



**PREPARATION OF ACTIVATED CARBON NANOMATERIAL FROM PALM OIL
KERNEL SHELL FOR REMOVAL OF HEAVY METAL IONS**

By

BABY RABIA

**This thesis submitted to the School of Graduate Studies Universiti Putra Malaysia,
in the Fulfilment of the Requirements for the Degree of Doctor of Philosophy**

September 2022

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DEDICATION

I dedicated my thesis to my father Late. Dr. Capt. Mohammad Iqbal Shaikh, my dearest mother Mrs. Shahmeer Shaikh, who always encouraged me for higher studies, my kids and loving Husband Dr. Saifullah Bullo who was always beside me with his full support.



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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September 2022

Chairman : Professor Mohd Zobir bin Hussein, PhD
Institute : Nanoscience and Nanotechnology

Environmental pollution is greatly caused by industrialization as well as human beings and water contamination is one of the major hazards for living. Many kinds of contaminants have been studied such as bacteria, viruses, organic molecules, dyes and heavy metal ions, such as Cr^{6+} , Pb^{2+} , Cd^{2+} , Zn^{2+} , As^{3+} , Cu^{2+} and Hg^{2+} , etc. Among all these contaminants, heavy metal ions are non-biodegradable in nature and can accumulate in the human body continuously over time, which results in severe adverse effects like a breakdown of the nervous system, skin diseases, liver damage, kidney failure, anemia, hepatitis, ulcers and carcinogenic. The key objectives of this study are to prepare the PKS powder, the functionalization of the PKS powder, preparation of activated carbon nanomaterial (ACN) and functionalized ACN and application for the treatment of heavy metal-contaminated water. In this study, four heavy metal ions namely chromium (Cr^{6+}), lead (Pb^{2+}), cadmium (Cd^{2+}) and zinc (Zn^{2+}) were selected as the water contaminants. Furthermore, four different bio-sorbents were prepared namely PKS powder, functionalized PKS (PKS-Sulfo), activated carbon (AC) and functionalized activated carbon (FAC). All of these adsorbents were successfully applied for the treatment of heavy metal-contaminated water. They showed good adsorption affinities for the Cr^{6+} , Pb^{2+} , Cd^{2+} and Zn^{2+} heavy metal ions. Adsorption of Cr^{6+} and Pb^{2+} was determined to be maximum almost reach to 99% in case of each adsorbent. The Cd^{2+} whose adsorption was found to be 80-85% , followed by 70-75% of Zn^{2+} ions adsorption in all four applied adsorbents. Two isotherm models namely Freundlich and Langmuir and three kinetics models namely the pseudo-first-order, pseudo-second-order and Parabolic diffusion were applied for the determinations of kinetic interactions between the adsorbents; (PKS, PKS-Sulfo, AC and FAC) and the adsorbates; (Cr^{6+} , Pb^{2+} , Cd^{2+} and Zn^{2+}). The adsorption followed Pseudo Second Order in all cases of the adsorbents. Langmuir adsorption isotherm was found to be followed in case of PKS-Sulfo, and only zinc adsorption in PKS, apart from this Activated carbon (AC, Functionalized Activated Carbon (FAC) as well as PKS were found to follow Freundlich isotherm model. PKS-Sulfo The adsorption efficiency among all four adsorbents was found to be in the order of $\text{FAC} > \text{AC} > \text{PKS-Sulfo} > \text{PKS}$. This is a greener approach that can serve the dual

function of the protection environment from smoke resulting from the burning of PKS waste. The first function was by converting the PKS into a useful material instead of burning it and secondly it is applied for the treatment of heavy metal-contaminated water. The designed materials can further be exploited for dye-contaminated water, the chemical industry, the food industry, catalysis, air and gas purifications and automobile industries, etc.



Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENYEDIAAN BAHAN NANO KARBON AKTIF DARIPADA TEMPURUNG ISIRONG SAWIT UNTUK PENYINGKIRAN ION LOGAM BERAT

Oleh

BABY RABIA

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Pencemaran alam sekitar banyak disebabkan oleh perindustrian dan juga manusia dan pencemaran air adalah salah satu bahaya utama untuk kehidupan. Banyak jenis bahan cemar telah dikaji seperti bakteria, virus, molekul organik, pewarna dan ion logam berat seperti Cr^{6+} , Pb^{2+} , Cd^{2+} , Zn^{2+} , As^{3+} , Cu^{2+} dan Hg^{2+} , dsb. Di antara semua ini, ion logam berat tidak boleh terbiodegradasi dan boleh terkumpul dalam tubuh manusia secara berterusan mengikut masa, yang mengakibatkan kesan buruk yang teruk seperti kerosakan sistem saraf, penyakit kulit, kerosakan hati, kegagalan buah pinggang, anemia, hepatitis, ulser dan kesan karsinogenik. Dalam kajian ini empat ion logam berat iaitu kromium (Cr^{6+}), plumbum (Pb^{2+}), kadmium (Cd^{2+}) dan zink (Zn^{2+}) yang merupakan bahan cemar air telah dipilih. Tempurung isirong sawit (PKS) telah dihaluskan menjadi serbuk dengan menggunakan pengisar elektrik dan seterusnya dirawat dengan kepekatan asid sulfurik yang berbeza untuk menghasilkan kumpulan berfungsi. PKS juga telah dirawat dengan asid fosforik untuk sintesis karbon teraktif diikuti dengan pengoptimuman masa dan suhu di dalam relau tiub elektrik dengan aliran gas nitrogen yang berterusan pada kadar alir $150 \text{ cm}^3/\text{minit}$. Karbon teraktif juga telah difungsikan lagi dengan menggunakan asid nitrik pada kepekatan berbeza yang menghasilkan karbon teraktif berfungsi (FAC). Kajian kelompok telah dijalankan untuk rawatan air tercemar logam berat dan parameter yang berbeza seperti pH larutan, dos penjerap, masa sentuhan, dan kepekatan ion logam telah dioptimumkan untuk setiap penjerap. Mereka menunjukkan pertalian penjerapan yang baik untuk ion logam berat Cr^{6+} , Pb^{2+} , Cd^{2+} dan Zn^{2+} . Penjerapan Cr^{6+} dan Pb^{2+} ditentukan untuk maksimum hampir mencapai 99% dalam kes setiap penjerap. Cd^{2+} yang penjerapannya didapati 80-85%, diikuti oleh 70-75% penjerapan ion Zn^{2+} dalam keempat-empat penjerap yang digunakan. Dua model isoterma iaitu Freundlich dan Langmuir dan tiga model kinetik iaitu pseudo-first-order, pseudo-resapan tertib kedua dan Parabolik digunakan untuk penentuan interaksi kinetik antara penjerap; (PKS, PKS-Sulfo, AC dan FAC) dan bahan penjerap; (Cr^{6+} , Pb^{2+} , Cd^{2+} dan Zn^{2+}). Penjerapan mengikut Pseudo Second Order dalam semua kes penjerap. Isoterma penjerapan Langmuir didapati diikuti dalam kes PKS-Sulfo, dan hanya penjerapan zink dalam PKS, selain daripada karbon Aactivated (AC, Karbon Aktif

Berfungsi (FAC) serta PKS didapati mengikuti model isoterma Freundlich. Kecekapan penjerapan di antara keempat-empat penjerap adalah mengikut urutan $FAC > AC > PKS-Sulfo > PKS$. Kaedah ini merupakan pendekatan yang lebih hijau yang dengan dwifungsi untuk perlindungan persekitaran daripada asap akibat pembakaran sisa PKS. Fungsi pertama ialah menukar PKS menjadi bahan berguna dan bukannya membakarnya dan kedua ia digunakan untuk rawatan air tercemar logam berat. Bahan-bahan yang direka bentuk boleh terus dieksploitasi untuk rawatan air tercemar pewarna, industri kimia, industri makanan, pemangkinan, penulenan udara dan gas dan industri automobil.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

Abbreviation	Full-Form
PKS	Palm Kernel Shell
PKS-Sulfo	Palm Kernel Shell-Sulfonated
AC	Activated Carbon
FAC	Functionalized Activated Carbon
XRD	X-Ray Diffraction
FTIR	Fourier Transformed Infrared
BET	Brunauer-Emmett-Teller
FESEM	Field Emission Scanning Electron Microscopy

CHAPTER 1

INTRODUCTION

1.1 Background of Studies

Pollution is termed as the presence of undesirable chemical entity/entities preventing the natural process or causing adverse effects on living organisms and the environment (Ayawei Nimibofa, 2018; Poorva Mehndiratta, 2013; Wu et al., 2019). Industrialization and the immense increase in population led to growing urbanization causing an increase in pollution at an alarming rate (Poorva Mehndiratta, 2013; X. Zhang et al., 2017). Improving the water, soil and air quality is an immense challenge of the modern era. Identification and treatment of environmental pollutants and their prevention is a key step in the protection of the environment. Pure and clean water is getting scarce due to industrialization, and the world is facing a shortage of clean water, especially in developing countries (World Health Organization (WHO) and the United Nations Children's Fund (UNICEF), 2017). Water contaminants can be organics, bacteria, viruses, dyes, and heavy metal ions such as lead, cadmium, zinc, nickel, arsenic, chromium and mercury with non-biodegradable nature posing a great risk to human health. Heavy metal ions can cause many adverse affects like cancer, kidney damage, hepatitis, miscarriages, anemia, encephalopathy and nephritic syndrome, etc. (Sardans et al. 2011; Tlili, 2018; Yang et al. 2018). Lead ions are released in the environment generally from metal mining industries of acid lead batteries, paper, glass and polishing industries. Cadmium is generally found in water discharged from the electroplating designing of batteries, photovoltaic cells, metallurgy processes, and fabric factories (Xiangtao Wang, 2012). Nickel ions can cause skin diseases when contacted with jewelry trashing, zips, watches, and coins, etc. Cr(VI) ions cause diseases like liver damage, nephritis, stomach distresses and a nasal mucous ulcer (Moreno-Castilla et al. 2004). Because of these severe adverse effects, the removal of heavy metal ions from water is of prime importance for saving human lives from such problematic health issues. Toxic metal ions could be removed by numerous methods, like ion exchange, reverse osmosis, precipitation filtration, biosorption, coagulation, and extraction, etc. (Fu & Wang, 2011; Huang et al., 2018). Adsorption is considered the best method as it is cost-effective, highly efficient, and easy to operate for removing trace levels of heavy metal ions (Czikkely 2018).

Nanotechnology plays a vital role in realizing the clean environmental goal and has progressed exponentially in the last decade (Culetu et al., 2014; Wu et al., 2019). Nanotechnology platform finds application almost in every field such as environmental science, health sciences, electronics, industrial separation, portable water treatment large/small scale plants, catalysts, energy storage, and energy generation, etc. (Liu et al., 2018; G. Zeng et al., 2018; G. Zeng et al., 2016). Nanomaterials provide a special platform for the purification of contaminated water due to their high surface area, their capability of chemical modification and easier regeneration. Nanomaterials are being exploited more and more for the removal of different types of pollutants namely organic, metal ions, biological contaminants, and arsenic, etc. from the water (Al-Senani & Al-Fawzan, 2018; Bozbaş & Boz, 2016; Bradder et al.2011; Brunet et al. 2009). Carbon

nanomaterials namely graphene, graphene oxide, carbon nanotubes, fullerenes, and activated carbons, etc. have been widely used in energy storage, sensor, electronics, water purification, drug delivery, and disease diagnosis, etc. owing to their exceptional chemical, mechanical, thermal and electrical characteristics. In this study (PKS) has been selected for the preparation of adsorbent materials namely PKS powder, functionalized PKS, activated carbon (AC) and functionalized activated carbon (FAC). Functionalization will confer on the more chemically active sites on the above material will enhance the adsorption efficiency of the functionalized material. And these adsorbent materials were successfully used for the treatment of heavy metal-contaminated water. Their kinetics as well as their isotherms studies were out in detail. The mechanisms of interactions of heavy metal ions with all these four-designed adsorbents (PKS, PKS-Sulfo, Activated carbon and Functionalised activated carbon) were determined. All of these four adsorbents had a lot of active sites for the interactions with heavy metal ions and therefore were found to be efficient in removing the heavy metal-contaminated water.

1.2 Problem Statement

Clean water is getting scarce because of industrialization, and the world is confronting a deficiency of clean water. Among the different water contaminants, heavy metals are non-biodegradable and they get accumulated in the body over time and result in severe illness and disorders in humans as well as in other living creatures. Consumption of heavy metal-contaminated water or food can cause numerous health problems like a malignant growth, kidney damage, hepatitis, unsuccessful labor, iron deficiency, encephalopathy, nephritic disorder, and so forth (Anubhav et al., 2022; Rehman et al. 2018). Chromium metal (Cr^{6+}) can cause maladies like liver harm, nephritis, stomach problems and nasal mucous ulcers (Jacobs 2005). The concentration of Cr^{6+} in groundwater and surface water is exceeding World Health Organization (WHO) set limit of 50 μg per liter (Velma, 2009). Lead (Pb^{2+}) is a carcinogenic element declared by the Environmental Protection Agency (EPA). Lead poisoning is the term used for its toxicity and it may be acute or chronic. Lead poisoning can cause mental retardation, birth defects autism, allergies, dyslexia, paralysis, brain damage, kidney damage and may also result in death etc. (Martin, 2009). Cadmium-induced toxicity to kidneys, respiratory systems, and skeleton and is a carcinogen to humans (Monisha Jaishankar, 2014; Waalkes M, 2001). Cadmium is ranked 7th most toxic metal by the Agency for Toxic Substances and Disease Registry (ATSDR) (Jaishankar et al. 2014). Although zinc is essential to trace metals for humans, excessive absorption of zinc can suppress iron absorption. Zinc ions are highly toxic to plants, vertebrates, fishes and invertebrates, etc. (Brita, 2006; Fosmire et al., 2003).

Agricultural waste of oil palm is produced in millions of tons from Malaysia, Indonesia, Cameron, Africa, China and Nigeria (Nicholas et al. 2018). Malaysia is among the biggest producer and suppliers of oil palm with about 4.5 million hectares of cultivation and produces about 90×10^6 tons of agricultural waste oil palm (PKS) is a sustainable source and usually, it is burned to dispose of causing the greenhouse effect and resulting in a negative impact the environment (Nicholas et al., 2018).

Different methods are widely applied for the removal of heavy metal ions from the aqueous solutions namely reverse osmosis, evaporation, colorimetric, ion exchange, precipitation, membrane and coagulation (Bradder et al., 2011; Cimxe, 2019; Hung et al., 2014; Wu et al., 2019). Because of the high cost, energy consumption and low concentration of metal ions are problems in the above techniques.

1.3 Objectives of the Study

The following are the key objectives of this research study.

- A). To prepare the four different adsorbents Palm Kernel Shell (PKS) Powder, Functionalized PKS powder, Activated Carbon from PKS and Functionalized activated Carbon using physical blending, chemical method of oxidation, carbon vapour deposition method followed by chemical oxidation respectively.
- B). To optimize the preparation parameters of the above four adsorbents to get the best samples and their detailed characterization.
- C). To perform adsorption study for the removal of heavy metal ions namely Cr^{6+} , Pb^{2+} , Cd^{2+} , and Zn^{2+} from the water using the prepared adsorbents.
- D). To optimize the adsorption parameters; the pH of the solution, concentration of metal ions, contact time and the adsorbent dosages.
- E). To analyze the kinetics studies using the models of pseudo-first-order, pseudo-second-order and parabolic-diffusion-order and isotherm models namely Freundlich and Langmuir, to study the kinetics and mechanism of interactions between metal ions and adsorbents.

1.4 Significant of studies

In this study, agricultural waste of Palm Kernel Shell (PKS) which is produced in millions of tons every year as an agricultural waste of oil palm upstream industries is produced in Malaysia, Indonesia, Cameron, Africa, China and Nigeria. Malaysia is among the biggest producer and suppliers of oil palm with about 4.5 million hectares of cultivation and produces about 90×10^6 tons of agricultural waste oil palm. Most commonly the waste is disposed of by burning, causing a lot of smoke and haze resulting in a negative impact on the environment. The haze and smoke from the burning of the oil palm waste result in different eye diseases, skin diseases, and causing lung diseases.

On other hand, heavy metals are non-biodegradable environmental contaminants coming into the environment and water from industrial waste. Consumption of these heavy metals by living creatures from water heavy metals are also the main pollutants in groundwater.

In this study, agricultural waste of oil palm PKS was utilized for the preparation of the different adsorbents namely PKS Powder, Sulfonated PKS powder, Activated carbon (AC), and Functionalized activated carbon (FAC). The designed Adsorbents were successfully applied for the treatment of heavy metal-contaminated water through the adsorption process. The adsorption process is economical, more effective and user-friendly (easy to use) compared to the other water treatment methods such as reverse osmosis, evaporation, colorimetric, ion exchange, precipitation, membrane and coagulation. The designed materials have the potential to be applied for the treatment of other water pollutants such as dyes and bacteria, etc. This study is a greener approach for the conversion of agricultural waste into useful advanced material and it will serve dual purposes; protecting the environment from the smoke resulting from the burning of PKS and treatment of heavy metal-contaminated water. This study opens up new horizons for greener nanotechnology where other agricultural waste can also be converted into useful materials.

1.5 Scope of the Study

The focus of this study is to use the waste from the oil palm upstream industry for environmental remediation, firstly by converting the agricultural waste into useful advanced material. Agricultural waste from oil palm plantations is produced in millions of tons every year which can serve as a sustainable material. Most of them are dumped by burning them off causing haze, and environmental pollution and resulting in health issues for living beings.

In this study, the shell (PKS) of the oil palm was grounded into fine powder for adsorption of heavy metal ions and selected to convert it into useful advanced material namely activated carbon using the technique of carbon vaporized depositor (CVD) and was further functionalized with nitric acid and sulfuric acid resulting in the functionalized activated carbon. Different parameters of activated carbon synthesis concentration of activating agent (phosphoric acid), temperature and holding time were optimized. These parameters were optimized to get the activated carbon with a high specific surface area. The best sample, i.e. the sample with the highest surface area was selected for further functionalized with nitric acid and sulfuric acid to get the nitro- and sulfo- functionalized activated carbon. In addition, the PKS was also directly functionalized with sulfuric acid, which resulted in PKS-Sulfo. This is a green approach for environmental remediations where agricultural waste has been converted to useful materials, namely PKS powder, PKS-Sulfo, activated carbon and functionalized activated carbons; instead of burning them off, this approach immensely contribute to avoiding air pollution. Furthermore, the designed useful materials have been applied for the treatment of heavy metal-contaminated water. The different parameters of the adsorption study; pH of the solution, contact time, adsorbent dosage and heavy metal ions solutions were varied to get the optimized condition for the maximum removal of heavy metal ions. This research serves dual purposes; first converting agricultural waste into useful and advanced materials and secondly, it also offers a solution for the treatment of heavy metal-contaminated water. Generally, this work only covers the use of PKS for the preparation of activated carbon and its functionalized products and their treatments for the removal of heavy metal ions from water.

The novelty of this study is that a waste material of agriculture produced in millions of tons every year causing a lot of environmental pollution due to burning off for its disposal; has been converted into four different advanced useful materials. Not only this, but this material is also further utilized for environmental remediation for the removal of heavy metal ions from water. Therefore, this study serves a dual purpose conversion of waste material to use advanced materials and applying them for further protection of the environment.

The limitations of this study are, the designed materials can be applied for the removal of the dyes from water. Furthermore, these can also be used as a stationary phase of the glass columns for the removal of contaminants from water both dyes as well heavy metal ions. Recycling of adsorbents can also be done and reuse of the material for further experiments can be done which was not done due to limit access to laboratories due to covid-19 MCO. we have not conducted the antimicrobial and antifungal studies of design materials but we do expect these activities from prepared material.

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