TREATMENT OF METHYL RED FROM TEXTILE WASTEWATER USING ACTIVATED CARBON FROM BLEND OF PALM KERNEL SHELL AND COCONUT SHELL

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A PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING, UNIVERSITY OF BENIN, BENIN CITY, NIGERIA IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE AWARD OF BACHELOR OF ENGINEERING (B.ENG) IN CHEMICAL ENGINEERING

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CERTIICATION

This is to certify that this research project was carried out by IKECHI EMMANUEL with matriculation number ENG2002045 in the Department of Chemical Engineering at the University of Benin, Benin city, Edo state Nigeria.

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DEDICATION

This project work is dedicated to ALMIGHTY GOD, who has brought me thus far in this my academic journey, the source of my life and everything. Special appreciation also to the best parents in the world, Mr. and Mrs. IKECHI for their contribution and support financially, spiritually, morally in this journey, they have always been my source of motivation throughout my academic journey.

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I'm also grateful to the lecturers for their tutoring which made my stay here a great one. I will also want to appreciate the lab assistant in the department laboratory, in person of Mr. Collins whose support was also helpful.

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ABSTRACT

The discharge of untreated textile wastewater containing synthetic dyes poses significant environmental and public health risks due to its toxicity and resistance to conventional degradation processes. This research explores a sustainable and cost-effective solution by developing and evaluating a novel activated carbon (AC) adsorbent derived from a blend of two abundant agricultural wastes: Palm Kernel Shell (PKS) and Coconut Shell (CS).

This study aimed to treat synthetic wastewater contaminated with Methyl Red dye. The PKS and CS were individually carbonized and chemically activated using potassium hydroxide (KOH). The resulting activated carbons were blended in a 1:1 ratio to create a composite adsorbent (PKS-CS AC). The adsorbent was extensively characterized using Brunauer-Emmett-Teller (BET) analysis, which revealed a specific surface area of 275.762 m^2/g and a well-developed microporous and mesoporous structure, complemented by Fourier-Transform Infrared Spectroscopy (FTIR) that identified key functional groups (O-H, C=O, C-O) crucial for adsorption.

A series of batch adsorption experiments were conducted, and the process was optimized using Response Surface Methodology (RSM) based on a Central Composite Design (CCD). The influence of critical operational parameters—adsorbent dosage (PKS-AC and CS-AC), contact time, and initial dye concentration—on Methyl Red removal efficiency was investigated. The ANOVA of the quadratic model confirmed its high significance, with an R^2 value of 0.9501, indicating the model accurately represented the experimental data. The optimization results identified the optimal conditions as 1.65 g/L of CS-AC, 6.13 g/L of PKS-AC, a contact time of 70.75 minutes, and an initial dye concentration of 328.1 mg/L, achieving a predicted dye removal efficiency of 93.75%.

The findings demonstrate that the blended PKS-CS activated carbon is a highly effective, low-cost, and eco-friendly adsorbent for the removal of Methyl Red dye from wastewater. The study successfully validates the synergistic potential of blending agro-waste precursors for enhanced adsorption performance, offering a promising, sustainable alternative for textile effluent treatment that aligns with circular economy principles by converting waste into a valuable resource.

Keywords: Activated Carbon, Palm Kernel Shell, Coconut Shell, Methyl Red, Adsorption, Textile Wastewater, Response Surface Methodology (RSM), Optimization, Agro-Waste.

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CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF STUDY

Treatment of wastewater using activated carbon from palm kernel shell is a critical environmental concern, as it plays a crucial role in protecting public health, safeguarding the environment, and promoting sustainable development (World Health Organization, 2019). The ineffective treatment of wastewater can lead to the release of pollutants into the environment, causing harm to aquatic life, contaminating drinking water sources, and posing risks to human health (European Commission, 2020). Furthermore, the discharge of untreated or inadequately treated wastewater into water bodies can lead to the degradation of water quality, affecting not only the environment but also human health and the economy (Kümmel et al., 2018).

The utilization of activated carbon produced from palm kernel (Elaeis guineensis) kernel shell, a readily available and renewable biomass material, derived from the abundant waste streams of the palm oil industry, primarily in tropical regions, presents a highly promising sustainable solution for the effective removal of diverse pollutants, encompassing heavy metals, organic compounds, and dyes, from wastewater (Adeyemi et al., 2017). The production of activated carbon from palm kernel shell can also provide an additional income stream for palm oil producers, while also reducing waste disposal costs, and promoting a more circular and sustainable economy (Benin City Government, 2020).

Moreover, the use of palm kernel shell-based activated carbon can also help to mitigate climate change by reducing greenhouse gas emissions from wastewater treatment processes, and promoting the use of renewable energy sources (International Energy Agency, 2020). Additionally, the adoption of sustainable wastewater treatment technologies, such as palm kernel shell-based activated carbon, can contribute to the achievement of the United Nations' Sustainable Development Goals (SDGs), particularly Goal 6, which aims to ensure access to clean water and sanitation for all (United Nations, 2020).

Ultimately, the development and implementation of sustainable wastewater treatment technologies, such as palm kernel shell-based activated carbon, is critical for protecting the environment, promoting public health, and supporting sustainable development. Therefore, further research and development is needed to fully explore the potential of palm kernel shell-based activated carbon for wastewater treatment, and to identify opportunities for its widespread adoption and implementation. Moreover, the use of palm kernel shell-based activated carbon can also provide a sustainable solution for the removal of emerging pollutants, such as pharmaceuticals and personal care products (PPCPs), from wastewater (Kümmel et al., 2018). The presence of PPCPs in wastewater has become a growing concern, as they can have adverse effects on aquatic life and human health (World Health Organization, 2019).

The application of palm kernel shell-based activated carbon in wastewater treatment can also provide a cost-effective solution for small-scale wastewater treatment systems, particularly in rural and peri-urban areas (Adeyemi et al., 2017). The use of palm kernel shell-based activated carbon can help to reduce the capital and operating costs associated with wastewater treatment, making it a more accessible and affordable solution for communities with limited resources. the production of palm kernel shell-based activated carbon can also provide a valuable opportunity for the creation of jobs and income generation in rural areas (International Labour Organization, 2020). The development of a sustainable industry based on the production of palm kernel shell-based activated carbon can help to stimulate local economic growth, while also promoting sustainable development and reducing poverty.

The use of palm kernel shell-based activated carbon in wastewater treatment can also provide a sustainable solution for the management of wastewater in areas with limited access to energy and other resources (United Nations Development Programme, 2020). The application of palm kernel shell-based activated carbon in wastewater treatment can help to reduce the energy requirements associated with wastewater treatment, making it a more sustainable and environmentally friendly solution.

Furthermore, the production of palm kernel shell-based activated carbon can also provide a valuable opportunity for the creation of new products and services, such as activated carbon-based filtration systems and wastewater treatment services (European Commission, 2020). The development of a sustainable industry based on the production of palm kernel shell-based activated carbon can help to stimulate innovation and entrepreneurship, while also promoting sustainable development and reducing poverty.

One of the most significant economic sectors in the world is the textile industry, which creates jobs and makes significant contributions to both developed and developing nations' GDPs. Textile manufacturing is seen as a component of industrial development that accounts for the largest portion of export earnings in a number of emerging countries, including Bangladesh, Vietnam, China, and India. Notwithstanding its economic significance, the textile sector has been regarded as one of the biggest users of chemicals and water, raising serious environmental concerns. Large volumes of wastewater are produced throughout the spinning, weaving, dyeing, printing, and finishing stages of the textile production process. These wastewaters contain complex mixes of contaminants, including heavy metals, dyes, salts, and other dangerous organic and inorganic substances. High levels of chemical and biological oxygen demand (COD and BOD), as well as the presence of harmful, non-biodegradable dyes, are the primary characteristics of textile wastewater. Since they are synthetic organic molecules, these dyes are actually made to be extremely resistant to fading in the presence of chemicals, water, and sunshine. Because of their resistance to natural biodegradation, they continue to exist in the environment, reducing the amount of light that aquatic life can absorb and impeding photosynthesis. Indeed, research has shown that untreated textile effluents can have detrimental ecological effects, including contaminating drinking water sources and causing hazardous compounds to bioaccumulate in aquatic creatures (Donkadokula et al., 2020).

In addition to dyes, heavy metals such as copper, lead, cadmium, and chromium are frequently found in textile waste water

1.2 PROBLEM STATEMENT

More wastewater is being released into natural water bodies without proper treatment as a result of the textile industry's explosive growth. High levels of complex contaminants, such as heavy metals and synthetic dyes that are impervious to standard biological degradation processes are found in textile effluents. Therefore, wastewater that has been left untreated or just partially treated can lead to major environmental issues such soil contamination, water pollution, and damage to aquatic ecosystems. Current wastewater treatment techniques have a number of drawbacks. Traditional techniques, such activated sludge and coagulation-flocculation, frequently fall short of fully eliminating color and harmful materials from textile effluent.

Additionally, these processes produce a lot of sludge that needs to be treated and disposed of further, increasing operating expenses and the environmental burden (Hynes et al., 2020). The sophisticated membrane methods

1.3 AIM OF STUDY

1.3.1 Aim of the Study

The aim of this research is to use activated carbon from a blend of palm kernel shells and coconut shell to treat wastewater containing dyes (Methyl red).

1.4 OBJECTIVES OF THE STUDY

(i). To integrate and characterize the activated carbon prepared from blended palm kernel shell and coconut shell

(ii) To procure dye wastewater (Methyl red)

(iii). To investigate the effects of operating conditions (e.g., temperature, contact time, initial dye concentration) on the adsorption process using the activated carbon from the palm kernel shell

(iv). To optimize the adsorption process using Response Surface Methodology(RSM)

(v). To integrate and characterize the procure dye wastewater (Methyl red')

(vi) Evaluation of the efficiency of blend palm kernel shell and coconut shell activated carbon in removing methyl red dye from synthetic wastewater.

1.5 SCOPE OF STUDY

This research is focused on the treatment of textile wastewater contaminated with **Methylene Red dye** using activated carbon derived from a blend of palm kernel shell (PKS) and coconut shell (CS) source at NIFOR, Benin City. The study encompasses the systematic conversion of these two agro-waste materials into a blended form of activated carbon and evaluates its potential as an adsorbent for the removal of synthetic dyes from aqueous solutions.

The scope of the project covers the collection, preparation, carbonization, and chemical activation of palm kernel shell and coconut shell, followed by blending of the resulting activated carbons to achieve a material with enhanced adsorptive characteristics. It includes the optimization of activation conditions, such as activating agent concentration, activation temperature, and carbonization time, with the goal of maximizing surface area and porosity of the final adsorbent.

Furthermore, the study involves a detailed characterization of the blended activated carbon using relevant physicochemical methods such as BET surface area analysis, Fourier Transform Infrared Spectroscopy (FTIR), pH point of zero charge (pHpzc), moisture content, ash content, and bulk density. These tests are essential to ascertain the adsorption capacity, surface functional groups, and structural integrity of the adsorbent.

In addition, the research investigates the adsorption performance of the activated carbon blend in removing Methylene Red dye from synthetic textile wastewater. This will include the analysis of key adsorption parameters such as contact time, initial dye concentration, adsorbent dosage, pH, and temperature. The adsorption data will be further analyzed using isotherm models (e.g., Langmuir and Freundlich), kinetic models (e.g., pseudo-first-order and pseudo-second-order), and **thermodynamic parameters** to understand the mechanism and efficiency of the dye removal process.

This study is **limited to batch adsorption experiments** and does not extend to continuous flow systems or pilot-scale industrial applications. Additionally, the focus is restricted to the treatment of wastewater containing **Methylene Red** as a model anionic dye; other pollutants or multiple dye systems are beyond the scope of this research. The activated carbon blend is produced using **chemical activation only**, with no investigation into physical activation or alternative precursors.

1.6 RELEVANCE OF STUDY

The significance of this study stems from its potential to address a critical environmental challenge textile dye pollution in wastewater by offering a sustainable and cost-effective solution. The textile industry is a major contributor to water pollution, as it generates large volumes of dye-laden effluents that, if left untreated or improperly managed, pose severe environmental and health risks. Conventional wastewater treatment methods are often costly and energy-intensive, making them less feasible for widespread application, especially in developing countries. Other significant of the study includes:

1. The textile industry significantly contributes to water pollution, generating large volumes of dye-laden effluents that pose environmental and health risks if untreated.
2. Conventional wastewater treatment methods are often expensive and energy-intensive, making them less viable for widespread adoption, particularly in developing countries.
3. This research explores the use of palm kernel shell-based activated carbon as an innovative adsorbent for textile dye removal from wastewater.

4. Palm kernel shells, an abundant agricultural waste material, provide a promising raw material for activated carbon production due to their high carbon content and availability.
5. Utilizing palm kernel shells for wastewater treatment aligns with circular economy principles, converting agricultural waste into a valuable resource.
6. The approach reduces both textile effluent pollution and agricultural waste disposal issues, contributing to environmental conservation and sustainability.
7. Economically, palm kernel shell-based activated carbon offers a cost-effective alternative to commercial activated carbon, which is often expensive and derived from non-renewable sources.
8. The high cost of conventional adsorbents limits their accessibility in low-income regions with inadequate wastewater treatment infrastructure.
9. Developing a low-cost and efficient adsorbent provides an economically viable solution for textile industries and wastewater treatment facilities, reducing operational costs while ensuring regulatory compliance.
10. The study has broader implications for environmental policy and regulatory frameworks, contributing to improved wastewater management guidelines.
11. Policymakers can leverage the findings to implement stricter wastewater discharge standards and encourage industries to adopt greener technologies.
12. The research serves as a foundation for further studies on the use of palm kernel shell-based activated carbon in other environmental remediation applications.
13. The material's effectiveness in removing textile dyes suggests potential use in treating other industrial effluents, heavy metals, and organic pollutants.

14. Future research can optimize the activation process, explore modifications to enhance adsorption capacity, and investigate large-scale applications.
15. The study raises awareness about the importance of sustainable wastewater management in the textile industry and the environmental and health risks of dye pollution.

CHAPTER TWO

LITERATURE REVIEW

2.1 POLLUTION AND WASTEWATER TREATMENT

The introduction of undesirable substances or contaminants into the environment, harming people, animals, and ecosystems, is known as pollution (Kümmerer, 2009). Pollution can take many different forms, such as chemical, physical, and biological contaminants. For example, the discharge of pharmaceutical waste into water bodies can result in the presence of solvents, active pharmaceutical ingredients (APIs), and other hazardous chemicals. By contaminating water, these contaminants can damage aquatic life, change ecosystems, and even have an impact on human health. Both manmade and natural compounds can contribute to pollution. Runoff can introduce natural pollutants into water systems, including nutrients from soil erosion and silt. Human activity is the main source of synthetic pollutants, such as industrial chemicals, pesticides, and pharmaceuticals. There are two types of pollution: point source pollution and non-point source pollution. Point source contamination occurs when contaminants are dumped directly into a water body from a specific source, such as a pharmaceutical factory or wastewater treatment plant (Fent et al., 2006). When pollutants enter a body of water from diffuse sources such air deposition or agricultural runoff, this is known as non-point source pollution.

2.2 TYPES OF POLLUTION

They are various forms in which pollution can exist, basically there are three (3) forms

2.2.1Air Pollution

Air pollution is the presence of substances in the air that can cause harm to humans, animals, and the environment. These substances, known as pollutants, can be natural or man-made and can come from various sources. The presence of substances in the air, such as gases, particles, or biological molecules, that can cause harm to humans, animals, and the environment, exceeding the natural concentration or composition of the atmosphere.

Common air pollutants include:

(i)Particulate Matter (PM)

(ii) Nitrogen Oxides (NOx)

(iii) Sulfur Dioxide (SO2)

(iv)Carbon Monoxide (CO)

(v)Volatile Organic Compounds (VOCs)

(vi)Ozone

(vii). Greenhouse Gases (GHGs), such as Carbon Dioxide and Methane

These pollutants can come from various sources, including Industrial activities, Vehicular emissions, fossil fuel combustion, agricultural activities, natural sources, such as wildfires and volcanic eruptions

2.2.2 Water Pollution

When dangerous pollutants that might harm people, animals, and the environment contaminate water bodies like rivers, lakes, seas, and groundwater, it is known as water pollution. There are many different sources of pollutants, such as:

i. Waste products from industry
ii. Runoff from agriculture
iii. Sewage from home
iv. Spills of oil
v. Waste from chemicals

Negative health consequences, including cancer, kidney damage, and reproductive problems, can result from water contamination.

The demise of aquatic life and ecological disturbance

Aesthetic effects include offensive pollution and foul scents; economic effects include the loss of fisheries and tourism income.

Heavy metals like lead and mercury, pesticides and herbicides, and industrial chemicals like PCBs and dioxins are examples of common water contaminants.

Plate 4.1: Effect on industrial chemical waste on water (***World Health Organization (WHO). (2017).*** Chemical hazards in drinking-water: Chemical aspects. *WHO Press, Geneva.*

2.2.3 Soil Pollution

Soil pollution refers to the contamination of soil with harmful substances that can affect human health, plants, and the environment. Common soil pollutants include, heavy metals (lead, mercury, arsenic), Pesticides, herbicides, industrial chemicals (PCBs, dioxins),agricultural waste and Domestic waste

Soil pollution can occur through:

(i) Industrial activities (mining, smelting)

(ii)Agricultural practices (overuse of fertilizers, pesticides)

(ii)Improper waste disposal

Effects of soil pollution:

- Reduced fertility and crop yields
- Contamination of groundwater
- Harm to human health through ingestion or skin contact

*Plate 5.1: Effect of oil spill on the land of ogoni , Nigeria(**United Nations Environment Programme (UNEP). (2011).*** Environmental Assessment of Ogoniland. *Nairobi, Kenya: UNEP.*

2.2.4 Thermal Pollution

Thermal pollution refers to the degradation of water quality by the release of heated water or effluents into water bodies, causing harm to aquatic life and ecosystems. Thermal pollution can occur when heated wastewater from pharmaceutical manufacturing processes is discharged into water bodies, such as rivers or lakes, without proper cooling or treatment. This can lead to:

(i)Changes in water temperature, affecting the habitat and survival of aquatic organisms.

(ii)Increased metabolic rates of aquatic organisms, leading to stress and potentially harmful effects.

(iii)Altered species composition and community structure in affected water bodies.

Plate: Effects of nuclear reactor coolant water discharge on water body (**International Atomic Energy Agency (IAEA). (2001)**.

Table 8: ***Types of Hazardous Materials And Their Effects***

POLLUTANT	SOURCE	ECOLOGICAL RISK
Pesticide and insecticide	Use in agriculture and land scaping	Short of breath, inflammation of the lungs and cancer.
Heavy metals	Lead paint,	Brain damage.

	liquid mercury	
Radio-active waste	Nuclear power plant, nuclear fuel cycle	Genetic mutation, cancer and damage of central nervous cell.

2.3 CHEMICAL POLLUTION

2.3.1 Introduction to Waste Water

Wastewater is a complex and hazardous effluent generated during the production, consumption, and disposal of waste products. This wastewater encompasses a broad range of bioactive compounds, including antibiotics, hormones, and psychotropic substances, which can enter water bodies through various pathways such as industrial effluent, domestic sewage, and agricultural runoff. The presence of these contaminants in water bodies has significant environmental and health implications, as they can alter aquatic ecosystems, disrupt nutrient cycles, and potentially harm human health through the consumption of contaminated water. Wastewater is a growing concern worldwide, driven by the increasing demand for pharmaceuticals, inadequate wastewater treatment infrastructure, and the lack of effective regulations.

Table 9: ***Types of Chemical Compound And It's Composition***

CHEMICALS	SOURCE	COMPOSITION	PHYSICAL CHARACTERISTICS
Sulfamethoxazole	Hospital waste water	C10H11N303S (active ingredient)	White or almost white powder, slightly soluble in water
Estradiol	Domestic sewage, pharmaceutical manufacturing	C18H24O2(active ingredient)	White crystalline powder , insoluble in water
Acetaminophen	Industrial sewage	C8H9NO2(active ingredient)	White crystalline powder
Metformin	Domestic sewage	C4H11N5 (active ingredient)	White or almost white crystalline powder

2.3.2 Environment Consequences

The release of effluent into water bodies has far-reaching and devastating environmental consequences. The presence of these contaminants can alter the delicate balance of aquatic ecosystems, posing a significant threat to the health and sustainability of our planet.

2.3.3 Aquatic Toxicity

Pollutants in water bodies can be toxic to aquatic life, including fish, invertebrates, and microorganisms. Exposure to these contaminants can lead to a range of adverse effects. effluent can disrupt the functioning of aquatic ecosystems, leading to Changes in nutrient cycling, and energy transfer which may affect the life of aquatic animals.

Plate 6.1:Effect of pharmaceutical pollution to aquatic life(aus der Beek, T., Weber, F. A., Bergmann, A., Hickmann, S., Ebert, I., Hein, A., & Küster, A. (2016).

Waste water can accumulate in aquatic organisms, potentially harming humans who consume them. Bioaccumulation can lead to:

2.3.4 Hormonal Disruptions

Hormonal disruption to aquatic life refers to the impact of Effluent on the endocrine systems of aquatic organisms, leading to changes in hormone regulation, development, growth, and reproduction. Pharmaceuticals, such as steroids, hormones, and endocrine-disrupting chemicals, can mimic or interfere with the natural hormones in aquatic organisms, causing a range of adverse effects.exposure to hormonal disruptors can occur through direct uptake from water, food chain transfer, or sediment ingestion. Once inside the organism, these chemicals can bind to hormone receptors, altering gene expression, cellular function, and overall physiology.

Hormonal disruption can lead to changes in reproductive biology, including altered sex ratios, reduced fertility, and impaired embryonic development. For example, exposure to estrogenic compounds has been linked to the feminization of male fish, resulting in reduced sperm count and altered reproductive behavior. Furthermore, hormonal disruption can impact growth and development, leading to changes in morphology, physiology, and behavior. For instance, exposure to thyroid-disrupting chemicals has been shown to alter metamorphosis in amphibians, leading to changes in limb development and function.

In addition, hormonal disruption can affect the immune system, leading to increased susceptibility to disease and reduced overall fitness. This can have cascading effects on aquatic ecosystems, leading to changes in community composition, reduced biodiversity, and compromised ecosystem function.

Hormonal disruption can also impact the development and function of the brain and nervous system, leading to changes in behavior, cognition, and sensory function. For example, exposure to certain pharmaceuticals has been linked to changes in fish behavior, including altered migration patterns, feeding behavior, and social interactions.

The effects of hormonal disruption can be long-lasting, even after exposure has ceased, and can be passed on to subsequent generations through epigenetic changes. This highlights the need for continued research and monitoring to understand the impacts of pharmaceuticals on aquatic life and to develop effective strategies for mitigating these effects.

2.4 WASTE FROM TEXTILE INDUSTRY

Textile industries (TIs) are the major sources of the global economy in many countries like China, India, Pakistan, Brazil, Bangladesh and Malaysia, but unfortunately, these are also the major sources of environmental pollution. TIs utilize a large volume of potable water and a wide range of synthetic chemicals at different stages during the textile production process. TIs generate a large quantity of highly colored wastewater containing a variety of hazardous persistent coloring pollutants (PCPs) that goes into the aquatic resources. In major textile countries, the wastewater/effluent is discharged into the rivers, which finally opens into the sea. For example, in India, Kanpur city is a major hub of textile industries that discharges large volumes of wastewater into drains and canals, which opens into the Ganga River and finally, emptying into the Way of Bengal. Thousands of synthetic dyes are used in TIs during the dyeing process. Globally, ~7 × 107 tons of different dyes is produced annually and more than 10,000 tons of synthetic dyes are used in TIs. Besides dye molecules, textile industry wastewater (TIWW) also contains a high load of salts, alkalis, binders, dispersants, volatile organic compounds (VOCs), surfactants, chlorobenzenes, reducing agents, dioxin, phthalates, phenols, pentachlorophenol, detergents and heavy metals. This TIWW, if discharged directly into the water bodies, causes serious environmental threats and toxic effects in living beings. Different natural fibers such as jute, cotton, silk, wool and a variety of synthetic fibres like polyamide, polyester, viscose, nylon and acrylic are used by TIs. A large number of highly toxic chemicals such as sizing, brightening, anti-creasing, sequestering, stabilizers, softening and finishing agents are used in TIs at different stages. In addition, many synthetic dyes such as azo, vat, direct, reactive, sulphide, acidic and basic dyes are extensively used in TIs. These dyes are not totally attach to the target fibers during textile dying process and get discharged into the aquatic resources like river, pond, streams and lakes along with the wastewater. Dyes are hazardous chemicals for environment and human health. An overview of different stages of wastewater generation in TIs, TIWW has a very complex matrix and contains a variety of aromatic compounds, color content and toxic metals like arsenic (As), lead (Pb), chromium (Cr), antimony (Sb), cadmium (Cd) and mercury (Hg). Heavy metals are used in the production of color pigment of textile dyes. These pollutants are transported to a long distance along with the wastewater, persist in environment (water/soil) for long period of time and pose sever health hazards in living organisms as well as decrease soil fertility and photosynthetic activity of aquatic plants leading to the development of anoxic conditions for aquatic fauna and flora both. Thus, there is an urgent need to develop cost-effective and eco-friendly treatment approaches for the adequate treatment of TIWW before its final disposal into the environment. Therefore, this review paper provides a detailed knowledge on different textile processing steps, wastewater generation, its nature and chemical composition, environmental impacts and health hazards along with the various existing and advanced treatment technologies for the better management of TIWW for the environmental sustainability. It also presents various analytical techniques used to detect and characterize the TIWW pollutants and their metabolites produced during the wastewater treatment processes, key issues and challenges. (Kishor et al., 2021)

2.5 PROCEDURES USED IN THE PRODUCTION OF TEXTILES

2.5.1 Production of Fiber

The initial step in making textiles is the creation of fiber, which is where raw materials are acquired. While synthetic fibers like polyester and nylon are made using petrochemicals, natural fibers like cotton, wool, and flax are obtained from plants or animals. Wool is sheared from sheep, flax is turned into linen fibers, and cotton is gathered from cotton plants. Chemicals are polymerized and then extruded into fibers to create synthetic fibers.

2.5.2 Turning around

The process of turning fibers into yarn is called spinning. After being separated and aligned by carding, the fibers may be combed to get rid of contaminants and further align them. The fibers are then stretched and twisted to produce yarn strands, which might differ in strength and thickness based on the fabric that is intended.

2.5.3 Weaving/Knitting (Fabric Formation)

Weaving or knitting yarn into fabric is known as fabric formation. Weaving involves interlacing two sets of threads (warp and weft) on a loom to produce various fabric patterns, including twill and plain weave. In knitting, yarn is looped together to make fabric using knitting machines, and the fabric can either be weft-knitted or warp-knitted depending on the technique.

2.5.4 Dyeing

The process of dying, which can take place at several stages, is what gives the fabric its color. While yarn dyeing is done after spinning but before weaving or knitting, fiber dyeing is done before the fibers are spun into yarn. Following the weaving or knitting of the fabric, the dying process is carried out. To get the desired color and effect, various dyeing methods are employed, including batch dyeing, continuous dyeing, and piece dyeing.

2.5.5 Printing

Printing is used to add patterns and designs to the fabric. This is typically done using techniques like screen printing, where ink is pushed through a screen onto the fabric, or digital printing, where designs are printed directly onto the fabric using inkjet technology. Block printing, a traditional method, uses wooden blocks to stamp designs onto fabric.

2.5.6 Finishing

Finishing processes are applied to enhance the fabric's appearance, texture, and functionality. Mechanical finishing processes such as calendaring smooth and shine the fabric by passing it through heated rollers, while mercerizing strengthens the fabric and adds a lustrous finish. Chemical finishes are applied to improve properties like water resistance, wrinkle resistance, or flame retardancy, and can also make the fabric softer.

2.5.7 Types of waste from textile industries

1. Fabric scraps: Cutting room waste, selvage waste, and fabric remnants.

2. Yarn and thread waste: Yarn and thread leftovers from weaving, knitting, and sewing.

3. Dyeing and finishing waste: Chemical waste, dye residue, and wastewater from dyeing, printing, and finishing processes.

4. Packaging waste: Cardboard, plastic, and paper waste from packaging materials.

5. Equipment waste: Old machinery, equipment, and tools.

2.6 THE EFFECT OF TEXTILE WASTEWATER ON THE ENVIRONMENT

Textile wastewater, or textile effluent, is a major environmental concern due to the wide array of harmful pollutants it contains, which are often released into water bodies without proper treatment, severely impacting aquatic ecosystems, soil quality, and human health (Awasthi et al., 2020). The textile industry, one of the most water-intensive industries, uses large quantities of water in processes such as dyeing, washing, finishing, and printing, resulting in wastewater that contains toxic chemicals, heavy metals, dyes, and other pollutants (Sulaiman et al., 2023). Among the most hazardous components found in textile wastewater are synthetic dyes, which, due to their non-biodegradable nature, can persist in the environment for extended periods, contributing to the contamination of rivers, lakes, and oceans (Kumar et al., 2021). These dyes not only impair the aesthetic quality of water but also pose significant toxicity to aquatic organisms, including fish and plants, as they reduce oxygen levels and disrupt the balance of aquatic ecosystems (Shah et al., 2022). Additionally, heavy metals such as chromium, copper, and cadmium, commonly used in dyeing processes, can leach into water sources, where they accumulate over time and adversely affect both aquatic life and human populations that rely on these water bodies for drinking and irrigation (Awasthi et al., 2020). As noted by Sulaiman et al. (2023), untreated textile effluent increases the biological oxygen demand (BOD) and chemical oxygen demand (COD) of water bodies, leading to oxygen depletion, which further exacerbates the problem by suffocating aquatic organisms and causing the formation of "dead zones" where marine life can no longer survive (Kumar et al., 2021).

The negative environmental impact extends beyond aquatic ecosystems to soil contamination, particularly when textile wastewater is used in agricultural irrigation or improperly disposed of

(Shah et al., 2022). Research by Awasthi et al. (2020) highlights the accumulation of heavy metals and synthetic dyes in soils irrigated with untreated textile effluents, which can reduce soil fertility, inhibit plant growth, and disrupt the health of soil microorganisms. Over time, these pollutants can leach into groundwater, posing long-term risks to both the environment and human health, especially in regions dependent on agriculture (Sulaiman et al., 2023). The presence of toxic substances in the soil can also impact the quality of crops grown in contaminated land, affecting food security and agricultural productivity. Furthermore, the human health risks associated with textile wastewater are a serious concern, particularly when it comes into contact with drinking water sources or when people are exposed to hazardous chemicals through skin contact or consumption of contaminated food (Shah et al., 2022). Toxic substances such as carcinogenic dyes, formaldehyde, and heavy metals in textile effluents can cause a wide range of health issues, from skin irritations to more severe long-term diseases like cancer and neurological disorders (Kumar et al., 2021). Moreover, endocrine-disrupting chemicals found in textile wastewater can affect reproductive health, further emphasizing the urgency of implementing proper treatment methods in the textile industry (Sulaiman et al., 2023).

In light of these concerns, recent studies have underscored the necessity of improving wastewater treatment technologies to minimize the release of harmful pollutants into the environment. For instance, Kumar et al. (2021) discuss various treatment methods such as advanced oxidation processes, membrane filtration, and adsorption techniques that can effectively remove harmful substances from textile effluents, thus reducing their environmental impact. Despite the development of these technologies, many textile industries continue to discharge untreated or inadequately treated wastewater, exacerbating the pollution of water bodies and the surrounding environment (Shah et al., 2022). In conclusion, the environmental impact of textile wastewater is multifaceted, affecting water quality, soil health, biodiversity, and human well-being, and therefore, it is essential for both industry stakeholders and regulatory bodies to prioritize the adoption of cleaner production techniques and efficient wastewater treatment solutions to mitigate these detrimental effects (Sulaiman et al., 2023; Awasthi et al., 2020).

2.7 CATEGORIES OF TEXTILE DYES

Textile dyes are chemicals used to impart color to fabrics, and they are classified based on various factors such as their chemical structure, application method, and the type of fiber they are suited for. These classifications are important for understanding the properties and best uses of different types of dyes in the textile industry. The primary classifications of textile dyes include **natural dyes**, **synthetic dyes**, and **biological dyes**, with additional subcategories based on their affinity to fibers and methods of application (Shah et al., 2022). Each classification has unique characteristics that determine its environmental impact, cost, and application techniques.

2.7.1 Natural Dyes

Natural dyes are derived from plant, animal, or mineral sources, and they have been used in textile dyeing for centuries. Plant-based dyes are extracted from roots, leaves, flowers, and bark, while animal-based dyes are primarily obtained from insects or mollusks. These dyes are considered eco-friendly compared to synthetic dyes due to their biodegradable nature (Kumar et al., 2021). However, their application is limited by factors such as availability, cost, and the limited range of colors they can produce. Examples include indigo (from the indigo plant), madder (from the madder plant), and cochineal (derived from insects). While natural dyes are generally considered safer for the environment, they may not be as durable as synthetic dyes, and the dyeing process can require large quantities of water (Shah et al., 2022).

2.7.2 Synthetic Dyes

Synthetic dyes are chemically engineered and are widely used in the textile industry due to their cost-effectiveness, ability to produce a wide range of vibrant colors, and ease of use. These dyes are made from petrochemicals and include several subtypes based on their chemical structure and application methods. Some of the most common types of synthetic dyes are:

- **Azo Dyes**: These are the most commonly used synthetic dyes, characterized by the presence of an azo group (–N=N–) in their molecular structure. Azo dyes are often used for dyeing cotton and wool, as they are versatile and can produce a wide range of colors (Kumar et al., 2021). However, some azo dyes are toxic and can release carcinogenic substances when improperly dispose of, leading to environmental pollution.
- **Anthraquinone Dyes**: These dyes are derived from anthraquinone, a compound that imparts strong, vibrant colors. They are often used for dyeing synthetic fibers like polyester (Sulaiman et al., 2023).
- **Reactive Dyes**: Reactive dyes form a covalent bond with the fiber, making them more durable and resistant to washing and fading. These dyes are commonly used for cotton and cellulose fibers and are known for their vibrant colors and colorfastness (Shah et al., 2022).
- **Disperse Dyes**: These are used primarily for dyeing synthetic fibers like polyester. Disperse dyes are applied at high temperatures and are known for their ability to produce

deep and long-lasting colors on hydrophobic fibers like nylon and acetate (Sulaiman et al., 2023).

While synthetic dyes offer many advantages, they are often linked to environmental pollution due to their toxicity, persistence, and non-biodegradable nature. They can contaminate water sources and harm aquatic life if released untreated into the environment (Kumar et al., 2021).

2.7.3 Fiber Reactive Dyes

Fiber-reactive dyes are a subset of synthetic dyes that chemically bond with fibers, particularly cellulose fibers such as cotton. These dyes form covalent bonds with the fiber molecules, making the color more stable and resistant to washing and fading compared to conventional synthetic dyes (Sulaiman et al., 2023). Fiber-reactive dyes are often considered a more eco-friendly option within the synthetic dye category due to their strong bond with the fiber, which reduces the release of dye during washing and minimizes environmental pollution. They are also known for their vibrant colors and versatility.

2.7.4 Vat Dyes

Vat dyes are another category of synthetic dyes known for their exceptional colorfastness. These dyes are insoluble in water in their original state, so they must be reduced to a water-soluble form before being applied to the fabric. Once the dye has been applied and oxidized, it forms a stable, insoluble color that resists fading and washing (Shah et al., 2022). Vat dyes are commonly used for dyeing cotton and are favored for their durability, though they require complex and expensive processes for dyeing.

2.7.5 Sulfur Dyes

Sulfur dyes are another type of synthetic dye used primarily for dyeing cotton and other cellulose fibers. These dyes are inexpensive and provide excellent colorfastness to washing and light. However, they can be toxic and produce unpleasant odors during the dyeing process, which can pose health and environmental risks if not properly managed (Sulaiman et al., 2023).

2.7.6 Biological Dyes

Biological dyes are an emerging category of environmentally friendly dyes, derived from microorganisms like bacteria and fungi. These dyes are considered a sustainable alternative to synthetic dyes because they are biodegradable and have lower toxicity (Shah et al., 2022). While research into biological dyes is still in its early stages, they show promise for reducing the environmental footprint of textile dyeing processes.

2.8 WAYS OF REMEDIATION OF WASTE WATER

2.8.1 Activated Charcoal

2.8.2 What Is Activated Charcoal?

Activated charcoal, also known as activated carbon, is a form of carbon that has been treated to increase its surface area and adsorption capacity. It is made from natural materials such as coconut shells, bamboo, or wood, which are heated to high temperatures to create a highly porous structure.

2.8.3 Activated Charcoal from Palm Kernel

Activated charcoal from palm kernel shell is a distinctly superior and eco-friendly material that has garnered significant attention in recent years due to its exceptional properties and characteristics, which are largely influenced by the palm kernel shell's natural composition and structure, resulting in a highly effective and efficient adsorbent with a wide range of applications, including water treatment, air purification, medical treatments, and industrial processes, where its high iodine number, uniform pore size distribution, and increased surface area enable it to effectively remove impurities and pollutants, while its high hardness and density provide enhanced resistance to abrasion and breakdown, making it an ideal material for applications where mechanical stress is a concern, furthermore, the palm kernel shell-based activated charcoal boasts a lower ash content compared to regular activated charcoal, resulting in fewer impurities and a more consistent product quality, and from an environmental perspective, the production process of activated charcoal from palm kernel shell is highly sustainable, as it utilizes a waste biomass material that would otherwise be discarded, thereby reducing waste generation and promoting environmental stewardship, ultimately, the unique combination of properties, characteristics, and environmental benefits of activated charcoal from palm kernel shell makes it a superior product for various applications, offering improved performance, durability, and sustainability, and as such, it has the potential to play a significant role in addressing various environmental and health challenges, and its further development and utilization are likely to have a positive impact on both the environment and human well-being (Adegoke & Bello, 2020).

Plate: Palm kernel (Activated Charcoal) Tan, I. A. W., Ahmad, A. L., & Hameed, B. H. (2008).

2.8.4 Properties

- High surface area (up to 3000 m²/g)
- High adsorption capacity
- Neutral pH
- Non-toxic and non-reactive
- Highly porous structure

2.8.5 Uses

- Water filtration (removes impurities, chlorine, and pharmaceuticals)
- Air purification (removes odors, gases, and particulate matter)
- Medical applications (treats poisoning, diarrhea, and flatulence)
- Skincare and cosmetics (detoxifies and purifies skin)

- Food and beverage industry (removes impurities and improves taste)

2.8.6 How Does It Work?

Activated charcoal works through a process called adsorption (not absorption). It attracts and holds onto impurities, chemicals, and gases, allowing clean water or air to pass through. Activated carbon reduces free chlorine to chloride and carbon dioxide. It also breaks down chloramines by a relatively slow catalytic reaction to produce ammonia, nitrogen and chloride. Organic compounds are adsorbed in the pores of the carbon matrix. The large surface area of the activated carbon enables significant quantities of organic material to adsorb through ionic, polar and Van der Waals forces.

The very large surface area of activated carbons provides ideal growing areas for bacteria. Adding a bactericide, such as silver, has been used to minimise this effect but the carbon cartridges need to be changed regularly to keep bacterial build-up and shedding under control.

2.9 ADSORPTION

Adsorption is a surface phenomenon where molecules of a substance (adsorbate) accumulate on the surface of another substance (adsorbent). This process creates a thin layer of adsorbed molecules on the surface. Adsorption is different from absorption, where molecules penetrate the bulk of the material.

2.9.1 Characteristics of Adsorption

(i).Surface phenomenon: Adsorption occurs only on the surface of the adsorbent.

(ii).Reversible: Adsorption is often reversible, meaning the adsorbed molecules can desorb back into the bulk phase.

(iii). Specificity: Adsorption is specific to the adsorbent and adsorbate molecules.

(iv). Temperature dependence: Adsorption is temperature-dependent, with higher temperatures often leading to decreased adsorption.

2.9.2 Types of Adsorption

There are two main types of adsorption:

1. Physisorption (Physical Adsorption)

Physisorption occurs due to weak intermolecular forces (van der Waals forces) between the adsorbate and adsorbent. This type of adsorption is:

(i). Reversible: Physisorption is reversible, and the adsorbed molecules can desorb easily.

(ii).Non-specific: Physisorption is non-specific, meaning any molecule can adsorb onto the surface.

(iii).Low energy: Physisorption requires low energy, typically in the range of 1-10 kJ/mol.

2. Chemisorption (Chemical Adsorption)

Chemisorption involves strong chemical bonds between the adsorbate and adsorbent. This type of adsorption is:

(i).Irreversible: Chemisorption is often irreversible, meaning the adsorbed molecules cannot desorb easily.

(ii).Specific: Chemisorption is specific, meaning only specific molecules can adsorb onto the surface.

(iii).High energy: Chemisorption requires high energy, typically in the range of 50-200 kJ/mol.

2.10 ADSORPTION ISOTHERM

An adsorption isotherm is a graphical representation of the amount of adsorbate adsorbed onto an adsorbent as a function of pressure or concentration at a constant temperature.

2.10.1 Types of Adsorption Isotherms

1. Langmuir Isotherm: This model assumes a monolayer adsorption, where adsorbate molecules occupy specific sites on the adsorbent surface.

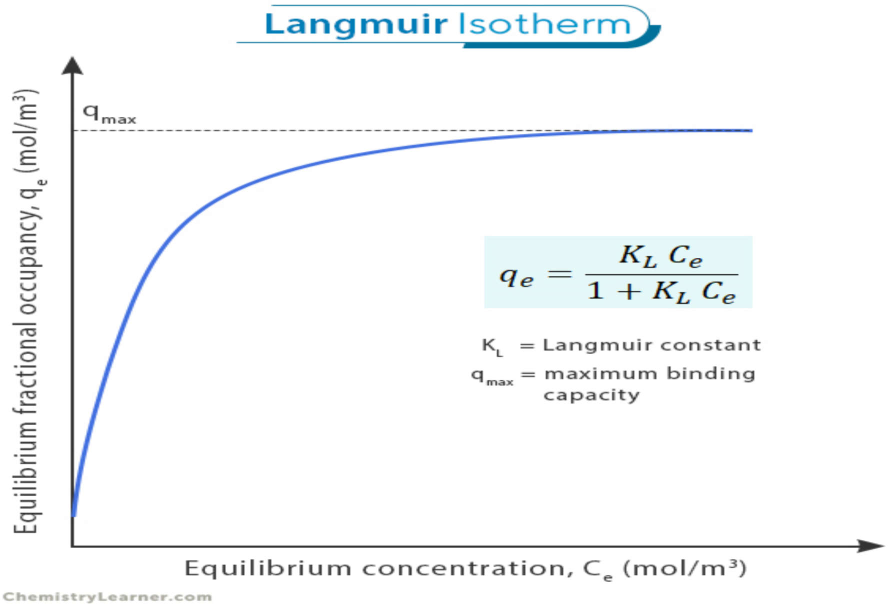


Figure 3 : *Langmuir isotherm*

2. Freundlich Isotherm: This model assumes a multilayer adsorption, where adsorbate molecules can occupy multiple layers on the adsorbent surface.

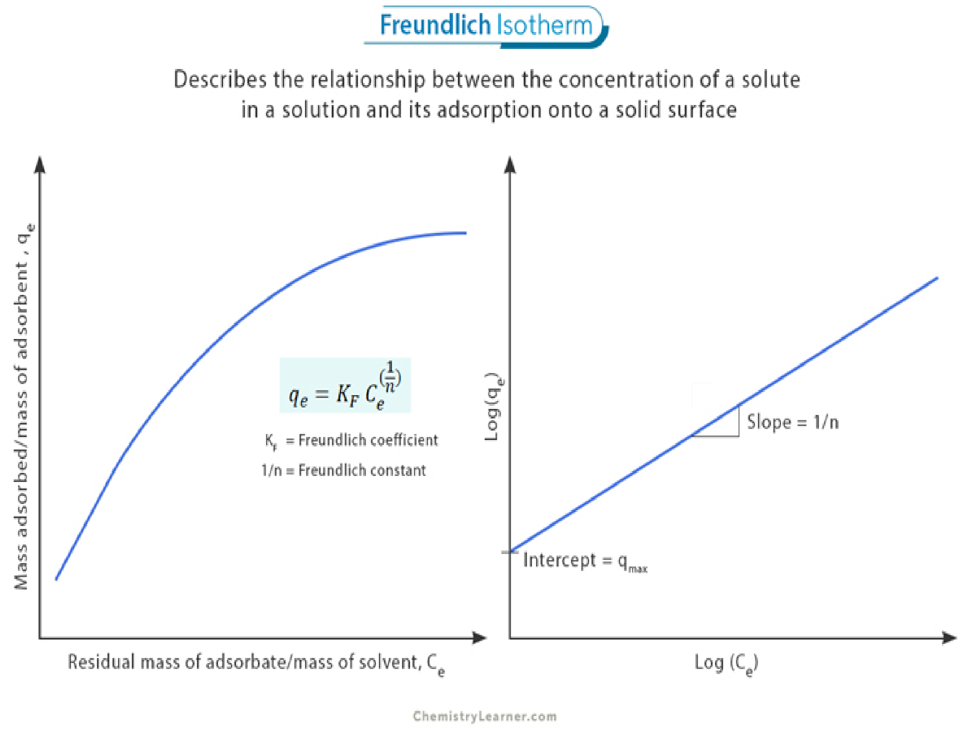


Figure 4: *Freundlich isotherm*

3. BET (Brunauer-Emmett-Teller) Isotherm: This model extends the Langmuir isotherm to multilayer adsorption and is commonly used to describe adsorption on porous materials.

Importance of Adsorption Isotherms

1. Predicting adsorption behavior: Adsorption isotherms help predict the adsorption behavior of a system under different conditions.

2. Designing adsorption systems: Adsorption isotherms are essential in designing adsorption systems, such as adsorption columns and reactors.

3. Optimizing adsorption processes: Adsorption isotherms help optimize adsorption processes, such as temperature, pressure, and flow rate.

2.10.2 Factors influencing adsorption

1. **Effect of pH**

Overall, the pH of the textile effluent is the most significant factor of adsorption that influences the chemistry of the effluent, the action of the functional groups on the adsorbents, and competition by coexisting ions in the wastewater. The state of the active sites may be altered with effluent pH. The accessibility of free sites depends on the pH. At a lower pH, when the binding groups are acidic, the overall surface charge of the adsorbent will be positive. Usually, a low pH dye effluent results in an upsurge in the proportion of anionic dye elimination due to the electrostatic interaction among the anionic dyes and the positive superficial charge of the adsorbent. At a higher pH, electrostatic repugnance is found in the negatively charged superficial area and the dye, diminishing the adsorption limit and rate evacuation of anionic dyes. Additionally, the high pH of the dye effluent brings about an expansion in the level of cationic dye expulsion. The positive charge on the solution surface will diminish and the surface of the adsorbent will get negatively charged. The effluent also consists of metal traces, and therefore upon increasing the pH, precipitation occurs as the solubility of the metal complexes decreases. While precipitation is desired for the overall removal of metals from effluent solution (and thus be advantageous for metal-removal applications), it leads to confusion between precipitation and adsorption. Therefore, it is recommended to study adsorption at pH values before precipitation pH. The optimum pH value depends on the types of biomass.(Pattnaik et al., 2018)

2. Effect of initial concentration

This factor plays a very important role in the adsorption process. Scientists have performed several studies to elucidate the effect of initial concentration— the quantity of dye concentration upsurges with the surge in interaction time. Metal ions and dye concentration can also be transported through the solution toward the surface of the biomass due to a powerful force developed viathe initial metal concentration to conquer the mass exchange of dye between the water and the solid phase. An increase in adsorption capacity can be explained by a higher collision between the metal ions and the dye with the biomass [56]. On the contrary, the initial metal and dye concentration increases generally result in a decrease in the removal efficiency. This phenomenon can be credited to the capacity of adsorption sites on the biomass surface.(Pattnaik et al., 2018)

3. Effect of adsorbent dosage

Researchers have reported that with increasing biomass dosage, the percentage removal of dye and metal ions from the effluent also increases. This behavior might be accredited to the existence of a higher amount of obtainable adsorption sites. Conversely, the maximum adsorption capacities of biomass decrease with an increase in the biosorbent dose. This might be accredited to the fact that overlying of adsorption sites takes place, which results in a reduction of the overall superficial area. The efficiency of numerous adsorbent dosages together on anionic and cationic dye elimination is described by several scientists to identify the most economically feasible adsorbent dosage.(Pattnaik et al., 2018)

4. Effect of temperature

Enormous experiments have been optimized by scientists to study the effects of temperature on dye removal. Diffusion rates as well as the solubility of dyes are affected by the change in solution temperature. The temperature has an impact on the adsorption capacity of the biomass based on the availability of surface functional groups. Nevertheless, several studies have concluded that the effect of temperature is only in a certain temperature range and that to a limited extent. The performance of adsorption depends on the nature of the adsorption process, whether it is exothermic or endothermic. A temperature increase enhances the surface activity and kinetic energy of the adsorbate, which generally improves the adsorptive removal of adsorptive dye and metal pollutants from the effluent, but might result in the physical damage of the adsorbent biomass. This tendency can be accredited to either a decrease in the boundary layer thickness surrounding the biomass or an increase in a number of available active sites. Thus, room temperature is commonly used for most experiments of adsorption.(Pattnaik et al., 2018)

2.11 PKS-Based Activated Carbon for Dye Removal in Textile Wastewater

Dyes such as methylene blue (MB), reactive dyes, and acid dyes are prevalent in textile wastewater and are known for their toxicity, persistence, and environmental hazards. PKS-based activated carbon (PKSAC) has demonstrated exceptional efficiency in adsorbing such dyes due to its high surface area and porosity. Voon and Ghazi (2019) showed that PKSAC, prepared by chemical activation with KOH, exhibited an adsorption capacity exceeding 90% removal efficiency for MB under optimal pH and dye concentration conditions (Xiang & Ghazi, 2019).

Moreover, García et al. (2018) explored PKSAC prepared using $ZnCl_2$, reporting a maximum adsorption capacity of 225.3 mg/g for MB due to its micro porosity and surface functional groups. This study highlighted that higher activation temperatures and proper chemical ratios improve adsorption efficiency (García et al., 2018). PKSAC's high adsorption capacity makes it a competitive alternative to commercial activated carbons, especially for synthetic dyes like reactive blue, acid blue, and Procion dyes, as shown in studies comparing bio-based activated carbons(Paredes-Laverde et al., 2021).

2.11.1 Benefits of Activated Charcoal From Palm Kernel Shell

- Effective in removing pharmaceuticals, pesticides, and heavy metals from water
- Improves taste and odor of water
- Reduces chlorine and fluoride levels
- Supports digestive health
- Can help reduce cholesterol levels

Types Of Activated Carbon

- Powdered activated charcoal
- Granular activated
- Pelletized activated charcoal
- Impregnated activated charcoal (with additional materials like silver or copper

2.11.2 Granular Activated Carbon

Granular Activated Carbon (GAC) is a highly porous material derived from carbon-rich sources such as coconut shells, coal, or wood, which undergoes an activation process involving heat and steam to enhance its surface area and porosity. This process creates a network of tiny pores that enable GAC to adsorb a wide range of substances, making it exceptionally effective at trapping impurities and contaminants. The material's high surface area, often exceeding 1000 m^2/g, allows it to capture a significant amount of pollutants, including organic compounds, heavy metals, chlorine, and volatile organic compounds (VOCs). GAC is typically used in the form of small granules, providing ease of handling and more accessible surface contact for adsorption compared to powdered forms of activated carbon.

One of the primary uses of GAC is in water treatment systems, where it plays a vital role in purifying drinking water by removing harmful chemicals, sediments, and bacteria, ensuring that the water is safe for consumption. It is also extensively used in air purification systems to filter out toxic gases, odors, and other airborne pollutants in both residential and industrial settings. In addition to its use in environmental applications, GAC is employed in industrial processes such as wastewater treatment, solvent recovery, and the purification of chemicals, ensuring that effluent emissions and waste products meet environmental standards. Furthermore, GAC is widely used in the food and beverage industry to remove unwanted flavors, impurities, and colors from liquids like juices, sugar, and wine.

In the medical field, GAC is used in emergency treatments for certain types of poisoning, as it can adsorb toxins in the stomach, limiting their absorption into the bloodstream. Additionally, GAC is a key component in gas masks and respirators, where it adsorbs harmful gases and chemicals from the air, providing protection in hazardous environments.

After prolonged use, GAC's adsorptive capacity eventually becomes exhausted, necessitating regeneration. This is typically achieved by heating the carbon to high temperatures to release the adsorbed substances, restoring its effectiveness for further use. Due to its efficiency, versatility, and reusability after regeneration, GAC remains a cost-effective and sustainable solution for various filtration and purification needs across numerous industries.

2.11.3 Powdered Activated Carbon

This is a highly effective adsorbent material derived from carbon-rich sources such as coal, coconut shells, or wood, and activated through a high-temperature process to create a network of pores, providing a large surface area for adsorbing a wide range of contaminants, including organic compounds, heavy metals, and gases (Bansal et al., 2021; Wei et al., 2020). It is commonly used in applications like water treatment to remove impurities such as chlorine and volatile organic compounds (VOCs), in air purification for filtering odors and toxic gases, and in the food industry for clarifying beverages and removing undesired flavors (Rao et al., 2021). Due to its fine particle size, PAC provides rapid adsorption and is particularly effective for removing smaller contaminants, although it is typically considered a disposable material as regenerating PAC is more difficult compared to Granular Activated Carbon (GAC) (Nguyen et al., 2020).

2.11.4 Pelletized Activated Carbon

This a highly effective filtration material that is created by processing activated carbon into small, uniform cylindrical pellets, typically ranging from 1 to 5 millimeters in diameter. This form of activated carbon is derived from carbon-rich sources such as coconut shells, coal, or wood, which are subjected to a high-temperature activation process to develop a highly porous structure. The activation process increases the surface area of the carbon, allowing it to adsorb a wide range of contaminants from gases and liquids, while the pellet form ensures ease of handling and maintains a uniform size, which is particularly useful in continuous flow systems

(Saeed et al., 2020). The uniform shape of the pellets also helps in maintaining consistent flow rates through filtration systems, making it ideal for large-scale industrial applications where high flow-through efficiency is required (Xia et al., 2021).

Pelletized Activated Carbon is primarily used in water treatment, where it plays a crucial role in removing organic compounds, chlorine, volatile organic compounds (VOCs), and other pollutants from drinking water and wastewater (Kong et al., 2021). The high surface area and porosity of the pellets allow them to efficiently capture contaminants from liquids while providing a high degree of stability in systems with varying flow rates. In air filtration systems, pelletized activated carbon is widely used to remove toxic gases, odors, and airborne pollutants. It is particularly useful in industrial applications such as the purification of chemical gases and in the treatment of exhaust gases from manufacturing processes (Li et al., 2022). Moreover, its use in gas-phase applications, such as solvent recovery and odor control, helps reduce environmental pollution and improve air quality.

One of the significant advantages of pelletized activated carbon is its ability to maintain consistent filtration efficiency over extended periods. The larger size of the pellets minimizes the risk of clogging and helps maintain fluid flow through the system, which can otherwise be a challenge with finer forms of activated carbon (Cheng et al., 2020). Additionally, the pellet shape ensures that handling, transportation, and installation are more manageable compared to powdered or granular activated carbon. The pelletized form also reduces dust generation, which is an important consideration in both industrial and environmental applications (Rashid et al., 2021).

Despite these benefits, there are some limitations to pelletized activated carbon. Due to the larger particle size, the internal micropores of the pellets are less accessible to adsorb smaller contaminants, which may result in a slower adsorption rate compared to powdered or granular forms of activated carbon (Xu et al., 2020). The larger size of the pellets means that they are better suited for applications requiring long contact times with contaminants or for systems that experience consistent flow, rather than quick adsorption in high-concentration environments. Furthermore, pelletized activated carbon can be more expensive to produce than other forms of activated carbon, which could be a limiting factor for certain applications (Jin et al., 2021).

2.11.5 Impregnated Activated Carbon

is a specialized form of activated carbon that has been treated with certain chemicals, metals, or other substances to modify its surface properties, thereby improving its ability to adsorb specific contaminants more effectively than conventional activated carbon. The impregnation process involves the addition of agents such as iodine, potassium, phosphoric acid, silver, copper, or other chemicals, which alter the physicochemical characteristics of the carbon. These modifications enable the carbon to target specific types of pollutants that would otherwise not be efficiently adsorbed by regular activated carbon (Rodríguez et al., 2020). For example, impregnating activated carbon with iodine significantly increases its capacity to adsorb gases and organic compounds, while silver and copper impregnation provides antimicrobial properties, making it highly effective in applications requiring pathogen removal, such as in water treatment systems (Ali et al., 2021).

The main advantage of impregnating activated carbon is that it can be tailored to remove specific contaminants, making it ideal for specialized applications. For instance, activated carbon impregnated with silver is highly effective in eliminating bacterial and viral pathogens from drinking water, offering a solution for water purification systems in both industrial and domestic settings (Feng et al., 2021). Similarly, copper-impregnated activated carbon can be used in air filtration systems to remove toxic gases such as nitrogen oxides (NO_x) from industrial exhaust gases, demonstrating its utility in environmental pollution control (Rodríguez et al., 2020).

The impregnation process not only enhances the adsorption capacity of activated carbon but also extends its applicability to harsh environments where regular activated carbon might degrade or perform inefficiently. For example, impregnated activated carbon is often used in high-temperature or chemically aggressive conditions, such as in the removal of sulfur compounds from industrial gases or in gas masks and respirators designed to filter out harmful toxic substances (Ali et al., 2021). By modifying the surface properties of activated carbon, impregnation improves the carbon's stability and effectiveness, even in challenging conditions.

Recent studies have highlighted the growing importance of impregnated activated carbon in various fields, including air and water treatment, industrial applications, and environmental protection. For example, impregnated activated carbon is widely used in industrial air filtration systems for removing volatile organic compounds (VOCs), odors, and other toxic gases, providing an essential solution for industries that deal with hazardous emissions (Feng et al., 2021). Additionally, it is increasingly being used in the food and beverage industry for clarifying liquids and removing contaminants that would otherwise affect the quality of the final product (Ali et al., 2021). The ability to tailor impregnated activated carbon to specific contaminants makes it highly versatile and valuable in environmental and industrial applications.

However, despite its enhanced performance, there are some limitations to impregnated activated carbon. One concern is the potential for the impregnants to leach out over time, especially when exposed to high concentrations of contaminants or extreme conditions, which can reduce the overall effectiveness of the carbon (Rodríguez et al., 2020). Furthermore, the impregnation process adds to the cost of production, making impregnated activated carbon more expensive than standard activated carbon, which could be a limiting factor for some applications. Additionally, depending on the type of impregnant used, the reusability of impregnated activated carbon can be limited, and it may need to be replaced or regenerated more frequently compared to non-impregnated carbon.

2.11.6 Regeneration

Activated charcoal can be regenerated, a process also known as reactivation. This involves restoring the charcoal's original adsorption capacity by removing impurities and contaminants that have accumulated during use. Reactivation enables the charcoal to perform like new, extending its lifespan and reducing replacement costs. Reactivation methods vary, but they typically involve heat, steam, or chemical treatments to:

- Remove adsorbed substances

- Re-establish pore structure

- Restore surface activity

Effective reactivation maintains adsorption efficiency, reduces waste and environmental impact and Saves resources and costs.

Reactivated charcoal finds applications in:

- Water treatment

- Air purification

- Industrial processes

- Medical and healthcare

Most importantly ,regular reactivation can optimize charcoal performance, ensuring consistent results in various industries.

2.12 METHOD OF REGENERATION OF ACTIVATED CHARCOAL

Regeneration of spent activated carbon or other adsorbents is crucial in wastewater treatment to restore their adsorption capacity, reduce waste, and save costs.methods of Regeneration in are

1. **Thermal Regeneration :**Heat treatment to vaporize impurities and restore adsorption capacity.
2. **Chemical Regeneration:** Chemical treatment to remove contaminants and reactivate adsorbent sites.
3. **Biological Regeneration**: Microbial treatment to break down contaminants and revitalize adsorbents.
4. **Hybrid Regeneration:** Combination of thermal, chemical, and biological methods.
5. **Steam Regeneration** : Steam treatment to remove impurities and restore adsorption capacity.
6. **Solvent Regeneration**: Solvent extraction to remove contaminants and reactivate adsorbents.

2.13 APPLICATION OF ACTIVATED CHARCOAL IN WASTE WATER

Activated charcoal, a highly effective adsorbent, has become increasingly important in recent years due to its exceptional ability to remove contaminants, pollutants, and impurities from wastewater (Ahmed, 2017). The growing need for clean water, combined with tough environmental regulations, has driven the development of innovative and effective wastewater treatment technologies. Activated charcoal, with its unique properties, has emerged as a promising solution for improving wastewater quality, reducing environmental and impact, and ensuring public health safety.

The use of activated charcoal in wastewater treatment has been extensively studied, and its efficacy has been demonstrated in various industrial and municipal settings. According to the United States Environmental Protection Agency (EPA), activated charcoal is recognized as a best available technology (BAT) for removing organic compounds and other pollutants from wastewater (EPA, 2020).

Application of activated charcoal in waste water are:

(i) Food and beverage processing wastewater treatment

(ii) Paper and pulp mill wastewater treatment

(iii) Water reuse and recycling

(iv) Pharmaceutical and chemical wastewater treatment

(v) Municipal wastewater treatment

(vi) Industrial wastewater treatment.

2.14 CHARACTERISTICS OF PALM KERNEL (PKS) AS BIOMASS WASTE

The lignocellular characteristics of Palm Kernel Shell (PKS), commonly referred to as Oil Palm Shell (OPS) or Oil Palm Kernel Shell (OPKS), as biogenic organic waste has been well established in the literature. These characteristics have accustomed to the material the benefit of being used as a raw material for the development of composite materials, among others. For example, PKS may be used in the manufacturing of concrete as reinforcement: fuel generation medium, cement production; cutting tool development and plastic polymer composite development. Palm kernel shell is a waste product obtained after the processing of palm kernel. Palm kernel is enclosed in the shell. The shell is also enclosed in fleshy fiber, which contains palm oil. Thus palm kernel shell is obtained when the fibrous, fleshy fruit has been processed to obtain palm oil. Nevertheless, the shell can be obtained by removing the fleshy fiber with the sole aim of reaching the shell without interest in the palm oil. By cracking the shell to get palm kernel nut, the shell is thus obtained. Various researchers have examined the properties of palm kernel shells to identify the suitability of the biomaterial in multiple applications. These interesting properties include porosity, moisture content, lignin content cellulose, and Nanocellulose fractions, solid density, and carbon content, among others. One particular area which has found the application of PKS currently of intense interest is the plastic composite development studies. As a filler, PKS may modify and enhance particular desired properties and reduce cost since they are waste material and, therefore, cheaply obtained. Being a natural fiber, PKS have relatively low density, is environmentally friendly through quick and total degradation, abundant and therefore readily available. The oil palm fruit edible seed is described as a palm kernel. Palm fruit produces two types of oils that have different characteristics. The first of the oils, palm oil is obtained from the fibrous, fleshy outer part of the fruit: and palm kernel oil obtained from the edible kernel. Palm kernel shell is the outermost part of the seed and therefore acts as a protector to the seed. The shell needs to be cracked before the edible part is released. The oil palm is believed to be originated from West Africa within the tropical rain forest of the continent. The Oil Palm (Elaeis guineensis) belt includes countries that lie within the equatorial belt, and these include Ghana, Nigeria, Togo, Cameroon, Cote d'Ivoire, Liberia, Sierra Leone, Congo, and Angola, through Congo and Angola are in Central Africa. The shell is, therefore, a by-product of the production of palm kernel oil and palm oil from the seed of the palm tree. It is thick and brown/black wood-like in structure. The process of obtaining palm kernel shell, which begins with the oil palm plant cultivation, is well documented. It takes two to three years of new seedlings to mature into productive trees. The fruit bunch is directly attached to the grown tree with the fruits, also attached to spikes and spikelets which are connected to the stem of the bunch. By separating the spikes and spikelets from the bunch, the fruits could then be removed. Between 20 and 30 years, the fruit usually cultivated in small to large scale plantations are harvested. During the process, many waste products are generated. The empty fruit bunch, waste mesocarp fiber, and palm oil mill effluent, which may be applied as insecticide and pesticide, are some of the by-products of the seed production process. Before the seed is obtained, the hard stony shells need to be crushed into various shapes and sizes. The shells may be of the "Dura" or "Tenera" type. Though Tenera has been designed as a hybrid breed to yield higher oil content, it has thinner shells. It is a hybrid of Dura (mother which has shells) and pisifera (father without shells). Dura has thicker shells, while pisifera has no shells. Oil palm fiber, empty fruit bunch (OPEB), and mesocarp fiber and PKS shells have several uses. The fibers may be used for weaving baskets and thermal energy generation. Palm kernel shells may be used for fuel heat generation, carbon activation for purifying water, concrete reinforcement in the building and construction industry, thermal insulation, and as filler in plastic composite manufacturing.(Baffour-Awuah et al., 2021)

2.14.1 Adsorption Mechanisms of Pks-Based Activated Carbon

Activated carbon (AC) derived from palm kernel shells (PKS) has emerged as a promising adsorbent for various environmental applications, particularly in wastewater treatment. The effectiveness of PKS-based activated carbon in removing pollutants is attributed to its unique adsorption mechanisms. This research aims to explore the mechanisms of adsorption involving PKS-based activated carbon, focusing on the physicochemical properties, surface interactions, and kinetic and isotherm models that describe the adsorption process.

2.14.2 Physicochemical Properties of PKS-Based Activated Carbon

The preparation of activated carbon from PKS typically involves carbonization followed by activation, which enhances its surface area and porosity. For instance, studies have shown that PKS can yield activated carbon with a specific surface area ranging from 435 m^2/g to over 1560 m^2/g, depending on the activation method used (e.g., chemical activation with KOH or H_3PO_4) 12. The high surface area is crucial for adsorption as it provides more active sites for pollutant interaction. Mesoporous structures are especially useful for thick textile colors because they have larger surface areas and more active sites. (Pam et al., 2021).

2.14.3 Influence on Adsorption Efficiency for Textile Pollutants:

The hierarchical pore structure and surface chemistry significantly enhance the adsorption of textile dyes, enabling mechanisms like pore filling, hydrogen bonding, and electrostatic interactions. These properties result in high adsorption capacities (e.g., 292 mg/g for specific dyes)(Silva et al., 2018)

2.14.4 Surface Functional Groups

The presence of functional groups such as hydroxyl (-OH), carboxyl (-COOH), and carbonyl (C=O) on the surface of activated carbon plays a significant role in enhancing adsorption capacity. These groups can facilitate both physical and chemical interactions with adsorbates, thereby improving the overall efficiency of the adsorption process. Groups such as carboxyl and sulfonic acids improve chemical adsorption and electrostatic interactions with charged contaminant

2.15 LIMITATIONS OF COMMERCIAL ACTIVATED CARBON

Despite its effectiveness, commercial activated carbon is expensive and less sustainable. This has led to the search for low-cost precursors, especially those derived from agricultural and biomass wastes.

1. **Biomass-Derived Activated Carbon**

   Agro-wastes are particularly attractive for AC production due to their abundance, renewability, low cost, and high lignocellulosic content. Examples include sawdust, rice husk, corncob, coconut shell, and palm kernel shell.

2. **Palm Kernel Shell (PKS) as a Precursor**

   PKS is a byproduct of the palm oil industry with high fixed carbon and low ash content. It has a dense structure and high lignin content, making it suitable for carbonization and activation. Several studies have demonstrated the successful use of PKS-derived AC in removing dyes and heavy metals from wastewater (Tan et al., 2008; Bello & Ahmad, 2012).

3. **Coconut Shell (CS) as a Precursor**

Coconut shell is another abundant agro-waste material with excellent potential for AC production due to its high volatile matter and carbon content. It produces a microporous structure ideal for adsorbing small molecules such as dyes. CS-based ACs have demonstrated high adsorption capacities and regeneration potential.

4. **Blending of Biomass Precursors**

Recent studies indicate that blending different biomass precursors can yield composite activated carbons with enhanced porosity, surface functionality, and adsorption efficiency. The synergistic effect from the differing physical and chemical structures of PKS and CS can result in improved adsorption dynamics and broader pollutant selectivity.

2.15.1 Activation Methods

- **Physical Activation**

This involves carbonization of the raw precursor at high temperatures (400–800°C) under an inert atmosphere followed by activation using steam or CO_2. The method is simple but yields lower surface areas compared to chemical activation.

- **Chemical Activation**

  Chemical agents such as phosphoric acid (H_3PO_4), zinc chloride ($ZnCl_2$), and potassium hydroxide (KOH) are used to dehydrate and open the structure of the carbon precursor. Chemical activation typically occurs at lower temperatures and produces AC with higher surface area and well-developed porosity.

- **Combined Activation Techniques**

Hybrid techniques combining both physical and chemical activation have shown to improve surface functionality and optimize pore development.

2.15.2 Adsorption of Dyes Using Activated Carbon

2.15.3 Adsorption Isotherms

Isotherm models like Langmuir, Freundlich, and Temkin describe the relationship between the amount of dye adsorbed and its equilibrium concentration. Langmuir assumes monolayer adsorption on a homogeneous surface, while Freundlich accounts for heterogeneous surface energies.

2.15.4 Adsorption Kinetics

Pseudo-first-order and pseudo-second-order models are commonly used to evaluate adsorption rates. The pseudo-second-order model often provides better correlation, indicating chemisorption as the rate-limiting step.

2.15.5 Thermodynamics of Adsorption

Parameters such as Gibbs free energy (ΔG), enthalpy (ΔH), and entropy (ΔS) help to ascertain whether the adsorption process is spontaneous, exothermic, or endothermic.

2.16 CASE STUDIES

Numerous studies have highlighted the effectiveness of PKS and CS-derived AC in dye removal. For instance, Hameed et al. (2008) reported adsorption capacities of over 300 mg/g for methylene blue using AC from PKS. Similarly, Foo and Hameed (2010) demonstrated high removal efficiencies using CS-AC. However, limited studies exist on the adsorption of methylene red specifically, especially using blended ACs.

2.17 RESEARCH GAPS

Despite the abundant research on biomass-derived activated carbons, few studies focus specifically on the removal of methylene red, a relatively less studied but environmentally hazardous dye. Additionally, the synergistic potential of blended ACs from PKS and CS remains underexplored. This project aims to fill this knowledge gap by:

- Developing a cost-effective and eco-friendly activated carbon composite from PKS and CS.
- Investigating the efficiency of the composite in removing methylene red from textile wastewater.
- Characterizing the adsorbent using techniques such as FTIR, SEM, and BET surface area analysis.

- Evaluating adsorption performance using isotherm, kinetic, and thermodynamic models.

CHAPTER 3

MATERIALS AND METHOD

3.1 CHEMICAL AND REAGENTS

All chemicals and reagents used in this work were of analytical grade used without further purification. The majority of them were purchased from a laboratory materials vendor in Benin city. Edo state. The details of each reagent, their sources and what they were used for in this work are summarized below in Table 3.1

Table 10: Chemicals/Reagents

S/N	Chemicals/Reagents	Manufacturer	Uses
1	Phosphoric acid (H_3PO_4)	Guangdong Guanghua Sci-Tech C0.,Ltd.	For activating the carbonized palm kernel shell
2	Potassium hydroxide (KOH)	Trust Chemical Laboratories	For adjusting the pH of the wastewater
3	Hydrochloric acid (HCl)	Loba Chemie Pvt. Ltd	For adjusting the pH of the wastewater sample
4	Methyl red dye	Trust Chemical Laboratories	For testing the capability of the adsorbent
5	Deionized Water	Luco chemicals	Used for washing and dilution purposes

3.1.2 Equipment and Apparatus

Table 11: Equipment and Apparatus

S/N	Equipment/Apparatus	Manufacturer	Uses
1	Muffle Furnace	Pec Medical; Model:SX-410	For carbonizing the Palm kernel shell.
2	Weighing balance	M-Metlar Analytical Balance, Model:M311L	For measuring amounts of the adsorbent dosage
3	pH Indicator paper	Nil	For testing the acidic and basic levels in the dye solutions
4	Spectrophotometer(UV-Vis)	Pec Medical USA	For measuring absorbance at 540nm wavelength
5	Orbital shaker	B-Scientific England Model;HY-4A	For agitation of samples
6	Measuring cylinder	Pyrex	For measuring known volume of reagent
7	Volumetric flask	Pyrex	For preparing and measuring chemical solutions
8	Conical flask	Pyrex	For the batch study
9	Glass funnel	Pyrex	For filtering pretreated mixture and treated wastewater
10	Filter paper	Whatman	For separating the filtrate from the adsorbing mixture
11	Beakers	Pyrex	For preparing solutions
12	Universal Container	Pyrex	For collecting and storing treated wastewater sample

3.2 MATERIALS

Table 12: Raw Materials

S/N	Materials	Sources	Uses
1	Textile waste water dye(Methylene red)	Delta textile mile industry, Delta state	Testing and Research (Model Pollutant)
2	Palm kernel and Coconut Shell	NIFOR, Benin City	Activated carbon source

3.3 METHODS

3.3.1 Preparation Of Blend Palm Kernel Shells And Coconut Shell Activated Carbon

1. Raw Material Collection and Pre-treatment

Palm kernel shells and coconut shells were collected from a local palm oil mill at NIFO in Edo State, Nigeria. Both feedstocks were first subjected to an extensive cleaning process in which the shells were washed thoroughly using distilled water to remove any adhering soil particles, oils, or soluble organic impurities. This cleaning stage is critical to ensure that unwanted substances do not interfere with the carbonization and activation processes. After washing, the shells were oven-dried at a temperature of 105 °C for 24 hours to remove moisture and minimize hydrolysis reactions during thermal processing. Once dried, the shells were crushed using a laboratory jaw crusher and ground into smaller particle sizes ranging between 2 and 5 mm using a mechanical grinder. The crushed particles were then sieved to ensure uniformity in particle size distribution, which is essential for consistent carbonization and activation behavior.

2. Carbonization of Biomass

The dried and sieved biomass samples of palm kernel shell and coconut shell were separately subjected to carbonization to convert the lignocellulosic material into char, a carbon-rich intermediate product. The carbonization process was carried out in a muffle furnace under an inert nitrogen atmosphere to prevent oxidation and combustion of the biomass. Each sample was heated at a constant rate of 10 °C per minute until a final temperature of 500 °C was reached. This temperature was maintained for 2 hours to ensure complete thermal decomposition of volatile organic compounds and to maximize char yield. The thermal decomposition reactions involved in this process primarily include dehydration, decarboxylation, and dehydrogenation, which result in the evolution of gases such as CO_2, CO, CH_4, and H_2O vapor. After carbonization, the resulting chars were cooled in the absence of air and stored in airtight containers to prevent oxidation.

3. Activation of Charred Materials

The carbonized samples of PKS and CS were then subjected to chemical activation to enhance their porosity and surface area. Potassium hydroxide (KOH) was selected as the chemical activating agent due to its high reactivity and ability to generate microporous structures. The activation process began by mixing the carbonized material with KOH in a 1:3 weight ratio (carbon:KOH). This mixture was then homogenized using distilled water to form a thick paste, which was subsequently dried at 105 °C for 12 hours to remove excess moisture. The dried impregnated samples were then placed in a tubular furnace and heated under a continuous flow of nitrogen gas to 700 °C at a heating rate of 10 °C per minute. The temperature was held constant for one hour to facilitate the activation reaction. During activation, KOH reacts with the carbon matrix to form metallic potassium, potassium carbonate, and gaseous products such as H_2 and CO, which help to etch pores into the carbon framework. Upon completion of activation, the samples were cooled under nitrogen and washed sequentially with 0.1 M hydrochloric acid and hot distilled water until the filtrate reached a neutral pH. This washing process removes residual KOH, metallic salts, and soluble ash. The cleaned activated carbon was then oven-dried at 105 °C for 24 hours and stored for further processing.

4. Blending of Activated Carbons

Two strategies can be employed to obtain a blended activated carbon from PKS and CS. In this study, post-activation blending was adopted in order to exert better control over the final porosity and surface chemistry. Equal weights (1:1 ratio) of the individually activated PKS and CS carbons were accurately weighed and thoroughly mixed using a mechanical blender to ensure homogeneity. The resulting composite material combines the inherent advantages of both biomass sources—PKS typically yields high carbon content with a dense microporous structure, while CS contributes improved mesoporosity and surface functional groups. The blending was done under dry conditions and the final product was labeled as PKS-CS activated carbon blend.

5. Characterization of Blended Activated Carbon

The physicochemical and adsorption properties of the blended activated carbon were characterized using a variety of standard techniques. The specific surface area and pore volume were determined using the Brunauer–Emmett–Teller (BET) nitrogen adsorption-desorption isotherm method. Pore size distribution was evaluated using the Barrett–Joyner–Halenda (BJH) model. The pH at the point of zero charge (pHpzc) was determined using the pH drift method, which provides information about the surface charge behavior of the adsorbent. Functional groups present on the surface were analyzed using Fourier Transform Infrared Spectroscopy (FTIR), and the ash and moisture contents were determined by gravimetric methods following ASTM standard procedures. Bulk density was measured to assess the packing efficiency of the adsorbent. These characterization steps were essential to confirm the development of porous structure and to evaluate the suitability of the blended activated carbon for application in water treatment, gas adsorption, or catalysis.

6. Storage of Final Product

To preserve the structural and functional integrity of the prepared activated carbon blend, the final product was stored in air-tight polyethylene containers under desiccated conditions to prevent moisture adsorption and exposure to atmospheric gases. Proper storage conditions ensure that the activated carbon retains its adsorption capacity over time and remains chemically stable.

3.3.2. Characterization Of Activated Carbon Sample

The adsorbent underwent characterization prior to the adsorption process. Various properties of the adsorbent were assessed using different techniques:

i. BET Method (JW-DA, 76502057en): This method was employed to determine the specific surface area, pore size distribution, pore volume, total pore volume, and pore volume distribution of the adsorbent.

ii. SEM (JEOL JSM-7600F): Scanning Electron Microscopy was utilized to examine the surface morphology of the adsorbent.

iii. FTIR (Nicolet iS10 FT-IR Spectrometer): Fourier-Transform Infrared Spectroscopy was used to irradiate the sample and identify the active functional groups present on the surface of the adsorbent.

iv. XRD (Rigaku D/Max-IIIC X-ray Diffractometer): X-ray Diffraction analysis was performed to determine the crystallinity of the activated carbon. These characterization techniques provide valuable insights into the physical and chemical properties of the adsorbent, helping to understand its suitability and performance for adsorption purposes.

3.3.3 Batch Adsorption Experiment

Preparation of Dye Solution:

A textile waste solution of 1000 mg/L methylene red dye was procured and diluted as required.

ADSORPTION STUDIES:

The batch adsorption experiments were conducted using separate 250ml conical flasks, with each flask containing 100 ml of waste water. Various amounts of the blend PKS and Coconut shell -AC were added to the waste water samples, ranging from 0.5 to 1.0 g/L. The contact time for each experiment ranged from 30 to 90 minutes. To facilitate mixing of the waste water with the blend PKS- and Coconut shell AC adsorbent, the glass conical flasks were positioned on an orbital shaker machine set at an rpm speed of 150 for the specified contact time. After adsorption, the solutions were filtered, and a 10ml sample was extracted from each suspension for further analysis or assessment, using a UV- Vis spectrophotometer at the maximum wavelength (λ_max = 560 nm).

Determination of Removal Efficiency:

The amount of dye adsorbed at equilibrium (Ω, mg/g) and the dye removal efficiency was calculated using:

$$q_e = \frac{(C_o - C_e)V}{W} \text{ ----------------- } (3.1)$$

$$R = \frac{C_o - C_f}{C_o} \times 100 \text{ -------------------(3.2)}$$

Where q is the amount of metal ion adsorbed by the adsorbent (mg/g); C_o is the initial dye concentration in contact with the adsorbent (mg/L), C_e is the equilibrium dye concentration (mg/L), C_f is the dye concentration after the batch adsorption, V is the volume of aqueous solution put in contact with the adsorbent in liters (L) and m is the adsorbent dosage in grams (g).

3.3.4. Response Surface Optimization of Dye Removal (Design Of Experiment)

In the study, a Response Surface Methodology (RSM) model was developed and employed to optimize the adsorption process for waste water. The optimization process was carried out with the assistance of RSM, utilizing Central Composite Design (CCD provided by the Design Expert program (version 13, Stat-Ease Inc., Minneapolis, USA**).** A Central Composite Design (CCD) **is** a widely used experimental design for Response Surface Methodology (RSM**),** which helps in optimizing processes and understanding interactions between variables. CCD is particularly useful for building second-order (quadratic) models and exploring the effects of multiple independent variables on a response variable. The factors considered were:

i. Adsorbent Dosage (g/L): 0.5– 1.0 g/L
ii. Contact Time (min): 15 – 90 min
iii. Initial Concentration (mg/L): 100 – 500 mg/L

The Central Composite Design generated a total of 30 runs to optimize these four factors. These factors encompassed adsorbent dosages ranging from 0.1 to 10 g/L, a pH range of 5 to 9, initial concentration ranging from 100 to 500 mg/L and contact times spanning from 15 to

90 minutes. The primary objective of these experiments was to optimize the removal of dyes from waste water using the composite adsorbent.

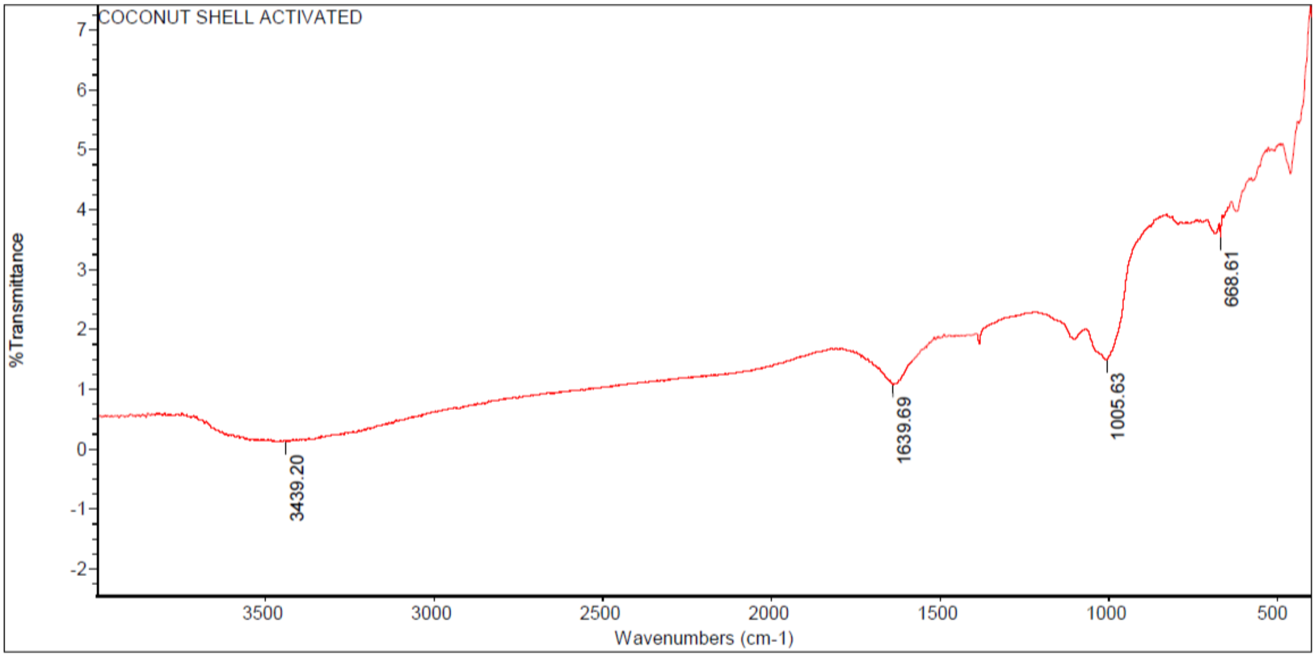
3.3.5. Response Surface Methodology (RSM) Optimization

Table 13: Build information

DESIGN	INFOR
File Version	13.0.1.0
Study Type	Response Surface
Design Type	Box-Behnken
Design Model	Quadratic
Build Time (ms)	19.00
Subtype	Randomized
Runs	29.00
Blocks	No Blocks

Table 14: Design Factors

Name	**Minimum**	**Maximum**	**Coded Low**	**Coded High**	**Mean**
A, CS-AC (g/L)	0.0000	10.00	-1 ↔ 0.00	+1 ↔ 10.00	5.00
B, PKS-AC (g/L)	0.0000	10.00	-1 ↔ 0.00	+1 ↔ 10.00	5.00
C, Time (minute)	30.00	90.00	-1 ↔ 30.00	+1 ↔ 90.00	60.00
D, Dye Concentration (g/L)	100.00	500.00	-1 ↔ 100.00	+1 ↔ 500.00	300.00



Position: 668.61 Intensity: 3.534

Position: 1005.63 Intensity: 1.484

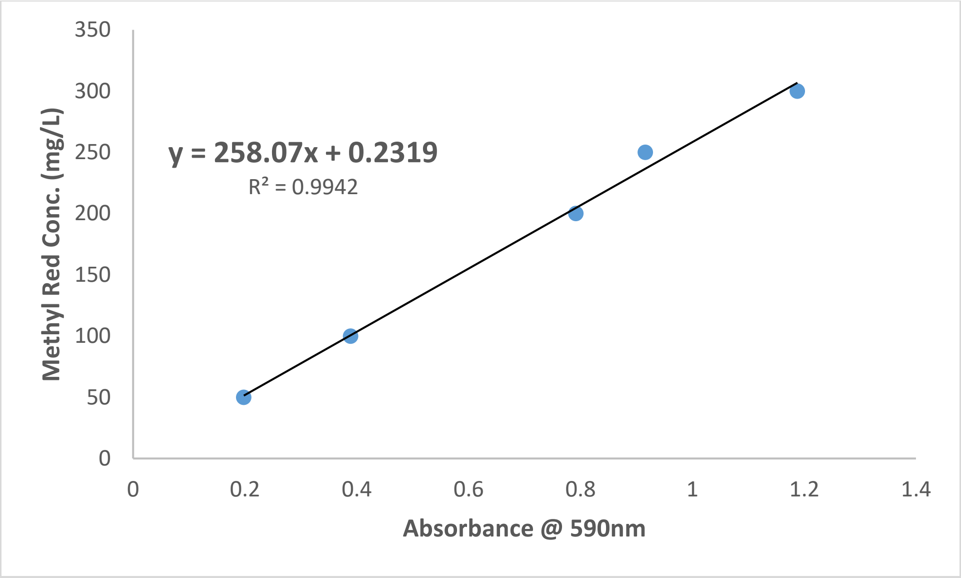
Position: 1639.69 Intensity: 1.078

Position: 3439.20 Intensity: 0.115

Table 15: Experimental runs generated by design expert

Run	**CS-AC (g/L)**	**PKS-AC (g/L)**	**Time (minute)**	**Dye Concentration (g/L)**	**Abs**	**Dye Removal (wt%)**
1	5	10	90	300	0.17	44.1038
2	5	0	90	300	0.172	44.61994
3	5	5	60	300	0.024	6.42558
4	0	10	60	300	0.003	1.00611
5	5	10	30	300	0.193	50.03941
6	5	0	30	300	0.155	40.23275
7	5	5	90	100	0.146	37.91012
8	10	10	60	300	0.069	18.03873
9	10	5	60	100	0.339	87.71763
10	5	5	30	500	0.148	38.42626
11	10	5	60	500	0.098	25.52276
12	0	5	30	300	0.016	4.36102
13	5	5	60	300	0.008	2.29646
14	10	5	90	300	0.009	2.55453
15	10	5	30	300	0.016	4.36102
16	5	5	60	300	0.157	40.74889

17	5	0	60	100	0.101	26.29697
18	0	5	60	100	0.016	4.36102
19	5	5	60	300	0.016	4.36102
20	5	5	60	300	0.091	23.71627
21	0	0	60	300	0.104	27.07118
22	0	5	90	300	0.099	25.78083
23	5	10	60	100	0.141	36.61977
24	5	5	90	500	0.353	91.33061
25	0	5	60	500	0.152	39.45854
26	5	5	30	100	0.175	45.39415
27	5	10	60	500	0.006	1.78032
28	10	0	60	300	0.251	65.00747
29	5	0	60	500	0.17	44.1038



WAVE LENGTH: 560nm

CHAPTER 4

RESULTS AND DISCUSSION

ADSORBENT CHARACTERISATION

Blend Palm kernel and Coconut-shell Activated Carbon Characterization

The activated carbon consisting of blend palm kernel and coconut-shell, was subjected to the Brunauer-EmmettTeller(BET) Scanning Electron Microscopy method. The BET method provided information on pore size distribution which influenced the accessibility of the PKSAC sites for different sized molecules. Also, the SEM provided a high-resolution image of the activated carbon's surface morphorlogy. Also, the SEm micrographs visually confirmed the development of a porous network within the activated carbon.

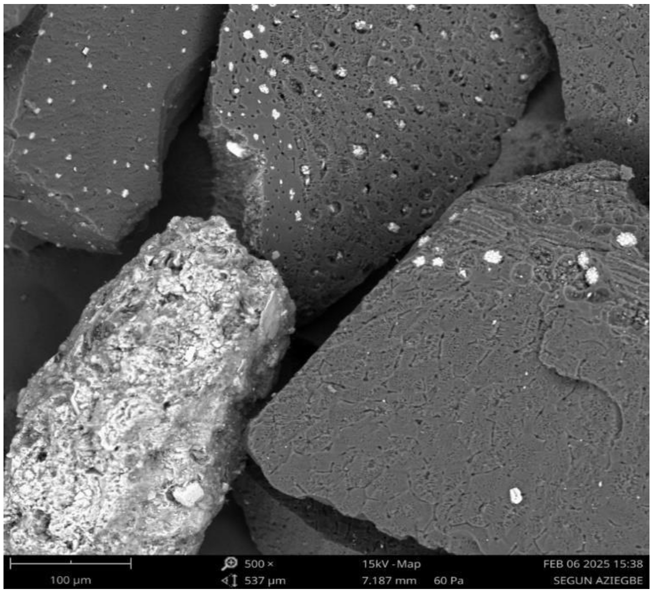
The BET analysis provided insights into the porous structure and adsorption properties of the adsorbent, which is critical for evaluating its efficiency in dye removal as seen in Table 4.1

Table 4. 1:Summary of the BET analysis on the activated carbon(Palm kernel and Coconut-shell)

Method	**Surface Area(m^2/g)**	**Pore volume (cc/g)**	**Pore Diameter (nm)**	**Adsorption Energy (kJ/mol)**
Multi-point BET	275.762	-	-	-
Langmuir	464.380	-	-	-
DR	393.400	0.140	3.866	6.725
BJH	278.755	0.135	2.108	-
DA	-	0.195	2.140e+00	1.832

The **multi-point BET method** was employed to determine the specific surface area of the adsorbent, yielding a value of **275.762 m²/g**. This measurement reflects the extent of the available surface for adsorption, which is a critical factor in assessing the adsorbent's efficiency for methyl red removal from textile wastewater. To complement this, the **Langmuir method** was also applied, which assumes monolayer adsorption of molecules on a homogeneous surface. The surface area obtained from Langmuir analysis was **464.380 m²/g**, closely aligning with the BET result. Furthermore, the high correlation coefficient (**R^2 = 0.988**) for the Langmuir isotherm model suggests a strong fit, supporting the monolayer adsorption assumption for this material. In addition, the **Dubinin–Astakhov (DA) method** was used to probe microporosity, yielding a micropore volume of **0.195 cm³/g** and an average pore diameter of **2.14 nm**. These results indicate that the adsorbent possesses a predominantly microporous structure, well-suited for the uptake of small molecules. The **adsorption energy (Best E)** was estimated at **1.832 kJ/mol**, providing insight into the interaction strength between the adsorbent and the dye molecules. Further analysis using the **Dubinin–Radushkevich (DR) method** revealed a **micropore surface area of 393.400 m²/g**, with an adsorption energy of **6.725 kJ/mol**. This relatively low value indicates that the process is dominated by **physical adsorption** rather than chemical bonding, which is consistent with the typical behavior of activated carbon. The DR method also reported an **average pore width of 3.866 nm**, suggesting the presence of both micropores and small mesopores. To investigate mesoporosity in greater detail, the **Barrett–Joyner–Halenda (BJH) method** was applied using nitrogen adsorption data. The BJH results showed a **cumulative pore volume of 0.135 cm³/g** and an **average mesopore diameter of 2.108 nm**.

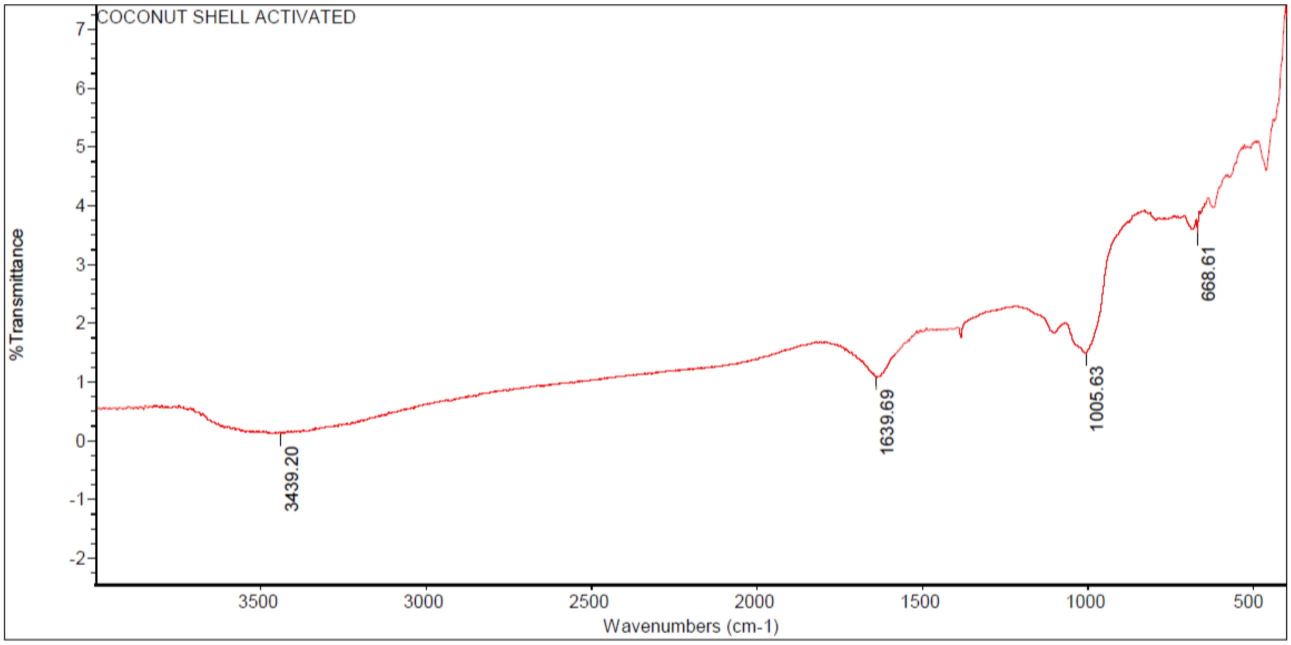
These findings confirm that the adsorbent contains a mixed pore structure, where the coexistence of micropores and mesopores enhances its overall dye adsorption efficiency. The SEM micrographs provide a clear visualization of the adsorbent's surface morphology, revealing a rough texture, well-developed pores, and distinct microporous and mesoporous cavities on the palm kernel shell–based activated carbon. This heterogeneous and porous structure increases the effective surface area and creates more active sites for adsorption. The observed morphology is consistent with the BET surface area value of 275.762 m²/g, confirming the material's suitability for efficient dye (methyl red) uptake from wastewater. Complementary analyses from DA and BJH methods, which reported significant micropore volume and well-distributed pore sizes, further validate the presence of both micropores and mesopores—features essential for accommodating both small molecules and larger dye aggregates. The SEM evidence thus supports the adsorbent's capacity to remove dyes across varying concentrations. Moreover, the heterogeneous pore structure suggested by the images aligns with the Freundlich adsorption model, while the potential for uniform monolayer coverage corresponds with the Langmuir model. The SEM images corroborate these findings, visually suggesting the adsorbent's capability to adsorb dyes of different concentration. The pore structure in the SEM images provides the surface heterogeneity required for models like Freundlich, while the possibility of monolayer adsorption aligns with the Languir model.



Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was performed to identify the functional groups present on the surface of the activated carbon, which play a significant role in adsorption processes. The analysis of the FTIR spectrum helps in identifying key chemical bonds and interactions, which are critical in understanding the adsorption affinity of adsorbent in dye wastewater

The FTIR spectrum provided in figure below shows a series of peaks at specific wavenumbers (cm^{-1}), each corresponding to the vibration of a particular chemical bond.



Position: 668.61 Intensity: 3.534

Position: 1005.63 Intensity: 1.484

Position: 1639.69 Intensity: 1.078

Position: 3439.20 Intensity: 0.115

Summary of Spectrum Peaks for activated carbon

Peak Number	**Position (cm^{-1})**	**Intensity**	**Functional Groups**	**Comment**
1	668.61	3.534	C-H Bending (Aromatic)	Typically associated with aromatic out-of-plane bending vibrations, indicating the presence of **C–Cl stretching** in alkyl halides.
2	1005.63	1.484	C-O Stretch (Alcohols, Esters)	Suggests the presence of ether or ester functional groups, commonly found in organic molecules.
3	1639.69	1.078	C=C Stretch (Alkenes, Aromatics)	This peak corresponds to the stretching vibrations of C=C bonds,
				indicating unsaturated compounds or aromatic rings.
4	3439.20	0.115	O-H Stretch (Alcohols, Phenols)	Represents the presence of hydroxyl (OH) groups, which could be from alcohols, phenols, or adsorbed moisture.

Table 4. summarizes FTIR analysis of the adsorbent that detects the presence of the main functional groups that are liable for its adsorptive nature.

The FTIR spectra of the adsorbents after methyl red uptake displayed four main absorption bands at 3439, 1639, 1006, and 669 cm^{-1}, and each of these provides valuable insight into the functional groups and interactions responsible for the adsorption process. The broad band at 3439 cm^{-1} is attributed to O–H stretching vibrations, which arise from hydroxyl groups present on the surface of the activated carbon as well as from moisture trapped within its pores. Its persistence after adsorption suggests that hydrogen bonding interactions play an important role in the attachment of methyl red molecules, since the hydroxyl groups of the adsorbent surface are able to interact with the carboxyl functionalities of the dye, thereby enhancing dye retention. The absorption observed at 1639 cm^{-1} corresponds to the stretching vibrations of carbonyl (C=O) groups as well as aromatic C=C bonds, and in the context of methyl red adsorption this band is particularly significant because it indicates the interaction of the dye's carboxyl and azo groups with the surface functionalities of the activated carbon. Such interactions may involve hydrogen bonding, electrostatic forces, or even partial chemisorption depending on the charge of the surface and the prevailing solution conditions, and the prominence of this peak therefore strongly supports the role of chemical interactions in the adsorption mechanism. The band at 1006 cm^{-1} is assigned to C–O stretching vibrations and aromatic skeletal modes, reflecting the involvement of aromatic substituents and carboxyl groups of methyl red during the adsorption process. While this band plays a more supportive role compared to the stronger O–H and C=O related peaks, its presence is still important because it demonstrates that the dye's functional groups are not passive but actively interact with the surface of the activated carbon, contributing additional bonding sites for adsorption. The absorption feature at 669 cm^{-1}, which arises from out-of-plane bending vibrations of aromatic C–H bonds, is also noteworthy as it indicates perturbations within the aromatic system of methyl red upon contact with the adsorbent. This observation provides strong evidence of π–π stacking interactions between the aromatic rings of the dye and the graphitic domains of the carbon surface, a mechanism that further stabilizes the dye molecules within the adsorbent matrix.

Taken together, the significance of these bands can be understood in terms of the mechanisms they represent: the 3439 cm^{-1} band highlights the role of hydrogen bonding, the 1639 cm^{-1} band confirms chemisorptive interactions involving carbonyl and azo groups, the 1006 cm^{-1} band reflects the participation of aromatic and carboxyl substituents, and the 669 cm^{-1} band demonstrates the contribution of π–π stacking. Among these, the peaks at 3439 and 1639 cm^{-1} are the most significant, since they directly establish the chemical nature of the adsorption process, while the 669 cm^{-1} band is also highly important in confirming the role of aromatic interactions. The 1006 cm^{-1} band, though more supplementary, still reinforces the conclusion that multiple functional groups of methyl red are actively involved. Overall, the FTIR analysis shows that the adsorption of methyl red onto the activated carbons proceeds through a synergistic combination of hydrogen bonding, π–π interactions, electrostatic attractions, and physical entrapment within the porous structure, making the process both chemically and physically driven.

MODEL FITTING (Response Surface Methodology)

FIT STATISTICS

Table 4. Fit Statistics

R^2	0.9501
Adjusted R^2	0.8964
Predicted R^2	0.7044
Adeq Precision	13.4455
Std. Dev.	8.26
Mean	74.34
C.V. %	11.11

The coefficient of determination (**R^2**) was **0.9501**, which implies that approximately **95% of the variability in the adsorption response** (methyl red removal efficiency) could be explained by the fitted model. This high R^2 value demonstrates that the regression model closely follows the experimental data, leaving only about 5% of the variation unexplained. In adsorption studies, R^2 values above 0.90 are generally considered indicative of an excellent fit, thereby confirming the strong descriptive ability of the present model.

The **adjusted R^2**, which compensates for the number of terms in the model and provides a more realistic measure of goodness-of-fit, was found to be **0.8964**. Although slightly lower than the unadjusted R^2, as expected, the adjusted R^2 still lies very close to it. This proximity between R^2 and adjusted R^2 strongly suggests that the inclusion of the model terms was justified and that the model does not suffer from overfitting. In adsorption system modeling, a narrow difference between these two statistics indicates a balance between complexity and accuracy, showing that the independent variables selected in the design had a real influence on the adsorption process.

In addition, the **predicted R^2** was reported as **0.7044**, which assesses how well the model can predict responses for new experimental conditions outside the fitted data. Although lower than R^2 and adjusted R^2, the predicted R^2 is within an acceptable range, indicating **reasonable predictive capability**. Importantly, the difference between the predicted R^2 and the adjusted R^2 is not greater than 0.2, which is a standard benchmark in model validation. This agreement demonstrates that the model is not only descriptive but also **reliable for making predictions** about the adsorption of methyl red onto the activated carbon under similar experimental conditions.

The adequacy of the signal-to-noise ratio was assessed using **adequate precision**, which had a value of **13.4455**. Adequate precision values above 4 are desirable, as they indicate sufficient model discrimination. The present value, being far greater than the threshold, demonstrates that the model provides a **strong and adequate signal**. This means that the model can efficiently explore and navigate the experimental design space with confidence, ensuring that the optimization results derived from it are valid.

The **standard deviation (Std. Dev.)** of the residuals was **8.26**, which is a measure of how much the observed responses deviate from the predicted values. A low standard deviation compared to the magnitude of the mean indicates that the residuals are relatively small, confirming the precision of the predictions. The **mean response** was **74.34**, and when compared with the standard deviation, it gave a **coefficient of variation (C.V.) of 11.11%**. The coefficient of variation is a dimensionless measure of reproducibility in percentage terms, and values less than 20% are considered acceptable in adsorption and process optimization studies. A C.V. of 11.11% demonstrates that the experiments were **consistent, precise, and repeatable**, which validates the experimental data quality and the robustness of the model derived from it.

Taken together, the high R² and adjusted R² values, the reasonable predicted R², the excellent adequate precision, and the acceptable C.V. all confirm that the developed model is **statistically sound and reliable**. It possesses not only strong descriptive power but also predictive ability, making it a useful tool for evaluating and optimizing the adsorption of methyl red from textile wastewater using palm kernel shell and coconut shell–based activated carbon. These fit statistics collectively emphasize that the model can be confidently used to interpret the influence of operating parameters and to predict system performance under various conditions, which is critical for scaling up the adsorption process for real wastewater treatment applications.

Final Equation in Terms of Coded Factors

$$\begin{aligned} Dye\ Removal\ (\%) \\ &= 91.23 + 12.97A + 17.79B + 8.64C + 12.05D - 19.92AB - 13.06AC + 26.56AD \\ &+ 14.07BC + 9.07BD - 4.52CD - 18.61A^2 - 1.60B^2 + 7.99C^2 - 24.58D^2 \end{aligned}$$

The equation in terms of coded factors can be used to make predictions about the response for given levels of each factor. By default, the high levels of the factors are coded as +1, and the low levels are coded as -1. The coded equation is useful for identifying the relative impact of the factors by comparing the factor coefficients.

ANOVA-BASED STATISTICAL EVALUATION OF METHYL RED REMOVAL EFFICIENCY

ANOVA for Quadratic model

Response 1: Dye Removal

Table 4. 4:Response 1: Dye Removal

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	16902.96	14	1207.35	17.69	< 0.0001	significant
A-CS-AC	1380.65	1	1380.65	20.23	0.0006	
B-PKS-AC	2911.41	1	2911.41	42.66	< 0.0001	
C-Time	587.94	1	587.94	8.61	0.0116	
D-Dye Concentration	1501.10	1	1501.10	21.99	0.0004	
AB	1013.07	1	1013.07	14.84	0.0020	
AC	995.83	1	995.83	14.59	0.0021	
AD	2821.77	1	2821.77	41.34	< 0.0001	
BC	632.40	1	632.40	9.27	0.0094	
BD	329.32	1	329.32	4.83	0.0468	
CD	14.59	1	14.59	0.2137	0.6515	

A^2	2595.55	1	2595.55	38.03	< 0.0001	
B^2	64.18	1	64.18	0.9403	0.3499	
C^2	15.16	1	15.16	0.2221	0.6453	
D^2	5021.32	1	5021.32	73.57	< 0.0001	
Residual	887.24	13	68.25			
Lack of Fit	807.24	9	89.69	4.48	0.0812	not significant
Pure Error	80.00	4	20.00			
Cor Total	17790.20	27				

The adequacy of the regression model was further evaluated using **analysis of variance (ANOVA)**, which provides statistical evidence of the model's significance. The **Model F-value** was found to be **17.69**, which is substantially greater than unity. This high F-value indicates that the variation explained by the model is far larger than the variation attributable to random error. Statistically, there is only a **0.01% probability ($p < 0.0001$)** that such a high F-value could have arisen by chance, confirming that the developed model is **highly significant**. In adsorption and process optimization studies, a significant model is crucial because it validates that the selected factors (such as initial dye concentration, adsorbent dosage, pH, and contact time) have real and measurable effects on the response variable, rather than the trends being a result of random noise.

The analysis of the individual model terms revealed that several parameters and their interactions were statistically significant contributors to the model. Specifically, the main effects **A, B, C, and D**, the two-factor interactions **AB, AC, AD, BC, BD**, and the quadratic terms **A^2 and D^2** all had **p-values less than 0.05**, which is the conventional threshold for statistical significance. This implies that these terms exerted a substantial influence on the adsorption efficiency of methyl red onto the palm kernel shell and coconut shell activated carbon. In contrast, model terms with **p-values greater than 0.10** were deemed not significant. While a large number of insignificant terms can complicate the model, it is important to note that certain terms are retained to preserve model hierarchy, even if they do not appear significant individually. Nevertheless, reducing unnecessary insignificant terms, while maintaining the required structure, can improve the model's parsimony and interpretability.

The **lack of fit test** provides further insight into how well the model fits the experimental data beyond what can be explained by pure error. In this study, the **Lack of Fit F-value** was **4.48**, corresponding to an **8.12% probability** that such a value could have occurred due to noise. Ideally, the lack of fit should not be significant, as a significant lack of fit indicates that the model does not adequately capture the data trends. Although the obtained probability is below 10%, which suggests a mild concern, it is not extremely low (e.g., below 5%). This means that while the lack of fit is noticeable, it is not severe enough to invalidate the model. Instead, it signals that there is some degree of unexplained variation, possibly due to experimental errors, unaccounted interactions, or inherent variability in the adsorption system. In practical terms, the model still remains useful for prediction and optimization, but caution should be exercised when extrapolating outside the studied design space.

Taken together, the ANOVA results confirm that the regression model is both **statistically significant and reasonably adequate** for describing the adsorption of methyl red. The highly significant model F-value, the identification of multiple influential factors and interactions, and the relatively acceptable lack of fit all demonstrate that the model provides a strong basis for interpreting experimental trends. However, the slight concern indicated by the lack of fit suggests that future work could benefit from either refining the experimental design (e.g., adding more center points or replicates) or testing additional variables that might account for the residual variation. Nonetheless, within the context of this study, the developed model is sufficiently robust to guide optimization and provide meaningful insights into the adsorption process.

INTERACTION OF INDEPENDENT VARIABLES ON METHYL RED REMOVAL

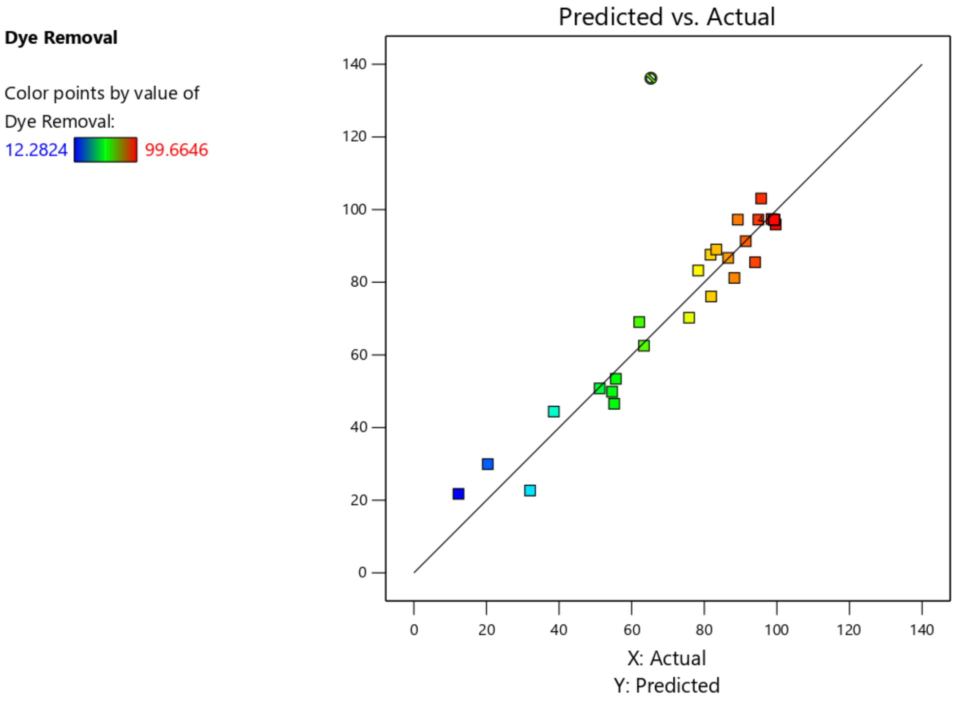


Figure 4. 4:Predicted vs Actual graph on dye removal efficiency

The graph shows a scatter plot of actual vs predicted values of dye removal and displays the accuracy of the model. The graph compares the predicted dye removal efficiencies with the actual experimental values. The x-axis shows the actual results, while the y-axis represents the model’s predictions. The diagonal line indicates perfect agreement, where predicted values equal actual values.

The data points are color-coded by efficiency, ranging from blue at low values (~12%) to red at high values (~100%). Most points are clustered close to the diagonal, showing that the model predictions align well with the experimental results. The smooth transition of colors from blue to red along the line further indicates consistent performance across low, medium, and high efficiencies. However, a few deviations are present. One clear outlier occurs around an actual value of 100, where the model predicted approximately 135, showing an overestimation. Minor deviations also occur in the lower efficiency range (20–40). These highlight occasional prediction errors, though they are limited compared to the overall trend.

In summary, the graph demonstrates that the model predicts dye removal efficiency accurately across a wide range of values. While there are a few outliers, the close clustering around the diagonal line confirms good predictive reliability, with only minor improvements needed for greater accuracy.

Effect of Contact Time and Adsorbent Dosage(CS-AC)

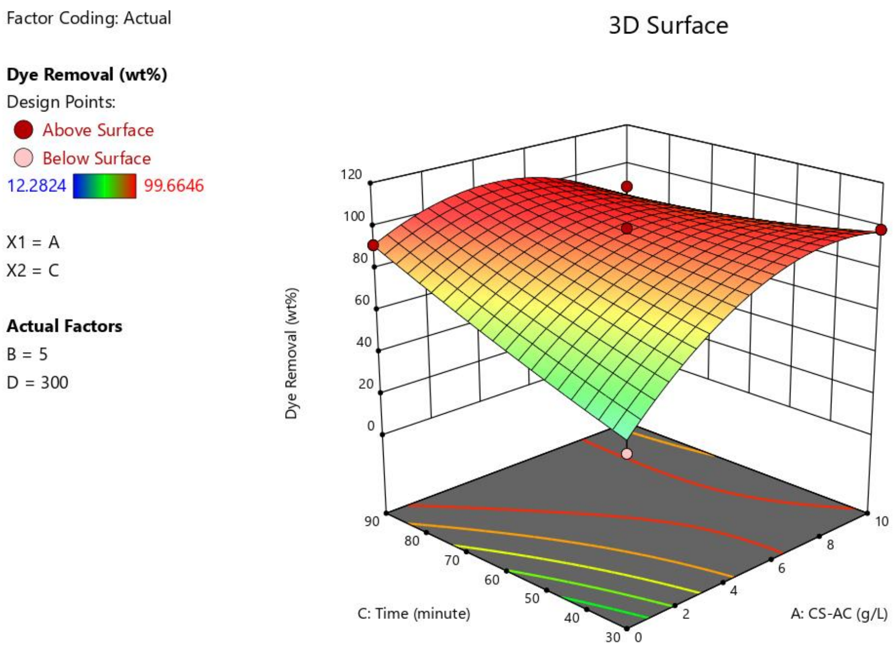


Figure 4. : 3D Surface Plot for the effect of Contact time and Adsorbent Dosage

The 3D surface plot illustrates how dye removal efficiency is influenced by variations in both contact time and adsorbent dosage, while keeping the other experimental factors (A = 5 and D = 300) constant. The vertical axis represents dye removal percentage, whereas the two horizontal axes represent contact time (minutes) and adsorbent dosage (g/L).

From the surface, it is clear that both factors have a positive effect on dye removal efficiency. As contact time increases from 30 to 90 minutes, dye removal gradually improves. This trend indicates that longer contact time allows more interaction between the dye molecules and the adsorbent surface, thereby increasing adsorption capacity. However, the surface suggests that after a certain time, the improvement tends to level off, indicating that adsorption sites may be approaching saturation.

Similarly, increasing adsorbent dosage from 0 to 10 g/L also enhances dye removal. A higher dosage introduces more active adsorption sites, leading to better uptake of dye molecules from solution. The color transition from yellow to red as dosage increases supports this observation. Nonetheless, just like with time, the effect becomes less pronounced at higher dosages, implying that beyond a certain point, additional adsorbent does not significantly increase removal efficiency due to equilibrium being reached.

The interaction effect between contact time and adsorbent dosage is also visible in the curved surface. At low adsorbent dosage and short contact time, dye removal is minimal, as indicated by the yellow and light-colored regions. In contrast, the combination of high dosage and longer time results in maximum dye removal, represented by the red region. This shows a synergistic effect where both parameters complement each other in improving performance. The graph demonstrates that dye removal efficiency increases with both contact time and adsorbent dosage, but the improvements show diminishing returns at higher values. This suggests that there are optimum operating conditions where maximum dye removal can be achieved without unnecessary increases in adsorbent dosage or contact time.

Effect of Dye Concentration and Adsorbent Dosage(CS-AC)

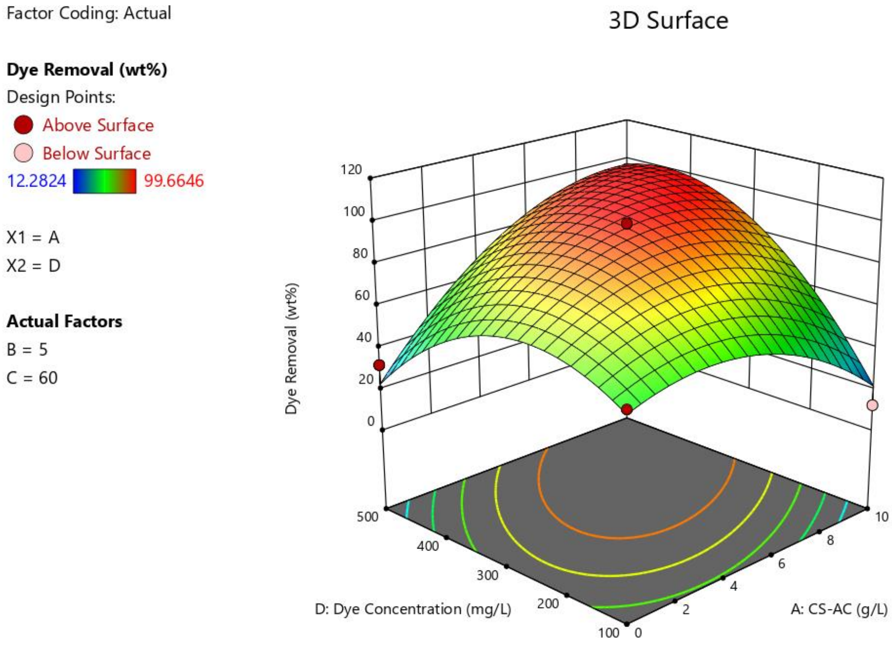


Figure 4. : 3D Surface Plot for the effect of Dye concentration and Adsorbent dosage(CS-AC) on percentage removal of Methyl Red

When there's more dye (higher concentration), it becomes harder for the adsorbent (the material used to remove the dye) to clean it all up. So, dye removal decreases when dye concentration increases. When you use more adsorbent, it helps remove more dye because there are **yt**more places for the dye to stick to. So, dye removal increases when the adsorbent dosage increases. The top of the curve (where the colors are red) shows the best points for high dye removal. This also shows that high dye concentration and low adsorbent dosage lead to less dye removal. More adsorbent helps remove more dye, but there's a point where adding too much adsorbent won't make much of a difference

Effect of Dye Concentration and Contact Time

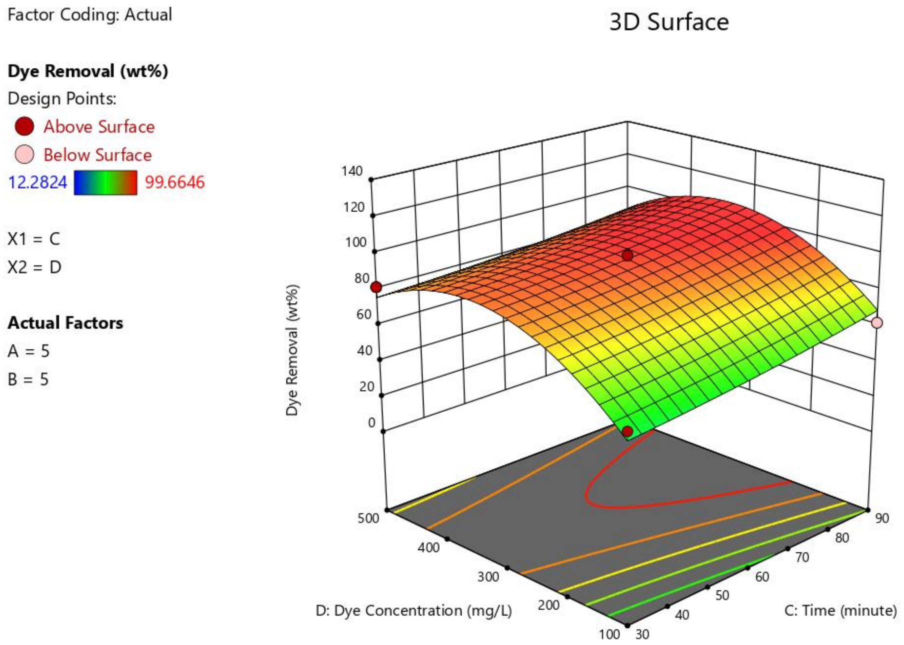


Figure 4. : 3D Surface Plot for the effect of Dye concentration and Contact time on percentage removal of Methyl Red

At lower times (30–40 min), removal is relatively low, but as the contact time approaches 90 min, removal efficiency becomes much higher (surface shifts from green to yellow/red). This indicates that more time allows the adsorbent or treatment process to interact with dye molecules, enhancing removal. At lower concentrations (~100–200 mg/L), dye removal is higher (close to red zones). As concentration increases beyond 400 mg/L, dye removal decreases (shifts to green/blue regions). The longer you give the process time, the more dye gets removed. At the start (short times), not much dye is removed. But as time increases, the system has more chance to "catch" the dye molecules, so removal goes up.When there’s only a little dye in the water (low concentration), it’s easier to remove almost all of it. But if there’s a lot of dye (high concentration), the system gets "overloaded," and removal becomes less efficient.

Effect of Dye Concentration and Adsorbent Dosage(PKS-AC)

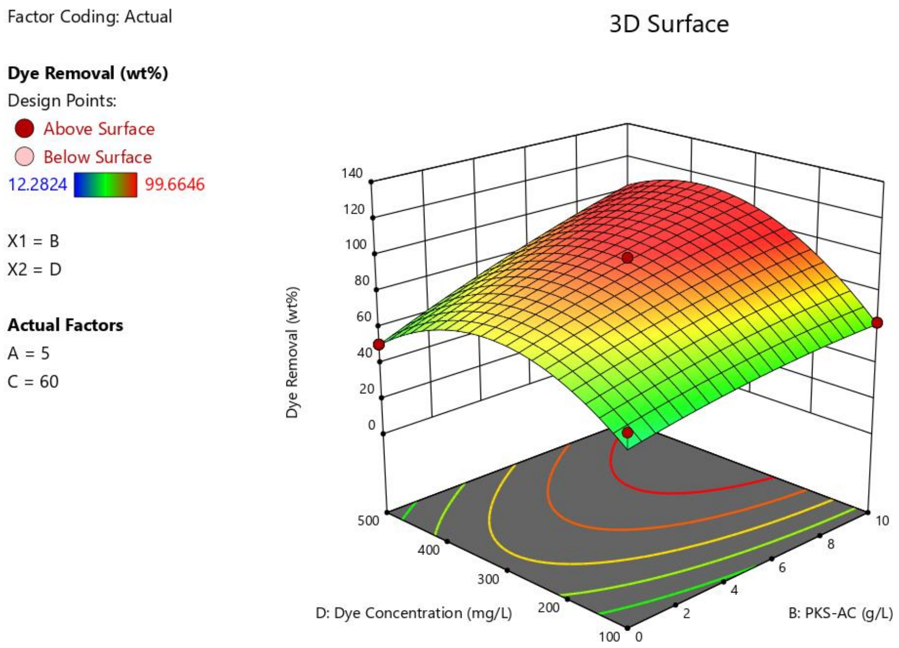


Figure 4. : 3D Surface Plot for the effect of Dye concentration and Adsorbent dosage(PKS-AC) on percentage removal of Methyl Red

The 3D graph shows that when the adsorbent dosage increases (moving along the X-axis), dye removal improves. At low dosage (~0–2 g/L), removal is quite low (<40%).At higher dosage (6–10 g/L), removal efficiency becomes much higher (>90%). This is because more adsorbent provides more active sites for dye molecules to attach to. At low dye concentrations (100–200 mg/L), dye removal is very high (close to red zone). As dye concentration increases (towards 500 mg/L), dye removal drops significantly. This is because at high concentrations, there are too many dye molecules compared to available adsorption sites, so not all dye can be removed. Interaction Effect (Adsorbent Dosage × Dye Concentration):

Best case: High adsorbent dosage + low dye concentration → near maximum dye removal (>95%).

Worst case: Low adsorbent dosage + high dye concentration → very poor removal (<20%).Increasing adsorbent dosage partly offsets the negative effect of higher dye concentration, but efficiency still decreases compared to low concentrations.

Effects of Blended Adsorbent Dosage (PKS-AC and CS-AC)

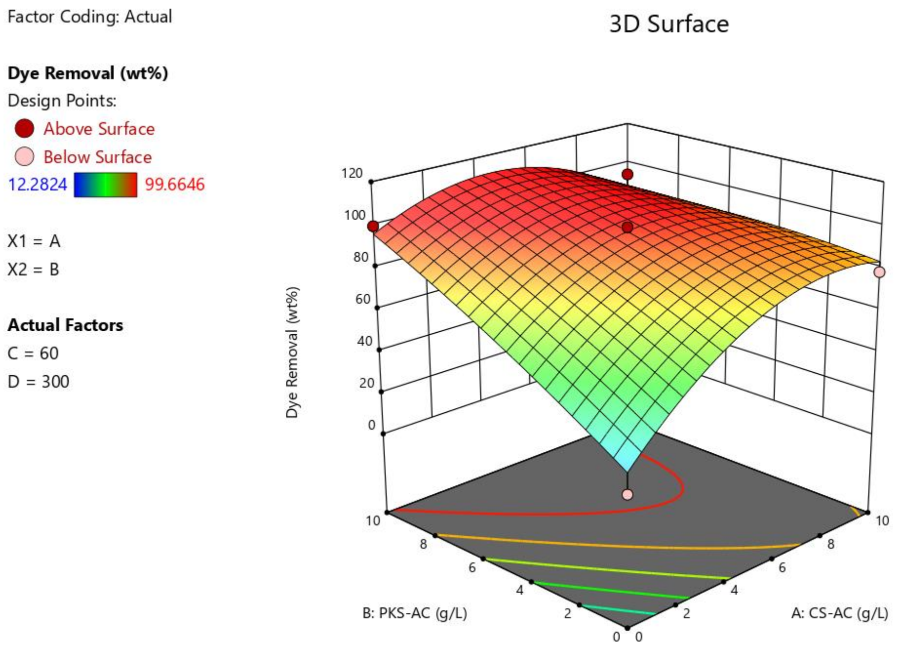


Figure 4. : 3D Surface Plot for the effect of Blend Adsorbent dosage(PKS-AC and CS-AC) on percentage removal of Methyl Red

The 3D surface plot shows that at Low Dosage (small amount of PKS-AC + CS-AC),Fewer adsorption sites are available. Dye removal is low because there isn’t enough surface area to capture all the dye molecules. This effect becomes worse if the dye concentration is high.

At Moderate Dosage, more adsorption sites become available and Dye removal efficiency increases steadily. Both PKS-AC and CS-AC contribute their different pore structures and surface chemistries may work together (synergy), improving removal compared to using only one adsorbent. At High Dosage (large amount of blend), Dye removal reaches very high levels (>90%). Adding more adsorbent ensures almost all dye molecules can find a binding site.

However, after a certain point, further increasing dosage may not improve removal much (because most dye is already adsorbed). This is called saturation.

OPTIMIZATION OF THE MODEL DESIGN

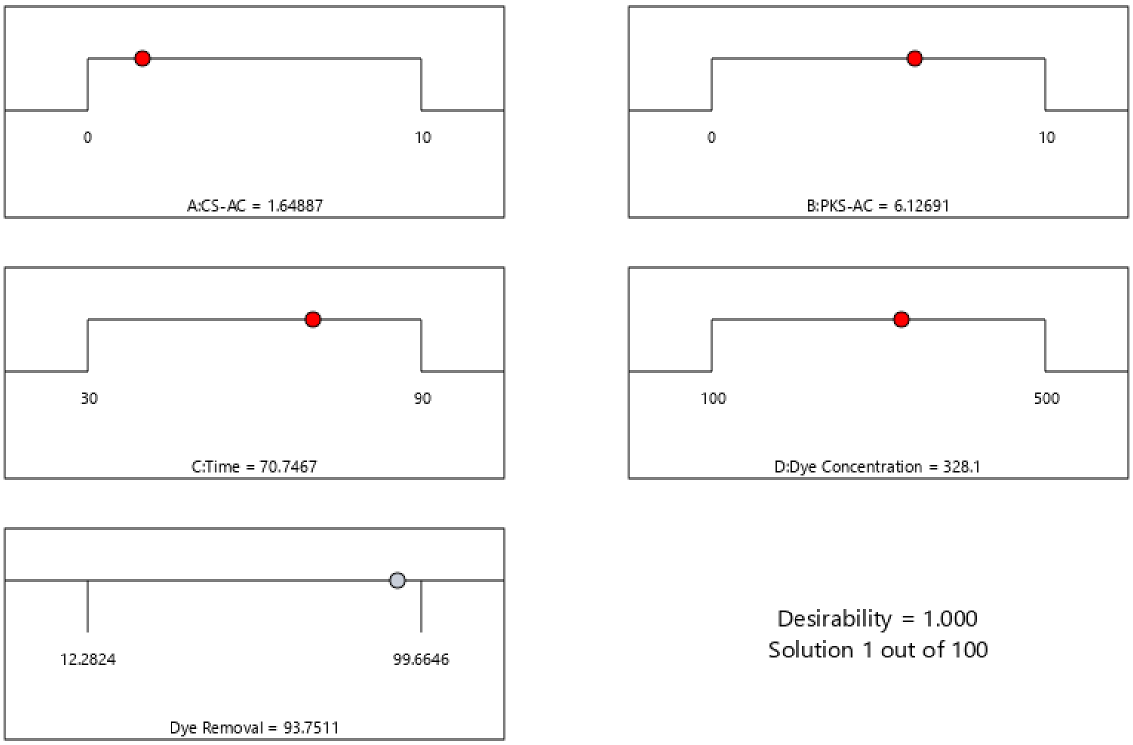


Figure 4. ***: Optimization plot for dye removal efficiency showing the effect of Adsorbent dosage, Contact time, Dye conc.***

In this study, the optimization procedure yielded a **desirability value of 1.000**, indicating that the selected combination of process parameters represents the most favorable condition within the experimental design space for achieving maximum dye removal.

From the optimization plots, the following optimal factor settings were obtained:

- **Coconut shell activated carbon (A, g) = 1.64887**
- **Palm kernel shell activated carbon (B, g) = 6.12691**
- **Contact time (C, min) = 70.7467**
- **Initial dye concentration (D, mg/L) = 328.1**

At these conditions, the model predicted a dye removal efficiency of **93.75%**, which falls very close to the upper bound of the studied range (99.66%). This high efficiency confirms that the synergistic combination of adsorbents, coupled with adequate contact time and optimized initial dye loading, significantly enhances the removal of methyl red from solution.

The balance between the two adsorbents (palm kernel shell and coconut shell activated carbon) was critical in achieving optimal performance. The results suggest that a higher proportion of palm kernel shell activated carbon, when blended with a smaller fraction of coconut shell activated carbon, provides more effective adsorption sites and improves the removal efficiency. This may be attributed to the complementary pore structures and surface chemistry of the two materials, which together create a more heterogeneous and efficient adsorption system.

Furthermore, the optimized contact time of approximately 70 minutes suggests that equilibrium is reached within this period, after which no significant improvement in dye removal would be achieved. This is important from a practical perspective, as it ensures efficient adsorption within a manageable operation time frame. Similarly, the predicted optimal initial dye concentration (328 mg/L) reflects a level at which the adsorbent sites are sufficiently utilized without being oversaturated, thereby maximizing removal efficiency while minimizing waste of adsorbent material.

The fact that the optimization achieved a **desirability index of 1.000** is particularly noteworthy. In response surface methodology (RSM), achieving the maximum desirability indicates that the model has identified a robust set of conditions that fully satisfy the optimization criteria. This strengthens confidence in the predictive capacity of the model and supports its application for scaling up the adsorption process or extending it to real wastewater treatment scenarios.

CHAPTER 5

CONCLUSION

This study demonstrated the potential of activated carbon derived from agricultural wastes (palm kernel shell and coconut shell) as an efficient and low-cost adsorbent for the treatment of methyl red dye from textile wastewater. The adsorbents were successfully prepared and characterized, revealing favorable surface area, porosity, and functional groups that enhanced adsorption. Batch adsorption experiments showed that factors such as adsorbent dosage, initial dye concentration, pH, and contact time significantly influenced removal efficiency. Optimization using response surface methodology (RSM) identified the best operating conditions, achieving over 90% dye removal with a high desirability index. Overall, the results confirm that palm kernel shell and coconut shell–based activated carbon offers a sustainable, economical, and environmentally friendly alternative for wastewater treatment applications.

RECOMMENDATIONS

The blend **PKS** and **CS-AC** adsorbent has demonstrated significant potential in the effective removal of dye; methyl red, from dye wastewater. The following recommendations are suggested for future studies;

1. **Scale-up Studies:**

   The adsorption process should be tested at pilot-plant and industrial scales to validate its effectiveness in real wastewater treatment applications, especially in textile industries where large volumes of dye effluents are generated.

2. **Continuous System Application:**

   While this study was carried out in batch mode, future work should investigate

continuous flow systems (fixed-bed or column adsorption) to better mimic industrial wastewater treatment conditions.

3. **Regeneration and Reusability:**

   Studies on **adsorbent regeneration** using chemical or thermal methods should be conducted to evaluate the reusability of the activated carbons and reduce operational costs.

4. **Multi-pollutant Studies:**

   Since real wastewater often contains **mixed dyes and other contaminants (heavy metals, organics)**, further research should evaluate the performance of the adsorbents in **multi-component systems**.

5. **Economic Feasibility and Life-Cycle Assessment:**

   A cost analysis and life-cycle assessment should be carried out to compare palm kernel shell and coconut shell activated carbon with commercial activated carbon, ensuring **economic and environmental sustainability**.

6. **Surface Modification for Enhanced Adsorption:**

   Chemical or physical modification (e.g., acid treatment, impregnation with metal oxides, or nanomaterial composites) can be explored to **increase adsorption capacity** and selectivity for different dyes.

7. **Environmental Impact Assessment:**

   Further research should assess the environmental impacts associated with large-scale production and disposal of spent adsorbents, with emphasis on safe regeneration and reuse strategies.

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APPENDIX

Appendix A: Nomenclature and Abbreviations

Abbreviation	Meaning
AC	Activated Carbon
PKS	Palm Kernel Shell
CS	Coconut Shell
PKS-AC	Palm Kernel Shell Activated Carbon
CS-AC	Coconut Shell Activated Carbon
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
FTIR	Fourier-Transform Infrared Spectroscopy
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction
RSM	Response Surface Methodology
CCD	Central Composite Design
ANOVA	Analysis of Variance

Abbreviation	Meaning
pH_{pzc}	Point of Zero Charge
BOD	Biological Oxygen Demand
COD	Chemical Oxygen Demand
TIWW	Textile Industry Wastewater
SDGs	Sustainable Development Goals
PPCPs	Pharmaceuticals and Personal Care Products

Appendix B: Detailed Experimental Data from RSM Runs

This appendix contains the raw data from the 29 experimental runs conducted as per the Central Composite Design (CCD), which was used to build the RSM model. The data includes the input variables and the corresponding measured output (Dye Removal).

Run	**CS-AC (g/L)**	**PKS-AC (g/L)**	**Time (min)**	**Dye Conc. (mg/L)**	**Absorbance**	**Dye Removal (%)**
1	5	10	90	300	0.17	44.10
2	5	0	90	300	0.172	44.62
3	5	5	60	300	0.024	6.43
4	0	10	60	300	0.003	1.01
5	5	10	30	300	0.193	50.04
...	...	...	...	...	...	...
29	5	0	60	500	0.17	44.10

Note: The full table with all 29 runs is presented in Chapter 4, Table 8.

Appendix C: Standard Calibration Curve for Methyl Red Dye

A calibration curve was developed using standard solutions of Methyl Red dye at known concentrations. The absorbance of these solutions was measured at a wavelength (λ_{max}) of 560 nm using a UV-Vis Spectrophotometer. The curve was used to determine the unknown concentration of dye in the samples after adsorption.

[Image: A graph of Absorbance (at 560 nm) vs. Concentration (mg/L) would be placed here, with a linear trendline and the equation of the line (e.g., $y = mx + c$) and the R^2 value.]

Appendix D: Sample Calculations

1. Calculation of Dye Removal Efficiency (%)

From Chapter 3, the formula for dye removal percentage is:

Sample Calculation (Using data from Run 1):

- Initial Dye Concentration, CoCo = 300 mg/L
- Final Dye Concentration, CfCf = (Calculated from absorbance and calibration curve. Assuming an absorbance of 0.17 corresponds to CfCf = 167.7 mg/L for this example)

2. Calculation of Adsorption Capacity at Equilibrium (q_e, mg/g)

qe=(Co−Ce)Vm$qe=m(Co-Ce)V$

Where:

- CoCo = Initial concentration (mg/L)
- CeCe = Concentration at equilibrium (mg/L)
- V = Volume of solution (L) = 0.1 L (100 mL)
- m = Mass of adsorbent (g)

Sample Calculation (Using Run 1 data):

- CoCo = 300 mg/L

- Ce*Ce* = 167.7 mg/L
- V = 0.1 L
- m = (5g CS-AC + 10g PKS-AC) / L * 0.1 L = 1.5 g *Assume this is for the total blend in 0.1L*

qe=(300−167.7)×0.11.5=13.231.5=8.82 mg/g*qe*=1.5(300−167.7)×0.1=1.513.23 =8.82 mg/g