



Faculty of Engineering

**DEVELOPMENT OF BIOMASS DERIVATIVES NANOCARBON AND
ITS COMPOSITE FOR REMOVAL OF THE HEAVY METAL**

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**Development Of Biomass Derivatives Nanocarbon And Its Composite
For Removal Of The Heavy Metal**

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A dissertation submitted in partial fulfilment
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ABSTRACT

Heavy metal contamination is a severe issue and potential harm to the ecosystem. There are numerous reports on the utilization of activated carbon/nanocarbon as adsorbents of heavy metals. Biomass such as sawdust as a precursor is used to synthesize activated carbon which is developed into nano-activated carbon (NAC). It is studied that activated carbons have been globally recognized as the oldest, widely used, and most popular adsorbent. Meanwhile, nanoparticles are used to remove the heavy metal pollutants and it is called new generation nano-adsorbents. Besides, the effective nanocomposites consisting of metal oxides, carbon, and polymers are also created as promising adsorbents for the sake of wastewater treatment and prevention of pollution. Therefore, this research is carried out to study the characterization and effectiveness of nanocarbon and its composites as an adsorbent for heavy metal removal. To conclude, the experimental procedures of the development of nanocarbon through a ball-milling process using Planetary Ball Mill (PM 400), and the composites is developed used solution mixing. The findings from this project may contribute to the advancement of activated carbon as nanocarbon composites in order to enhance its role as heavy metal adsorbents, which can aid in the efficacy of heavy metal contamination treatment. The nano-activated carbon and its composites with PLA/TiO₂ were successfully characterized using BET, SEM, EDS, FTIR, and TGA. Lastly, the analysis of the performance of nanocarbon composites as adsorbents in the adsorption of heavy metals in POME is analysed by using Atomic Absorption Spectroscopy (AAS) and it is found that composite with the highest content of NAC has a better adsorption capability.

Keywords: Heavy metals, Adsorbents, Ball-milling, Nano-activated carbon, Nanocomposites

ABSTRAK

Pencemaran logam berat adalah isu yang teruk dan berpotensi membahayakan ekosistem. Terdapat angka laporan mengenai penggunaan karbon teraktif/nanokarbon sebagai penjerap logam berat. Biojisim seperti habuk papan sebagai pendahulu digunakan untuk mensintesis karbon teraktif yang dibangunkan menjadi karbon teraktif nano (NAC). Dikaji bahawa karbon teraktif telah diiktiraf secara global sebagai penjerap tertua, digunakan secara meluas dan paling popular. Sementara itu, zarah nano digunakan untuk membuang bahan pencemar logam berat dan ia dipanggil penjerap nano generasi baharu. Selain itu, nanokomposit berkesan yang terdiri daripada oksida logam, karbon, dan polimer juga dicipta sebagai penjerap yang menjanjikan demi rawatan air sisa dan pencegahan pencemaran. Oleh itu, kajian ini dijalankan untuk mengkaji pencirian dan keberkesanan nanokarbon dan kompositnya sebagai penjerap untuk penyingkiran logam berat. Sebagai kesimpulan, prosedur eksperimen pembangunan nanokarbon melalui proses pengilangan bola menggunakan Planetary Ball Mill (PM 400), dan komposit dibangunkan menggunakan campuran larutan. Penemuan daripada projek ini mungkin menyumbang kepada kemajuan karbon teraktif sebagai komposit nanokarbon untuk meningkatkan peranannya sebagai penjerap logam berat, yang boleh membantu dalam keberkesanan rawatan pencemaran logam berat. Karbon teraktif nano dan kompositnya dengan PLA/TiO₂ berjaya dicirikan menggunakan BET, SEM, EDS, FTIR, dan TGA. Akhir sekali, analisis prestasi komposit nanokarbon sebagai penjerap dalam penjerapan logam berat dalam POME dianalisis dengan menggunakan Spektroskopi Serapan Atom (AAS) dan didapati komposit dengan kandungan NAC tertinggi mempunyai keupayaan penjerapan yang lebih baik.

Kata kunci: *Logam berat, Penjerap, Bola penggilingan, Karbon teraktif nano, Nanokomposit*

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LIST OF ABBREVIATIONS

AAS	Atomic Absorption Spectroscopy
AC	Activated Carbon
BET	Brunauer-Emmett-Teller
COD	Chemical oxygen demand
CVD	Chemical vapour deposition
EDS	Energy dispersive X-ray spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
NAC	Nano-activated carbon
PLA	Polylactic acid
POME	Palm oil mill effluent
PSA	Particle Size Analyzer
SEM	Scanning electron microscopy
TGA	Thermogravimetric Analysis

LIST OF NOMECLATURE

As	Arsenic
Cd	Cadmium
CO ₂	Carbon dioxide
Cu	Copper
Hg	Mercury
Pb	Lead
Mn	Manganese
Ni	Nickel
TiO ₂	Titanium oxide
Zn	Zinc
°C	Celcius
µm	Micrometer
nm	Nanometer

CHAPTER 1

INTRODUCTION

1.1 Overview Biomass in Malaysia

Malaysia is blessed with significant biomass resources that may be turned into renewables energy or beneficial eco-products among the countries with active agricultural business. According to BE-Sustainable Magazine (2012), Malaysia generates a minimum of 168 million tonnes of biomass waste per year. Furthermore, palm oil waste contributes for 94% of biomass feedstock, with agricultural and natural resources by-products such as wood waste (4%), rice (1%), and sugarcane sector wastes contributing for the remaining (1%). Besides, Malaysia has both non-renewable and renewable energy resources, such as oil and gas, as well as biomass, hydroelectric, and wind energy. In terms of biomass resources, Malaysia has enormous agricultural biomass and wood waste resources that are ready to be used immediately. However, the biomass resource's energy potential has yet to be fully realized in Malaysia. Biomass resources have increasingly been known as a reliable alternative energy source, especially in countries with a large agricultural sector. In Malaysia, substantial use of biomass as a renewable energy source might reduce reliance on non – renewable energy in which provide a major benefit in lowered CO₂ emissions to the environment, resulting in a lower greenhouse impact. However, increasing competitiveness would necessitate huge financing and technological advancements in the efficient and cost-effective conversion of biomass to energy.

An approximately 2 million m³ of wood wastes are created each year in Malaysia. Moreover, wood wastes are classified as types of wood that cannot be profitably sold from a certain forestry or industrial activity owing to the present economic circumstances and technical advancement. According to Osman et al. (2014), the national biomass plan is broadening its biomass collection, which was primarily centred on the palm oil business, to incorporate biomass from other resources like as rubber, wood, and rice husk. For example, the woody biomass available in Sarawak by itself generated millions of

tonnes from current forest areas. Malaysia generates a lot of forest and wood processing waste, which offers a lot of renewable energy possibilities such as sawdust in which it is a component of saw milling by-products. Kong (2000) stated that it is reported that the number wood waste created yearly in Peninsular Malaysia is relatively large, with the sawmilling sector producing around 13% of the sawdust. Presently, the most practical wood sources are industrial wastes and wood waste. Wood waste is easily accessible, cost-effective, and ecologically acceptable biomass supplies available. Zafar (2020) mentioned that biomass generated wood production sites, such as plywood mills and sawmills, is mostly concentrated in these industries. Moreover, the quantity of waste created by wood processing companies varies based on the kind of raw material used and the completed product produced. According to Shafie et al. (2017), there are numerous kinds of tree types present in Malaysia's tropical forest areas of heavy rainfall that affect the growth of the timber manufacturing sectors, particularly in the operations of plywood that create waste in large quantities, such as sawdust and wood chips. Furthermore, Lim & Hii (2004) stated that the timber business in Bintulu, Sarawak, capitalises on the significant volumes of sawdust generated by sawmills, where the sawdust collected comes from about 40 wood types and is utilised to manufacture charcoal. In this research, biomass such as sawdust as a precursor is used to synthesize activated carbon which then will be developed as nanocarbon.

1.2 Overview Biomass Derivatives as Adsorbent

As stated by Tay et al. (2009), lignocellulosic components are excellent and inexpensive foundation materials for the synthesis of activated carbon. In addition, the major components of wood biomass are lignocellulosic components. Moreover, the accessibility of economical wastes from the biomass industrial processing sector has shown to be a promising raw material for the development of activated carbons. Wood's well-structured fiber elements offer an important role in the synthesis of fine-grained activated carbons (Danisha & Ahmad, 2018). Besides, activated carbon is a carbonaceous substance that has been treated to contain numerous tiny pores that may improve specific surface area for adsorption or chemical reactions. It is feasible to make it from low-cost and widely available biomass precursors such as crop production and timber. Besides, activated carbon may be used for a variety of purposes, including contaminant gas

adsorption, water treatment, metal removal, and catalyst or catalyst support (Mazlana, et al., 2016). Furthermore, activated carbons have been globally recognized as the oldest, widely used and most popular adsorbent in water and wastewater treatment industries (Danisha & Ahmad, 2018). Besides, activated carbon has been employed as a flexible adsorbent for a variety of liquid and gaseous contaminants. In addition, processing wood waste into useful carbon material may also help to alleviate environmental challenges such as agricultural residues piling, which affects air and water pollution throughout the natural deterioration process. Along with activated carbon, it is studied that carbon nanomaterials such as nanocarbon has excellent potential for removal of heavy metal owing to its particular and versatile features provide prospective advancements in ecological processes such as pollution reduction and toxicity management. Besides, Kausar (2018) stated that carbon nanomaterials are reported to be very efficient in eliminating numerous contaminants in water and air. Therefore, this research will emphasize more about nanomaterials such as nanocarbon as adsorbent for heavy metal removal.

1.3 Problem Statement

Every alkaline metal compound with a high density that is harmful at low concentrations is described as heavy metal. Heavy metal contamination is a severe issue and a potential harm to the ecosystem. Ali et al. (2019) stated that heavy metals are regarded as environmental contaminants because of their adverse effects, chemical stability in the environment, and gradual accumulation of organism-derived chemicals. Natural sources of heavy metals include ore depletion and volcanic explosions, while manmade sources include mining and different industrial and agricultural operations. The Earth's worldwide tendencies of industrialization and urbanisation have resulted in a rise in the man-made contribution of heavy metals to the ecosystem. Ruparelia et al. (2008) mentioned that excessive manufacturing and inappropriate dumping are regarded as major contributors to the emission of heavy metals into environments. Besides, heavy metals emitted into the environment throughout industrial activities are deposited on land via dry and wet precipitation. Heavy metals are released into the environment when wastewaters such as industrial wastes and household waste are disposed. There are numerals of reports on the utilization of activated carbon or nanocarbon as well

nanocomposite as adsorbents. It is well known that activated carbons can be prepared using a variety of raw materials. The most commonly used precursors are coal, wood, coconut shells and agricultural by-products. Activated carbon may be used as an adsorbent to remove unpleasant taste, odour, colour, and other inorganic and organic contaminants from industrial and urban wastewater, as well as for solvent treatment and air pollution management in populated areas. Regardless the immense various application of activated carbon, the most significant barrier to widespread usage of activated carbons in business areas is the high cost of manufacturing and the ambiguous procedures involved with manufacturing and conversion processes. Throughout the initial stage of the invention of activated carbons, coal was considered to be a sufficient resource of carbon, but the perspective changed to renewable supplies such as biomass or other carbonaceous waste for activated carbon synthesis. Ruparelia et al. (2008) studied that activated carbon is one of the sorbents that has been researched for metal ion adsorbent. However, it has been shown that activated carbon adsorbents have weak removal effectiveness. Next, nanotechnology is a cutting-edge method of research that has the potential to overcome a wide range of environmental problems by regulating the size and form of composites on a nanostructured (Baby, Saifullah, & Hussein, 2019). Moreover, carbon nanoparticles have distinctive characteristic in such that they are non-toxic, simpler to biodegrade, have a greater surface area, and are especially beneficial for pollutants removal. Besides, carbon nanoparticles are rapidly growing owing to their excellent physical properties and chemical structure which may be used to enhanced heavy metal water treatment. Furthermore, carbon nanomaterials such as carbon nanotubes and activated nanocarbon have a strong capability for heavy metal absorption from water due to their huge surface area, and nanoparticle size, as well as their ease of chemical modification and regeneration. Besides, the effective nanocomposites consisting of metal oxides, carbon, and polymers also created as promising adsorbents for the sake of wastewater treatment and prevention of pollution. Therefore, this research is carried out to study the characterization and effectiveness of nanocarbon and its composites as an adsorbent for heavy metal removal.

1.4 Research Questions

This project will be focused on the development of biomass derivatives nanocarbon and its composites for removal of heavy metal. Therefore, below are the research questions:

- i. What are the characterizations of nanocarbon and its composite analyse by analytical techniques?
- ii. How is the performance of nanocarbon composites as an adsorbent of heavy metals in Palm Oil Effluent Mill (POME)?
- iii. How effective nanocarbon composites in the removal of heavy metal in Palm Oil Effluent Mill (POME)?

1.5 Objectives of the Study

The main aim of this research is to develop nanocarbon from biomass derivative. Next, the nanocarbon is further reinforced with PLA/TiO₂ to develop into nanocomposite. Besides, the nanocarbon and its composites will be characterized by using various analytical techniques to apply in the removal of heavy metal. For this research, there are three objectives that have been emphasized in this research. Therefore, the objectives are scheme as follows:

- i. To develop the nanocarbon from the biomass derivatives.
- ii. To characterize the nanocarbon and its composites.
- iii. To analyse the application of nanocarbon composites in removal of heavy metals.

1.6 Scopes of the Study

The approach to develop nanocarbon using mechanical-mill activated carbon as adsorbent of heavy metals was conducted to determines its characterization and applications. The analysis of characterization of nanocarbon is focused on using analytical techniques such as Brunauer-Emmett-Teller (BET), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS). Furthermore, the milling process of activated

carbon is conducted at Engineering Materials: Physical Metallurgy Laboratory in Mechanical Engineering Department. Next, the composites of nanocarbon consists of PLA/TiO₂ is developed and analyze by using Thermogravimetric Analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy (EDS). Lastly, the analysis of performance of nanocarbon composites in the applications removal of heavy metals is analyzed by using Atomic Absorption Spectroscopy (AAS).

1.7 Significance of Study

Heavy metal contamination is a serious problem that might affect the environment and humans. Furthermore, heavy metals are considered environmental pollutants due to their negative effects, chemical stability in the environment, and slow accumulation of organism-derived compounds. According to research, the use of activated carbon for heavy metal removal is widely available and highly effective. Moreover, surface-engineered nano-adsorbent has shown quick adsorption rate and improved sorption effectiveness for removing heavy metal pollutants in wastewater according to research. This study project contributes to the advancement of activated carbon develop as nano-activated carbon reinforced with PLA/TiO₂ in order to enhance its role as heavy metal adsorbents, which can aid in the efficacy of heavy metal contamination treatment. Therefore, the findings of this study will immediately aid those who wish to filter substances in a wide range of chemical purposes, such as public water supplies, odour elimination, and industrial pollution management.

1.8 Summary

This chapter introduces the term biomass which is essential in the development of activated carbon and nanocarbon. In terms of biomass resources, Malaysia has enormous agricultural biomass and wood waste resources that are ready to be used immediately. Moreover, the accessibility of economical wastes from the biomass industrial processing sector has shown to be a promising raw material for the development of activated carbons. Along with activated carbon, it is studied that carbon nanomaterials such as nanocarbon has excellent potential for removal of heavy metal owing to its particular and versatile

features provide prospective advancements in ecological processes such as pollution reduction and toxicity management. Therefore, this research is carried out to study the characterization of nanocarbon and its performance as heavy metal removal. The findings from this project may contribute to the advancement of activated carbon developed as nano-activated carbon reinforced with PLA/TiO₂ in order to enhance its role as heavy metal adsorbents, which can aid in the efficacy of heavy metal contamination treatment.

Chapter 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, literature review will be conducted to study in details about activated carbon and nanocarbon in terms of their synthesis, and characterization. In this study, the general and treatment of heavy metal will be discussed as well. As studied, the prepared activated carbon is synthesized through chemical activated method from biomass such as sawdust. Furthermore, the studies about activated carbon develop into nano-activated carbon will be discussed in terms of its role and synthesis. Moreover, the nano-activated carbon and its composite will be discussed generally. Lastly, the analytical technique used to analyze the characterization and performance of activated carbon, nanocarbon, and its composite are also studied such as SEM, EDS, BET, FTIR, TGA, and AAS.

2.2 General of heavy metal

The emission of heavy metals such as Ni^{2+} , Cu^{2+} , Zn^{2+} , Cd^{2+} , Pb^{2+} , and As is common place in a variety of industries, particularly in the chemical industry. Purification of effluents from any industries before they are discharged into a waterway or lake is critical to preventing the negative environmental and human health impacts of active or passive ingestion from those metals. According to the Mariana et al. (2021), heavy metal concentrations in various rivers, lakes, and groundwater exceeded regulatory standards as a consequence of rapid urbanisation, global economic growth, and rapid manufacturing. As a result of these factors, water contamination has increased. Heavy metals may be present in the environment in a multitude of ways, particularly water, which can be consumed actively or passively, and plants treated with heavy metal-infected water. Furthermore, Fu & Wang (2011) added that heavy metal effluents are