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Optimization and Application of Date Palm Seed Derived Biochar for Augmented Adsorption of Volatile Organic Compounds: A Specialized Inquiry into Trichloroethylene (TCE) and Tetrachloroethylene (PCE) Remediation

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Abstract

This research delves into the meticulous optimization and characterization of biochar (BC) derived from date palm seeds (DPS) in the Biskra region of Algeria. Guided by Response Surface Methodology (RSM), the study explores the dynamics of BC preparation, emphasizing the interplay between pyrolysis time and temperature. The success of the optimization process is validated through a desirability index of 0.843, showcasing the precision required for tailoring BC to specific environmental and economic contexts. Structural and compositional analyses, including X-ray Diffraction (XRD), Fourier Transform Infrared (FTIR), and Scanning Electron Micrograph (SEM), unravel the intricate relationship between precursor material and BC structure. Energy Dispersive X-ray Spectroscopy (EDS) results provide insights into BC's elemental composition. Surface properties and reactivity assessments, including BET surface area analysis and pH at the point of zero charge (pH_{pzc}), underscore BC's potential as a versatile adsorbent. The study extends to adsorption assessments, revealing BC's superior affinity for volatile organic compounds (VOCs) (perchloroethylene (PCE) and trichloroethylene (TCE)). Kinetic studies employing the PSO model and isotherm studies utilizing Freundlich and Langmuir models elucidate BC's adsorption behavior. The research contributes not only to the field of BC synthesis but also to the broader discourse on sustainable and tailored adsorbents, positioning BC as a multifaceted material with applications in environmental science, materials engineering, and catalysis.

Key-words :

Biochar, Date Palm Seeds, Response Surface Methodology, Optimization, Characterization, Adsorption, Volatile Organic Compounds, Sustainability.

Résumé

Cette étude approfondie se consacre à l'optimisation et à la caractérisation méticuleuse du biochar (BC) dérivé des graines de palmier-dattier (DPS) dans la région de Biskra en Algérie. Guidée par la méthodologie de surface de réponse (RSM), l'investigation explore de manière systématique la dynamique du processus de préparation du BC, mettant en exergue l'interaction sophistiquée entre le temps de pyrolyse et la température. La réussite du processus d'optimisation est formellement validée par un indice de désirabilité de 0,843, démontrant la précision essentielle pour adapter le BC à des contextes environnementaux et économiques spécifiques. Les analyses structurales et compositionnelles, comprenant la diffraction des rayons X (XRD), la spectroscopie infrarouge à transformée de Fourier (FTIR) et la micrographie électronique à balayage (SEM), révèlent la complexité intrinsèque de la relation entre le matériau précurseur et la structure du BC. Les résultats de la spectrométrie de dispersion d'énergie des rayons X (EDS) fournissent des données élémentaires sur la composition du BC. Les évaluations des propriétés de surface et de la réactivité, notamment l'analyse de la surface spécifique BET et le pH au point de charge nulle (pH_{pzc}), soulignent le potentiel du BC en tant qu'adsorbant polyvalent. L'étude s'étend à des évaluations approfondies de l'adsorption, mettant en lumière la supériorité du BC pour les composés organiques volatils (COV) (le perchloroéthylène (PCE) et le trichloroéthylène (TCE)). Des études cinétiques, employant le modèle PSO, et des études d'isothermes, utilisant les modèles de Freundlich et Langmuir, apportent des éclairages significatifs sur le comportement d'adsorption du BC. Cette recherche ne se limite pas seulement à la synthèse du BC, mais enrichit également le discours global sur les adsorbants durables.

DEDICATION

In profound dedication, I commit this work to the memory of the late **Prof. Boutarfaia Ahmed**, my esteemed supervisor, mentor, and guide, whose untimely departure earlier this year has left an indelible void in my academic journey. His wisdom, support, and scholarly insights have been the cornerstone of this endeavor, and though my heart is shattered by his loss, I am profoundly grateful for the profound impact he had on shaping my intellectual pursuits.

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In dedication and gratitude.

Rania Remmani

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List of Abbreviations

BAC: Biological Activated Carbon

BET: Brunauer-Emmett-Teller

BC: Bacterial Cellulose

BIS: Business Integrity Services

CCD: Central Composite Design

COD: Chemical Oxygen Demand

C°: Degrees Celsius

cv.: Cultivar

DO: Dissolved Oxygen

DPS: Date Palm Seeds

ECS: Electrical Conductivity

EDS: Energy Dispersive X-ray Spectroscopy

Exp: Experimental value

FTIR: Fourier Transform Infrared Spectroscopy

GAC: Granular Activated Carbon

ICMR: Indian Council of Medical Research

PAC: Powdered Activated Carbon

PAC-MBR: Powdered Activated Carbon Membrane Bioreactors

pH: Potential of Hydrogen

pHpzc: pH of Zero Point Charge

Pred: Predicted value

PSO: Particle Swarm Optimization

RSM: Response Surface Methodology

SEM: Scanning Electron Micrograph

SSA: Specific Surface Area

TCE: Trichloroethylene

USPHS: U.S. Public Health Service

V_m : Mesopore Volume

VOCs: Volatile Organic Compounds

V_t : Total Pore Volume

WHO: World Health Organization

XRD: X-ray Diffraction

X_1 : Parameter representing Time

X_2 : Parameter representing Temperature

Y : Yield

Y_1 : Parameter representing Specific Surface Area

Y_2 : Parameter representing Total Pore Volume

Y_3 : Parameter representing Mesopore Volume

Y_4 : Parameter representing Yield

General Introduction

In the contemporary discourse on sustainable environmental practices, the exploration of innovative solutions has taken center stage, and among these solutions, biochar—an intricately structured carbonaceous material derived through the pyrolysis of organic substrates—stands as a potential linchpin. This research endeavors to contribute substantively to the discourse by engaging in the systematic optimization of biochar production, focusing on date palm seeds as a distinctive substrate. Employing the methodological rigor of Response Surface Methodology (RSM), the study extends beyond the bounds of procedural refinement, seeking to illuminate the latent potential of date palm seed biochar as a judicious adsorbent for Volatile Organic Compounds (VOCs).

The imperative for sustainable environmental practices arises from an acute awareness of the ecological challenges facing the contemporary world. The depletion of natural resources, exacerbation of climate change, and the proliferation of pollutants underscore this context, biochar has emerged as a subject of considerable scientific inquiry. Defined as a carbonaceous residue resulting from the pyrolysis of organic materials, biochar possesses unique physicochemical properties that make it a promising candidate for diverse environmental applications, including soil amendment, carbon sequestration, and pollution mitigation.

The specific focus on date palm seeds as a feedstock for biochar production reflects a deliberate effort to diversify the sources of this valuable material. Date palm trees, ubiquitous in various regions, generate significant agricultural residues in the form of seeds. While these seeds have traditionally been regarded as waste, their potential as a biochar precursor has yet to be comprehensively explored. Thus,

the research positions date palm seeds as a distinct substrate, warranting meticulous investigation into their biochar production potential and subsequent application in VOC adsorption.

The methodological framework chosen for this research, namely Response Surface Methodology (RSM), is characterized by its systematic and mathematical approach to optimizing complex processes.

RSM facilitates the exploration of the relationship between input variables (factors) and the desired response, offering a structured pathway to uncover the optimal conditions for biochar production. By leveraging RSM, this study endeavors not only to refine the biochar production process but also to elucidate the intricate interplay of factors influencing the characteristics of date palm seed biochar.

Within the expansive realm of biochar applications, the focus on VOC adsorption stands as a distinctive and consequential facet of this research. Volatile Organic Compounds represent a diverse group of organic chemicals that can vaporize into the air, contributing to air pollution and posing potential health risks. The adsorption of VOCs onto biochar surfaces presents an innovative and environmentally sustainable strategy for mitigating their impact. Therefore, this research aspires to elevate the utility of date palm seed biochar beyond conventional applications, positioning it as a valuable adsorbent for addressing contemporary environmental challenges.

The contextualization of this research within the broader discourse on environmental challenges and sustainable alternatives underscores its significance. As environmental issues become increasingly complex and intertwined, the need for multifaceted solutions grows imperative. The exploration of biochar from date palm seeds, guided by RSM, not only seeks to address this imperative but also endeavors to contribute new knowledge to the scientific community.

To substantiate the significance of this research, it is crucial to delineate the specific niche it occupies within the existing body of knowledge. While biochar re-

search is well established, the application of RSM to optimize the production process from date palm seeds is relatively unexplored. The dearth of comprehensive studies in this specific domain underscores the novelty and timeliness of the research endeavor. By articulating the unique attributes of date palm seeds, the study aims to carve out a distinctive niche, offering insights that extend beyond procedural refinements to encompass broader applications and implications.

Chapter I

Literature Review

I.1 Water pollution: Sources, Impacts on the Environment, and Health

I.1.1 Introduction

Since time immemorial, carbon and water have served as the elemental pillars sustaining life on our planet. While the environmental conditions prevailing on other celestial bodies within the Solar System are inhospitable to life, Earth stands as the solitary "habit-able planet sensu stricto" where water maintains its stability at the planetary surface (?). The steadfast presence of liquid water on Earth's surface stems from its propitious climatic circumstances and atmospheric parameters, setting it apart from the extreme environments encountered elsewhere. This moderation in temperature and atmospheric pressure fosters an environment conducive to the existence of liquid water—a crucial prerequisite for the manifold biochemical processes and intricate interplay essential for life's emergence and proliferation. The solvent properties inherent in water underpin diverse chemical reactions while enabling pivotal biological functions encompassing nutrient transportation, waste expulsion, and temperature regulation. Moreover, Earth boasts an unrivaled abundance of water resources within our Solar System. These bountiful freshwater reservoirs, encompassing expansive lakes, meandering rivers, and subsurface groundwater, sustain intricate ecosystems and serve as a vital life-sustaining lifeline for hu-

man civilization. The intricate interconnectedness of the hydrological cycle ensures the perpetual circulation and replenishment of this invaluable resource, facilitating its equitable distribution across disparate geographical regions while fostering an array of diverse habitats. By acknowledging the distinctive stability and copiousness of water on Earth, we gain a heightened appreciation for the extraordinary habitability of our planet. Such recognition underscores the pressing need for judicious custodianship to safeguard and prudently manage this invaluable resource, with far-reaching implications for our own survival and the preservation of Earth's exceptional ecosystems and biodiversity. In-depth exploration of water's properties, behaviors, and multifaceted interactions serves to enrich our comprehension of our planet's intricacies while potentially yielding invaluable insights into the prospects for extraterrestrial life forms.



Figure I.1: Water in nature: Mediterranean Sea, Algeria.

Groundwater, as an omnipresent and abundant source of pristine water

on Earth's surface, holds immense potential for broad-scale development without the need for extensive infrastructure (Figure I.2). Its widespread availability makes

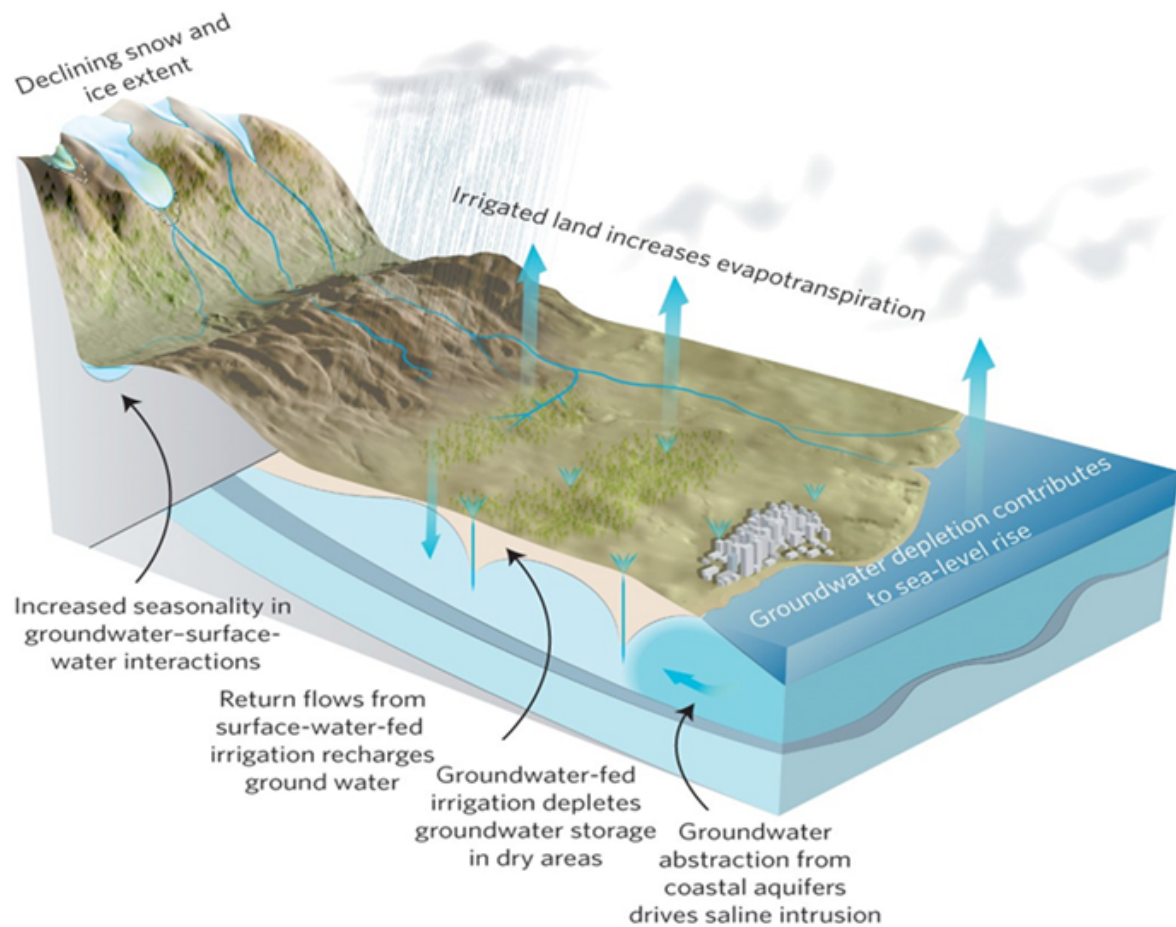


Figure I.2: Graphical representation of the hydrologic cycle: fundamental interchanges between groundwater and environment Taylor et al..

it a highly desirable re-source, capable of meeting diverse demands while maintaining water quality standards. Global freshwater withdrawals derive approximately one-third of their supply from groundwater, serving as the primary source for household, agricultural, and industrial uses, constituting 36%, 42%, and 27% respectively (Taylor et al., Döll et al. [2012]). To gain insights into the long-term dynamics of groundwater systems in response to climatic influences, researchers often turn to palaeohydrological evidence obtained from localized aquifer systems in arid and semi-arid regions. These valuable records enable an understanding of the intrinsic mechanisms governing groundwater behavior, decoupled from human interference. By analyzing the palaeohydrological data, we can project and anticipate

the responses of groundwater resources to natural climate variations over extended temporal scales, shedding light on their resilience and vulnerability.

An illustrative example showcasing the vast expanse of groundwater resources is the Northwest Sahara aquifer system, spanning multiple countries in northern Africa, including Algeria, Tunisia, Libya, and Egypt (Figure I.3) (Richits et al. [2011]). This extensive aquifer system offers a compelling case study, highlighting the significance and magnitude of groundwater reservoirs in arid regions. In-depth investigations into the hydrogeological characteristics and dynamics of such systems contribute to a comprehensive understanding of water availability, resource management, and sustainability, particularly in regions heavily reliant on groundwater resources. By harnessing the knowledge gained from studying groundwater responses to climatic variations, policymakers and stakeholders can devise informed strategies to effectively manage and safeguard this invaluable resource. Moreover, a nuanced comprehension of groundwater dynamics in diverse geographical contexts enhances our ability to address water scarcity challenges, promote sustainable water usage practices, and ensure the preservation of both human water needs and ecological integrity.

The objective of this comprehensive literature review is to conduct a systematic investigation into the myriad origins of water pollution, conduct a comprehensive examination of its wide-ranging environmental impacts, and elucidate the consequential health implications. By thoroughly reviewing and synthesizing existing scholarly works, this study endeavors to advance the collective understanding of water pollution as a significant and pressing environmental predicament, accentuating the imperative for efficacious interventions and evidence-based policies that can effectively mitigate pollution, preserve fragile ecosystems, and ensure the well-being and safety of human populations.

I.1.2 Water Pollution, Pollutants

I.1.2.1 Water Pollution definition

1. Definition N°1

Water pollution encompasses the contamination of water sources by various pollutants, leading to the degradation of water quality and rendering it unsuitable for essential activities such as drinking, cooking, cleaning, and recreational purposes. These pollutants can manifest in the form of chemicals, solid waste, pathogenic bacteria, and parasitic organisms. The introduction of any type of pollutant ultimately contributes to the pollution of water bodies. Broadly defined, a substance qualifies as a pollutant if its release into the environment results in the depletion of resources or exerts adverse effects on the ecosystem. The presence of pollutants can inflict both immediate and long-term damage to aquatic environments. Notably, nonbiodegradable pollutants pose a significant environmental threat, as their persistence can lead to sustained harm, while biodegradable pollutants typically exert only transient adverse effects (Chaudhry and Malik [2017]).

2. Definition N°2

Water pollution arises when detrimental substances, primarily chemicals or bacteria, infiltrate and permeate various water bodies, including streams, rivers, lakes, oceans, aquifers, and other aquatic systems. The introduction of these harmful constituents significantly compromises the quality of water, rendering it potentially hazardous to both human health and the ecological balance (Dakota, Remmani [2022]).

3. Definition N°3

Pollution encompasses the intentional or unintentional introduction of chemicals or energy into the natural environment by human beings. Through a variety of human activities, diverse pollutants are released into aquatic environments, posing significant threats to the well-being of organisms, and inducing ecological degradation that renders water bodies unsuitable for their intended purposes (Bashir et al. [2020], Remmani et al. [2021]).

I.1.2.2 Sources of Water Pollution

Water pollution is a multifaceted issue stemming from a wide array of causative factors, necessitating a classification into two primary classifications: direct and indirect sources of pollutants. Direct sources pertain to distinct points of discharge where pollutants are introduced into water bodies, encompassing industrial facilities, wastewater treatment plants, and sewage systems. Such point sources enable relatively straight forward identification and regulation. In contrast, indirect sources encompass diffuse inputs arising from diverse land-based activities, such as urban runoff, agricultural practices, and atmospheric deposition. These diffuse sources pose distinct challenges due to their dispersed nature, necessitating comprehensive management strategies to effectively mitigate their adverse impacts on water quality(Owa [2014],?).

Moreover, pollutants responsible for water contamination can be broadly categorized into distinct groups based on their chemical composition and properties. Organic pollutants encompass compounds originating from living organisms or their byproducts, including pesticides, pharmaceuticals, and petroleum-derived hydrocarbons. Inorganic pollutants comprise chemical substances lacking carbon atoms, such as heavy metals, toxic ions, and acids. Radioactive pollutants stem from radioactive materials, potentially posing significant health hazards. Additionally, acid/base pollutants contribute to alterations in water pH levels, frequently as a result of industrial processes or the occurrence of acid rain. A comprehensive understanding of the diverse origins and characteristics of pollutants is essential for effective pollution management and the formulation of targeted mitigation strategies aimed at preserving water resources and safeguarding ecosystems(Owa [2014],?).

Human activities exert substantial influence on the quality of aquatic ecosystems, including rivers, lakes, oceans, and groundwater. The degradation of water quality stems from diverse anthropogenic sources, necessitating a comprehensive understanding to effectively address the issue. These sources can be broadly

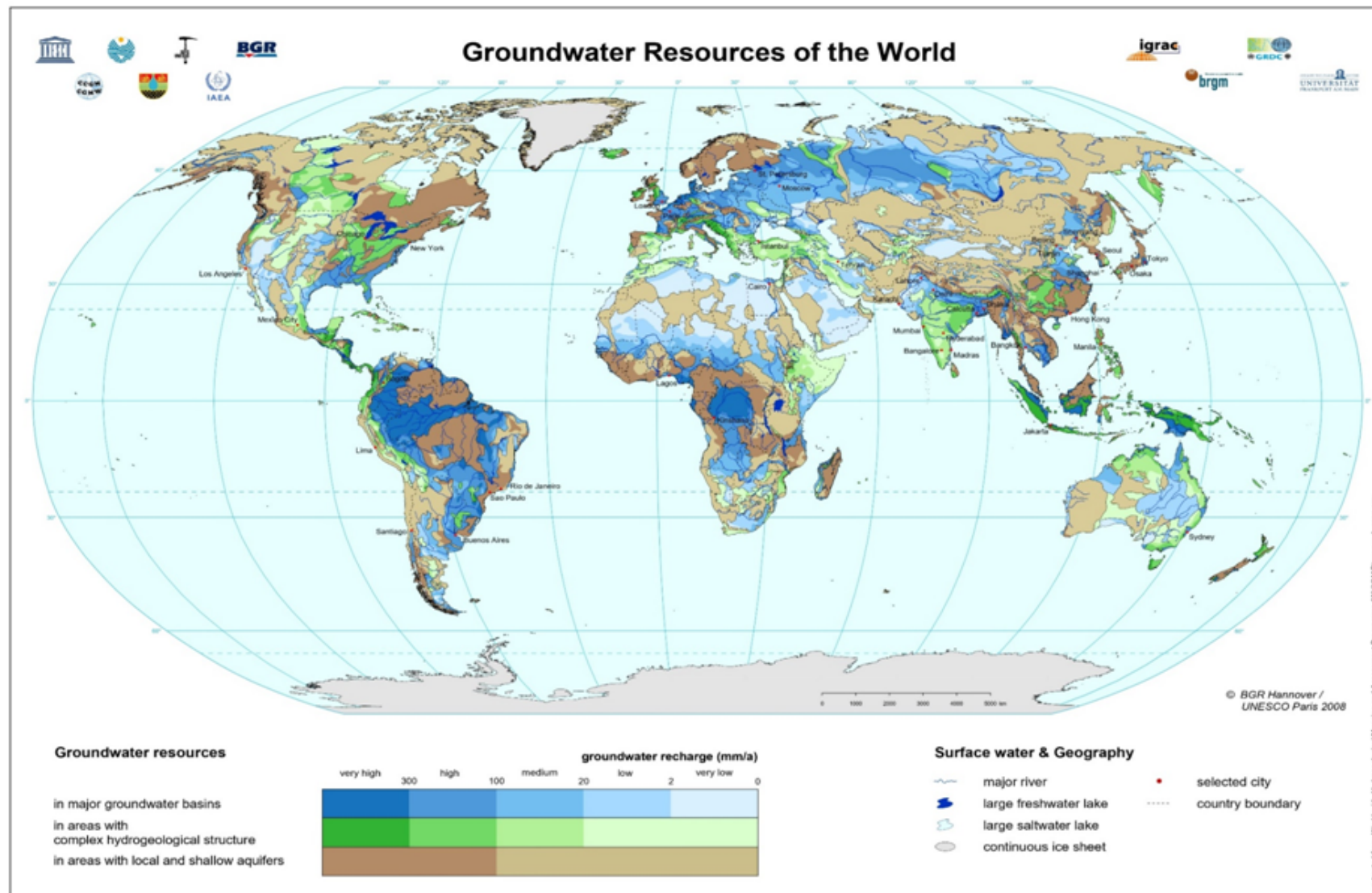


Figure I.3: Simplified version of a global groundwater resources map showcasing local aquifer systems (Richits et al. [2011])

categorized into two distinct classifications: point sources and diffuse sources. Point sources of pollution encompass specific discharge points, such as industrial facilities and sewage treatment plants, which release pollutants directly into water bodies. These inputs result in localized contamination and immediate environmental impacts. Notably, regulatory measures targeting point sources have demonstrated efficacy in mitigating direct pollution load and safeguarding aquatic ecosystems.

In addition to point sources, water quality is profoundly affected by diffuse pollution originating from multiple sources. Agricultural activities contribute significantly to diffuse pollution through the discharge of nutrients and pesticides. Improper management of fertilizers and animal waste can lead to nutrient runoff, fostering eutrophication and harmful algal blooms in aquatic systems. Moreover, industrial activities emit pollutants into the atmosphere, which subsequently settle on land and water, perpetuating the phenomenon of diffuse pollution. These pollutants encompass a wide range of substances, including heavy metals, volatile organic compounds, and particulate matter, among others, posing significant risks to water quality and ecosystem health. Developing comprehensive strategies to address both point and diffuse sources of pollution is paramount to ensuring the sustainable management and conservation of water resources. Implementing targeted measures to mitigate the impacts of point sources, along with adopting best practices in agricultural and industrial sectors, are imperative for preserving the integrity of aquatic ecosystems. By understanding and effectively managing the diverse sources of water pollution, it is possible to promote the long-term health and resilience of these invaluable ecosystems.

Sewage and wastewater treatment facilities emerge as prominent contributors to water pollution, while diffuse pollution predominantly stems from agricultural practices and emissions from fossil fuel power plants. It is crucial to note that although sewage treatment plants are classified as "point sources," they do not serve as the primary origins of pollution, but rather function as remedial entities targeting the waste discharged from human activities such as toilets, sinks, and industrial pro-

cesses. Their purpose revolves around the treatment and removal of contaminants to mitigate potential environmental repercussions.

On the other hand, diffuse pollution, which poses a substantial challenge to water quality preservation, predominantly emanates from agricultural activities and emissions from fossil fuel power plants. The agricultural sector contributes to diffuse pollution through the introduction of fertilizers, pesticides, and sediments into nearby water bodies, facilitated by surface runoff or infiltration into groundwater. Similarly, fossil fuel power plants release a range of air pollutants, including sulfur dioxide, nitrogen oxides, and particulate matter, which undergo atmospheric transport and subsequent deposition in water ecosystems. This form of pollution necessitates comprehensive strategies for mitigation to ensure the conservation of water resources and the preservation of ecological integrity.

Comprehending the distinct sources of pollution, encompassing both point and diffuse origins, holds paramount significance in the pursuit of effective water management and conservation endeavors. By acknowledging the intermediary role of sewage treatment plants and addressing the underlying human activities that generate waste, opportunities arise to enhance wastewater treatment systems and curtail associated environmental impacts. Simultaneously, the adoption of sustainable agricultural practices and the implementation of cleaner technologies in power generation can contribute significantly to the reduction of diffuse pollution, fostering the safeguarding of water ecosystems and their intrinsic value.

I.1.2.3 Water quality standards

Water quality standards are developed and recommended by reputable health institutions to ensure the adequacy and safety of various water sources. Noteworthy organizations in this regard include the United States Public Health Service Drinking Water Standards (USPHS), the Indian Council of Medical Research (ICMR), and the World Health Organization (WHO). These esteemed institutions have established precise guidelines and parameters to govern the quality of water, recognizing its direct impact on human health and well-being. Compliance with these standards

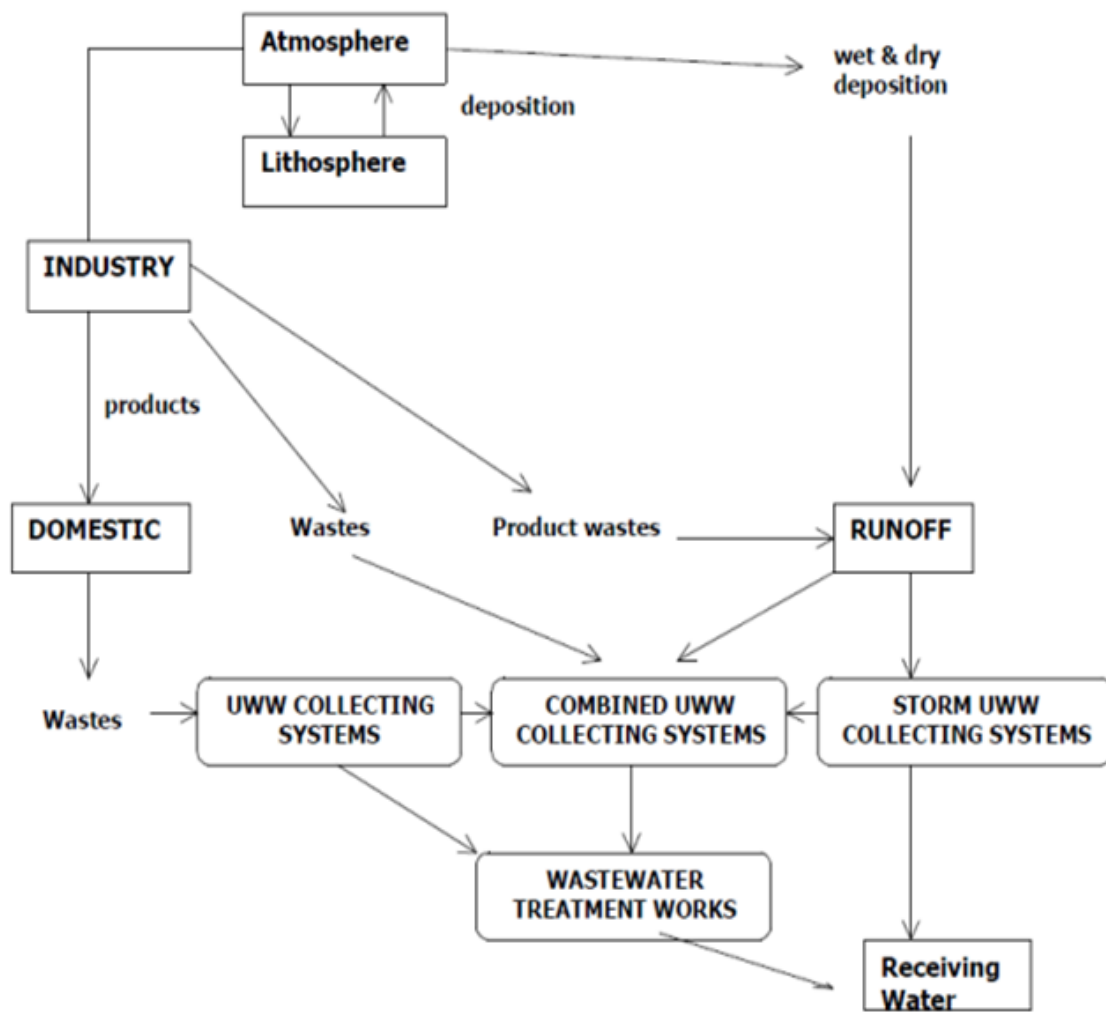


Figure I.4: Graphical representation of sources of pollution(McMonagle [2013]).

is imperative to mitigate potential health risks associated with the consumption and utilization of water resources.

Table I.1 presents a comprehensive overview of the water quality requirements prescribed by diverse authoritative entities for inland water sources. These requirements encompass a wide range of criteria, encompassing physical, chemical, and microbiological aspects, which are critical for assessing and monitoring water quality. Adhering to these established standards allows policymakers, regulatory bodies, and water management authorities to ensure that water resources fulfill the necessary criteria for human use, thus minimizing potential adverse effects on public health. These standards serve as fundamental tools for the evalua-

tion, assessment, and effective management of water quality, facilitating informed decision-making and the implementation of appropriate measures to safeguard human well-being.

Table I.1: Water quality standards (Dwivedi [2017]).

Parameter	USPHS	BIS	WHO	ICMR
Temperature C°	-	40.0	-	-
$EC\text{Sm}^{-1}$	0.03	0.075	-	-
pH	6.0-8.5	6.5-8.5	7.0-8.5	6.5-9.2
$DO\text{mg.L}^{-1}$	>4.0	>5.0	-	-
$BOD\text{mg.L}^{-1}$	-	<3.0	-	-
$COD\text{mg.L}^{-1}$	-	<20.0	-	-
$Chlorid\text{mg.L}^{-1}$	250	250	200	250
$Alkalinity\text{mg.L}^{-1}$	-	-	-	81-120
$Nitrat\text{mg.L}^{-1}$	10.0	50.0	45.0	20.0
$Phosphat\text{mg.L}^{-1}$	0.1	-	-	-
$Sulphat\text{mg.L}^{-1}$	250	150	200	200
$Total\text{hardness}\text{mg.L}^{-1}$	500	300	100	300
$Total\text{solids}\text{mg.L}^{-1}$	500	-	500	-
$Calcium\text{mg.L}^{-1}$	100	75	75	75
$Magnesium\text{mg.L}^{-1}$	-	30	-	50
$Potassium\text{mg.L}^{-1}$	-	-	-	20
$Sodium\text{mg.L}^{-1}$	-	-	50	-

- Not available; USPHS: U.S. Public Health Service Drinking Water; BIS: Business Integrity Services; WHO: World Health Organization; ICMR: Indian Council of Medical Research; EC: Electrical Conductivity; pH: Potential of Hydrogen; DO: Dissolved Oxygen; BOD: Biochemical Oxygen Demand; COD: Chemical Oxygen Demand.

I.1.3 Effects on the Environment and human health

I.1.3.1 Health risk of water pollution

Water pollution poses significant concerns primarily due to its adverse impacts on human health resulting from various anthropogenic activities. Extensive empirical evidence, as presented in Table I.2 and Figure I.5, elucidates the manifold risks associated with diverse types of pollutants, which represent a tangible threat to human life and future well-being (Lin et al. [2022], Haseena et al. [2017], Xue et al. [2022]). With respect to organic pollutants, although their concentrations conform to permissible limits for drinking water, a comprehensive assessment of carcinogenic risks undertaken for the district's heterogeneous communities and the overall population unveils an escalated risk of cancer (Callahan and Sexton [2007]). This risk is explained in detail within Table I.2, elucidating the potential health hazards linked to organic pollutants.

Conversely, inorganic pollutants engender substantive health concerns due to the emission of trace elements, which accumulate within the human body via the food chain (Camargo and Alonso [2006]). Among these pollutants, heavy metals constitute a particularly pernicious subset, giving rise to a myriad of severe illnesses, as comprehensively delineated in Table I.2 (Lin et al. [2022]). Importantly, the presence of organic contaminants in water and their prevailing concentrations do not contribute to an augmented incidence of non-carcinogenic ailments among residents relying on monitored water sources. These findings underscore the critical importance of discerning the specific health risks tied to distinct pollutant types and concentrations, thus facilitating targeted interventions and effective mitigation strategies to safeguard human health.

I.1.3.2 Environmental risk of water pollution

The utilization of contaminated water for irrigation purposes in low-income nations worldwide poses a significant challenge to achieving food and livelihood security. Extensive research indicates that more than 80% of available water sources in these regions suffer from contamination, necessitating the use of polluted water

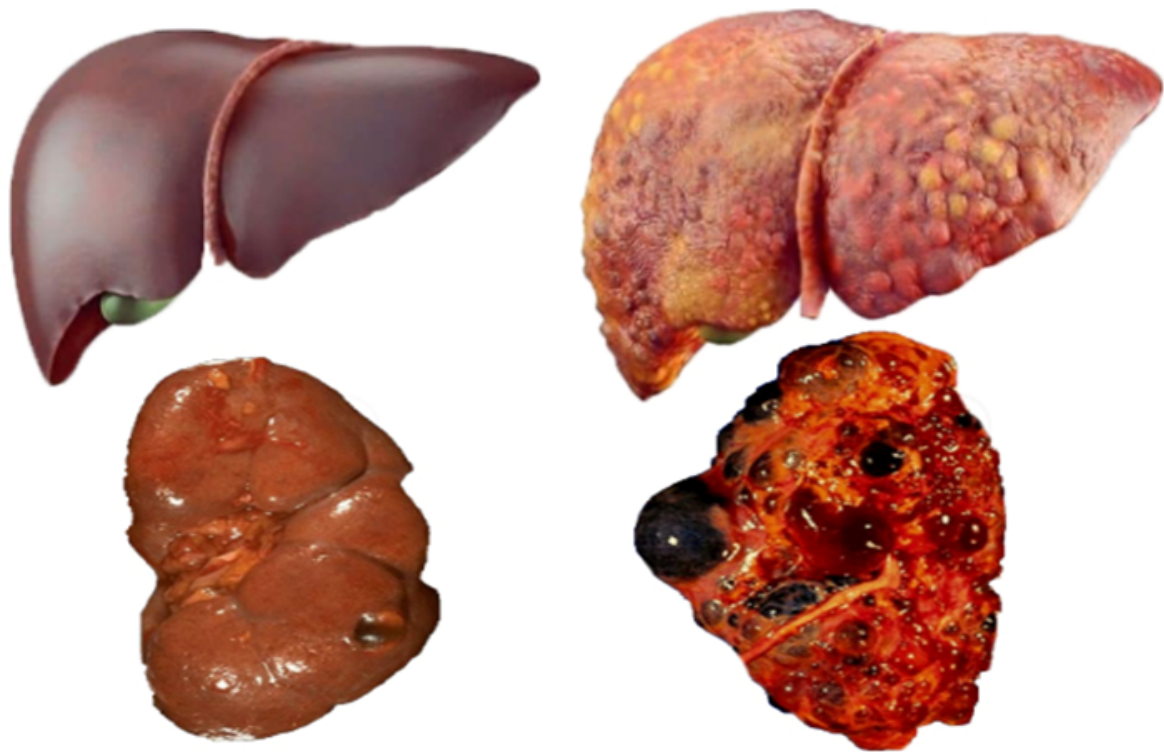


Figure I.5: Effects of water pollution on human organs.

for agricultural activities. In contrast, industrialized metropolitan and semi-urban areas, where food and livelihood security are achieved at a rate of approximately 70% to 80%, also face the pressing issue of water pollution (Stavi et al. [2021], Khan and Ghouri [2011]). Water pollution exerts detrimental effects on the health and quality of soils and vegetation, with some impacts being observable yet challenging to reverse. The presence of pollutants in water systems contributes to soil contamination, leading to reduced fertility and compromised nutrient content. Moreover, exposure of vegetation to polluted water sources can result in diminished crop yields, impaired growth, and disrupted ecosystem functioning. The persistent nature of pollutants in the environment further hampers the restoration of affected ecosystems (Luo et al. [2019]).

The consequences of water pollution extend across various water bodies, including seas, lakes, rivers, and drinking water sources. These vital aquatic ecosystems and sources of freshwater face significant threats due to pollution. The accumulation of contaminants poses risks to aquatic life, disrupting ecosystems and

Table I.2: Health effects of several water pollutants (Lin et al. [2022]).

Pollutant	Health effects
Nitrate	Shortness of breath and blue baby syndrome for infants
Chromium tot	Allergic problems
Arsenic	Skin damage and circulatory system problems; increased risk of lung, bladder, or skin cancer
Fluoride	Bone fluorosis
Copper	Gastrointestinal effects, liver, and kidney damage
Lead	Children: delay in physical and mental development; Adults: kidney damage
Cadmium	Kidney damage
Mercury	Kidney damage
Manganese	Neurological effect
Nickel	Allergic effect
DDT	Liver cancer
DDD	Liver cancer
DDE	Liver cancer
$\gamma - HCH$	Liver and kidney problems

DDT: p,p'-dichlorodiphenyltrichloroethane; DDD: p,p'-dichlorodiphenyl dichloroethane; DDE: p,p'-dichlorodiphenyldichloroethylene; $\gamma - HCH$: gamma-hexachlorocyclohexane.

undermining biodiversity. Moreover, the conimpaction of drinking water sources directly impacts human populations, leading to the spread of waterborne diseases and associated health issues. As such, water pollution has emerged as a global challenge with far-reaching implications for environmental preservation and public health (Callahan and Sexton [2007]). Numerous studies and reports emphasize the severity of water pollution as a worldwide concern, necessitating collaborative efforts and effective strategies to address its causes and mitigate its impacts. Sustain-

able water management practices, encompassing improved wastewater treatment, pollution control measures, and comprehensive public awareness campaigns, are pivotal in combatting water pollution. By adopting interdisciplinary approaches and fostering international cooperation, it is possible to safeguard the quality of water resources and protect the well-being of communities on a global scale (Lin et al. [2022], Yan et al. [2016]).

I.1.4 Conclusions

In conclusion, water pollution is a pressing global issue with significant implications for the environment and human health. The contamination of water sources by various pollutants poses a threat to the quality of water, rendering it unsuitable for essential activities and endangering ecosystems. Water pollution arises from both direct and indirect sources, including industrial facilities, sewage systems, agricultural practices, and atmospheric deposition. Understanding the diverse origins and characteristics of pollutants is crucial for effective pollution management and the formulation of targeted mitigation strategies. The impacts of water pollution on human health are significant, ranging from immediate risks to long-term health effects. Pollutants, such as organic compounds and heavy metals, can pose carcinogenic and non-carcinogenic health risks, highlighting the need for comprehensive assessment and targeted interventions. Additionally, contaminated water used for irrigation purposes can threaten food and livelihood security, particularly in low-income nations. Water pollution also harms the environment by compromising soil fertility, vegetation health, and aquatic ecosystems, leading to biodiversity loss and the spread of waterborne diseases. Addressing water pollution requires a multi-faceted approach that includes improved wastewater treatment, pollution control measures, sustainable agricultural practices, and public awareness campaigns. Compliance with water quality standards set by reputable health institutions is essential to ensure the safety of water sources for human use. Collaboration and international co-operation are vital in combating water pollution and safeguarding the integrity of water resources worldwide. By recognizing the significance of water as a vital resource and taking proactive measures to mitigate pol-

lution, we can protect the environment, promote human health, and secure the sustainability of our precious water ecosystems for future generations.

I.2 Several Common Wastewater Treatment Approaches

I.2.1 Introduction

The historical lineage of wastewater treatment finds its origins in antiquity, notably marked by the pioneering efforts of the Mesopotamian Empire in the establishment of rudimentary drainage systems. The evolution of wastewater treatment processes, however, transcends the temporal expanse, influenced by the confluence of scientific advancements, imperatives of public health, and geopolitical dynamics (Lofrano and Brown [2010], Aiello et al. [2008]). Scientific progress engendered a transformative phase in wastewater treatment practices. The post-Second World War era, despite momentarily impeding advancements until 1948, emerged as a catalyst for urgent and comprehensive wastewater management. The ensuing period witnessed a precipitous acceleration of wastewater treatment initiatives, particularly notable in the United Kingdom and the United States. Political ideologies, exemplified by Germany's adherence to the "Blood and Soil" doctrine of the national socialist party, profoundly shaped the discourse on wastewater management. This ideological inclination towards prioritizing agricultural utilization over rigorous treatment methodologies left an enduring imprint on the trajectory of wastewater practices within the nation. Internationally, the implementation of wastewater treatment processes assumed diverse contours. The United Kingdom, for instance, prominently featured the deployment of trickling filters, juxtaposed against the United States, where the expeditious adoption of activated sludge plants found impetus in legislative milestones such as the Clean Water Act of 1972 (Lofrano and Brown [2010], Angelakis and Spyridakis [1996]).

Historically, the allocation of resources and focus often skewed towards potable water infrastructure, a predilection underscored by the pragmatic fiscal appeal of restoring ancient Roman pipelines. This historical inclination mirrors soci-

etal exigencies and contributes substantively to the broader historical narrative of wastewater treatment evolution. This historical expedition serves as a foundational backdrop for the nuanced examination of contemporary wastewater management practices. The lessons gleaned from historical antecedents furnish invaluable insights as the contemporary milieu navigates the exigencies of sustainable and efficacious wastewater treatment modalities (Lofrano and Brown [2010], Angelakis and Spyridakis [1996]).

I.2.2 Sociological Aspects

The sociological dimensions inherent in wastewater management serve as a profound conduit for elucidating societal perspectives on waste and environmental concerns. Heidegger's conceptualization of "enframing" captures the utilitarian relationship between humanity and the environment, portraying both nature and humans as commodified resources. This philosophical paradigm has historically contributed to the systemic neglect of wastewater within the broader context of waste management. Wastewater management practices, when examined through a sociological lens, unveil intricate social relationships and power dynamics. A poignant illustration is observed in the manual collection of waste by the marginalized "untouchables" in India, embodying deeply ingrained social hierarchies and discriminatory practices inherent in the societal fabric (Lofrano and Brown [2010], Viale [2000], Heidegger [2003]).

Furthermore, wastewater transcends its utilitarian function to become a sociological repository, offering profound insights into the customs and behaviors of a society. In alignment with Victor Hugo's assertion, sewers emerge as historical archives reflecting societal narratives of protest, crime, and normative frameworks, underscoring the symbolic significance attached to wastewater systems. The historical trajectory of wastewater management bears the indelible imprint of societal influences, encompassing manifestations of corruption, pursuit of economic interests, and adherence to cultural taboos. These societal factors wield considerable influence in shaping the nuanced evolution of wastewater practices, thereby introducing layers of complexity to its historical development. The endeavor to en-

hance wastewater management, particularly in developing nations, necessitates a nuanced comprehension of sociocultural boundaries, reservations, and prevailing taboos. The efficacy of interventions and improvements hinges upon adept navigation and eventual transcending of deeply ingrained societal norms and beliefs (Lofrano and Brown [2010], Viale [2000], Heidegger [2003]).

In summation, wastewater management, far from being confined to its technical and environmental dimensions, assumes a profound sociological significance. It not only encapsulates the pragmatic handling of waste but also functions as a nuanced reflection of societal values, power structures, and cultural norms. Hence, a comprehensive and interdisciplinary approach is warranted for the nuanced understanding and amelioration of wastewater management practices.

1.2.3 The Vital Role of Wastewater Treatment

Wastewater treatment assumes a pivotal role in upholding human health and environmental integrity by effecting the removal of pollutants and contaminants from wastewater prior to its discharge into water bodies. Beyond its overt environmental function, wastewater treatment substantiates a critical nexus with multifaceted implications. Foremost among its considerations is the profound impact on public health, where the systematic eradication of pathogens and contaminants serves as a potent deterrent against waterborne diseases. The fortification of public health frameworks is especially salient in averting the contamination of drinking water sources, underscoring the essentiality of wastewater treatment in safeguarding community well-being.

Moreover, the custodial responsibility of wastewater treatment extends to the preservation of ecological equilibrium within aquatic ecosystems. Its capacity to mitigate the deleterious effects of pollutants on aquatic life and habitats positions wastewater treatment as an instrumental actor in biodiversity preservation, contributing significantly to the resilience of ecosystems grappling with anthropogenic pressures.

The import of effective wastewater treatment reverberates into the realm of sustainable water resource management. By ensuring the availability of purified water for diverse applications—including agriculture, industry, and domestic use—wastewater treatment becomes an indispensable cornerstone for societal resilience and the judicious allocation of this finite resource.

In a testament to its evolving role, wastewater treatment transcends its conventional purview to become a nexus for resource recovery. The retrieval of valuable resources, including energy, nutrients, and water, exemplifies a harmonious integration of environmental stewardship with resource efficiency. This paradigm not only aligns with the precepts of a circular economy but also underscores wastewater treatment facilities as strategic loci for sustainable resource management.

The historical trajectory of wastewater treatment emerges as an instructive tableau, bearing witness to the intricate influences of political, societal, and cultural factors. This historical lens accentuates the imperative of incorporating social dimensions into wastewater management paradigms, recognizing the nuanced interplay between technological advancements and the sociocultural milieu.

In summation, the indispensable role of wastewater treatment spans realms encompassing human health, ecological preservation, and resource sustainability. It stands as a linchpin in fortifying public health, preserving environmental equilibrium, ensuring water security, and championing resource efficiency—a testimony to its irrefutable centrality in contemporary environmental stewardship.

I.2.4 Common Wastewater Treatment Methods

The unregulated discharge of untreated wastewater constitutes a grave peril to environmental integrity, engendering the contamination of aquatic ecosystems. This unbridled effluent release introduces a spectrum of contaminants, spanning pathogens, chemical agents, and nutrients, thereby imperiling aquatic biota, and compromising the overall quality of water resources. In contrast, the systematic treatment of wastewater antecedent to environmental discharge, expounded in Fig-

ure I.6 and I.7, delineates a methodically orchestrated process aimed at the removal or mitigation of contaminants. The graphical representation elucidates a comprehensive treatment paradigm encompassing physical, biological, and chemical modalities. These encompass sedimentation for solid particulate removal, biological methodologies such as activated sludge treatment for organic matter degradation, and chemical procedures like coagulation to address dissolved constituents. The visual exposition in Figure I.6 and I.7 affords a discerning perspective on the methodical and nuanced approach underpinning wastewater purification preceding its introduction into the environment. This strategic intervention serves to uphold ecological equilibrium and ameliorate the deleterious consequences associated with indiscriminate and untreated discharges.

1.2.4.1 Physical Treatment

The physical treatment of wastewater constitutes an integral facet within the broader schema of conventional wastewater treatment, synergistically interacting with chemical and biological counterparts. Its principal aim resides in the expeditious removal of solids, encompassing colloids and organic constituents, from the intricate matrix of wastewater. A repertoire of techniques, notably screening, sedimentation, filtration, and flotation, is deployed to ensure the meticulous elimination of impurities. Screening entails the deployment of screens or sieves to extract large particulates, sedimentation capitalizes on gravity for the precipitative settling of suspended solids, and filtration employs porous mediums such as sand to effectuate the extraction of smaller particles (Crini and Lichtfouse [2018]). Concurrently, flotation introduces air bubbles, inducing the ascent of solids for subsequent removal. The orchestrated integration of these physical treatment modalities assumes paramount significance in effecting the comprehensive purification of wastewater, particularly in the eradication of solid and particulate constituents. Remarkably, these physical processes often constitute inaugural steps, amplifying the efficacy of ensuing biological or chemical treatment modalities within the overarching wastewater treatment paradigm (Crini and Lichtfouse [2018], Bhargava [2016]).

1. Grit Removal

Grit removal methodologies employed in wastewater treatment plants encompass a spectrum of techniques, including medium-energy forced vortex grit units, gravity settling detritors, aerated grit systems, hydro-cyclones, classifiers, wet sieve separation, and grit concentration through hydro-cyclone systems. An empirical investigation across five wastewater treatment plants delineated discernible variations in efficacy, with gravity settling detritors exhibiting optimal grit removal efficiency (McNamara et al. [2009]). The hydro-cyclone system assumes a pivotal role in grit concentration, ameliorating hydraulic load on the classifier. Systematic analysis and rectification of issues in select hydro-cyclone systems yielded marked enhancements in overall grit removal performance. Positioned as a seminal precursor in wastewater treatment, grit removal entails the meticulous separation and elimination of diminutive, dense particles such as sand and gravel prior to subsequent treatment phases. Traditional modalities, including detritus pits and constant-velocity channels, have been complemented by contemporary approaches, yet persisting challenges encompass the assessment of system efficiency and unseparated grit volumes. Methodological selection historically hinged on subjective predilection and mechanical dependability, owing to a paucity of empirically validated alternatives (McNamara et al. [2009], Valo et al. [2004]).

2. Sedimentation

The sedimentation method, integral to wastewater treatment, serves the purpose of segregating solid particles from the liquid medium through the gravitational settling principle. Particularly pertinent in the treatment of chemical organic wastewater, sedimentation treatment tanks are meticulously engineered to facilitate the extraction of settling impurities and extraneous matter from a designated treating pond. This infrastructure encompasses a sophisticated design featuring a bottom plate, axle seats, driving shafts linked to rotation motors, a principal bevel gear, supporting blocks, threaded screw rods, secondary bevel gears, and a treating pond body (Bezirgiannidis et al. [2019]). The operational sequence involves the introduction of wastewater into the treating pond body, initiating the sedimentation process whereby suspended

particles settle under gravitational influence, effecting their subsequent separation from the liquid phase. A subsequent refinement stage employs a filtering mechanism to further eliminate impurities from the liquid medium. The delineated sedimentation tank design is characterized by its commitment to efficiency, operational convenience, and structural stability, aligning with the imperative of achieving optimal sedimentation in the context of wastewater treatment paradigms (Bezirgiannidis et al. [2019], Novikov et al. [2021]).

3. Filtration

The wastewater treatment mechanism involves a sophisticated mechanical filtration device, featuring a structural composition with a shell containing a mediating layer positioned between two filter screens. Engineered to enhance impurity filtration in wastewater, this configuration contributes to heightened efficiency and velocity in the filtration process. Crucial elements include a coagulant feeding pipe to expedite the condensation and precipitation of impurities, along with a flushing water inlet pipe and a flushing water drainage pipe to maintain filtration efficacy by preventing blockages. The mediating layer, propelled by a third fan blade, rotates to intensify the flushing process, thereby augmenting the operational viability of the filtration apparatus. Tailored for industrial wastewater treatment, the equipment systematically addresses harmful constituents, including insoluble matters, small particulate matters, and suspended solids. Comprising a shell, support base, and multiple filtering units, it orchestrates a nuanced and multi-modal filtration process, incorporating high-speed centrifugation filtration, sand-stone filtration, adsorption filtration, and settling filtration. The initial stage employs centrifugation to eliminate larger particles, while the subsequent stage refines the wastewater by extracting smaller particulate matters. The subordinated settling unit, positioned beneath the second-stage filtering unit, facilitates the separation and removal of any residual suspended solids, ensuring adherence to optimal wastewater discharging standards (Zhang et al. [2018], Patwardhan [2017]).

1.2.4.2 Biological Treatment

Biological wastewater treatment constitutes a significant domain within the realms of biochemistry, biotechnology, and environmental engineering, relying on microorganisms to enzymatically degrade organic matter and eliminate pollutants. Despite its merits, including operational simplicity, potential bioproduct generation, and expeditious treatment, this approach is not without inherent challenges. Concerns encompass potential air pollution proximal to bio-lagoons and associated health risks for personnel involved in the treatment process (Vijatov et al. [2020], Cort [2019]). Diverse methodologies can be employed in biological wastewater treatment. Bioaugmentation involves the deliberate introduction of a specific consortium of microorganisms, cultivated from waste sourced at a Wastewater Treatment Plant (WWTP), to facilitate the decomposition of organic matter and pollutant removal. The strategic implementation of a flow control device serves to prolong the residence time for biological treatment, thereby facilitating controlled water discharge. Furthermore, the high-rate clarification system, leveraging flocculating polymers, proves effective in eliminating fine suspended solids. The resultant clarified water undergoes supplementary treatment within containment structures, optimizing treatment residence time (Cort [2019]). The synergistic application of these methodologies augments the overall efficiency of biological wastewater treatment, ensuring a comprehensive removal of pollutants and organic constituents.

1. Activated Sludge

The Activated Sludge Method constitutes a prevalent biological treatment paradigm within wastewater treatment plants, utilizing a consortium of microorganisms termed activated sludge to enzymatically degrade organic matter and mitigate pollutants in wastewater (?). The procedure involves deliberate aeration of the wastewater to furnish requisite oxygen for the metabolic activities of the microorganisms, facilitating the breakdown of organic constituents. Concurrently, the microorganisms generate flocs—comprising microorganisms and organic matter—which sediment in a dedicated tank, forming a sludge layer subsequently reintroduced into the aeration tank for sustained treatment. The treated effluent undergoes controlled discharge or fur-

ther processing prior to environmental release. Recognized for its efficacy in organic matter, nutrient, and pollutant removal, the activated sludge method capitalizes on the enzymatic capabilities of microorganisms, thereby fostering a proficient purification mechanism for wastewater (Ohki and Rich [2019]).

2. Trickling Filter

The Trickling Filter Method represents a wastewater treatment modality predicated on the utilization of a biofilm to effectuate the removal of organic matter and contaminants. Operationalizing this method involves the systematic distribution of wastewater across a bed of solid media, including substrates such as rocks or plastic, within the trickling filter reactor (Liang et al. [2020]). The ensuing percolation of wastewater through the media prompts the development of a biofilm comprised of microorganisms, orchestrating the breakdown of organic matter into carbon dioxide, water, and ancillary byproducts. Notably, this biofilm acts as a substrate conducive to the proliferation of bacteria instrumental in nitrification and denitrification processes, thereby facilitating the removal of nitrogen compounds. Subsequent to traversing the media, the treated wastewater is methodically collected as effluent (Liang et al. [2020]).

3. Anaerobic Digestion

Anaerobic digestion, a method intrinsic to biological wastewater treatment, serves a dual mandate of pollution mitigation and the recuperation of resources. This process engages microbial consortia to effectuate the conversion of pollutants into inert byproducts while yielding valuable metabolites, notably methane and hydrogen. Positioned as a linchpin in the realization of a "zero-waste" society and a circular economy, anaerobic digestion demonstrates proficiency in the conversion of organic waste, including wastewater sludge, into biogas primarily comprised of methane and carbon dioxide (Kundu et al. [2021], Nabaterega et al. [2021]). Operationalized through two distinct stages, namely acidogenesis and methanogenesis, this process exhibits a capacity to break down complex organic compounds into simpler entities, such as volatile fatty acids (VFAs), which subsequently undergo conversion

into methane and carbon dioxide under the influence of methanogenic bacteria. Optimization of this process involves the implementation of a two-stage system, effectively segregating the acidogenesis and methanogenesis stages to exert precise control over operating conditions and enhance overall system performance (Nabaterega et al. [2021]).

I.2.4.3 Chemical Treatment

The method denoted as chemical treatment, expounded upon in the provided sources, constitutes a comprehensive strategy employed for the treatment of chemical wastewater, amalgamating a spectrum of chemical processes to effectually eliminate both organic and inorganic constituents. This method is characterized by the integration of physical-chemical and biological treatment paradigms, contributing to an elevated level of treatment efficacy (van Loosdrecht et al. [2016]). The procedural sequence encompasses a series of meticulously designed steps: initial rough filtration and precipitation for pretreatment, pre-oxidation treatment to condition the wastewater, micro-electrolysis treatment incorporating pH adjustment and aeration, re-oxidation treatment utilizing hydrogen peroxide to induce Fenton oxidation, activated sludge treatment, flocculating settling to facilitate the sedimentation of suspended particles, and deep treatment involving a membrane separator to yield water of heightened purity. The method, embodying a synergistic fusion of physical-chemical and biological mechanisms, adeptly addresses the nuanced challenge of impurity removal from chemical wastewater. This complex process involves primary and secondary filtering, a regulating reservoir to abate salt content, oxidation of ammonia nitrogen, pretreatment employing catalyzed iron inner electrolysis, equilibrium modulation of microorganism growth through magnetic powder addition, continuous phenol oxidation utilizing magnetic immobilized enzyme, and MBR membrane biochemical treatment, culminating in the generation of water meeting high-quality standards (van Loosdrecht et al. [2016], Ahmed et al. [2021]).

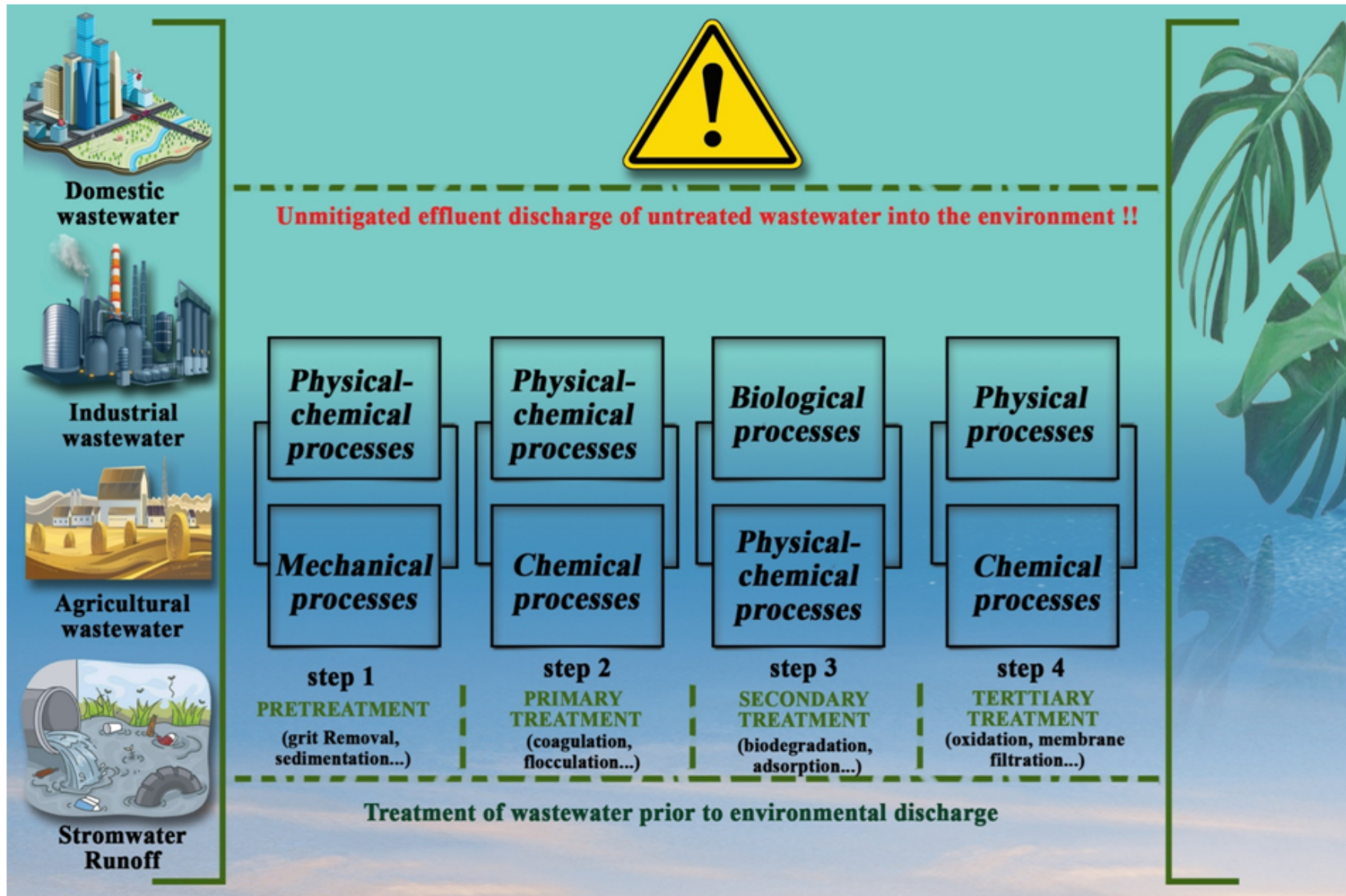


Figure I.6: Environmental discharge of untreated and treated wastewater.

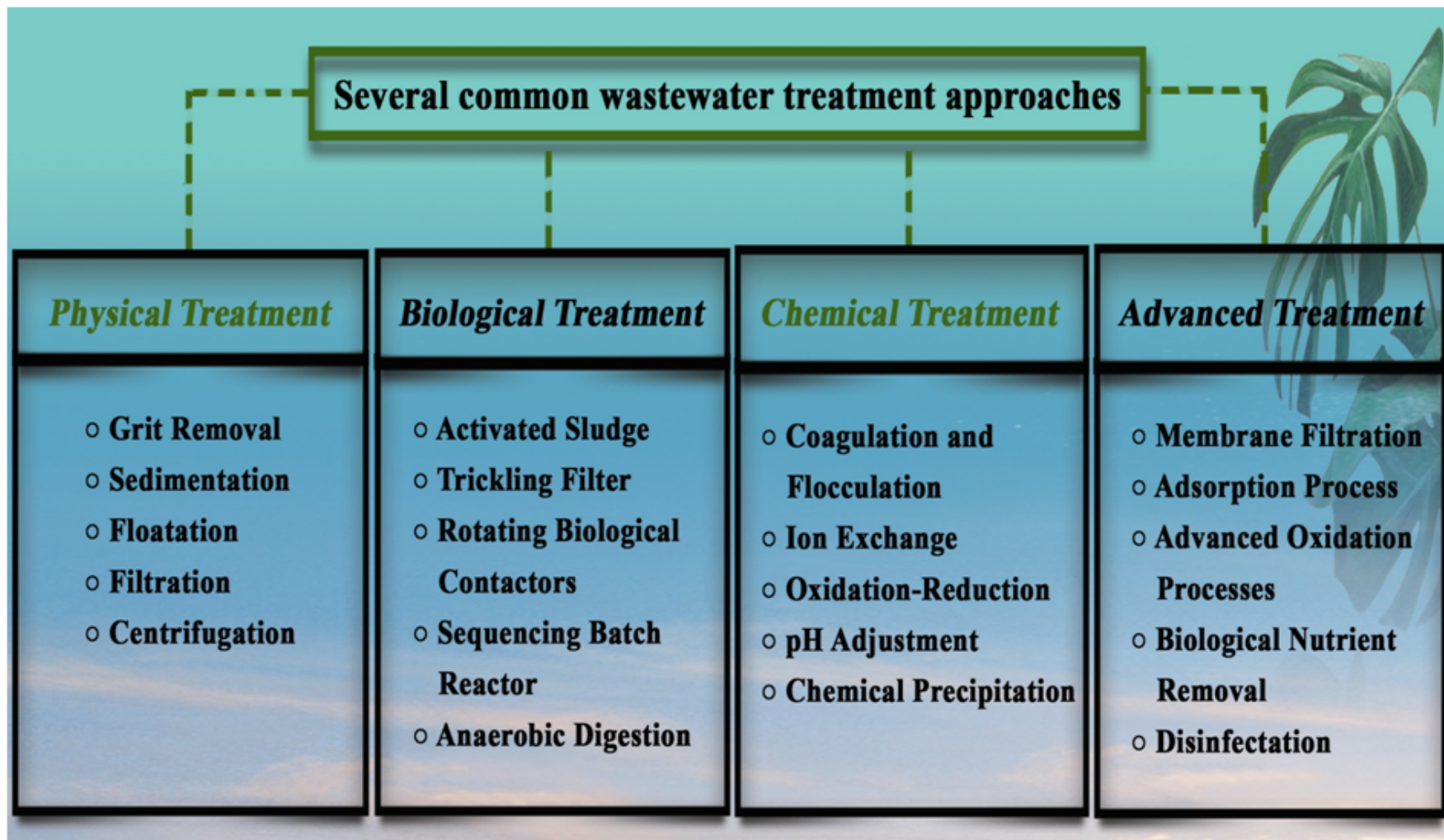


Figure I.7: Several common wastewater treatment approaches.

1. Coagulation and Flocculation

The Coagulation and Flocculation method emerges as a fundamental paradigm in water and wastewater treatment, strategically devised for the extraction of suspended particles and impurities from aqueous sources. Coagulation instigates this process by introducing chemicals, such as aluminum sulfate or ferric chloride, to induce particle destabilization, thereby precipitating the formation of larger aggregates denoted as flocs. Subsequently, the Flocculation stage involves judiciously stirring or mixing the water medium, instigating the collision and aggregation of destabilized particles, culminating in the genesis of larger and denser flocs conducive to settling or facile removal (Guimarães et al. [2020]). The synergistic interplay of these processes culminates in the effective elimination of suspended solids, turbidity, and certain dissolved organic constituents from the water milieu, concomitantly augmenting its translucency and overarching quality. Predominantly employed in wastewater treatment facilities and potable water treatment plants, this method substantively amplifies the operational efficiency of filtration and sedimentation mechanisms. In the specific context of wastewater discharge into surface water, Coagulation involves the addition of compounds such as ferric chloride and polymers to destabilize colloidal materials, thereby fostering the agglomeration of minute particles into larger, readily settleable flocs. Succeeding this, Flocculation, executed through gentle stirring, orchestrates the formation of enlarged flocs, streamlining their subsequent separation from the water matrix via sedimentation or filtration modalities. This method exhibits pronounced efficacy in the removal of heavy metallic ions, chemical contaminants, and turbidity endemic to industrial wastewater. The selection of compounds for coagulation-flocculation is intricately contingent upon the specific contaminants manifest in the wastewater and the desired treatment objectives. The omnipresence of Coagulation and Flocculation methodologies within the purview of water and wastewater treatment underscores its established efficacy in pollutant removal and the amelioration of water quality (Guimarães et al. [2020], Iwuozor [2019]).

2. Ion Exchange

The Ion Exchange Method stands as a pivotal water treatment process, delineated by the dynamic exchange of ions between a solid resin and the water subject to treatment. Leveraging ion exchange resins, typically manifested as diminutive beads or particles composed of polymer materials with appended functional groups, this method orchestrates the selective attraction and interchange of ions within the aqueous medium (Mohammed et al. [2018], Koliehova et al. [2019]). As the water permeates the resin bed, ions therein are magnetically drawn to the functional groups on the resin beads, instigating an exchange with counterpart ions present in the resin matrix. Released ions seamlessly integrate into the treated water, while undesired ions undergo sequestration by the resin. The operational efficiency of ion exchange is contingent upon variables encompassing resin mass, agitation duration, temperature, and inherent resin attributes, including moisture content and particle dimensions. Notably adept at abating pollutants such as sulfate, chloride, and sodium from wastewater, this method assumes particular prominence in its capacity for pollutant removal. Comprehensive investigations into kinetic and thermodynamic parameters are requisite to unravel the intricacies of the adsorption process, thereby facilitating the optimization of treatment efficiency (Koliehova et al. [2019]).

3. Oxidation-Reduction

The Oxidation-Reduction Method, colloquially known as the redox method, represents a chemical process characterized by the exchange of electrons between reactants. Within the realm of wastewater treatment, this method assumes a pivotal role in the elimination of contaminants (Lucas et al. [2021], Koliehova et al. [2019]). Post-oxidation, the introduction of a coagulant precipitates the formation of aggregates within the wastewater matrix, facilitating subsequent removal through the process of solid/liquid separation. Significantly, the Oxidation-Reduction Method distinguishes itself through its pronounced cost-effectiveness in comparison to conventional methodologies employed in wastewater treatment, thereby underscoring its efficacy as a resource-efficient solution for ad-

dressing water quality imperatives (Koliuhova et al. [2019]).

1.2.4.4 Advanced Treatment

Advanced Treatment in wastewater management represents a crucial stage of water purification, strategically introduced subsequent to primary, secondary, and tertiary treatment phases within conventional wastewater treatment frameworks (Gedda et al. [2021],Paliwal [2020]). Distinct from conventional methodologies such as ion exchange, thermal treatment, coagulation, electrochemical degradation, and chemical precipitation, which manifest certain inefficiencies in water purification, Advanced Treatment processes are meticulously designed to address specific pollutants. The deployment of these supplementary processes is orchestrated with the explicit goal of augmenting the quality of treated wastewater, thereby ensuring alignment with mandated standards for either conscientious discharge into the environment or judicious reuse (Paliwal [2020],Ul-Islam et al. [2023]). As an integral component of wastewater treatment, Advanced Treatment methodologies play an instrumental role in attaining elevated water quality benchmarks

1. Membrane Filtration

The Membrane Filtration Method stands as an advanced and pivotal facet within contemporary water and wastewater treatment paradigms, employing semi-permeable membranes to selectively segregate solids and contaminants from aqueous matrices (Ismail et al. [2022],Abdel-Fatah et al. [2021]). This method encompasses various filtration processes, including pressure-driven, osmotic-driven, electrical-driven, and thermal-driven modalities. Notably, the widely adopted pressure-driven variant involves the exertion of force to propel water through the membrane, facilitating the passage of clean water while sequestering solids and contaminants (Abdel-Fatah et al. [2021]). Alternatives leverage osmotic gradients, electric fields, or thermal differentials for analogous purposes. The efficacy of these processes is contingent upon diverse factors, encompassing membrane modules, concentration polarization, and the propensity for fouling. Despite the potential for fouling, membrane filtration remains instrumental in industrial water treatment. To ameliorate fouling con-

cerns, hybrid processes, such as membrane bioreactors and reverse osmosis, have been innovated, striving to augment resilience and curtail energy consumption in industrial wastewater treatment (Ismail et al. [2022], Abdel-Fatah et al. [2021], Bera et al. [2022]).

2. Disinfestation

The Disinfestation Method is a systematic approach to eradicate or control pests, specifically insects or rodents, within defined environments. In the context of water and wastewater treatment, disinfestation methods play a pivotal role in eliminating or regulating organisms that present potential risks to public health or environmental integrity. Electrochemical disinfection, emerging as a prominent method, involves the generation of chemical oxidants through redox reactions occurring at the surface of an electrode. Recent scholarly attention has been directed toward assessing the efficacy of this method in disinfecting water and wastewater, wherein its performance is intricately linked to variables such as water quality and operational parameters (Hand and Cusick [2021], Ragazzo et al. [2020], Ragazzo et al. [2013]).

3. Adsorption Process

The adsorption process, integral to contemporary wastewater treatment strategies, encompasses the adherence of contaminants to the surface of a designated solid substrate, identified as an adsorbent. Governed by physical principles, this mechanism relies on intermolecular forces whereby contaminants in aqueous solutions form attachments to the adsorbent's surface. Prevailing adsorbent materials encompass activated carbon, zeolites, clay minerals, or other porous substrates characterized by substantial surface areas. This characteristic porosity significantly augments the adsorption process's efficiency by affording an expansive surface for the attachment of contaminants, ensuring their efficacious removal from the water matrix. Notably versatile, the process extends its utility to an array of contaminants, spanning dyes, heavy metals, surfactants, personal care products, pesticides, and pharmaceuticals. Celebrated for its simplicity, cost-effectiveness, and ecological compatibility,

adsorption emerges as a sustainable and viable solution in wastewater treatment protocols. However, the continual evolution of this methodology necessitates sustained research and development initiatives, fostering optimization and adaptable integration across a diverse spectrum of wastewater treatment applications (Sales et al. [2019], Erdogan [2022], Rashid et al. [2021]).

Operationally, the adsorption process unfolds as contaminants in the aqueous milieu interact with the surface of the adsorbent, forming attachments via weak intermolecular forces. The pronounced porosity of the adsorbent material, a pivotal aspect, substantiates a larger surface area, thereby amplifying the efficacy of the adsorption process. Notably effective against an extensive repertoire of contaminants, including organic compounds, heavy metals, and dyes, the process exhibits particular prowess in addressing soluble and insoluble organic constituents. Post adsorption, separation of water from the adsorbent is executed through filtration or analogous separation methodologies, facilitating the extraction of adsorbed contaminants from the aqueous medium. The intrinsic advantage of adsorption resides in the regenerative capacity and the potential for multiple uses of the adsorbent material, bolstering the economic viability of adsorption as a sustainable and recurrent facet within wastewater treatment paradigms. While acknowledged for its fundamental robustness, the adsorption process is subject to continual refinement through ongoing research endeavors, which aim to delineate nuanced applications and elevate performance standards across diverse and dynamic wastewater treatment scenarios (Rashid et al. [2021]).

I.2.5 Conclusions

In conclusion, the discerning selection of the adsorption process as the focal method for wastewater treatment is underscored by its manifold advantages, cementing its status as a cornerstone in contemporary remediation strategies. The process stands out for its exceptional versatility, exhibiting efficacy across a spectrum of pollutants encompassing organic compounds, heavy metals, dyes, surfactants, pharmaceuticals, and more. This attribute renders it applicable to a wide array of

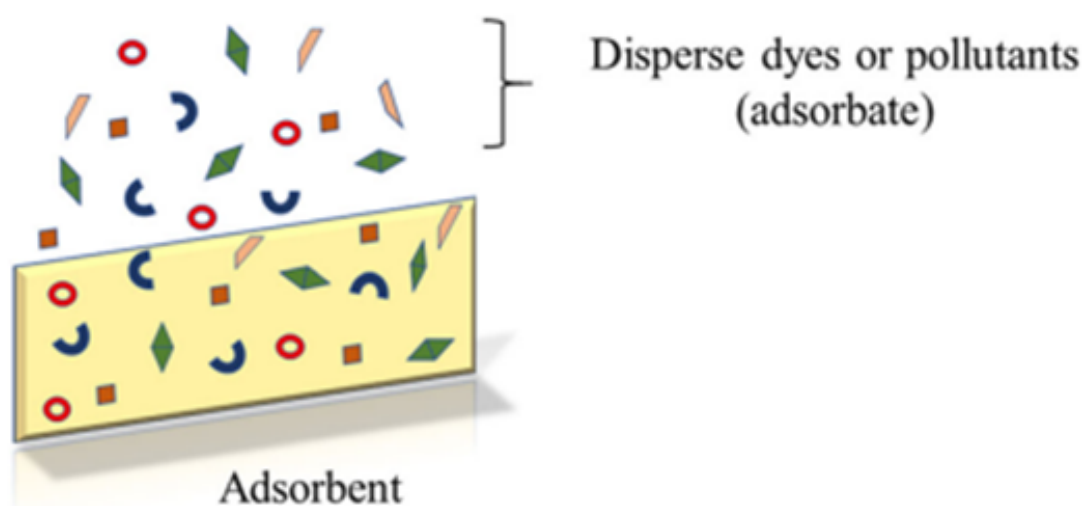


Figure I.8: General mechanism of adsorption to remove contaminants (Rashid et al. [2021]).

industrial and municipal wastewater treatment contexts. The physical mechanism of adsorption, facilitated by the porous structure of adsorbent materials, endows it with a notable removal efficiency, enhancing its performance in capturing and immobilizing diverse pollutants. Notably, the economic sustainability of adsorption is accentuated by the regenerability and reusability of adsorbent materials, contributing to the mitigation of operational costs and aligning with imperatives of resource conservation.

Environmental considerations further commend the adoption of adsorption, given its propensity to entail minimal chemical usage, thereby minimizing the chemical footprint associated with wastewater treatment. The operational simplicity and seamless integration of adsorption into existing treatment infrastructures contribute to its pragmatic utility and cost-effectiveness. Its scalability ensures adaptability to varying scales of application, from decentralized systems to large-scale industrial treatment plants, underscoring its relevance across a spectrum of operational contexts. Moreover, the energy-efficient nature of adsorption, with lower energy inputs compared to certain alternative advanced treatment methodologies, enhances its standing in the wastewater treatment paradigm.

Crucially, the selective removal capability of adsorption allows for the customization of treatment strategies, aligning them with specific contaminants based on the choice of adsorbent material. The process's amenability to integrate into hybrid systems further expands its utility, allowing synergies with other treatment methodologies to comprehensively address specific challenges. In light of these considerations, the judicious selection of the adsorption process over alternative methods is contingent upon meticulous evaluation of wastewater characteristics, treatment objectives, and operational exigencies. Thus, the adsorption process emerges not merely as a singular technique but as a dynamic and adaptable cornerstone, offering a tailored and sustainable approach to contemporary wastewater treatment.

I.3 Adsorption on Carbonaceous materials

I.3.1 Generalities

Adsorption on carbonaceous materials represents a pivotal facet in contemporary wastewater treatment strategies, prominently featuring activated carbon formulations such as granular activated carbon (GAC) and powdered activated carbon (PAC). These carbonaceous materials, characterized by a porous structure boasting a high surface area ranging from 500 to 1500 m^2g^{-1} , serve as robust agents for the adsorptive removal of contaminants from water. The adsorption process, a cardinal mechanism wherein pollutants in water selectively adhere to and accumulate on the surface of activated carbon, unfolds due to the presence of diverse functional groups such as hydroxyl, carboxyl, and phenolic groups on the carbon surface. These groups facilitate interactions with pollutants through a combination of physical and chemical forces, thereby effectuating the transfer of pollutants from the liquid phase to the solid phase of the activated carbon (Rashid et al. [2021], Barua et al. [2021], Sherugar et al. [2022]).

Crucially, the efficacy of adsorption on carbonaceous materials is intricately influenced by a gamut of factors, including the concentration of pollutants, contact time, temperature, pH, and the nuanced surface properties of the activated carbon.

This process stands as a versatile remediation mechanism, demonstrating prowess in the removal of an expansive array of organic pollutants such as dyes, hormones, phenols, pharmaceuticals, aromatic compounds, herbicides, pesticides, and industrial by-products. Moreover, the dynamic landscape of wastewater treatment has witnessed a recent integration trend wherein adsorption onto activated carbon harmonizes with biological treatment processes, giving rise to innovative techniques like powdered activated carbon membrane bioreactors (PAC-MBR) and biological activated carbon reactors (BAC). These hybrid systems ingeniously leverage the adsorption capacity of activated carbon synergistically with the biological degradation capabilities of microorganisms, presenting a holistic approach to wastewater treatment (Barua et al. [2021], Sherugar et al. [2022]).

The purview of adsorption on carbonaceous materials extends beyond activated carbon, encompassing diverse carbon-based substrates with high surface area and pore volume. This category includes materials derived from biomass, exemplified by activated carbon sourced from the mangrove fruit of *Rhizophora mucronata*, which has showcased exceptional efficacy in heavy metal and dye removal owing to its substantial surface area and pore volume. The adsorption kinetics of carbonaceous materials are aptly described by models such as the Langmuir and Freundlich models, offering insights into the intricate mechanisms and efficiency of adsorption processes (Hussain et al. [2021], Ambreen et al. [2021]).

Furthermore, within the realm of low-cost alternatives to conventional activated carbon, carbonaceous materials derived from agricultural and industrial waste have emerged as promising candidates. Hazelnut shell activated carbon, for instance, has been investigated for its potential in removing heavy metals like chromium from aqueous solutions. This underscores the pragmatic shift towards sustainable and cost-effective adsorbents in the quest for efficient wastewater treatment solutions. Mechanistic studies into heavy metal removal by various types of activated carbons, notably carbonaceous materials, have contributed to a nuanced understanding of their adsorptive capabilities, paving the way for optimized and tailored applications in diverse wastewater treatment scenarios (Sherugar et al.

[2022], Hussain et al. [2021], Ambreen et al. [2021]).

In a more futuristic vein, the exploration of nanocarbon-based aerogels introduces a realm of materials with exceptionally high surface areas and porosity, rendering them highly effective for adsorption processes. The intricate dance between accessible surface area and adsorption dynamics defines the efficacy of nanocarbon-based aerogels in selectively capturing and immobilizing water pollutants, ranging from heavy metal ions to dyes. The adsorption capacity of carbonaceous materials in these advanced forms is meticulously influenced by characteristics such as surface area, pore size, and volume, underscoring the need for a nuanced understanding of material properties for optimal deployment in wastewater treatment strategies (Barua et al. [2021], Sherugar et al. [2022], Hussain et al. [2021], Ambreen et al. [2021]).

I.3.2 Adsorption on Carbonaceous materials

Adsorption on carbonaceous materials epitomizes a pivotal facet within contemporary wastewater treatment methodologies, characterized by the preeminent usage of activated carbon as an adept adsorbent. This method, rooted in the physical adherence of contaminants to the surface of activated carbon, delineates an intricate process influenced by a confluence of factors. Paramount among these are the molecular structure, solubility, ionization, and temperature, alongside the inherent composition of adsorbed substances. The expansive domain of carbonaceous adsorbents encompasses a spectrum of influences, including the type of adsorbent, temperature, mineral content, concentration of oxidizing gas, and intrinsic chemical properties. Activated carbon, a vanguard in this realm, manifests a robust affinity for binding organic substances, thus presenting itself as a potent tool for the treatment of organic-laden wastewater (Saravanan et al. [2022], Hardyanti et al. [2023]).

These adsorbents, hewn from diverse raw materials such as waste tyres, banana trunks, tea leaves, and date seeds, undergo synthesis to yield activated carbon. The judicious selection of these source materials crucially modulates the quality of adsorption, with waste tyres, in particular, emerging as noteworthy for their

elevated surface area and pore volume. The subtleties of moisture content in the adsorbent emerge as a critical determinant of adsorption efficiency, wherein materials with diminished moisture content, like waste tyres, furnish augmented surface area for adsorption, amplifying their efficacy in removing contaminants from wastewater (Hardyanti et al. [2023]).

The strategic manipulation of dosage concentration, a cardinal parameter in the adsorption process, dynamically dictates its efficiency. Elevated concentrations of activated carbon in wastewater orchestrate a proportional increase in available surface area, thereby facilitating the removal of dissolved solids. This nuanced orchestration of factors encapsulates the intricate landscape of adsorption on carbonaceous materials, delineating a complex interplay that governs the adsorption capacity and efficiency of these materials (Saravanan et al. [2022], Hardyanti et al. [2023]).

In essence, adsorption on carbonaceous materials, with activated carbon as its vanguard, crystallizes as a sophisticated mechanism within wastewater treatment. This approach, characterized by the inherent predilection of activated carbon for organic substances, articulates a nuanced paradigm with demonstrable efficacy across diverse wastewater compositions. As a fundamental definition within the wastewater treatment lexicon, adsorption on carbonaceous materials underscores both its versatility and efficacy as an integral facet of contemporary wastewater treatment methodologies.

I.3.2.1 Activated Carbonaceous materials

Activated Carbonaceous materials stand as an illustrious archetype within the realm of adsorbent materials, bearing intrinsic characteristics that render them paramount in applications spanning water and air pollutant removal across diverse industries. Distinctively porous and endowed with enhanced adsorption capabilities, these materials are the cornerstone of myriad industrial processes. Synthesized from an array of precursors including fossil fuels, agricultural wastes, and lignocellulosic residues, the production of Activated Carbonaceous materials involves a so-

phisticated tandem of carbonization and activation processes. The precursor undergoes carbonization at elevated temperatures, expunging volatile components, followed by activation to augment pore size and surface area (Hardyanti et al. [2023], Sharma et al. [2022], Ofomata et al. [2022]).

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The synthesis of activated carbon involves the intricate manipulation of precursors, among them coconut shells, employing the activation process to instill the material with the requisite attributes. This treatment furnishes the activated carbon with an advantageous crystalline structure and extensively developed internal pore configurations, culminating in materials that exhibit remarkable adsorption capacities. Activation emerges as a pivotal process, wherein carbonaceous precursors are meticulously subjected to elevated temperatures, imparting upon them the distinctive features that render activated carbonaceous materials integral to diverse industrial landscapes (Hardyanti et al. [2023], Sharma et al. [2022], Ofomata et al. [2022], Ansari and Sayem [2022]).

Activated carbonaceous materials, drawn from a diverse spectrum of sources encompassing bituminous coal, bones, coconut shells, lignite, peat, pecan shells, petroleum-based wastes, pulp mill black ash, sugar, wastewater treatment sludge, and wood, embody the epitome of high specific surface area and micropore volume. These structural attributes underpin their efficacy as chemical adsorbents, facilitat-

ing the retention and removal of a spectrum of chemical species. The high specific surface area and micropore volume of these materials engender a propitious environment for chemical interactions, making them adept at adsorbing diverse pollutants and contaminants (Saravanan et al. [2022], Hardyanti et al. [2023], Sharma et al. [2022], Ofomata et al. [2022], Ansari and Sayem [2022]).

Beyond the purview of environmental applications, the multifarious deployment of activated carbonaceous materials spans water purification, food-grade products, cosmetics, automotive applications, industrial gas purification, petroleum refining, and precious metal recovery. Their exceptional versatility is further highlighted in gold recovery processes, attesting to their robust adsorptive capabilities across precious metal domains. In these applications, activated carbonaceous materials emerge not merely as cost-effective adsorbents but as dynamic entities with athletic and catalytic propensities, navigating a broad spectrum of industries with discernible efficacy (Saravanan et al. [2022], Hardyanti et al. [2023], Sharma et al. [2022], Ofomata et al. [2022], Ansari and Sayem [2022]).

In summation, Activated Carbonaceous materials represent an apotheosis of adsorbent materials, meticulously synthesized to embody exceptional porosity, expansive surface area, and discernible sorption capabilities. Their versatile applications, spanning from environmental remediation to catalytic support and diverse industrial sectors, underscore their indispensability in contemporary material science. The intricacies of their synthesis processes, rooted in carbonization and activation, delineate a sophisticated paradigm, affirming their status as stalwarts in the pursuit of sustainable and effective adsorption.

1. Commercial Activated Carbonaceous materials

Commercial Activated Carbonaceous epitomizes a pivotal facet within the ambit of activated carbon, representing a commercially available form that finds widespread application across diverse industrial domains. Renowned for its profound porosity and expansive surface area, this variant of activated carbon stands as a stalwart in the realm of adsorption, particularly for the purification of gases and liquids. The nuanced efficacy of commercial activated

carbonaceous materials is underscored by their ability to adsorb and eliminate impurities, a characteristic that aligns seamlessly with the stringent purification demands of various industries (Ansari and Sayem [2022], Lewoyehu [2021]). The defining attributes of commercial activated carbonaceous materials, notably their specific surface area and porosity, are subject to a dynamic interplay of factors intricately linked to the manufacturing processes and the envisaged applications. The commercial landscape offers a spectrum of these materials, each tailored to meet the distinct requirements of specific industries (Lewoyehu [2021]). Consequently, the specific surface area and porosity become pivotal parameters that delineate the performance of commercial activated carbonaceous materials. The variability in these characteristics is intricately tied to the methods employed in their production, serving as a testament to the tailored nature of these materials for diverse applications.

It is imperative to recognize that the nuanced intricacies of commercial activated carbonaceous materials may result in variations in their specific surface area, porosity, and adsorption performance when juxtaposed with their counterparts described in scholarly sources. The commercial viability of these materials often necessitates optimization for specific industrial applications, a process that may entail compromises in certain structural attributes for enhanced practical utility. Therefore, stakeholders navigating the expansive landscape of commercial activated carbonaceous materials must be cognizant of the contextual nuances that shape their specific attributes and performance characteristics.

In summation, Commercial Activated Carbonaceous materializes as a dynamic and commercially viable iteration of activated carbon, celebrated for its porous architecture and significant surface area. This commercial variant, shaped by diverse manufacturing processes, aligns seamlessly with the rigorous demands of industries reliant on adsorption for the purification of gases and liquids. The nuanced variations in specific surface area and porosity, inherent to the manufacturing intricacies, are emblematic of the tailored

nature of these materials for multifaceted industrial applications. Recognition of these contextual intricacies becomes paramount for stakeholders seeking to harness the full spectrum of commercial activated carbonaceous materials in diverse industrial settings.

2. Alternative Activated Carbonaceous materials

Alternative Activated Carbonaceous materials represent a paradigm shift in the production of activated carbon, offering sustainable and cost-effective alternatives to conventional precursors like coal, wood, and coconut shell. These materials serve as promising substitutes, particularly advantageous in regions with limited access to traditional precursor sources. A pantheon of unconventional materials has been explored for this purpose, each presenting unique advantages and potential applications (Ansari and Sayem [2022], Lewoyehu [2021], Kosheleva et al. [2018]).

Olive-waste cakes, derived from agricultural by-products, stand as a noteworthy exemplar in this cadre. The olive-waste cakes, abundantly available from olive processing, exhibit potential as green precursors for activated carbon synthesis. Their integration into the realm of alternative activated carbonaceous materials aligns with the broader ethos of sustainability, harnessing agricultural residues for value-added applications (Kosheleva et al. [2018], Zubrik et al. [2017]).

Cattle-manure compost emerges as another distinctive member of this alternative cohort. Originating from agricultural waste, cattle-manure compost demonstrates versatility as a precursor for activated carbon production. The recycling of organic waste materials in this manner not only addresses environmental concerns associated with waste disposal but also contributes to the synthesis of activated carbon through a more sustainable paradigm (Kosheleva et al. [2018], Zubrik et al. [2017]).

Bamboo materials, owing to their fast growth and renewable nature, constitute a compelling alternative. Bamboo, a resilient and abundant resource,

finds application as a precursor for activated carbon synthesis. This not only taps into the potential of a rapidly renewable resource but also diversifies the precursor base, enhancing the sustainability quotient of the activated carbon production process (Kosheleva et al. [2018],Zubrik et al. [2017]).

Apple pulp, a by-product of the apple processing industry, emerges as an unconventional yet viable candidate. Leveraging an industrial by-product for activated carbon production aligns with principles of circular economy, repurposing waste streams into valuable materials. Apple pulp, in this context, offers a dual benefit of waste reduction and activated carbon synthesis (Kosheleva et al. [2018],Zubrik et al. [2017]).

Potato peel, a waste product from potato processing, joins the league of alternative precursors. This agricultural residue, often overlooked, reveals promise as a precursor for activated carbon. Its inclusion in the repertoire of alternative activated carbonaceous materials not only addresses waste management concerns but also opens avenues for sustainable production practices (Kosheleva et al. [2018],Zubrik et al. [2017]).

Expanding the gamut, alternative activated carbonaceous materials also include crop straw. Rice straw or wheat straw, conventional agricultural residues, find novel application in the synthesis of modified activated carbon. The utilization of crop straw broadens the scope of precursor materials, integrating diverse agricultural by-products into the activated carbon production process (Kosheleva et al. [2018],Zubrik et al. [2017]).

Tea dregs, the residue left after brewing tea, present another unconventional yet viable option. This carbonaceous material, often treated as waste, assumes a new identity as a raw material for modified activated carbon. The conversion of tea dregs into activated carbon contributes to the valorization of waste streams, aligning with sustainable practices (Kosheleva et al. [2018],Zubrik et al. [2017]).

Peanut shells and chestnut shells, both regarded as agricultural waste materials, further enrich the array of alternative precursors. These materials, often overlooked in traditional contexts, become instrumental in the preparation of modified activated carbon. The integration of peanut shells and chestnut shells broadens the repertoire of alternative activated carbonaceous materials, tapping into diverse agricultural residues (Kosheleva et al. [2018], Zubrik et al. [2017]).

In essence, these alternative activated carbonaceous materials represent a paradigmatic departure from conventional precursor sources. Their adoption underscores a shift towards sustainable and diversified sources for activated carbon production. By repurposing agricultural residues and industrial by-products, this alternative paradigm aligns with principles of circular economy, waste valorization, and environmental stewardship. These materials, often derived from abundant and renewable sources, present viable alternatives for regions facing constraints in accessing traditional precursors. As the landscape of activated carbon production evolves, the inclusion of these alternative materials offers a nuanced and sustainable trajectory for the synthesis of this invaluable adsorbent.

I.3.2.2 Biochar derived carbonaceous materials

Biochar-derived carbonaceous materials represent a class of solid carbonaceous products derived from the pyrolysis or thermal decomposition of diverse biomass sources such as agricultural residues, wood, sewage sludge, waste products, litters, and animal manures under controlled conditions with limited oxygen. This versatile category of carbon materials, often referred to as biochar, exhibits unique properties that render them suitable for a myriad of applications across diverse industries. The distinctive attributes of biochar-derived carbonaceous materials encompass a mesoporous structure, high surface area, porosity, and functional groups, offering a valuable suite of characteristics for applications in soil remediation, hazardous contaminant removal, wastewater treatment, electrode materials, and catalysis (Zubrik et al. [2017], Sarmah et al. [2023], Vinodha et al. [2022], Tian et al. [2022], Wijitkosum

[2022]).

Biochar, as a parent material, derives its properties from the controlled pyrolysis process, ensuring that organic matter undergoes thermal decomposition under limited oxygen conditions. The resulting carbonaceous materials are characterized by their mesoporous structure, a critical feature contributing to their high surface area and adsorption capabilities (Sarmah et al. [2023], Vinodha et al. [2022], Tian et al. [2022]). The biochar-derived carbonaceous materials have demonstrated efficacy in soil remediation by influencing the migration and transformation of soil elements and pollutants. Their specific physicochemical properties, such as surface area, porosity, and functional groups, position them as effective sorbents for the removal of toxic chemicals, including organic dyes, heavy metals, and phenolic compounds in wastewater treatment (Tian et al. [2022], Wijitkosum [2022], Seow et al. [2022], Remmani et al. [2021]).

While biochar presents inherent advantages, including sustainability and suitability for diverse applications, its heterogeneity, low porosity, and limited adsorption capacity necessitate refinement. Various physical and chemical techniques are employed to enhance the physicochemical properties of biochar, expanding its potential applications. Techniques like steam activation, a physical modification method, have demonstrated the ability to augment the properties of biochar without the use of additional chemicals. Nanoscale engineering techniques, such as ball milling, microwave irradiation, and magnetic modification, represent innovative approaches to fabricate biochar-derived carbonaceous materials into nano-sized forms, overcoming the limitations associated with bulk biochar. This nanoscale modification enhances stability and efficiency, opening avenues for advanced waste management techniques (Tian et al. [2022], Wijitkosum [2022], Seow et al. [2022]).

Biochar-derived carbonaceous materials extend their versatility into electrode materials for batteries, including lithium-ion batteries, sodium-ion batteries, lithium-sulfur batteries, and Zn-air batteries. Their unique properties, including high surface area and porosity, contribute to their effectiveness as electrode materials, facilitating energy storage and retrieval in various battery configurations. Addi-

tionally, these carbon materials serve pivotal roles as catalysts and catalyst supports in fuel cells, further expanding their utility in sustainable energy applications (Wijitkosum [2022], Seow et al. [2022]).

The application range of biochar-derived carbonaceous materials, while expansive, underscores the ongoing need for improvement and optimization. The inclusion of alternative precursors, such as agricultural waste, food waste, and industrial waste, in the pyrolysis process broadens the repertoire of biochar-derived carbonaceous materials. These materials, rich in organic matter, have the potential to contribute to sustainable waste management practices by repurposing various waste streams into valuable carbon materials (Sarmah et al. [2023], Vinodha et al. [2022], Tian et al. [2022], Wijitkosum [2022], Seow et al. [2022]).

The characterization of biochar-derived organic matter becomes imperative for tailoring these materials to specific applications. The choice of extraction solvent, including dichloromethane, acetone, methanol, and water, influences the molecular characteristics of the organic matter extracted. This nuanced approach to characterizing biochar-derived organic matter aids in selecting appropriate extractants for analysis, contributing to the theoretical basis for biochar-based soil remediation (Vinodha et al. [2022], Tian et al. [2022], Wijitkosum [2022], Seow et al. [2022]).

In conclusion, biochar-derived carbonaceous materials, arising from the pyrolysis of diverse biomass sources, stand as versatile and sustainable carbon materials with applications ranging from soil remediation to advanced energy storage. Their mesoporous structure, high surface area, and functional groups contribute to their efficacy in adsorption, making them invaluable for environmental remediation. The application of physical and chemical techniques for modification, especially at the nanoscale, enhances their properties, addressing the limitations associated with bulk biochar. The incorporation of alternative precursors further diversifies the biochar-derived carbonaceous materials, contributing to sustainable waste management practices. As research in this field progresses, a nuanced understanding of the molecular characteristics and optimal applications of biochar-derived carbonaceous

materials will continue to drive advancements in environmental and energy-related domains.

The choice between biochar-derived carbonaceous materials and activated carbonaceous materials for the adsorption process in wastewater treatment involves a nuanced consideration of their respective properties, advantages, and application contexts. Both materials possess unique characteristics that contribute to their efficacy in adsorption processes, yet the selection depends on specific treatment goals, economic considerations, and sustainability concerns.

Activated carbonaceous materials, renowned for their high porosity and large surface area, have long been the stalwart in adsorption applications. Their extensive micropore structure allows for efficient adsorption of a wide range of contaminants, including organic dyes, heavy metals, and various pollutants present in wastewater. Activated carbon exhibits excellent adsorption capacity and is particularly effective in treating industrial effluents with diverse and complex compositions. However, the high cost associated with the production and regeneration of activated carbon, along with concerns about its sustainability, has led researchers to explore alternative materials that offer comparable or superior performance.

Biochar-derived carbonaceous materials emerge as a viable and sustainable alternative to activated carbon in wastewater treatment adsorption processes. The pyrolysis of biomass sources results in biochar with a mesoporous structure, high surface area, and functional groups conducive to adsorption applications. One significant advantage lies in the sustainable and renewable nature of the precursors, such as agricultural waste and wood residues, contributing to a more environmentally friendly approach. The lower production costs associated with biochar, compared to activated carbon, make it an economically attractive option, especially in large-scale wastewater treatment operations.

Furthermore, the diverse range of biomass precursors available for biochar production allows for tailoring its properties to specific adsorption requirements. This adaptability ensures that biochar-derived carbonaceous materials can be opti-

mized for the removal of particular contaminants, providing a level of customization that is advantageous in addressing the unique challenges posed by different wastewater compositions. The mesoporous structure of biochar, though distinct from the microporous structure of activated carbon, offers substantial surface area for adsorption, and the modification of biochar at the nanoscale enhances its efficiency, overcoming limitations associated with bulk biochar.

In terms of sustainability, biochar production not only repurposes agricultural and industrial waste but also aids in carbon sequestration when applied to soils, contributing to broader environmental benefits. The integration of biochar-derived carbonaceous materials into wastewater treatment aligns with the principles of circular economy and sustainable resource management, making it an appealing choice for environmentally conscious applications.

In conclusion, the selection between biochar-derived carbonaceous materials and activated carbonaceous materials for wastewater treatment adsorption processes involves a meticulous evaluation of their respective attributes. While activated carbon remains a robust and effective adsorbent, the growing emphasis on sustainability, cost-effectiveness, and customization favors the adoption of biochar-derived materials. The ability to repurpose waste biomass, coupled with the lower economic burden, positions biochar as a promising alternative that aligns with contemporary environmental and economic imperatives. As research in this field progresses, continued optimization of biochar production processes and a deeper understanding of its adsorption mechanisms will likely enhance its role in shaping the future of wastewater treatment.

I.3.3 Adsorption of Volatile Organic Compounds

I.3.3.1 Volatile Organic Compounds

Volatile Organic Compounds (VOCs) constitute a complex and diverse array of organic chemicals characterized by their low boiling points and high vapor pressures at room temperature. These compounds, present in both natural and anthropogenic sources, play a significant role in the atmospheric and indoor environments.

Their diverse applications in various industries, such as solvents, base raw materials, and chemical processing, underscore their ubiquity in contemporary society (Chiang and Gao [2022], Jianyin and Huang [2022]).

Within the realm of VOCs, numerous chemical species contribute to this group. Examples include ketones, such as acetone and methylethyl ketone, alkanes exemplified by hexane, alcohols including ethanol, and aromatics such as benzene and toluene. The collective impact of VOCs on human health and the environment is profound. Beyond their versatile industrial applications, these compounds pose inherent risks due to their potential for toxicity and carcinogenicity (Jianyin and Huang [2022], Li et al. [2015]).

VOCs often find release into the environment through various processes, particularly from commonplace sources like furniture and building materials. This emission contributes to indoor air pollution, rendering VOCs a prominent focus of research in the fields of indoor environments and public health. As such, the mitigation of adverse health effects stemming from exposure to VOCs necessitates a comprehensive understanding of their properties, hazards, and influencing factors (Jianyin and Huang [2022]).

The adverse health effects associated with exposure to VOCs are well-documented and vary depending on the specific chemical species involved. While short-term exposure may lead to symptoms such as eye, nose, and throat irritation, long-term exposure has been linked to more serious health concerns, including damage to the liver, kidneys, and the central nervous system. Additionally, certain VOCs are recognized as carcinogens, underscoring the importance of stringent guidelines and measurement methods to assess their concentrations and emission characteristics (Jianyin and Huang [2022], Li et al. [2015]).

In response to the potential risks posed by VOCs, guidelines and standards have been developed to monitor and regulate their presence. These guidelines are essential for assessing air quality, particularly in enclosed spaces where concentrations can accumulate. Measurement methods involve sophisticated analytical tech-

niques that enable the accurate detection and quantification of VOCs. Gas chromatography, mass spectrometry, and infrared spectroscopy are among the tools employed in environmental monitoring, occupational safety, healthcare, and disease diagnosis (Jianyin and Huang [2022]).

Addressing the complex nature of VOCs also requires an examination of their contribution to air pollution. These compounds, released into the atmosphere, can undergo photochemical reactions to form secondary pollutants, including ground-level ozone and particulate matter. The role of VOCs in air pollution is particularly significant in urban areas where industrial activities, vehicular emissions, and residential sources collectively contribute to their release (Li et al. [2015]).

To effectively mitigate the adverse effects of VOCs on human health, understanding the factors influencing their emission and concentration is imperative. The emission characteristics of VOCs depend on a multitude of variables, including temperature, humidity, and ventilation rates. Additionally, the type of material emitting VOCs, the specific chemical composition of the VOC mixture, and the duration of exposure all play crucial roles in determining the potential health risks (Chiang and Gao [2022], Jianyin and Huang [2022], Li et al. [2015]).

In conclusion, VOCs represent a diverse group of organic compounds with far-reaching implications for human health and environmental quality. The multifaceted nature of these compounds, originating from both natural and anthropogenic sources, demands a comprehensive understanding of their properties and impacts. Ongoing research, coupled with stringent guidelines and advanced measurement methods, remains critical for monitoring and regulating VOC concentrations, thereby safeguarding public health and environmental well-being.

1.3.3.2 Adsorption of VOCs

The adsorption of VOCs represents a critical process in mitigating the deleterious effects of these harmful compounds on both the environment and human health. VOCs, pervasive air pollutants stemming from industrial chemical processes, pose significant risks to the well-being of ecosystems and individuals alike. The process

of adsorption, a fundamental mechanism, involves the adherence of VOC molecules to the surface of a solid material, referred to as an adsorbent, through weak intermolecular forces (Siu et al. [2023]). Distinct classes of VOCs, including aliphatic, aromatic, oxygenated, and sulfur-containing compounds, exhibit varied mechanisms of capture when subjected to alternative adsorbents. This variability arises from the diverse molecular structures and chemical properties inherent to each class of VOC. Aliphatic VOCs, characterized by straight or branched carbon chains, may engage in adsorption through van der Waals forces, whereby transient dipoles induce attraction between molecules. In contrast, aromatic VOCs, featuring cyclic structures, may form $\pi - \pi$ interactions with adsorbents, while oxygenated compounds, which include alcohols and ketones, can participate in hydrogen bonding or dipole-dipole interactions. Sulfur-containing VOCs, such as mercaptans and sulfides, may exhibit unique interactions based on the electron-donating or electron-withdrawing nature of sulfur atoms. The adsorption process provides an effective means of capturing and sequestering these diverse VOCs, preventing their uncontrolled release into the environment (Siu et al. [2023], Xiao et al. [2023]).

In essence, the adsorption of VOCs onto solid adsorbents is a nuanced process that intricately depends on the specific characteristics of both the VOCs and the adsorbent material. This understanding is crucial for the development of efficient adsorption systems tailored to different VOC classes, contributing to the broader goal of mitigating the environmental and health hazards posed by these compounds.

I.3.4 Research Gaps and Justification

The research endeavors embarked upon in this thesis are characterized by a comprehensive exploration of novel facets surrounding the preparation and application of biochar (BC) derived from date palm seeds in the Biskra region of Algeria. At the heart of this scientific pursuit lies the innovative utilization of Response Surface Methodology (RSM) for the optimization of BC preparation. RSM serves as a sophisticated and systematic framework, enabling the intricate dissection of multifaceted parameters in the preparation process. This deliberate departure from

conventional methodologies ensures the nuanced optimization of BC, specifically tailored to the unique attributes of date palm seeds from the Biskra region. This groundbreaking approach injects a fresh methodological perspective into the preparation of BC, thereby contributing to the evolving landscape of sustainable adsorbents.

The centrality of this research is underscored by its profound focus on adsorption processes facilitated by carbonaceous materials, with a particular emphasis on BCs derived from date palm seeds. This strategic emphasis underscores a deliberate alignment with the ethos of sustainability, as the investigation champions the utilization of locally sourced, agricultural waste-derived biochar for wastewater treatment. In adopting date palm seeds as a precursor, this research strategically aligns itself with the environmental and economic realities of the Biskra region, thereby cultivating an indigenous approach to adsorbent development. The academic discourse has thus far recognized the efficacy of various carbonaceous materials; however, this research elevates the dialogue by spotlighting the untapped potential of biochar sourced from a distinct regional biomass, thereby transcending the established paradigms of adsorbent preparation.

A salient feature of this research lies in its dedicated exploration of Volatile Organic Compounds (VOCs) as a contemporary case study, positioned in deliberate juxtaposition to the traditional examination of classic organic pollutants. The inclusion of VOCs as a focal point stems from their recognition as pervasive and deleterious pollutants in contemporary environments. This deliberate shift in focus reflects an astute responsiveness to evolving environmental challenges. While classic organic pollutants have been the subject of extensive study, the distinct characteristics and challenges posed by VOCs demand dedicated inquiry. This strategic alignment with the forefront of environmental challenges further propels this research into the realm of pioneering studies.

Moreover, the originality of this research is manifest in its innovative application of BCs as a potent medium for VOC removal. VOCs, owing to their volatility and potential health implications, represent a distinct class of pollutants, necessitat-

ing nuanced remediation strategies. In this context, the research casts a pioneering spotlight on the application of BCs as an effective adsorbent for VOCs. The novelty here resides not merely in the exploration of biochar as an adsorbent but in its specific application to the uniquely challenging realm of VOC removal. In doing so, the research extends beyond the conventional utility of biochar, demonstrating its versatility and efficacy in addressing contemporary environmental challenges.

In conclusion, this research project significantly contributes to the extant knowledge base by addressing a nexus of critical research gaps. The methodological innovation introduced through the application of RSM for BC preparation sets a precedent for sophisticated optimization in the realm of adsorbent development. The deliberate focus on locally sourced and sustainable adsorbents, especially BCs derived from date palm seeds, not only advances the discourse on carbonaceous materials but also aligns with the imperative of localized and eco-friendly solutions. The dedicated exploration of VOCs as a contemporary case study breaks new ground, offering insights into a class of pollutants that is increasingly becoming the focus of environmental concern. Finally, the unique application of BCs for VOC removal positions this research at the forefront of innovative remediation strategies. Collectively, these contributions not only enrich the academic dialogue but also offer tangible and actionable insights with implications for the broader domains of environmental science and engineering.

Chapter II

Methodology

II.1 Procurement and Refinement of Raw Materials

II.1.1 Sourcing of DPS

Date fruit, scientifically known as *Phoenix dactylifera* L., has been a nutritional mainstay for millennia, particularly in arid and semi arid terrains such as the Middle East and North Africa. This botanical resilience is essential, given the formidable climatic challenges—characterized by elevated temperatures, scarce precipitation, and arid soils—confronting edible plant species (Tang et al. [2013]), (Al Juhaimi et al. [2014]). The taxonomy of date palm cultivars, discerned by flesh texture, delineates three distinctive categories: soft, dry, and semi-dry (Biglari et al. [2008]). Noteworthy among the semi-dry variants flourishing in the Ziban region of Algeria, within the Biskra Province, are the Deglet Nour cv. and Arechti cv. (Allaith [2019]). Deglet Nour, with its ovoid morphology and a chromatic spectrum from reddish to yellow, possesses flesh characterized by exceptional transparency, tenderness, fibrous consistency, and delicately perfumed nuances (Akkak et al. [2009]).

Cultivation and production of Deglet Nour are paramount in Algeria and Tunisia, constituting nearly 90% of the global yield. Algeria strategically concentrates its cultivation endeavors in Ziban, Oued Souf, and M'zab (Amziane [2016], Bouguedoura et al. [2015]). In stark contrast, Arechti cultivars, distinguished by a distinctive brown hue and an ovoid contour, present a unique sensory experience

with commendable tenderness and an enticing acidulous taste (Akkak et al. [2009]). The predominant production hub for Arechti in Algeria resides in the Ziban and Oued Souf regions, significantly shaping the local agricultural landscape (Bouguedoura et al. [2015]). The dichotomy between Deglet Nour and Arechti cultivars not only sparks intrigue but also serves as a compelling avenue for the exploration of diverse attributes and culinary applications intrinsic to these date palm varieties in the Ziban region. A meticulous examination of each cultivar's distinct characteristics holds promise for unveiling valuable insights into their agronomic potential, nutritional composition, and prospective applications spanning culinary and industrial domains.

Within the ambit of sourcing DPS for this study, a noteworthy consideration is the substantial byproduct generated by date fruit processing industries on a daily basis. This residual material, primarily comprising date seeds, stands as a prolific reservoir poised for transformation into valuable food products. The selected DPS for this investigation emanates from the Deglet Noor cultivar, a prevalent semi-dry variant of date fruits endemic to the Zyban region in Biskra, Algeria, as illustrated in Figure II.1. This cultivar, with its unique characteristics, serves as a pertinent subject for examination, given its widespread presence and significance within the local agricultural landscape. The discernment of DPS from the processing residue of date fruit industries not only aligns with sustainable practices but also unveils a reservoir rich in bioactive compounds and essential oils, fostering its applicability across diverse industries, encompassing realms such as food, medicine, and cosmetics (Alharbi et al. [2021]).

II.1.2 Preprocessing of DPS Powder

In the nascent phase of pretreatment, the foundational raw material undergoes a meticulous transformation process. Initially, the raw DPS are subjected to a grinding procedure, followed by sieving to attain a particle size below 200 μm , a critical parameter enhancing the efficacy of subsequent thermo-treatment processes. Subsequent to this size refinement, the DPS undergo a thorough cleansing with deionized water, a procedure essential for the removal of extraneous impurities. Following

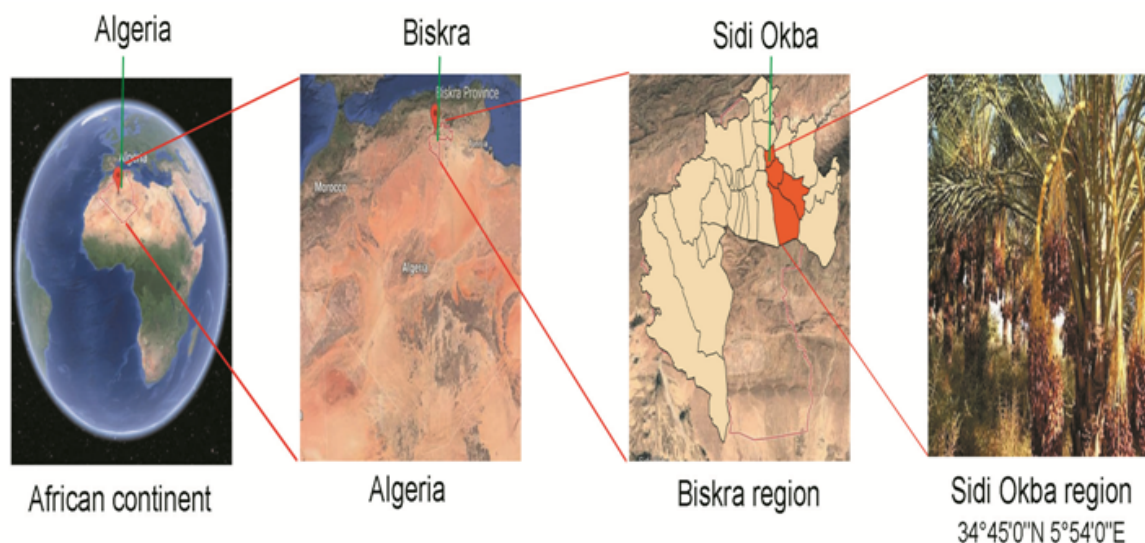


Figure II.1: Location map of the study area.

the washing stage, the DPS are subjected to an oven-drying process utilizing a UF 160 Memmert apparatus located in Schwabach, Germany. The oven-drying is executed over a duration of 12 hours at a controlled temperature of 105 °C, ensuring the elimination of residual moisture from the DPS. The resultant fine DPS powder, a product of this meticulous pretreatment, is judiciously preserved within an air-tight propylene plastic receptacle, thereby mitigating the risk of molding and maintaining the integrity of the pretreated DPS throughout subsequent analytical and synthetic processes.

II.2 BC Synthesis

The synthesis of BC commences with the carbonization of the meticulously pretreated DPS powder within an oxygen-limited environment, facilitated through the use of a covered porcelain crucible (Remmani et al. [2021], Remmani et al., Mesnoua et al.). The carbonization process unfolds with a deliberate and controlled ascent in temperature, implemented at a rate of 10 °C per minute. This gradual heating profile is instrumental in ensuring an oxygen-limited milieu and facilitating the thorough evaporation of entrapped pore water within the material. Following the attainment of the predetermined temperature, the material is maintained at this

thermal threshold for a specified duration, promoting the completion of the carbonization process.

Post-carbonization, the resulting BC entities are allowed to undergo natural cooling to room temperature. Subsequently, a stringent purification protocol is enacted, involving multiple rinses with hot deionized water to expunge superfluous impurities. The meticulously purified BC is then subjected to a drying regimen at 110 °C for a duration of 12 hours. The resultant dried BC is judiciously stored within compliant sealed containers, ensuring its sustained integrity until its subsequent utilization in ensuing phases of the study. This methodological precision not only underscores the commitment to rigorous procedural standards but also assures the production of BC of optimal purity and quality.

II.3 Statistical Optimization Utilizing RSM

In this investigation, the pursuit of an optimal BC synthesis from DPS is underpinned by the application of a meticulously constructed statistical model known as the central composite design (CCD) within the overarching framework of RSM. The principal focus of this study revolves around elucidating the nuanced interplay of factors influencing key characteristics of BC, specifically its specific surface area (SSA), total pore volume (V_t), mesopore volume (V_m), and overall yield (Y). These vital parameters are intricately linked to the independent variables governing the BC preparation process, notably the factors of time (t) and temperature (T).

The central composite design emerges as a judicious choice for this investigation due to its inherent capacity to model a quadratic surface, enabling a comprehensive exploration of the multifaceted relationship between the aforementioned factors. Importantly, the CCD approach facilitates not only the fitting of a quadratic surface but also the optimization of critical parameters, all achieved with a minimized number of experimental iterations. This efficiency is achieved through a structured arrangement of experimental runs, encompassing 2^n factorial runs, 2^n axial runs, and n_c center runs. The total number of experiments (N) is mathemati-

cally expressed as $N = 2^n + 2n + nc$, wherein n denotes the number of factors. In the context of this study, the preparation of BC involves two variables, translating to four factorial points, four axial points, and five replicates at the center points. Consequently, the total number of experiments for BC synthesis amounts to thirteen, a judiciously determined number balancing efficiency with statistical robustness.

The integration of center points within the experimental design serves a dual purpose: estimation of experimental error and assessment of data repeatability. To standardize the interpretation of the independent variables, they are coded to the $[-1, 1]$ interval, with -1 and $+1$ signifying low and high values, respectively. Axial points, strategically positioned at $(\pm\alpha, 0)$ and $(0, \pm\alpha)$ for BC synthesis, introduce a rotational aspect to the design, enhancing its robustness.

Table II.1 encapsulates the range and levels of the independent parameters for BC synthesis. The dependent variables (Y_j), representing the predicted responses in the RSM designs, are crucial in unraveling the intricate correlation between independent and dependent variables across the studied parameter space. In this comprehensive analysis, SSA (Y_1), V_t (Y_2), V_m (Y_3), and Y (Y_4) collectively form the crux of the investigation.

Table II.1: Range and Levels of Independent Parameters for BC Synthesis.

Variable	Range and levels				
	$-\alpha$	-1	0	$+1$	$+\alpha$
Time (h), (X_1)	0.6	1	2	3	3.4
Temperature ($^{\circ}\text{C}$), (X_2)	417	500	700	900	983

Experimental determination of SSA and V_t for BC at all points is executed through the meticulous application of methylene blue and iodine number techniques (Nunes and Guerreiro [2011], Saka [2012]), methods renowned for their precision and reliability in quantifying these crucial BC characteristics. The thorough integration of these methodological elements, guided by the principles of RSM and CCD, positions this study at the forefront of systematic exploration and optimization in BC synthesis.

The intricate orchestration of the RSM and CCD in this study is complemented by the utilization of cutting-edge statistical software. The statistical analyses, encompassing the establishment of the central composite design, parameter optimization, and the exploration of intricate relationships between variables, are conducted with precision and efficiency using the latest version of Design-Expert software (Figure II.2). This software, revered for its robust capabilities in experimental design and optimization, provides a user-friendly interface coupled with advanced statistical algorithms, ensuring a seamless integration of the Response Surface Methodology framework. Design-Expert's capacity to handle complex experimental designs, such as those involving multiple factors and response variables, aligns seamlessly with the sophisticated requirements of this study. The latest iteration of Design-Expert stands as an invaluable tool, facilitating a rigorous and comprehensive statistical exploration of the BC synthesis process from pretreated date palm seed powder.

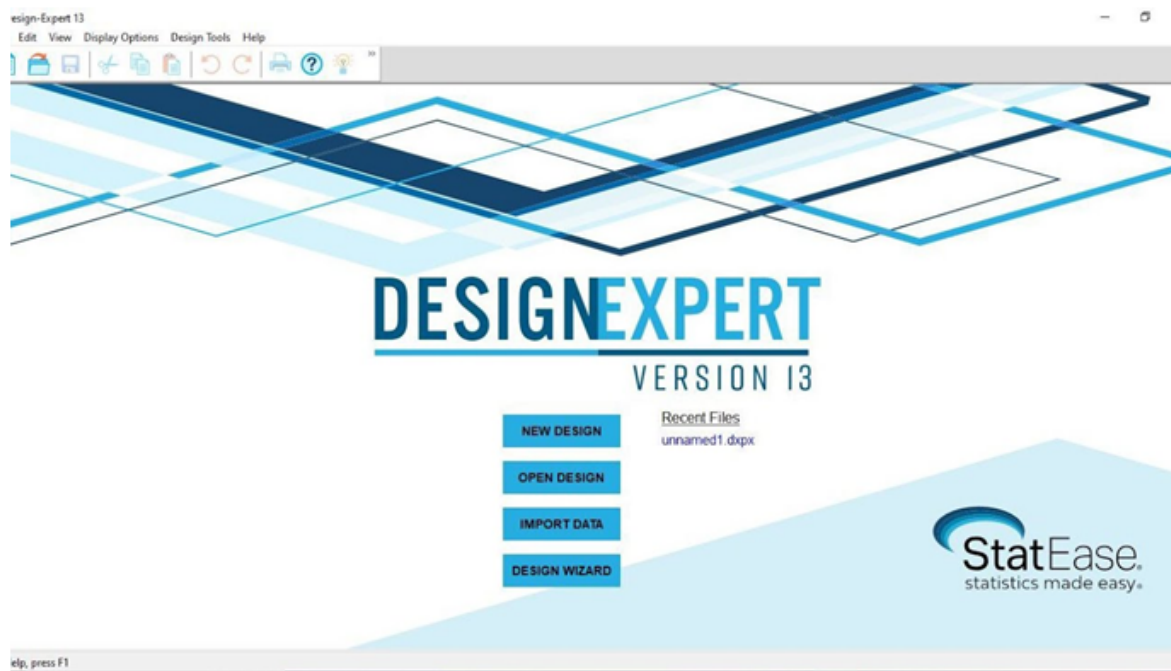


Figure II.2: Design-Expert software version 13.

II.4 Regression and Optimization Analysis

The culmination of experimental data was subjected to a rigorous regression analysis, with the objective of formulating predictive equations for each response variable—SSA (Y_1), Vt (Y_2), Vm (Y_3) and Y (Y_4)—within the BC synthesis process derived from pretreated DPS powder. Notably, the limited independent variables, denoted as (X_1) (time) and (X_2) (temperature), prompted the adoption of a simplified second-order regression model:

$$Y_j = b_0 + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2$$

Herein, the coefficients b_0 , b_1 , b_2 , b_{11} , b_{22} , and b_{12} signify the regression coefficients, while X_1 and X_2 represent the coded independent variables or regressors, aligning with the parameters of time and temperature, respectively.

Leveraging state-of-the-art statistical analysis software, specifically the Design-Expert software, the determination of coefficients for this refined second-order model was facilitated. These equations played a pivotal role in predicting the values of Y_2 , Y_3 , and Y_1 across a spectrum of time and temperature combinations.

The derived predictive values were instrumental in the generation of contour plots, providing insightful visualizations of the response surfaces for Y_2 , Y_3 , and Y_1 . These plots, underpinning the optimization process, played a critical role in identifying optimal conditions that concurrently maximize specific surface area, total pore volume, and overall yield in the synthesis of BC from pretreated date palm seed powder.

This tailored approach, marked by the integration of sophisticated mathematical modeling and meticulous prediction techniques, underscores the precision and depth of the regression and optimization analyses, establishing a robust founda-

tion for the nuanced interpretation of the intricate relationships between the coded independent variables and the targeted responses.

In the pursuit of a comprehensive understanding of the synthesized BC's properties, a graphical presentation (Figure II.3) has been included to visually encapsulate the synthesis process and the strategic application of RSM. The presentation delineates the sequential progression, commencing from the acquisition of date palm seeds to the systematic implementation of RSM for the optimization of BC preparation. The assimilation of RSM into the synthesis process represents a methodological innovation, ushering in a paradigm of sophistication and precision for the optimization of pivotal parameters. This visual representation serves not only as a scholarly guide through the intricate stages of the synthesis journey but also stands as a testament to the methodological intricacies inherent in the strategic application of RSM in BC preparation.

II.5 Characterization of BC

II.5.1 Structural and Compositional Analysis

II.5.1.1 X-Ray Diffraction (XRD)

In the meticulous characterization of BC, XRD analysis emerges as a cornerstone method, drawing from its intrinsic capability to unravel the crystalline intricacies of materials. This sophisticated analytical technique, as elucidated by various scholarly sources (Zheng et al. [2016], Nzediegwu et al. [2021]), plays a pivotal role in deciphering the internal crystalline structure of BC, thereby offering profound insights into the carbonization process. XRD spectroscopy, renowned for its prowess in delineating crystal structures and mineral compositions, has become particularly relevant in the comprehensive study of BC (Zheng et al. [2016], Nzediegwu et al. [2021]). The fundamental principle underpinning XRD analysis involves the strategic application of X-rays directed at the sample, capturing the ensuing diffraction pattern as these X-rays engage with the crystal lattice of the material (Nzediegwu et al. [2021]). The resulting diffraction pattern serves as an intricate

window into the arrangement of atoms within the crystal lattice, thereby facilitating the identification of minerals intricately woven into the BC matrix (Nzediegwu et al. [2021]). This includes a discerning analysis of crystalline phases such as graphite, quartz, and potentially other mineral constituents embedded within BC (Nzediegwu et al. [2021]). Beyond unraveling the mineralogical tapestry, XRD analysis extends its purview to offer insights into the degree of crystallinity and the dimensions of crystalline domains within the BC structure (Nzediegwu et al. [2021]). This nuanced examination aids in unraveling the structural intricacies that contribute to BC's unique properties. The information gleaned from XRD analysis is instrumental not only in enhancing our understanding of BC's inherent properties but also in providing a foundational platform for exploring its diverse potential applications in a myriad of scientific domains (Nzediegwu et al. [2021]). Consequently, the integration of XRD within the characterization toolkit stands as a critical step towards a comprehensive comprehension of BC's structural, functional, and morphological attributes, offering valuable insights that extend beyond the boundaries of conventional analysis.

In the experimental phase dedicated to elucidating the phase state of the BC sample through XRD analysis, a "Bruker D-8 advance diffractometer" took center stage. This state-of-the-art instrument, renowned for its precision, was deployed with $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) at a current intensity of 30 mA and voltage of 40 kV (Remmani et al. [2021]). The selection of $\text{CuK}\alpha$ radiation, distinguished by its characteristic wavelength, ensured meticulous probing into the crystalline intricacies of BC. Under these controlled conditions, the diffractometer functioned as a discerning lens, capturing the nuanced XRD distribution of BC sample. This experimental design, marked by stringent parameters and cutting-edge instrumentation, attests to the dedication to analytical precision. The Bruker D-8 advance diffractometer, as the chosen apparatus, epitomizes a commitment to methodological rigor in exploring the crystalline composition and internal structure of BC. This experimental undertaking represents a significant advancement in the holistic characterization of BC, positioning XRD analysis as an indispensable tool for unraveling its material intricacies and potential applications in scientific domains.

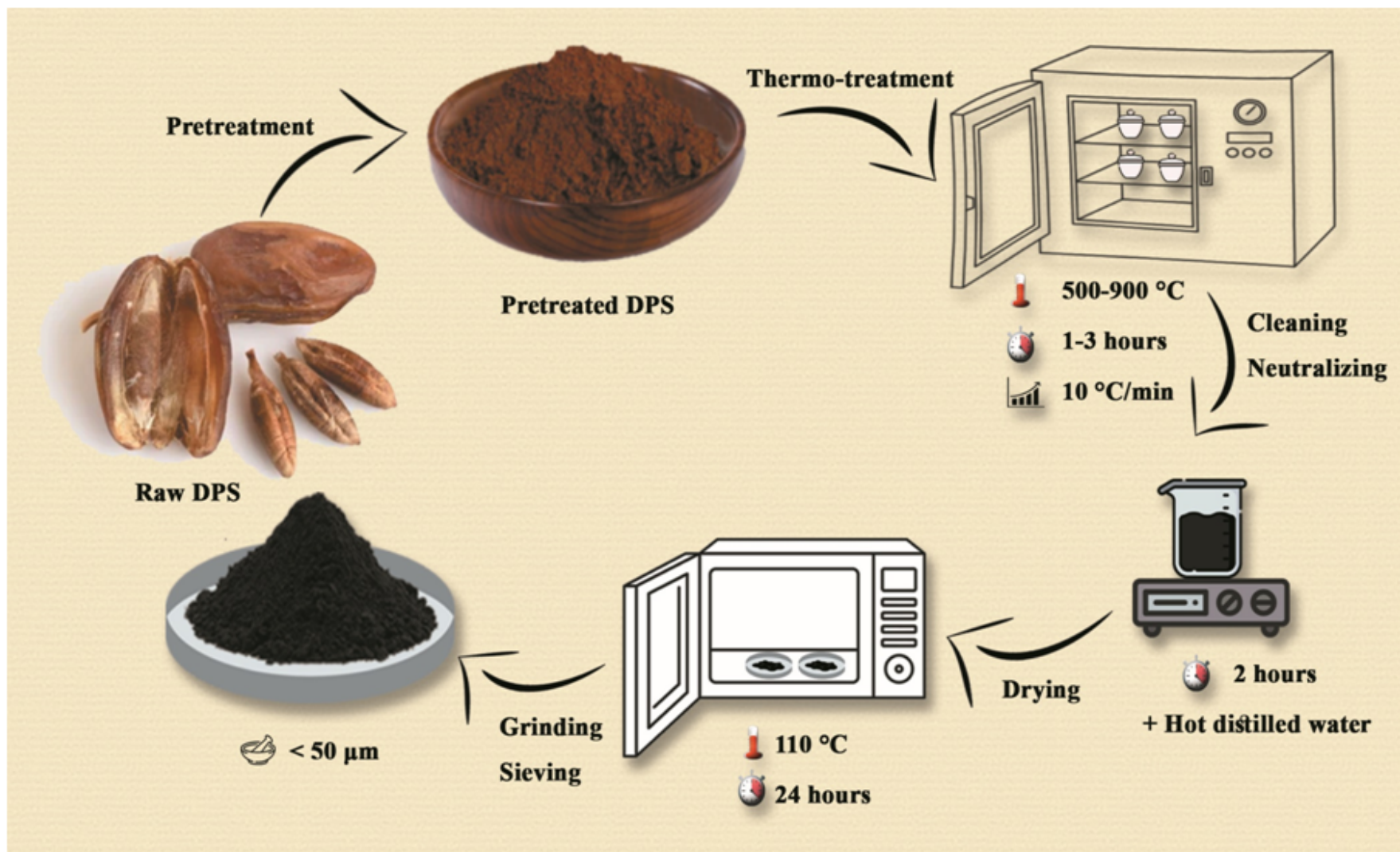


Figure II.3: Synthesis Journey and Methodological Innovation in BC Preparation.



Figure II.4: Bruker D8 Advance Diffractometer Setup for X-Ray Diffraction Analysis (Research Laboratory on Thin Films and Their Applications, University of Biskra, Algeria).

II.5.1.2 FTIR

In this section, FTIR Spectroscopy utilizing the state-of-the-art Spectrum Two FT-IR Spectrometer played a pivotal role in characterizing BC. This sophisticated analytical method provided high-resolution spectra, offering insights into the chemical composition and functional groups within the BC matrix. The Spectrum Two, renowned for its precision and versatility, facilitated the identification of crucial functional groups such as hydroxyls, carbonyls, and aliphatic C-H stretches. This detailed molecular analysis is foundational to understanding BC's reactivity and potential applications, particularly in environmental contexts such as adsorption processes. The Spectrum Two FT-IR Spectrometer (Figure II.5) emerged as an indispensable tool, enriching our comprehension of BC's chemical attributes and paving the way for its diverse applications across scientific domains.

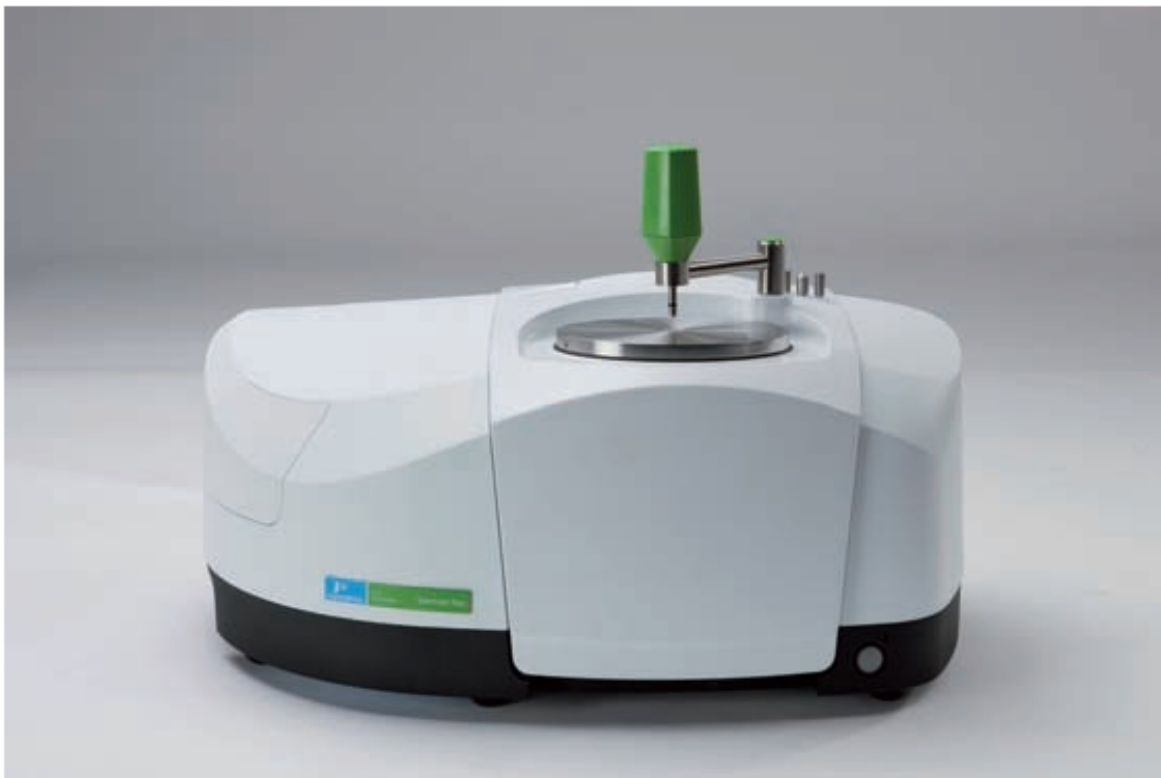


Figure II.5: PerkinElmer Spectrum Two FT-IR Spectrometer with LiTaO₃ Detector (Photonics and Multifunctional Nanomaterials Research Laboratory, University of Biskra, Algeria).

II.5.1.3 SEM-EDS

In the comprehensive characterization of BC, the utilization of the advanced High-Resolution SEM JSM-7610F Plus (Figure II.6) from JEOL proves pivotal. This section delves into the amalgamated capabilities of SEM-EDS, facilitated by this cutting-edge instrument, to unveil both the morphological intricacies and elemental composition of the BC matrix. Through meticulous SEM imaging, the JSM-7610F Plus provides intricate details on BC's surface morphology, encompassing pore structure, particle size, and topography. Concurrently, the Energy-Dispersive X-ray Spectroscopy (EDS) component enables precise elemental analysis, identifying and mapping elements such as carbon, oxygen, and trace elements. This tandem approach emerges as a powerful tool, offering a holistic perspective on BC's structural and elemental attributes. The JSM-7610F Plus, with its advanced imaging and analytical capabilities, not only enriches our understanding of BC but also provides

a foundation for diverse scientific applications, marking a significant stride in the advancement of BC characterization



Figure II.6: High-Resolution SEM JSM-7610F Plus (CRTSE, Algeria).

II.5.2 Morphological and Surface Analysis

II.5.2.1 BET

In the pursuit of a nuanced exploration of BC's surface characteristics, the Brunauer-Emmett-Teller (BET) surface area analysis was meticulously conducted using the Micromeritics 3Flex Version 4.05 instrument. Employing nitrogen as the adsorptive, the analysis was performed at a bath temperature of 77.371 K. The sample, with a density of 1.000 g/cm³, underwent controlled conditions at 200°C for 24

hours (noted as 200C-24h). The precisely measured sample mass stood at 0.8426 g, ensuring the accuracy of the BET analysis. This sophisticated instrumental setup, specifically the Micromeritics 3Flex Version 4.05 (Figure II.7), with its detailed analysis of adsorption isotherms and controlled conditions, contributes to a robust understanding of the BC's surface area characteristics, paving the way for informed interpretations and implications in adsorption processes.



Figure II.7: Micromeritics 3Flex Version 4.05 (dipartimento ingegneria chimica materiali ambiente, Sapienza University, Italy).

II.5.3 pH_{pzc}

In accordance with reference (Gatabi et al. [2016]), the experimental methodology employed to ascertain the pH of Zero Point Charge (pH_{pzc}) meticulously adhered to a pH drift method, intricately integrated with the co-precipitation process subsequent to each surface modification. The experimental configuration encompassed the meticulous preparation of solutions, consisting of 0.01 mol /L NaCl

in 10 ml test tubes, wherein pH values spanning from 2 to 8 ($\text{pH}_{\text{initial}}$) were precisely adjusted through incremental additions of 0.1 mol/L NaOH and 0.1 mol /L HCl. Subsequently, 30 mg of BC sample was introduced into the test tubes, subjected to agitation at 250 rpm, and maintained at a constant temperature of 25°C, facilitated by the Hanna Basic pH/ORP Benchtop Meter - HI2211. Following an incubation period of 48 hours, the final pH (pH_{final}) of the samples was meticulously determined using the same meter. The resultant curve, portraying pH_{final} against $\text{pH}_{\text{initial}}$, discerned nuanced variations, with the pHPZC value pinpointed at the intersection point of the curve with the line passing through the origin. Rooted in reference (Gatabi et al. [2016]), this methodological framework ensures a systematic and dependable assessment of pHPzc values, offering profound insights into the surface charge properties of the optimized BC powder under scrutiny.

II.6 Adsorption Experiments

II.6.1 Preparation of Stock Solution

In the context of VOCs adsorption tests, a meticulous approach was undertaken to prepare a mono-component solution within a Tedlar bag®, with the deliberate exclusion of headspace formation. A volume of ACS $\geq 99.5\%$ pure VOCs from Sigma-Aldrich® was precisely measured and introduced into the Tedlar bag®, ensuring the attainment of the predetermined concentration required for subsequent adsorption experiments (Rossi et al. [2021]). This careful spiking process, conducted in a sealed environment, aimed to maintain the integrity of the concentration throughout the preparation.

The solution, now enriched with VOCs, underwent a horizontal shaking process for one day, promoting thorough homogenization to achieve a uniformly distributed composition. This rigorous agitation played a crucial role in enhancing the representativeness of the stock solution for subsequent tests, ensuring that the characteristics of the VOCs were evenly dispersed.

To establish a baseline for evaluating adsorption efficiency, one volume of

the prepared contaminated solution was consistently sampled at the initiation of each test. This initial effective concentration served as a reference point, allowing for a comprehensive assessment of the optimized BC powder's adsorption capabilities over the course of the experiments.

The preparation process incorporated stringent quality assurance measures, including the use of certified pure VOCs and controlled shaking conditions (Rossi et al. [2021]). These measures were integral to ensuring the reliability and accuracy of the initial concentration, laying a robust foundation for the subsequent investigation into the adsorption capabilities of the optimized BC powder in addressing VOC contamination in aqueous solutions.

II.6.2 Kinetic and Isotherm Study

In the pursuit of a comprehensive understanding of the adsorption kinetics and isotherms, an intricate experimental design was implemented. The kinetic study commenced with an initial theoretical concentration (C_0) of 50 mg/L, employing a solid/liquid ratio of 0.5 in triplicate experiments. Aliquots were judiciously sampled at specific time intervals of 0.5, 1, 2, 3, 5, 24, and 28 hours. The collected samples were promptly transferred to hermetically sealed glass vials for subsequent analysis.

For the isotherm study, a deliberate design was employed to create a solution with a theoretical initial concentration spanning 10–50–100–200–500 mg/L. These solutions were brought into contact with varying amounts of BC ranging from 0.001 to 0.010 g. Sampling at time 0 (C_0) enabled the assessment of the initial concentration, while additional sampling at equilibrium time (C_e) provided crucial data points for the isothermal curve. It is noteworthy that, based on kinetic tests, a 24-hour duration was deemed sufficient to ensure the attainment of equilibrium, validating the reliability and efficiency of the chosen experimental timeframe (Rossi et al. [2021]).

To benchmark the results, kinetic and isothermal curves were also obtained

for a commercial Norit AC, type Darco Sigma-Aldrich®. This comparative analysis served as a valuable reference for evaluating the performance of the optimized BC powder in comparison to a commercially available adsorbent (Rossi et al. [2021]). The meticulous design and execution of these experiments contribute to a nuanced understanding of the adsorption kinetics and isotherms, laying the groundwork for insightful comparisons and assessments in the realm of wastewater treatment and environmental remediation.

II.6.3 Determination of VOCs

In the meticulous determination of VOCs, a sophisticated approach was employed utilizing gas chromatography (GC) with a DANI MASTER instrument, equipped with a DANI 86.50 headspace auto-sampler (Figure II.8). The GC setup featured a capillary column (30 m × 0.53 mm ID × 3 μm, TRB624) coupled with a Flame Ionization Detector (FID). The headspace analysis program was meticulously executed, with parameters set as follows: oven temperature at 80 °C, manifold temperature at 120 °C, transfer line temperature at 180 °C, and gentle shaking for 1 minute. GC conditions included helium as the carrier gas with a flow rate of 10 mL/min, an injector temperature of 180 °C, split injection at a ratio of 1:2, and a detector temperature of 300 °C with a combination of air, N_2 , and H_2 for the FID (flows 240, 25, 60 mL/min).

The temperature programming for the oven followed a sequence of 70 °C for 0.5 minutes, ramping up at 30 °C/min to 90 °C, and further increasing at 30 °C/min to reach 180 °C. For the quantitative determination of VOCs, a meticulous calibration curve was constructed by diluting a VOC/Ethanol stock solution into standards with a concentration range of 0.1–5 mg/L. This comprehensive experimental setup, integrating precise instrumental configurations and calibration procedures, ensured the accurate and reliable determination of VOC concentrations in the context of adsorption experiments. The utilization of gas chromatography, a powerful analytical technique, underlined the commitment to precision and analytical rigor in assessing the efficacy of BC in removing VOCs from aqueous solutions.



Figure II.8: DANI MASTER Instrument with DANI 86.50 Headspace Auto-sampler (dipartimento di chimica, Sapienza University, Italy).

II.7 Modeling of Adsorption Processes

In the rigorous modeling of adsorption processes, we employed two well-established kinetic models: the pseudo-first-order (PFO) and pseudo-second-order model (PSO), as detailed in references (Ahmad et al. [2013], Kołodyńska et al. [2012]). These models provide a nuanced understanding of the temporal dynamics of VOCs adsorption onto BC. The PFO model, represented by Equation (1), incorporates the natural logarithm of the difference between the equilibrium adsorption capacity (q_e) (mg/g) and the adsorption capacity at a given time (q_t) (mg/g), unveiling the rate

constant (k_1) (min^{-1}) governing the initial adsorption phase.

$$q_t = q_e(1 - e^{-k_1 t})(1)$$

On the other hand, the PSO model, encapsulated in Equation (2), elucidates the intricate interplay between (q_e) (mg/g), (k_2) (min^{-1}), and the elapsed time (t) (min). The PSO kinetics delve deeper into the adsorption mechanism, offering insights into the chemisorption nature of the process.

$$q_t = (q_e^2 k_2 t) / (1 + q_e k_2 t)(2)$$

To quantify VOC adsorption per gram of BC at equilibrium q_e and at specific sampling times q_t , we employed Equations (3) and (4), respectively. These equations allow for a comprehensive exploration of VOC adsorption dynamics over time, with C_t indicating the VOC concentration at the time of sampling.

$$q_e = ((C_o - C_{eq}) \times V) / w(3)$$

$$q_t = ((C_o - C_t) \times V) / w(4)$$

Additionally, we harnessed the power of Langmuir and Freundlich models to discern the underlying adsorption isotherms. Equation (5) showcases the Langmuir model, incorporating parameters such as q_{max} (maximum adsorption capacity) (mg/g) and K_L (Langmuir constant) (L/mg), while Equation (6) portrays the Freundlich model, characterized by K_F (Freundlich constant) (L/mg) and n (heterogeneity factor) (Rossi et al. [2021], Liu et al. [2011]).

$$q_e = (q_{max} \times K_L \times C_e) / (1 + K_L \times C_e)(5)$$

$$q_e = K_F \times C_e^{1/n}(6)$$

Through these intricate models, our analysis transcends mere adsorption kinetics, delving into the underlying mechanisms and equilibrium dynamics, providing a robust foundation for understanding the complex interplay between BC and VOC

in aqueous solutions.

Chapter III

Results and Discussion

III.1 BC Preparation

Embarking on the intricate journey of BC preparation unveils a meticulous dance between innovation and precision, guided by the sophisticated principles of RSM. Situated in the heart of the Biskra region in Algeria, this section undertakes a thorough exploration of the optimal parameters that govern the synthesis of BC derived from date palm seeds. RSM, a strategic and systematic framework, assumes a central role in steering this scientific endeavor away from conventional approaches. This intentional departure allows for the tailored optimization of BC, intricately aligned with the distinctive characteristics of date palm seeds sourced from the Biskra region. As we delve into the depths of this groundbreaking methodology, a new chapter in sustainable adsorbent development unfolds—one characterized by tailored precision and an unwavering commitment to the unique environmental and economic realities of the locale. The comprehensive design matrix, accompanied by the corresponding values of the four responses extracted from the experimental endeavors, is meticulously detailed in Table III.1.

III.1.1 Exploring the Dynamics of BC preparation process

Embarking on the intricate exploration of the BC preparation process, the outcomes of this scientific journey are meticulously presented in Tables III.1, III.2, III.3, and Figure III.1. These comprehensive visualizations encapsulate the dynamic inter-

Table III.1: Experimental Design Matrix and Results for BC

Runs	Variables		Responses			
	X_1	X_2	Y_1	Y_2	Y_3	Y_4
1	-1.414	0	463.832	0.1991	0.0874	41.98
2	1	1	780.457	0.1969	0.0975	21.13
3	0	1.414	868.197	0.1952	0.0917	21.24
4	0	0	520.286	0.1825	0.0933	37.06
5	0	-1.414	230.461	0.1545	0.0089	55.58
6	1.414	0	515.618	0.1757	0.0904	36.184
7	0	0	538.027	0.1879	0.0914	36.352
8	0	0	534.203	0.1804	0.0875	37.67
9	-1	1	679.649	0.1925	0.2988	26.63
10	0	0	526.571	0.1841	0.0828	35.33
11	0	0	516.188	0.1793	0.947	36.89
12	1	-1	467.867	0.1697	0.2489	31.56
13	-1	-1	397.278	0.1546	0.1857	53.072
Goal	minimize	minimize	maximize	In range	In range	maximize

play between pyrolysis time (X_1) and temperature (X_2), skillfully harnessed through the intricate framework of RSM. Within these tables and figures lie the nuanced details of SSA, V_m , V_t , and the final Y of BC. As we navigate through these data-rich representations, a deeper understanding of the optimized parameters and their impact on BC synthesis unfolds, paving the way for a comprehensive interpretation of the dynamics inherent in the synthesis process.

The intricate orchestration of X_1 and X_2 in the synthesis of BC from DPS unfolds through the lens of RSM. This journey is not only about optimizing BC but also about gaining profound insights into the interplay of critical parameters.

- SSA (Y_1)

The analysis of SSA, denoted by Y_1 , showcases a robust model. With an Adjusted R^2 of 0.8991 and a Predicted R^2 of 0.8189, the model demonstrates its efficacy. Both X_1 and X_2 play pivotal roles in maximizing SSA, highlighting the importance of fine-tuning time and temperature during pyrolysis.

- V_m (Y_2)

Venturing into V_m , represented by Y_2 , the model's goodness of fit is notable,

albeit not as robust as SSA. With an Adjusted R^2 of 0.7022 and a Predicted R^2 of 0.4783, the model emphasizes the moderate influence of X_2 . This insight is invaluable for tailoring BC for applications where microporosity is crucial.

- V_t (Y_3)

V_t , captured by Y_3 , presents a unique challenge in the modeling process. The model's Adjusted R^2 is at its minimal value, signifying a lack of significance in the selected parameters. The high coefficient of variation (CV) points towards additional factors influencing V_t , warranting a more intricate exploration.

- Yield (Y_4)

The Y of the BC synthesis process, embodied by Y_4 , stands out as a success in modeling. With an Adjusted R^2 of 0.8623 and a Predicted R^2 of 0.5897, the model showcases a robust fit. The harmonious dance between time and temperature underscores their synergistic impact on the final yield.

- Coefficients Analysis

Delving into the coefficients of the models provides further clarity (Table III.3). The linear model for SSA (Y_1) exhibits a strong positive relationship with both X_1 and X_2 , indicating that an increase in time and temperature positively influences specific surface area. In contrast, the model for V_m (Y_2) reflects a delicate balance, with X_2 exerting a more pronounced effect. The model for V_t (Y_3), represented as a mean model, raises questions about the robustness of the selected parameters in influencing V_t . The need for additional explorations into factors impacting V_t becomes apparent. Yield (Y_4), modeled with a 2-Factor Interaction, unfolds a complex scenario. X_1 , X_2 , and their interaction contribute significantly to the final Y, emphasizing the necessity for meticulous control of both time and temperature for optimal yield.

In conclusion, the RSM analysis provides a multifaceted understanding of the BC preparation process. While SSA and Yield demonstrate strong predictability and optimization potential, V_m and V_t pose challenges that beckon further exploration. Future directions call for a nuanced exploration of additional factors influencing

micropore and total pore volumes, promising a sustainable and tailored adsorbent with applications across diverse scientific domains.

Table III.2: Analysis of variance (ANOVA) for the fitted models for BC preparation

Source	SS	DF	MS	F-value	P-value
(a) For Y_1 (Adj $R^2 = 0.8991$; Pred $R^2 = 0.8189$)					
Model	2.875	2	1.437	54.45	<0.0001
Coefficient of variation (CV), 9.49%					
(b) For Y_2 (Adj $R^2 = 0.7022$; Pred $R^2 = 0.4783$)					
Model	0.0019	2	0.0010	15.15	0.0009
Coefficient of variation (CV), 4.38%					
(c) For Y_3 (Adj $R^2 = 0.0000$; Pred $R^2 = -0.1736$)					
Model	0.0000	0			
Coefficient of variation (CV), 130.27%					
(d) For Y_4 (Adj $R^2 = 0.8623$; Pred $R^2 = 0.5897$)					
Model	0.1131	1	0.3771	26.04	
Coefficient of variation (CV), 1.51%					

SS, sum of squares; DF, degree of freedom; MS, mean square; Adj R^2 , Adjusted R^2 ; Pred R^2 , Predicted R^2 .

Table III.3: The Coefficients values of fitted models for BC preparation.

	Fitted Model	R^2	P-value	Intercept	X_1	X_2	X_1X_2
Y_1	Linear	0.9159	<0.0001	541.45	30.58	187.08	-
Y_2	Linear	0.7519	<0.0009	0.1810	-0.0017	0.0153	-
Y_3	Mean	0.0000	0.0170	0.1855	-	-	-
Y_4	2FI	0.8967	0.0647	36.21	-4.40	-10.68	4.00

III.1.2 Model's validation for BC preparation process

The exploration of optimal processing conditions for BC synthesis, guided by RSM, culminates in a desirability (D) of 0.843. This metric reflects the convergence of predicted and experimental values, a critical aspect in the precision of the predictive models. The derived conditions, underpinning BC synthesis, are characterized by a pyrolysis time (X_1) of 1.700 hours and a pyrolysis temperature (X_2) of 828.155°C.

Within this framework, a meticulous examination of response variables at these optimal conditions unveils intriguing insights. The specific surface area (Y_1),

a paramount determinant of adsorption efficiency, is predicted at $652.146 \text{ m}^2/\text{g}$, closely aligning with the experimental value of $654.785 \text{ m}^2/\text{g}$. This close correspondence attests to the reliability of the predictive model. Moving to micropore volume (Y_2), a critical indicator for adsorption, the model predicts $0.191 \text{ cm}^3/\text{g}$, mirroring the experimental value of $0.190 \text{ cm}^3/\text{g}$ with remarkable precision. This congruence reinforces the efficacy of the model in capturing the dynamics of micropore formation during BC synthesis.

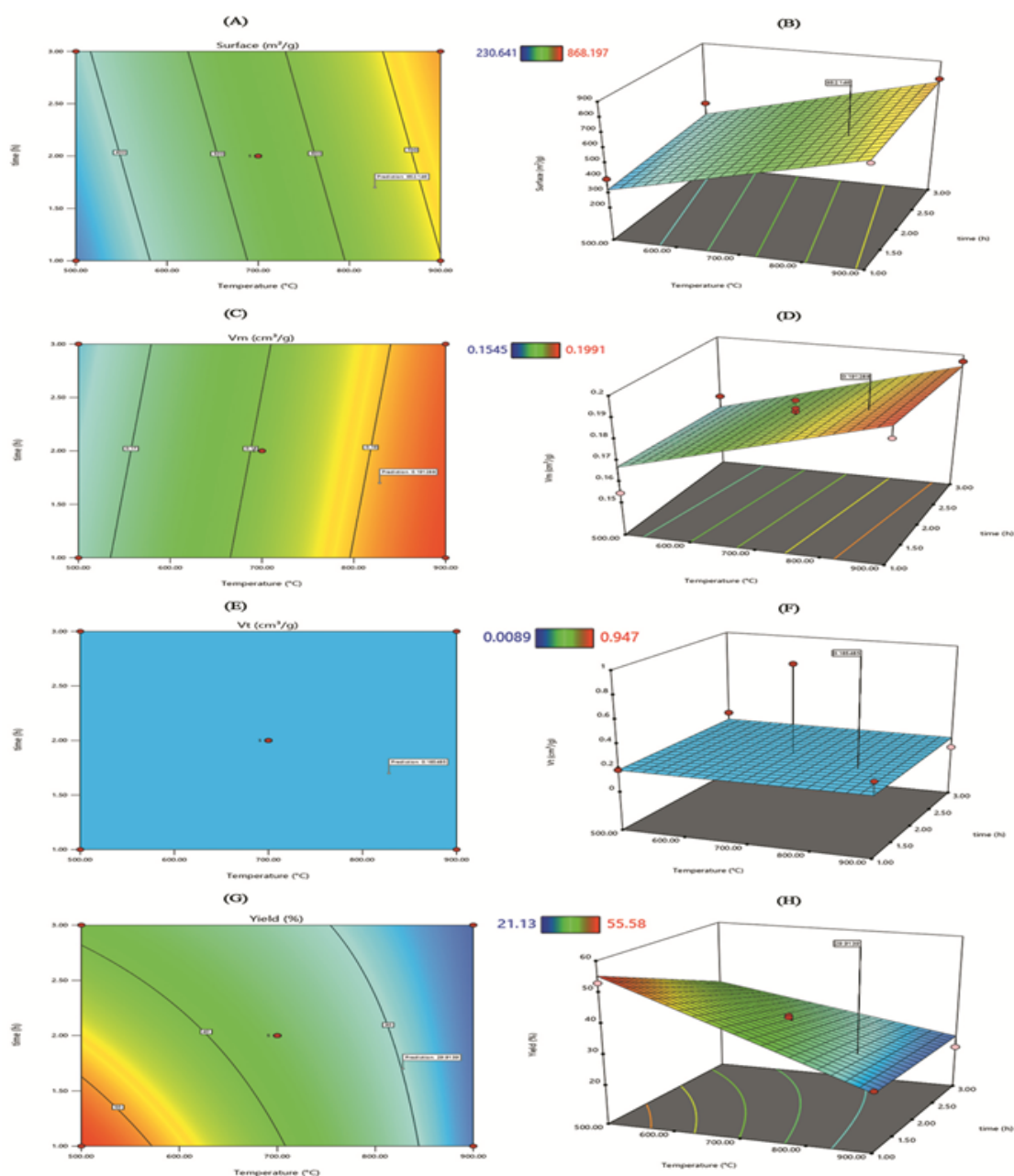


Figure III.1: Plots of respectively contour and response surfaces highlighting the influence of X_1 and X_2 on A&B: SSA (Y_1), C&D: V_m (Y_2), E&F: V_t (Y_3) and Y (Y_4).

Table III.4: Optimal processing conditions from predicted optimization and experimental model validation

Parameters	X_1 (h)	X_2 (°C)	Y_1		Y_2		Y_3		Y_4	
			Pred	Exp	Pred	Exp	Pred	Exp	Pred	Exp
BC (D= 0.843)										
Conditions	1.700	828.155	652.146	654.785	0.191	0.190	0.0947	0.185	29.914	30.546

D, desirability; Pred, predicted value; Exp, Experimental value.

However, the evaluation of total pore volume (Y_3) reveals a noteworthy discrepancy. While the predicted value stands at $0.0947 \text{ cm}^3/\text{g}$, the experimental determination yields $0.185 \text{ cm}^3/\text{g}$. This incongruity suggests a potential area for refinement in the model, urging further exploration into the factors influencing total pore volume. In the realm of yield (Y_4), a pivotal parameter for economic viability, the model predicts 29.914%, closely converging with the experimental yield of 30.546%. This alignment underscores the model's proficiency in predicting optimal conditions that maximize BC yield.

Statistical scrutiny further strengthens the narrative. The desirability index of 0.843 emphasizes the overall coherence between the predicted and experimental outcomes. Statistical validation reinforces the robustness of the model, providing a reliable framework for optimizing BC synthesis.

From a chemical perspective, the high specific surface area of $654.785 \text{ m}^2/\text{g}$ signifies an ample surface available for adsorption, augmenting BC's potential as an effective adsorbent. The congruence between predicted and experimental micropore volumes ($0.191 \text{ cm}^3/\text{g}$) underscores the precision in optimizing conditions for maximizing microporosity. However, the observed discrepancy in total pore volume suggests a nuanced interplay of factors governing porosity, demanding further chemical analyses to unveil the intricacies of total pore volume dynamics.

III.1.3 A Scientific and Environmental Impact of Optimizing BC Preparation

The optimization of BC preparation through RSM represents a remarkable convergence of predictive accuracy and experimental validation. The achieved D value of 0.843 attests to the model's effectiveness in steering the synthesis process towards optimal conditions. The precise calibration of pyrolysis time to 1.7 hours and temperature to 828.155°C showcases the model's accuracy in guiding the synthesis process.

Examining the impact of these optimized conditions on crucial parameters, the SSA emerges as a pivotal indicator of BC's adsorption efficiency. The predicted SSA of 652.146 m^2/g closely aligns with the experimental value of 654.785 m^2/g , affirming the model's reliability in predicting and optimizing BC's surface area for enhanced adsorption capabilities. Shifting focus to BC yield, the optimization process predicts a yield of 29.914%, closely matching the experimental yield of 30.546%. This congruence underscores the model's proficiency in predicting optimal conditions for maximizing BC yield, a parameter with significant implications for economic viability.

Exploring micropore volume, the model predicts 0.191 cm^3/g , mirroring the experimental value of 0.190 cm^3/g . This close correspondence underscores the model's ability to capture the intricate dynamics of micropore formation during BC synthesis, further enhancing its suitability as an adsorbent. However, the observed discrepancy in total pore volume, where the predicted value is 0.0947 cm^3/g while the experimental determination yields 0.185 cm^3/g , suggests a nuanced interplay of factors influencing total porosity. This incongruity serves as a crucial avenue for refinement in the model, warranting additional chemical analyses to unravel the complexities governing total pore volume dynamics.

From a scientific standpoint, the high SSA and micropore volume signify an extensive surface area available for adsorption, augmenting BC's potential as an effective adsorbent. The meticulous optimization of time and temperature showcases

the pivotal role of these parameters in influencing not only porosity but also yield. The enhanced surface area and yield, driven by optimized parameters, contribute to the efficacy of BC in environmental remediation applications.

In the realm of environmental impact, BC's ability to sequester atmospheric carbon holds profound significance. The optimization process, informed by RSM, contributes to reducing global warming by enhancing BC's carbon sequestration capabilities. The synthesis of BC from DPS, guided by RSM, thus emerges not only as a scientifically optimized process but also as a key player in climate mitigation and biogeochemical cycles (Patel and Panwar [2023]).

Economically, the optimization of BC production from DPS bears implications for sustainable and cost-effective BC production. The precision achieved through RSM ensures resource efficiency, contributing to the economic viability of BC synthesis.

In conclusion, the optimization of BC preparation through RSM demonstrates not only scientific rigor but also a pathway towards sustainable and tailored adsorbents. The synergistic impact of time and temperature optimization on SSA and yield underscores the multifaceted benefits of this approach. As BC stands poised for applications in environmental remediation, its economic and environmental implications underscore the transformative potential of RSM-guided synthesis in addressing global challenges.

III.2 BC Characterization

III.2.1 Structural and Compositional Analysis

The XRD analysis of optimized BC sample, as depicted in Figure III.2, reveals essential insights into its crystalline characteristics. The absence of distinct, sharp reflections in the patterns underscores the noncrystalline nature inherent to carbon materials. Notably, the broad reflections observed at approximately $2\theta = 26^\circ$ and 42° are attributed to the (002) and (100) planes of graphite crystalline, respectively.

This crystalline signature aligns with findings from a prior study (Remmani et al. [2021]) on BC preparation, suggesting a consistent presence of graphite crystalline phases. Despite variations in the preparation conditions, the resemblance in mineral composition points to a robust correlation with the composition and initial structure of DPS. This intriguing connection implies that the unique mineral composition of DPS profoundly influences the resulting BC, emphasizing the intricate relationship between precursor material and the derived BC structure. The observed similarities in the crystalline phases provide valuable chemical insights, opening avenues for a deeper understanding of BC's structural attributes.

In Figure III.3, the FTIR spectrum of the BC sample reveals distinctive peaks indicative of various functional groups. Within the range of $500\text{-}900\text{ cm}^{-1}$, the presence of alkanes is suggested, characterized by C-H bending, C-H wagging, and C-C stretching. Moving to the interval of $900\text{-}1050\text{ cm}^{-1}$, the spectrum displays a peak around 1010 cm^{-1} , suggesting C-O stretching of the ether (R-O-R') functional group. Additionally, this interval may be associated with C-C stretching or C-H bending of alkynes. The region from $1330\text{ to }1540\text{ cm}^{-1}$ indicates the presence of C-H bending in alkanes and C-N stretching in amines. Shifting to the range of $1670\text{-}1550\text{ cm}^{-1}$, the spectrum suggests C=C stretching of alkenes. Finally, in the interval of $1880\text{-}2080\text{ cm}^{-1}$, the presence of C=O stretching in aromatics is highlighted. These observations collectively provide valuable insights into the chemical composition of BC, offering a nuanced understanding of the functional groups present in the sample. Moreover, the identified functional groups in the FTIR spectrum of BC hold significant implications for its potential as an adsorbent in various adsorption processes. The presence of alkanes, ethers, and aromatics suggests a diverse range of surface functionalities that could enhance adsorption capabilities. Alkane groups are known for their hydrophobic nature, indicating a potential affinity for nonpolar adsorbates. The ether functional group's C-O stretching band may contribute to interactions with polar compounds. Additionally, the presence of aromatic structures hints at the possibility of $\pi - \pi$ interactions, further broadening BC's adsorption versatility. Understanding these functional groups provides a foundation for tailoring BC's surface properties, optimizing its performance as an efficient adsorbent across

a spectrum of applications.

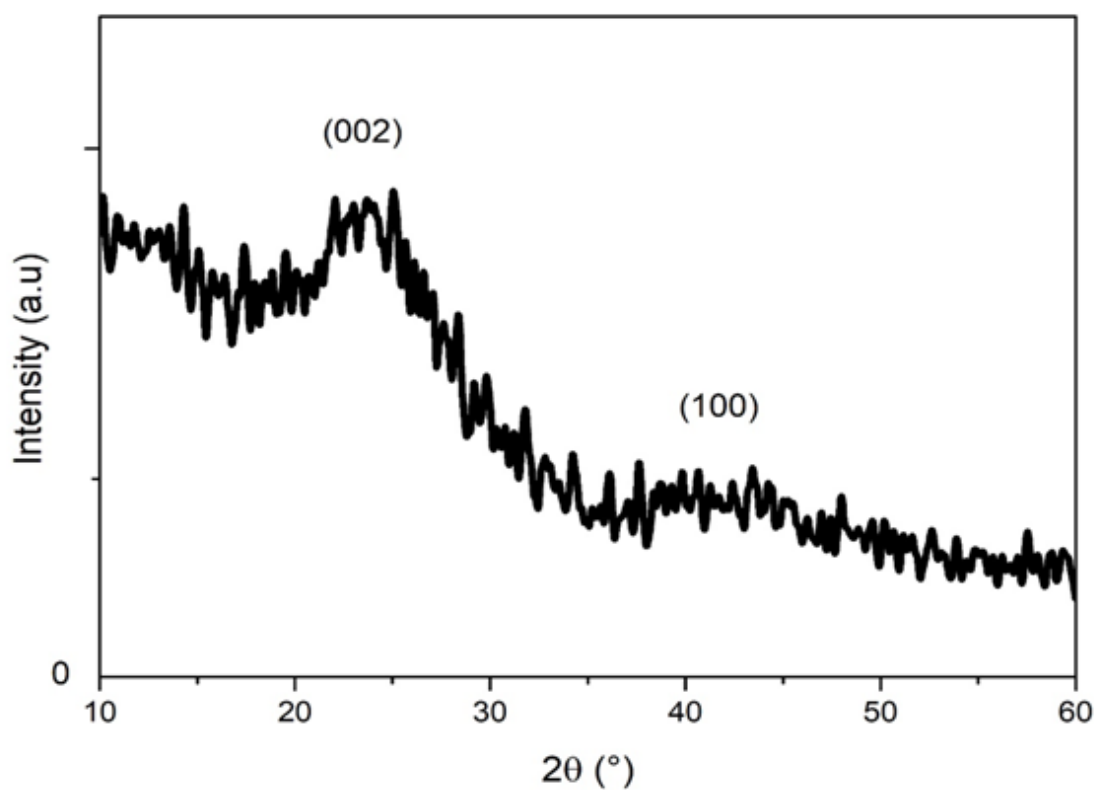


Figure III.2: X-ray Diffraction Pattern of BC Sample.

Figure III.4 presents a captivating SEM image that unveils the intricate microstructural landscape of BC sample. The image vividly captures the presence of small microskeletons distributed unevenly throughout the field of view, forming intriguing random conglomerates. These microskeletons exhibit a fascinating level of heterogeneity, suggesting diverse compositions or stages of carbonization within the BC matrix. The random clustering of these microskeletons implies intricate spatial relationships, potentially influenced by the complex interplay of precursor materials and processing conditions. Moreover, the conglomerates encapsulate a myriad of nano and micro pores, adding an additional layer of complexity to the microstructural architecture. These pores, characterized by their irregular distribution and varied dimensions, play a crucial role in influencing BC's surface area and porosity. The presence of unsystematic nano and micro pores underscores the dynamic nature of the carbonaceous structures, suggesting potential pathways for adsorption,

catalysis, or other environmentally relevant processes. The inhomogeneous distribution of microskeletons and the intricate pore network depicted in Figure III.4 provide valuable insights into the morphology and microscale architecture of BC. Such details are pivotal for understanding the BC's functional properties, making it a compelling material for diverse applications, including environmental remediation and sustainable resource utilization.

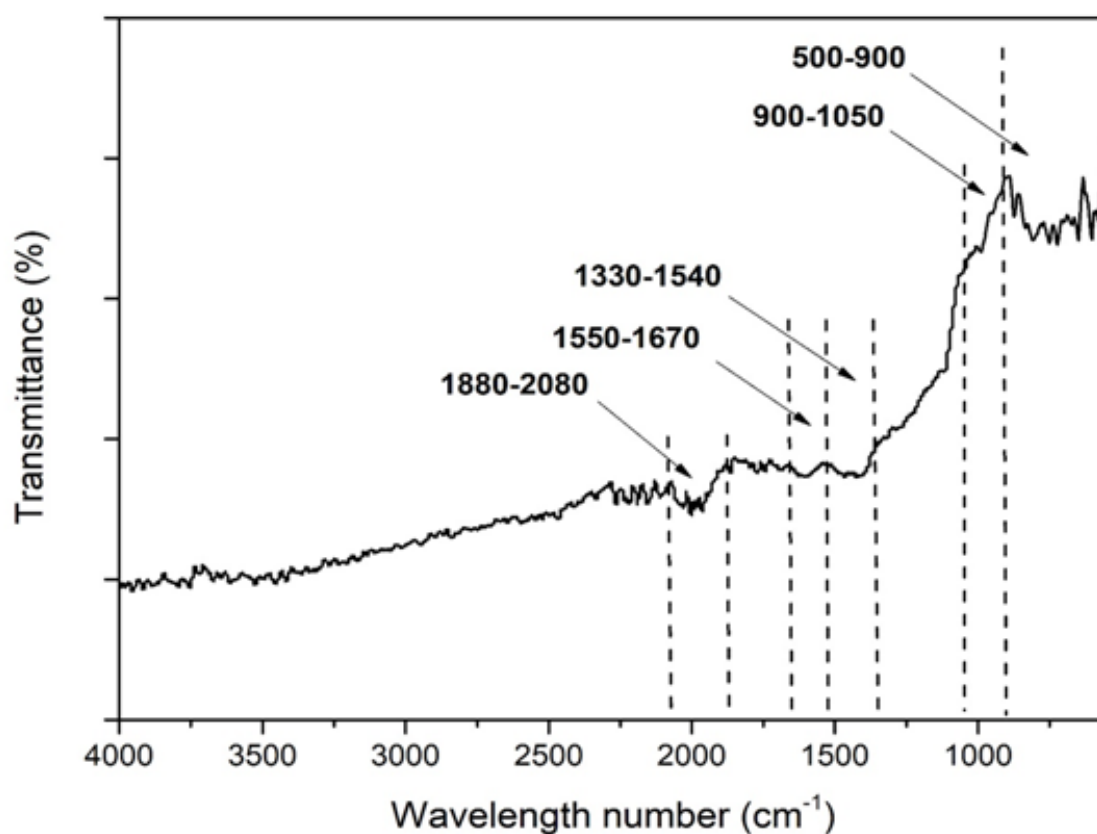


Figure III.3: FTIR Spectrum of BC Sample.

In Figure III.5, a meticulous zoom to 10 μm at a magnification of X1,000 unveils a captivating micrograph that delves into the finer details of the BC microstructure. The image distinctly showcases an abundance of microskeletons, each intricately arranged to form a visually striking pattern. Notably, these microskeletons serve as hosts to a fascinating array of sporadic nanotubes, characterized by diameters ranging between 4 to 10 nm. The organized series of nanotubes within each microskeleton hints at a well-defined structural hierarchy, suggesting a controlled

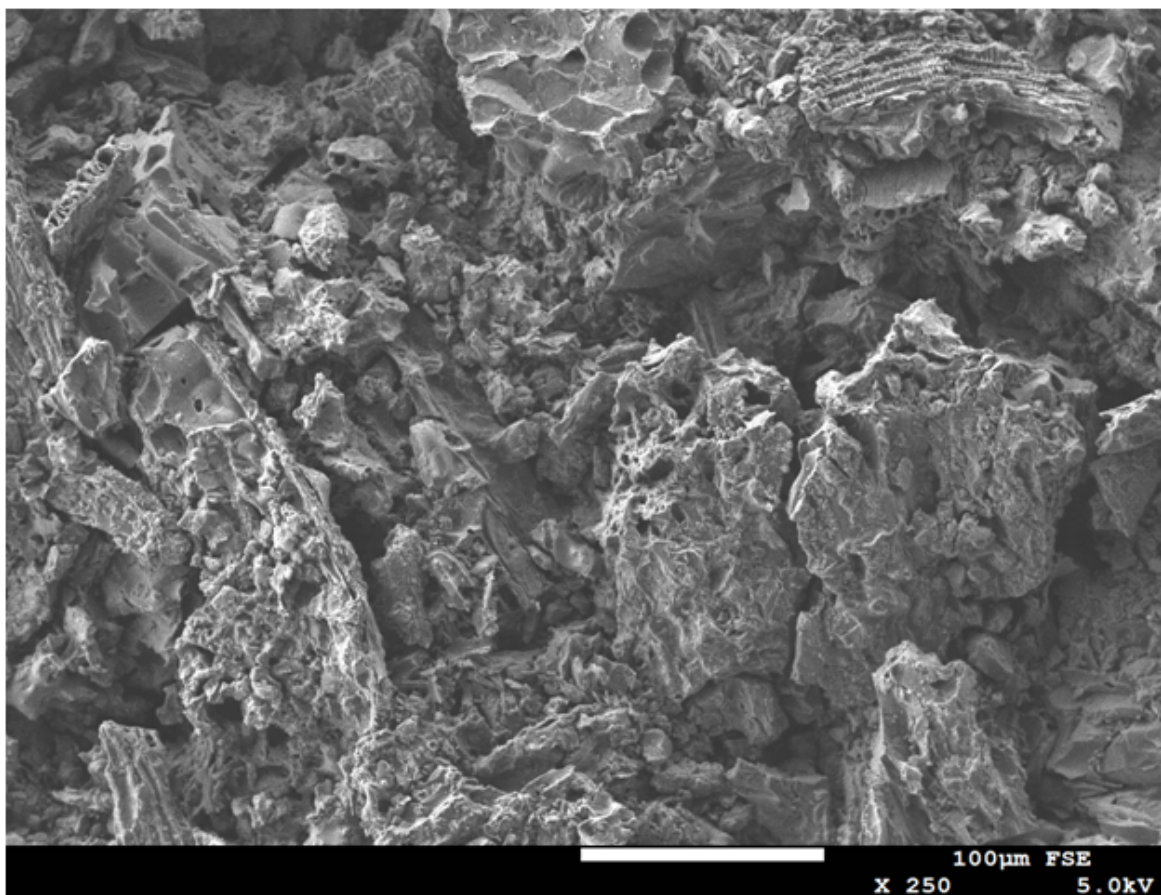


Figure III.4: SEM Micrograph of BC Sample at $100\mu\text{m}$, Magnification X2,500.

synthesis process or a unique carbonization pathway. The diametric diversity of the nanotubes introduces an additional layer of complexity, potentially signifying variations in growth conditions or precursor materials. The observed nanotubes, with dimensions within the nanoscale range, hold promising implications for BC's reactivity, adsorption capacity, and catalytic potential. This high-resolution micrograph in Figure III.5 provides a glimpse into the intricacies of BC's nanoscale architecture, unraveling the ordered interplay between microskeletons and sporadic nanotubes. The detailed exploration of these structures is instrumental in advancing our understanding of BC's functionalities, paving the way for tailored applications in nanotechnology, environmental science, and materials engineering.

The EDS results, as elucidated in Table III.5 and depicted in Figure III.6, offer a comprehensive insight into the elemental composition of BC. Carbon dominates the composition, constituting 81.87% by mass and a slightly higher proportion



Figure III.5: SEM Micrograph of BC Sample at $10\mu\text{m}$, Magnification X1,000.

of 86.64% by atom. This prevalence underscores the carbonaceous nature of BC, aligning seamlessly with the FTIR findings that highlighted the presence of alkanes, aromatics, and other carbon-based functional groups. Oxygen, the second most abundant element, contributes 13.23% by mass and 10.51% by atom. This presence underscores the role of oxygen-containing functional groups, corroborating with the FTIR's indication of C-O stretching in ethers and ketones. Nitrogen, at 1.71% by mass and 1.55% by atom, aligns with FTIR peaks indicative of C-N stretching in amines, providing further evidence of BC's heterogeneity in functional groups. Minor elements, including magnesium, calcium, phosphorus, and chlorine, collectively account for the remaining percentage. The presence of these elements, though in relatively small amounts, could influence BC's physicochemical properties and reactivity. The correlation between EDS and FTIR results offers a holistic understanding of the BC's elemental composition and functional groups, enriching our comprehension of its potential applications in adsorption, catalysis, and environmental

remediation.

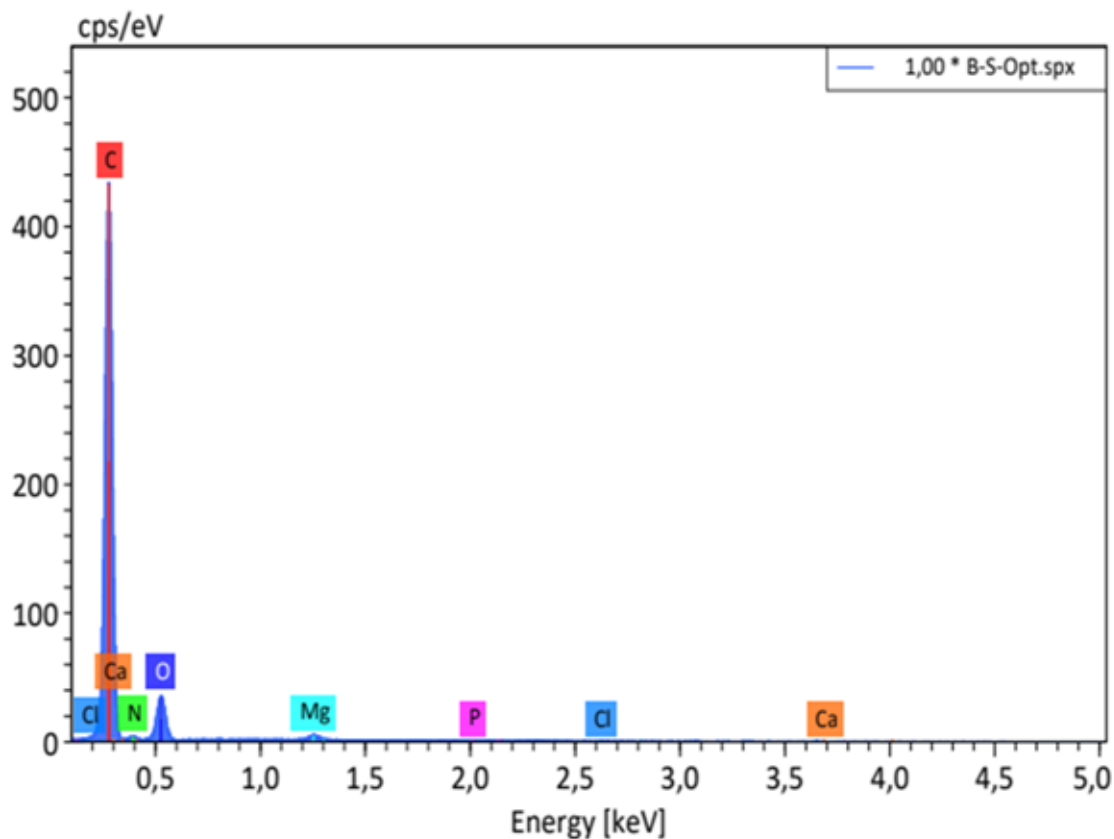


Figure III.6: EDS result of BC Sample.

Table III.5: EDS Results of BC Sample.

Element	Mass (%)	Atom (%)
Carbon	81.87	86.64
Oxygen	13.23	10.51
Nitrogen	1.71	1.55
Magnesium	1.06	0.55
Calcium	1.18	0.37
Phosphorus	0.56	0.23
Chlorine	0.39	0.14
Total	100.00	100.00

III.2.2 Morphological and Surface Analysis

The BET surface area analysis unveils the remarkable porous architecture of BC, shedding light on its potential as an effective adsorbent. At a single-point surface area measurement ($p/p^\circ = 0.072791378$), BC exhibits a substantial surface area of $647.5560 \text{ m}^2/\text{g}$. The BET surface area, calculated at $654.7851 \text{ m}^2/\text{g}$, further emphasizes the extensive interfacial regions available for adsorption processes.

Delving into the pore structure, the BJH adsorption cumulative surface area of pores within the diameter range of 1.7000 nm to 300.0000 nm is measured at $131.6077 \text{ m}^2/\text{g}$, showcasing a diverse range of pore sizes contributing to the overall porosity of BC. The corresponding desorption cumulative surface area is slightly lower at $119.9999 \text{ m}^2/\text{g}$, suggesting reversible adsorption-desorption dynamics within the pores.

Pore volume, a critical parameter influencing adsorption capacity, is quantified through BJH adsorption and desorption cumulative volumes within the 1.7000 nm to 300.0000 nm diameter range. BC exhibits an adsorption cumulative pore volume of $0.101663 \text{ cm}^3/\text{g}$ and a desorption cumulative pore volume of $0.094737 \text{ cm}^3/\text{g}$, underscoring the substantial void spaces available for molecular interactions.

The pore size distribution analysis provides further insights into BC's porous nature. The BJH adsorption and desorption average pore diameters, calculated using the formula $(4V/A)$, are found to be 3.0899 nm and 3.1579 nm, respectively. This suggests a well-defined and consistent pore structure, crucial for accommodating diverse adsorbates.

The synergy between the BET results and BC's morphological features, as observed in SEM and EDS analyses, underscores its potential as a high-surface-area adsorbent. The rich porous network, coupled with the diverse elemental composition, positions BC as a promising candidate for applications in environmental remediation and adsorption processes.

The comparison between results derived from RSM and BET analysis offers a comprehensive evaluation of the predictive model's efficacy in optimizing BC synthesis. The close alignment between the RSM-predicted SSA and the experimental BET SSA, registering at $652.146 \text{ m}^2/\text{g}$ and $654.7851 \text{ m}^2/\text{g}$ respectively, underscores the model's precision in capturing critical surface area dynamics. Similarly, the agreement in predicted and experimental micropore volumes at $0.191 \text{ cm}^3/\text{g}$ and $0.190 \text{ cm}^3/\text{g}$ respectively showcases the model's effectiveness in characterizing micropore formation. Notably, the discrepancy in total pore volume, where the RSM-predicted value of $0.0947 \text{ cm}^3/\text{g}$ differs from the experimental BET value of $0.185 \text{ cm}^3/\text{g}$, prompts a nuanced consideration. However, the emphasis on SSA as a primary indicator for BC's adsorption efficiency underscores the overarching success of the model in optimizing key parameters, prioritizing SSA over total pore volume in delineating BC's potential applications.

In Figure III.7, the pivotal pHpzc for the BC is graphically represented, standing at 7.58. This result serves as a cornerstone in understanding the dynamic surface charge properties of the BC, crucial for its applications in adsorption processes. The neutral surface charge at pHpzc implies an equilibrium between positive and negative charges, with the BC transitioning from a propensity to attract positively charged species before pH 7.58 to a preference for negatively charged substances beyond this point. This nuanced insight, visually captured in Figure III.7, establishes a foundation for interpreting the electrochemical behavior of the BC. It signifies its adaptability and selectivity in adsorbing contaminants, paving the way for tailored applications in environmental remediation and other adsorption-driven processes. The pHpzc result, intricately woven into the visual representation in Figure III.7, encapsulates the versatility and potential efficacy of BC as a dynamic adsorbent.

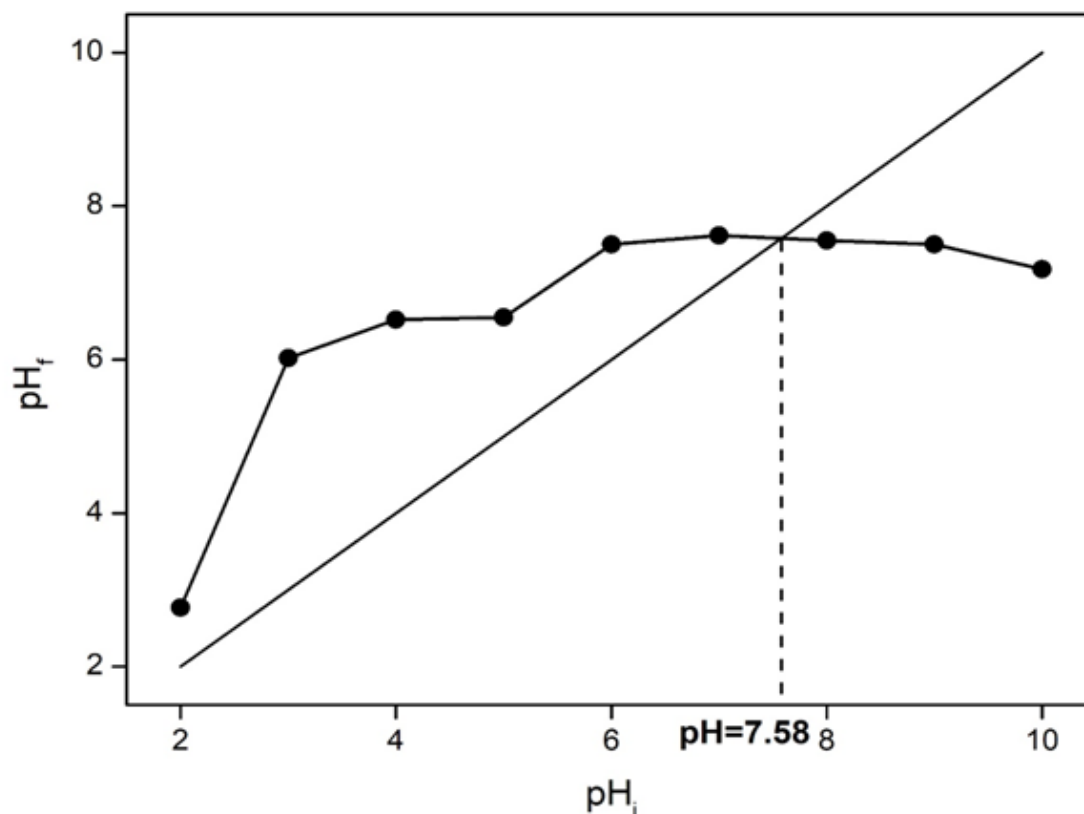


Figure III.7: pH_{PZC} Graphical presentation of BC Sample.

III.3 Adsorption Assessment - Unveiling BC's Affinity for VOCs

III.3.1 Kinetic Studies: Probing BC's Affinity for VOCs

In a comprehensive exploration of adsorption kinetics, our meticulous experimental design sought to unravel the intricate interplay between BC and volatile organic compounds VOCs, particularly PCE and TCE. Figures III.8 and III.10 intricately illustrate the kinetic presentation of PCE and TCE adsorption into BC, respectively. These figures showcase the experimental data alongside fits generated by the PFO and PSO models, providing a detailed analysis of the adsorption kinetics.

For PCE removal (Figure III.8 and III.9), BC exhibited a profound affinity, scientifically substantiated by the PSO model, revealing a maximum q of 85.9675 mg/g,

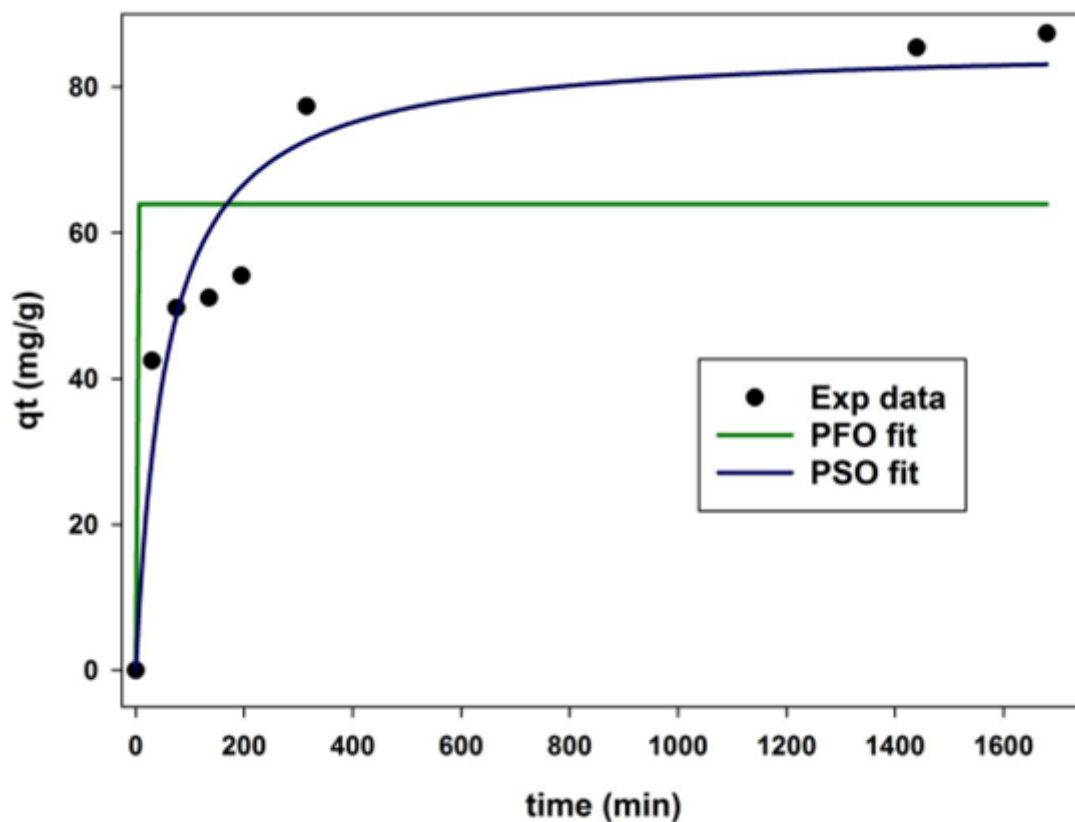


Figure III.8: Kinetic presentation of PCE adsorption into BC: Experimental data, PFO fit and PSO fit.

Table III.6: Fitted Models Data for Kinetic Study of VOC Adsorption into BC and AC.

PCE Removal		
Adsorbent	BC	AC
Fitted Model	PSO	PSO
q (mg/g)	85.9675	44.7409
K (min ⁻¹)	0.0002	26826.0932
R ²	0.9594	0.9998
TCE Removal		
Adsorbent	BC	AC
Fitted Model	PSO	PSO
q (mg/g)	86.6836	47.5400
K (min ⁻¹)	0.0003	0.0031
R ²	0.9810	0.9998

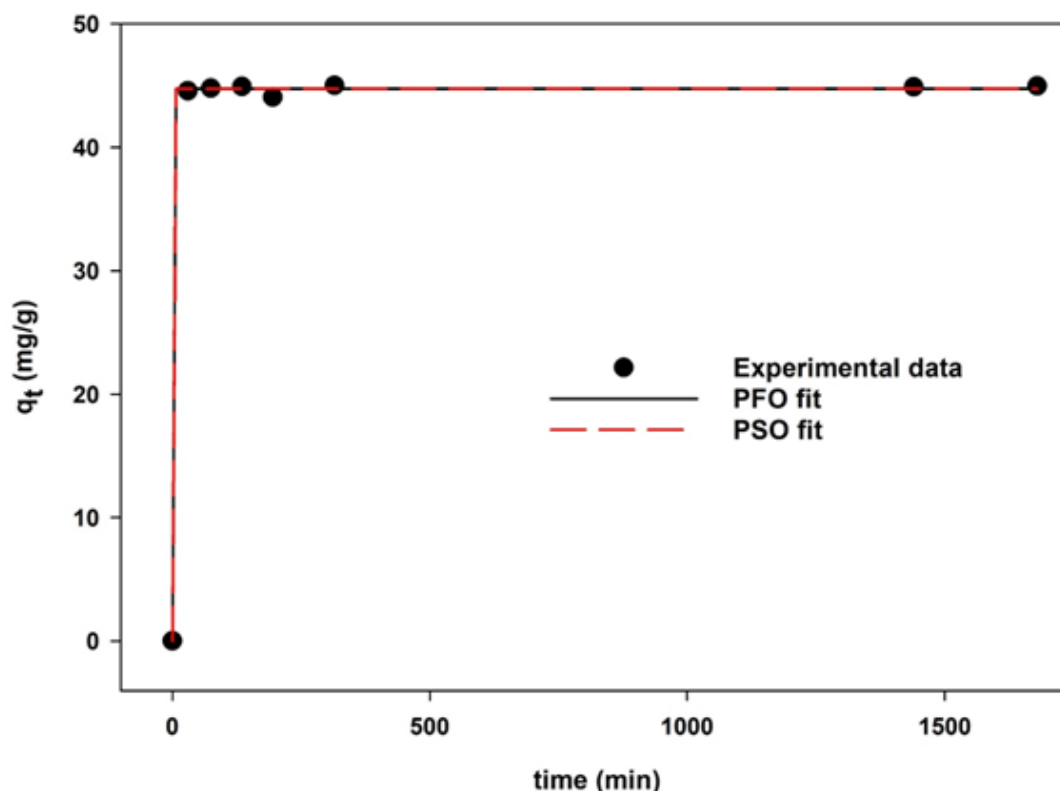


Figure III.9: Kinetic presentation of PCE adsorption into AC: Experimental data, PFO fit and PSO fit.

K of 0.0002, and an impressive R of 0.9594. This emphasizes BC's robust capability in PCE adsorption. Notably, in comparison with commercial AC, BC showcased a significantly higher q value under the PSO model, underscoring its superior adsorption potential.

Extending the kinetic study to TCE removal (Figure III.10 and III.11), BC, under the PSO model, revealed a remarkable q of 86.6836 mg/g, K of 0.0003, and R of 0.9810, highlighting its efficacy in TCE adsorption. In contrast, commercial AC exhibited a lower q (47.5400 mg/g) and a comparable K (0.0031) under the PSO model. The findings, meticulously presented in Table III.6, accentuate BC's superiority over commercial AC in both PCE and TCE removal, providing a nuanced understanding of their respective kinetic behaviors.

This thorough study not only establishes BC's prowess in VOC removal but

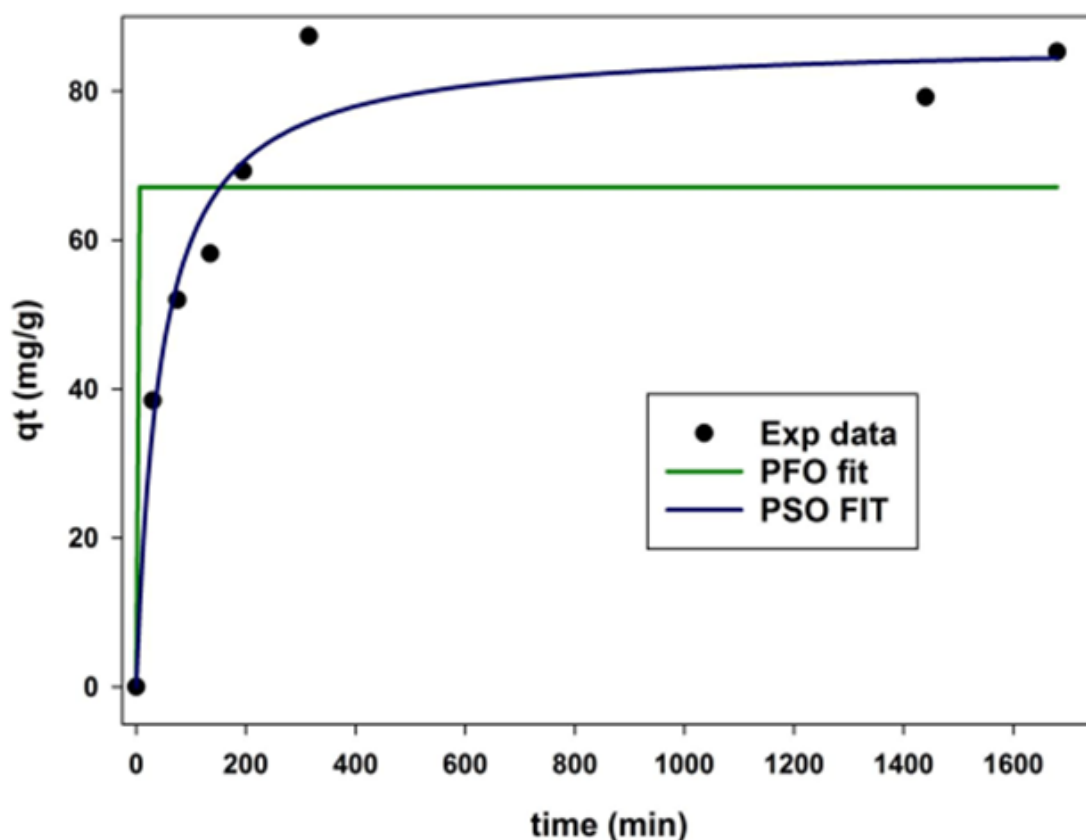


Figure III.10: Kinetic presentation of TCE adsorption into BC: Experimental data, PFO fit and PSO fit.

also chemically relates its kinetic performance to the intricacies of its morphology, as elucidated in previous analyses. The superior adsorption capacities of BC, evident in the kinetic models, find correlation with its specific surface area, pore volume, and morphology, highlighting the interconnected aspects influencing its efficacy as an adsorbent for VOCs.

III.3.2 Isotherm Studies: Unveiling BC's Interaction with VOCs

In the realm of isotherm studies, the nuanced interaction between BC and VOCs, specifically PCE and TCE, is meticulously unraveled through experimental scrutiny. Figures III.12 and III.14 intricately depict the isotherms of PCE and TCE adsorption into BC, respectively, juxtaposing the experimental data with the well-established Freundlich and Langmuir isotherms.

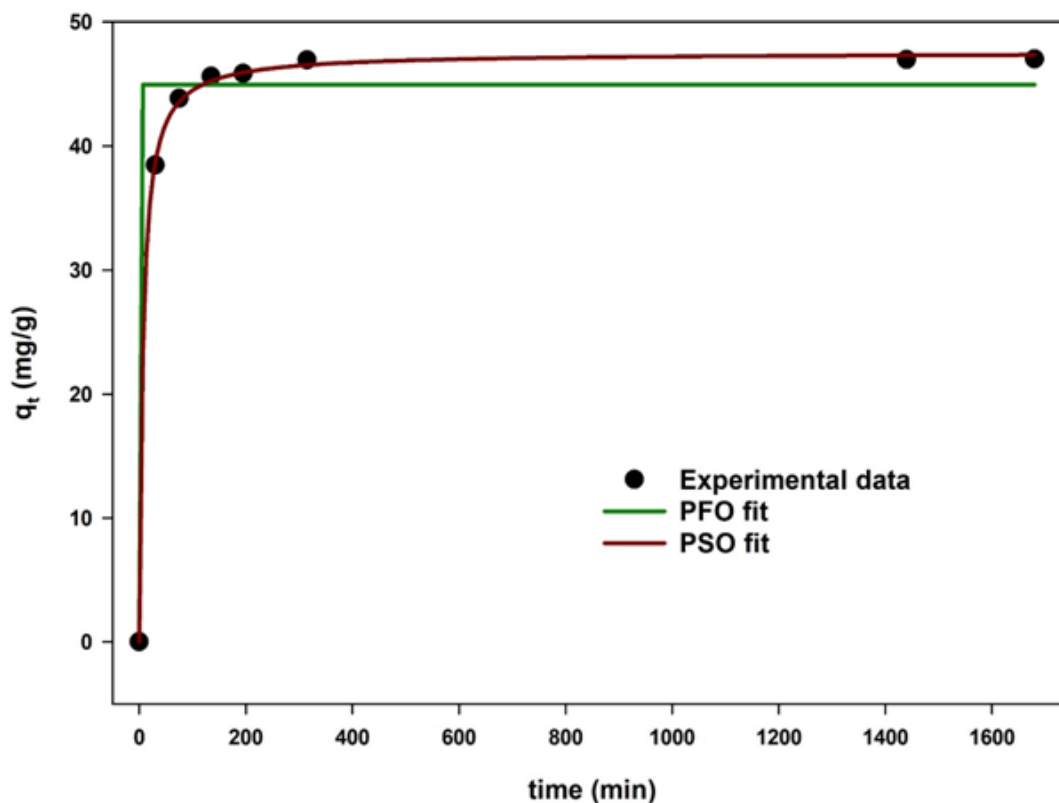


Figure III.11: Kinetic presentation of TCE adsorption into AC: Experimental data, PFO fit and PSO fit.

For PCE removal (Figure III.12 and III.13), BC exhibits a remarkable fit under the Freundlich model with parameters k at 43.5601, n at 0.6167, and an impressive R of 0.9940. The Langmuir model for BC reveals a Q_{\max} of 283.2054mg/g, k of 0.1496, and R of 0.9862. These findings underscore the favorable adsorption characteristics of BC for PCE, outperforming commercial AC as evidenced by higher k values in both Freundlich and Langmuir models.

The trend continues in TCE removal (Figure III.14 and III.15), where BC demonstrates a robust Freundlich fit (k : 72.3580, n : 0.4866, R : 0.9826) and Langmuir fit (Q_{\max} : 586.1234mg/g, k : 0.0777, R : 0.9984). BC consistently surpasses AC in both models, emphasizing its superior affinity for TCE. The Freundlich model for AC yields k at 7.8316, n at 0.8534, and R at 0.9990, while the Langmuir model displays Q_{\max} of 713.4764mg/g, k of 0.0085, and R of 0.9991.

Table III.7 serves as a consolidated repository of these results, facilitating a comprehensive comparison of BC and AC in terms of isotherm models for both PCE and TCE removal. The intricacies unveiled in this isotherm study not only enrich our comprehension of BC's adsorption behavior but also elucidate its potential as an exceptionally effective adsorbent for VOCs, a prowess that distinctly surpasses the performance of commercial AC. This aligns with the earlier BET results, where the superior surface area and pore characteristics of BC were identified, establishing a chemical connection between its morphology and exceptional adsorption capabilities for VOCs.

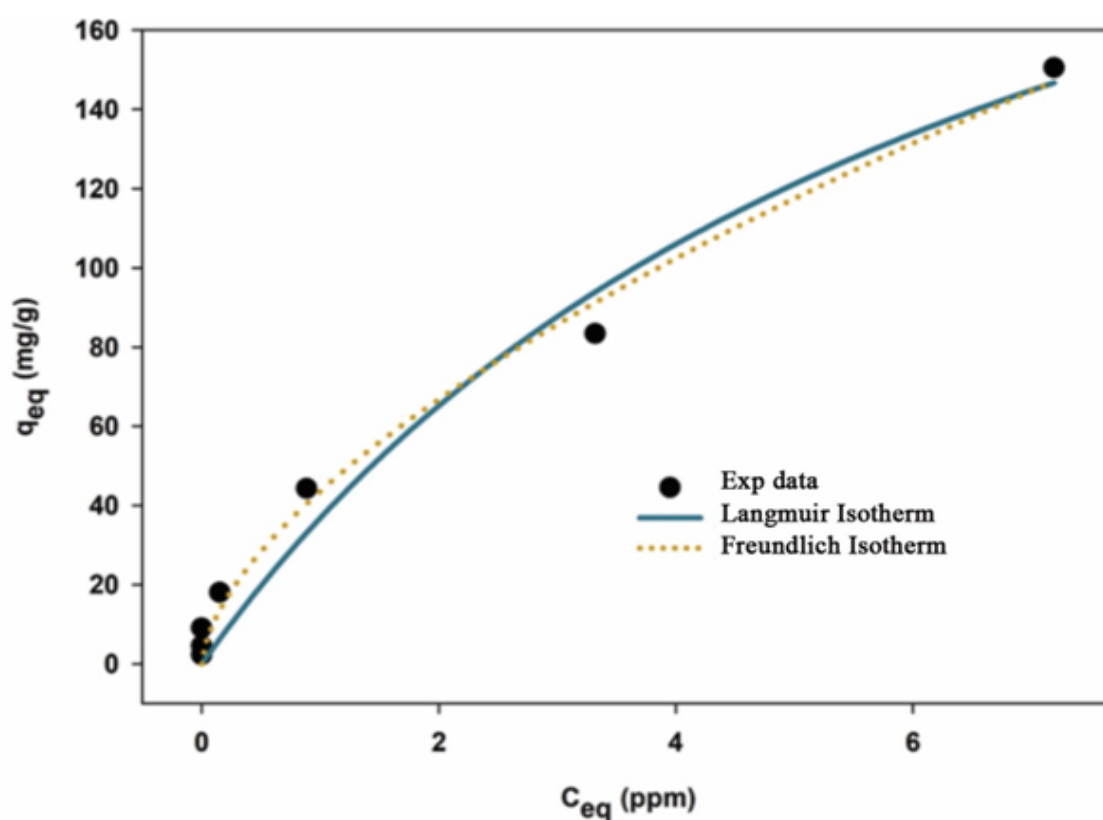


Figure III.12: Isotherms presentation of PCE adsorption into BC: Experimental data, Freundlich and Langmuir isotherms.

BC has been found to exhibit superior adsorption capabilities for TCE PCE, with a particularly pronounced efficiency under the PSO kinetic model. The Freundlich and Langmuir isotherms have been employed to aptly characterize BC's multi-layered and monolayered adsorption processes for TCE and PCE, respectively.

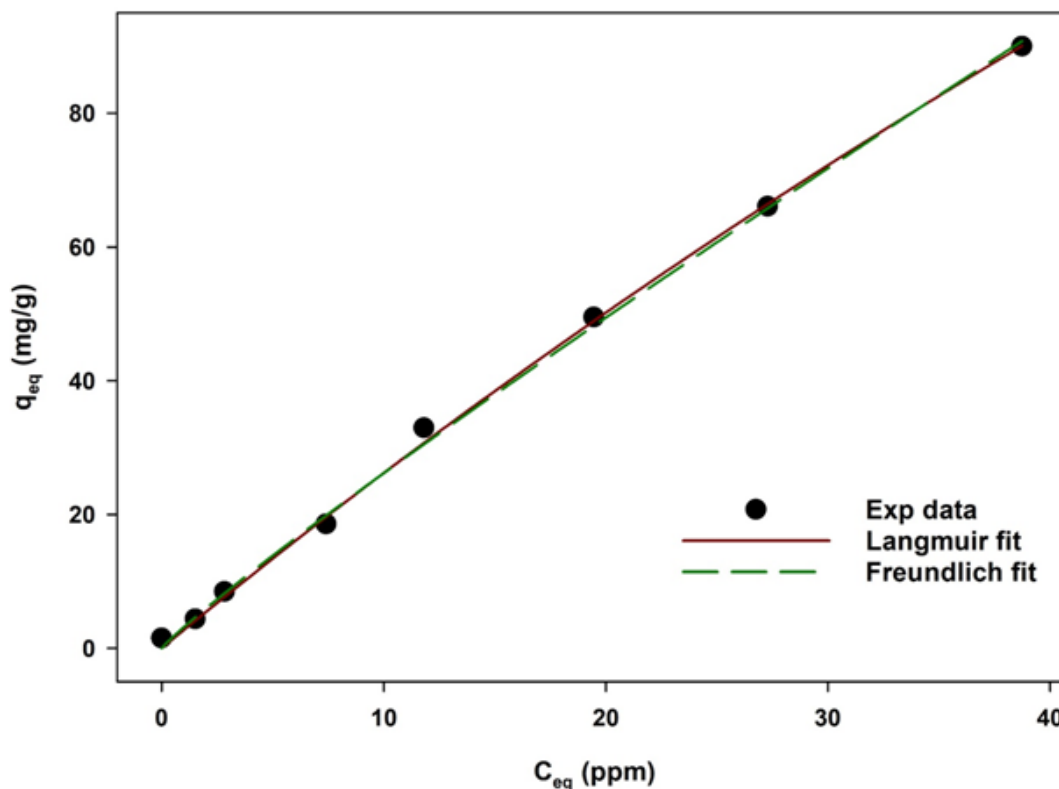


Figure III.13: Isotherms presentation of PCE adsorption into AC: Experimental data, Freundlich and Langmuir isotherms.

The nanotube morphology revealed by SEM further elucidates BC's exceptional adsorption kinetics, emphasizing its potential as a highly effective and promising adsorbent for the removal of VOCs.

The fit with the PSO model accentuates the robustness of BC's adsorption kinetics, providing a comprehensive understanding of the temporal evolution of the adsorption process. The PSO model, based on the second-order rate equation, considers the concentration of adsorbate and the number of available sites for adsorption. The superior fit of BC with the PSO model suggests a prominent role of chemisorption in the adsorption mechanism, indicating a strong chemical affinity between BC and the chlorinated solvents (Capra et al. [2017]).

The Freundlich isotherm, applicable to heterogeneous systems, describes

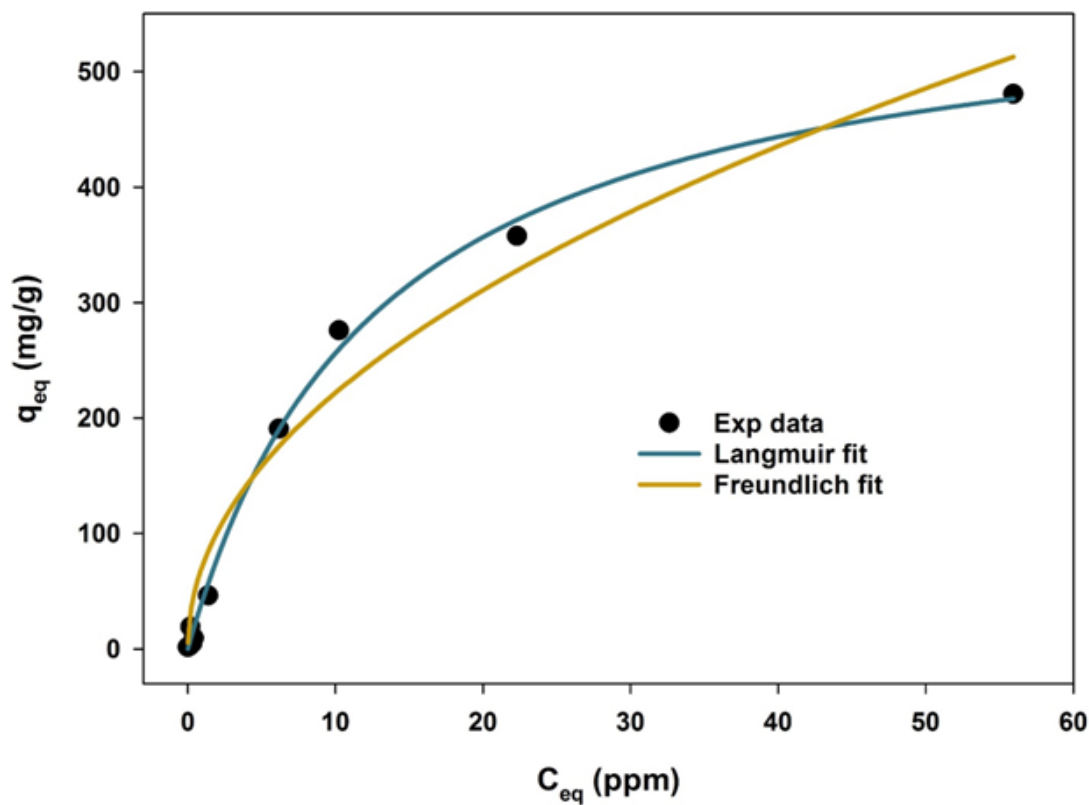


Figure III.14: Isotherms presentation of TCE adsorption into BC: Experimental data, Freundlich and Langmuir isotherms.

the adsorption process on a multi-layered surface. In the case of TCE, the elevated rate constants (k values) and a favorable fit under the Freundlich model imply a higher affinity and extensive adsorption on BC's surface. This is indicative of the heterogeneous nature of BC, allowing for varied adsorption energies across its surface. On the other hand, the Langmuir isotherm, which assumes a monolayer adsorption process on a homogeneous surface, aligns seamlessly with the adsorption behavior of PCE on BC. The elevated affinity observed for PCE under the Langmuir model suggests a more uniform distribution of adsorption sites on BC's surface, accommodating PCE molecules in a monolayer fashion (Ji et al. [2022]).

The molecular distinction between TCE and PCE plays a pivotal role in influencing their interactions with BC. The linear structure of TCE with three chlorine atoms and the additional chlorine atom in the configuration of PCE contribute to

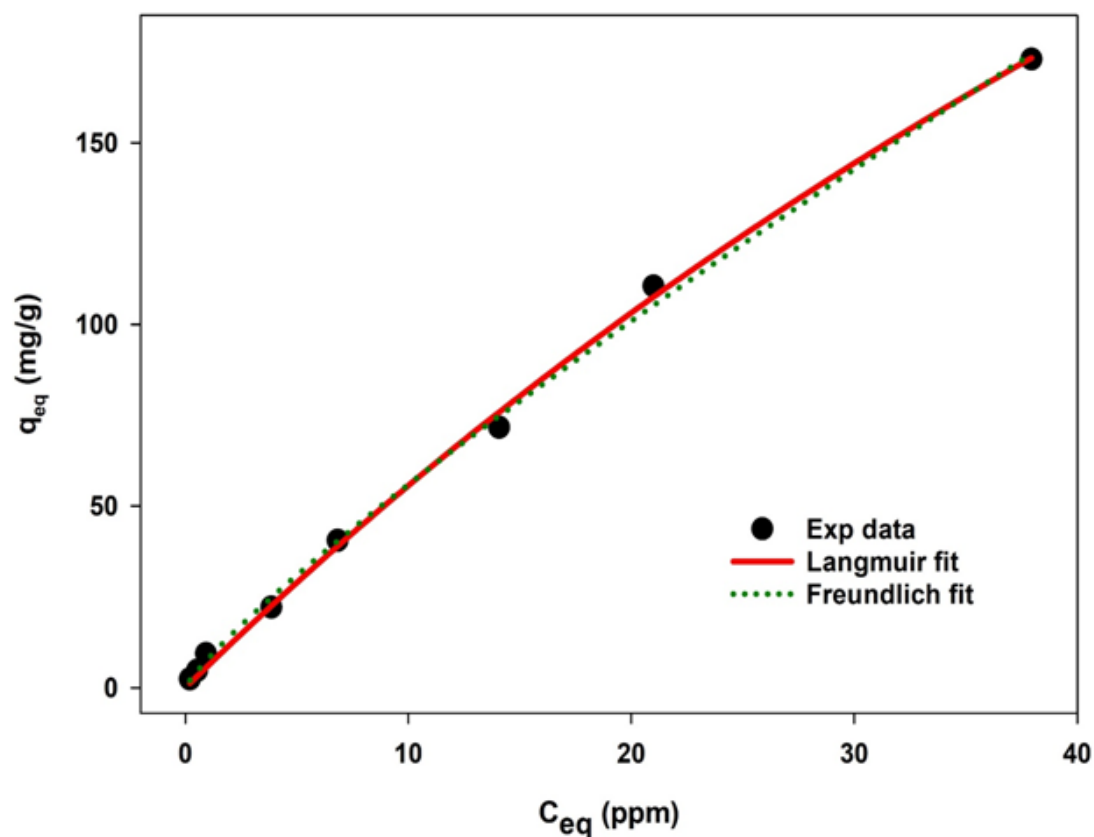


Figure III.15: Isotherms presentation of TCE adsorption into AC: Experimental data, Freundlich and Langmuir isotherms.

their disparate molecular weights and structures. The nuanced differences in their molecular architectures likely influence their adsorption behaviors on BC, as corroborated by the SEM analysis revealing nanotubes and a complex porous structure on BC's surface.

In conclusion, the comprehensive scientific analysis of BC's adsorption capabilities for TCE and PCE, elucidated through fitting with the PSO model and the Freundlich and Langmuir isotherms, provides valuable insights into the specific molecular interactions governing the adsorption process. The heightened affinity of BC for both VOCs, driven by its unique surface morphology and chemical composition, underscores its potential as a promising adsorbent for a wide spectrum of chlorinated contaminants. This study emphasizes the necessity of considering

Table III.7: Fitted Models Data for Isotherm Study of VOC Adsorption into BC and AC.

PCE Removal			
Adsorbent		BC	AC
Freundlich	k (L/mg)	43.5601	3.1559
	n	0.6167	0.9187
	R ²	0.9940	0.9954
Langmuir	Qmax (mg/g)	283.2054	593.1827
	k (L/mg)	0.1496	0.0046
	R ²	0.9862	0.9957
TCE Removal			
Adsorbent		BC	AC
Freundlich	k (L/mg)	72.3580	7.8316
	n	0.4866	0.8534
	R ²	0.9826	0.9990
Langmuir	Qmax (mg/g)	586.1234	713.4764
	k (L/mg)	0.0777	0.00856
	R ²	0.9984	0.9991

molecular idiosyncrasies for tailoring effective environmental remediation strategies using BC as an adsorbent.

General Conclusion

In conclusion, the exploration of BC synthesis from DPS in the Biskra region of Algeria has been a nuanced scientific endeavor, guided by precision and innovation. The journey through the optimization of BC preparation using RSM has unearthed a plethora of insights into the delicate interplay of pyrolysis time and temperature. The success of the optimization process, as evidenced by a D value of 0.843, underlines the meticulous control required for tailoring BC to specific environmental and economic contexts.

The in-depth analysis of the BC preparation process has shed light on the critical parameters influencing SSA, V_m , V_t , and final Y. The examination of these parameters not only serves as a testament to the efficacy of the predictive models but also provides a roadmap for further exploration and refinement. The validation of optimized conditions against experimental values reinforces the reliability of the predictive model, offering a robust framework for BC synthesis.

Moving beyond the laboratory, the scientific and environmental impact of optimizing BC preparation becomes evident. The emphasis on SSA as a key determinant of BC's adsorption efficiency underscores its potential in addressing global warming through enhanced carbon sequestration capabilities. The economic viability of BC synthesis, facilitated by RSM-guided precision, aligns with the broader goals of sustainable resource utilization. This optimization journey, therefore, represents a harmonious blend of scientific rigor and practical applicability.

The characterization of BC delves into its structural and compositional intricacies, unraveling a complex tapestry of graphite crystalline phases, diverse func-

tional groups, and a microstructural landscape that holds promise for various applications. XRD and FTIR analyses showcase the intimate relationship between precursor material and derived biochar structure. SEM imagery paints a vivid picture of BC's microstructural architecture, revealing unsystematic nano and micro pores and nanotubes that hold potential for catalytic applications.

EDS results provide a comprehensive understanding of BC's elemental composition, with carbon dominating alongside oxygen, nitrogen, and minor elements. This elemental composition enriches our comprehension of BC's potential in diverse fields such as adsorption, catalysis, and environmental remediation.

The assessment of surface properties and reactivity, through BET surface area analysis and pH at the point of zero charge pHPzc, further solidifies BC's potential as a versatile adsorbent. The alignment between RSM-predicted SSA and experimental BET SSA underscores the precision of the model in capturing critical surface area dynamics. The electrochemical behavior highlighted by pHPzc establishes BC's adaptability and selectivity in adsorbing contaminants.

In the broader academic context, this research contributes not only to the field of BC synthesis but also to the evolving narrative of sustainable and tailored adsorbents. The integration of RSM as a guiding framework provides a methodological template for researchers exploring similar avenues. The nuanced exploration of BC's characteristics opens avenues for interdisciplinary collaborations, as its applications span environmental science, materials science, and catalysis.

As we navigate the intricate landscape of BC synthesis and characterization, this research serves as a testament to the power of precision and innovation in advancing scientific knowledge. The optimization journey undertaken, the structural intricacies unveiled, and the potential applications discovered collectively contribute to a comprehensive understanding of BC's role in addressing contemporary environmental challenges.

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