CHARACTERIZATION OF COCONUT HUSK AS ADSORBENT FOR METHYLENE BLUE DYES

NURUL NATRAH BINTI ABDUL RAHMAN

SCHOOL OF CIVIL ENGINEERING

UNIVERSITI SAINS MALAYSIA

2022

CHARACTERIZATON OF COCONUT HUSK AS ADSORBENT FOR METHYLENE BLUE DYES

By

NURUL NATRAH BINTI ABDUL RAHMAN

This dissertation is submitted to
UNIVERSITI SAINS MALAYSIA
As partial fulfilment of requirement for the degree of

BACHELOR OF ENGINEERING (HONS)
(CIVIL ENGINEERING)

School of Civil Engineering

Universiti Sains Malaysia

August 2022

Appendix A8

UNIVERSITI SAINS MALAYSIA

SCHOOL OF CIVIL ENGINEERING
ACADEMIC SESSION 2021/2022

FINAL YEAR PROJECT EAA492/6
DISSERTATION ENDORSEMENT FORM

Title: CHARACTERIZATION OF COCONUT HUSK AS ADSORBENT FOR METHYLENE BLUE DYES

Name of Student: NURUL NATRAH BINTI ABDUL RAHMAN

I hereby declare that all corrections and comments made by the supervisor(s)and examiner have been taken into consideration and rectified accordingly.

Signature:

Approved by:

(Signature of Supervisor)

Date : 10 / 8 / 2022

Name of Supervisor :DR NIKAZIMATOLAKMA AWANG

Date : 10 / 8 / 2022

Approved by:

(Signature of Examiner)

Name of Examiner : DR ROSNANI ALKARIMIAH

Date : 10 / 8 / 2022

ACKNOWLEDGEMENT

Today is the culmination of a demanding six-month period: penning this note of gratitude is the finishing touch on my dissertation. I want to thank everyone who has supported and encouraged me over this challenging period. All praise is due to ALLAH SWT, the Almighty, for the blessings, strength, opportunities, and fortitude expected to accomplish this study.

I want to thank my research advisor, Dr Nikazimatol Akma Binti Awang, for her time, compassionate direction, perseverance, and encouragement throughout the entire dissertation work, from which I have gained so much. This dissertation could not have been completed without her direction and enthusiastic participation at every stage. My most profound appreciation goes to my cherished parents, Abdul Rahman Bin Abdul Rashid and Sarimah Binti Ismail, for their selflessness, steadfast support, and encouragement. Their love, continuous prayer, and dua' have strengthened me daily as I struggle to complete my course.

I want to express my heartfelt gratitude to all of my friends for their wise counsel and assistance during my undergraduate studies. Thank you for four incredible years of love, friendship, and support. Finally, I would like to thank the members of School of Civil Engineering at Universiti Sains Malaysia for their assistance.

Thank you all very much!

Penang, Malaysia, Jun 24, 2022 Natrah Rahman

ABSTRAK

Pewarna digunakan dalam sektor tekstil untuk mewarnakan bahan fabrik seperti kapas dan nilon. Prosedur pencelupan mencemarkan bekalan air dengan memendapkan efluen pewarna ke dalamnya. Antara strategi yang paling berkesan untuk penyahwarnaan ialah penjerapan karbon teraktif. Sekam kelapa karbon aktif (ACCH) telah dibangunkan untuk pengurangan metilena biru (MB) dalam penyelidikan ini. Kadar penyingkiran pewarna dikaji menggunakan pelbagai faktor, termasuk pH, suhu, dos penjerap, masa sentuhan, dan kepekatan awal pewarna (Oliveira et al., 2020). pH rawatan optimum, dos MB dan ACCH ditentukan masing-masing ialah pH 5, 15mg/mL dan 1.0g/mL, menghasilkan penyingkiran pewarna MB sebanyak 84.02%. Kesan kepekatan pewarna awal (15mg/mL - 75mg/mL) telah dikaji. Apabila kepekatan awal pewarna dan pH meningkat, begitu juga dengan penjerapan MB pada ACCH. Dengan menggunakan model resapan intrapartikel, mekanisme penjerapan ditentukan. Keputusan imej SEM juga menunjukkan bahawa bentuk penjerap bio yang dirawat adalah lebih berliang dan lebih kasar untuk meningkatkan proses penjerapan. Imej EDX boleh menunjukkan unsur yang boleh ditemui selepas kajian penjerapan kelompok. 82.58% karbon (C), 0.12% aluminium (Al), 11.60% oksigen (O), 4.51% nitrogen (N), silikon (Si), 0.07% sulfur (S), 0.75 % kalsium (Ca), dan 0.24% fosforus (P) dikesan oleh analisis unsur EDX dalam ACCH. Sementara itu, pemeriksaan FTIR mendedahkan penurunan pada graf akibat penyingkiran lignin dan hemiselulosa. Ciri-ciri sabut kelapa berdasarkan titik Iso-elektrik (IEP) menggunakan Zeta Sizer dan analisis pembelauan sinar-X (XRD) juga telah dikaji dalam kertas penyelidikan ini.

ABSTRACT

Dyes are used in the textile sector to colour fabric materials such as cotton and nylon. The dyeing procedure polluted water supplies by depositing dye effluents into them. Amongst the most efficient strategies for decolorization is activated carbon adsorption. Activated carbon coconut husk (ACCH) was developed for methylene blue (MB) reduction in this research. Dyes removal rates were studied using a variety of factors, including pH, temperature, adsorbent dosage, contact time, and initial dye concentration (Oliveira et al., 2020). The optimal treatment pH, dosage of MB and ACCH were determined to be pH 5, 15mg/mL and 1.0g/mL respectively, resulting in MB dye elimination of 84.02%. The impact of initial dye concentration (15mg/mL - 75mg/mL) was studied. As the initial dye concentration and pH rose, so did the adsorption of MB on ACCH. By utilizing the intraparticle diffusion model, the adsorption mechanism was determined. The SEM images results also indicate that the shape of the treated bio adsorbent is more porous and rougher to enhance the adsorption process. EDX images can indicate which element can be found after batch adsorption studies. 82.58% carbon (C), 0.12% aluminium (Al), 11.60% oxygen (O), 4.51% nitrogen (N), silicon (Si), 0.07% sulphur (S), 0.75 % calcium (Ca), and 0.24% phosphorus (P) were detected by element EDX analysis in the ACCH. In the meantime, FTIR examination reveals a decline in peaks due to the elimination of lignin and hemicelluloses. Characteristic of coconut husk based on Iso-electric point (IEP) using Zeta Sizer and X-ray diffraction analysis (XRD) also been studied in this research paper.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF EQUATIONS

LIST OF ABBREVIATION

ACCH – Activated Carbon Coconut Husk

MB – Methylene Blue

CH – Coconut Husk

AAS - Atomic Absorption Spectroscopy

PPS – Peganum Harmala Seeds

FITR - Fourier Transform Infrared Spectroscopy

XRD – X-ray Diffraction Analysis

CHAPTER 1

INTRODUCTION

1.1 Background

Water is necessary for flora and fauna to survive. Today, degradation of waterbodies with a diverse spectrum of contaminants is a serious cause of worry. Dyes are one of the most common types of contaminants found in industrial effluents, and they may be detected with the naked eye. The most prevalent classifications are negative charge (anion), positive charge (cationic), and non-ionic dyes (Carney Almroth et al., 2021). Dyes are organic molecules that produce colour and attach to textiles or surface layers. Depolarization of effluent from fabric and other sectors is a critical challenge for pollution control since dyes are water-soluble and yield particularly vibrant colours in acidic water (Carney Almroth et al., 2021). Dyes are disposed of into wastewater as pollutants throughout colouring activity.

Water contamination due to the failure of textile manufacturers to properly dispose of their wastewater is currently one of the world's greatest challenges. In several nations, like China and South African estuaries, the textile industry substantially contributes to the world economy and environmental pollution (Olisah et al., 2021). The effluent containing dyes is a significant environmental pollutant that also affects human health, as the textile industry generates vast quantities of highly coloured wastewater carrying a variety of persistent toxins (Ali et al., 2022; Carney Almroth et al., 2021). Over 10000 dyes are widely viable globally and used in the fabric and industrial sectors, with dye demand in the fabric sectors more than 1000 tonnes annually (Chandanshive et al., 2020). According to their origin, composition, and use, dyes are frequently divided into several categories (Holkar et al., 2016, Akpomie and Conradie, 2020). Textile industries extensively use azo, direct, reactive, mordant, acid, basic, disperse, and synthetic sulphide dyes. Wool, cotton, silk, polyester, polyamide, and acrylic are used in natural and synthetic textile manufacturing (Deopura and Padaki, 2015, Silva et al., 2021). In addition, textile businesses use numerous highly harmful chemicals at various phases of production, such as sizing, softening, desizing, brightening, and finishing agents (Kishor et al., 2021). However, textile dyes do not stick to fabric securely. Without prior treatment, they are deposited as effluent and wastewater into aquatic habitats such as lakes, rivers, streams, and ponds, creating major ecotoxicological concerns with toxic effects on living species (Parmar et al., 2022).

The bulk of these colours are hazardous and artificial, with prediction of malignant and mutagenic consequences (Chatterjee et al., 2005; Daneshvar et al., 2007). Even at very low concentrations, the presence of these colours is highly visible and unwanted. Due to their artificial sources and predominantly inorganic structures that are highly hazardous and biologically non-degradable, textile wastewater is tough to control from an ecological point of view (Ali et al., 2022). Researchers around the world have employed several methods to extract dye from industrial effluents. The adsorption mechanism is one of most successful methods to degrade colour from wastewater. Basic dye is hydrophobic as it has a weaker hydrogen bond. It is water-soluble because of the existence of alcohol or acetic acid. Basic dye, also known as a cationic dye, is an artificial colour that can react with anion molecules. The properties of methylene blue is shown in Figure 1.1.

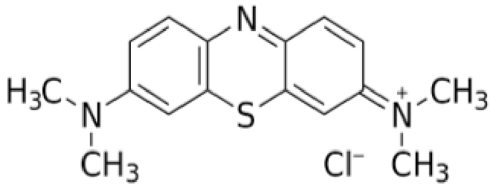
Properties	Cationic Azo Dye
Synonym name	Basic Blue 9
Molecular formula	$C_{16}H_{18}ClN_3S$
Molecular weight	319.851 g/mol
Absorbance wavelength(λ_{max})	664 nm
Molecular structure	

Figure 1.1 Properties of MB (Azim et al., 2018)

Adsorption, oxidation, electrocoagulation, and photocatalytic degradation have already been utilized to degrade colours from effluent. Adsorption has attracted extensive interest in those techniques because of their large benefits in terms of budget, simplicity of handling, flexibility of design and efficiency, and responsiveness to hazardous pollutants (Rashid et al., 2018). Activated carbon may be regarded as the most preferred adsorbent for decolorization between the numerous adsorbents due to its exceptional high adsorption capacity. However, due to its expensive cost, activated carbon is not widely used. As a result, the focus has switched to finding cheaper and more efficient activated carbon replacements. Biological sources, manufacturing and livestock waste, and bio-sorbents are several alternatives for low-cost adsorbents (Anderson et al., 2022).

Adsorption is a first-rate method for eliminating soluble organic pollutants such as colours from effluent discharge. Adsorption is the concentration of molecules on the surface of inorganic compounds. Adsorption is a surface phenomenon that primarily applies the principles of hydrophobic interactions. When an adsorbate directly interacts with the high porosity of the adsorbent, the solute is concentrated at the surface of the adsorbent due to liquid-solid intermolecular attractions (Kandisa & Saibaba KV, 2016). Adsorption is a chemical process used to extract impurities from dye effluent.

The economical and easily accessible adsorbent is made from coconut husk. The coconut mesocarp, which comprises roughly 33% to 35% of the husk, is known as coconut husk (CH) (Aziz et al., 2016). Multiple studies on the use of agricultural by-products such as rice straw, oil palm fibre, and rubberwood sawdust as adsorbent compounds in water purification have been conducted, but coconut husk tends to become the most favoured, with cellulose and lignin accounting for 60% of the husk (Sivapragasam 2008). The hydroxyl groups of these two compounds act as dye adsorbent surfaces (Neto et al. 2011; Wong et al. 2013). After the palm oil industry, rubber, and paddy field, coconut is Malaysia's fourth-biggest commercial production in total cultivation area (Aziz et al., 2016). If proper dumping and treatment processes are not taken seriously, the environmental footprint of husk and shell by-products will be severe. As an outcome, turning the coconut husk into an adsorbent has become a viable value-added alternative (Aziz et al., 2016).

The dosage of activated carbon coconut husk (ACCH) and methylene blue dyes (MB), and pH are all varied in constant temperature and agitation rate batch mode adsorption studies to optimize the process. Within an hour of operation, the solution reached equilibrium. The adsorption of dye content is significantly influenced by pH and initial dye concentration of the wastewater. Maximum adsorption was observed at a pH of 5, which represents optimum process variables. There are two ways of processing adsorbents, physical and chemical. Heat up the adsorbent in an oven to activate the carbon is the physical treatment of wastewater while adding acid or alkali is the chemical method (Budianto et al., 2021).

The use of an adsorbent such as activated carbon to remove dyes from industrial wastewater effluents is effective, but its cost limits its usage in large-scale applications (Kandisa & Saibaba KV, 2016). Experiments have shown that numerous inexpensive non-conventional adsorbents may also be used to remove dyes effectively. As a result, research into finding effective and economical adsorbents generated from natural resources is becoming more important for dye removal (Kandisa & Saibaba KV, 2016). Parameters that are going to be observe, are pore sizes and surface area, turbidity, pH, colour, and dosage of the activated carbon coconut and methylene blue dyes.

The surface area and pore volume are two important properties for determining the adsorption capacity of carbon adsorbents such as coconut husk. The larger the surface area, the greater the potential for absorption (Liao et al., 2013). UV–visible spectroscopy validated the creation of methylene blue, whereas Fourier Transform Infrared Spectroscopy and X-ray Diffraction Spectroscopy established the integration of magnetite with highly porous carbon. Using a Scanning Electron Microscope, the morphological properties of magnetite nanoparticles and very porous carbon were examined (Mohamed Khalith et al., 2021). It is common to practice utilizing FTIR spectroscopy to determine a surface's functional groups, as functional groups play a crucial role in dye adsorption processes (Janani et al., 2022). They fitted pseudo-second-order equation with kinetics and Langmuir monolayer favouring isotherm. This work demonstrates the efficacy of removing MB dyes from wastewater by adsorption batch study, expanding their utility in wastewater treatment.

1.2 Problem Statement

Dyes are emitted into effluent by a variety of industries, including paper, food colouring, cosmetics, dyestuffs, publishing, and flooring. The fabric industry and dyeing businesses have adopted vast amounts of dye, and these colouring pollutants are discharged into the ecosystem as untreated wastewater. These colours must be treated before discharging into water bodies due to their high toxicity to bacterial diversity and even hazardous to animals. They are among the most complex groups to eliminate from effluent discharge due to their resistance to aerobic digestion and light-stable and biodegradable constituents. As a result, adsorption has emerged as one of the most successful ways for removing colours from dyes effluent. Adsorption is a popular approach since it is quick, economical, easy, and requires little commitment. For this research, coconut husk is used to remove basic dyes such as methylene blue in the wastewater.

1.3 Objectives

1) To determine the characteristic of coconut husk based on Iso-electric point (IEP), Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction analysis (XRD), and Image analyzer (SEM and digital microscope).
2) To study the optimum condition of activated carbon coconut husk to degrade the methylene blue dyes.
3) To evaluate the adsorption capacity of activated carbon coconut to remove methylene blue from aqueous solutions.

1.4 Scope of Work

In this study, the wastewater sample was prepared in the laboratory. Activated carbon coconut was selected as adsorbent and methylene blue as adsorbate studied in terms of pH, colour, turbidity, and the efficiency of the MB removal using ACC. This study focused on the characterization of the adsorbents upon completing the batch study of adsorption to evaluate its removal. As for the molecular size of ACC, FTIR and XRD are applied to evaluate the porosity and surface area.

1.5 Dissertation Outline

This thesis is organized into five parts and appendices that seek to convey the work done on the subject in a way that is compatible with the development of the subject being discussed. Each contribution to the thesis is addressed in its section.

This research is introduced in Chapter 1. The first chapter outlines the study's background and defines the thesis's basic framework in terms of issue statement, objectives, and scope of work.

Chapter 2 is a review of the literature. The second chapter focuses on some of the past research relating to this research study.

Chapter 3 is the methodology of the research. The third chapter explain in detail the techniques used to carry out the research through batch study of adsorption.

Chapter 4 is result and discussion. The fourth chapter reports the results obtained from the batch study of adsorption and discuss the outcomes.

The findings and conclusions are included in Chapter 5. The fifth chapter summarises the work and offers suggestions based on the study's objectives. Furthermore, this chapter tackles several unresolved difficulties and suggests future study approaches.

1.6 Expected Outcomes

1) Successfully reduce the number of dyes in the wastewater along with other pollutants by using sorption process
2) Successfully solve issues regarding clogging of sewer by dyes
3) Successfully utilize palm coir to test sorption process

1.7 The Importance and Benefits of the Research

Problems due to the properties of dyes can be solved by utilizing coconut husk to test the sorption process. By understanding the mechanism of methylene blue contacted with coconut husk, dyes can be reduced massively in wastewater. Dyes cause major issues during wastewater treatment because they are insoluble in water. Adsorption is a basic and cost-effective technology for treating wastewater.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

The coconut husk activated carbon used for industrial wastewater treatment can achieve a discharge concentration of 9 mg/L. The refinery wastewater purified by the coconut husk activated carbon used for industrial sewage treatment fully meets the national emission requirements.

2.2 Adsorbent

Several adsorbents have demonstrated success in removing colours from effluent. Numerous natural low-cost adsorbents have been studied to minimize dye concentrations in dilute conditions. Depending on their origin, structural composition, and processing conditions, these substances exhibited distinct surface attributes such as pore volume, size, surface functional groups, surface area, and surface charge (Sultana et al., 2022). Organic waste and livestock manure are economical natural resources that can be employed as synthetic dye adsorbents. They have had excellent results in removing colours from effluents due to the high lignocellulose content of their compounds (Chen et al., 2011; Sen et al., 2011; Wang and Yan, 2011).

As contrasted to other activated carbons, these wastes are renewable, accessible, and economical. They exceed other adsorbents since crop residues are frequently used without or with low-rate processing such as washing, drying, and grinding, cutting manufacturing costs by using cheap raw material and reducing the emission of carbon dioxide associated with heat processing (Franca et al., 2009). Many studies have explored economical adsorbents for colouring degradation from effluent discharge.

2.2.1 Coconut Husk

The separation of coconut fibre from the coconut husk and the manufacturing of final products from the extracted fibre led to an accumulation of coconut husk dust. Coconut husk dust is the darkish, porous, low-weight particle that comes out once the fibre is separated from the husk. Husk dust accounts for approximately 70% of the content of coconut husk (Tejano, 1985). Husk dust is rich in lignins and tannins.

Hasany et al. (2005) studied the potential of CH adsorbent in the elimination of heavy metals such as Cd (II), Cr (III), and Hg (II). Jain et al. (2016) employed CH adsorbent to extract tropaeoline dye, and it was discovered that the activated CH sorbent performed as well as industrial activated carbon (Anderson et al., 2022). However, since pure adsorbents have a poor adsorption performance, numerous treatments and alterations are created to improve their efficiency. Chemical methods of alteration include alkaline treatment, acid treatment, decolorization treatment, and mixing the adsorbent with the other compound. Manju et al significantly increased the adsorption performance of arsenic by adsorbing CH adsorbent with copper (Anderson et al., 2022). CH powder can be used extensively as a possible adsorbent for degrading organic dyes from industrial effluent. (S. Sultana et al., 2022).

2.2.2 Activated Carbon

Activated carbon is a strong yet expensive adsorbent due to its high manufacturing expenses. Activated carbon (AC) is a carbon-containing property such as high pore volume, physicochemical stability, better adsorption capacity, high hardness, significant surface reactivity, and greater surface areas that differs from pure carbon due to the oxidation of carbon molecules on the external and internal surfaces (Rashid et al., 2018). AC is one of the best solutions for treating wastewater because it can adsorb many pollutants from the medium, including dyes, toxic substances, pesticides, and vapours.

The supply of raw materials, processing and treating settings, surface properties, surface tension, porous structure, surface areas, and the contaminants' availability to the adsorbent's internal layer are all factors that can influence adsorption capability (Rashid et al., 2018).

Activated carbon is created by pyrolysis, and chemical activation of sewage sludge by Otero et al. (2021). Research on the advantages of using indigo carmine and phenol adsorbents is carried out in liquid-phase adsorption. They proposed using activated carbons from sewage sludge to separate organic contaminants from water. Dye removal from contaminated water and wastewater can be done using treated sewage sludge, as confirmed through the study. For example, the maximum adsorption capacity (Q_{max}) for indigo carmine dye adsorption was higher than that of crystal violet dye (263.2 mg/g using Atomic Absorption Spectroscopy (AAS), and 184 mg/g using Peganum harmala seeds, PPS). It was significantly lower for indigo carmine dye adsorption (S. Sultana et al., 2022).

Adsorbents can be produced from waste products, particularly those with challenging handling and disposal, for a more sustainable solution, as their use gives a double environmental benefit (Huang et al., 2022; Mohamed et al., 2022). Due to the massive global production and high carbonaceous organic matter content of sewage sludge and biosolids from municipal wastewater treatment facilities (Huang et al., 2022), these materials are used for the pyrolysis-based production of activated carbon (AC) (Mu’azu et al., 2017)

Recently, there has been a rise in interest in the cost-effective treatment of wastewater containing dyes by utilizing activated carbon derived from agricultural and industrial waste as an adsorbent for removing these stubborn organic compounds (Mohan et al., 2014; Regti et al., 2017). According to reports, activated carbon is derived from carbon-rich industrial and agricultural waste such as nutshells, coconut shells, rice husk, peat, wood, coir, lignite, and coal (Aljeboree et al., 2017; Mullick et al., 2018).

Coconut husk activated carbon used in industrial wastewater treatment has a good adsorption effect on complex pollutants. In addition to petroleum and cyanide, there are polycyclic aromatic compounds, aromatic amine compounds, heterocyclic compounds and so on. Certain pollutants in wastewater, such as phenol and cyanide, are very toxic, and can also be effectively treated with the coconut husk activated carbon for industrial wastewater treatment (Liu et al., 2020).

Activation conditions, physical and chemical activities, and the activation method all play a role in determining the quality of AC. In the second step of physical activation, carbonised substrates are activated with oxidising gases such as vapour, carbon dioxide, or their mixes (Okman et al., 2014). This crude sample was combined with chemical catalysts using the chemical technique and simultaneously carbonised at multiple temperatures. In contrast, both existing methods employ conduction or convection to transfer heat to cementite, which wastes a great deal of energy and requires a great deal of time to achieve the appropriate activation level (Hejazifar et al., 2011).

Coconut husk-derived ACH with high porosity is a good adsorbent for wastewater's subsequent degraded F^- ion. This analysis focuses on two essential aspects: synthesising activated carbon from wasted coconut husk and employing the resulting activated carbon as a suitable adsorbent for F^- ion removal from contaminated water (Talat et al., 2018).

M. Sultana et al. (2022) investigated the removal of methylene blue (MB) dyes and phenol with activated carbon adsorbent derived from moderate standard coal.

Various activated carbons were generated from Rawdon coal using the following procedures:

i) direct activation/carbonization of coal; and

ii) coal demineralization prior to activation/carbonization.

iii) coal pre-carbonization before activation/carbonization

iv) coal demineralized and pre-carbonized before activation or carbonization.

These activated carbons were designated AC800, D-AC800, AC500-800, and D-AC500-800, respectively. Following acid treatments involving HCl and HF, the coal was demineralised. At 500 °C, one hour of pre-carbonisation was performed. For activation, samples saturated with KOH were heated in a nitrogen gas flow at 800 °C for 60 minutes. The manufactured adsorbents were characterised by surface area, pore and particle size distributions, FTIR, and XRF investigations. The surface characteristics of AC800, D-AC800, AC500-800, and D-AC500-800 were 1059, 1869, 1385, and 1951 m2 g-1, respectively (M. Sultana et al., 2022). Both demineralisation and pre-carbonisation enhanced the surface area of activated carbon. After demineralisation, the raw coal ash percentage decreased from 28.8% to 0.91%. The demineralised materials lacked mineral characteristics, yet the FTIR spectra confirmed the presence of equivalent organic groups.

Table 2.1 The research focus on adsorbent and adsorbate (Soonmin, 2018)

Adsorbent	**Adsorbate**	**Description**	**Citation**
Coconut shell	Rhodamine-B	Higher surface area (1200 m^2/g) and iodine number (600 mg/g) were observed in the produced activated carbon.	Balasubramani and Sivarajasekar

		The optimal conditions for dye removal are pH, starting dye concentration (150 mg/L), and carbon content (1g).	
Coconut Husk	Maxilon blue GRL and Direct Yellow DY 12	Adsorption is a thermodynamically driven isothermal reaction. The adsorption analysis proves the Fritz-Schlunder model.	Asel and co-workers

2.3 Characterization of Adsorbent

2.3.1 Scanning Electron Microscope (SEM)

The scanning electron microscope is a type of microscope used to visualise the porous structure of a material by scanning a sample with a beam of highly energised electrons and forming an image. The electron beam is focussed by a pair of magnetic condenser lenses in an electron gun, which serves as an electron source. These magnets can alter the course of electrons (M. Sultana et al., 2022). The material was initially deposited in the sample chamber for analysis. The electron beam then impacts the sample and decelerates.

This will produce several indicators, including secondary electrons, backscattered electrons, diffracted backscattered electrons, photons, visible light, and heat. The detectors then collected the secondary electrons, creating images of the object's surface on the monitor. This entire procedure took place in a vacuum chamber. The sample of activated carbon was examined in an SEM to visualise the porous structure, and the magnification was altered to get a more distinct and clear image (Dinh et al., 2019).

2.3.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy, often known as FTIR spectrums or FTIR Scanning, is a method for identifying organic, polymeric, and inorganic compounds. This approach analyses the chemical properties of test samples using infrared light (Debi,2014). The FTIR equipment will transmit between 10,000 and 100 cm^{-1} infrared radiation through a specimen, with some energy being absorbed and the remainder flowing through. Subsequently, the sample molecules convert the received radiation into rotational or vibrational energy.

The resultant indicator at the analyzer is typically displayed as a spectral range between 4000 cm-1 and 400 cm^{-1}, revealing the molecular fingerprint of the material. Because each molecule or chemical composition generates a unique spectral fingerprint, FTIR analysis is an ideal technique for chemical characterization (Debi,2014). A deviation from the typical pattern of absorption bands suggests a change in the composition of the material or the presence of pollutants. If the visual evaluation of a product reveals defects, FTIR microanalysis is often employed to determine its origin.

2.3.3 X-Ray Diffraction Analysis (XRD)

X-ray diffraction analysis (XRD) is utilised in substance research to determine a material's crystallographic structure. The principal role of XRD analysis is to identify compounds based on their diffraction patterns. In addition to identifying phases, XRD exposes how the fundamental structure deviates from the ideal structure due to internal stresses and defects.

Atoms are arranged in crystals, while X-rays are electromagnetic radiation vibrations. Atoms in a crystal diffract transmitted X-rays due mostly to interactions between electrons. This phenomenon is known as elastic scattering, with the electron serving as the scatterer. A collection of scatterers generates a collection of spherical vibrations with the same consistency. Bragg's law states that destructive interference causes these waves to cancel out in most directions, yet they add in a few specific directions.

$$2d\sin\theta = n\lambda \tag{2.1}$$

Where d represents the distance between diffracting planes, θ indicates the incident angle, *n* is an integer and λ indicates the beam's wavelength. The particular directions emerge as reflections on the diffraction pattern. Therefore, X-ray diffraction patterns result from electromagnetic waves striking a regular array of scatterers.

2.4 Adsorbate

2.4.1 Methylene Blue Dye

One of the major challenges with textile wastewater is its colour. The colour of textile industry wastewater, especially that resulting from the dying or printing operations, is unappealing. The relationship between watercolour and how humans perceive water quality is direct. Humans can quickly determine whether a river is poisoned based on its hue (West et al., 2016). Even when the concentration of contaminants in the water is low, textile effluent can have an undesirable hue. Highly coloured dye effluents contaminate sources of groundwater and surface water. About one hundred thousand dyes are on the market now with various chemical structures (Singh et al., 2018).

Anionic, cationic, or non-ionic dyes are identified by the ionic charge on their dye molecules. Cationic dyes are more harmful than anionic colours (Etim et al., 2016). Due to their artificial sources and predominantly reactive compounds, which are biologically non-degradable and possibly toxic, dye effluents are challenging to handle from an environmental perspective. Cationic dyes contain amine-group and have strong solubility in alcohol, typically used along with ethanol to make dye paste. Dyeing can be through an ionic bond between the dye and the negatively-charged component of the fibres, limiting its usefulness because the overwhelming bulk of natural fibres is positively-charged.

Cationic dye methylene blue (MB) is widely used to dye linen, cotton, and satin. Aside from its use in surgical procedures and diagnostic tests, it is also used as a staining agent (Etim et al., 2016). Due to the positive charge being distributed throughout the chromophoric system, basic dyes are cationic. It derives its name from its affinity for basic synthetic products with negatively charged functional groups.

Methylene blue (MB) is an instance of a basic dye that, after brief exposure, is toxic to living creatures (Rashid et al., 2018). Although it is not considered extremely harmful, it can cause aesthetically and environmental problems. MB impeded the penetration of sunlight into water bodies, impeding the photosynthesis of aquatic flora and the oxygen saturation of water reservoirs (Rashid et al., 2018). Aside from the fact that it has a wide range of applications, this dye has several adverse reactions in humans and animals. As it pertains to health, MB has carcinogenic properties that can result in various diseases, including allergic dermatitis and skin irritation. Ingestion of MB through the mouth causes a burning sensation and may result in nausea, vomiting, diarrhoea, and gastritis.

Equally enormous quantities of effluent containing high concentrations of dissolved solids, organics, metals, salts, and stubborn colour result from the use of vast volumes of water in the production of textiles (Ismail and Sakai, 2022) due to the outstanding durability and solubility of synthetic dyes in water, standard treatment strategies are frequently inadequate (Shindhal et al., 2021). Secondary pollution and poor removal of organic load upon discolouration need the adoption of innovative techniques (Samsami et al., 2020).

Table 2.2 Literature Review on Dyes

Title	Research Focus	References
1. Textile wastewater dyes: Toxicity profile and treatment approaches	Presented multiple strategies for addressing textile wastewater and highlighted biological treatments as the preferred method that can be acceptable for sustainable and environment-friendly treatment solutions.	(Mani et al., 2019)
2. **A critical review on recent developments in the low-cost adsorption of dyes from wastewater**	A comprehensive analysis of removal efficiency technologies focuses on identifying effective, economically adsorbents that can replace the present AC (activated carbon).	(Kumara et al., 2019)
3. High surface area and mesoporous activated carbon from KOH-activated dragon fruit peels for methylene blue dye adsorption: Optimization and mechanism study	With a maximum adsorption capacity (q_m) of 195.2 mgg^{-1}, DFPAC was utilised as an excellent adsorbent to degrade MB dye. These results demonstrate the viability of DFP as an economic precursor for producing AC with excellent adsorption affinity for cationic dyes and high surface area.	(Jawad et al., 2021)

2.5 Adsorption Method

The surface of a solid adsorbent captures the adsorbate or ion molecules, whether in a gaseous or liquid condition. Physisorption and chemisorption are the two basic forms of adsorption methods. This classification is determined by how dye molecules or other components are adsorbed onto the surface of the adsorbent. During the adsorption of dye molecules, hydrogen bonding, van der Wall forces, and electrostatic and hydrophobic interactions may be present (Samsami et al., 2020).

Bioremediation requires additional time to eliminate contaminants up to a particular concentration. Due to its scientific feasibility, affordability, commercial applicability over a wide range of contaminants' concentrations, parametric controllability, and simplicity of handling and maintenance, adsorption appears to be an effective treatment technique (Sayen et al., 2018). Adsorption process is also not affected by the potential toxicity in the case of all biological-based processes (Dassharma et al., 2020).

Table 2.3 Brief Explanation of Dye Removal Technology (Ismail & Sakai, 2022)

Process	Description	Advantages	Disadvantages
Coagulation and Flocculation	The effluent is treated with a chemical to coagulate the colour pigments. The coagulated dye is subsequently extracted as sludge.	Low capital and operating expenditures Easy to operate	High sludge production (hazardous waste) Potential of colour re-appearance
Adsorption	Wastewater is passed through an adsorption agent (often activated carbon) to degrade the colour and other contaminants, such as volatile chemicals with a pungent odour and organic debris. Required periodic regeneration.	Low capital and operational cost. High performance and flexibility	Does not degrade the substances. Required continuous maintenance since saturating the adsorbent drastically diminishes its performance.

Table 2.4 Advantages and disadvantages of treatments (Al-Gheethi et al., 2022)

Method of Treatment	**Advantages**	**Disadvantages**
Adsorption and Biosorption	Design simplicity and widespread availability of materials Excellent colour degradation	This procedure requires oversight and maintenance expenses. Enable carbon price

	Excellent and highly dependable rapid filmmaking procedure Selected dyes possess an exceptional affinity for microbial biomass.	Chemical modification to enhance their absorption ability Regeneration concerns
Biological processes	Decolourize a multitude of dye kinds while remaining inexpensive and foam-free. Environmentally friendly and cost-effective Minimal sludge generation Nonhazardous metabolic products	Does not eradicate all dye particles. Low biodegradability of resistant substances The necessary land area is more significant than for other techniques.

2.5.1 Adsorption Capacity

Water contamination mitigation has been examined regarding adsorption kinetics, which is affected by the adsorbate–adsorbent contact and system settings. An adsorption process control unit's mechanism and reaction rate are vital components to evaluate. Kinetic analysis can calculate the solute uptake capacity, indicating the retention taken to finish the adsorption process. When choosing an adsorbent, the adsorption efficiency is critical because the adsorbent must have a high adsorption capability and a rapid adsorption rate. The effectiveness of raw coconut husks as an adsorbent was evaluated using the adsorption kinetics of methylene blue saturation in an aqueous medium. Pseudo-first- and second-order models of the mechanisms' reactions are possible.

The adsorption rate was tracked over time to better comprehend methylene blue (MB) elimination's kinetics. Equilibrium studies used a common concept in this one. The

MB levels in the reaction flasks were determined at the pre-planned time intervals by removing the reaction flask samples. The equation, which is mathematically identical to Eq. 2.2, was used to compute the amount of MB at time t.

$$q_t = \frac{C_o - C_t}{m} V \tag{2.2}$$

2.5.2 Mechanism of adsorption

The interaction between the cell surface and dye-positive ions explains the most straightforward bio-adsorption mechanism for living biomass. The cell surface of live biomass is composed of negatively charged polysaccharides, proteins, and lipids, which accumulate enough positive ions from dyes contained in wastewater (Sahu & Singh, 2019). Including hydroxyl, nitro, and azo groups in the dye molecule increases adsorption, while sulfonic acid groups diminish it. It is now recognized that ion-exchange mechanisms are responsible for microbial biomass's efficiency and selectivity of adsorption (Sahu & Singh, 2019). The figure depicts the interaction between biomass and dye.

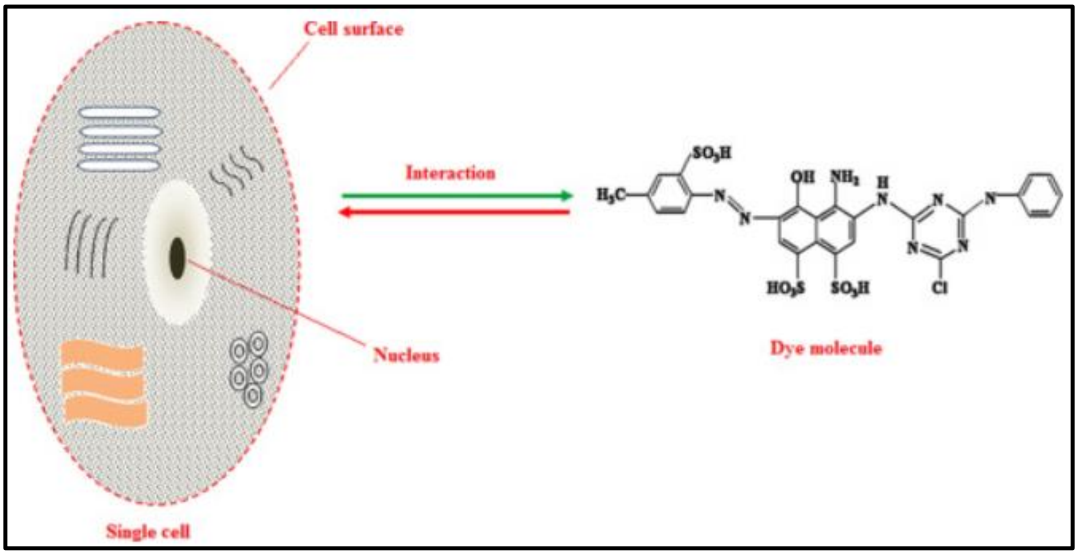


Figure 2.1 Interaction between dyes and adsorbent (Hoc Thang et al., 2021)

2.5.3 PORE SIZES AND SURFACE AREA

The physicochemical and chemical activation approaches are two of the most common ways to make activated carbon. Because of its large surface area and many pores, activated carbon is a good choice for catalysts and adsorbents. As adsorbents, carbon can absorb both colour and odour because the impurities or pollutants are attached to the carbon surface until it is complete. Non-carbon particles including oxygen, hydrogen, sulphides, and other volatiles are extracted from the feedstock during the carbonization process, leaving a carbonized product which is mainly carbon or char. A lack of pore structure means this char has a poor adsorption capability. In contrast, an oxidative treatment is used in the activation step to enhance the char's pore structure. Activated carbon manufacturing procedures that maximize yield while maintaining proper pore characteristics are the most desirable. The yield is most strongly influenced by the activation temperature and duration, and the physical method of generating activated carbon was shown to be less than 17% effective. Activated carbon, a porous material with an extraordinarily large surface area, possesses tremendous interaction potential (Yu et al., 2016) due to its high permeability, adequate pore size distribution, and even functional surface groups. The functional groups of modified activated carbon considerably impact the adsorption mechanisms of this substance (Mondal et al., 2016).

2.6 Parameter

Several factors affect the adsorption process. Dyes removal rates were studied using a variety of factors, including pH, temperature, adsorbent dosage, contact time, and initial dye concentration (Oliveira et al., 2020). When a single dose of adsorbent material is administered, the amount of dyes absorption is measured using the initial dyes concentration as a starting point. The contact time is used to compute the kinetics rate. Temperature is essential in regulating the adsorption process and determining whether it is endothermic or exothermic. When the saturation of adsorbent composites increases, the adsorption rate will rise. When the adsorption proportion rises with temperature, the interaction between the adsorbent and adsorbate is endothermic. However, when the temperature lowers, the relationship is exothermic. It takes longer to adsorb when there are too many places for dye ion binding, and only a few dye ions engage with the adsorbed material. These parameters must be tuned to achieve a significant adsorption rate.

CHAPTER 3

MATERIALS AND METHODS

3.1 Overview

In this study, laboratory experiments were done to assess the effectiveness of activated carbon coconut husk (ACCH) in removing pollutants, colour, and turbidity in an adsorption batch study. pH was also assessed throughout the studies. Before and after adsorption, ACCH is evaluated using Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and X-ray Diffraction (XRD).

After laboratory experiments, the removal performance of the chosen parameters was determined by calculating their percentage of removal. However, for parameters exhibiting negative removal %, desorption tests were undertaken at optimal conditions of 1.0g ACCH, 150 rpm shaking speed, and 30 minutes of contact time. This experiment was undertaken to examine whether contaminants from activated carbon coconut husk (ACCH) are discharged into the stream, affecting removal efficiency. The overall research technique is summarised in Figure 3.1.