A Study on Calcium Alginate Encapsulation of Hibiscus cannabinus L. biochar on Methylene Blue Removal

Nurin Nazifa binti Abu Bakar
J20B0586

A thesis submitted in fulfilment of the requirements for the degree of Bachelor of Applied Science (Materials Technology) with Honours

FACULTY OF BIOENGINEERING AND TECHNOLOGY
UMK

2024

DECLARATION

I declare that this thesis entitled "A Study on Calcium Alginate Encapsulation of Hibiscus cannanibus L. biochar on Methylene Blue Removal" is the results of my own research except as cited in the references.

Signature : ______________________________

Student's Name : Nurin Nazifa Binti Abu Bakar

Date : ______________________________

Verified by:

Signature : ______________________________

Supervisor's Name : Dr. Nur Nabilah Binti Shahidan

Stamp : ______________________________

Date : ______________________________

ACKNOWLEDGEMENT

Alhamdulillah, praise to Allah, I am very grateful to be able to complete my final year project. I would want to express my gratitude to everyone whose help made this project possible. I want to sincerely thank my supervisor for the support. Dr. Nur Nabilah Binti Shahidan and for the insight, constructive comments and advice in discussion of my project which greatly assisted me with the completion of this research.

My sincere thanks also go to all of the lecturers from Department of Bioengineering and Technology for their teaching and guidance over these past four years in University Malaysia Kelantan (UMK). A special thanks to Ts. Dr. Nor Hakmin Bin Abdullah even though I am not mentoring by him but as an academic adviser he offered at the beginning of my research journey had put an impact to my research.

My time doing experiment was productive and overwhelming with laboratory assistants from all three faculties, Faculty of Bioengineering and Technology (FBKT), Faculty of Earth Science (FSB) and Faculty of Agro-Based Industry (FIAT) especially Ts. Hanisah Izati Binti Adli that kindly assisted me with the experimental and computational facilities provided by the UMK. Most of the results described in this research would not be obtained without their helps.

I also wish to show my thankfulness to my friends, Mohd Faizal Bin Abd Majid and Nurul Asyiqin Binti Ahmad Hisham who willingly and selflessly devoted so much time in helping me to run my samples and with all the related submission of this project before deadlines.

I also want to express my sincere gratitude to all of my classmates, my SEB program partners, and my friends who are spread out around the globe for your support and encouragement during this entire research process. The connections we made through all these years during our degree have enriched my life and I look forward to continuing our relationships.

I also want to express my gratitude to both of my parents for their unwavering love and genuine support, which has been greatly helpful to me. They are the reason I am here. Last but not least, I wish to acknowledge the huge support and love from my family members that kept me going and lifting me uphill during thisphase. Without all of these precious supports, it would be very difficult for me to finish this project.

A Study on Calcium Alginate Encapsulation of Hibiscuscannanibus L. biochar on Methylene Blue Removal

ABSTRACT

This thesis contains analysis of the manufacturing and application of calcium alginate-encapsulated biochar made from Hibiscus cannabinus L. for the purpose of the removal of Methylene blue, a dye that is widely used in a variety of industries. In order to improve the durability and effectiveness of the biochar in water treatment applications, the study analyses the encapsulation procedure. The study focuses on optimizing key encapsulation parameters, including temperature of carbonization, size of biochar particles, dosage of biochar, pH of Methylene Blue solution, and agitation speed. To improve stability and adsorption efficiency, the encapsulation process is methodically adjusted, which aids in the creation of an efficient and sustainable water treatment solution. The objectives of this study were to prepare calcium alginate kenaf core biochar beads, (CAKCB) to obtain the optimum condition of CAKCB beads in methylene blue removal and to characterize CAKCB beads using UV-visible spectrophotometer, and Fourier-Transform Infrared (FTIR) before and after the methylene blue removal process. Consequently, by optimizing the parameters, batch experiments were conducted to determine the ideal adsorption conditions of the kenaf core biochar coated with calcium alginate gel. The calcium alginate beads were created by the cross-linking method while the kenaf core was carbonized to generate KC biochar. The optimum conditions obtained for highest methylene blue removal were at temperature, size, and dosage of 700 º C, 45 µm and 1.0 g respectively. The percentages of dye removal also increase with increasing of pH and agitation speed. Results indicate that optimal encapsulation parameters significantly improve the adsorption performance of the biochar, establishing it as a promising adsorbent for methylene blue removal. This shown that kenaf core biochar is an excellent adsorbent for the removal of dyes, able to boost kenaf usage while also assisting in the reduction of water pollution issues, improving both the general health of people and the quality of the water.

Keywords: Kenaf core, methylene blue, adsorption, wastewater treatment

Kajian mengenai Penyelaputan Kalsium Alginat bagi biocar Hibiscus cannabinus L. dalam Penyingkiran Methylene Blue

ABSTRAK

Tesis ini mengandungi analisis menyeluruh tentang pembuatan dan penggunaan biochar berkapsul kalsium alginat yang diperbuat daripada Hibiscus cannabinus L. untuk tujuan penyingkiran metilena biru, pewarna yang digunakan secara meluas dalam pelbagai industri. Untuk meningkatkan ketahanan dan keberkesanan biochar dalam aplikasi rawatan air, kajian menganalisis prosedur enkapsulasi. Kajian ini memberi tumpuan kepada mengoptimumkan parameter pengkapsulan utama, termasuk suhu pengkarbonan, saiz zarah biochar, dos biochar, pH larutan metilena biru, dan kelajuan pengadukan. Untuk meningkatkan kestabilan dan kecekapan penjerapan, proses pengkapsulan dilaraskan secara kaedah, yang membantu dalam penciptaan penyelesaian rawatan air yang cekap dan mampan. Objektif kajian ini adalah untuk menyediakan pengkapsulan kalsium alginat bagi manik biochar teras kenaf (CAKCB), untuk mendapatkan keadaan optimum manik CAKCB dalam penyingkiran metilena biru dan untuk mencirikan manik CAKCB menggunakan spektrofotometer UV-visible, dan Fourier-Transform Infrared (FTIR) sebelum dan selepas proses penyingkiran metilena biru. Akibatnya, dengan mengoptimumkan parameter, eksperimen kelompok telah dijalankan untuk menentukan keadaan penjerapan yang ideal bagi biochar teras kenaf yang disalut dengan gel kalsium alginat. Manik-manik kalsium alginat dicipta melalui kaedah penghubung silang manakala teras kenaf dikarbonkan untuk menghasilkan biochar KC. Keadaan optimum yang diperolehi untuk penyingkiran metilena biru tertinggi adalah pada suhu, saiz, dan dos 700 º C, 45 µm dan 1.0 g masing-masing. Peratusan penyingkiran pewarna juga meningkat dengan peningkatan pH dan kelajuan pengadukan. Keputusan menunjukkan bahawa parameter enkapsulasi optimum meningkatkan prestasi penjerapan biochar dengan ketara, menjadikannya sebagai penjerap yang menjanjikan untuk penyingkiran metilena biru. Ini menunjukkan bahawa biochar teras kenaf adalah penjerap yang sangat baik untuk penyingkiran pewarna, mampu meningkatkan penggunaan kenaf di samping membantu dalam pengurangan isu pencemaran air, meningkatkan kesihatan umum dan kualiti air.

Kata kunci: Inti kenaf, metilena biru, penjerapan, rawatan air sisa

TABLE OF CONTENTS

LIST OF FIGURES

CHAPTER 1

INTRODUCTION

1.1 Background of study

Wastewater pollution has long been recognized as an enduring issue and the treatment of wastewater has become increasingly important. Metal pollutant in water is one of the main causes of pollutant in water. Industries involved in metal mining, metal processing, electroplating, manufacturing, and other metal-related activities can release metal pollutants into water bodies through their wastewater discharges. Another one of the main pollutants found in wastewater are dyes used in industries such as textiles, leather, and paper. Methylene blue is toxic to aquatic organisms and can cause severeharm to the environment. Consequently, it is essential to create effective and sustainable methods for its removal from wastewater. Therefore, numerous research has been carried out to address the issue of unattractive colouring in natural bodies of water. It has been documented that the use of activated carbon for adsorption, as well as membrane filtration, biological treatment and chemical coagulation and flocculation, are successful methods for eliminating pollutants from wastewater.

The use of adsorbents such as activated carbon and biochar has shown promising results for removing contaminants from wastewater, particularly for organic and inorganic pollutants (Cong Nguyen et al., 2021). Wastewater treatment is a critical process that aims to remove pollutants and contaminants from water to make it safe for discharge into the environment or reuse. However, traditional wastewater treatment methods can be expensive, energy intensive, and generate significant amounts of waste. As a result, interest is rising in exploring alternative and sustainable solutions, such as the use of biochar for wastewater treatment. Biochar has a high surface area and porosity, which allows it to adsorb and trap various contaminants present in wastewater. As indicated by its growing usage in many environmental applications, biochar, a substance that is environmentally beneficial and reasonably priced and is often made from organic wastes such as municipal wastes, forestry leftovers, and agricultural wastes, has gained increasing interest.

1.2 Problem statement

Wastewater treatment has been a critical issue that society has been grappling with for a considerable time, with increasing concerns about water pollution and the impact on human health and the environment. One major challenge in wastewater treatment is the removal of coloured pollutants such as methylene blue, which can have harmful effects on aquatic ecosystems and public health. Kenaf core biochar is a potential candidate to use as an adsorbent for wastewater treatment. It is a renewable and sustainable material that canbe produced from agricultural waste and has been shown to be effective in removing pollutants from wastewater.

Wastewater treatment is a critical process that aims to remove pollutants and contaminants from water to make it safe to discharge into the environment or reuse. However, traditional wastewater treatment methods can be expensive, energy-intensive, and generate significant amounts of waste. As a result, there is a growing interest in exploring alternative and sustainable solutions, such as the use of biochar for wastewater treatment. *Kenaf Hibiscus cannabinus L.* is a fast growing, high yielding plant that has been shown to produce a high-quality biochar from its stems. Kenaf biochar has been demonstrated to be an effective adsorbent for a wide range of contaminants, including heavy metals, organic compounds, and nutrients, making it a promising material for wastewater treatment applications. In this study, kenaf stem core parts will be used as a biochar for water treatment.

1.3 Objectives

i. To prepare the Kenaf core biochar (KCB) to achieve maximum efficiency in removing methylene blue by different parameters.
ii. To prepare sodium alginate encapsulation of kenaf core biochar KCB beads.
iii. To characterize KCB using UV-visible spectrophotometer, SEM and FTIR before and after the methylene blue removal process.

1.4 Scope of study

This research aims to prepare calcium alginate encapsulated kenaf stem core biochar and utilize it as an adsorbent to remove methylene blue dye from aqueous solutions. The study will focus on preparing Kenaf core biochar and encapsulating it with calcium alginate to enhance its adsorption capacity. The effects of various parameters such as time, initial methylene blue concentration, pH, and the dosage of the adsorbent on the removal efficiency will be evaluated

1.5 Significant of study

The ultimate outcome of this research was associated with the aim of enhancing the constraints of using kenaf core biochar as an adsorbent. A new adsorption technique was developed to improve the current system by trapping kenaf core biochar in a polymer made from sodium alginate. This technique was designed to increase the capacity for removing methylene blue from wastewater that was discolored or colored.

CHAPTER 2

LITERATURE REVIEW

2.1 Wastewater

Water contamination is caused by a variety of toxins, primarily released by industrial and agricultural operations. It is including harmful elements such as lead, mercury, arsenic, cadmium, chromium, copper, and iron, which, if not properly treated, can contaminate water bodies and adversely affect ecosystems and human health. Untreated coloured wastewater discharge into bodies of water can cause serious aesthetic and ecological issues since it changes the way things seem and interferes with the aquatic ecosystems' ability to maintain their natural balance. Additionally, certain industrial colorants may include poisonous substances that might endanger both aquatic life and people if they get into drinking water supplies or are eaten by humans through the food chain. Sharma reported some other treatments for wastewater have been found such as Electrocoagulation Treatment of Electroplating Wastewater. The electrocoagulation one of the most innovative and promising techniques technologies to treat different types of wastewaters including organics and inorganics molecules contained in a variety of industrial effluents (Sharma et al., 2020). It can efficiently remove heavy metals, such as chromium, copper, nickel, zinc, and cadmium, which are commonly found in electroplating effluents. Other treatments that have been studied were biological treatment and oxidation processes. There is also colored wastewater, which is released by dyeing businesses like textile manufacturers and is rich in harmful chemicals. The presence of these colored compounds in wastewater can have environmental and health impacts, as they can reduce the transparency of water bodies, interfere with photosynthesis and aquatic life, and affect the quality of drinking water.

In accordance with the recent survey, there have been 100,000 commercially available dyes used all over the globe with a record of 10,000 tons per year of consumption (Mary Ealias & Saravanakumar, 2019). This is to ensure they can stay longer and exhibit brighter color, which is why many textile businesses favour synthetic colouring agents over natural

coloring. In this study, the adsorption of dye treatment is a propose to remove a dye that contains in wastewater.

2.2 Adsorption

The process by which contaminants present in water, such as organic pollutants, heavy metals, and nutrients, are eliminated or decreased by their attachment to the surface of biochar materials is known as adsorption in biochar wastewater treatment. As far as dye removal from water is concerned, adsorption has been found as one of the best treatment processes among other conventional water treatment methods due to its low-cost, affordability, greater efficiency, and the fact that it requires minimum maintenance (Sirajudheen et al., 2020)

Adsorption techniques involve using materials like activated carbon, zeolites, or other specially designed adsorbents to attract and trap metal ions from the wastewater. In wastewater treatment, biochar is used as an adsorbent due to its porous structure and high surface area. The adsorption process occurs when the contaminants in the water come into contact with the biochar surface, and they adhere to the biochar through various mechanisms such as chemical activation, surface functionalization, or impregnation with specific compounds. These modifications can increase the affinity of biochar towards certain contaminants and improve its adsorption efficiency works of mechanism.

2.3 Biochar

Biochar is a high carbon content material produced by thermochemical treatment of organic materials under the absence of oxygen condition (Banitalebi et al., 2019) Biochar is a substance resembling charcoal, produced through the controlled process of pyrolysis, wherein organic material derived from agricultural and forestry wastes (commonly known as biomass) is burned. Biochar is generated through a distinct method to minimize pollution and securely sequester carbon, even though it bears a striking resemblance to regular charcoal. In this process, known as pyrolysis, organic materials like wood chips, leaf litter, or deceased plants are burned within a controlled environment with minimal oxygen. This controlled combustion results in the release of no harmful contaminants. Some studies have found various materials that can be used as biochar, such as rice husk. Other organic materials, including agricultural residues like corn stalks, wheat straw, sugarcane bagasse, and coconut shells, have also been explored as viable sources for biochar production. Additionally, food processing waste like fruit peels and nut shells, as well as forestry waste like sawdust and wood chips, have showed potential as substitute feedstocks for the manufacture of biochar. Kenaf biochar is produced by carbonizing kenaf core at 1000 °C in an oven. The value range of the pyrolysis temperature for kenaf from 300 to 600°C (Harussani & Sapuan, 2022)

Kenaf stem biochar can be a valuable material for various applications. Biochar, including kenaf stem biochar, is a form of charcoal produced through the pyrolysis or heating of biomass, such as agricultural waste or plant materials. It is known for its porous structure and high carbon content, which give it unique properties and make it useful in different fields. Kenaf waste hasthe potential for various uses and applications, including the stalks and leaves, can be used as a biomass feedstock for energy production through processes such as combustion and gasification. Kenaf stalks and leaves also serve as supplementary feed for livestock due to their nutritional content. For biochar, kenaf stem core can be used for water waste treatment to remove any contaminants in water such as heavy metal or dye.

2.4 Kenaf *Hibiscus cannabinus L.*

Encapsulation is a process or technique of enclosing or encapsulating something within a protective or containment structure. It involves enclosing an object, substance, or component within a barrier or shell to isolate it from its surroundings or to provide protection from external factors such as physical damage, moisture, temperature fluctuations, or chemical reactions. Kenaf stems core, a fibrous agricultural waste material, offer a promising option for encapsulating biochar. Kenaf stems core are abundant, renewable, and low-cost resources that can be processed to obtain fibrous structures suitable for encapsulation purposes. By encapsulating biochar within the kenaf stem fibers, the resulting composite material can exhibit improved handling and controlled release properties.

Figure 2.1: *Kenaf Hibiscus cannabinus*

2.5 Dye

A dye refers to a pigmented substance employed to add color to different materials, including textiles, paper, plastics, or leather. Dyes are typically soluble in the medium in which they are applied, allowing them to bind with the material, resulting in a permanent or semi-permanent coloration. Dyes can originate from natural sources, such as plants, animals, or minerals, or they can be synthesized using chemical processes. Wastewater containing dyes can be discharged into water bodies by businesses engaged in the manufacture of textiles, the production of dyes, printing, and other dye industries. These discharges have the potential to pollute the aquatic environment with various colours and other chemicals if they are not correctly handled. A few examples of synthetic dyes are aniline blue, alcian blue, basic fuchsin, methylene blue, crystal violet, toluidine blue, and congo red. In this study the dye that's going to be use is a methylene blue.

2.5.1 Methylene blue

The pollution of water bodies by untreated MB dye effluents discharged from industries has been recently associated with the shortage of clean water in the society (Walker et al., 2019) Under typical environmental circumstances, the synthetic dye methylene blue is reasonably long-lasting and resistant to breakdown. This implies that it has the ability to stay in water bodies for quite a bit of time, potentially contaminating the water. the intense blue coloration of methylene blue can impact the aesthetic value of water bodies. There can be potential health risks to humans as well. Direct exposure to high concentrations of methylene blue in water of contaminated water may cause irritation, allergies, or other adverse effects.

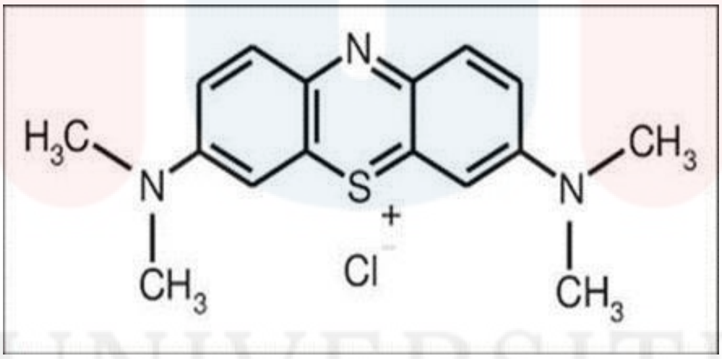

Figure 2.2: Microstructure of Methylene Blue

2.6 Encapsulation

Encapsulation is a technique or method for enclosing or encapsulating anything inside of a containment or protective structure. This enhances the stability and longevity of the biochar material, allowing it to retain its adsorption capacity fora longer period. here are several materials that have been researched to make encapsulations such as gelation approach and silica gel. Using gelatin as an encapsulating material can make the final formulation cost-effective because gelatin is cheap, readily available, and can be solubilized easily in water (Ci et al., 2015) Gelatin is a protein-based material derived fromcollagen. The application of a different formulated encapsulating product consisting of an aqueous silica and acidic polymer solution, that the resulting amorphous silica material which is the by-product of the treatment process does not exhibit the characteristics of the waste material (Akpoveta, 2020). For this research, calcium alginate will be used as a material for encapsulation. The encapsulation of biochar within calcium alginate beads provides, a protective barrier around the biochar particles, preventing their disintegration and loss during wastewater treatment.

2.7 Calcium alginate

Calcium alginate is a gel-like substance derived from the polysaccharide alginate, which is extracted from brown seaweeds. It is composed of long chains of repeating units of mannuronic acid and guluronic acid. Calcium alginate acts as a matrix or binder that encapsulates the biochar particles. Materials based on biochar may benefit from controlled release features provided by calcium alginate encapsulation. The gel matrix can control how quickly nutrients, organic compounds, or active ingredients in the biochar are released, enabling sustained. Calcium alginate, a biopolymer derived from algae or bacteria, which has been widely used for encapsulation purposes. It is biocompatibility, nontoxic and ability to form gel like structures.

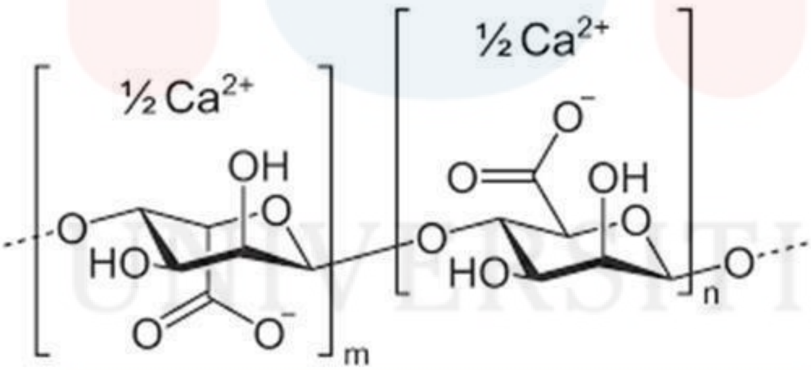

Figure 2.3: Microstructure of Calcium Alginate

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

Calcium alginate powder was purchased from the HmBG Chemicals. While methylene blue powder of 319.865 g/mol was purchased from HmBG Chemicals. Other chemicals are hydrochloric acid (HCl), calcium chloride and Sodium hydroxide (NaOH) was used as an activating agent in determining the characterization of carbon. Distilled water is also used to remove any impurities. The stem core of Kenaf (*Hibiscus cannabinus L.*) as a biochar material from Kenaf And Tobacco State (Lembaga Kenaf Dan Tembakau Negara Negeri Kelantan).

3.2 Preparation of kenaf stems core biochar

The biochar was prepared using kenaf stems that were collected from Kenaf And Tobacco State (Lembaga Kenaf Dan Tembakau Negara Negeri Kelantan) in Kubang Kerian, Kelantan. Only the core was collected. The dried kenaf stem core was then processed through sieved using a various size of sieve such as 250 μm 125 μm, 63 μm and 45 μm to create a fine powder desired mesh size small piece.

The kenaf mass loss was assigned to three main stages drying and evaporation of light particles, happened at temperatures below 150°C, (2) started outgassing from 150 to 375 °C, and (3) decomposition of lignin, at temperatures above 400 °C and a 1:1 sodium hydroxide NaOH ratio. The mass of the kenaf stem core powder was taken before and after the oven drying to calculate the percentage of the moisture removal using equation.

Under the following circumstances 550 °C of pyrolysis temperature, 180 minutes of heating duration, and a 1:1 NaOH ratio. The difference parameters will affect how biochar will be activated. The parameters were pyrolysis temperature, pyrolysis time, and the impregnation ratio of NaOH:KF. The yield, removal efficiency, and adsorption capacity of kenaf-based biochar were calculated according to equations:

$$\text{Biochar Yield\%} = wb/wrX100 \quad \text{Equation 3.1}$$

$$Removal\ Efficiency\ \% = Ci - CeX100 \quad \text{Equation 3.2}$$

3.3 Beads preparation

Codium alginate powder (5g) was put into a beaker containing 200 mL deionized water to obtain a sodium alginate solution and mixed until they became a homogenous mixture using a magnetic stirrer and a hot plate. Then, 20 mL of the mixture was added drop by drop into 2 wt% of NaOH solution while stirring slowly. The filtered beads were washed with distilled water and stored inside a falcon tube at 5 °C until further use. The initial ion concentration and solution temperature were increased to increase the adsorption capability. The use of iron oxide to give biochar a property allowed for straightforward separation and regeneration using an external magnet. The maximum percentage elimination of methylene blue to be 95% at a concentration of 50 mg/L.

3.4 Preparation of methylene blue (MB) calibration curve

A few series of methylene blue concentrations are created from the methylene blue standard stock solution using the dilution technique in order to create the calibration curve using dilution equation

$$M_1 V_1 = M_2 V_2 \qquad \text{Equation 3.4}$$

The methylene blue solution of concentration 2, 4, 6, and 8 mg/L were prepared. the MB were prepared into different pH which regulated using 0.1 M of HCl (hydrochloric acid) and 0.1 M NaOH (Sodium hydroxide) solution into pH of 4, 7 and 9.

3.5 Kenaf core biochar size

The core or pith of the kenaf stem typically consists of a softer, spongy material surrounded by the fibrous outer layer. When the kenaf stems are subjected to pyrolysis, a thermochemical process involving the heating of organic materials in the absence of oxygen, biochar is produced. The biochar derived specifically from the core of the kenaf stem is referred to as kenaf core biochar

Kenaf core biochar was sieved using siever into four different sizes instead which were from 250 μm to 45 μm. The following parameter research employed the size of the kenaf core that provided the maximum percentage of methylene blue elimination.

3.6 Characterization

In the context of using biochar kenaf stem encapsulation for water treatment, characterization involves assessing and comprehending the composite material's physical, chemical, and structural composites. Characterization techniques are employed to assess the quality, performance, and suitability of the encapsulated biochar and kenaf stem core composite for water treatment applications. In this study, UV-Vis, SEM, and FTIR are going to use for characterization.

3.6.1 Ultraviolet-visible (UV-Vis) spectrophotometry

Ultraviolet-visible (UV-Vis) spectrophotometry is a commonly used analytical technique that measures the absorption of ultraviolet and visible light by a sample. UV-Vis spectroscopy can be used to characterize the optical properties of biochar. By measuring the absorbance spectrum of biochar, information about its light absorption behavior and potential interaction with pollutants can be obtained. This characterization helps understand the intrinsic properties of biochar before its encapsulation. UV-Vis spectrophotometry can determine the encapsulation efficiency of biochar within encapsulating material.

3.6.2 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is another technique that valuable in studying the encapsulation of biochar for wastewater treatment. SEM allows for high resolution image of the surface morphology of biochar and encapsulating materials. It provides detailed information about the structure, texture, and porosity of the encapsulated biochar. SEM can be used to evaluate the integrity and uniformity of the encapsulating material surrounding the biochar. It can reveal any cracks, voids, or defects in the encapsulation layer, which may impact the performance and stability of the encapsulated biochar. (Liu et al., 2019) reported that ash promotes agglomeration of biochar.

3.6.3 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy is particularly useful for identifying and analyzing the functional groups present in biochar and the encapsulating material. The technique provides information about the chemical bonds and molecular structure of the materials. FTIR can help identify surface modifications or chemical transformations in the encapsulated biochar. By comparing the FTIR of the encapsulated biochar before and after wastewater treatment, any changes in absorption bands or peak can indicate the interaction or reaction between the encapsulated biochar and pollutants. FTIR spectroscopy can monitor chemical changes occurring during the encapsulation process, such as the presence of cross-linking agents, polymerization reactions, or chemical modifications of the encapsulating material. This information helps to understand the encapsulation mechanisms and the encapsulation process.

3.7 RESEARCH FLOWCHART

3.7.1 Research Flow

The research flow chart will be divided into three stages. Stage 1 is material preparation and stage 3 is for analysis, evaluation and comparison of the obtained experimental data as shown in Figure 4.1.

S

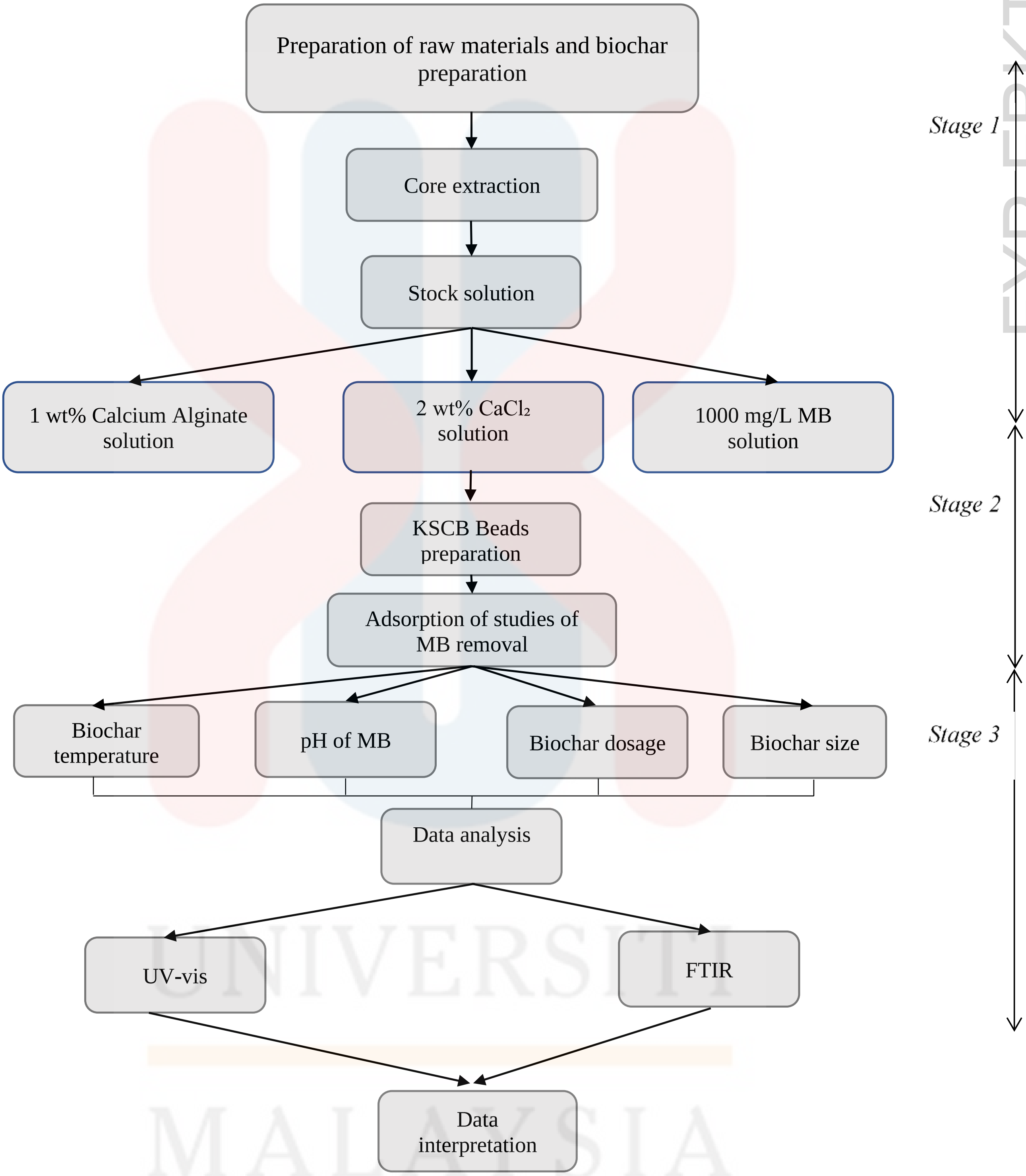

Figure 4.1: Research flowchart for *Kenaf (Hibiscus cannabinus L.)*

CHAPTER 4

RESULTS AND DISCUSSION

No of Sample	Name	MB Removal (%)
1	400°C/250µm/0.5g/100rpm/10mlMB/1wt%SA	87.90
2	400°C/125µm/0.5g/100rpm/10mlMB/1wt%SA	88.32
3	400°C/63µm/0.5g/100rpm/10mlMB/1wt%SA	96.19
4	400°C/45µm/0.5g/100rpm/10mlMB/1wt%SA	98.63
5	500°C/250µm/0.5g/100rpm/10mlMB/1wt%SA	96.37
6	500°C/125µm/0.5g/100rpm/10mlMB/1wt%SA	97.64
7	500°C/63µm/0.5g/100rpm/10mlMB/1wt%SA	98.13
8	500°C/45µm/0.5g/100rpm/10mlMB/1wt%SA	98.72
9	600°C/250µm/0.5g/100rpm/10mlMB/1wt%SA	98.31
10	600°C/125µm/0.5g/100rpm/10mlMB/1wt%SA	98.61
11	600°C/63µm/0.5g/100rpm/10mlMB/1wt%SA	98.72
12	600°C/45µm/0.5g/100rpm/10mlMB/1wt%SA	98.97
13	700°C/250µm/0.5g/100rpm/10mlMB/1wt%SA	98.91
14	700°C/125µm/0.5g/100rpm/10mlMB/1wt%SA	98.96
15	700°C/63µm/0.5g/100rpm/10mlMB/1wt%SA	99.10
16	700°C/45µm/0.5g/100rpm/10mlMB/1wt%SA	99.43
17	700°C/45µm/1g/100rpm/10mlMB/1wt%SA	99.70
18	700°C/45µm/0.5g/0rpm/10mlMB/1wt%SA	69.09
19	700°C/45µm/0.5g/50rpm/10mlMB/1wt%SA	73.08
20	700°C/45µm/1g/100rpm/pH9/10mlMB/1wt%SA	99.80
21	700°C/45µm/0.5g/100rpm/pH7/10mlMB/1wt%SA	99.43
22	700°C/45µm/0.5g/100rpm/pH4/10mlMB/1wt%SA	95.83

4.1 Calibration curve of methylene blue

By diluting the methylene blue stock dilution with distilled water, a range of methylene blue concentrations, from 2 to 10 mg/L, was created. The absorbance reading of the methylene blue concentrations was then obtained at a wavelength of 665 nm using a UV-vis spectrophotometer, as shown in Figure 4.1.

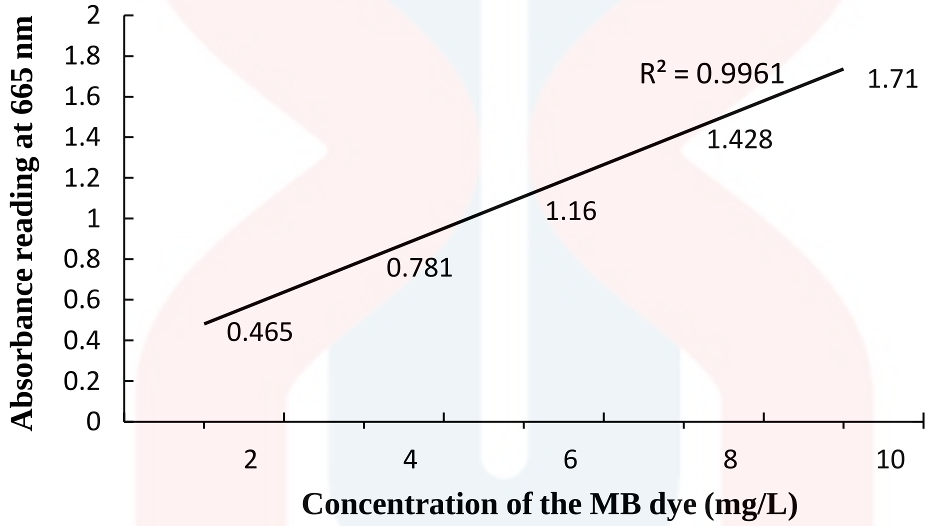

Figure 4.1: Concentration of the MB dye (mg/L)

The graph's coefficient of determination, or R-squared (R2), was close to 1, at 0.9961, indicating that the calibration curve is suitable and the standard curve used for this work was y = 0.00845x + 0.1002. As Figure 4.1 shows, the concentration of the methylene blue solution also increased along with its color intensity.

Figure 4.2: The observation of colour changes in methylene blue solution at different concentration. Cuvette arrangements from left to right are methylene blue at concentration of 2, 4, 6, 8 and 10 mg/L.

4.2 Parameter effect on the methylene blue adsorption

4.2.1 Effect carbonization temperature of kenaf core biochar

Figure 4.3 show how different carbonization temperatures affect the adsorption of methylene blue. Using a furnace, the carbonization process was set for one hour at a steady heating rate of 10 ºC per minute at each temperature.

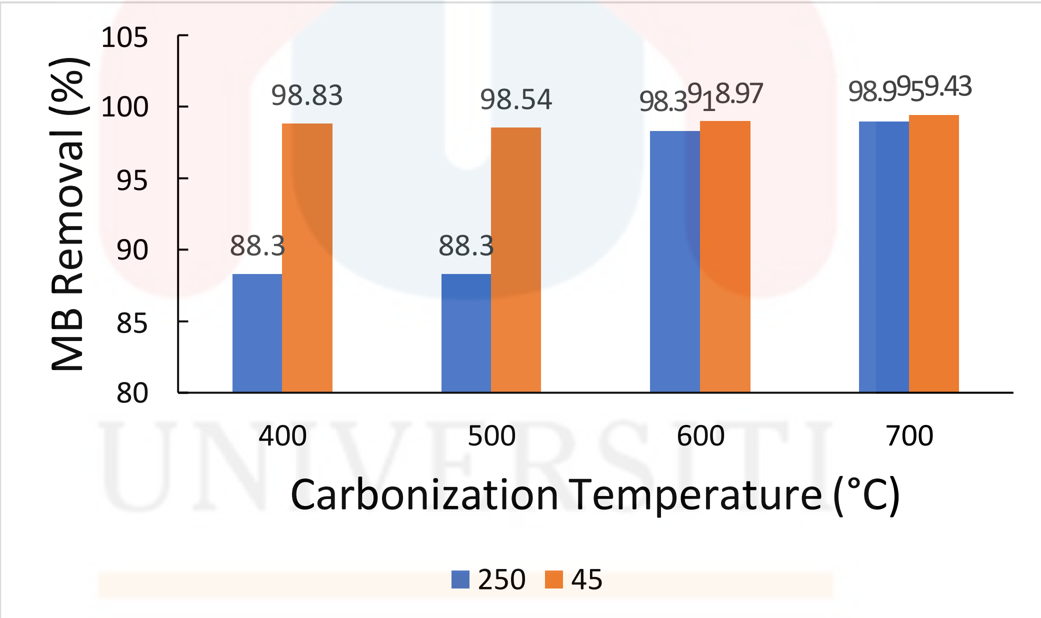

Figure 4.3 Effect of carbonization temperature on removal of MB dye using KC biochar of 250and 45 µm on 50 mL of MB solution of concentration 10mg/L after 90minutes.

Figure 4.3 shows the result of KC biochar carbonization temperature on the removal of MB dyes. The graph shows that as the temperature of carbonization rises from 400 to 700 ºC.

The MB removal percentage also increased from 88.30% to 98.95% and from 98.83% to 99.43% for KC biochar size 250 and 45 µm respectively. This indicated that the KC biochar that undergo carbonization at 700ºC had the maximum efficacy in removing MB dye for both sizes. This is a result of the high heat produced during the carbonization process affecting the KC biochar's microstructure. The KC biochar's cell walls shattered, shrank, and then regenerated at high temperatures. Additionally, the components of kenaf core 20.1% hemicellulose, 57.2% cellulose, and 77.3% of holocellulose will all decomposition more quickly at higher temperatures during the carbonization process. This is because cellulose is able to break down into a stable a hydrocellulose at lower temperatures, which is useful in the making of biochar. Conversely, cellulose depolymerizes at high temperatures and produces volatile materials that are released during carbonization. The surface of the biochar grew rougher and more porous as the pyrolysis temperature high. The organic component of pyrolyzed kenaf at high temperatures vanished owing to devolatilization/decomposition, resulting in pyrolyzed kenaf with a rough surface (Saba et al., 2015)

The calcium alginate beads were successfully encapsulated KC biochar using a crosslinking, straightforward and cost-effective method. The following is the procedure to prepare for 600c/250u/0.5g/100rpm/1% where 600c carbonization blend of 1 wt% Na-alg solution and KC biochar into a 1 wt% $CaCl_2$ solution, circular calcium alginate beads were promptly and instantly produced. Because calcium ions (Ca2+) bonded to alginate polymer and replaced sodium ions (Na+), the shape was able to be preserved. The gelation process is complete and the gel shells thicken after all of the Ca2+ ions within a gel bead have been used up (Zeeb et al., 2015). In addition to maintaining a high viability, this calcium alginate gel coating can protect the KC biochar from damage from the environment so that it won't break easily when high agitationspeed is used.

The effects of varying carbonization temperatures on kenaf core biochar (CAKCB) beads encapsulated in calcium alginate are also being studied. The size of the KC biochar applied to make the beads was 125µm. When the temperature rises in contrast to the control, KC biochar, the same pattern of methylene elimination is seen.

According to the graph in Figure 4.5, after two hours, the percentage of MB removal from CAKCB beads at 700 °C carbonization temperature was its highest at 99.27%; the percentages for carbonization temperatures of 600 °C and 500 °C were 92.81% and 91.78%, respectively. On the other hand, only 22.42% of the methylene blue was removed from the control calcium alginate (CA) beads without the KC biochar or blank beads.

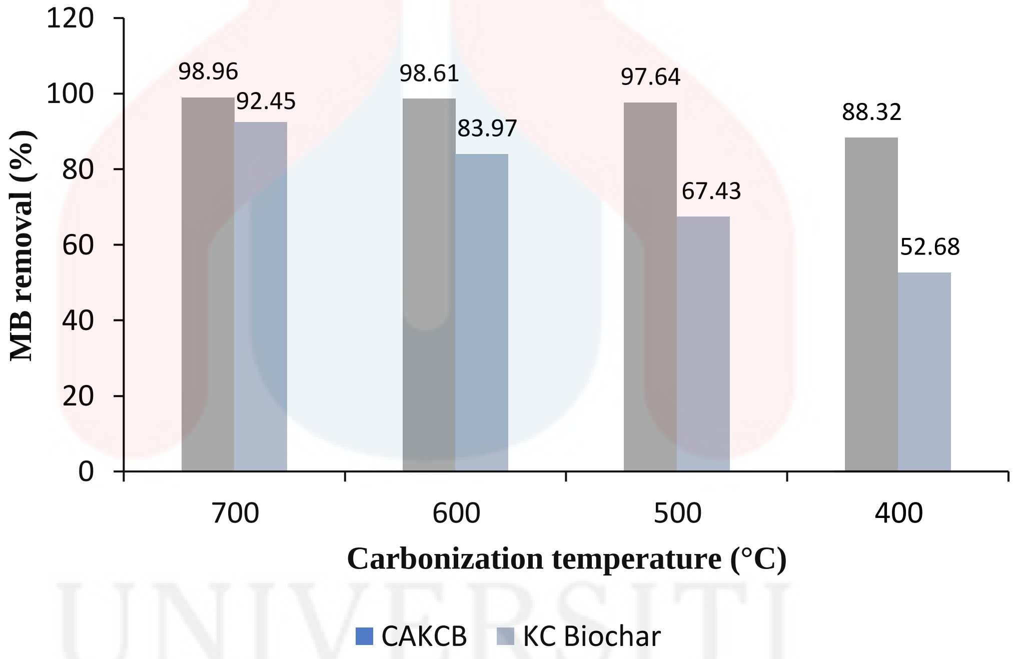

Figure 4.4: The effect of different carbonization temperature of the CAKCB on 50 mL of MB solution of concentration 10mg/L after 2 hours in comparison with the control parameters.

After two hours, the trapped methylene blue dyes inside the CA beads caused the beads to change color from colorless (Figure 4.6(a) to blue (Figure 4.6(b), indicating that adsorption had taken place. Therefore, it is anticipated that the adsorption performance will enhance when alginate and KC biochar are combined. In addition to that, they are simpler to work with and recycle than charcoal.

(a) (b) (c)

Figure 4.5: The prepared calcium alginate beads: (a) Blank calcium alginate (CA) beads, (b) CA beads after treated with methylene blue solution, (c) The successfully prepared CAKCB beads.

Based on Figure 4.6 (a), (b), (c), the control parameters that used KC biochar at various carbonization temperatures show the level to which the mixed methylene blue solution turned black. In the meantime, the color shifts in Figure 4.7(d), (e), (f), and (g), where all of these were done with calcium alginate beads, are simpler to see with the eye on its own. This proved the benefit of encasing the biochar in calcium alginate gels, which allow for simple handling and post-use filtering of the beads. Because of this, when KC biochar is used, absorbance readings can be collected more quickly and precisely at every point in time rather than requiring centrifugation, filtration, and a lengthy waiting period for the biochar to dry out to the bottom surface.

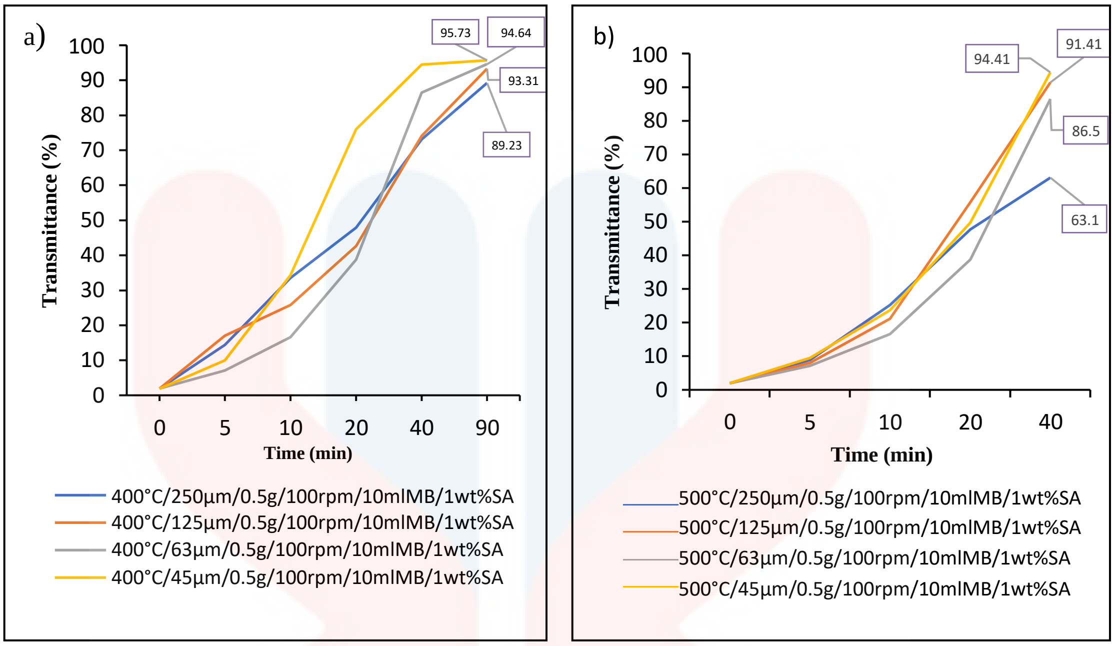

Figure 4.7: (a) Transmittance of methylene blue removal for 400°C, (b) Transmittance of methylene blue removal for 500°C

For 400°C of carbonization temperature, the beads of 250µm, 125µm, 63µm, 45µm was prepared and the removal of methylene blue is 87.90%, 88.32%, 96.19%, 98.63%, respectively. For 500°C of carbonization temperature, the beads of 250µm, 125µm, 63µm, 45µm was prepared and the removal of methylene blue is 96.37%, 97.64%, 98.13%, 98.72%, respectively. For 500°C of carbonization temperature, the beads of 250µm, 125µm, 63µm, 45µm was prepared and the removal of methylene blue is 96.37%, 97.64%, 98.13%, 98.72%, respectively.

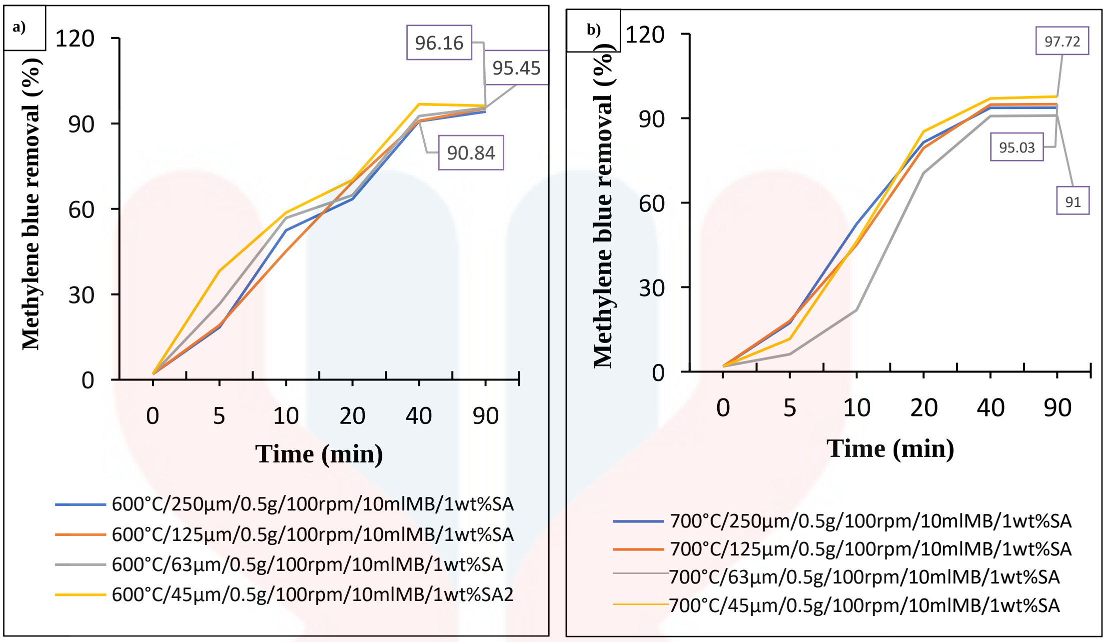

Figure 4.8: (a) Transmittance of methylene blue removal for 600°C, (b) Transmittance of methylene blue removal for 700°C

For 600°C of carbonization temperature, the beads of 250µm, 125µm, 45µm was prepared and the removal of methylene blue is 98.31%, 98.61%, 98.79%, respectively. For 700°C of carbonization temperature, the beads of 250µm, 125µm, 63µm, 45µm was prepared and the removal of methylene blue is 98.91%, 98.96%, 99.10%, 99.43%, respectively.

For 400°C, the trend from 250µm to 45µm, the efficiency of removal of methylene blue is increased. The same trend can be seen for carbonization temperature for 500°C which is at 250µm the efficiency is 87.90% and at 45µm the efficiency is 98.83%. For 700°C for 250µm and 45µm is 98.91% and 99.15%. The biochar yield during the pyrolysis process depends on the type and nature of biomass used. Temperature is the main operating process condition that decides the product efficiency (Yaashikaa et al., 2020) It can be seen that the smaller size of the beads will result in the absorption of methylene blue. This is because of the surface area of the beads. By sieving the biochar into smaller particle sizes has been used to improve the adsorptive capacity for dye. Small particles have a large surface area.

a)

b)

c)

d)

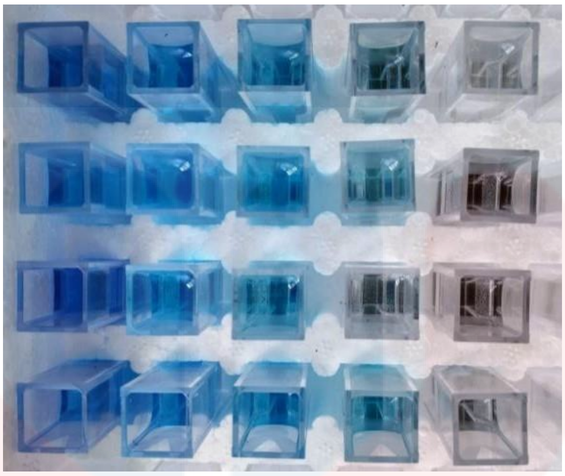

Figure 1: The observation of colour changes in methylene blue solution in different sizes. (a) 500°C/45μm/0.5g/100rpm, (b) 500°C/63μm/0.5g/100rpm, (c) 500°C/125μm/0.5g/100rpm, (d) 500°C/250μm/0.5g/100rpm

a)

b)

Figure 2: The observation of colour changes in methylene blue solution in different sizes. (a) 500°C/250μm/0.5g/100rpm, (b) 500°C/125μm/0.5g/100rpm

4.2.3 Effect of kenaf core biochar size

The purpose of this parameter is to figure out how the size of the biochar affects the removal of MB. The percent of MB removal using CAKCB beads produced using different KC charcoal sizes at 700 ºC 45 and 250 μm was displayed in Figure 4.8.

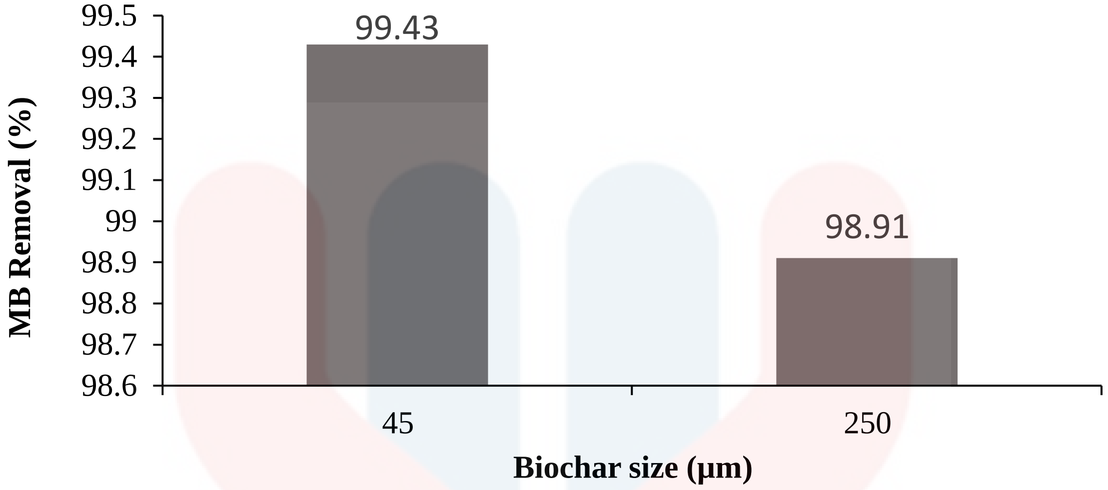

Figure 4.8: The effect of kenaf core biochar size in CAKCB beads in removal of 50 mL of MB of 10 mg/L after 90 minutes.

The graph shows that the efficiency of removing methylene blue dye with CAKCB beads of KC biochar size of 250 µm was 98.91%, and it rose slightly to 99.43% with KC biochar size of 45 µm. This might be because smaller KC biochar has more surface area exposed to the MB dye particles, increasing the number of adsorbent sites.

4.2.5 Effect of pH of methylene blue

The pH of the dye solution has a major impact on the adsorption process's capacity and efficacy. The methylene blue solution's pH has an effect on the binding site's chemistry and surface charge. While keeping the initial dye concentration at 10 mg/L and fixing the other parameters in accordance with the results of earlier studies,(Pathania et al., 2017) the impact of pH on the removal of methylene blue was examined in the pH of 4, 7 and 9. Figure 4.9 demonstrated how the dye pHscaled from 4 to its maximum at 9, progressively increasing the percentage of methylene blue removal from 98.47% to 99.46%.

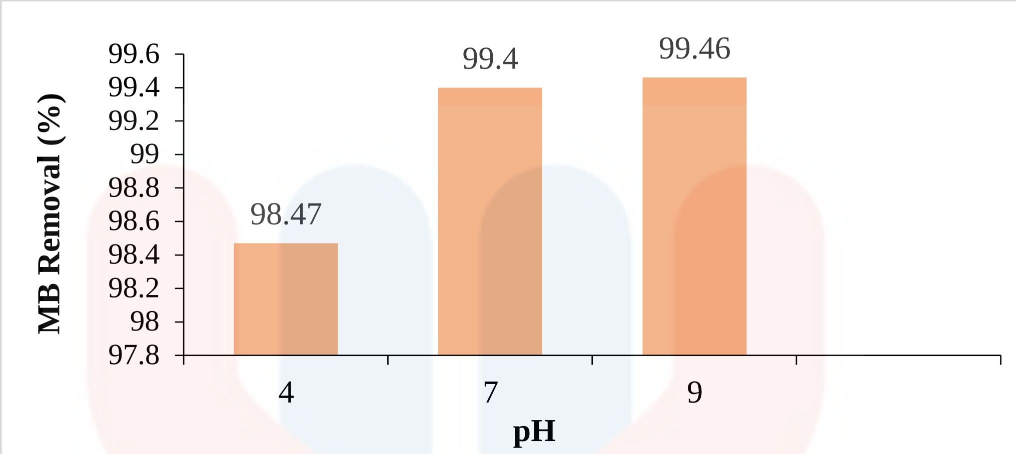

Figure 4.9: Effect on pH of methylene blue adsorption

Methylene blue is a cationic dye, and alginate has a pKa value between 3.4 and 4.5, meaning that both the adsorbent and the adsorbate have positively charged surfaces. It is important for the charges to be opposing in order to maximize the electrostatic attraction and allow for optimal adsorption. Thus, the quantity of positive charges, or H+ (hydrogen) ions, dropped as the pH of the methylene blue increased. This indicates that the dye solution is beginning to include OH- (hydroxide) ions.

4.2.6 Effect of agitation speed

Using a hot plate, the impact of methylene blue removal on CAKCB beads has been studied at three distinct agitation speeds. One of the most important factors affecting the effectiveness of adsorption is agitation. As the agitation speed increased, Figure 4.14's findings indicated that more methylene blue dye particles were adsorbed onto the CAKCB beads.

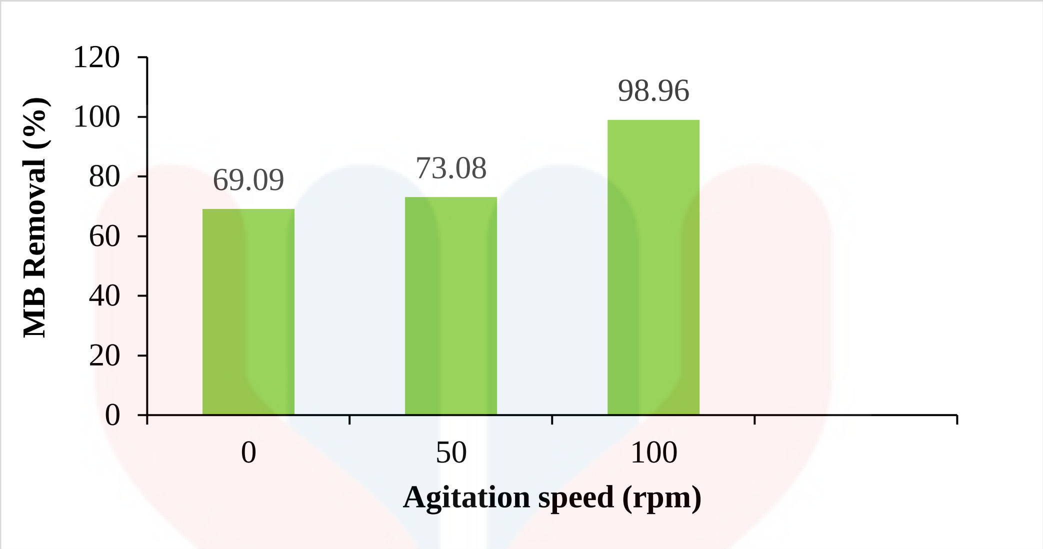

Figure 4.10: The effect of different agitation speed of the CAKCB beads on 50 mL of MB solution of concentration 10mg/L after 90 minutes.

These findings show that dye uptake continued with a 69.09% methylene blue removal rate in the absence of agitation. This indicates the CAKCB beads' capacity for bio-sorption, or the ability to draw and bind dye particles when they come into contact with methylene blue solution. Then, when the agitation speed was increased from 50 to 100 rpm, the methylene blue removal efficiency went from 73.08% to 98.96%. The low methylene blue removal at low agitation speeds can be attributed to the agglomeration of CAKCB beads, which causes incorrect adsorbent-dye molecule particle interaction (Almethen et al., 2022)

Next, applying a high stirring rate resulted in a still higher increase in the percentage of methylene blue removal. According to the graph, the CAKCB beads ability to remove methylene blue at 100 rpm nearly approached 100% efficiency, with a percentage of 98.96%. This demonstrated that high agitation speed improved appropriate and better contact between the CAKCB beads and the methylene blue solution, leading to efficient dye particle transfer onto the adsorbent sites.

More agitation speed, more binding sites on the adsorbent's surface, the availability of CAKCB beads, and less resistance can all contribute to the successful movement and diffusion of methylene blue particles into the KC biochar's pores. The time effect on removal justifies the use of this biochar in terms of the rate at which the dye can transfer from the aqueous phase to the solid adsorbent (Alharbi et al., 2023)

4.2.6 Effect of KC biochar dosage

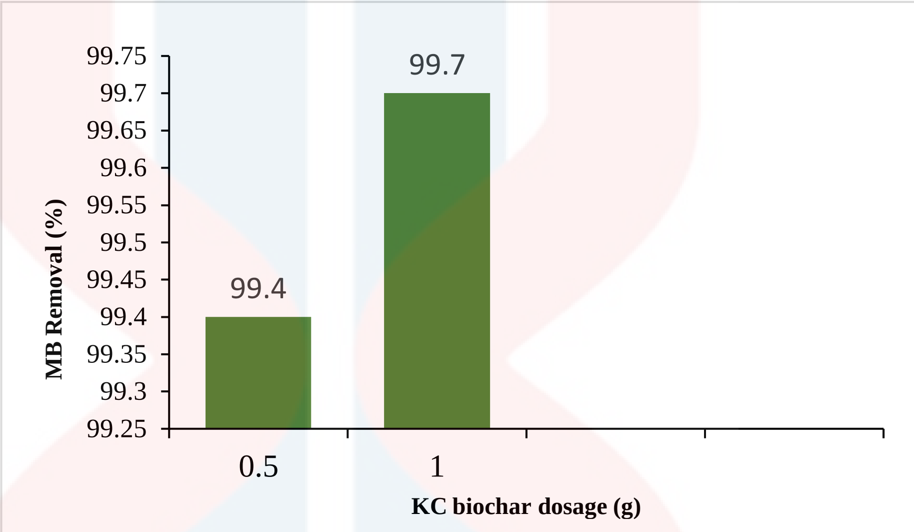

Figure 4.11: The effect of KC biochar dosage in removal of 50 mL MB of 10 mg/L after 90 minutes.

4.3 Characterization with Fourier-Transform Infrared (FTIR)

Using a Fourier transform infrared spectroscope, the surface functional groups of the samples were identified in order to study their surface chemistry and the unknown organic substance and observe the chemical properties that present inside a sample. As a result, we will be able to determine the general kinds of functional groups that are present in a sample molecule. In the mid- infrared range, the spectra were captured with resolutions ranging from 4000 to 400 cm-1. The FTIR spectra for the raw sample of kenaf core biochar were then displayed in Figure 4.21. Based on the findings, the composition of KC, which included cellulose and lignin, contributed for the existence of functional groups of inorganic phosphate, Alharbi, A. F., aliphatic primary amines, aliphatic hydrocarbon, and primary aliphatic alcohols.

The FTIR spectra of kenaf core biochar treated methylene blue solution at 500 and 700 ºC, indicated by the blue and green lines, respectively, in Figure 4.19. In the meanwhile, the FTIR spectra for KC biochar at 700 ºC alone were shown by the red line. The information about the included functional groups might be obtained using this FTIR spectrum. Based on the infrared spectrums show distinct peaks at low transmittance percentages, indicating that the sample was absorbing light at that specific energy or wavelength of the IR light. Because there were specific, unidentified functional groups present inside the sample that absorbed the IR light, the IR light transmittance did not manage to pass through and was not recorded by the detector, which explained the low transmittance %. Therefore, significant information can be noticed with each steep peak that is observed. However, the intensity of absorbance at particular IR wavelengths depends on the strength of the bonds within the functional groups.

Figure 4.19 illustrates the FTIR spectra of KC biochar at temperatures of 700 ºC and 600 ºC, with adsorbed methylene blue represented by red line. In the raw material of KC biochar at 700 ºC, an inorganic carbonate peak was observed at 1567.72 cm-1. Meanwhile at for 600 ºC is at peak 1809.09 cm-1. This means that the carbonate-carbon present in the biochar originated from minerals within the kenaf core powder during the carbonization process. The presence of this carbonate-carbon contributes to the biochar possessing acidic characteristics. Then, the hydroxide region involving absorption at 3200 observed the -OH stretching vibration. After being treated with methylene blue solution, the infrared absorption bands for KC biochar at 600 and 700 ºC were discovered at 2358.96 and 2341.06 cm-1, respectively. These two bands demonstrated the analysis of O-H stretching.

Based on the graph analysis, the modified treatment resulted in an increase in the typical peak intensity of −OH. This indicates that that the surface of the biochar exhibited an increase in oxygen-containing functional groups following acid–base alteration. In general terms, biochar had aromatic structure materials and was enriched in oxygen-containing groups. These oxygen-containing groups provided certain active sites for the adsorption of organic matter, which could promote the adsorption of biochar (Liu et al., 2020)

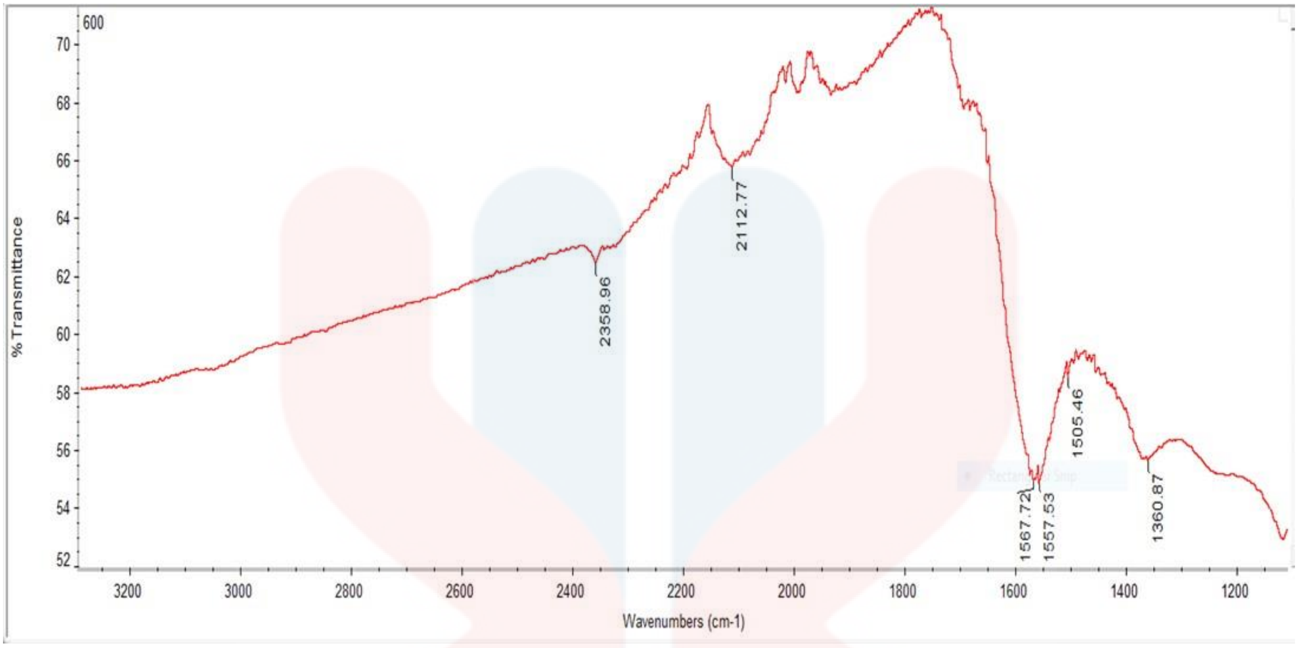

Figure 4.12: FTIR spectra of biochar kenaf core powder for 600°C and its functional groups.

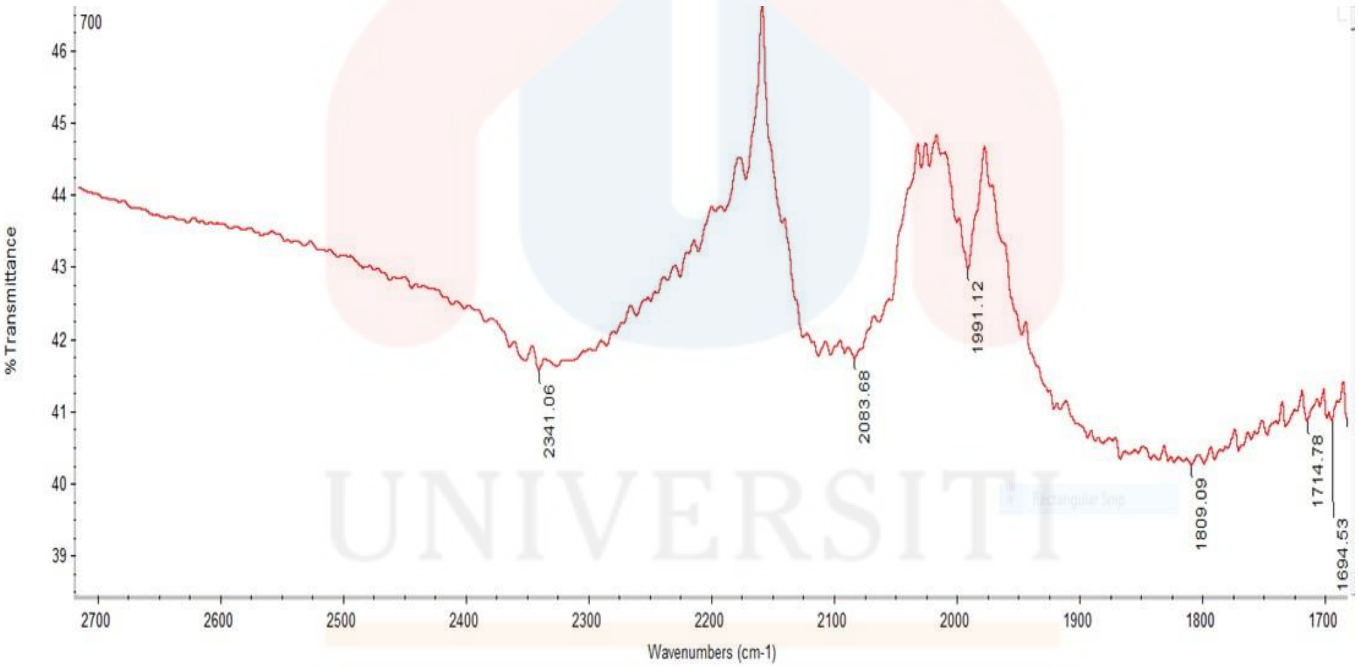

Figure 4.13: FTIR spectra of biochar kenaf core powder for 700°C and its functional groups.

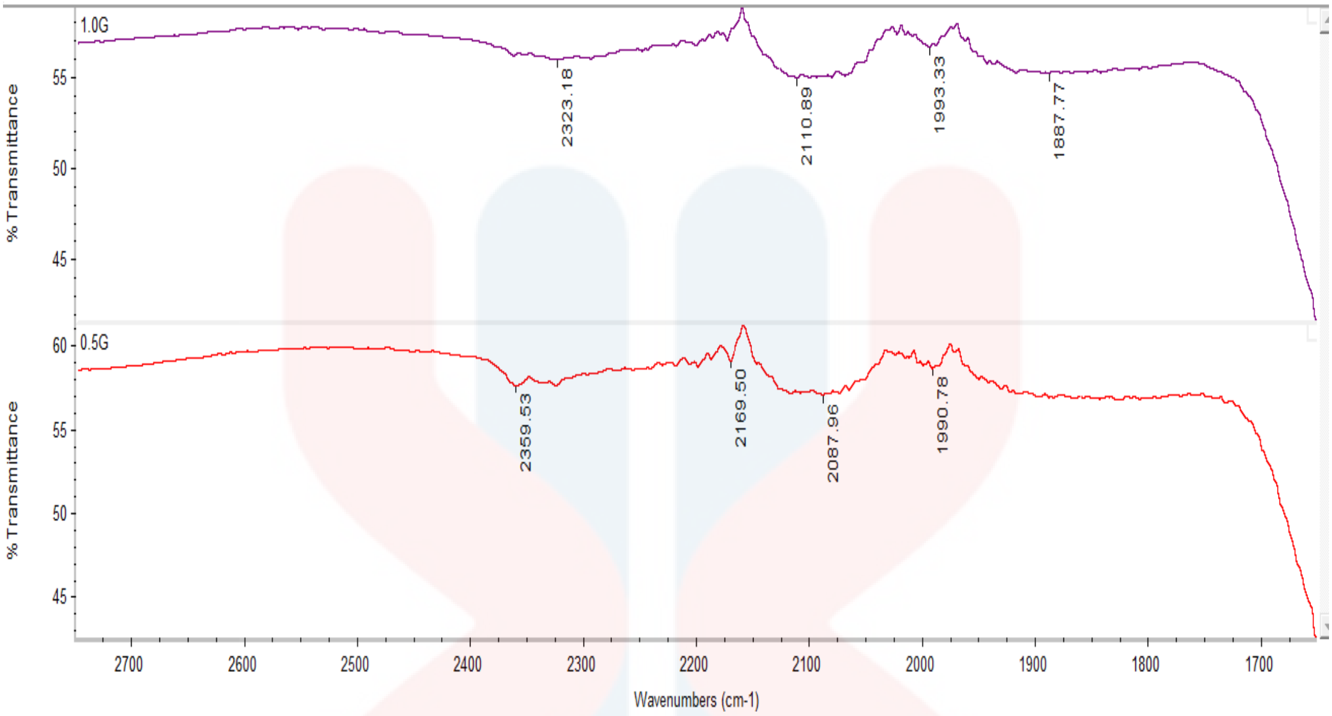

Figure 4.14: FTIR spectra of blank CA beads and CA and CAKCB beads adsorbed methylene blue of dosage 0.5 and 1.0 g.

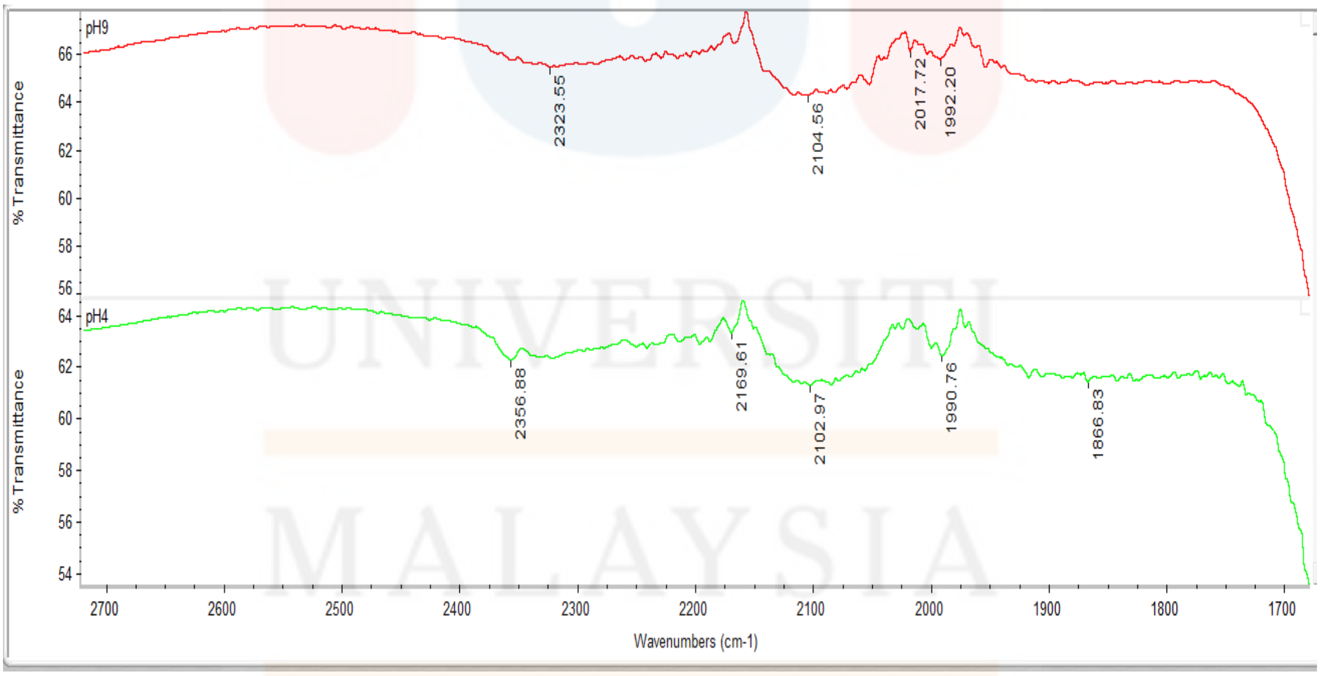

Figure 4.15: FTIR spectra of CAKCB beads adsorbed methylene blue at pH9 and pH4.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The present research has shown that the removal of methylene blue dye solution can be treated using CAKCB beads prepared in an effective manner. Kenaf core are widely available in Malaysia and other Asian nations. They can be safely used and thermally converted into biochar through an incremental carbonization process. Because calcium alginate contains functional groups that facilitate the adsorption process, it has the potential to absorb dyes. On the other hand, the high porosity of KC biochar assists the removal of dyes more easily. The adsorption efficiency and rapid methylene blue removal were improved by combining calcium alginate and KC biochar, resulting in the preparation of enhanced beads. In the prior batch studies, it was determined that the optimal conditions for utilizing CAKCB beads in the removal of methylene blue from an aqueous solution included a carbonization temperature of 700 ºC, a size of 45 µm, a dosage of 1.0 g, a pH of 9 for methylene blue, and an agitation speed of 100 rpm. Under these parameters, the dye removal percentage reached an impressive 99.70%. Because of the higher carbonization temperature, biochar has more porosity, which enhances dye particle entrapment and adsorption within its enlarged pores. Furthermore, a higher dosage of KC biochar increases the availability of adsorption surfaces, and the smaller particle size of KC biochar exposes a larger surface area to the methylene blue solution. The methylene blue with a pH of 9 then showed more OH- ions, which improved the electrostatic interaction of the dye particles with the adsorbent. Then, when the agitation speed was high, the boundary layer thickness around the CAKCB beads shrank, which made it easier for the dye particles to pass through into the adsorbent. The overall results of these studies indicate that the problems related to the removal of dyes from wastewater can be successfully handled by a green trick that involves simple in preparation measurements.

5.2 Recommendations

To enhance the research, one recommended improvement involves introducing in this study's parameter, specifically employing a syringe with various needle sizes during the bead formation. In the current investigation, all beads were crafted using a fixed 0.1mm dropper needle. Introducing smaller bead sizes could potentially elevate the surface-to-volume ratio, facilitating more effective interaction with methylene blue dye. This adjustment may lead to an improved adsorption rate and increased diffusion due to the enhanced accessibility of the adsorption sites. Needles offer enhanced precision during biochar bead formation, allowing for fine control over the release of the biochar solution. This results in the creation of beads with uniform sizes, reducing variations that may affect absorption characteristics. Uniform bead sizes achieved through needle-based techniques optimize the adsorption capacity of biochar for methylene blue. This consistency ensures a predictable and efficient adsorption process, enhancing the overall performance of biochar materials in pollutant removal. Next is the increased the agitation speed. In this study, the agitation speed is only until 100rpm. The higher speed of stir can increase the efficiency of methylene blue absorption into the beads. Furthermore, the adsorption approach can be utilized to investigate the possibility of many bead reuses. The goal of this is to determine how many adsorption cycles with a methylene blue solution can be completed without affecting the mass and shape of the biosorbent or the adsorbent's ability to bio selectively absorb substances.

REFERENCES

Alharbi, A. F., Alotaibi, A. A., Gomaa, H. E. M., Abahussain, A. A. M., & Abdel Azeem, S. M. (2023). Magnetic Biochar by One-Step Impregnation Pyrolysis of Peganum harmala L. for Removal of Rhodamine B. *Adsorption Science & Technology*, *2023*, 1–17. https://doi.org/10.1155/2023/9993465

Akpoveta, V. O. (2020). Process optimization of silica encapsulation technique as a unique remediation technology for the treatment of crude oil contaminated soil. Egyptian Journal of Petroleum, 29(2), 113–119.

Almethen, A. A., Alotaibi, K. M., Alhumud, H. S., & Alswieleh, A. M. (2022). Highly Efficient and Rapid Removal of Methylene Blue from Aqueous Solution Using Folic Acid-Conjugated Dendritic Mesoporous Silica Nanoparticles. *Processes*, *10*(4), 705. https://doi.org/10.3390/pr10040705

Banitalebi, G., Mosaddeghi, M. R., & Shariatmadari, H. (2019). Feasibility of agricultural residues and their biochars for plant growing media: Physical and hydraulic properties. Waste Management, 87, 577–589.

Ci, Y., Wang, L., Guo, Y., Sun, R., Wang, X., & Li, J. (2015). Original Article Study on encapsulation of chlorine dioxide in gelatin microsphere for reducing release rate. In Int J Clin Exp Med (Vol. 8, Issue 8).

Cong Nguyen, N., Thi Nguyen, H., Cong Duong, H., Chen, S.-S., Quang Le, H., Cong Duong, C., Thuy Trang, L., Chen, C.-K., Dan Nguyen, P., Thanh Bui, X., Guo, W., & Hao Ngo, H. (2021). A breakthrough dynamic-osmotic membrane bioreactor/nanofiltration hybrid system for real municipal wastewater treatment and reuse. Bioresource Technology, 342, 125930.

Harussani, M. M., & Sapuan, S. M. (2022). Development of Kenaf Biochar in Engineering and Agricultural Applications. In Chemistry Africa (Vol. 5, Issue 1). Springer Science and Business Media Deutschland GmbH.

Liu, C., Wang, W., Wu, R., Liu, Y., Lin, X., Kan, H., & Zheng, Y. (2020). Preparation of Acid- and Alkali-Modified Biochar for Removal of Methylene Blue Pigment. *ACS Omega*, *5*(48), 30906–

30922. https://doi.org/10.1021/acsomega.0c03688

Liu, H., Chen, Y., Yang, H., Gentili, F. G., Söderlind, U., Wang, X., Zhang, W., & Chen, H. (2019). Hydrothermal carbonization of natural microalgae containing a high ash content. Fuel, 249, 441–448.

Mary Ealias, A., & Saravanakumar, M. P. (2019). A critical review on ultrasonic-assisted dye adsorption: Mass transfer, half-life and half-capacity concentration approach with future 20industrial perspectives. Critical Reviews in Environmental Science and Technology, 49(21), 1959–2015.

Oladoye, P. O., Ajiboye, T. O., Omotola, E. O., & Oyewola, O. J. (2022). Methylene blue dye: Toxicity and potential elimination technology from wastewater. Results in Engineering, 16, 100678.

Saba, N., Jawaid, M., Hakeem, K. R., Paridah, M. T., Khalina, A., & Alothman, O. Y. (2015). Potential of bioenergy production from industrial kenaf (Hibiscus cannabinus L.) based on Malaysian perspective. *Renewable and Sustainable Energy Reviews*, *42*, 446–459. https://doi.org/10.1016/j.rser.2014.10.029

Sharma, D., Chaudhari, P. K., Dubey, S., & Prajapati, A. K. (2020). Electrocoagulation Treatment of Electroplating Wastewater: A Review. Journal of Environmental Engineering, 146(10).

Sim, D., Tan, I. B., Lim, L., & Hameed, B. (2021). Encapsulated biochar-based sustained release fertilizer for precision agriculture: A review. Journal of Cleaner Production, 303, 127018.

Singh, R. K., Gupta, D. K., Kumar, S., & Kumar, S. (2013). *Utilization of Fly Ash As Biologically Activated Carbon on Microbial Consortium for the Removal of*. *4*(1), 20–28.

Sirajudheen, P., Karthikeyan, P., Vigneshwaran, S., & Meenakshi, S. (2020). Synthesis and characterization of La(III) supported carboxymethylcellulose-clay composite for toxic dyes removal: Evaluation of adsorption kinetics, isotherms and thermodynamics. International Journal of Biological Macromolecules, 161, 1117–1126.

Walker, D. B., Baumgartner, D. J., Gerba, C. P., & Fitzsimmons, K. (2019). Surface Water

Pollution. In Environmental and Pollution Science (pp. 261–292). Elsevier.

Yang, X., Nguyen, X., Tran, Q. T., Nguyen, T. N., Ge, S., Nguyen, D., Nguyen, V., Cuong, L. P., Rene, E. R., Singh, P., Singh, P., Ahamad, T., Alshehri, S. M., Shi, S. Q., Kim, S. H., & Van Le, Q. (2022). Machine learning-assisted evaluation of potential biochars for pharmaceutical removal from water. Environmental Research, 214, 11395

Yaashikaa, P. R., Kumar, P. S., Varjani, S., & Saravanan, A. (2020). A critical review on the biochar production techniques, characterization, stability and applications for circular bioeconomy. *Biotechnology Reports*, *28*, e00570. https://doi.org/10.1016/j.btre.2020.e00570

Pathania, D., Sharma, S., & Singh, P. (2017). Removal of methylene blue by adsorption onto activated carbon developed from Ficus carica bast. *Arabian Journal of Chemistry*, *10*, S1445–S1451. https://doi.org/10.1016/j.arabjc.2013.04.021

Zeeb, B., Saberi, A. H., Weiss, J., & McClements, D. J. (2015). Formation and characterization of filled hydrogel beads based on calcium alginate: Factors influencing nanoemulsion retention and release. *Food Hydrocolloids*, *50*, 27–36. https://doi.org/10.1016/j.foodhyd.2015.02.041

APPENDIXS

RAW MATERIAL PREPARATION

A.1 The raw materials of the kenaf core

(a)

Figure A.1.1: (a) Kenaf core powder

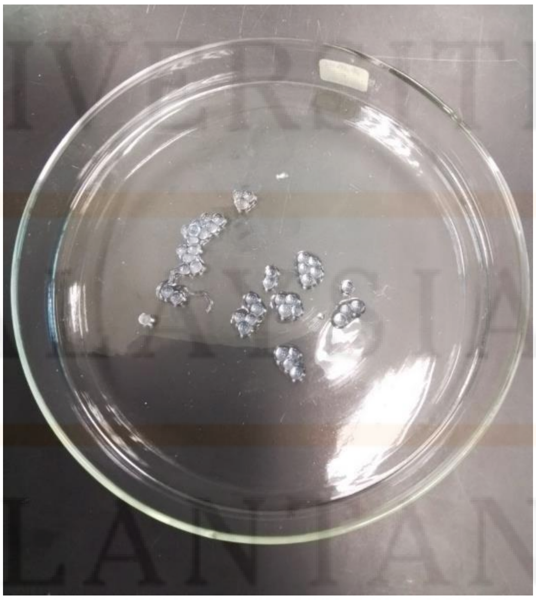

Figure A.1.2: (b) Blank beads

Figure A.1.3: (c) Kenaf Core biochar