Faculty of Engineering

SYNTHESIS OF SAGO HAMPAS ACTIVATED CARBON AEROGEL (SHACA) FOR SAGO WASTEWATER (SWW) TREATMENT

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REMOVAL OF HEAVY METAL IN SOLUTION BY SAGO HAMPAS ACTIVATED CARBON AEROGEL AS AN ADSORBENT

MADELIN ANAK DEMONG

A dissertation submitted in partial fulfilment

of the requirement for the Degree of

Bachelor of Engineering with Honours

(Chemical Engineering)

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Dedicated to my beloved parents, who always support, giving motivations and encourage me throughout this research study

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ABSTRACT

Sago hampas is well known with its special component in treating waste water effluent. An alternative was made on this raw material to transform sago hampas into activated carbon Aerogel. Aerogel can be produced from a wide range of raw materials of agricultural origin and it has a wide of usage, which includes as an adsorbent that act as medium in adsorbing organic pollutant content in waste water. These technologies can reduce the waste especially produced from the agricultural activities such as straws, bagasse, pith, and others. Apart from that, a low-cost and eco-friendly product can be generated, hence, minimizes the hazardous. Activated carbon Aerogel is found to be highly effective product to remove organic pollutants. Two techniques of drying were used which are pyrolysis and freeze-drying that give different properties in the activated carbon aerogel from sago hampas analysed by FTIR, FESEM, BET and particle size analyser (PSA). Samples were used to treat sago wastewater (SWW). There are three types of organic pollutant that is remove from the SWW and measurements were taken in terms of Chemical Oxygen Demand (COD), turbidity and Total Suspended Solid (TSS). The findings prove that freeze-drying give the best the Aerogel adsorbent that removed pollutants up to 80%. Meanwhile, the product from pyrolysis has less performance in the of pollutants. Results shows also showed that the higher the dosage of aerogel used, the more efficient the removal performance. However, tested for smaller particle size of adsorbent showed lower performance compared to those observed for particle sizes at 1.5 cm and 1mm. Outcomes of this wor indicates the potential of converting sago hampas become aerogel for wastewater treatment.

Keywords: activated carbon, Aerogel, SWW, pyrolysis, freeze-drying COD, turbidity, TSS

ABSTRAK

Hampas sago sangat dikenali dengan keistimewaan komponennya dalam merawat air sisa. Satu alternatif telah dibuat dari bahan ini dengan mengubahnya menjadi Aerogel. Aerogel boleh dihasilkan dari pelbagai jenis bahan mentah asal pertanian dan ia mempunyai banyak penggunaannya termasuk sebagai penjerap yang bertindak menjadi medium untuk menyerap logam berat yang terkandung dalam air sisa. Teknologi ini dapat mengurangkan sisa yang dihasilkan terutama dari aktiviti pertanian seperti jerami, tebu, pith, dan lain-lain. Selain itu, produk kos rendah dan mesra alam juga dapat diperbaharui kerana ia meminimumkan teknologi yang mengandungi bahan merbahaya dan berisiko tinggi dalam aplikasi rawatan air sisa. Dua teknik telah diguna pakai iaitu pirolisis dan pengeringan beku yang memberi perbezaan ketara didalam ciri struktur carbon aktif Aerogel sampel dibuktikan dengan FTIR, BET dan partikel saiz analisis. Selanjutny, sample tersebut digunakan untuk merawat air sisa sago. Tiga jenis pencemaran sisa organik akan dirawat iaitu COD, kekeruhan dan TSS. Keputusan menunjukkan teknik pengeringan beku memberi sample carbon aktif Aerogel (SHACA 2) yang terbaik dalam merawat sisa organik dengan jumlah purata melebihi 80%. Manakala, sample SHACA 1 yang menggunakan teknik pirolisis, memberi prestasi merawat sisa organik dengan purata kurang daripada 20%. Beberapa faktor yang mempengaruhi perawatan tersebut iaitu dos carbon aktif dan saiz sample. Keputusan menunjukkan semakin tinggi dos yang dipakai, semakin meninggi prestasi perawatan tersebut. Walau bagaimanapun, untuk saiz sample terdapat had saiz yang kecil untuk perawatan. Saiz yang kecil daripada 400 mikro meter tidak memberikan prestasi yang baik kerana ia menyumbang kepada pencemaran air sisa daam bentuk kekeruhan dan TSS. Hasil dari penyelidikan ini menunjukkan potensi menukar sagu hampas menjadi aerogel untuk rawatan air sisa

Kata kunci: karbon aktif, Aerogel, air sisa sago, pirolisis, pengeringan beku COD, kekeruhan, TSS

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CHAPTER 1

INTRODUCTION

This chapter introduced the overview of the biomass waste activated carbon Aerogel production. Research aim, objectives and scope of study are briefly explained in the following section. The usage and methodology of biomass waste as an adsorbent is also discussed in the aspect of waste water treatment application.

1.1 Research Overview

Biomass waste activated carbon is well known in the application of water treatment. Apart from low cost production, their adsorption effectiveness has been proved in many research studies. These ACs can be derived from any potential raw materials that depends on the physical and chemical methods. Due to their beneficial products, these ACs are further modified to form an Aerogel which has more unique properties. Aerogel at first was used in engineering technology such as insulation, electric components, encapsulation medium and others. The adsorbent potential is demonstrated in the chemical properties of the Aerogel with a high total surface area and a very high porosity volume. The Aerogel 's structured network has a good encapsulation of micro and nano particles. Apart from that, Aerogels are well performed in waste water treatment as an adsorbent or generally known as a solution filter. By having unique physical and chemical properties, the aerogel has the potential in adsorbed waste water contaminant such as organic pollutant, heavy metals, oils and other chemicals pollutions. These aerogels can be derived from any raw materials or precursors which will generate varies of physical and chemical properties.

In this study, a production of activated carbon aerogels from sago waste is proposed to study the efficiency of adsorbing organic pollutants in sago waste water. The sago waste or sago hampas is used as raw material because the waste production is excessive especially in Sarawak area. Further, sago waste still contained beneficial characteristics that can be fully applied as an adsorbent. Biomass waste from agriculture crops is more applicable in science and technology nowadays due to biodegradable and eco-friendly products. Instead of disposing the biomass waste naturally, an initiative of converting these wastes into more valuable products is safer and environmentally sustainable. This alternative can help to reduce the waste especially produced from the agriculture activities. So, this waste is suitable to be converted into carbon Aerogel.

The product which is sago waste activated carbon Aerogel is then used to adsorb organic pollutant in waste water from industry effluent or agriculture waste water. The adsorption capacity is studied by several parameters, such as dosage of adsorbent and particle size of adsorbent. Therefore, choosing a suitable activation treatment and synthesised technique will determine the adsorption effectiveness in terms of removal of organic pollutant performance.

1.2 Problem Statement

In Malaysia, especially in the state of Sarawak, the sago industry is well established and becomes one of the major industries exporting between 25,000 and 40,000 tons of sago products (Tek-Ann et al,1998). The wastes generated from sago hampas processing is huge. This excessive sago waste is unmanageable and some of the wastes is dumped to nearby rivers. Sago hampas is one kind of sago waste, extensive fibrous by-products from the rasped pit of the sago palm after the extraction of sago starch. Some of sago hampas is traditionally used for animal feeding, become a compost for mushroom cultivation and for particleboard, but the production of sago hampas exceeds the demand for this traditional usage. The fiber residue from sago waste is made up of celluloses and lignins that have the potential as a bio-adsorbent as well as Aerogel a commercially applied as thermal insulation, drug delivery, catalysts and carrier materials for catalysis and electrocatalysis as well as biocatalysis (Smirnova, 2018) .Hrubesh (1998) stated that, Aerogel is a promising adsorbing media applied in wastewater treatment. Hence, converting sago hampas to AC or Aerogel will not only reducing the impact of improper disposal of sago hampas, but also create business opportunity for the local sago processing entrepreneurs.

1.3 Aim and Objective

The research study aims to produce an Aerogel (ACA) carbon activated from biomass waste for wastewater treatment. Production of the carbon Aerogel as an adsorbent is depending on the chemical and physical preparation method. The research objective are as follows:

i. To evaluate the process of converting sago 'hampas' into activated carbon Aerogel (SHACA) by using pyrolysis and freeze-drying techniques.
ii. To study the characteristics of SHACA samples and its performance to adsorb organic pollutant (COD, turbidity and TSS)
iii. To study the kinetic of pollutants adsorption in terms of COD, turbidity and TSS reduction.

1.4 Scope of Study

In this study, the procedure SHACA was produced by using freezing-drying and pyrolysis techniques. Temperature range used was -40 °C for freezing-drying while 700 - 750 °C for pyrolysis. The characteristics of samples were determined using Fourier-Transform Infrared Spectroscopy (FTIR), Field Emission Scanning Electron Microscopy (FESEM), Brunauer, Emmett and Teller (BET) and particle size analyser (PSA). Subsequently, the performance of the Aerogel sample in absorbing pollutants was studied in terms of Chemical Oxygen Demand (COD), turbidity and Total suspended Solid (TSS). The experimental study is including evaluation on the contact time and adsorbent dose to performance of the Aerogel to treat wastewater.

1.5 Summary

This chapter introduces the project done that includes the aim and objectives of this research, and brief description on the procedure used. The project scope describes briefly the work included in this research, the techniques and equipment used as well as the parameters evaluated.

CHAPTER 2

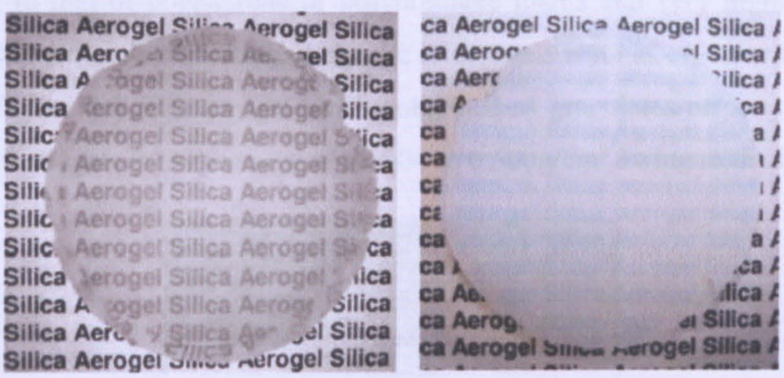
LITERATURE REVIEW

This chapter presents information to produce an Aerogel from biomass waste, various relevant aspects to the process, aerogel characteristics, application, preparation method and the raw materials. Apart from that, the kinetics study for the adsorption was also included based on the fundamental theory of adsorption and the relevant models used. At the end of this chapter, a summary on procedures for the experimental work is presented based on overall literature reviews.

2.1 Aerogel

Aerogel is a sponge-like structure gel derived from the nanostructures less than 1 to 100 nanometers pores in diameter, and densities ranging from 0.001 to 0.5 g/cm^3. The liquid in the gel is replaced by gas phase during drying to form a solid with extreme properties. The first aerogel was discovered by Samuel Stephens Kistler in late 1930s where an experiment that replaced the liquid phase with the gas phase was done on jellies without causing shrinkage was successfully accomplished, and in 1940, aerogel was commercially marketed. Since then, more research was developed to discover its usage for new technology in science and engineering field. Throughout the following years, aerogel filled the interest in chemist and engineers as its usage is more widely known. This new material is a promising solution to the environmental issues as new advance nanotechnology. There are several types of aerogels that depend on the precursor to create the aerogel with specific individual properties. Carbon, cellulose and polymer as shown in **Figure 2.1**, are the most common aerogel synthesized as they are applied in most of new technology recently and others are oxides, starch and any chemical that can be gelled. **Figure 2.2** represents silica aerogel appearance with different raw materials during synthesis. Different aerogels exhibit varies of physical and chemical properties depend on its precursor which will be discussed in next sub-chapter.

Figure 2.1. Different type of transition metal oxide aerogel (AEROGEL.ORG, 2018).



(a) (b)

Figure 2.2. (a) transparent-made (b) opaque-made Silica aerogels with different raw materials. (Ratke, 2011)

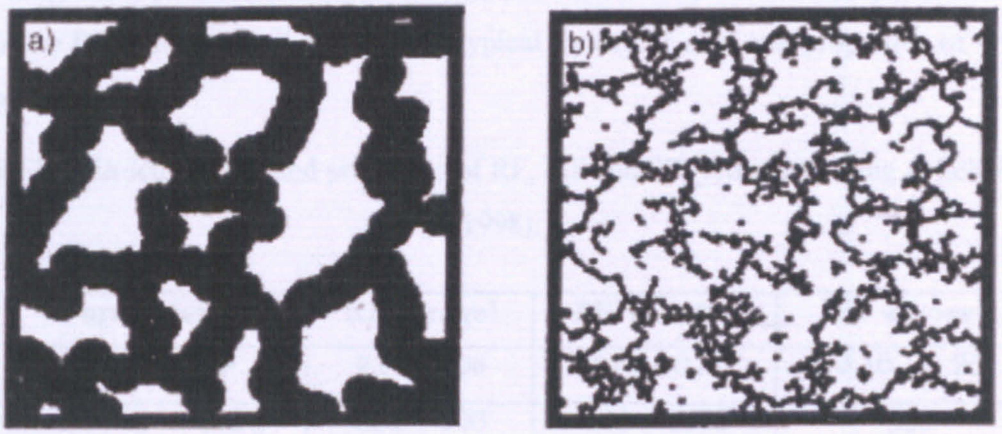
2.2 Characteristic of Aerogel

Aerogel is acknowledged with its porous material, ultra-low density, large specific surface area as well as other physical and chemical properties. With high volume porosity range of 80 to 99.8%, ultra-low density as low as 0.003 to 0.500 g.cm^{-3}, and large specific surface area, 100 to 1600 m^2 per gram, the gel is in the dried gel phase (Lin-Yu, Yun-Xuan, & Yu-Zhong, 2018). Aerogels characteristics depend on the performance of the aerogel types and there are two main types of aerogels, organic and inorganic aerogels. The combination of both aerogels generates the hybrid inorganic-organic aerogels which is depending on the precursor and processing method. One example of inorganic aerogel is SiO2, where it looks like glass with a high transparency, a thermal conductivity corresponding to that of polystyrene or polyurethane foams and very high surface areas. (Husing & Schubert, 1998). The unique feature of aerogels is it has effective heat insulation as shown in **Figure 2.3**. The aerogel can be obtained as granulates or powders where the bulk density of aerogels is in the range of 0.0004 to 0.500g/cm^3 owing to the high porosity.

Figure 2.3. Silica Aerogel shows excellent insulating properties (Husing & Schubert, 1998)

Theoretically, the three-dimensional structure of aerogel indicates that all the covalent bonded gels are between colloidal and polymeric gels, resulting the properties as shown in **Figure 2.4**. The three-dimensional network was formed by connecting primary particles or known as clusters. As shown in **Figure 2.5(a)**, dense colloidal particles are interconnected like string pearls in which linear or branched polymer chains form through the condensation of small polymer gels clusters, as shown in **(b)**. Husing and Schubert

(1998) claimed that each of the microstructure aerogel is strongly depends on the preparation methods. Due to the differences in the structure of primary and network formation caused by the variability of the reaction mechanism, SiO2 aerogel 's macroscopic properties differ widely. Thus, SiO_2 properties was listed in **Table 2.1** as mentioned in Husing and Schubert (1998) research study.



(a) (b)

Figure 2.4. Two-dimensional aerogel structures: (a) colloidal, (b) polymeric.

Table 2.1. Structural properties of SiO_2 aerogels.

Property	Range	Typical value
Bulk density, g/cm^3	0.003 – 0.500	0.100
Skeletal density, g/cm^3	1.700 – 2.100	-
Porosity, %	80 – 99.8	-
Mean pore diameter, nm	20 – 150	-
Inner surface area, m^2/g	100 – 1600	60
Refractive index	1.007 – 1.24	1.02
Thermal conductivity, λ (in air, 300 K), W/mK	0.017 – 0.021	1.02
Modulus of elasticity, E, MPa	0.002 – 100	1
Sound velocity, c_L, m/s	<20 – 80	100
Acoustic impedance, Z, kg/m^{2}s	-	10^4