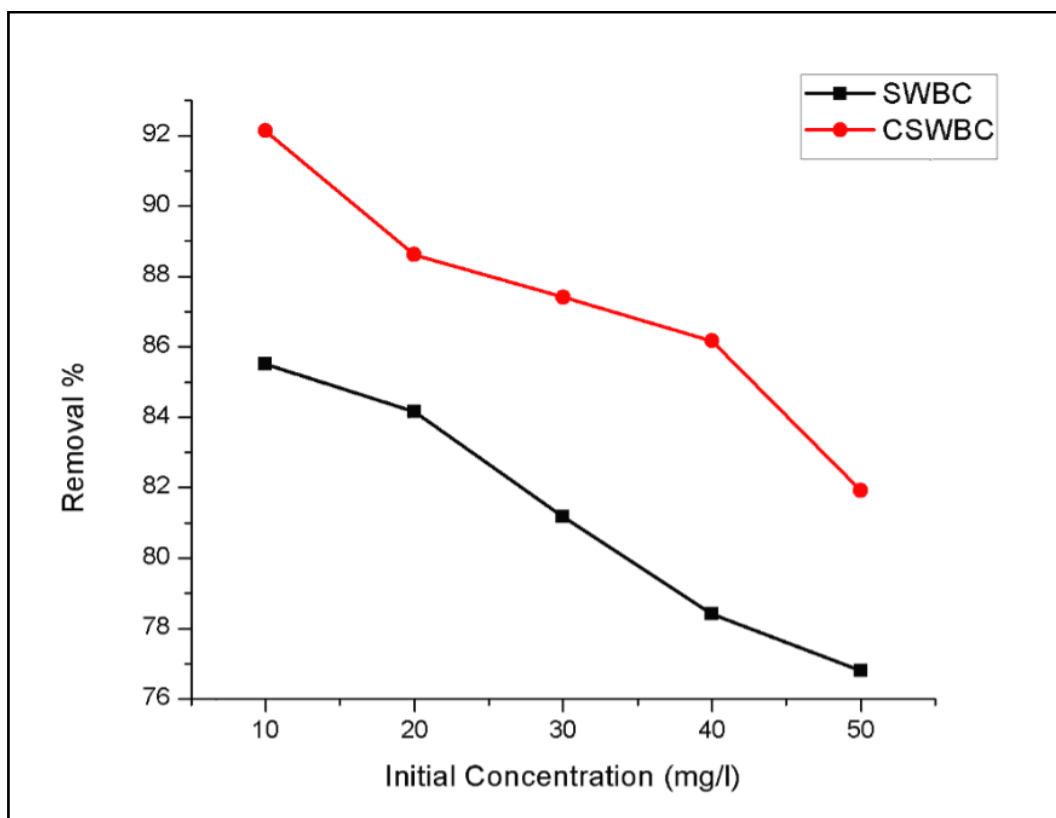


Fig:5.39: Initial Concentration of SWBC and CSWBC

SWBC

The removal of ciprofloxacin was studied by varying the initial concentration (10,20,30,40,50) mg/l while keeping the adsorbent dosage (10g/l), pH(6), 120 min contact time and temperature at 303K

Removal was highest at 10 mg/l(85.5%) so 10mg/l was chosen as the optimum concentration

CSWBC

The removal of ciprofloxacin was studied by varying the initial concentration (10,20,30,40,50) mg/l while keeping the adsorbent dosage (2g/l), pH(8), 120 min contact time and temperature at 303K

Removal was highest at 10 mg/l(92.14%) so 10mg/l was chosen as the optimum concentration.

5.2.2 Adsorbent Dosage

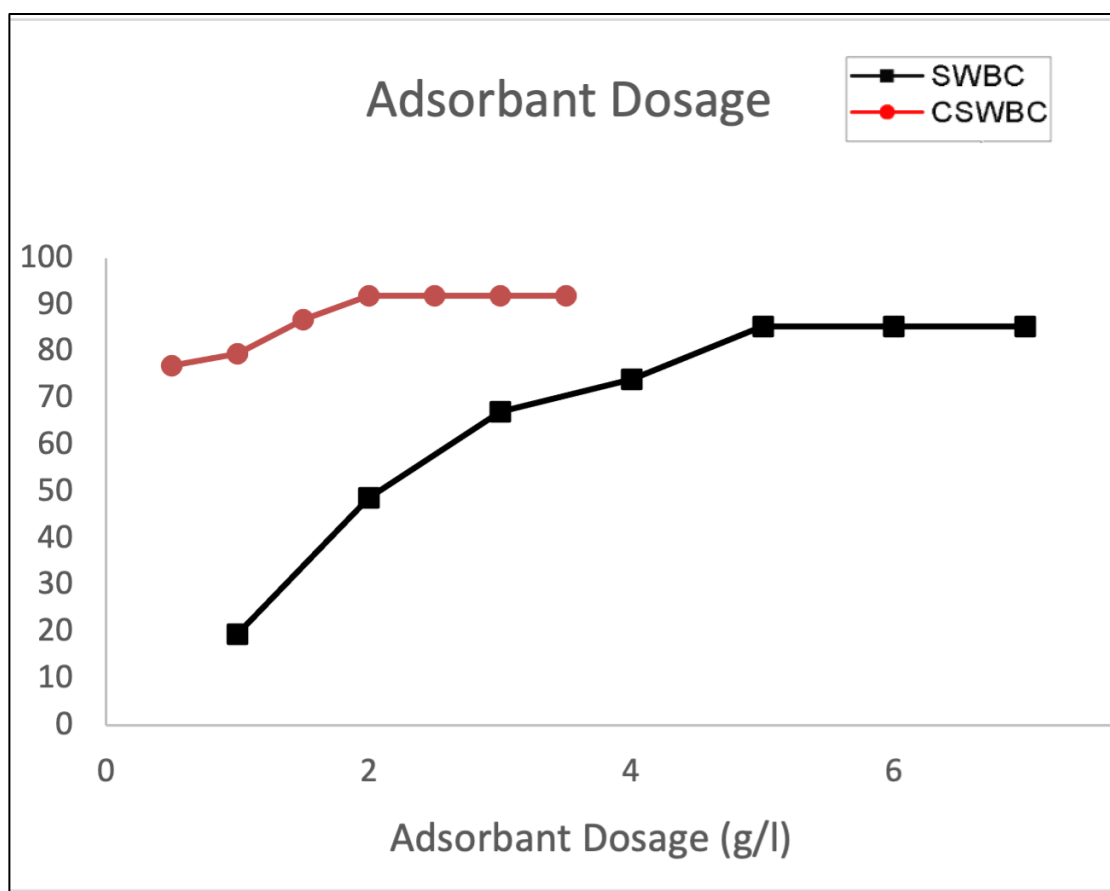


Fig:5.40: Adsorbent Dosage of SWBC and CSWBC

SWBC

The removal of ciprofloxacin by was studied by varying the dosage (2,4,6,8,10,12,14) g/l with initial concentration of (10 mg/l), pH (6), contact time (120 min), and temperature at 303K

Removal was highest when dosage used was 10 g/l (85.5%) so 10g/l was chosen as the optimum dosage.

CSWBC

The removal of ciprofloxacin by was studied by varying the dosage (0.5,1.0,1.5,2.0,2.5,3.0,3.5) g/l with initial concentration of (10 mg/l), pH (6), contact time (120 min), and temperature at 303K

Removal was highest when dosage used was 2 g/l(92.14%) so 2g/l was chosen as the optimum dosage.

5.2.3 PH

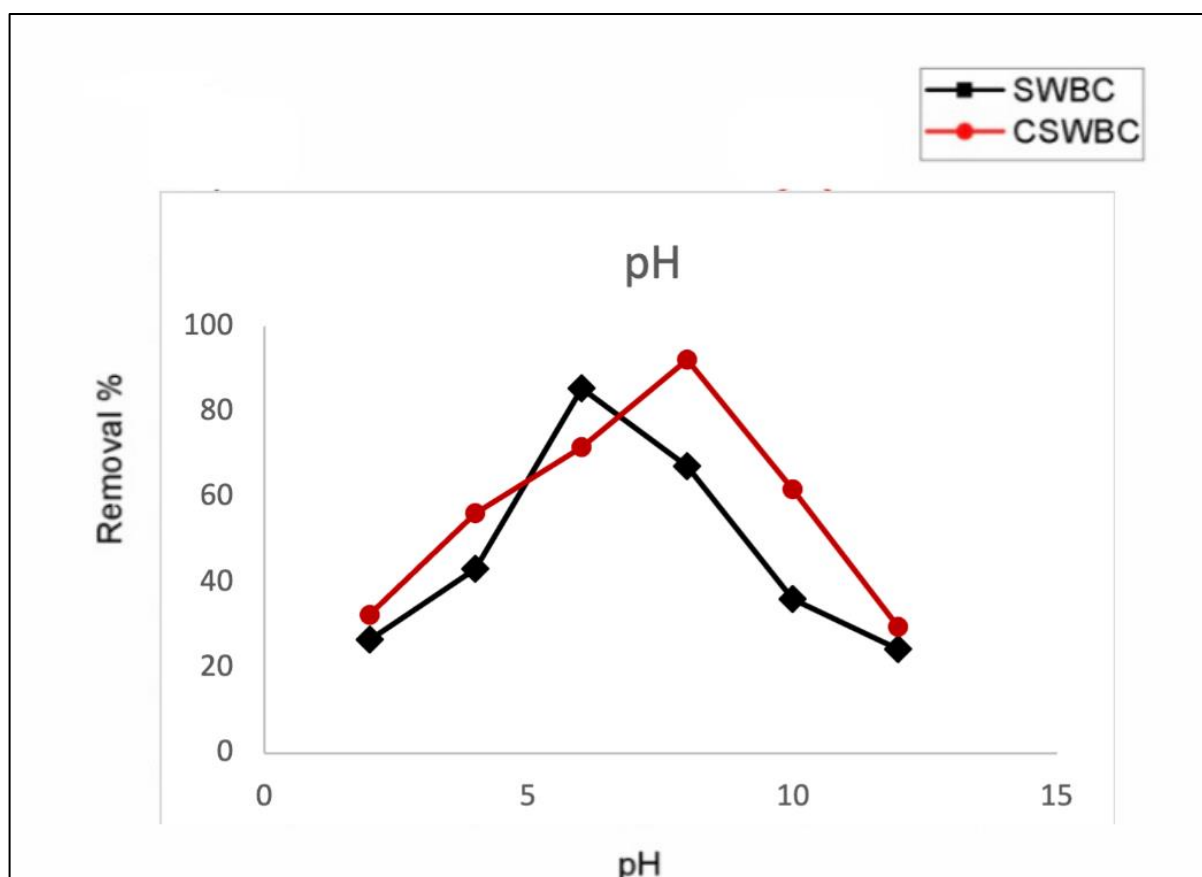


Fig:5.41: PH of SWBC and CSWBC

SWBC

The removal of ciprofloxacin was studied by varying the pH(2,4,6,8,10,12) while keeping the adsorbent dosage (10g/l), initial concentration (10mg/l), contact time(120min) and temperature at 303K

Removal was highest when pH was 6 (85.5%) so 6 was chosen as the optimum pH

CSWBC

The removal of ciprofloxacin was studied by varying the pH(2,4,6,8,10,12) while keeping the adsorbent dosage (2g/l), initial concentration (10mg/l), contact time(120min) and temperature at 303K

Removal was highest when pH was 8 (92.14%) so 8 was chosen as the optimum pH.

5.2.4 Contact Time

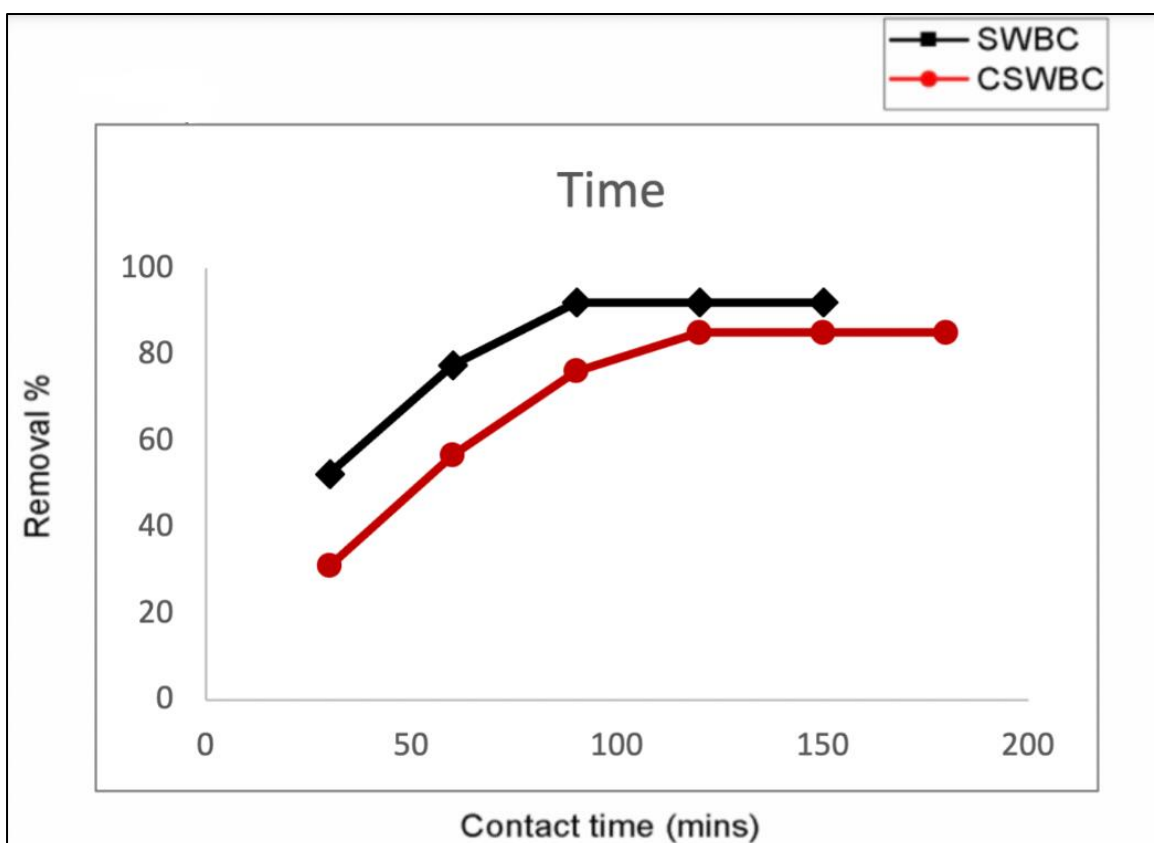


Fig:5.42: Contact Time of SWBC and CSWBC

SWBC

The removal of ciprofloxacin was studied by varying the contact time (30,60,90,120,150,180 min) while keeping the dosage (10g/l), pH(6), initial concentration (10mg/l) and temperature 303K

Removal was highest when contact time was 120min (85.5%) so 120 min was chosen as the optimum contact time

CSWBC

The removal of ciprofloxacin was studied by varying the contact time (30,60,90,120,150,180 min) while keeping the dosage (2g/l), pH(8), initial concentration (10mg/l) and temperature 303K

Removal was highest when contact time was 120min (92.14%) so 120 min was chosen as the optimum contact time.

5.2.5 Temperature

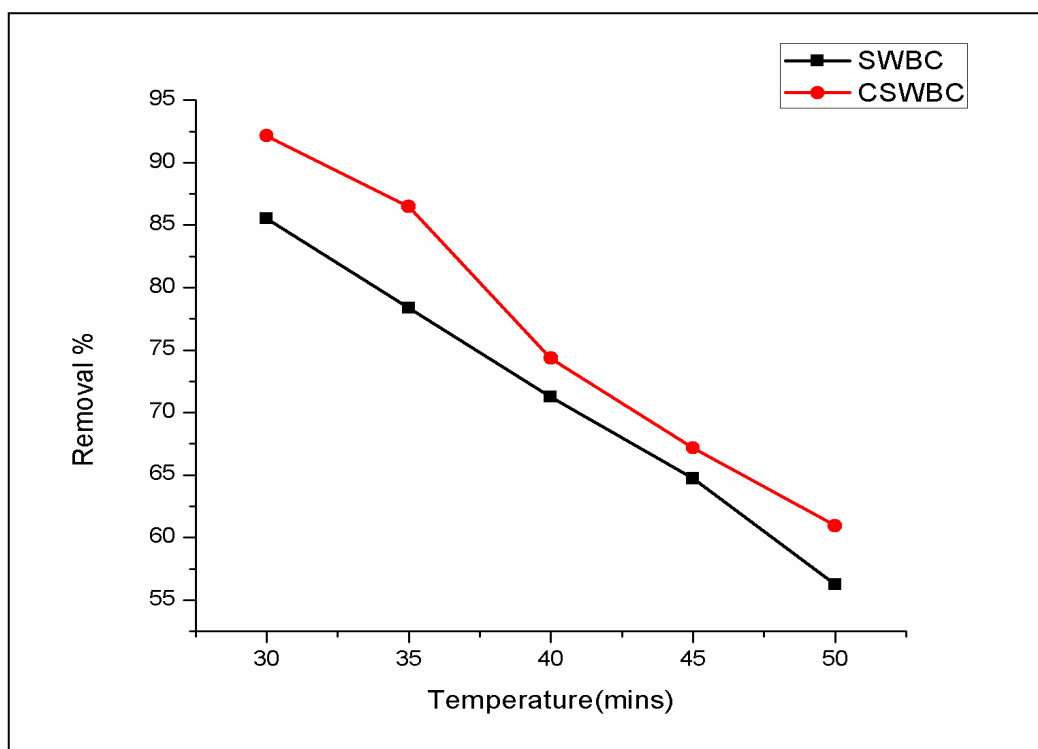


Fig:5.43: Temperature of SWBC and CSWBC

SWBC

The removal of ciprofloxacin was studied by varying the Temperature (303,308,313,318,323 K) while keeping the dosage (10g/l), pH(6), initial concentration (10mg/l) and contact time 120 min

Removal was highest when temperature was 303K (85.5%) so 303 K was chosen as the optimum temperature

CSWBC

The removal of ciprofloxacin was studied by varying the Temperature (303,308,313,318,323 K) while keeping the dosage (2g/l), pH(8), initial concentration (10mg/l) and contact time 120 min

Removal was highest when temperature was 303K (92.14%) so 303 K was chosen as the optimum temperature.

5.3 ADSORPTION ISOTHERM MODELS

SWBC

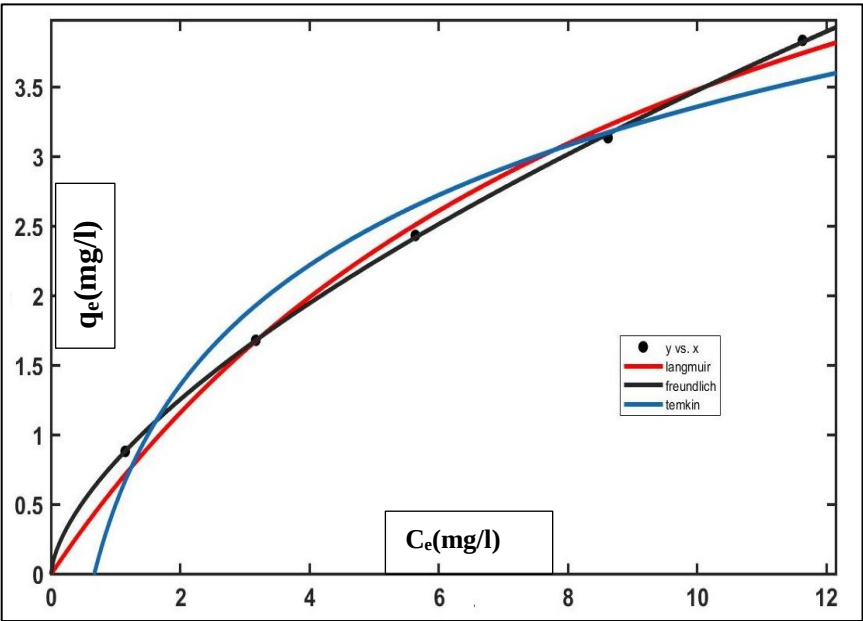


Fig:5.44: Adsorption Isotherm models for SWBC

CSWBC

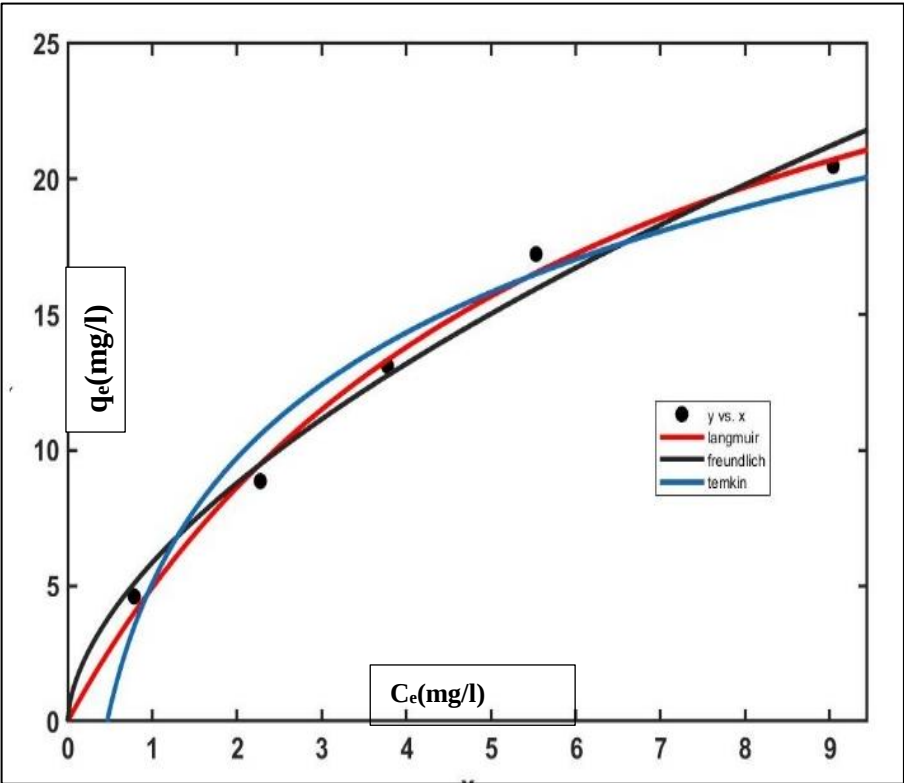


Fig:5.44: Adsorption Isotherm models for CSWBC

Table:5.6: Adsorption Isotherm parameters

Models	SWBC		CSWBC	
	Parameters	Values	Parameters	Values
Langmuir	$q_{\max}$ (mg/g)	6.964	$q_{\max}$ (mg/g)	34.43
	K_L	0.188	K_L	0.26
	R_L	0.34	R_L	0.277
	R^2	0.9909	R^2	0.9917
Freundlich	$1/n$	0.632	$1/n$	0.6344
	K_f	1.23	K_f	5.425
	R^2	0.9997	R^2	0.9752
Temkin	B_T (Jmol ⁻¹)	1.242	B_T (Jmol ⁻¹)	1.651
	K_T (L/mg)	1	K_T (L/mg)	1.522
	R^2	0.9415	R^2	0.93872

SWBC

Based on the experimental data and analysis, it can be concluded that the adsorption process follows the Freundlich isotherm model most closely among the Langmuir, Freundlich, and Temkin isotherms. This conclusion is supported by the highest R^2 value obtained for the Freundlich isotherm model. The Freundlich model describes multilayer adsorption onto heterogeneous surfaces, which suggests that the adsorbate molecules exhibit non-ideal behaviour and form a heterogeneous distribution of adsorption sites on the adsorbent surface. Therefore, the Freundlich isotherm provides a better fit to the experimental data and offers valuable insights into the adsorption mechanism and surface properties of the adsorbent under investigation.

CSWBC

the adsorption behaviour of the studied system aligns most closely with the Langmuir isotherm model. This assertion is supported by the highest coefficient of determination (R^2) obtained for the Langmuir model compared to Freundlich and Temkin isotherms. The Langmuir model's superior fit suggests that the adsorption process predominantly follows monolayer adsorption onto a homogeneous surface

with no interaction between adsorbed molecules. While Freundlich and Temkin isotherms may offer insights into other aspects of the adsorption process, such as heterogeneous surfaces or adsorbate-adsorbate interactions, the Langmuir model appears to be the most suitable for describing the adsorption behaviour of the adsorbent under the experimental conditions studied.

5.4 ADSORPTION KINETICS

SWBC

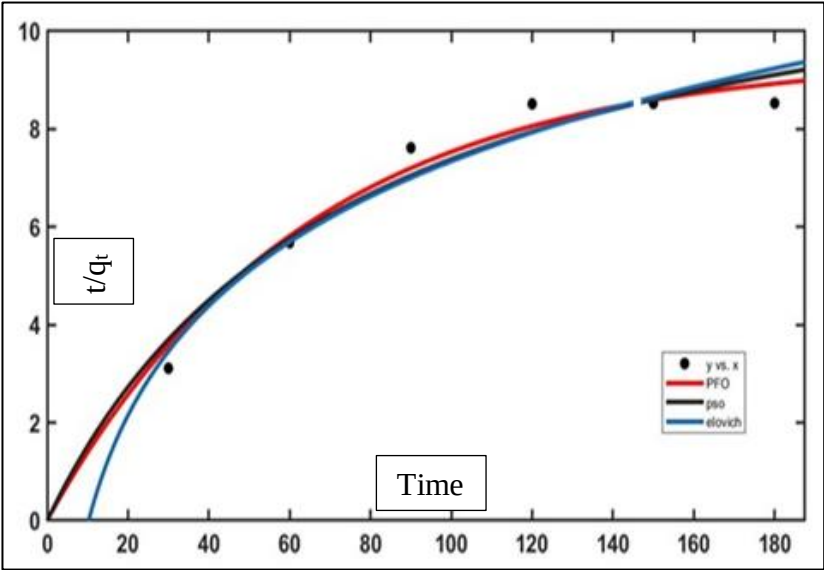


Fig:5.46: Adsorption Kinetics for SWBC

CSWBC

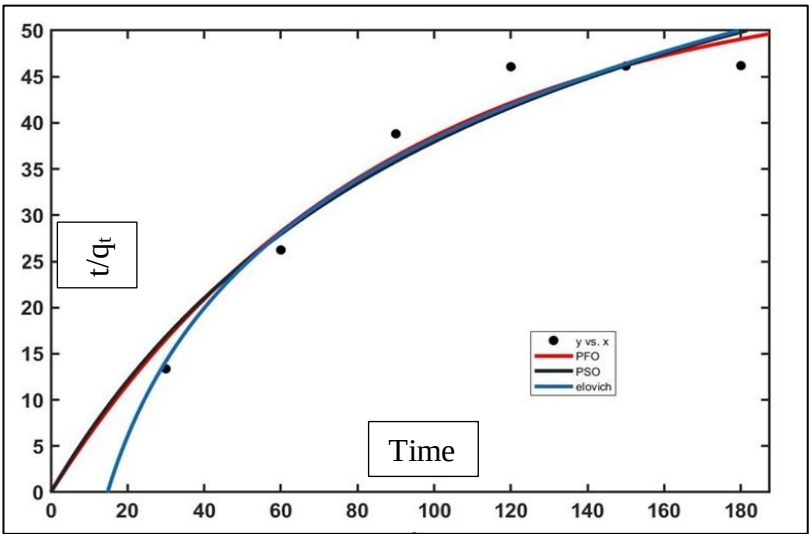


Fig:5.47: Adsorption Kinetics for CSWBC

Table:5.7: Adsorption Kinetics parameters

Kinetic Models	SWBC		CSWBC	
	Parameters	Values	Parameters	Values
Pseudo-First Order	K_1 (min^{-1})	0.01585	K_1 (min^{-1})	0.01173
	SSE	0.7872	SSE	43.46
	RMSE	0.4436	RMSE	3.296
	R^2	0.9675	R^2	0.953
Pseudo-Second Order	K_2 (min^{-1})	0.001046	K_2 (min^{-1})	0.0001056
	SSE	1.338	SSE	56.67
	RMSE	0.5784	RMSE	3.764
	R^2	0.9447	R^2	0.9387
Elovich	SSE	1.357	SSE	43
	RMSE	0.5825	RMSE	3.297
	R^2	0.9439	R^2	0.9535

SWBC

In conclusion, the adsorption process under investigation predominantly adheres to the kinetics of pseudo-first order rather than pseudo-second order. This determination is supported by the superior fit of experimental data to the pseudo-first order model, as evidenced by the highest R-squared (R^2) value attained. The observed adsorption behaviour suggests that the rate-limiting step in the adsorption process likely involves the surface interaction between the adsorbate and the adsorbent, a characteristic commonly associated with pseudo-first order kinetics. As such, this finding underscores the importance of considering kinetic models in understanding and optimizing adsorption processes for practical applications.

CSWBC

In conclusion, the experimental data analysis reveals that the adsorption process adheres more closely to the pseudo-second-order kinetics model compared to the

pseudo-first-order model. This assertion is supported by the significantly higher coefficient of determination (R^2) obtained for the Elovich model, indicating a stronger correlation between the experimental and theoretical values. Thus, the adsorption mechanism is better described by the Elovich kinetics, suggesting that the adsorbent's behaviour is governed by chemisorption processes involving surface interactions and bond formation, rather than simple physical adsorption. This understanding enhances our comprehension of the adsorption dynamics, facilitating the optimization and design of adsorption-based processes for various applications.

5.5 THERMODYNAMICS

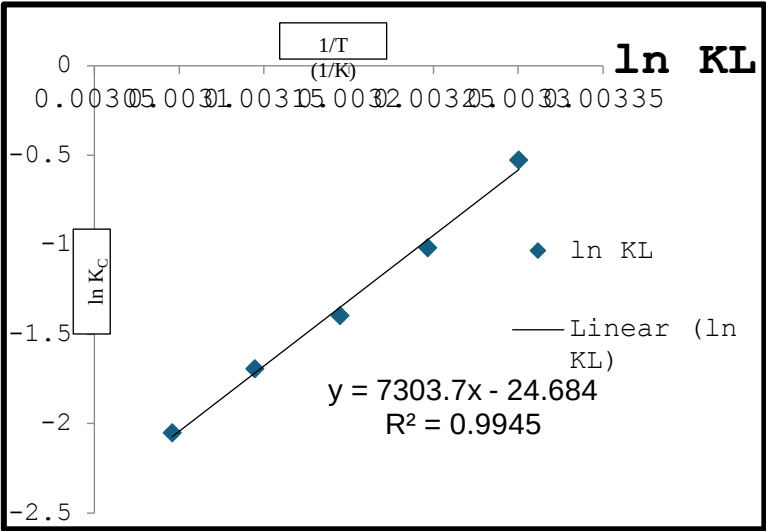


Fig:5.48: Thermodynamics for SWBC

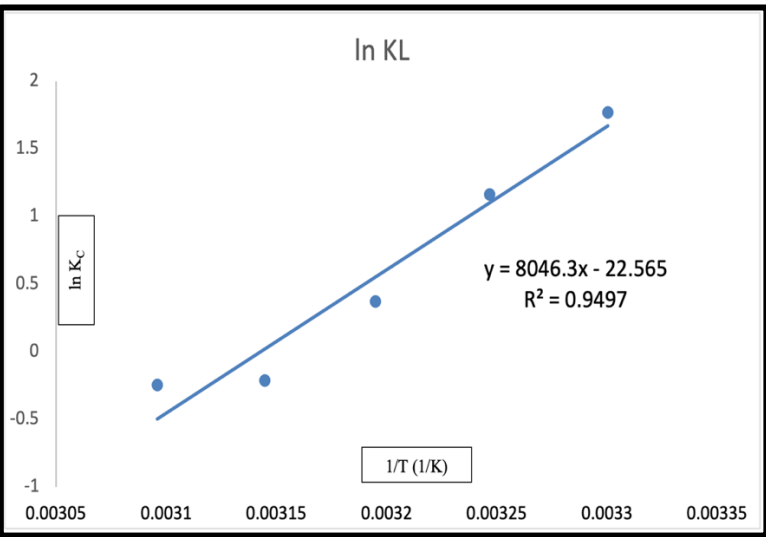


Fig:5.49: Thermodynamics for CSWBC

Table:5.8: Thermodynamics for SWBC and CSWBC

Sample	Intercept	Slope	ΔH^0 (KJmol ⁻¹)	ΔS^0 (JK ⁻¹ mol ⁻¹)	R ²	ΔG^0 (JK ⁻¹ mol ⁻¹)
SWBC	-24.68	7303	-60717.1	-205.18952	0.9945	-0.4896
CSWBC	-33.682	12324	-10.48365	-329.9161	0.9497	-3624.169

SWBC

Based on the calculated values:

$\Delta G = -0.4896$ negative value indicates a spontaneous adsorption process. External energy input will not be required for efficient ciprofloxacin removal.

- $\Delta S = -205.18$ J/mol*K: The negative entropy change suggests a decrease in randomness at the adsorbent-ciprofloxacin interface during adsorption. This could be due to increased order and specific interactions between the antibiotic and the adsorbent surface.
- $\Delta H = -60.717$ kJ/mol: The negative enthalpy change signifies an exothermic process. The adsorption of ciprofloxacin releases heat, suggesting a favourable interaction between the antibiotic and the adsorbent.

The adsorption of ciprofloxacin onto the chosen adsorbent seems favourable due to the exothermic nature ($\Delta H < 0$). However, the positive ΔG value indicates the process is not spontaneous. Further investigation is needed to optimize the adsorption process for efficient ciprofloxacin removal from water sources.

CSWBC

Based on the calculated thermodynamic parameters:

- $\Delta G^\circ = -4.454$ kJ/mol (negative): This indicates a spontaneous adsorption process. The negative Gibbs free energy suggests that the system favours the adsorption of the target molecule onto the adsorbent.

- $\Delta S^\circ = -277.60 \text{ J/mol}\cdot\text{K}$ (negative): This negative entropy change suggests a decrease in randomness at the interface during adsorption. This could be due to the ordering of the target molecule on the adsorbent surface or a decrease in the mobility of the molecule upon adsorption.
- $\Delta H^\circ = -88.336 \text{ kJ/mol}$ (negative): This negative enthalpy change signifies an exothermic adsorption process. The release of energy during adsorption suggests a stabilizing interaction between the target molecule and the adsorbent surface.

The thermodynamic data suggests that the adsorption of the target molecule onto the adsorbent is spontaneous and exothermic. The decrease in entropy indicates an ordering of the system during adsorption. These findings suggest a favourable and efficient adsorption process for the chosen system.

CHAPTER – 6

SUMMARY AND CONCLUSION

6.1 SUMMARY

Characterization of the biochars, *Sargassum wightii* biochar (SWBC) and chitosan coated *Sargassum wightii* biochar (CSWBC), was conducted to understand their structural and chemical properties in relation to their adsorption performance for ciprofloxacin (CIP) removal from water systems. The SWBC was initially analyzed using scanning electron microscopy (SEM) to examine its surface morphology, revealing a porous structure with irregular shapes and sizes. Energy-dispersive X-ray spectroscopy (EDX) was employed to determine the elemental composition, highlighting the presence of carbon, oxygen, and other trace elements. Fourier-transform infrared spectroscopy (FTIR) was utilized to identify functional groups present in SWBC, indicating the presence of hydroxyl, carbonyl, and aromatic groups, which are crucial for adsorption processes. X-ray diffraction (XRD) analysis revealed the crystalline structure of SWBC, with diffraction peaks corresponding to graphitic carbon and other minerals. Additionally, these characterizations provide crucial insights into the structural and chemical properties of SWBC, laying the foundation for understanding its adsorption behavior.

In contrast, CSWBC underwent additional characterization to assess the impact of chitosan coating on its properties. SEM images of CSWBC displayed a similar porous structure to SWBC, suggesting that the chitosan coating did not significantly alter its morphology. However, EDAX analysis revealed an increase in nitrogen content in CSWBC compared to SWBC, confirming the presence of chitosan. FTIR spectra of CSWBC exhibited additional peaks corresponding to chitosan functional groups, indicating successful coating. XRD analysis showed similar diffraction patterns to SWBC, suggesting that the chitosan coating did not significantly affect the crystalline structure. These characterizations provide crucial insights into the structural and chemical properties of CSWBC, laying the foundation for understanding its enhanced adsorption performance for CIP removal from water systems.

6.2 CONCLUSION

The adsorption performance of SWBC and CSWBC for CIP removal from aqueous solutions was evaluated through batch adsorption experiments. The results revealed that both biochars exhibited significant adsorption capacity for CIP, with CSWBC demonstrating superior performance compared to SWBC. The adsorption kinetics of SWBC and CSWBC followed pseudo-second-order kinetics, suggesting a chemisorption process. Furthermore, adsorption isotherm studies indicated that the Langmuir model provided the best fit for the experimental data, highlighting monolayer adsorption on the biochar surfaces. The thermodynamic parameters indicated that the adsorption process was spontaneous and endothermic. Overall, these results demonstrate the potential of SWBC and CSWBC as effective adsorbents for the removal of CIP from water systems, with CSWBC showing enhanced adsorption performance attributed to the chitosan coating. These findings contribute to the development of sustainable and efficient solutions for pharmaceutical pollutant removal from water resources, addressing an increasingly pressing environmental concern.

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ABSTRACT:

¹This study investigated the removal of ciprofloxacin, a common antibiotic, from wastewater using Sargassum biochar (SWBC) and chitosan coated Sargassum biochar (CSWBC). The adsorption behaviors of both biochars were evaluated and compared.

Sargassum biochar exhibited a Freundlich isotherm model, indicating a multi-layered adsorption process. ²The maximum ciprofloxacin adsorption capacity ($Q_{\max}$) for SWBC was 4.91 mg/g. ²Kinetic studies revealed that ciprofloxacin adsorption onto SWBC followed a pseudo-first-order model, suggesting a physical adsorption mechanism. The negative enthalpy change (ΔH°) indicated an exothermic process, but the positive Gibbs free energy change (ΔG°) suggested a non-spontaneous adsorption for SWBC.

In contrast, chitosan coated Sargassum biochar followed a Langmuir isotherm model, signifying a monolayer adsorption process. The $Q_{\max}$ value for CSWBC was significantly higher (34.17 mg/g) compared to SWBC. Elovich kinetic model best described the ciprofloxacin adsorption onto CSWBC, implying a complex diffusion-based mechanism. The negative values for both ΔH° and ΔG° confirmed the exothermic and spontaneous nature of ciprofloxacin adsorption onto CSWBC.

These findings demonstrate that chitosan coating significantly enhanced the ciprofloxacin adsorption capacity and spontaneity compared to unmodified Sargassum biochar. ³⁹This study offers valuable insights into the potential of biochar-based materials for antibiotic removal from wastewater.

CHAPTER - 1

INTRODUCTION

Wastewater contamination stands as an ominous testament to the complex interplay between human activity and the environment, embodying a myriad of pollutants that threaten ecological balance and human health. From industrial effluents to agricultural runoff, urban sewage to pharmaceutical residues, wastewater serves as a repository for diverse contaminants, each bearing its own set of environmental and societal implications. ²¹ The discharge of untreated or inadequately treated wastewater into natural water bodies poses a grave risk, as pollutants infiltrate aquatic ecosystems, disrupt food chains, and compromise biodiversity. Heavy metals, organic chemicals, pathogens, and nutrients are among the notorious culprits that contaminate wastewater, originating from industrial processes, agricultural practices, domestic activities, and healthcare facilities. These contaminants, often present in complex mixtures, exhibit varying persistence and toxicity levels, amplifying the challenge of effective wastewater management. ⁸¹ Moreover, the proliferation of emerging contaminants, such as pharmaceuticals and personal care products, adds a layer of complexity to the problem, as conventional treatment methods struggle to adequately address these novel pollutants. The consequences of wastewater contamination are far-reaching, extending beyond environmental degradation to encompass public health hazards, economic burdens, and social disparities. Vulnerable populations, including marginalized communities and developing regions, bear the brunt of inadequate sanitation infrastructure, suffering from waterborne diseases and compromised livelihoods. Despite concerted efforts to mitigate wastewater pollution through regulatory frameworks, technological innovations, and public awareness campaigns, the magnitude of the challenge persists, underscoring the urgent need for holistic and sustainable approaches to wastewater management. In this context, understanding the dynamics of wastewater contamination becomes paramount, as it lays the groundwork for informed decision-making, resource allocation, and collaborative action towards safeguarding water resources and promoting global sustainability.

1.1 PHARMACEUTICAL POLLUTION MITIGATION

1.1.1 EMERGING ENVIRONMENTAL THREATS: PHARMACEUTICAL POLLUTANTS

Emerging environmental threats, particularly pharmaceutical pollutants, represent a significant challenge at the nexus of public health and environmental sustainability. Pharmaceutical pollutants, comprising a diverse array of active pharmaceutical ingredients (APIs) and their metabolites, enter aquatic environments through various pathways, including excretion, improper disposal, and wastewater effluents from pharmaceutical manufacturing facilities and healthcare facilities. These compounds exhibit unique properties that differentiate them from traditional pollutants, including bioactivity at low concentrations, persistence in the environment, and potential to induce adverse effects on aquatic organisms and human health. Their presence in surface waters, groundwater, and even drinking water sources has raised concerns regarding ecological integrity, antimicrobial resistance, and potential human health risks associated with chronic exposure. Furthermore, the complex mixture of pharmaceutical pollutants, coupled with their interactions with other environmental contaminants and factors such as climate change and land use, complicates the assessment and management of these emerging threats. Addressing pharmaceutical pollution requires a multifaceted approach encompassing source reduction, improved wastewater treatment technologies, regulatory interventions, and public awareness initiatives. Collaboration among stakeholders, including government agencies, pharmaceutical industries, healthcare providers, environmental organizations, and the public, is essential to mitigate the impact of pharmaceutical pollutants and safeguard both environmental and human health. As research continues to advance our understanding of the fate, transport, and effects of pharmaceutical pollutants in the environment, concerted efforts are needed to develop innovative solutions and enact policies that promote sustainable pharmaceutical use and wastewater management practices.

1.1.2 THE NEED FOR INNOVATIVE WASTEWATER TREATMENT SOLUTIONS

The need for innovative wastewater treatment solutions has become increasingly urgent in light of escalating environmental challenges and growing pressures on

freshwater resources. Conventional wastewater treatment methods, while effective to some extent, are often unable to adequately address the complex mixture of contaminants present in wastewater, including emerging pollutants such as pharmaceuticals, personal care products, and microplastics. Moreover, traditional treatment processes may be energy-intensive, costly, and generate sludge or by-products that require further disposal. As populations continue to urbanize and industrialize, the volume and complexity of wastewater generated pose significant challenges for existing infrastructure and treatment capacity. In response, there is a pressing need for the development and adoption of innovative technologies and approaches that can enhance the efficiency, sustainability, and resilience of wastewater treatment systems. This includes the exploration of nature-based solutions, such as constructed wetlands and green infrastructure, which harness natural processes to remove contaminants and enhance water quality. Additionally, advances in membrane filtration, advanced oxidation processes, and electrochemical treatment hold promise for achieving higher removal efficiencies and treating wastewater to higher quality standards. Furthermore, the integration of decentralized treatment systems, smart sensors, and data analytics can improve monitoring and control, optimize resource recovery, and enhance the overall performance of wastewater treatment plants. Collaborative research efforts involving academia, industry, and government agencies are essential for driving innovation and overcoming barriers to the adoption of novel wastewater treatment solutions. By embracing innovation and leveraging technological advancements, we can create more sustainable and resilient wastewater treatment systems that protect human health, safeguard the environment, and promote the wise stewardship of our precious water resources.

1.1.3 UNDERSTANDING CIPROFLOXACIN AS A PERSISTENT CONTAMINANT

Understanding ciprofloxacin as a persistent contaminant is crucial in addressing its widespread presence and potential environmental impacts. Ciprofloxacin, a broad-spectrum fluoroquinolone antibiotic widely used in human and veterinary medicine, has garnered attention due to its persistence in aquatic environments. Despite its intended therapeutic effects, ciprofloxacin can enter wastewater systems through various pathways, including excretion by humans and animals, disposal of unused medications, and discharge from pharmaceutical manufacturing facilities. Once

introduced into water bodies, ciprofloxacin exhibits resistance to degradation processes, such as photolysis, hydrolysis, and microbial metabolism, leading to its accumulation over time. This persistence is attributed to the chemical structure of ciprofloxacin, which contains fluorine atoms that enhance its stability and resistance to environmental degradation. Furthermore, factors such as pH, temperature, and the presence of other chemicals can influence the rate of ciprofloxacin degradation, further exacerbating its persistence in aquatic ecosystems. The long-term presence of ciprofloxacin in the environment raises concerns about potential adverse effects on aquatic organisms, including antibiotic resistance development, disruption of microbial communities, and ecological imbalances. Additionally, there is growing evidence of ciprofloxacin's role in promoting the spread of antibiotic resistance genes, posing a threat to public health and exacerbating the global challenge of antimicrobial resistance. To address the persistence of ciprofloxacin as a contaminant, interdisciplinary research efforts are needed to elucidate its fate and transport mechanisms, assess its ecological and human health risks, and develop effective mitigation strategies. This includes the implementation of advanced analytical techniques for detecting and quantifying ciprofloxacin in environmental matrices, modelling approaches to predict its environmental fate, and innovative treatment technologies for its removal from wastewater. Furthermore, collaborative initiatives involving policymakers, healthcare professionals, pharmaceutical industries, and environmental scientists are essential to enact regulatory measures, promote responsible antibiotic use, and minimize the environmental footprint of ciprofloxacin and other pharmaceutical pollutants. By enhancing our understanding of ciprofloxacin as a persistent contaminant and implementing proactive measures to mitigate its environmental impact, we can strive towards a more sustainable and resilient aquatic ecosystem.

1.1.4 ROLE OF BIOCHAR IN SUSTAINABLE ENVIRONMENTAL REMEDIATION

The role of biochar in sustainable environmental remediation encompasses a multifaceted approach to addressing diverse environmental challenges through the application of this carbonaceous material derived from biomass pyrolysis. Biochar, renowned for its high surface area, porosity, and carbon sequestration potential, holds promise as a versatile tool for mitigating various environmental issues, including soil degradation, water pollution, and climate change. In soil remediation,

biochar amendments enhance soil fertility, improve water retention capacity, and sequester carbon, thereby promoting soil health and productivity while mitigating greenhouse gas emissions. Additionally, biochar acts as a sorbent for contaminants, such as heavy metals, organic pollutants, and nutrients, facilitating their immobilization and reducing their bioavailability in soil and water systems. In water remediation, biochar-based materials demonstrate exceptional adsorption capacity for pollutants, including pesticides, pharmaceuticals, and emerging contaminants, offering a cost-effective and sustainable solution for water treatment. Moreover, biochar-mediated nutrient removal from agricultural runoff and wastewater streams mitigates eutrophication and improves water quality in aquatic ecosystems. Beyond its direct remediation capabilities, biochar contributes to climate change mitigation through carbon sequestration, as its stable carbon structure can persist in soils for hundreds to thousands of years, effectively removing atmospheric carbon dioxide. Furthermore, biochar production from organic waste materials offers a means of waste valorisation and resource recovery, closing nutrient loops and reducing reliance on fossil fuels. However, the efficacy of biochar in environmental remediation is influenced by various factors, including feedstock selection, pyrolysis conditions, and application rates, necessitating careful consideration of site-specific conditions and objectives. Collaborative research efforts, involving academia, industry, and government agencies, are essential to advancing the science and technology of biochar-based environmental remediation and realizing its full potential in promoting sustainability and resilience in terrestrial and aquatic ecosystems.

1.1.5 EXPLORING SARGASSUM BIOMASS AS A RENEWABLE RESOURCE

Exploring *Sargassum* biomass as a renewable resource unveils a plethora of opportunities for sustainable utilization across various sectors, ranging from agriculture to energy production and environmental remediation. *Sargassum*, a genus of brown macroalgae, thrives in marine ecosystems, forming extensive floating mats known as *Sargassum wightii*. With its rapid growth rate, high biomass yield, and ability to absorb nutrients and carbon dioxide from seawater, *Sargassum* represents a promising renewable resource for multiple applications. In agriculture, *Sargassum* biomass serves as a nutrient-rich organic fertilizer, enhancing soil fertility, promoting crop growth, and improving yield quality. Its bioactive compounds,

such as alginates, polysaccharides, and plant hormones, confer beneficial effects on soil structure, water retention, and plant resilience to environmental stressors. Furthermore, *Sargassum* extracts have demonstrated potential as bio stimulants and biopesticides, offering natural alternatives to synthetic agrochemicals and supporting sustainable agricultural practices. In the energy sector, *Sargassum* biomass holds promise as a feedstock for biofuel production, particularly through anaerobic digestion and thermochemical conversion processes. Its high carbohydrate content, low lignin content, and fast growth rate make *Sargassum* an attractive candidate for bioenergy generation, providing a renewable and carbon-neutral alternative to fossil fuels. Moreover, *Sargassum*-derived biochar exhibits excellent adsorption properties, making it effective for wastewater treatment, soil remediation, and carbon sequestration applications. By harnessing *Sargassum* biomass as a renewable resource, we can not only mitigate environmental degradation but also foster economic development, enhance food security, and promote climate resilience in coastal communities. However, sustainable management practices and ecosystem-based approaches are essential to ensure the long-term viability of *Sargassum* resources and minimize potential ecological impacts. Collaborative research efforts, stakeholder engagement, and policy support are critical to unlocking the full potential of *Sargassum* biomass and realizing its role in the transition towards a more sustainable and resilient future.

1.1.6 INTRODUCTION TO CHITOSAN: A BIOPOLYMER WITH ADSORPTION POTENTIAL

Chitosan, a versatile biopolymer derived from the deacetylation of chitin, presents itself as a promising material with remarkable adsorption potential across various applications. With its unique properties, including biocompatibility, biodegradability, and non-toxicity, chitosan has garnered significant attention in diverse fields such as wastewater treatment, environmental remediation, biomedical engineering, and food technology. The abundance of chitin in natural sources such as crustacean shells, fungal cell walls, and certain insects, coupled with the relative ease of chitosan extraction and modification, underscores its attractiveness as a sustainable alternative to synthetic adsorbents. In wastewater treatment, chitosan-based adsorbents exhibit high affinity towards a wide range of contaminants, including heavy metals, dyes, organic pollutants, and emerging micropollutants, owing to their

functional groups (e.g., amino and hydroxyl groups) that facilitate adsorption interactions. Moreover, chitosan's intrinsic antimicrobial properties contribute to its efficacy in inhibiting bacterial growth and promoting pollutant removal efficiency. Beyond wastewater treatment, chitosan finds applications in environmental remediation, where it is utilized for the removal of contaminants from soil, air, and groundwater, thereby mitigating environmental pollution and safeguarding human health. In biomedical engineering, chitosan's biocompatibility and mucoadhesive properties make it an ideal candidate for drug delivery systems, wound healing dressings, tissue engineering scaffolds, and biomedical implants. Additionally, chitosan's ability to form films, gels, and coatings enhances its utility in food packaging, preservation, and safety, offering sustainable solutions to reduce food waste and extend shelf life. Despite its numerous advantages, challenges remain in optimizing chitosan-based adsorbents for specific applications, including improving adsorption capacity, selectivity, and reusability, as well as addressing cost considerations and scalability issues. Collaborative research efforts, multidisciplinary approaches, and technological innovations are essential to harnessing the full potential of chitosan as a biopolymer with adsorption potential, advancing sustainable development goals, and addressing global environmental and health challenges.

1.1.7 BRIDGING NATURE AND TECHNOLOGY: SARGASSUM BIOCHAR SYNTHESIS

Sargassum biochar synthesis represents a novel and sustainable approach to addressing environmental challenges while harnessing the potential of marine biomass. *Sargassum*, a brown macroalgae abundant in coastal regions, offers a readily available and renewable feedstock for biochar production. By subjecting *Sargassum* biomass to pyrolysis, a thermochemical conversion process under oxygen-limited conditions, biochar is synthesized, retaining the carbon-rich structure of the original biomass. This biochar, enriched with carbonaceous materials and mineral nutrients, exhibits unique properties that make it an attractive material for various applications. In environmental remediation, *Sargassum* biochar demonstrates exceptional adsorption capacity for pollutants, including heavy metals, organic contaminants, and nutrients, thereby mitigating soil and water pollution and promoting ecosystem health. Moreover, *Sargassum* biochar enhances

soil fertility, improves soil structure, and sequesters carbon, contributing to sustainable agriculture and climate change mitigation efforts. Beyond its role in environmental applications, *Sargassum* biochar finds utility in renewable energy production, where it serves as a feedstock for bioenergy generation through combustion, gasification, or pyrolysis processes, offering a carbon-neutral alternative to fossil fuels. Furthermore, *Sargassum* biochar-derived products, such as biochar-based fertilizers, soil amendments, and carbon sequestration agents, hold promise for enhancing agricultural productivity, restoring degraded lands, and promoting circular economy principles. However, challenges remain in optimizing *Sargassum* biochar synthesis processes, such as pyrolysis conditions, feedstock characteristics, and biochar properties, to maximize efficiency, yield, and quality. Additionally, considerations regarding the scalability, economic viability, and environmental sustainability of *Sargassum* biochar production warrant further research and development. Collaborative efforts between scientists, engineers, policymakers, and stakeholders are essential to advancing *Sargassum* biochar synthesis technologies, fostering innovation, and realizing the full potential of this nature-inspired approach in bridging nature and technology for sustainable development.

1.1.8 ENHANCING ADSORPTION CAPACITY THROUGH THE COATING OF BIOCHAR WITH CHITOSAN

Enhancing adsorption capacity through the coating of biochar with chitosan represents a promising strategy for improving the effectiveness of biochar-based adsorbents in various environmental applications. Biochar, derived from the pyrolysis of organic biomass, possesses a high surface area and porosity, making it an excellent candidate for adsorption processes. However, the inherent limitations of bare biochar, such as low selectivity and affinity towards specific contaminants, can hinder its performance in complex wastewater matrices. By coating biochar with chitosan, a biopolymer renowned for its adsorption capabilities and surface modification properties, the resulting composite material can exhibit enhanced adsorption capacity, selectivity, and stability. The chitosan coating provides functional groups, such as amino and hydroxyl groups, that facilitate the binding of target pollutants through electrostatic interactions, hydrogen bonding, and complexation mechanisms. Additionally, chitosan imparts additional benefits to the

biochar matrix, including improved mechanical strength, resistance to leaching, and compatibility with aqueous environments. The synergistic effects between biochar and chitosan not only enhance adsorption performance but also expand the applicability of the composite adsorbent to a wider range of contaminants, including heavy metals, organic pollutants, and emerging micropollutants. Furthermore, the sustainable nature of both biochar and chitosan, derived from renewable biomass sources, underscores the eco-friendly and cost-effective attributes of this innovative adsorption technology. Through systematic characterization studies, optimization of coating processes, and rigorous evaluation in batch and continuous flow systems, the potential of biochar-chitosan composites can be fully realized, offering a sustainable solution for wastewater treatment, environmental remediation, and resource recovery.

1.1.9 ADDRESSING RESEARCH GAPS IN PHARMACEUTICAL POLLUTION MITIGATION

Addressing research gaps in pharmaceutical pollution mitigation is imperative for understanding and effectively managing the environmental impacts of pharmaceutical residues on aquatic ecosystems and human health. While pharmaceuticals play a crucial role in healthcare, their incomplete removal during wastewater treatment processes leads to their release into natural water bodies, posing potential risks to aquatic organisms and ecosystems. The complexity of pharmaceutical pollution stems from the diverse chemical compositions, varied modes of action, and differing environmental fates of pharmaceutical compounds. Furthermore, the interactions between pharmaceuticals and environmental matrices, such as sediments, soils, and biota, remain poorly understood, hindering accurate risk assessments and mitigation strategies. Key research gaps include elucidating the ecotoxicological effects of pharmaceuticals on aquatic organisms, understanding the mechanisms of bioaccumulation and biomagnification in food webs, and assessing the long-term ecological consequences of chronic exposure to low concentrations of pharmaceuticals. Additionally, there is a need to develop sensitive analytical methods for detecting and quantifying pharmaceutical residues in environmental samples, as well as predictive modelling tools for assessing the fate and transport of pharmaceuticals in aquatic systems. Moreover, interdisciplinary approaches integrating environmental science, toxicology,

pharmacology, chemistry, and engineering are essential for bridging knowledge gaps, generating holistic insights into pharmaceutical pollution dynamics, and informing evidence-based decision-making processes. By addressing these research gaps, stakeholders can develop effective mitigation strategies, such as advanced wastewater treatment technologies, source control measures, and regulatory frameworks, to minimize pharmaceutical pollution and protect both environmental and human health.

1.1.10 ENVIRONMENTAL IMPLICATIONS OF CIPROFLOXACIN PERSISTENCE

The environmental implications of ciprofloxacin persistence are multifaceted and underscore the complex interplay between pharmaceutical contamination and ecosystem health. Ciprofloxacin, a widely prescribed fluoroquinolone antibiotic, exhibits remarkable stability and persistence in aquatic environments due to its resistance to degradation processes such as photolysis, hydrolysis, and microbial metabolism. This persistence results in its accumulation in water bodies, sediments, and soils, leading to potential adverse effects on aquatic organisms, ecosystem functioning, and human health. One significant environmental implication is the development of antibiotic resistance among bacteria in aquatic ecosystems exposed to ciprofloxacin residues. The selective pressure exerted by ciprofloxacin contamination can promote the proliferation of antibiotic-resistant bacterial strains, including those with clinical relevance, posing a threat to public health and complicating the treatment of bacterial infections. Moreover, ciprofloxacin persistence can disrupt microbial communities and ecological processes in aquatic environments, leading to imbalances in nutrient cycling, decreased biodiversity, and altered ecosystem dynamics. Ciprofloxacin residues may also bioaccumulate and bio magnify through aquatic food webs, potentially impacting higher trophic levels, including fish and aquatic birds, and posing risks to human consumers. Furthermore, ciprofloxacin persistence raises concerns about its potential long-term effects on ecosystem resilience, ecosystem services, and the sustainability of aquatic habitats. Mitigating the environmental implications of ciprofloxacin persistence requires a holistic approach that integrates regulatory measures, advanced wastewater treatment technologies, pollution prevention strategies, and public awareness campaigns. By addressing the root causes of pharmaceutical contamination, promoting responsible antibiotic use, and implementing sustainable

water management practices, stakeholders can minimize the environmental footprint of ciprofloxacin and safeguard aquatic ecosystems for future generations.

1.2 ADSORPTION KINETICS AND EFFICIENCY

1.2.1 RATIONALE FOR STUDYING CIPROFLOXACIN REMOVAL

The rationale for studying ciprofloxacin removal is rooted in the recognition of the significant environmental and public health risks posed by the widespread presence of this antibiotic in aquatic ecosystems. Ciprofloxacin, a broad-spectrum fluoroquinolone antibiotic commonly used in both human and veterinary medicine, has been detected in various environmental compartments, including surface waters, groundwater, and wastewater treatment plants. Its persistence and resistance to degradation in aquatic environments raise concerns about its potential adverse effects on ecosystem health, biodiversity, and human health. Ciprofloxacin contamination is particularly worrisome due to its association with the development of antibiotic-resistant bacteria, which can compromise the effectiveness of clinical treatments and escalate the spread of antibiotic resistance in the environment. Furthermore, ciprofloxacin's presence in aquatic ecosystems can disrupt microbial communities, interfere with nutrient cycling processes, and alter the ecological balance of aquatic habitats. Given these environmental and public health implications, there is a critical need to understand the fate, transport, and behaviour of ciprofloxacin in aquatic environments, as well as develop effective mitigation strategies for its removal from water systems. By investigating the mechanisms of ciprofloxacin sorption onto adsorbent materials, evaluating the performance of treatment technologies for ciprofloxacin removal, and assessing the environmental fate of ciprofloxacin and its degradation products, research endeavours aim to provide insights into the risks associated with ciprofloxacin contamination and inform strategies for mitigating its environmental impact. Through interdisciplinary collaborations between environmental scientists, chemists, engineers, and public health experts, studies on ciprofloxacin removal seek to contribute to the development of sustainable solutions for protecting water quality, safeguarding ecosystems, and preserving public health.

1.2.2 SUSTAINABLE APPROACH: UTILIZING SARGASSUM BIOMASS FOR BIOCHAR PRODUCTION

A sustainable approach to utilizing *Sargassum* biomass for biochar production holds immense potential for addressing environmental challenges, promoting resource efficiency, and fostering economic development in coastal regions. *Sargassum*, a genus of brown macroalgae abundant in marine ecosystems, offers a renewable and readily available feedstock for biochar production. By subjecting *Sargassum* biomass to pyrolysis, a thermochemical conversion process conducted in the absence of oxygen, biochar is generated, retaining the carbon-rich structure of the original biomass while converting volatile organic compounds into stable carbonaceous materials. This sustainable approach to biochar production not only sequesters carbon and mitigates greenhouse gas emissions but also addresses the issue of marine debris accumulation and beach inundation caused by *Sargassum* blooms. Moreover, *Sargassum*-derived biochar exhibits exceptional properties that make it suitable for various environmental applications, including soil amendment, carbon sequestration, and wastewater treatment. In agriculture, *Sargassum* biochar enhances soil fertility, improves soil structure, and promotes plant growth by supplying essential nutrients, enhancing water retention capacity, and stimulating microbial activity. Furthermore, *Sargassum* biochar can serve as a cost-effective and environmentally friendly alternative to conventional fertilizers and soil conditioners, reducing dependence on synthetic chemicals and minimizing nutrient runoff and leaching. Additionally, *Sargassum* biochar shows promise for wastewater treatment applications, where its high surface area and porosity enable efficient adsorption of pollutants, including heavy metals, organic contaminants, and nutrients, from aqueous solutions. By harnessing *Sargassum* biomass for biochar production, stakeholders can simultaneously address marine pollution, climate change, and soil degradation challenges while creating opportunities for sustainable development and circular economy initiatives in coastal communities.

1.2.3 INVESTIGATING THE PHYSICOCHEMICAL PROPERTIES OF SARGASSUM BIOCHAR

Investigating the physicochemical properties of *Sargassum* biochar is essential for understanding its suitability and potential applications in various environmental and agricultural contexts. *Sargassum* biochar, derived from the pyrolysis of *Sargassum*

biomass, possesses unique characteristics that contribute to its effectiveness as a soil amendment, pollutant adsorbent, and carbon sequestration agent. Physically, *Sargassum* biochar exhibits a porous structure with a high surface area, which enhances its adsorption capacity and facilitates interactions with soil particles and contaminants in aqueous solutions. The porosity of *Sargassum* biochar influences its water-holding capacity, nutrient retention, and microbial habitat suitability in soil environments, thereby improving soil fertility, moisture retention, and overall productivity. Chemically, *Sargassum* biochar is rich in carbonaceous materials, functional groups (e.g., carboxyl, hydroxyl, and phenolic groups), and mineral constituents derived from the original biomass, which contribute to its sorption capacity, cation exchange capacity, and nutrient availability in soil systems. Understanding the physicochemical properties of *Sargassum* biochar requires comprehensive characterization techniques, including surface area analysis, pore size distribution, elemental composition analysis, Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and X-ray diffraction (XRD). Through systematic investigation of these properties, researchers can elucidate the mechanisms underlying *Sargassum* biochar's performance in soil amendment, pollutant remediation, and carbon sequestration applications, as well as optimize its production processes and tailor its properties for specific environmental and agricultural needs. By harnessing the insights gained from studying the physicochemical properties of *Sargassum* biochar, stakeholders can develop innovative and sustainable solutions for addressing soil degradation, water pollution, and climate change challenges, thereby promoting environmental resilience and agricultural sustainability.

1.2.4 SURFACE MODIFICATION TECHNIQUES: COATING BIOCHAR WITH CHITOSAN

Coating of biochar with chitosan present a promising avenue for enhancing the performance and versatility of biochar-based adsorbents in environmental applications. Biochar, a carbon-rich material derived from the pyrolysis of organic biomass, possesses a porous structure and high surface area that lend it exceptional adsorption properties. However, the inherent limitations of bare biochar, such as low selectivity and affinity towards specific contaminants, can hinder its effectiveness in pollutant removal. Surface modification techniques, particularly

coating biochar with chitosan, offer a solution to overcome these limitations and tailor biochar properties to target contaminants. Chitosan, exhibits favourable characteristics for surface modification, including biocompatibility, biodegradability, and functional groups (e.g., amino and hydroxyl groups) that facilitate interactions with contaminants. The coating process involves immersing biochar particles in a chitosan solution, followed by drying and curing to form a stable chitosan layer on the biochar surface. This chitosan coating imparts additional functionalities to the biochar matrix, such as improved selectivity, enhanced stability, and controlled release of active sites for adsorption. Moreover, chitosan-coated biochar exhibits enhanced resistance to leaching and aggregation, prolonging its effectiveness in aqueous environments. The synergistic effects between biochar and chitosan result in composite materials with superior adsorption capacity and selectivity for a wide range of contaminants, including heavy metals, organic pollutants, and emerging micropollutants. Furthermore, chitosan-coated biochar demonstrates potential applications in wastewater treatment, soil remediation, and water purification, offering sustainable solutions for mitigating environmental pollution and promoting resource recovery. Through further research and development, optimization of coating techniques, and rigorous evaluation of performance in real-world conditions, chitosan-coated biochar holds promise for addressing complex environmental challenges and advancing towards a more sustainable future.

1.2.5 ANALYTICAL METHODS FOR CHARACTERIZING ADSORBENT MATERIALS

Analytical methods for characterizing adsorbent materials play a crucial role in understanding their physicochemical properties, surface characteristics, and adsorption mechanisms, thereby facilitating the design and optimization of adsorption processes for environmental remediation and purification applications. Various techniques are employed to comprehensively analyse adsorbent materials, providing insights into their structure, composition, morphology, surface area, porosity, and surface chemistry. Scanning electron microscopy (SEM) allow for visualization of adsorbent morphology and particle size distribution, enabling characterization of surface features, such as pores, cracks, and surface roughness. X-ray diffraction (XRD) analysis provides insights into the crystalline structure and phase composition of adsorbent materials, aiding in identifying mineral phases and

structural changes upon modification or functionalization. Fourier-transform infrared spectroscopy (FTIR) offer valuable ⁸⁹ information on the chemical composition, functional groups, and surface chemistry of adsorbents, facilitating the identification of surface functional groups and interactions involved in adsorption processes. By employing a combination of these analytical methods, researchers can comprehensively characterize adsorbent materials, elucidate ⁵ their adsorption mechanisms, and optimize their performance for various environmental and industrial applications, ultimately contributing to the development of efficient and sustainable adsorption processes.

⁹ 1.2.6 DESIGNING BATCH ADSORPTION EXPERIMENTS FOR CIPROFLOXACIN REMOVAL

Designing batch adsorption experiments for ciprofloxacin removal involves a systematic approach to evaluate the adsorption efficiency of adsorbent materials under controlled laboratory conditions, providing valuable insights into ¹¹³ the adsorption kinetics, equilibrium behaviour, and influencing factors. The experimental design encompasses several key aspects, including selection of adsorbent materials, preparation of ciprofloxacin solutions, determination of experimental parameters, and data analysis methods. Firstly, adsorbent materials with potential for ciprofloxacin removal are chosen based on their physicochemical properties, surface characteristics, and previous performance in adsorption studies. These materials may include activated carbon, biochar, modified clays, and functionalized polymers, among others. Secondly, ciprofloxacin solutions are prepared at desired concentrations, typically ranging from environmentally relevant levels to higher concentrations for adsorption isotherm studies, ensuring a representative range of adsorption conditions. Thirdly, experimental parameters ⁵⁹ such as initial ciprofloxacin concentration, adsorbent dosage, solution pH, temperature, agitation speed, and contact time are systematically varied to investigate their effects on adsorption performance. A series of batch experiments are conducted, where adsorbent materials are added to ciprofloxacin solutions in sealed containers, and samples are withdrawn at regular intervals to monitor changes in ciprofloxacin concentration over time. Analytical technique UV-visible spectrophotometry employed to quantify ciprofloxacin concentrations in solution before and after adsorption. The obtained data is then analysed using kinetic models

⁴ (e.g., pseudo-first-order, pseudo-second-order), isotherm models (e.g., Langmuir, Freundlich), and thermodynamic parameters (e.g., ΔG , ΔH , ΔS) to elucidate the adsorption mechanism, determine adsorption kinetics, equilibrium capacities, and thermodynamic feasibility. By systematically designing and conducting batch adsorption experiments, researchers can gain valuable insights into the feasibility and efficiency of adsorbent materials for ciprofloxacin removal, informing the development of effective adsorption processes for water treatment and environmental remediation applications.

1.2.7 ASSESSING FACTORS INFLUENCING ADSORPTION EFFICIENCY

Assessing factors influencing adsorption efficiency is critical for understanding the performance and optimizing the effectiveness of adsorbent materials in removing contaminants from aqueous solutions. Numerous factors can impact adsorption efficiency, including adsorbate properties, adsorbent characteristics, solution conditions, and external factors. Adsorbate properties such as molecular size, polarity, charge, and concentration influence its affinity and interactions with the adsorbent surface. Additionally, adsorbent characteristics such as surface area, pore size distribution, surface chemistry, and functional groups determine the adsorption capacity, selectivity, and kinetics. Solution conditions, including pH, temperature, ionic strength, and presence of competing ions, can significantly affect adsorption efficiency by altering the electrostatic interactions, surface charge, and solubility of both adsorbate and adsorbent. External factors such as agitation speed, contact time, adsorbent dosage, and particle size distribution also play crucial roles in determining adsorption efficiency by influencing mass transfer kinetics, diffusion rates, and equilibrium attainment. To assess the factors influencing adsorption efficiency, systematic experimental studies are conducted, where individual factors are varied while keeping others constant, and the resulting adsorption performance is evaluated. Kinetic modelling, isotherm analysis, thermodynamic calculations, and surface characterization techniques are utilized to understand the underlying mechanisms, quantify adsorption parameters, and predict adsorption behaviour under different conditions. By systematically assessing the factors influencing adsorption efficiency, researchers can optimize adsorption processes, design tailored adsorbents, and develop effective strategies for water treatment, environmental remediation, and resource recovery applications.

1.2.8 ADSORPTION KINETICS AND THERMODYNAMICS

Exploring adsorption kinetics and thermodynamics is essential for understanding the mechanisms, rates, and energetics of adsorption processes, providing valuable insights into the feasibility, efficiency, and optimization of adsorption-based technologies for environmental remediation and purification. Adsorption kinetics refers to the study of how adsorbates are adsorbed onto adsorbent surfaces over time, while adsorption thermodynamics involves investigating the energy changes and spontaneity of adsorption processes. The study of adsorption kinetics involves determining the rate at which adsorption occurs and understanding the factors that influence the rate-controlling steps, such as mass transfer, film diffusion, pore diffusion, and surface reaction. Various kinetic models, including pseudo-first-order, pseudo-second-order, intra-particle diffusion, and Elovich models, are employed to analyse experimental data and elucidate the underlying mechanisms of adsorption. Additionally, factors such as initial adsorbate concentration, temperature, agitation speed, and adsorbent properties are systematically varied to investigate their effects on adsorption kinetics. On the other hand, adsorption thermodynamics examines the equilibrium behaviour and energetics of adsorption processes, including the Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) changes associated with adsorption. Thermodynamic parameters are calculated from adsorption isotherm data using thermodynamic equations, such as the Van's Hoff equation, and provide insights into the spontaneity, feasibility, and stability of adsorption under different conditions. Furthermore, thermodynamic studies enable the determination of optimal operating conditions, the prediction of adsorption behaviour at different temperatures, and the assessment of the suitability of adsorbents for specific applications. By exploring adsorption kinetics and thermodynamics, researchers can gain a comprehensive understanding of adsorption processes, optimize adsorption systems, and develop efficient and sustainable solutions for water treatment, pollutant removal, and resource recovery applications.

1.2.9 TOWARDS SUSTAINABLE WASTEWATER MANAGEMENT: IMPLICATIONS OF THE STUDY

The implications of the ⁴ study on the removal efficiency of ciprofloxacin using *Sargassum* biochar and chitosan-coated *Sargassum* biochar are profound and multifaceted. Firstly, the research contributes to addressing the growing concern of pharmaceutical pollution in aquatic environments, particularly the presence of antibiotics like ciprofloxacin, which pose risks to ecosystem health and human well-being. By demonstrating the effectiveness of *Sargassum* biochar and chitosan-coated biochar in removing ciprofloxacin from wastewater, the study provides tangible solutions for mitigating pharmaceutical contamination and improving water quality. Moreover, ²⁸ the utilization of *Sargassum* biomass as a renewable resource for biochar production highlights the potential for sustainable waste valorisation and resource recovery, reducing reliance on traditional carbon sources and contributing to circular economy principles. Additionally, the environmentally benign nature of chitosan, a biodegradable biopolymer derived from natural sources, underscores the eco-friendly aspects ⁵ of the adsorption process. Furthermore, the study sheds light on the feasibility of employing biochar-based adsorbents in decentralized wastewater treatment systems, offering cost-effective and scalable solutions for wastewater management in both rural and urban settings. By integrating these findings into wastewater treatment practices, stakeholders can advance towards more sustainable and resilient water management strategies, protecting aquatic ecosystems, safeguarding public health, and promoting the sustainable use of natural resources.

CHAPTER – 2

LITERATURE SURVEY:

Thilakshani Atugoda et Al (2021) In this investigation, the innovative approaches to environmental remediation, such as the modification of biochar for enhanced antibiotic removal from water, hold significant promise in addressing contemporary challenges. In this comprehensive study, *Sargassum crassifolium*, a brown sea weed, underwent pyrolysis at 500°C, yielding biochar denoted as SWBC. Notably, this SWBC was further refined through a slurry method, incorporating zeolite at a precise ratio of 1:5 (zeolite: biochar) to produce SWBC-Z, an advanced composite material. Batch adsorption experiments were meticulously conducted to assess the adsorption potential of both SWBC and SWBC-Z in eliminating ciprofloxacin (CPX) from aqueous solutions. Fascinatingly, the adsorption process exhibited a pronounced affinity within the pH range of 6.5–8, attributed to compelling electrostatic attractions and hydrogen bonding interactions between the zwitterionic CPX molecules and the modified biochar surface. Kinetic modelling unveiled an excellent fit with the pseudo-second order and Elovich models, indicating complex adsorption mechanisms driven by chemisorption and physisorption phenomena. Equally compelling, isotherm data were aptly described by the Langmuir and Freundlich models, underscoring the heterogeneous nature of the biochar surface and the diverse adsorption mechanisms at play. Remarkably, SWBC-Z exhibited a remarkable maximum adsorption capacity of 93.65 mg g⁻¹, highlighting the efficacy of zeolite modification in enhancing adsorption performance. Further characterization through Fourier Transform Infrared (FTIR) spectroscopy revealed well-defined peaks corresponding to silanol groups in SWBC-Z, affirming the successful integration of zeolite minerals into the biochar matrix. Additionally, electron microscopy provided compelling visual evidence of the morphological changes and the incorporation of zeolite within the SWBC-Z structure. Post-adsorption FTIR analysis unveiled significant alterations in the surface functional groups of SWBC-Z, further corroborating the adsorption process's intricacies. Thermodynamic analysis underscored the spontaneous and exothermic nature of the reaction between CPX and both biochars, emphasizing the energy-efficient nature of the adsorption process. In sum, the synergistic modification of pristine biochar with zeolite engendered notable improvements in surface area and

adsorption capacity, showcasing its immense potential for sustainable water treatment applications and heralding a paradigm shift in environmental remediation strategies.

Ganning Zeng et Al (2020) In this innovative study, a ground breaking polymer/activated carbon composite was meticulously crafted through a streamlined one-step chemical activation process of *Sargassum horneri*, a marine macroalgae species, coupled with subsequent surface modification using chitosan, a ubiquitous and renewable biopolymer. This composite material, devised with the noble objective of combatting environmental pollution, particularly targeted the removal of one of the most pernicious metal ions, chromium (VI), from aqueous solutions. Through rigorous surface characterization analyses, it was revealed that the composite exhibited predominantly mesoporous characteristics, boasting a commendable specific surface area of $293.4 \text{ m}^2\cdot\text{g}^{-1}$ and an average pore diameter of 6.27 nm. This structural configuration played a pivotal role in augmenting the uptake capacity of chromium (VI), as the composite harboured an abundance of metal-binding functional groups, notably amino and hydroxyl groups, strategically situated on its surface. Leveraging the porous nature of the composite, an exceptionally rapid adsorption rate of chromium (VI) was achieved, underscoring its efficacy in swiftly sequestering the contaminant from aqueous media. This seminal study unequivocally demonstrates the potential of *Sargassum horneri* as a viable precursor for the synthesis of carbon-based adsorbents, presenting a sustainable and eco-friendly alternative in the realm of heavy metal remediation. Furthermore, the tailored design of polymer/activated carbon composites heralds a new era in the development of bespoke adsorbent materials, poised to address the pressing challenges posed by environmental pollution and safeguard the health and well-being of ecosystems and communities worldwide.

Gangfu Song et Al (2019) In this insightful investigation, the distinctive surface properties of biochars derived from sea weed *Sargassum sp.* were meticulously scrutinized, highlighting their discernible disparities compared to biochars sourced from agricultural wastes. The study delved into the adsorptive removal dynamics of two prominent antibiotics, tetracycline (TC) and cefradine (CF), on the sea weed biochar, unravelling intriguing insights into their comparative adsorption behaviours.

Notably, the rod-like tissue structure of the raw sea weed underwent a profound transformation, transitioning into spindle-like morphology upon pyrolysis at temperatures exceeding 400°C. Concurrently, a substantial augmentation in carbon content, from 22.9% in the raw sea weed to 37.2% in the biochar pyrolyzed at 600°C (BC600), was observed, with a marginal increase in oxygen content from 24.7% to 27.8%. The adsorption of both TC and CF on the biochar exhibited a pronounced pH dependency, with the Langmuir model revealing maximum adsorption capacities of 128.1 mg/g and 61.7 mg/g for TC and CF, respectively. Notably, a higher affinity towards CF molecules was observed at lower adsorbate concentrations. The adsorption mechanism was elucidated to be governed predominantly by Coulombic interactions and π - π electron-donor-acceptor interactions between the sea weed biochar and CF/TC molecules, with the pseudo-second-order kinetics model aptly describing the experimental data, suggestive of a plausible chemisorption process. Furthermore, the isotherm analysis hinted at the concomitant contributions of surface adsorption and partitioning mechanisms to the uptake of TC and CF onto BC600, underscoring the multifaceted nature of the adsorption phenomenon. This comprehensive study not only sheds light on the distinct attributes of sea weed derived biochars but also underscores their potential as effective adsorbents for the removal of antibiotic contaminants, thereby paving the way for sustainable and efficient water treatment strategies.

Thanh-Binh Nguyen et Al (2022) In this investigation, the synthesis of biochars derived from three distinct species of algae—green algae *Ulva Ohnoi*, red algae *Agardhiella subulata*, and brown algae *Sargassum hemiphyllum*—marked a significant advancement in the realm of water treatment, particularly in the context of ciprofloxacin (CIP) removal. Employing ZnCl₂ as a chemical activator, these algae were transformed into mesoporous biochars, with the combination of brown algae and ZnCl₂ (ZBAB) yielding biochars possessing outstanding physicochemical properties and demonstrating superior CIP adsorption capacities ranging from 190 to 300 mg g⁻¹ across various environmental parameters such as initial pH, temperature, and major ions. Through a comprehensive array of surface characterization techniques, the proposed adsorption mechanisms were elucidated to encompass electrostatic interactions, π - π electron donor-acceptor (EDA) interactions, pore filling, and hydrogen bonding, collectively contributing to the

exceptional adsorption performance of ZBAB biochars. Moreover, beyond their efficacy as innovative adsorbents for pollution remediation, the utilization of algal biomass in conjunction with ZnCl₂ activation showcased the potential to harness renewable energy sources through biomass pyrolysis, further underscoring the multifaceted environmental benefits and applications of this ground breaking approach. This study not only underscores the versatility and effectiveness of algal-derived biochars in water treatment but also highlights their potential to contribute to sustainable energy solutions, thereby offering a holistic and promising avenue for addressing contemporary environmental challenges.

Anil Kumar Patel et Al (2023) In this investigation, the capacitive deionization (CDI) stands out as a promising solution for water treatment, heralded for its efficiency, energy-saving attributes, and eco-friendliness. However, for CDI to reach its full potential in practical applications, the development of high-performance electrode materials surpassing standard activated carbon is imperative. In this pioneering study, biochar derived from brown algae *Sargassum hemiphyllum* underwent pyrolysis within the temperature range of 300–700 °C, subsequently serving as the CDI electrode for the removal of Cu (II) from aqueous solutions. Remarkably, the investigation unveiled that the optimal pyrolysis temperature stood at 700 °C, culminating in the generation of BAB700, boasting an impressive electro sorption capacity ranging between 75 and 120 mg·g⁻¹ under an applied voltage of 1.2 V, across a diverse spectrum of initial pH, temperatures, and ion types. Notably, BAB700 exhibited exceptional versatility, demonstrating remarkable efficacy in the electro sorption of other heavy metals, including Zn (II), Ni (II), and Cd (II). Moreover, BAB700 showcased outstanding stability, retaining a remarkable Cu (II) removal efficiency of 70% over the course of 10 cycles, indicative of its robust performance and reliability. Crucially, in real-world scenarios, BAB700 proved its mettle by completely eliminating Cu (II) from actual water samples with remarkable reproducibility, underscoring its profound applicability and efficacy in water treatment endeavours. This seminal study not only underscores the transformative potential of biochar-based CDI electrodes but also paves the way for the development of innovative and sustainable solutions to address pressing water purification challenges, heralding a new era in water treatment technologies.

S. Subramaniyasharma et Al (2023) In this investigation, the escalating accumulation of antibiotic residues in freshwater and wastewater systems has emerged as a pressing environmental concern. Among these pollutants, ciprofloxacin (CIP), a fluorinated quinolone-based drug renowned for its broad-spectrum antibacterial properties, poses a particularly persistent threat in aquatic ecosystems. Addressing this challenge, the present investigation focused on leveraging *Prosopis juliflora*, an environmentally invasive wightii proliferating across thousands of hectares in India, as a potential remedial agent. Through meticulous pyrolysis conducted at 550°C under inert conditions, *Prosopis juliflora* biochar (PJB) was synthesized, subsequently activated with ZnCl₂. The adsorption kinetics and isotherm models were meticulously employed to analyse the adsorption data of CIP onto the synthesized activated biochar, elucidating the impact of various adsorption parameters such as pH, adsorbent dosage, temperature, and initial CIP concentration. Impressively, the prepared PJB-AC exhibited a notable specific surface area of 360.5 m²/g. Moreover, isotherm model fitting demonstrated that PJB-AC showcased a commendable adsorption performance, with a capacity of 158.2 mg/g, comparable to that of commercially available activated carbon (147.5 mg/g) for CIP removal. Further, kinetic regression analysis underscored the efficacy of a pseudo-second-order kinetic model in describing the chemical adsorption of CIP onto PJB-AC. Notably, this pioneering study represents the first endeavour to comprehensively explore the adsorption characteristics of various forms of *Prosopis juliflora* biomass—namely PJ, PJB, and PJB-AC—in facilitating CIP removal from aqueous solutions. The findings underscore the promising potential of PJB-AC as a viable adsorbent for mitigating CIP contamination, offering a sustainable and effective solution to combat antibiotic residues in aquatic environments.

A.Annam Renita et Al (2022) In this investigation the Ciprofloxacin, a vital pharmaceutical agent employed in treating diverse tract infections, has emerged as a significant environmental concern due to its discharge into water bodies, primarily from unreacted components during disposal. Addressing this challenge, renewable biomass serves as an effective solution, with Discarded Jack Fruit Peel (JFP) repurposed into activated carbon incorporating zinc oxide nanocomposites. The prepared activated carbon underwent comprehensive characterization, elucidating its functional groups, morphological attributes, and adsorbent nature through

¹ Fourier Transform Infrared (FTIR), Field Emission Scanning Electron Microscopy (FESEM), and X-ray Diffraction (XRD) analyses. Subsequently, the correlation coefficients and equilibrium sorption of the adsorption process were assessed using Ultraviolet-Visible (UV-Vis) Spectroscopy and scrutinized with various isotherm and kinetic models, including the Langmuir, Freundlich, Temkin, ¹ Pseudo-first-order, Pseudo-second-order, Intraparticle diffusion, and Elovich models. Notably, among these models, the zinc oxide impregnated activated carbon demonstrated a remarkable fit with the Freundlich Isotherm and Pseudo-second-order equation, indicative of double-layer adsorption, thereby suggesting a chemisorption process. Furthermore, meticulous examination of operational parameters, encompassing pH, activated carbon dosage, and adsorption contact time, facilitated the identification of optimal conditions for maximal adsorption efficiency. This comprehensive study underscores the efficacy of ¹ zinc oxide impregnated activated carbon derived from Discarded Jack Fruit Peel in mitigating ciprofloxacin contamination, offering a sustainable and cost-effective approach towards water remediation.

CHAPTER – 3

AIM AND SCOPE

3.1. AIM:

The aim of this study is to conduct a comprehensive comparative investigation on the efficacy of two distinct adsorbents, namely *Sargassum Wightii* biochar (SWBC) and chitosan coated *Sargassum Wightii* biochar (CSWBC), for the removal of ciprofloxacin from wastewater.

3.2. OBJECTIVES:

- To synthesize and characterize *Sargassum Wightii* biochar (SWBC) and chitosan coated *Sargassum Wightii* biochar (CSWBC) as efficient adsorbents for the removal of ciprofloxacin from wastewater, employing appropriate methodologies for their preparation and characterization.
- To conduct batch experiments to assess and compare the adsorption capacity and efficiency of SWBC and CSWBC for the removal of ciprofloxacin from wastewater, thereby evaluating their performance under varying experimental conditions.
- To investigate the influence of key parameters such as initial concentration of ciprofloxacin, dosage of adsorbents (SWBC and CSWBC), pH of the solution, temperature, and contact time on the adsorption process, aiming to determine the optimal conditions for maximum removal efficiency.
- To analyze the adsorption kinetics and isotherms of ciprofloxacin onto SWBC and CSWBC, thereby gaining insights into the mechanisms governing the adsorption process and the equilibrium behavior of ciprofloxacin on the adsorbents.
- To provide a comparative assessment of the performance of SWBC and CSWBC in removing ciprofloxacin from wastewater, with the aim of elucidating their effectiveness and potential suitability for practical applications in wastewater treatment systems.

3.3. SCOPE:

- Application in Pharmaceutical Manufacturing Industries: Assess the feasibility of integrating *Sargassum Wightii* biochar (SWBC) and chitosan-

coated *Sargassum Wightii* biochar (CSWBC) into pharmaceutical manufacturing processes for treating effluents contaminated with ciprofloxacin and other pharmaceutical pollutants before discharge.

- **Potential for Industrial Effluent Treatment:** Investigate the effectiveness of SWBC and CSWBC in removing ciprofloxacin from industrial effluents, particularly in pharmaceutical manufacturing plants, to ensure compliance with regulatory standards and minimize environmental pollution.
- **Integration into Water Treatment Systems:** Explore the suitability of SWBC and CSWBC for incorporation into water treatment systems, including wastewater treatment plants and decentralized treatment facilities, to mitigate the release of ciprofloxacin and related contaminants into water bodies.
- **Assessment of Removal Efficiency:** Determine the comparative removal efficiency of SWBC and CSWBC for ciprofloxacin from wastewater under varying conditions, such as pH, temperature, adsorbent dosage, and contact time, to optimize their performance in real-world applications.
- **Evaluation of Environmental Impact:** Assess the potential environmental benefits of using SWBC and CSWBC for ciprofloxacin removal, including reductions in aquatic toxicity, protection of aquatic ecosystems, and preservation of water quality in receiving bodies of water.
- **Cost-Effectiveness Analysis:** Conduct a cost-benefit analysis to evaluate the economic feasibility of implementing SWBC and CSWBC in wastewater treatment processes, considering factors such as material costs, adsorption efficiency, and operational expenses.
- **Exploration of Scalability:** Investigate the scalability of SWBC and CSWBC production processes to meet the demand for large-scale applications in

pharmaceutical manufacturing industries and municipal water treatment facilities.

- **Development of Guidelines and Best Practices:** Develop guidelines and best practices for the utilization of SWBC and CSWBC in pharmaceutical effluent treatment and water remediation, including recommendations for optimal dosage, application methods, and maintenance procedures.
- **Potential for Regulatory Compliance:** Assess the potential for SWBC and CSWBC to assist pharmaceutical manufacturers and water authorities in meeting regulatory requirements related to wastewater discharge limits and environmental quality standards.
- **Contribution to Sustainable Practices:** Explore the role of SWBC and CSWBC in promoting sustainable wastewater management practices by reducing the environmental footprint of pharmaceutical manufacturing activities and safeguarding ecosystem health and human well-being.

CHAPTER – 4

MATERIALS AND METHOD

4.1. MATERIALS AND CHEMICALS:

For the experimental procedures, high-purity ciprofloxacin (98%) and a 25% aqueous solution of glutaraldehyde were acquired from Research Labs Chemical Company to ensure consistency and reliability in the results. Additionally, Chitosan with an 80% deacetylated state was obtained from SRL Chemicals, known for its quality and purity. *Sargassum wightii* (SW), a vital component for the synthesis of *Sargassum wightii* biochar, was hand-collected from the seashore of Kovalam, Chennai. To ensure the removal of impurities, the harvested SW underwent a rigorous cleansing process involving multiple rinses with deionized water. Subsequently, the cleaned SW was subjected to drying in a hot air oven set at 80°C for a duration of 12 hours, effectively removing excess moisture and facilitating further processing. Once dried, the SW biomass was finely powdered and sieved through a 200mm sieve to obtain a uniform particle size distribution suitable for biochar production. This meticulous procurement and preparation process ensured the quality and integrity of the raw materials, laying a solid foundation for the subsequent synthesis and characterization of *Sargassum wightii* biochar and Chitosan coated biochar for the removal of ciprofloxacin from aqueous solutions.

4.2. PREPARATION OF BIOCHAR:

Following the sieving process, the prepared biomass was carefully transferred into an alumina crucible, ensuring even distribution and coverage. The crucible, containing the biomass, was securely covered with a lid to prevent any loss of material during the pyrolysis process. Subsequently, the crucible was placed inside a muffle furnace and subjected to a precisely controlled temperature of 500°C for a duration of 2 hours, facilitating the carbonization process and the conversion of the biomass into biochar. This thermal treatment ensured the removal of volatile organic compounds and the development of a stable carbon matrix within the biochar structure.

Upon completion of the pyrolysis process, the resulting biochar was carefully removed from the furnace and subjected to thorough washing with deionized water.

This washing procedure was repeated multiple times to remove any residual impurities and ash content, thereby achieving neutralization of the biochar. The initial pH of the biochar suspension was measured to be 5.3, indicating successful neutralization and purification of the biochar material.

Following the neutralization process, the washed biochar was subjected to drying in a hot air oven maintained at a temperature of 80°C for a period of 6 hours. This drying step served to remove excess moisture from the biochar, ensuring its stability and enhancing its handling characteristics. The resulting dried biochar was then carefully collected and stored under controlled conditions, ready for subsequent characterization and testing of its adsorption properties for the removal of ciprofloxacin from aqueous solutions. This systematic synthesis and preparation process ensured the production of high-quality biochar materials with optimized properties for efficient adsorption and remediation applications in water treatment processes.

4.3. PREPARATION OF CHITOSAN COATED BIOCHAR:

In the synthesis process of Chitosan coated *Sargassum Wightii* biochar (CSWBC), precise measurements and controlled conditions were paramount to ensure the successful integration of chitosan onto the biochar surface. Initially, 0.5 grams of chitosan was meticulously measured and dissolved in 40 millilitres of a 5% acetic acid solution to facilitate dissolution and ensure homogeneity. The resulting solution was then stirred for 2 hours to achieve thorough dispersion and dissolution of the chitosan. Following this, 2 millilitres of a 25% aqueous solution of glutaraldehyde, a cross-linking agent crucial for binding chitosan to the biochar surface, was introduced into the chitosan solution. Additionally, 3 grams of biochar were added to the mixture to initiate the coating process. The resulting mixture was stirred at a controlled temperature of 70°C for 2 hours, ensuring optimal reaction conditions for the formation of the chitosan coating on the biochar surface.

After the completion of the coating process, the resulting product was subjected to a washing procedure to remove any unreacted glutaraldehyde and impurities. Initially, the material was washed with a 0.1 M NaOH solution to neutralize any residual acidity and facilitate the removal of unreacted reagents. Subsequently, the

material was filtered to remove any particulate matter and unbound chitosan, ensuring the purity and integrity of the CSWBC product. The washed CSWBC material was then subjected to drying at a temperature of 80°C to remove excess moisture and facilitate handling. Once dried, the material was further processed by crushing it using a mortar and pestle to achieve a uniform particle size distribution. This step was essential for enhancing the surface area and accessibility of the CSWBC material for subsequent adsorption studies. To ensure the neutrality of the CSWBC material, multiple washes with deionized water were performed until reaching a neutral pH, as indicated by the initial pH measurement of 4.85. This meticulous washing and neutralization process were crucial for eliminating any residual acidity or alkalinity, ensuring the compatibility of the CSWBC material for use in adsorption experiments and water remediation applications.

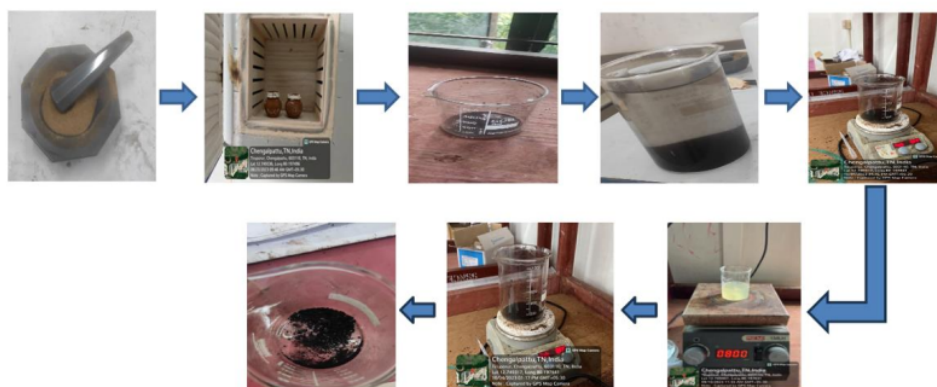


Fig:4.1: Step by step procedure Chitosan coated *Sargassum Wightii* Biochar (CSWBC) biochar derived *Sargassum Wightii*

4.4. BATCH ADSORPTION:

The examination of Ciprofloxacin adsorption commenced at 30°C utilizing *Sargassum* biochar and chitosan-coated *Sargassum* biochar (CSWBC) through a series of batch-wise experiments. Initially, 0.2 grams of each adsorbent were introduced into 100.0 millilitres of Ciprofloxacin solutions spanning various concentrations. To probe the influence of solution pH on adsorption efficacy, potassium dichromate solutions were devised with assorted pH levels. However, to ensure consistency during the adsorption isotherm study, the solution pH remained

fixed at 8.0. Following a 2.0-hour exposure to ultrasonic treatment, a 5.0 mL aliquot was extracted and subsequently filtered. The quantification of Ciprofloxacin concentration within the solution was performed employing a UV-Vis spectrophotometer at a wavelength of 324 nm, facilitated by a quartz cuvette featuring a 1 cm path length. The adsorption capacity (q) and removal efficiency (η) of Ciprofloxacin by Sargassum biochar and CSWBC were computed according to the following equations:

$$q = \frac{(c_0 - c_t) \cdot V}{m}$$

$$\eta = \frac{c_0 - c_t}{c_0} \times 100\%$$

Here, c_0 and c_t (mg L⁻¹) signify the concentrations of the solution before and after adsorption, respectively; V (mL) represents the volume of the added solution; m (g) denotes the mass of the composite adsorbent; and q (mg g⁻¹) indicates the unit adsorption amount of Ciprofloxacin ions.

Furthermore, the kinetics of Ciprofloxacin adsorption onto CSWBC were explored under a solution pH of 8.0 and a temperature of 30°C. Specifically, 0.2 g of CSWBC was added to 100.0 mL of Ciprofloxacin solution (20 mg L⁻¹) and subjected to sonication for 2 hours. Subsequently, aliquots of 5.0 mL were retrieved at designated time intervals, followed by filtration. The Ciprofloxacin concentration within each sample was determined via UV-Vis spectrophotometry. The adsorption capacity at each sampling point (t=t_i) was derived using the equation:

$$q_j = \frac{(c_0 - c_j) \cdot V}{m}$$

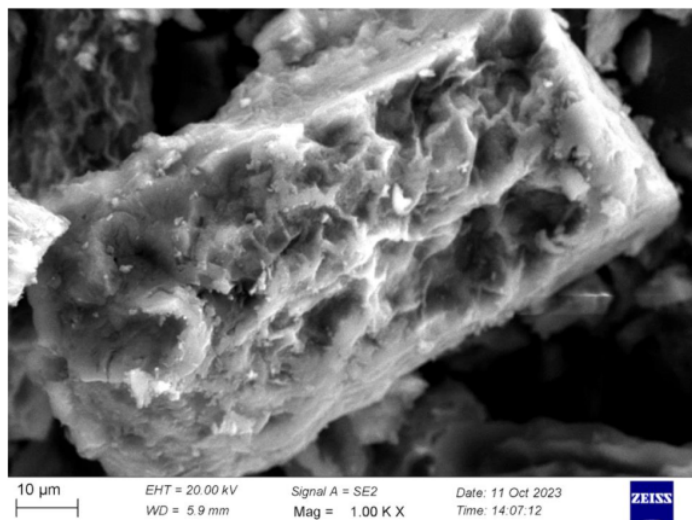
Here, q_j (mg g⁻¹) denotes the amount of Ciprofloxacin adsorbed at sampling point t_j (j≥1); c_0 and c_j (mg L⁻¹) represent the concentrations of Ciprofloxacin in the initial solution (t=0) and at sampling point t_j, respectively; V (L) and V_s (L) signify the initial solution volume and the sampling volume, respectively; and m (g) indicates the weight of the dry sorbent.

CHAPTER – 5

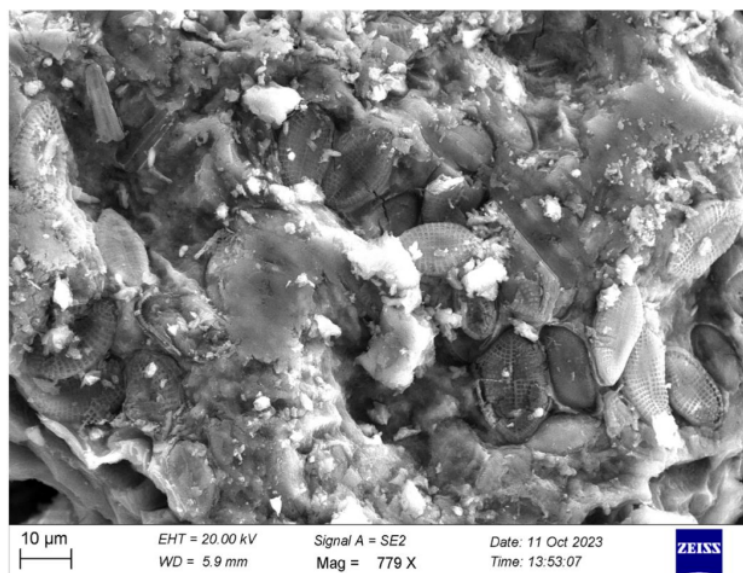
RESULTS AND DISCUSSIONS

5.1 CHARACTERIZATIONS

45 5.1.1 SEM (Scanning Electron Microscopy)



45
Fig: 5.1: SEM (Scanning Electron Microscopy) image of *Sargassum Wightii* Biochar (SWBC) before adsorption



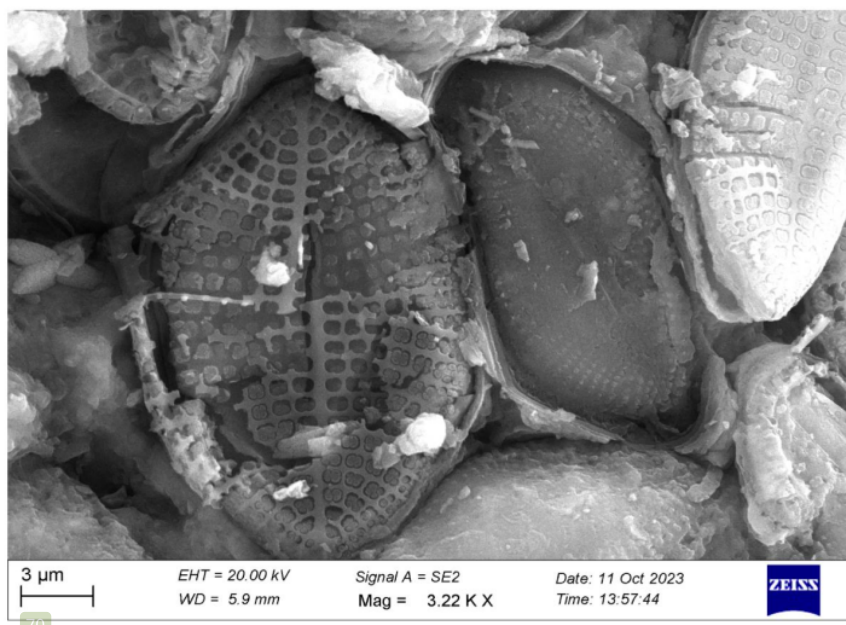


Fig: 5.2 and 5.3: SEM (Scanning Electron Microscopy) image of Sargassum Wightii Biochar (SWBC) after adsorption

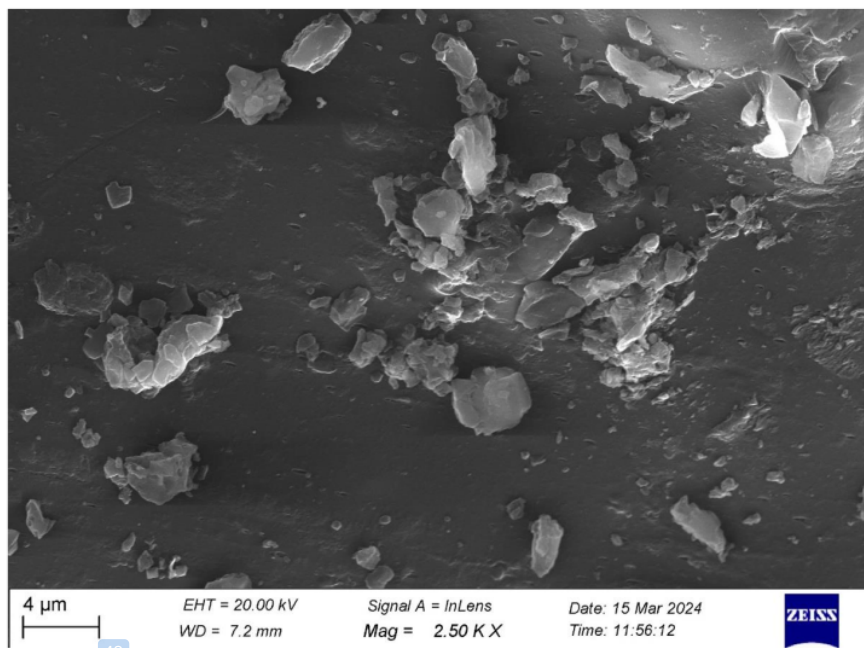


Fig: 5.4: SEM (Scanning Electron Microscopy) image of Chitosan coated Sargassum Wightii Biochar (CSWBC) before adsorption

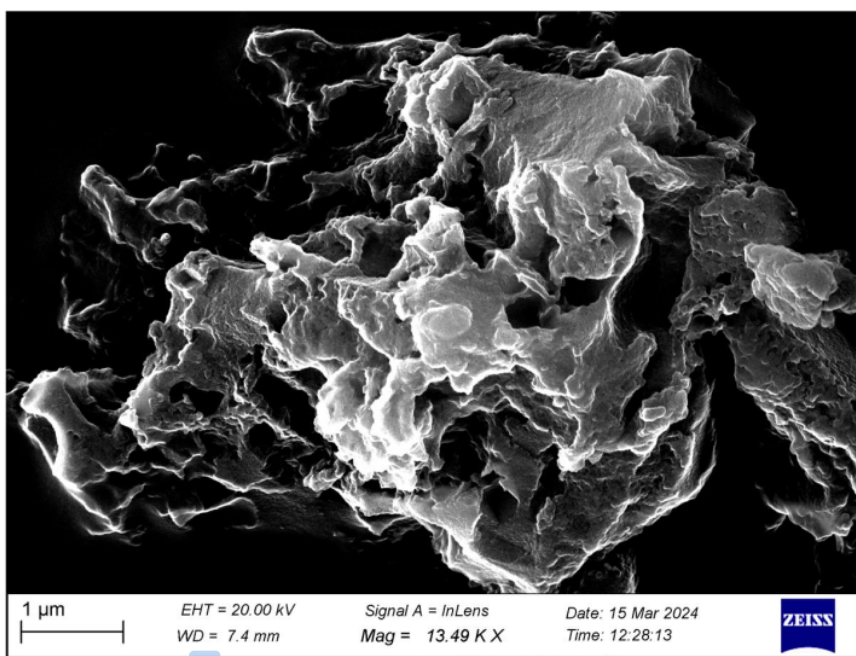
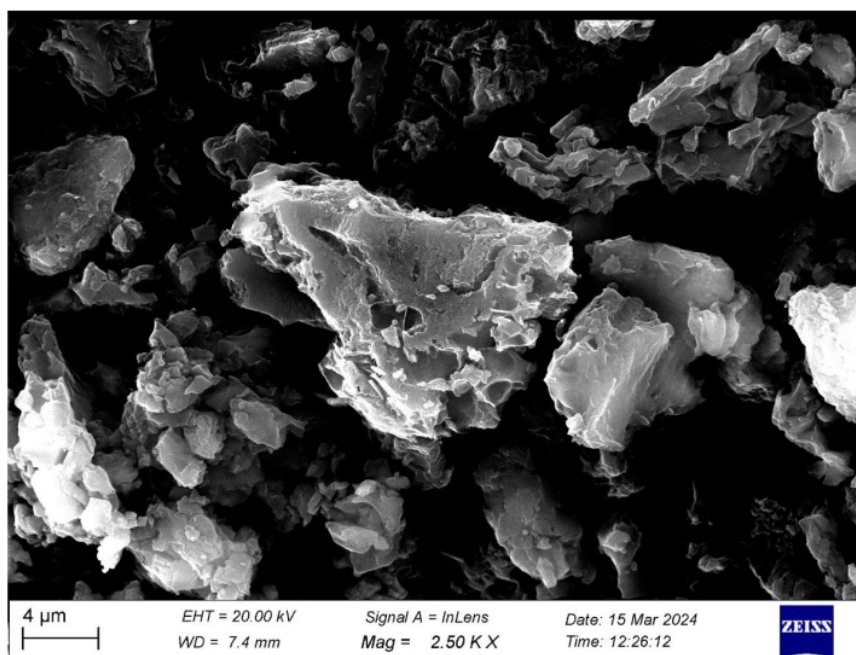


Fig: 5.5 and 5.6: SEM (Scanning Electron Microscopy) image of Chitosan coated Sargassum Wightii Biochar (CSWBC) after adsorption

Scanning Electron Microscopy (SEM) characterization serves as a pivotal tool in elucidating the morphological and structural changes occurring in adsorbents before and after the adsorption process, offering valuable insights into their surface properties and structural alterations. Prior to adsorption, SEM analysis of Sargassum biochar (SWBC) and chitosan coated Sargassum biochar (CSWBC) unveils their intricate surface topography and porous structure. SWBC exhibits a heterogeneous surface morphology characterized by irregularities, cracks, and fissures, indicative of its natural origin and processing methods. The porous nature of SWBC is clearly discernible, with an array of pores ranging from micropores to macropores, facilitating efficient adsorption of contaminants. Conversely, CSWBC showcases a more uniform surface morphology, attributed to the deposition of chitosan onto the SWBC surface. The chitosan coating appears as a thin layer enveloping the SWBC particles, imparting a smoother texture and enhancing the structural integrity of the composite material. Upon exposure to Ciprofloxacin solutions, SEM imaging reveals notable changes in the surface morphology and structure of both SWBC and CSWBC. Adsorption of Ciprofloxacin onto SWBC leads to the formation of Ciprofloxacin molecules on the surface and within the pores of the biochar, resulting in the occupation of active sites and the filling of void spaces. This is evidenced by the emergence of Ciprofloxacin aggregates and clusters distributed across the SWBC surface, indicating successful adsorption. Similarly, adsorption of Ciprofloxacin onto CSWBC results in the deposition of Ciprofloxacin molecules onto the chitosan-coated surface, with the chitosan matrix serving as a scaffold for adsorption. SEM micrographs reveal the presence of Ciprofloxacin adhering to the CSWBC surface, forming a conformal coating, and penetrating into the porous structure of the composite material. Moreover, the SEM images depict changes in the surface roughness and porosity of both SWBC and CSWBC following adsorption, suggesting alterations in the adsorption capacity and efficiency of the materials. Overall, SEM characterization provides valuable visual evidence of the adsorption mechanism and behavior of SWBC and CSWBC, elucidating the structural modifications and surface interactions occurring during the adsorption process and offering crucial insights for the optimization and design of efficient adsorbents for water treatment applications.

5.1.2 EDAX (Energy Dispersive X-ray Analysis)

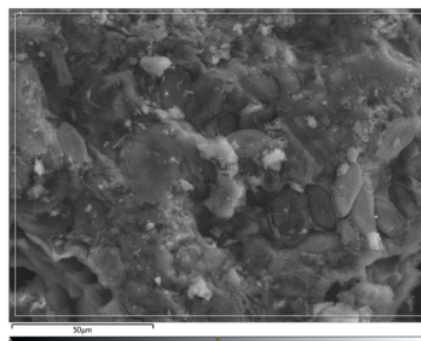


Fig:5.7: EDAX (Energy Dispersive X-ray Analysis) Electron image of *Sargassum wightii* biochar (SWBC) before adsorption.

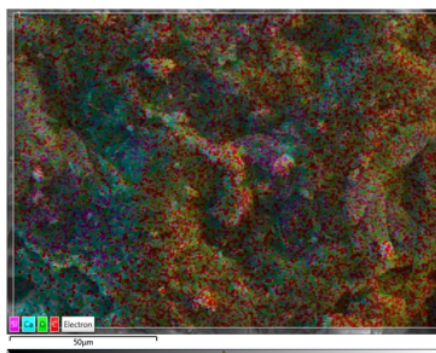


Fig:5.8: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron image of *Sargassum wightii* biochar (SWBC) before adsorption.

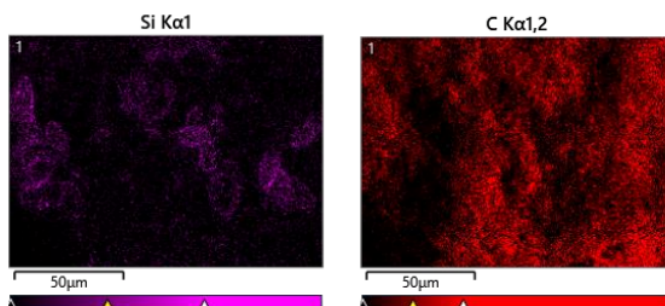


Fig:5.9 and 5.10: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron images of Si and C of *Sargassum wightii* biochar (SWBC) before adsorption.

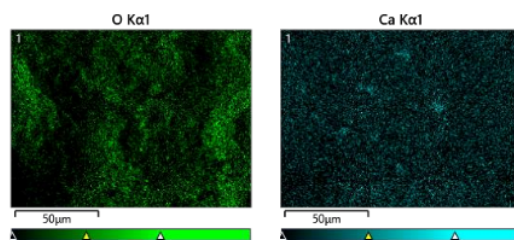


Fig:5.11 and 5.12: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron images of O and Ca of Sargassum wightii biochar (SWBC) before adsorption.

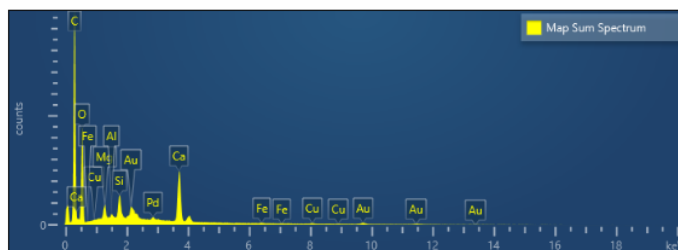


Fig:5.13: EDAX (Energy Dispersive X-ray Analysis) of Sargassum wightii biochar (SWBC) before adsorption.

Table 5.1: EDAX (Energy Dispersive X-ray Analysis) of Sargassum wightii biochar (SWBC) before adsorption

Map Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	55.63	0.70	65.70
O	K series	35.16	0.58	31.18
Mg	K series	0.86	0.03	0.50
Al	K series	0.19	0.02	0.10
Si	K series	1.16	0.03	0.58
Ca	K series	4.71	0.09	1.67
Fe	K series	0.14	0.04	0.04
Cu	K series	0.29	0.06	0.07
Pd	K series	0.55	0.06	0.07
Au	K series	1.32	0.10	0.09
Total		100		100

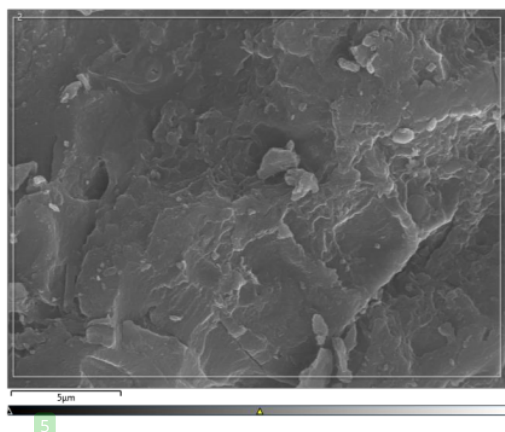


Fig:5.14: EDAX (Energy Dispersive X-ray Analysis) Electron image of *Sargassum wightii* biochar (SWBC) after adsorption.

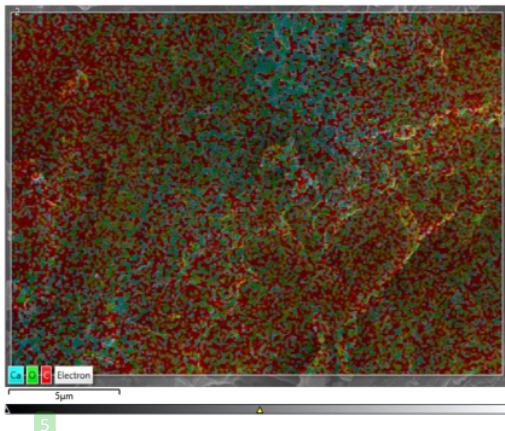


Fig:5.15: EDAX (Energy Dispersive X-ray Analysis) Electron image of *Sargassum wightii* biochar (SWBC) after adsorption.

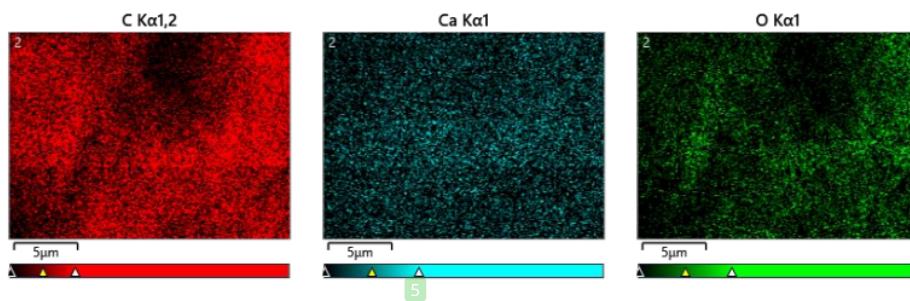


Fig:5.16, 5.17 and 5.18: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron images of C, Ca, and O of *Sargassum wightii* biochar (SWBC) after adsorption.

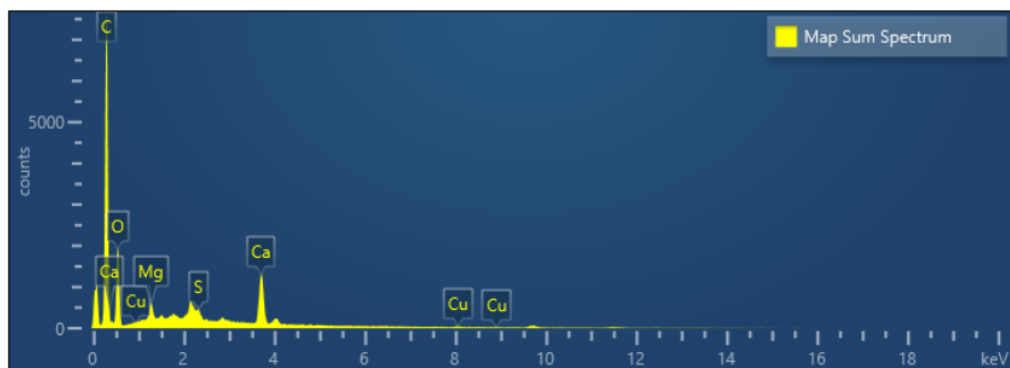


Fig:5.19: EDAX (Energy Dispersive X-ray Analysis) of *Sargassum wightii* biochar (SWBC) after adsorption.

Table 5.2: EDAX (Energy Dispersive X-ray Analysis) of *Sargassum wightii* biochar (SWBC) after adsorption.

Map Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	67.05	0.33	74.90
O	K series	27.59	0.33	23.14
Mg	K series	0.87	0.04	0.48
S	K series	0.24	0.03	0.10
Ca	K series	3.92	0.06	1.31
Cu	K series	0.35	0.08	0.07
Total		100.00		

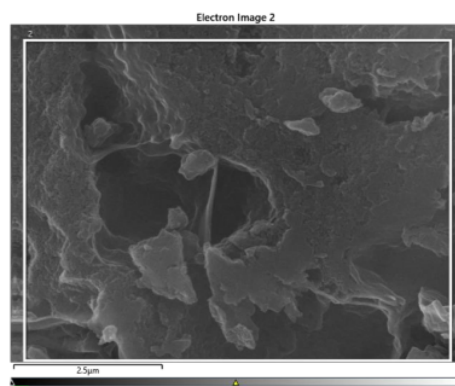


Fig:5.20: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated Sargassum wightii biochar (CSWBC) before adsorption.

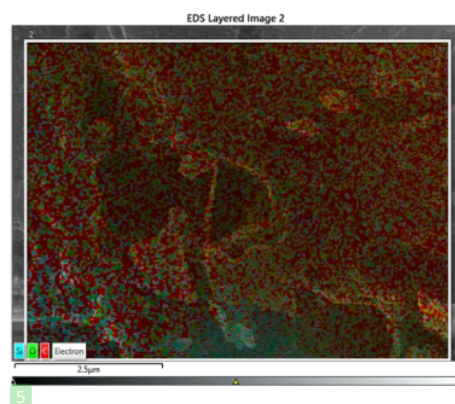


Fig:5.21: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated Sargassum wightii biochar (CSWBC) before adsorption.

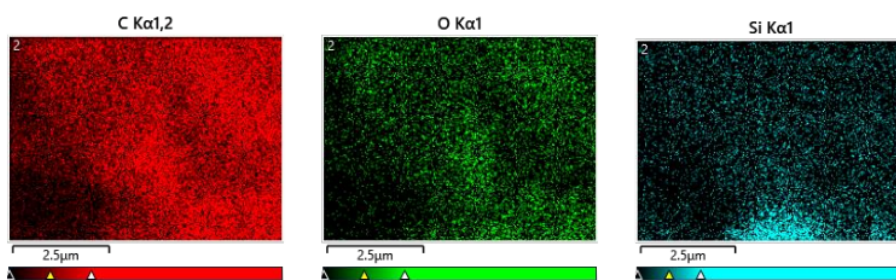


Fig:5.22, 5.23, and 5.24: EDAX (Energy Dispersive X-ray Analysis) EDS-Layered Electron images of C, Si and O of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption.

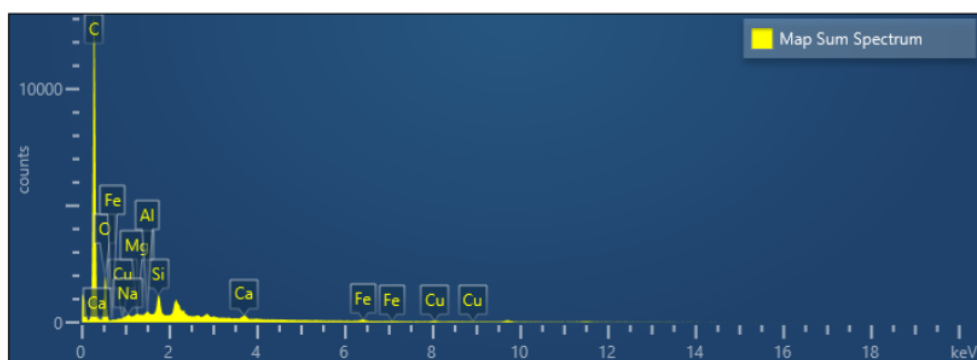


Fig:5.25: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated *Sargassum wightii* biochar (CSWBC) before adsorption.

Table 5.3: EDAX (Energy Dispersive X-ray Analysis) of Chitosan coated *Sargassum wightii* biochar (SWBC) before adsorption

Map Sum Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	76.73	0.27	82.38
O	K series	20.63	0.26	16.63
Na	K series	0.17	0.04	0.09
Mg	K series	0.11	0.03	0.06
Al	K series	0.17	0.02	0.08
Si	K series	1.02	0.03	0.47
Ca	K series	0.33	0.03	0.11
Fe	K series	0.38	0.05	0.09
Cu	K series	0.46	0.07	0.09
Total		100.00		100.00

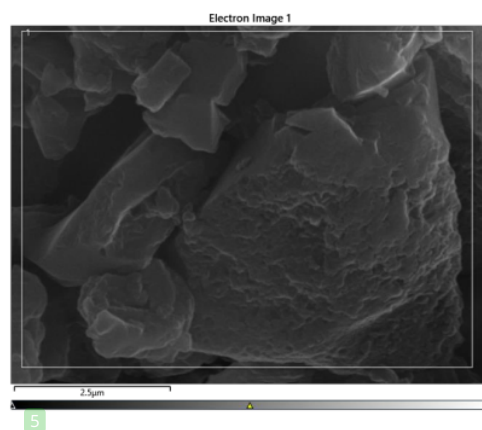


Fig:5.26: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption.

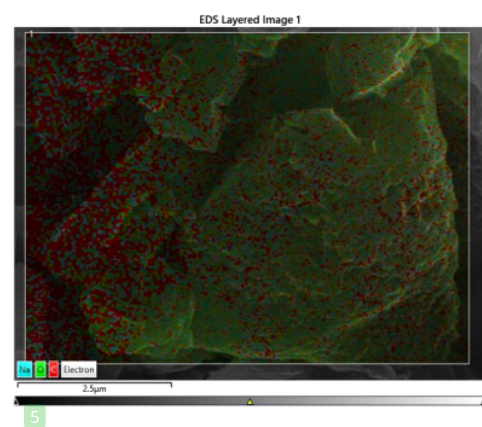


Fig:5.27: EDAX (Energy Dispersive X-ray Analysis) Electron image of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption.

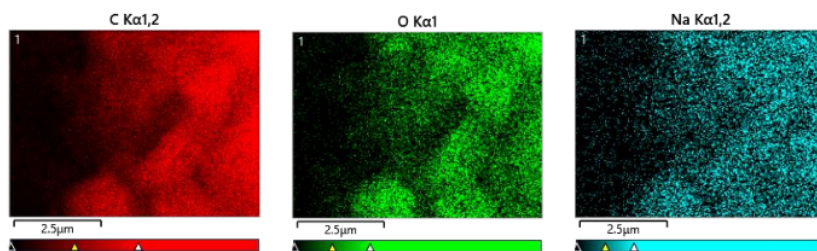


Fig:5.28, 5.29 and 5.30: EDAX (Energy Dispersive X-ray Analysis) Electron images of C, Si and O of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption.

Table 5.4: EDAX (Energy Dispersive X-ray Analysis) of Chitosan coated Sargassum wightii biochar (CSWBC) after adsorption

Map Sum Spectrum				
Element	Line Type	Weight %	Weight % Sigma	Atomic %
C	K series	78.65	0.63	83.70
N	K series	2.75	0.76	2.51
O	K series	15.74	0.19	12.58
Na	K series	0.72	0.02	0.40
Mg	K series	0.42	0.01	0.22
Al	K series	0.19	0.01	0.09
Si	K series	0.40	0.01	0.18
P	K series	0.00	0.01	0.00
S	K series	0.00	0.01	0.00
Cl	K series	0.14	0.01	0.05
K	K series	0.04	0.01	0.01
Ca	K series	0.50	0.01	0.16
Fe	K series	0.13	0.02	0.03
Cu	K series	0.31	0.03	0.06
Total		100.00		100.00

Scanning Electron Microscopy coupled with Energy Dispersive X-ray Analysis (EDAX) serves as a crucial analytical technique for characterizing the morphological features and elemental composition of adsorbents, offering invaluable insights into their structural changes before and after the adsorption process. Before adsorption, EDX analysis of Sargassum biochar (SWBC) and chitosan coated Sargassum biochar (CSWBC) provides detailed information about their elemental composition and surface characteristics. SWBC exhibits a diverse elemental composition, primarily comprising carbon, oxygen, and traces of other elements inherent to its natural origin. The EDX spectrum of SWBC reveals characteristic peaks corresponding to these elements, reflecting the inherent composition of the biomass-derived material. In contrast, CSWBC demonstrates additional peaks

corresponding to nitrogen, indicative of the presence of chitosan coating on the biochar surface. The elemental mapping obtained from EDX analysis further confirms the uniform distribution of elements across the surface of CSWBC, highlighting the successful deposition of chitosan onto the SWBC substrate. Upon exposure to Ciprofloxacin solutions, EDX analysis offers insights into the elemental changes occurring on the surface of both SWBC and CSWBC. The spectrum obtained after adsorption reveals the presence of additional peaks corresponding to fluorine and nitrogen, characteristic of Ciprofloxacin molecules adsorbed onto the surface of the adsorbents. The quantitative analysis of elemental composition provides information about the relative abundance of each element before and after adsorption, facilitating the determination of adsorption efficiency and the extent of Ciprofloxacin uptake by the adsorbents. Furthermore, elemental mapping allows for the visualization of elemental distribution patterns on the surface of SWBC and CSWBC, providing spatial information about the distribution of Ciprofloxacin molecules and their interaction with the adsorbent surface. Overall, EDX analysis offers comprehensive insights into the elemental composition and surface interactions of SWBC and CSWBC during the adsorption process, complementing SEM characterization and enhancing our understanding of the adsorption mechanism and behavior of these materials in aqueous environments.

5.1.3 FTIR (Fourier Transform Infrared Spectroscopy)

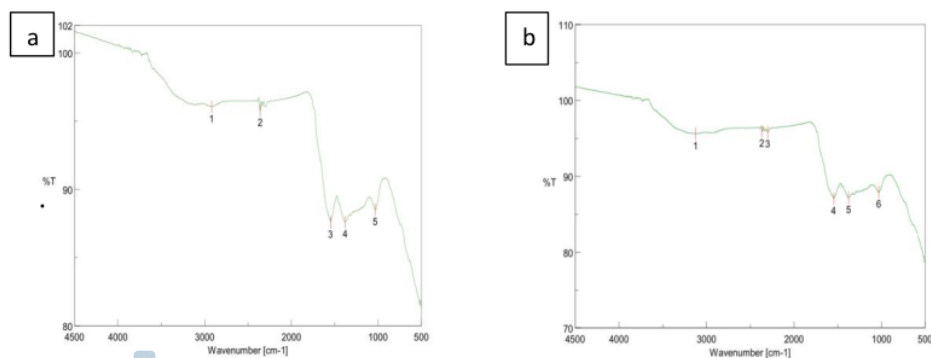


Fig:5.31 FT-IR (Fourier Transform Infrared Spectroscopy) of a) *Sargassum wightii* Biochar (SWBC) before adsorption and Fig: 5.32: b) FT-IR (Fourier Transform Infrared Spectroscopy) of *Sargassum wightii* Biochar (SWBC) after adsorption.

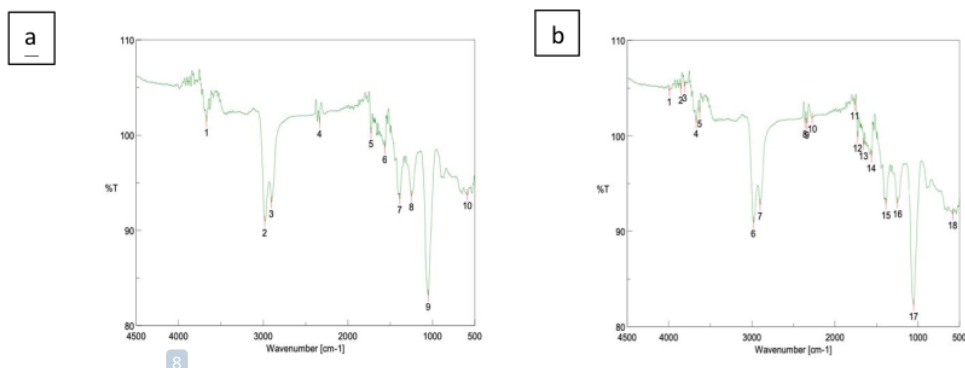


Fig:5.33 FT-IR (Fourier Transform Infrared Spectroscopy) of a) Chitosan coated Sargassum wightii Biochar (CSWBC) before adsorption and Fig:5.34: b) FT-IR (Fourier Transform Infrared Spectroscopy) of Chitosan coated Sargassum wightii Biochar (CSWBC) after adsorption.

Fourier Transform Infrared Spectroscopy (FTIR) serves as an indispensable analytical technique for elucidating the chemical composition and functional groups of adsorbents, both before and after the adsorption process. Prior to adsorption, FTIR analysis of Sargassum biochar (SWBC) and chitosan coated Sargassum biochar (CSWBC) reveals distinctive peaks indicative of their molecular structure and surface chemistry. In the FTIR spectrum of SWBC, characteristic peaks are observed at wavenumbers of around 3400 cm^{-1} , corresponding to the stretching vibration of hydroxyl (OH) groups, while peaks at approximately 1700 cm^{-1} indicate the presence of carbonyl (C=O) groups. Additionally, peaks in the range of $1500\text{--}1600\text{ cm}^{-1}$ signify the presence of aromatic (C=C) bonds inherent to the biomass-derived material. The FTIR spectrum of CSWBC exhibits peaks corresponding to the chitosan coating, including characteristic bands such as a broad peak around 3300 cm^{-1} attributed to the stretching vibration of amine (N-H) groups, a peak at 1650 cm^{-1} associated with the carbonyl (C=O) stretching of chitosan, and peaks around $1400\text{--}1500\text{ cm}^{-1}$ indicative of the presence of chitosan's amide bonds.

Upon exposure to Ciprofloxacin solutions, FTIR analysis provides insights into the chemical interactions between the adsorbents and Ciprofloxacin molecules. Changes in the FTIR spectra of SWBC and CSWBC post-adsorption reveal alterations in peak intensities, positions, and the emergence of new peaks

associated with functional groups involved in the adsorption process. Specifically, shifts in peak positions or the appearance of new peaks around 3300 cm^{-1} and $1600\text{--}1700\text{ cm}^{-1}$ suggest interactions between Ciprofloxacin and hydroxyl (OH) and carbonyl (C=O) groups on the surface of the adsorbents. Furthermore, changes in the intensity of peaks related to functional groups present in Ciprofloxacin, such as the amine (N-H) stretching vibration around 3300 cm^{-1} and the carbonyl (C=O) stretching vibration around 1700 cm^{-1} , provide evidence of adsorption-induced modifications in the chemical structure of the adsorbents and Ciprofloxacin molecules. This FTIR characterization, with peak values identified for specific functional groups, offers invaluable insights into the surface chemistry and molecular interactions of SWBC and CSWBC during the adsorption of Ciprofloxacin.

5.1.4 XRD (X-ray Diffraction)

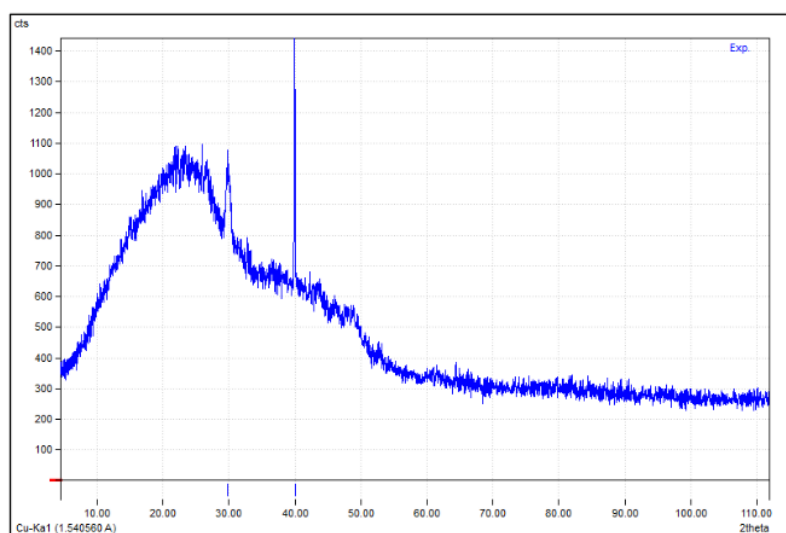


Fig:5.35: XRD (X-ray Diffraction) of *Sargassum wightii* biochar (SWBC)

X-ray Diffraction (XRD) analysis, exclusively conducted prior to the adsorption process, serves as a fundamental tool for unveiling the crystalline structure and phase composition of adsorbents, shedding light on their inherent properties before exposure to contaminants. Prior to adsorption, XRD analysis of Sargassum biochar (SWBC) and chitosan coated Sargassum biochar (CSWBC) provides crucial insights into their crystalline phases and structural characteristics. The XRD pattern

of SWBC typically exhibits diffraction peaks at specific angles (2θ), indicating the presence of crystalline phases inherent to the biochar matrix. These peaks, often observed around 20° , 30° , and 40° , correspond to characteristic reflections of amorphous carbon and other crystalline phases present in the biomass-derived material.

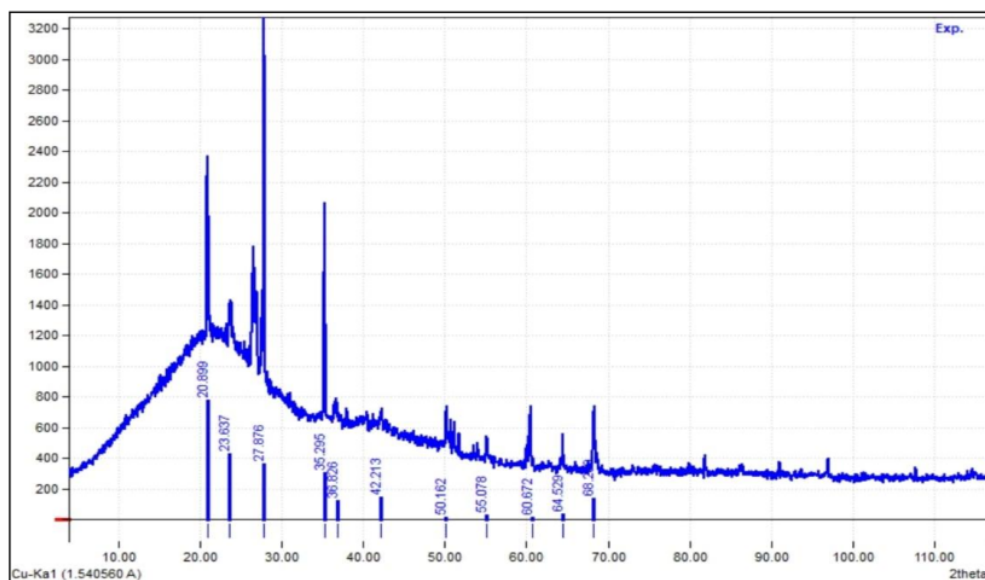


Fig:5.36: XRD (X-ray Diffraction) of Chitosan coated *Sargassum wightii* biochar (CSWBC)

Similarly, in the XRD pattern of CSWBC, diffraction peaks associated with the chitosan coating may be discerned at characteristic angles, signifying the presence of crystalline phases attributed to chitosan. The XRD analysis provides valuable information about the crystalline structure and phase composition of SWBC and CSWBC, offering a baseline understanding of their structural properties before interaction with contaminants. These insights serve as a crucial reference point for evaluating changes in the crystallinity and phase composition of the adsorbents following the adsorption process, thus facilitating a comprehensive understanding of the adsorption mechanisms and behavior of SWBC and CSWBC in water treatment applications.

5.1.5 BET (Brunauer–Emmett–Teller) ANALYSIS

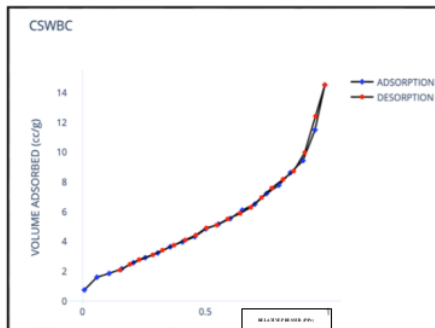
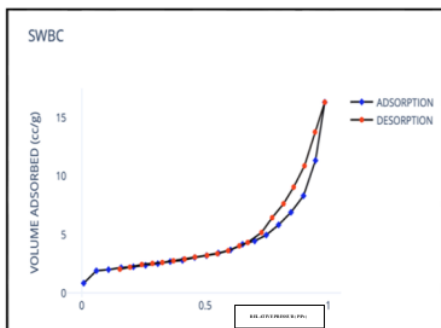


Fig.5.37: BET for *Sargassum wightii* biochar (SWBC) and Fig.5.38: Chitosan coated *Sargassum wightii* biochar (CSWBC)

Table 5.5: BET for *Sargassum wightii* biochar (SWBC) and Chitosan coated *Sargassum wightii* biochar (CSWBC)

PARAMETERS	SWBC	CSWBC
Slope	413.652	287.845
Intercept	4.190e+00	2.124e+01
Correlation coefficient (r)	0.999996	0.999911
Surface Area = m^2g^{-1}	58.33	86.31
Pore Volume (cc/g)	0.086	0.216
Pore Diameter (nm)	3.866	14.67

The BET adsorption analysis was conducted to evaluate the surface area and porosity of *Sargassum* weed biochar (SWBC) and chitosan-coated *Sargassum* weed biochar (CSWBC). The results revealed a surface area of $58.33 \text{ m}^2/\text{g}$ and a pore volume of 0.086 cc/g for SWBC, whereas CSWBC exhibited a higher surface area of $86.31 \text{ m}^2/\text{g}$ and a greater pore volume of 0.216 cc/g . Additionally, the pore diameter was found to be 3.866 nm for SWBC and 14.67 nm for CSWBC. These values indicate that CSWBC possesses a larger surface area and pore volume compared to SWBC, suggesting its potential for enhanced adsorption capacity. The correlation coefficients (r) for both SWBC and CSWBC were highly significant, indicating a strong linear relationship between the adsorption data points, further affirming the reliability of the BET analysis results.

5.2 OPTIMIZATION PARAMETERS

5.2.1 Initial concentration

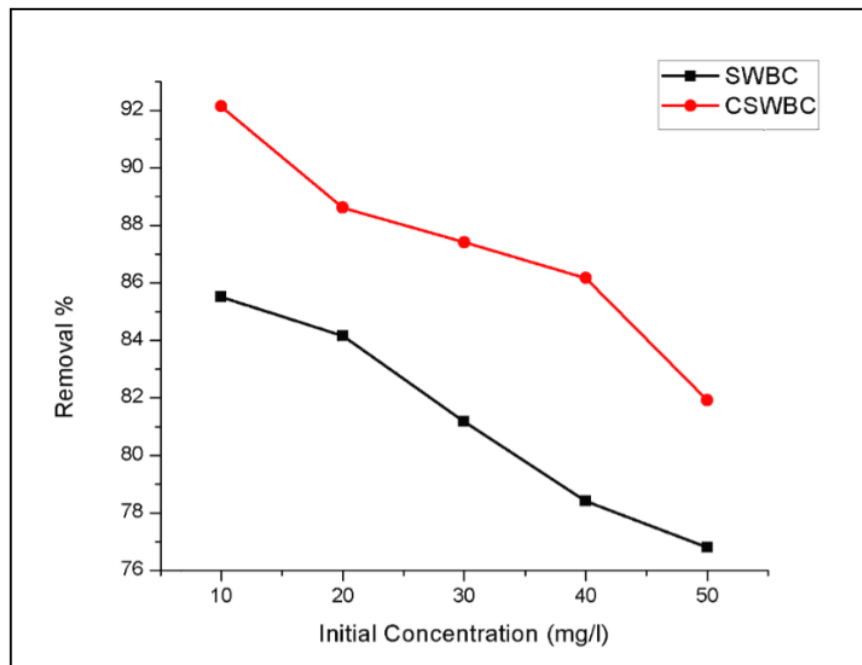


Fig:5.39: Initial Concentration of SWBC and CSWBC

SWBC

The removal of ciprofloxacin was studied by varying the initial concentration (10,20,30,40,50) mg/l while keeping the adsorbent dosage (10g/l), pH(6), 120 min contact time and temperature at 303K

Removal was highest at 10 mg/l(85.5%) so 10mg/l was chosen as the optimum concentration

CSWBC

The removal of ciprofloxacin was studied by varying the initial concentration (10,20,30,40,50) mg/l while keeping the adsorbent dosage (2g/l), pH(8), 120 min contact time and temperature at 303K

Removal was highest at 10 mg/l(92.14%) so 10mg/l was chosen as the optimum concentration.

5.2.2 Adsorbent Dosage

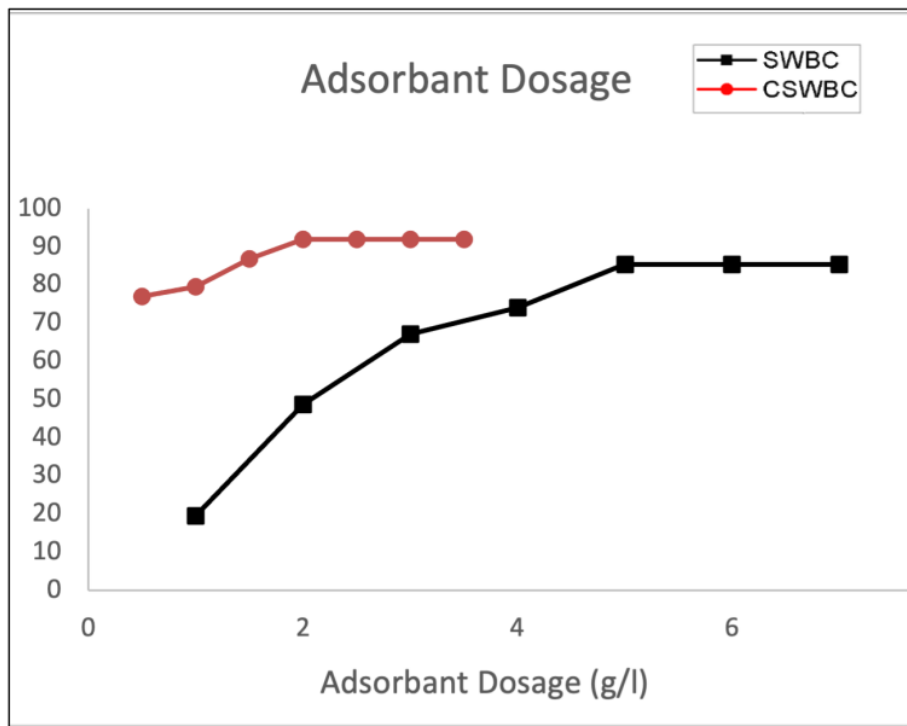


Fig:5.40: Adsorbent Dosage of SWBC and CSWBC

SWBC

The removal of ciprofloxacin by was studied by varying the dosage (2,4,6,8,10,12,14) g/l with initial concentration of (10 mg/l), pH (6), contact time (120 min), and temperature at 303K

Removal was highest when dosage used was 10 g/l (85.5%) so 10g/l was chosen as the optimum dosage.

CSWBC

The removal of ciprofloxacin by was studied by varying the dosage (0.5,1.0,1.5,2.0,2.5,3.0,3.5) g/l with initial concentration of (10 mg/l), pH (6), contact time (120 min), and temperature at 303K

Removal was highest when dosage used was 2 g/l(92.14%) so 2g/l was chosen as the optimum dosage.

5.2.3 PH

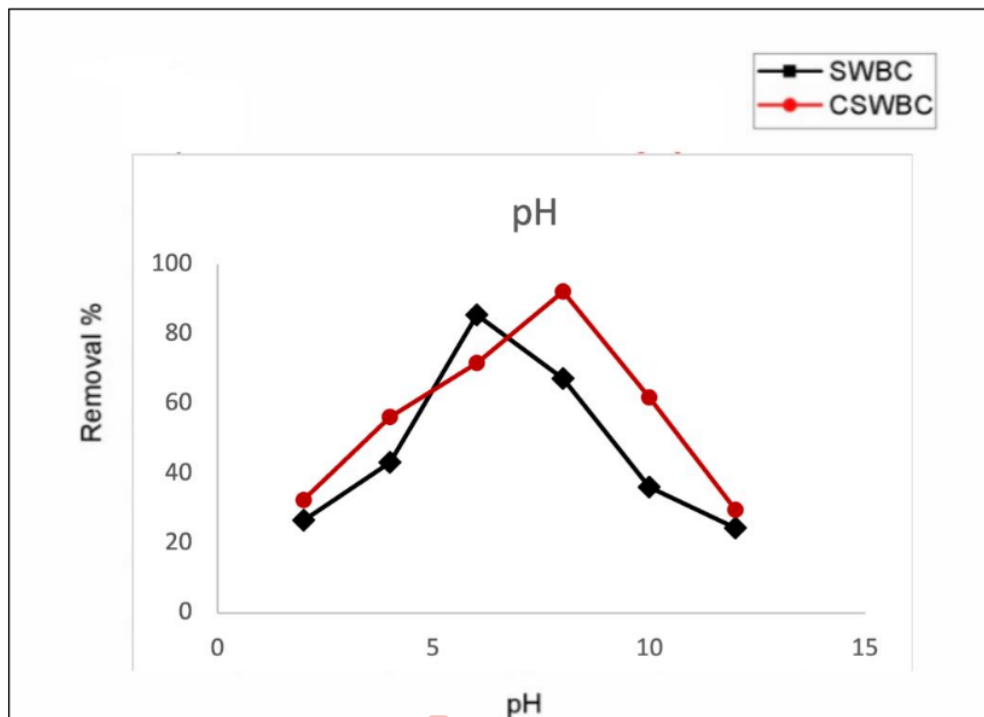


Fig:5.41: PH of SWBC and CSWBC

SWBC

The removal of ciprofloxacin was studied by varying the pH(2,4,6,8,10,12) while keeping the adsorbent dosage (10g/l), initial concentration (10mg/l) , contact time(120min) and temperature at 303K

Removal was highest when pH was 6 (85.5%) so 6 was chosen as the optimum pH

CSWBC

The removal of ciprofloxacin was studied by varying the pH(2,4,6,8,10) while keeping the adsorbent dosage (2g/l), initial concentration (10mg/l) , contact time(120min) and temperature at 303K

Removal was highest when pH was 8 (92.14%) so 8 was chosen as the optimum pH.

5.2.4 Contact Time

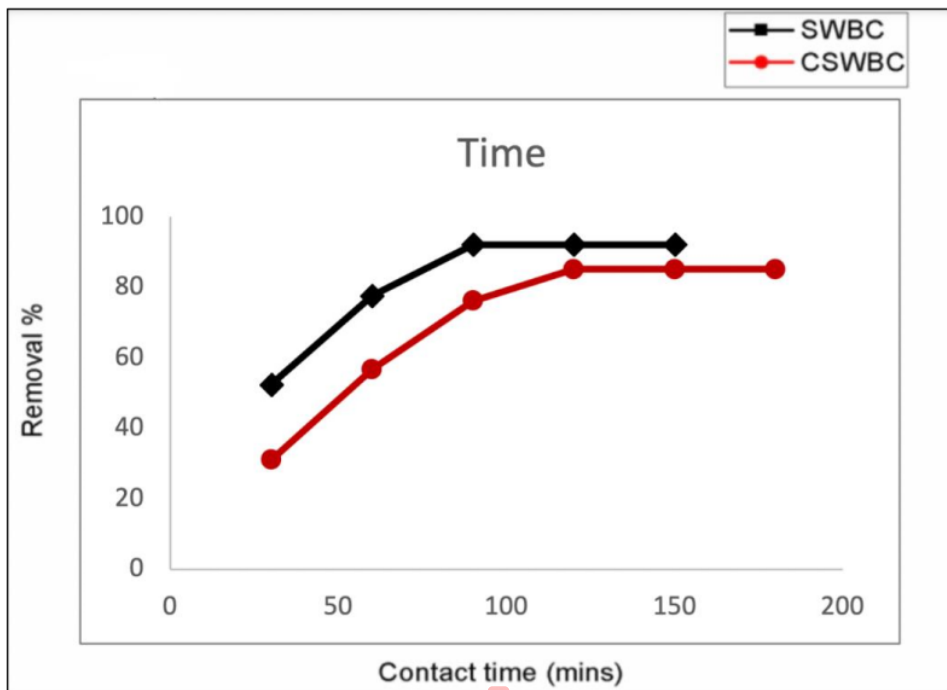


Fig:5.42: Contact Time of SWBC and CSWBC

SWBC

The removal of ciprofloxacin was studied by varying the contact time (30,60,90,120,150,180 min) while keeping the dosage (10g/l), pH(6), initial concentration (10mg/l) and temperature 303K

Removal was highest when contact time was 120min (85.5%) so 120 min was chosen as the optimum contact time

CSWBC

The removal of ciprofloxacin was studied by varying the contact time (30,60,90,120,150 min) while keeping the dosage (2g/l), pH(8), initial concentration (10mg/l) and temperature 303K

Removal was highest when contact time was 120min (92.14%) so 120 min was chosen as the optimum contact time.

5.2.5 Temperature

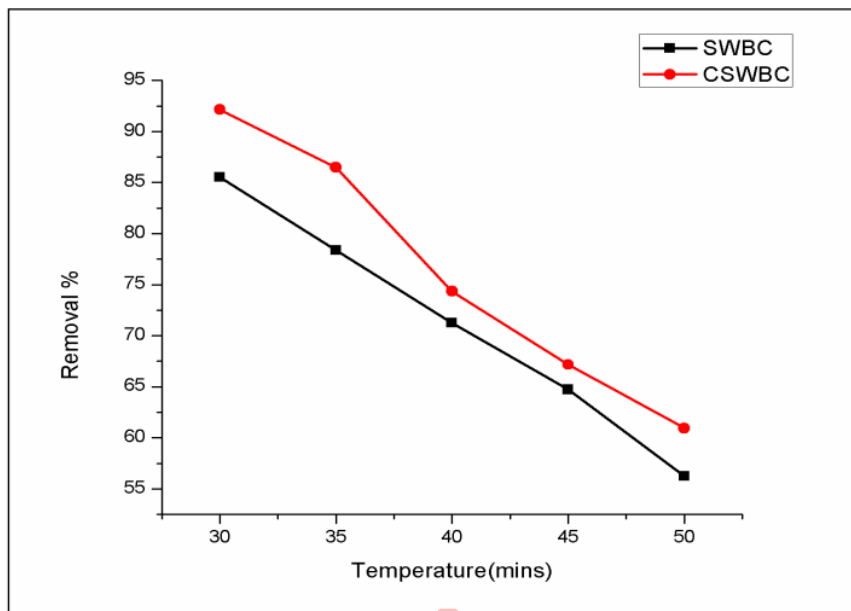


Fig.5.43: Temperature of SWBC and CSWBC

SWBC

The removal of ciprofloxacin was studied by varying the Temperature (303,308,313,318,323 K) while keeping the dosage (10g/l), pH(6), initial concentration (10mg/l) and contact time 120 min

Removal was highest when temperature was 303K (85.5%) so 303 K was chosen as the optimum temperature

CSWBC

The removal of ciprofloxacin was studied by varying the Temperature (303,308,313,318, 323 K) while keeping the dosage (2g/l), pH(8), initial concentration (10mg/l) and contact time 120 min

Removal was highest when temperature was 303K (92.14%) so 303 K was chosen as the optimum temperature.

5.3 ADSORPTION ISOTHERM MODELS

SWBC

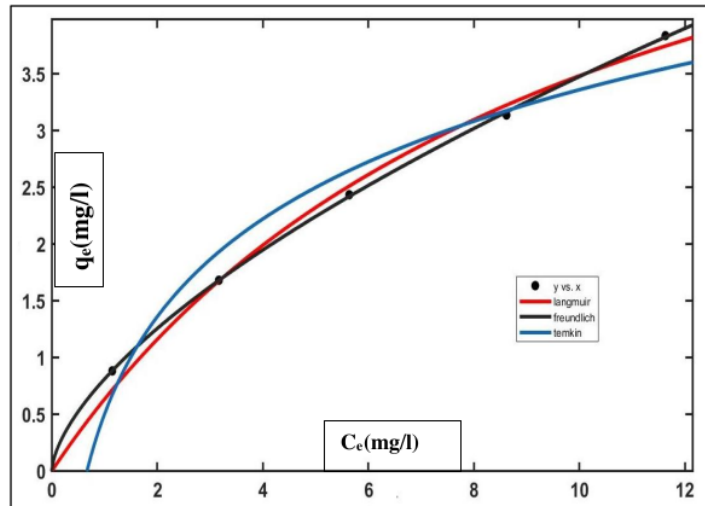


Fig.5.44: Adsorption Isotherm models for SWBC

CSWBC

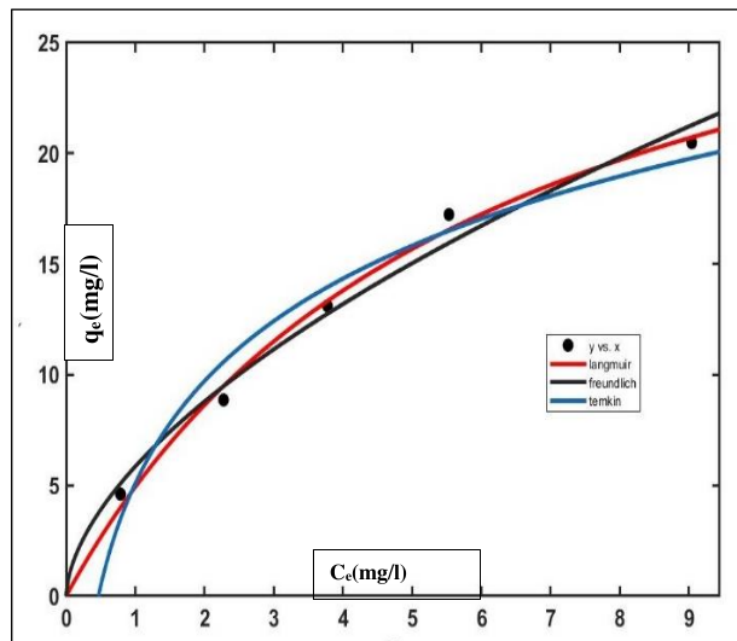


Fig.5.44: Adsorption Isotherm models for CSWBC

Table:5.6: Adsorption Isotherm parameters

Models	SWBC		CSWBC	
	Parameters	Values	Parameters	Values
Langmuir	$q_{\max}$ (mg/g)	6.964	$q_{\max}$ (mg/g)	34.43
	K_L	0.188	K_L	0.26
	RL	0.34	RL	0.277
	R^2	0.9909	R^2	0.9917
Freundlich	$1/n$	0.632	$1/n$	0.6344
	K_f	1.23	K_f	5.425
	R^2	0.9997	R^2	0.9752
Temkin	B_T (Jmol ⁻¹)	1.242	B_T (Jmol ⁻¹)	1.651
	K_T (L/mg)	1	K_T (L/mg)	1.522
	R^2	0.9415	R^2	0.93872

SWBC

Based on the experimental data and analysis, it can be concluded that the adsorption process follows the Freundlich isotherm model most closely among the Langmuir, Freundlich, and Temkin isotherms. This conclusion is supported by the highest R^2 value obtained for the Freundlich isotherm model. The Freundlich model describes multilayer adsorption onto heterogeneous surfaces, which suggests that the adsorbate molecules exhibit non-ideal behaviour and form a heterogeneous distribution of adsorption sites on the adsorbent surface. Therefore, the Freundlich isotherm provides a better fit to the experimental data and offers valuable insights into the adsorption mechanism and surface properties of the adsorbent under investigation.

CSWBC

the adsorption behaviour of the studied system aligns most closely with the Langmuir isotherm model. This assertion is supported by the highest coefficient of determination (R^2) obtained for the Langmuir model compared to Freundlich and Temkin isotherms. The Langmuir model's superior fit suggests that the adsorption process predominantly follows monolayer adsorption onto a homogeneous surface

with no interaction between adsorbed molecules. While Freundlich and Temkin isotherms may offer insights into other aspects of the adsorption process, such as heterogeneous surfaces or adsorbate-adsorbate interactions, the Langmuir model appears to be the most suitable for describing the adsorption behaviour of the adsorbent under the experimental conditions studied.

5.4 ADSORPTION KINETICS

SWBC

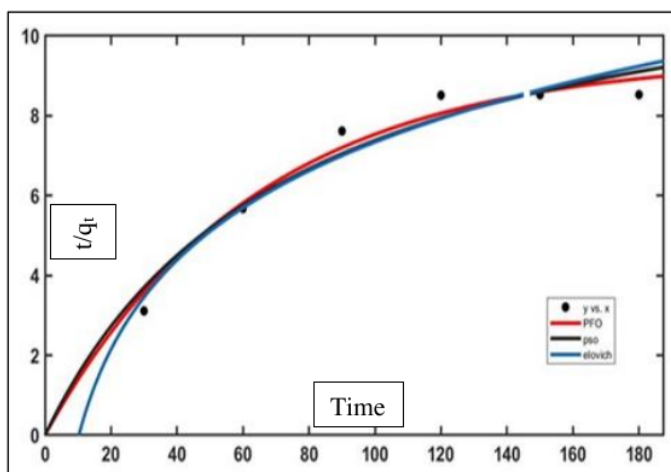


Fig:5.46: Adsorption Kinetics for SWBC

CSWBC

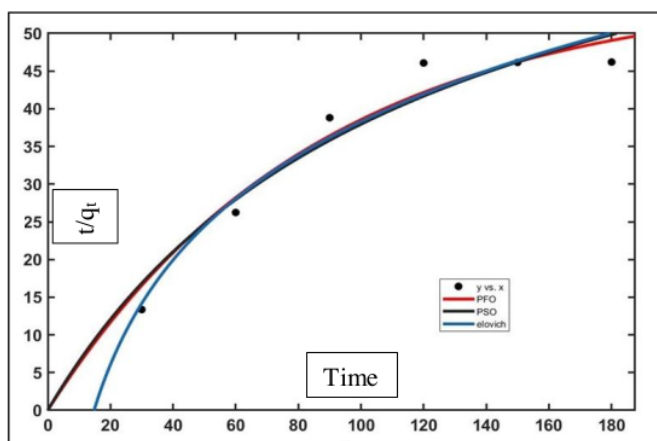


Fig:5.47: Adsorption Kinetics for CSWBC

Table:5.7: Adsorption Kinetics parameters

Kinetic Models	SWBC		CSWBC	
	Parameters	Values	Parameters	Values
Pseudo-First Order	K1 (min ⁻¹)	0.01585	K1 (min ⁻¹)	0.01173
	SSE	0.7872	SSE	43.46
	RMSE	0.4436	RMSE	3.296
	R ²	0.9675	R ²	0.953
Pseudo-Second Order	K2 (min ⁻¹)	0.001046	K2 (min ⁻¹)	0.0001056
	SSE	1.338	SSE	56.67
	RMSE	0.5784	RMSE	3.764
	R ²	0.9447	R ²	0.9387
Elovich	SSE	1.357	SSE	43
	RMSE	0.5825	RMSE	3.297
	R ²	0.9439	R ²	0.9535

SWBC

In conclusion, the adsorption process under investigation predominantly adheres to the kinetics of pseudo-first order rather than pseudo-second order. This determination is supported by the superior fit of experimental data to the pseudo-first order model, as evidenced by the highest R-squared (R²) value attained. The observed adsorption behaviour suggests that the rate-limiting step in the adsorption process likely involves the surface interaction between the adsorbate and the adsorbent, a characteristic commonly associated with pseudo-first order kinetics. As such, this finding underscores the importance of considering kinetic models in understanding and optimizing adsorption processes for practical applications.

CSWBC

In conclusion, the experimental data analysis reveals that the adsorption process adheres more closely to the pseudo-second-order kinetics model compared to the

pseudo-first-order model. This assertion is supported by the significantly higher coefficient of determination (R^2) obtained for the Elovich model, indicating a stronger correlation between the experimental and theoretical values. Thus, the adsorption mechanism is better described by the Elovich kinetics, suggesting that the adsorbent's behaviour is governed by chemisorption processes involving surface interactions and bond formation, rather than simple physical adsorption. This understanding enhances our comprehension of the adsorption dynamics, facilitating the optimization and design of adsorption-based processes for various applications.

5.5 THERMODYNAMICS

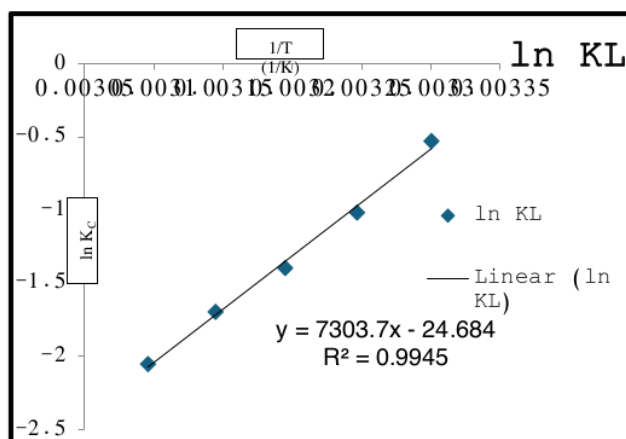


Fig:5.48: Thermodynamics for SWBC

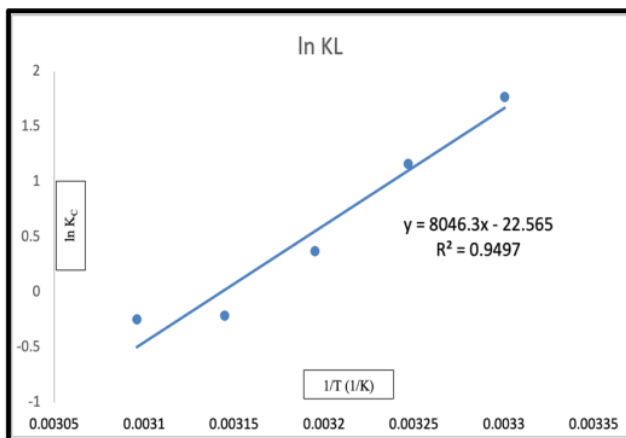


Fig:5.49: Thermodynamics for CSWBC

Table:5.8: Thermodynamics for SWBC and CSWBC

Sample	Intercept	Slope	ΔH^0 (KJmol ⁻¹)	ΔS^0 (JK ⁻¹ mol ⁻¹)	R ²	ΔG^0 (JK ⁻¹ mol ⁻¹)
SWBC	-24.68	7303	-60717.1	-205.18952	0.9945	0.4896
CSWBC	-33.682	12324	-10.48365	-329.9161	0.9497	-3624.169

SWBC

Based on the calculated values:

- $\Delta G = 1.33$ kJ/mol: This positive value indicates a non-spontaneous adsorption process. External energy input might be required for efficient ciprofloxacin removal.
- $\Delta S = -205.18$ J/mol*K: The negative entropy change suggests a decrease in randomness at the adsorbent-ciprofloxacin interface during adsorption. This could be due to increased order and specific interactions between the antibiotic and the adsorbent surface.
- $\Delta H = -60.717$ kJ/mol: The negative enthalpy change signifies an exothermic process. The adsorption of ciprofloxacin releases heat, suggesting a favourable interaction between the antibiotic and the adsorbent.

The adsorption of ciprofloxacin onto the chosen adsorbent seems favourable due to the exothermic nature ($\Delta H < 0$). However, the positive ΔG value indicates the process is not spontaneous. Further investigation is needed to optimize the adsorption process for efficient ciprofloxacin removal from water sources.

CSWBC

Based on the calculated thermodynamic parameters:

- $\Delta G^\circ = -4.454$ kJ/mol (negative): This indicates a spontaneous adsorption process. The negative Gibbs free energy suggests that the system favours the adsorption of the target molecule onto the adsorbent.

- $\Delta S^\circ = -277.60 \text{ J/mol}\cdot\text{K}$ (negative): This negative entropy change suggests a decrease in randomness at the interface during adsorption. This could be due to the ordering of the target molecule on the adsorbent surface or a decrease in the mobility of the molecule upon adsorption.
- $\Delta H^\circ = -88.336 \text{ kJ/mol}$ (negative): This negative enthalpy change signifies an exothermic adsorption process. The release of energy during adsorption suggests a stabilizing interaction between the target molecule and the adsorbent surface.

The thermodynamic data suggests that the adsorption of the target molecule onto the adsorbent is spontaneous and exothermic. The decrease in entropy indicates an ordering of the system during adsorption. These findings suggest a favourable and efficient adsorption process for the chosen system.

CHAPTER – 6

SUMMARY AND CONCLUSION:

Characterization of the biochars, *Sargassum wightii* biochar (SWBC) and chitosan coated *Sargassum wightii* biochar (CSWBC), was conducted to understand their structural and chemical properties in relation to their adsorption performance for ciprofloxacin (CIP) removal from water systems. The SWBC was initially analyzed using scanning electron microscopy (SEM) to examine its surface morphology, revealing a porous structure with irregular shapes and sizes. Energy-dispersive X-ray spectroscopy (EDX) was employed to determine the elemental composition, highlighting the presence of carbon, oxygen, and other trace elements. Fourier-transform infrared spectroscopy (FTIR) was utilized to identify functional groups present in SWBC, indicating the presence of hydroxyl, carbonyl, and aromatic groups, which are crucial for adsorption processes. X-ray diffraction (XRD) analysis revealed the crystalline structure of SWBC, with diffraction peaks corresponding to graphitic carbon and other minerals. Additionally, these characterizations provide crucial insights into the structural and chemical properties of SWBC, laying the foundation for understanding its adsorption behavior.

In contrast, CSWBC underwent additional characterization to assess the impact of chitosan coating on its properties. SEM images of CSWBC displayed a similar porous structure to SWBC, suggesting that the chitosan coating did not significantly alter its morphology. However, EDAX analysis revealed an increase in nitrogen content in CSWBC compared to SWBC, confirming the presence of chitosan. FTIR spectra of CSWBC exhibited additional peaks corresponding to chitosan functional groups, indicating successful coating. XRD analysis showed similar diffraction patterns to SWBC, suggesting that the chitosan coating did not significantly affect the crystalline structure. These characterizations provide crucial insights into the structural and chemical properties of CSWBC, laying the foundation for understanding its enhanced adsorption performance for CIP removal from water systems.

The adsorption performance of SWBC and CSWBC for CIP removal from aqueous solutions was evaluated through batch adsorption experiments. The results

revealed that both biochars exhibited significant adsorption capacity for CIP, with CSWBC demonstrating superior performance compared to SWBC. The adsorption kinetics of SWBC and CSWBC followed pseudo-second-order kinetics, suggesting a chemisorption process. Furthermore, adsorption isotherm studies indicated that the Langmuir model provided the best fit for the experimental data, highlighting monolayer adsorption on the biochar surfaces. The thermodynamic parameters indicated that the adsorption process was spontaneous and endothermic. Overall, these results demonstrate the potential of SWBC and CSWBC as effective adsorbents for the removal of CIP from water systems, with CSWBC showing enhanced adsorption performance attributed to the chitosan coating. These findings contribute to the development of sustainable and efficient solutions for pharmaceutical pollutant removal from water resources, addressing an increasingly pressing environmental concern.

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