

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

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Bachelor of Engineering with Honours (Chemical Engineering) 2019

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## **APPROVAL SHEET**

This final year report which entitled "<u>Synthesis Of Sago Hampas Activated Carbon</u> <u>Aerogel (Shaca) For Sago Wastewater (Sww) Treatment</u>" was prepared by Madelin Demong (50580) as a partial fulfilment for the Degree of Bachelor of Chemical Engineering is hereby read and approved by:

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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)

Faculty of Engineering

Universiti Malaysia Sarawak

2019

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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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## **TABLE OF CONTENTS**

	Pages
ACKNOWLEDGEMENT	vi
ABSTRACT	vii
ABSTRAK	viii
TABLE OF CONTENTS	ix
LIST OF TABLES	xii
LIST OF FIGURES	xiv

### CHAPTER 1 INTRODUCTION

1.1	Research Overview	1
1.2	Problem Statement	2
1.3	Aim & Objectives	3
1.4	Scope of Study	3
1.5	Summary	3

### CHAPTER 2 LITERATURE REVIEW

2.1	Aerogel	4
2.2	Characteristics of Aerogel	6
2.3	Application of Aerogel	12
2.4	Preparation of Aerogel	14
	2.4.1 Freeze drying method	14

		2.4.2 Supercritical Drying	15
	2.5	Activated Carbon (AC) for water treatment	18
	2.6	Availability of biomass material in Malaysia	20
	2.7	Sago Palm Processes and Waste	21
	2.8	Bio-conversion of sago waste to an adsorbent	25
	2.9	Summary	35
CHAPTER 3	MET	THODOLOGY	
	3.1	Material and Apparatus	36
	3.2	Experimental Procedure	37
		3.2.1 Preparation of Sago Hampas Activated	37
		Carbon Aerogel (SHACA)	
		3.2.2 Aerogel Sample Characterization	39
		3.2.3 Aerogel for Heavy Metal Adsorption Studies	40
	3.3	Summary	42
CHAPTER 4	RES	ULTS AND DISCUSSIONS	
	4.1	Introduction	43
	4.2	Synthesis of Sago Hampas Activated Carbon and	43
		Aerogel (SHACA)	
		4.2.1 SHACA Characteristics	45
	4.3	Sago Wastewater Treatment	51
		4.3.1 Turbidity Analysis	51
		4.3.2 Total Suspended Solid (TSS) Analysis	53

		4.3.3	Chemical Analysis	Oxygen	Demand	(COD)	54
	4.4		Factor Affe Treatment	ecting the S	Sago Waste	water	56
		4.4.1	Particle Siz	e of SHAC	CA		56
		4.4.2	Dosage of	SHACA			60
	4.5		Correlation	of Turbid	ity, TSS and	d COD	63
	4.6		Kinetic Stu Models	dy of Line	ar Isotherm		65
CHAPTER 5	CON	NCLUSI	ONS AND	RECOMM	IENDATI	ONS	
	5.1	Conclu	sion				70
	5.2	Recom	mendations				73
BIBLIOGRAPH	Y						74

.

## LIST OF TABLES

Table		Page
2.1	Structural properties of SiO2 aerogels	8
2.2	Structural data and properties of RF, MF and PF Aerogel	9
2.3	Structural data and properties of RF, MF and PF Aerogel	12
2.4	Typical fluids critical point parameters	16
2.5	Aqueous phase of active carbon preparation using non - conventional waste	19
2.6	Sago palm growing area in different country	22
2.7	Proximate analysis of sago effluent and 'hampas'	25
2.8	Properties of the sago activated carbon	27
3.1	List of material and apparatus	36
3.2	RL value with correspond adsorption criteria	41
4.1	Characteristic of Activated Carbon and Aerogel (SHACA) Sample	45
4.2	Efficiency of SHACA samples in reducing the turbidity in SWW	52
4.3	Efficiency of SHACA samples in reducing the TSS in SWW.	53
4.4	Efficiency of SHACA samples in reducing the TSS in SWW	55
4.5	The efficiency of SHACA 2 sample treats the SWW with different particle sizes	59
4.6	The efficiency of COD, turbidity and TSS reduction for different weight of adsorbent aerogel sample.	63
4.7	Adsorption data of four different weight of adsorbents for COD, turbidity and TSS removal in SWW solution	65

.

4.8	Adsorption data of different analysis (COD, turbidity & TSS) with	66
	different initial weight of adsorbent	
4.9	. Linear Langmuir and Freundlich graphs form for COD, turbidity and TSS studies	67
4.10	Linear isotherm model equation for Langmuir and Freundlich	68
4.11	Separation factor, $R_L$ for COD, turbidity and TSS	69

## **LIST OF FIGURES**

Figure	Item	Page
2.1	Different type of transition metal oxide aerogel	6
2.2	Silica aerogels	6
2.3	Silica Aerogel shows excellent insulating properties	7
2.4	Two dimensional structures of aerogels (a) colloidal, (b) polymeric	8
2.5	FESEM images at different magnifications; (a) bamboo aerogel, (b) carbon aerogel	10
2.6	CA absorption capacity at different organic solvents and oils	11
2.7	Process flow for carbon aerogel preparation from bamboo pulp	15
2.8	CO <sub>2</sub> supercritical drying path in P-T diagram	17
2.9	Schematic illustration of a supercritical alcohol (ethanol) drying autoclave	17
2.10	Flow diagram of the supercritical alcohol drying process	18
2.11	Annual generation of biomass in Malaysia in million tons	21
2.12	Sago Palm adult tree	22
2.13	Sago processing flow diagram	24
2.14	Sago 'Hampas'	24
2.15	Relation of agitation time and different initial Hg (II) mercury adsorption concentrations	26
2.16	AC preparation process flow diagram from sewage	28
2.17	Process flow of sago waste adsorbent modification for methylene blue from aqueous solution	30

2.18	FESEM images of treated sago waste with (a) KOH, (b) $H_3PO_4$	31
2.19	Effect of agitation time on Cu (II) ion removal	32
2.20	Effect of carbon dosage on Cu(II) removal	32
2.21	Effect of pH on Cu(II) adsorption	32
2.22	DOE Standard Limit for heavy metals discharge	34
4.1	The process to obtain raw sago hampas from sago tree.	44
4.2	SHACA Preparation Process	44
4.3	Porous in sample SHACA 1(a) and 2(b)	46
4.4	FESEM images at different magnification for SHACA 1 (a-c) and SHACA 2 (c-f).	47
4.5 (a)	FTIR spectrum of raw sago hampas.	48
4.5 (b)	FTIR spectrum of SHACA 1 (Pyrolysis process).	49
4.5 (c)	FTIR spectrum of SHACA 2 (Freeze drying process).	50
4.6	Turbidity reduction for Aerogel sample vs treatment time	52
<b>4.</b> 7	TSS reduction for Aerogel sample vs. treatment time	53
4.8	COD reduction for Aerogel sample vs. treatment time.	55
4.9	Different particle size in treating the SWW for 5 hours	56
4.10	The COD removal by different particle diameter size with constant weight.	57
4.11	Turbidity removal by different particle diameter size with constant weight	57
4.12	TSS removal by different particle diameter size with constant weight	58

4.13	SWW treatment after 3 days with different dose of adsorbent	60
4.14	COD reduction at different weight of Aerogel	61
4.15	Turbidity reduction at different weight of Aerogel	61
4.16	TSS reduction at different weight of Aerogel	61
4.17	Correlation of COD and turbidity result	63
4.18	Correlation of COD and turbidity result	64
4.19	Correlation of turbidity and TSS result	65

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2

# **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<sup>3</sup>. 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

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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).

Silica	Aerogel	Sillen	Aerogel	Silica	ca Aerogel Silica	Aerogel Silica /
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Silica	Reroget	Sillea	Aerogel	Silica	caA	'ca /
Silic	Aerogel	Sillca	Aerogel	Stica	ca	a/
Silic	Aerogel	Silica	Aerogel	S/-ca	ca	11
Silie	Aerogel	Silica	Aerogel	Silea	Ca	11
Silic	Aerogel	Silica	Aerogel	SI :a	CE MARKET CARL	11
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Silica	A copel	Silica	Aeroos	silica	ca Ae.	dica /
Silica	Aer	Silica	Apr Jal	Silica	ca Aerog.	a Silica A
Silica	Aerogei	Since	rogel	Silica	ca Aerogel Silve	Herogel Silica A

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

(b)

(a)

#### 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<sup>-3</sup>, and large specific surface area, 100 to 1600 m<sup>2</sup> 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<sup>3</sup> 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<sub>2</sub> properties was listed in **Table 2.1** as mentioned in Husing and Schubert (1998) research study.



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

Property	Range	Typical value
Bulk density, g/cm <sup>3</sup>	0.003 - 0.500	0.100
Skeletal density, g/cm3	1.700 - 2.100	-
Porosity, %	80 - 99.8	-
Mean pore diameter, nm	20-150	-
Inner surface area, m <sup>2</sup> /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, cL, m/s	<20-80	100
Acoustic impedance, Z, kg/m <sup>2</sup> s	-	104

Table 2.1. Structural properties of SiO<sub>2</sub> aerogels.