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Synthesis and Characterization of Activated Carbon from Dabai *(Canarium odontophyllum)* Nutshell for Removal of Water Pollutants

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Synthesis and Characterization of Activated Carbon from Dabai (*Canarium odontophyllum*) Nutshell for Removal of Water Pollutants

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DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Malaysia Sarawak. Except where due acknowledgements have been made, the work is that of the author alone. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

…………………………………………

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ABSTRACT

This research is conducted to explore the potential of utilizing dabai nutshell as the precursor for the purpose of synthesizing biochar and activated carbon (AC) for the purpose of removing water pollutants. Dabai nutshell is commonly thrown away as waste instead of being utilized for industrial purpose. Previous studies on chemical activation of the biomass do not meet the required properties of the AC, and do not include the optimization of carbonization temperature. The potential of the AC from dabai nutshell for removal of heavy metal ions are not explored in prior studies. Inert gases and complex equipment set up to provide oxygen-less atmosphere for the synthesis of AC are not available in rural areas, thus further studies are required to determine the alternatives for the inert gas. This research aimed to synthesise the AC from Dabai nutshell using chemical activation and carbonization (both processes are without inert gas flow) and characterize it using several analytical techniques. This work also studies on the effect of different chemical activating agents with different concentration on the synthesis of AC, to optimize the carbonization temperature and to apply the AC on removal of selected water pollutants. The dabai nutshell underwent physical treatment to clean, dry and reduce its size down to 1 mm. The biomass then carbonized under different atmosphere, and at different temperature, in order to perform optimization study for the process's temperature. The resulting biochar is then activated using sodium hydroxide (NaOH), potassium chloride (KCl) or hydrochloric acid (HCl). The synthesized AC were then characterize using several analytical methods and applied on removal of dye methylene blue and heavy metal ions (zinc, chromium, and lead). The results showed that the biochar is dominated by carbon and oxygen, and the percentage yield and ash content of the biochar are more than 26% and less than 4.00%, respectively. The nitrogen adsorption isotherm of the biochar is similar to Type I of IUPAC classification, and biochar is observed to have the surface area more than 370 m^2/g. The optimization of the carbonization temperature on synthesizing biochar that met the set criteria concluded that 721.469 °C is the optimum temperature. The biochar derived without inert gas flow during carbonization have comparable properties with the biochar derived under nitrogen and argon gas flow. AC derived through chemical activation using NaOH have comparable BET surface properties with commercial AC, with micropores properties, BET surface area of more than 328 m^2/g, and total pore volume of more than 0.194 m^3/g. The nitrogen adsorption isotherms are similar to Type II isotherm of the IUPAC categorization across all replicates. AC replicates derived through HCl and KCl activation are dominated with carbon and oxygen and have ash content less than 3%. All AC replicates however are having low MB adsorption uptake, lower than the standard (60-120 mg/g), and the kinetic study of MB adsorption reveals that the most suited model to describe the MB adsorption on the AC replicates are pseudo-second-order model and Elovich kinetic model. All AC replicates have more than 35% removal of heavy metal ions with initial concentration of 30 ppm. These findings conclude that dabai nutshell are suitable to be utilized as precursor for AC, with carbonization and chemical activation are performed on the precursor without the use of inert gas flow. The resulting AC also performed excellent adsorption to reduce the concentration of heavy metal ions.

Keywords: Dabai nutshell, chemical activation, activated carbon, adsorption, carbonization temperature, optimization, dye methylene blue, heavy metal ions.

Sintesis dan Perwatakan Karbon Diaktifkan daripada Kulit Kacang Dabai (Canarium odontophyllum) untuk Penyingkiran Bahan Pencemaran Air

ABSTRAK

Kajian ini dilaksanakan bagi mengenali potensi penggunaan kulit kacang dabai sebagai bahan asas penghasilan bio-arang dan karbon teraktif (AC) untuk tujuan penyingkiran bahan pencemar air. Kulit kacang dabai sering dibuang dan tidak dimanfaatkan untuk tujuan industri. Kajian terdahulu ke atas pengaktifan kimia pada biojisim berkenaan tidak menepati ciri AC, dan tidak merangkumi pengoptimuman suhu pengkarbonan. Kajian terdahulu juga tidak mengenali potensi AC daripada kulit kacang dabai untuk penyingkiran ion logam berat. Gas lengai dan kelengkapan peralatan yang kompleks bagi menghasilkan keadaan tanpa oksigen untuk penghasilan karbon teraktif juga tiada di kawasan pedalaman, maka kajian selanjutnya diperlukan untuk menemui alternatif kepada gas lengai. Kajian ini bertujuan untuk menghasilkan AC daripada kulit kacang Dabai melalui pengaktifan kimia dan pengkarbonan (kedua-dua proses dilaksanakan tanpa aliran gas lengai) dan mencirikan AC berkenaan melalui beberapa kaedah analitikal. Kajian ini juga akan meneliti kesan penggunaan agen pengaktifan kimia yang berbeza, dengan kepekatan yang berbeza ke atas penghasilan AC, mengoptimumkan suhu pengkarbonan dan menggunakan AC berkenaan dalam penyingkiran bahan pencemar air yang terpilih. Kulit kacang dabai terlebih dahulu melalui rawatan fizikal, dengan membersih, mengering dan mengurangkan saiz sehingga 1 mm. Biojisim berkenaan kemudian dikarbonkan di bawah keadaan atmosfera yang berbeza, dan suhu yang berbeza bagi melaksanakan kajian pengoptimuman ke atas suhu proses berkenaan. Bio-arang yang telah dihasilkan kemudiannya diaktifkan menggunakan natrium hidroksida (NaOH), kalium klorida (KCl) atau asid hidroklorik (HCl). AC yang telah dihasilkan kemudiannya dicirikan *melalui beberapa kaedah analitikal dan digunakan untuk penyingkiran pewarna metilena biru dan ion logam berat. Keputusan dari kajian ini menunjukkan bahawa bio-arang yang terhasil didominasi oleh karbon dan oksigen, dan peratusan hasil dan kandungan abu pada bio-arang masing-masing adalah 26% dan kurang daripada 4.00%. Isoterm penjerapan nitrogen bagi bio-arang ini adalah serupa dengan Jenis I dalam klasifikasi IUPAC, dan bio-arang diperhatikan mempunyai luas permukaan melebihi 370 m^2/g. Pengoptimuman untuk suhu pengkarbonan yang menepati kriteria yang telah ditetapkan menyimpulkan bahawa 721.469 °C ialah suhu optimum untuk proses berkenaan. Bio-arang yang dihasilkan tanpa menggunakan aliran gas lengai mempunyai ciri-ciri yang setanding dengan bio-arang yang dihasilkan menggunakan aliran gas nitrogen dan argon. AC yang dihasilkan melalui pengaktifan kimia menggunakan NaOH adalah setanding dengan AC komersial, dengan mempunyai ciri-ciri mikropori, luas permukaan BET melebihi 328 m^2/g dan jumlah isi padu pori melebihi 0.193 m^3/g. Isoterm penjerapan nitrogen bagi kesemua replika AC berkenaan adalah serupa dengan Jenis II dalam klasifikasi IUPAC. Replika AC yang dihasilkan melalui pengaktifan HCl dan KCl didominasi oleh karbon dan oksigen, dan mempunyai kandungan abu kurang daripada 3%. Namun, kesemua replika AC mempunyai penjerapan MB yang rendah, lebih rendah daripada standard (60-120 mg/g) dan kajian kinetik terhadap penjerapan MB mendapati model yang paling sesuai menjelaskan penjerapan MB ke atas kesemua replika AC adalah model pseudo-urutan-kedua dan model kinetik Elovich. Kesemua replika AC telah mengurangkan sekurang-kurangnya 35% daripada kepekatan asal (30 ppm) ion logam berat. Dapatan ini menyimpulkan bahawa kulit kacang dabai sesuai digunakan sebagai bahan asas untuk AC, dengan pengkarbonan dan pengaktifan kimia dilakukan pada bahan asas tanpa menggunakan aliran gas lengai. AC yang terhasil* *juga melakukan penjerapan yang sangat baik untuk mengurangkan kepekatan ion logam berat.*

Kata kunci: *Kulit kacang dabai, pengaktifan kimia, karbon teraktif, penjerapan, suhu karbonisasi, pengoptimuman, pewarna biru methylene, ion logam berat*

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LIST OF ABBREVIATIONS

AC	Activated Carbon
ASTM	American Society for Testing and Materials
BET	Brunauer-Emmett-Teller
COD	Chemical Oxygen Demand
EDX or EDS	Energy Dispersive Xray Spectroscopy
FTIR	Fourier Transform Infrared Spectroscopy
HCl-AC	AC derived though hydrochloric acid, HCl activation
ID	Intra-particle diffusion kinetic model
IUPAC	International Union of Pure and Applied Chemistry
KCl-AC	AC derived though potassium chloride, KCl activation
MB	Methylene Blue
NaOH-AC	AC derived though sodium hydroxide, NaOH activation
PFO	Pseudo-first-order adsorption kinetic model
PSO	Pseudo-second-order adsorption kinetic model
SEM	Scanning Electron Microscope

LIST OF NOMENCLATURE

H_3PO_4	Phosphoric acid
HCl	Hydrochloric acid
KCl	Potassium Chloride
KOH	Potassium hydroxide
NaOH	Sodium Hydroxide

CHAPTER 1

INTRODUCTION

1.1 Study Background

Responsibility towards curing Mother Earth from pollution is one of the major concerns worldwide. Reducing the various pollutants through numerous methods are necessary for each nation on this planet, which induce countless efforts to explore all resources and procedures available. Current development in the combat against water pollution includes the physical treatment such as sedimentation and filtration, and chemical treatments such flocculation, coagulation, adsorption, and ion exchange methods (Saravanan et al., 2021). For adsorption, one of the common methods being deployed is the utilization of highly porous activated carbon.

Ukanwa et al. (2019) stated that activated carbon (AC) is a carbonaceous material produced by thermal or thermochemical processes from coal or biomass. ACs are distinguished by their well-developed pore morphology, extraordinary surface area, electron-conducting amphoteric tendencies, and high adsorptive capacity (Pezoti et al., 2016). The activated carbon has a variety of uses in the treatment of wastewater, including the removal of metal ions and dye from aqueous solutions as well as the sterilization of drinking water. (Ioannidou & Zabaniotou, 2007).

Conventionally, AC is developed using coal, wood and coconut shells as described by Crini et al. (2019). Non-conventional AC are synthesized from solid biomass waste containing high percentage of carbon, and industrial byproducts such as sewage sludges and waste tires. Utilization of solid biomass such as date stones, palm kernel husks, nutshells and palm fruit bunch is studied thoroughly to determine the suitability of the raw materials as the precursor in AC production (Crini et al., 2019).

One of the most underutilized lignocellulosic wastes in Borneo is Dabai (*Canarium odontophyllum*). The dabai fruit, leaves, and stem (in terms of extract), is proven by various studies to have pharmaceutical advantages, ranging from potential diabetic medication to cancer treatment (Fredalina Basri, 2015; Hamzah et al., 2022). According to Kasron et al. (2020), in 2018, Dabai had the greatest crop area and led overall indigenous fruit production in Malaysia, accounting for 29% of land area and 15% of production. However, it is different for the nutshell of the fruit, where this material is usually thrown away after its flesh being consumed, and very little research is made on the nutshell in order to determine its value. Dabai nutshell, for instance, has a lot of undiscovered value since it could potentially be used as a precursor in the production of activated carbon. Some folks like to boil and dry the nutshell before eating the nut within. The nutshell is very hard, where it requires nutcrackers to open it, and heavy-duty grinder to further crush it into powders. The biomass also took long time to be decomposed naturally. By looking at the possibility of using the Dabai nutshell as raw material for the synthesize of AC, it could open up fresh opportunities for using Dabai for commerce., instead of just being thrown away as waste. Additionally, this will result in a significant increase in the number of available positions for labour. Furthermore, the expansion of Dabai tree plantation areas would bring value to the state economy, particularly through the use of Dabai by-products as a precursor to the production of activated carbon. These advantages will increase the value of agricultural goods and enable a broader range of low-cost solutions to water pollution (Ioannidou & Zabaniotou, 2007).