



Faculty of Resource Science and Technology

**Adsorptive Properties of Biochar Derived from Sago Effluent Activated
Sludge Process**

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**Master of Science
2022**

Adsorptive Properties of Biochar Derived from Sago Effluent Activated Sludge Process

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A thesis submitted

In fulfillment of the requirements for the degree of Master of Science

(Organic Chemistry)

Faculty of Resources Science and Technology

UNIVERSITI MALAYSIA SARAWAK

2022

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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ACKNOWLEDGEMENT

First and foremost, praises and thanks to The Almighty Allah for His showers of blessings throughout my research work to complete the research successfully.

I would like to express my highest gratitude to my supervisor, Prof Dr Zainab Ngaini as well as my co-supervisor, AP Dr Rafeah Wahi who allowed me to do this research and helped me in so many ways. Their dynamism, vision, sincerity, and motivation have deeply inspired me to finish this research as excellently as I can. It was a great privilege and honor to work and study under their guidance.

An endless appreciation to Tun Openg Sago Research Chair: F07/TOC/1742/2018 for the financial support given for this research. I would like to express my gratitude specifically to Nurul Iwani Mahmut and Abigail Vanessa Ziem for their contribution to some parts of this research and endless help whenever I need them.

I am extremely grateful to my family members especially my parents for their love, care, and prayers for educating me and preparing me for a better future. Special thanks to all my colleagues for the best friendship, motivation, moral support and have helped me throughout my journey.

My sincere gratitude to the Centre for Graduate Studies, for the advice and support given during my period of study in Universiti Malaysia Sarawak. Finally, I would like to thank the management of the Universiti Malaysia Sarawak for making it possible for me to complete my study here in Sarawak. Thank you all.

ABSTRACT

Bioconversion of agricultural wastes into value-added products provides an eco-friendly alternative for a sustainable waste management system. In this study, activated carbon (AC) derived from the sago effluent activated sludge process was proposed for the removal of lead(II) (Pb(II)) and methylene blue (MB) dye from an aqueous solution. Sago effluent activated sludge was pyrolyzed in a tube furnace and underwent chemical activation with NaOH to yield sago AC. Batch experiments were used to study the Pb(II) adsorption by sago AC at adsorbent dosage (0.1 – 0.5 g), initial Pb(II) concentration (1 – 5 mg/L), and pH (2 – 10), and MB adsorption at initial MB concentration (2 – 10 mg/L) and contact time (30 – 90 min). Response surface methodology (RSM) with central composite design (CCD) was used to optimize the adsorption of Pb(II) and MB. Results showed that the AC has a large pore size (2.70 – 6.90 μm) and a surface area of 78.863 m^2/g . Adsorption of Pb(II) and MB can be represented by Langmuir isotherm with R^2 values of 0.9308 and 0.9225 respectively. The optimum Pb(II) removal (99.87%) occurred at pH 2, 0.5 g adsorbent dosage, 5 mg/L initial Pb(II) concentration and at 30 min. The optimum MB removal (92.69%) occurred at pH 6, 10 mg/L initial MB concentration, 0.5 g adsorbent dosage, and 60 min. In conclusion, sago AC derived from the sago effluent activated sludge process is an excellent adsorbent for Pb(II) and MB.

Keywords: Activated carbon, heavy metals, dyes, bioconversion, pyrolysis

Ciri Penyerap Biocar yang dihasilkan daripada Efluen Sagu Proses Enapcemar Aktif

ABSTRAK

Penggubahan biomas menjadi produk bernilai tambah telah memberikan alternatif yang mesra alam untuk sistem pengurusan sisa lestari. Dalam kajian ini, karbon aktif (AC) yang dihasilkan melalui proses enapcemar aktif sagu efluen diusulkan untuk penyingkiran Pb(II) dan biru metilena (MB) dari larutan akueus. Enapcemar efluen sagu dipirolisis dalam relau tiub dan menjalani pengaktifan kimia dengan NaOH untuk menghasilkan karbon aktif. Eksperimen proses kelompok dijalankan untuk mengkaji penyerapan Pb (II) oleh karbon aktif pada dos adsorben (0.1 – 0.5 g), kepekatan asal (1 - 5 mg/L), dan pH (2 - 10), dan penyerapan MB pada kepekatan asal (2 - 10 mg/L) dan masa (30 - 90 min). Kaedah Rangsangan Permukaan (RSM) berdasarkan reka bentuk komposit berputar (CCD) telah digunakan untuk mengoptimumkan penyerapan Pb(II) dan MB. Hasil kajian menunjukkan bahawa karbon aktif mempunyai ukuran pori besar (2.70 – 6.90 μm) dan luas permukaan 78.863 m^2/g . Penyerapan Pb(II) dan MB dapat diwakili oleh isoterma Langmuir dengan nilai R^2 masing-masing 0.9308 dan 0.9225. Penyingkiran Pb(II) optimum (99.87%) berlaku pada pH 2, 0.5 g dos penyerap, kepekatan Pb(II) asal 5 mg/L dan pada 30 min. Penyingkiran MB optimum (92.69%) berlaku pada pH 6, kepekatan MB asal 10 mg/L, dos penyerap 0.5 g, dan 60 min. Kesimpulannya, karbon aktif sagu yang berasal dari proses enapcemar aktif sagu efluen adalah adsorben yang sangat baik untuk Pb(II) dan MB.

Kata kunci: Karbon aktif, logam berat, pewarna, penukaran bio, pirolisis

TABLE OF CONTENTS

	Page
DECLARATION	i
ACKNOWLEDGEMENT	ii
ABSTRACT	iii
<i>ABSTRAK</i>	iv
TABLE OF CONTENTS	v
LIST OF TABLES	ix
LIST OF FIGURES	x
LIST OF ABBREVIATIONS	xii
CHAPTER 1: INTRODUCTION	1
1.1 Study Background	1
1.2 Sago Wastes	1
1.3 Activated Sludge Process	2
1.4 Activated Carbon	3
1.5 Pb(II) and methylene blue (MB) in water	3
1.6 Problem Statement	4
1.7 Objectives	6
CHAPTER 2: LITERATURE REVIEW	7
2.1 Sago Palm	7

2.2	Sago Starch Processing	8
2.3	Sago Processing Wastes	10
2.4	Sago Effluent	12
2.5	Activated Sludge Process (ASP)	14
2.6	Biomass from Agricultural Wastes	16
2.7	Pyrolysis of Biomass	17
2.8	Biochar	19
2.9	Activated Carbon from Biochar	22
2.10	Adsorption	23
2.10.1	AC for Heavy Metals Adsorption	24
2.10.2	AC for Dyes Adsorption	30
2.11	Chapter Summary	35
CHAPTER 3: METHODOLOGY		37
3.1	Materials	37
3.2	Preparation of Adsorbent	37
3.2.1	Activated Sludge Process (ASP)	37
3.2.2	Pyrolysis of SE-B	38
3.2.3	Activations of SE-BC	38
3.3	Characterization of Adsorbent	39
3.3.1	Proximate Analysis	39

3.3.2	Instrumental Analysis	40
3.4	Batch Adsorption	41
3.4.1	Adsorption of Pb(II)	41
3.4.2	Adsorption of MB	42
3.4.3	The Removal of Pb(II) and MB	43
3.4.4	Isotherm Study	44
3.5	Response Surface Methodology (RSM)	46
CHAPTER 4: RESULTS AND DISCUSSION		49
4.1	The Yield of Sago Effluent Activated Sludge Process (ASP)	49
4.2	Characterization of Sago Effluent Activated Carbon (SE-AC)	50
4.2.1	Proximate Analysis	51
4.2.2	Fourier Transform Infrared (FTIR) Analysis	52
4.2.3	Transmission Electron Microscope (TEM) Analysis	54
4.2.4	Scanning Electron Microscope (SEM) Analysis	55
4.2.5	Brunauer Emmett and Tellet (BET) Analysis	58
4.3	Adsorption of Pb (II) on SE-AC	59
4.3.1	The Effect of Adsorbent Dosage	59
4.3.2	The Effect of Initial Concentration	60
4.3.3	The Effect of pH	61
4.3.4	Isotherm Study on the Adsorption of Pb(II)	63

4.4	Adsorption of MB on SE-AC	67
4.4.1	The Effect of Initial Concentration	67
4.4.2	The Effect of Contact Time	69
4.4.3	Isotherm Study on the Adsorption of MB	70
4.5	Response Surface Methodology (RSM)	74
4.5.1	The Utilisation of RSM for Optimization of Pb(II) Adsorption by SE-AC	74
4.5.2	The Utilisation of RSM for Optimization of MB Adsorption by SE-AC	80
	CHAPTER 5: CONCLUSION AND RECOMMENDATIONS	84
5.1	Conclusion	84
5.2	Recommendations	85
	REFERENCES	86
	APPENDICES	119

LIST OF TABLES

	Page
Table 2.1 Physicochemical parameters of sago effluent (Ramesh et al., 2013)	14
Table 2.2 Pyrolysis stages	18
Table 2.3 Typical product weight yields obtained by different modes of pyrolysis of wood (Bridgwater, 2012)	19
Table 2.4 Biochar Pore Sizes at Different Mode of Pyrolysis	21
Table 2.5 The adsorption capacity on heavy metals adsorption of different types of AC with different activation conditions	27
Table 2.6 The adsorption capacity on dyes adsorption of different types of AC with different activation conditions	32
Table 3.1 Experimental Factors and Response for the Adsorption of Pb(II)	47
Table 3.2 Experimental Factors and Response for the Adsorption of MB	47
Table 4.1 Proximate analysis results for SE-B and SE-AC	52
Table 4.2 The BET surface area of SE-B, SE-BC and SE-AC	58
Table 4.3 Parameters and Values of Langmuir and Freundlich Isotherms for Pb(II) adsorption by SE-AC	64
Table 4.4 Comparison of maximum adsorption capacity, Q_{max} obtained from Langmuir isotherm for the removal of Pb(II) by different adsorbent	66
Table 4.5 Parameters and Values of Langmuir and Freundlich for MB adsorption by SE-AC	71
Table 4.6 Comparison of maximum adsorption capacity, Q_{max} obtained from Langmuir isotherm for the removal of MB by different adsorbent	73
Table 4.7 Experimental Factors and Response for the Adsorption of Pb(II) by SE-AC	74
Table 4.8 Experimental Factors and Response for the Adsorption of MB	80

LIST OF FIGURES

	Page
Figure 2.1 Semi-cultivated sago palm in Mukah	7
Figure 2.2 Commercial sago extraction process	9
Figure 2.3 Pre-cut sago trunk placed by the roadside to be transported to the sago mill	10
Figure 2.4 ASP basic model interpretation for biomass production	15
Figure 3.1 Flow chart of SE-AC preparation	38
Figure 4.1 ASP of SE with different C: N: P ratio of tank (a) 100:10:1; (b) 100:10:3; and tank (c) unmodified SE, at Day 7 of aeration and 5 days settling.	50
Figure 4.2 FTIR Spectrum of SE-B, SE-BC, and SE-AC	53
Figure 4.3 TEM image of (a) SE-BC and (b) SE-AC (Magnification: 100,000X)	55
Figure 4.4 The SEM images for (a) SE-B, (b) SE-BC, and (c) SE-AC at magnification x10,000	57
Figure 4.5 The graph of the effect of adsorbent dosage on the adsorption of Pb(II)	59
Figure 4.6 The graph of the effect of initial concentration on the adsorption of Pb(II)	61
Figure 4.7 The graph of the effect of pH on the adsorption of Pb(II)	63
Figure 4.8 Linear plot of (a) Langmir isotherm and (b) Freundlich isotherm for the adsorption of Pb(II)	65
Figure 4.9 The graph of the effect of initial concentration on the adsorption of MB	68
Figure 4.10 The graph of the effect of contact time on the adsorption of MB	69
Figure 4.11 The linear plot of (a) Langmuir isotherm and (b) Freundlich isotherm on the adsorption of MB	72
Figure 4.12 The combined effects of (a) adsorbent dosage-initial concentration, (b) contact time-initial concentration, and (c) contact time-adsorbent dosage during Pb(II) adsorption by SE-AC	76

Figure 4.13 The Combined Effect of (a) Initial Concentration-pH, (b) Contact Time-Initial Concentration, and (c) Contact Time-pH during MB adsorption by SE-AC

82

LIST OF ABBREVIATIONS

ASP	Activated sludge process
BET	Bruenur Emmet and Teller
FTIR	Fourier Transform Infrared
SE	Sago effluent
SEM	Scanning electron microscope
SE-AC	Sago effluent activated carbon
SE-B	Sago effluent biomass
SE-BC	Sago effluent biochar
TEM	Transmission electron microscope
UV-Vis	UV Visible

CHAPTER 1

INTRODUCTION

1.1 Study Background

Sago palm is one of the tropical crops that are widely grown in wet conditions, such as peat swamps (Ehara et al., 2018). Sago palm has high immunity towards drought, floods, and even strong winds and fire due to the extensive fibrous roots system of the plant (Amin et al., 2019). In addition, Sago palm contains a large amount of starch used as a staple food source for the people in South East Asia (Lim et al., 2020a).

In Malaysia, approximately 90% of sago planting areas are in the state of Sarawak, where the largest planting area (75%) is in Mukah (Bujang, 2014; Lim et al., 2020b). In 2005, approximately 45,300 metric tons of food-grade sago starch were produced, contributing RM40.4 million to the Malaysian economy (Karim et al., 2008). However, the massive productions lead to an uncontrollable number of residues or by-products. Wastes from sago starch processing are usually disposed into the nearby streams and empty land, causing enormous environmental disruption (Adeni et al., 2010; Ehara et al., 2018).

1.2 Sago Wastes

The wastes from sago starch processing include sago bark, sago *hampas*, and sago effluent. Sago bark is obtained from the peeling process of the sago trunk called debarking, which is the initial process before starch extraction. Sago *hampas* is a fibrous by-product produced during the crushing and sieving process, while sago effluent is the wastewater produced during the washing process in sago mill industries (Adeni et al., 2010; Lim et al.,

2020a). Sago effluent is the wastewater collected from the washing cycles of starch in sago mill.

Sago mill is a water-intensive industry as the whole process of producing sago starch requires a great amount of water for several washing cycles. Approximately 40,000 – 50,000 L of sago effluent per tons of sago flour was produced during the process (Thangavelu et al., 2020). Sago effluent is acidic with pH 4 – 5 and high with dissolved organic matter. The effluent has high biological oxygen demand (BOD) and chemical oxygen demand (COD), which causes infertility of soil in nearby farms and water pollution (Neethu & Murugan, 2018). Most sago mills released the effluent into the stream without proper treatment (Hassimi et al., 2020), which lead to water pollution, destruction of aquatic habitat, and soil pollutions.

1.3 Activated Sludge Process

The activated sludge process (ASP) is a process in water treatment industries to treat water from all sources before safely being released into the environment. It is one of the biological treatments that require bacteria during the sedimentation process *via* aerobic consumption of organic carbon content in the effluent. The adhesive fluid secreted by the bacteria will clothe minute particles present in the wastewater, creating layers of clear water at the top and sediments at the bottom of a tank (Ahansazan et al., 2014; Shchegolkova et al., 2016). The whole process is accompanied by an aeration process that supplies oxygen to the culture of bacteria and microorganisms for complete waste stabilization (Hassard et al., 2018). The biomass obtained from this process can be applied in various value-added products and applications.

1.4 Activated Carbon

In general, activated carbon (AC) is a carbon-rich material produced *via* oxygen-starved combustion like biomass pyrolysis and underwent further activation with acids or bases (Chon, 2021). AC has well-built internal pore structures, high surface area, and versatile applications (Li et al., 2018). AC from agricultural wastes is commonly used as an adsorbent for water-soluble pollutants in the wastewater as well as gaseous pollutants in the air (Danish & Ahmad, 2018).

There are numerous studies conducted employing AC as an adsorbent for lead (Pb(II)) and methylene blue (MB), as well as other pollutants. For example, AC derived from olive branches (Alkherraz et al., 2020), sugar molasses (Legrouri et al., 2017), and palm kernel shells (Gaya et al., 2015) were reported to have high adsorption capacity for the removal of Pb(II). Besides, corn cob AC (Jawad & Mohammed, 2019) and vinasses wastes (Li et al., 2021) reported to have high adsorption capacity and removal of MB from wastewaters.

1.5 Pb(II) and Methylene Blue (MB) in Water

Lead (Pb(II)) and methylene blue (MB) are among the most commonly reported water-soluble pollutants of wastewaters in the world (Saleh, 2016; Asuquo et al., 2017). Pb(II) is widely used in diverse applications such as electroplating, paint manufacturing, batteries, and building industries (Saleh, 2016; Asuquo et al., 2017). Closed mines and the abandoned mining sites also contributed to the rising level of Pb(II) pollutions (Asuquo et al., 2017).

Pb(II) has no significant biological function to the human body and is harmful (Alghamdi et al., 2019). Heavy metals like Pb(II) comes in contact with human being

through food chains such as food and water (Zhou et al., 2020). At low concentrations, Pb(II) disturbs the metabolism and body nutrients, while at high concentrations, Pb(II) causes encephalopathy, cognitive impairment, and anaemia (Asuquo et al., 2017; Sajjadi et al., 2019). Apart from humans, aquatic life such as fish is also vulnerable to Pb(II) pollution (Alipour et al., 2020).

MB is a chromatic dye with high pH and is toxic to the environment, and is most commonly used in textile, paper printing, paints manufacturing, rubber dyeing, and leather industries (Liu et al., 2018; Tong et al., 2018). High concentrations of MB in the waterlines hindered photosynthetic activities in aquatic plants by preventing sunlight from penetrating the water (Kuang et al., 2020; Utsev et al., 2020). MB is hazardous, and when in direct contact, it can affect humans' brain activities, eyes injury, nausea, jaundice, and various other diseases (Nallapan et al., 2019). This toxic nature of both Pb(II) and MB has brought an urgent need to remove from water bodies.

1.6 Problem Statement

The massive-scale production of sago starch produced a large amount of sago effluent. Sago effluent is commonly dumped into the nearby streams without treatment, causing river pollutions which affect aquatic life due to its large BOD and COD value (Neethu & Murugan, 2018). The activated sludge process is a potential way to reduce the high organic matter in sago effluent prior to its release to waterways. At the same time, it produces sludge that can be utilized as feedstock for activated carbon production.

Over the years, the solid wastes from sago processing such as sago bark and *hampas* have been utilized as adsorbents for various pollutants such as MB (Amode et al., 2016; Rajan et al., 2019), zinc (Zn(II)) (Karri & Sahu, 2018), and oily wastewater (Ngaini et al.,

2014). However, to the best of the author's knowledge, no studies have been published on the potential application of sago effluent to remove water pollutants due to its liquid nature. Current studies on sago effluent are limited to using sago effluent as the medium of cultivations for various bacteria and microorganisms (Mookiah et al., 2020; Phang et al., 2000). The utilization of sago effluent as the medium of cultivations produces secondary wastes that trigger another environmental management solution.

Previous studies showed that wastewater processing sludge can be converted to activated carbon with high adsorption performance (Bian et al., 2018). Therefore, in this study, activated sludge process (ASP) had been introduced to sago effluent to produce sludge (sago effluent biomass, SE-B), which in turn, was used as the precursor for activated carbon preparation. Based on precedent literature, sago effluent contains high organic matter, nutrients, and microorganisms, making sago effluent an ideal medium for ASP (Hammado & Utomo, 2018). The SE-B obtained underwent physical activation in a tube furnace and chemical activation with NaOH to produce sago effluent activated carbon (SE-AC). The adsorptive properties of SE-AC were determined, and its efficiencies for removing Pb(II) and methylene blue (MB) in an aqueous solution were studied.

This research promotes not only the repurposing of sago effluent as a value-added product that helps in waste management in sago industries but also suggests a low-cost alternative for sustainable removal of heavy metals and dyes.

1.7 Objectives

The objectives of this study are:

- i. To produce biomass (SE-B) from sago effluent *via* the activated sludge process.
- ii. To synthesize activated carbon (SE-AC) from SE-B *via* pyrolysis and chemical activation.
- iii. To determine the efficiency of SE-AC for removal of Pb(II) and MB in an aqueous solution.

CHAPTER 2

LITERATURE REVIEW

2.1 Sago Palm

Sago palm with the scientific name of *Metroxylon sagu* belongs to the Palmae family. Sago is also known as Rumbia (Malaysia/Indonesia), Thagu-bin (Burma), Sa Kuu (Cambodia), and Sa Khu (Thailand). Sago palm can be easily grown and sustained in peat soils (Ehara et al., 2018). It is a hardy plant that survives in extreme weather conditions like drought, floods, strong wind, and fire, regardless of the soil conditions (saline, submerged, acid, swampy) that only a few crops can survive (Adeni et al., 2010). Figure 2.1 shows semi-cultivated sago palm trees planted by the roadside of Mukah, Sarawak.



Figure 2.1: Semi-cultivated sago palm in Mukah

Sago is a plant species that produces valuable and high-quality starch from its stem (Riyadi et al., 2018). Sago has been known as humankind's oldest food plant and staple food supply for humans, especially in Asia (Mofu & Abbas, 2018). It is an essential source of primary dietary starch to millions of people throughout the Indo-Pacific region as it stores a large amount of starch in its trunk compared to other species in the genus (Azhar et al., 2018). Sago produces significantly more starch than other crops such as rice, corn, wheat, and

potatoes (Metaragakusuma et al., 2016). In Malaysia, the Melanaus in the Third Division of Sarawak consume sago as their staple dietary routine instead of rice. Sago pearls are food products made by local Melanaus as a healthy snack that can be boiled or eaten alone with a banana. Tebaloi, a sweet snack made from sago and coconut, is another popular food product from sago and widely marketed in Malaysia (Ehara et al., 2018).

Approximately 90% of sago plantation in Malaysia is in the state of Sarawak, where the largest area cultivated with sago is in Mukah (75%) (Bujang, 2014; Lim et al., 2020b). The Sarawak government had developed a plantation crop and established a research station for sago palm in 1996 (Adeni et al., 2010). In Mukah, about 7700 hectares of land were developed for sago palm plantation by Sarawak Land Development Agency, making the first world-scale commercial plantation. Research on sago palm grows extensively as it complements six advantages: economically acceptable, relatively sustainable, environmentally friendly, vigorous, and promotes socially stable agroforestry systems (Azhar et al., 2018; Ehara et al., 2018).

2.2 Sago Starch Processing

Traditionally, starch extraction was manually conducted by local village folks following a few simple but tiring steps. The fell-ripe trunk was chosen, cut, and floated back to the village using a waterway nearby. The trunk is then cut into half, and thick bark is removed to obtain a soft pith inside. Soft fibrous pith was then rasped by using bush knives and put in sago sheath for extraction. The sago extract was washed several times, kneaded with water, and stored wet in a basket made with sago leaflets (Flach, 2011).

Commercial sago processing industries involved several crucial steps: debarking, pulping, starch extraction, drying, and packing (Adeni et al., 2013; Ehara et al., 2018). Commercial sago processing industries are using modern extraction methods employing heavy machines to assist the extraction processes. The step-by-step process of sago processing is shown in Figure 2.2.

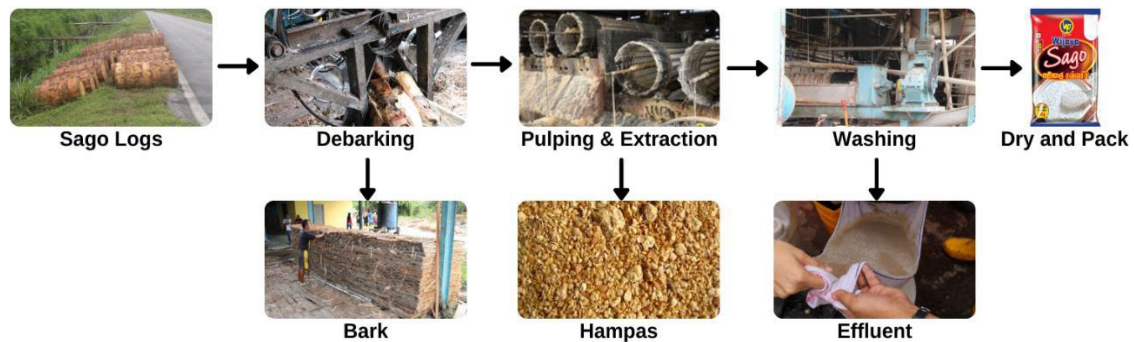


Figure 2.2: Commercial sago extraction process

The first step into obtaining sago starch is debarking. During debarking, 2 – 3 cm, fibrous bark is removed and pre-cut into 1 m sago trunk. The sago trunk was then transported to the nearby sago processing mill for the following process (Bujang, 2008). Figure 2.3 shows a pre-cut trunk placed by the roadside, ready to be transported to the sago mill. The next step involves pulping the soft pith into a shapeless mass for starch extraction. Sago starch is extracted and repeatedly washed during the process. The last step is drying sago starch, packing and ready to be used in many other industries (Flach, 2011; Ehara et al., 2018). Sago starch processing produces by-products such as sago bark, sago *hampas*, and sago effluent, which have no market value thus categorized as agricultural wastes.



Figure 2.3: Pre-cut sago trunk placed by the roadside to be transported to the sago mill

2.3 Sago Processing Wastes

Collectively, an estimated 10 - 50 tons of sago wastes are generated daily from sago starch processing. This amount is expected to increase every year due to the high demand for sago starch. During the debarking process, approximately 5 tons of sago barks are produced daily, contributing about 17% of the log process (Amin et al., 2019). Sago barks are strong and hard because it is made up of mostly peripheral cells consisting of sclereid cells with thick secondary cell wall. Lignin (29.4%) and cellulose (44.0%) are the main chemical components of sclereid cells dominated by the phenylpropane aromatic group (Siruru et al., 2019).

Besides, sago bark also contains a small fraction of hemicellulose (22.8%) and other extracts such as starch, minerals, tannins, and flavonoids (Danish & Ahmad, 2018). Due to its sturdy characteristics, sago bark is commonly used as a platform around the factory and footpaths for folk houses. Many studies reported on the utilization of solid sago bark into value-added products (Adeni et al., 2010).

Chemical modification of sago bark *via* esterification with stearic acid has been reported with excellent adsorbent on engine oil in an aqueous environment (Ngaini et al., 2014) and palm oil mill effluent (POME) (Wahi et al., 2016). Wahi et al. (2019a) produced pyrolytic oil from sago bark *via* microwave pyrolysis and improved its calorific value (21.57 KJ/kg) through esterification with ethanol and sulphuric acid, which was higher than pyrolytic oil from sago biomass (22.15 MJ/kg) (Rambli et al., 2019). This is due to the high lignin and cellulose content in sago bark that contributes to a high percentage of carbon composition, making it possible to be an alternative for biofuel productions (Amin et al., 2019)

Sago bark combined with other wastes such as empty fruit bunch shown good mineralization and germination index for the growth of water spinach and green mustard (Wahi et al., 2019b) as well as frond wastes for oyster mushroom cultivation replacing sawdust (Senghie et al., 2021). The utilization and extensive research conducted on sago bark help in reducing the wastes from the mill. Other application of sago bark includes building materials such as plywood, particleboards, and wall tiles.

Another type of waste in the sago industry is sago *hampas*. Sago *hampas* is a starchy by-product from the crushing of sago pith during starch extraction and composed of lignocellulosic materials (Husin et al., 2019). The quantity of sago *hampas* produced greatly depending upon the quality of the extraction process. In Mukah, Sarawak, 50 – 110 tons of sago *hampas* was produced daily. The texture of sago *hampas* is softer and not as rigid as sago bark due to the low lignin (6.11%) and cellulose (12.75%) content (Amin et al., 2019). Sago *Hampas* is composed of mostly hemicellulose (32.99%), starch (66.0%), and other non-structural components such as tannins and sugar (Siruru et al., 2019). Hemicellulose is an amorphous branching compound with more -OH functional groups responsible for the

hygroscopicity of sago *hampas* (Siruru et al., 2020). The high starch content of sago *hampas* is also used as feedstock for animals and compost for mushroom culture (Adeni et al., 2010). Unlike sago bark, sago *hampas* is dominated by parenchyma cells that function as photosynthesis storage for food reserves (Siruru et al., 2020)

There are several downstream studies reported on sago *hampas*, such as the production of fermented sugar (Kannan et al., 2013) and bioethanol (Adeni et al., 2013) *via* enzymatic and acid hydrolysis, adsorbent for the removal of heavy metals (Karthika et al., 2010) and biofuels using *Clostridium acetobutylicum* ATCC 824 (Husin et al., 2019). Reutilizing sago *hampas* into other value-added products can create proper management of wastes and reduce environmental pollutions.

Last but not least, sago effluent (SE) is another waste that being produced in the highest amount during sago processing (Adeni et al., 2010). The process of starch extraction requires a large amount of water for the washing process of starch. As a result, sago mills and manufacturers produce about 8 – 22 tons of effluent per day (Hammado & Utomo, 2018). Antithetical to the other wastes (bark and *hampas*), SE is in liquid form that does not have specific repurposing values and is often discarded to the nearby stream. There is also a very limited study conducted on the repurposing of SE to value-added products.

2.4 Sago Effluent

In general, SE is a pale white, grey, or yellowish-grey colour liquid. The pH of SE is acidic due to the discharge of hydrocyanic acid during the extractions of starch from sago pith (Priya & Ramesh, 2021). It has an obnoxious odour, high chemical oxygen demand (COD), and high biological oxygen demand (BOD), which affects the health of the soil,

disturb the ecosystem of the streams, animals, plants and even human being (Illi et al., 2020).

Table 2.1 shows the parameters of sago effluent produce by a typical sago starch processing mill.

SE is composed of discarded materials and compounds from the activity of crushing and pulping of sago pith. It contains extracts from sago pith that are soluble in water (starch, tannins, sugar) and washed away during the washing cycle of sago starch (Srinivasan et al., 2020). SE is believed to have high minerals and inorganic residue which are stable in high temperatures that contributed to SE's high ash content. SE also contains a very high carbon to nitrogen ratio (105:0.12) and a high concentration of suspended solids, which makes it challenging to treat the effluent after every process thus, sago mills commonly release effluent directly into the nearby streams without proper treatment (Ramesh et al., 2013).

Limited studies reported on the repurposing of SE due to its liquid in nature which required further treatment and containment (Illi et al., 2020). However, some studies employed SE as the medium for the cultivations of various organisms. Many years ago, SE was utilised as the medium of spirulina cultivation (Phang et al., 2000). Recently, SE had been studied for *Yarrowia lipolytica* (yeast strain) and *Chlorella vulgaris* (microalga) cultivation as lipid sources for biofuel production (Mookiah et al., (2020). Besides, SE was also employed to produce fermentable sugar (Illi et al., 2020), biocellulose (Yanti et al., 2019), bioflocculant (Hassimi et al., 2020), and biohydrogen production using *Enterobacter aerogenes* (Ulhiza et al., 2018).

The employment of SE as the medium for various processes is due to the high C:N:P (24:0.14:1) ratio with high COD and BOD values that makes it suitable for bacterial growth and respirations. However, the employment of SE as the medium for cultivations produced

secondary pollutants which contaminate the water system. Thus, appropriate treatment on the SE is needed to eliminate the formation of secondary pollutants.

Table 2.1: Physicochemical parameters of sago effluent (Ramesh et al., 2013)

No.	Parameters	Values (m)
1.	pH	3.5 – 5.6
2.	Dissolved Solod (DS)	10,475 mg/L $\pm$ 22.23
3.	Suspended Solid (SS)	4,645 mg/L $\pm$ 33.67
4.	Biological Oxygen Demand (BOD)	2,600 mg/L $\pm$ 30.12
5.	Chemical Oxygen Demand (COD)	3, 570 L $\pm$ 27.49

2.5 Activated Sludge Process (ASP)

There are many methods reported for the treatment of wastewater. One of them is by using ASP. ASP is a common method in sewage and wastewater treatment before releasing into the environment. ASP is usually used to remove organic carbon and phosphorus as well as to carry out nitrification and denitrification of waste (Orhon, 2014). The basic mechanism of ASP involves the decomposition of complex in dissolved and suspended solid substrates into simple end products such as carbon dioxide and water (Shchegolkova et al., 2016). Generally, ASP involves two types of treatments which are primary treatment and secondary treatment. The primary treatment of wastewater involves removing suspended solid through screening. The sludge obtained is known as primary sludge.

In the secondary treatment, wastewater is mixed with activated sludge from the secondary clarifier in an aerated tank (Ahansazan et al., 2014). In this tank, the wastewater is aerated to provide oxygen to the heterogeneous mixture of microorganisms and help in the mixing process. Microorganisms and bacteria available in the wastewater utilized the organic waste substrates to synthesize their cell material, thus reducing the amount of

organic and suspended solids in the effluent (Kumar & Chopra, 2017). In addition, aerobic bacteria played an important role in oxidizing organic matters and forming flocs (Orhon, 2014; Am et al., 2019).

Flocculation and coagulation of bacteria and the separation of biomass occurred in the secondary clarifier (Ahansazan et al., 2014). The size of floc particles is determined by the agglutination strength of the floc-forming bacteria, the existence of filamentous organisms, and the turbulence level in the tank (Anbalagan et al., 2016; Fergala et al., 2018). Typically, bacteria species such as *Achromobacter* sp., *Aerobacter* sp., *Pseudomonas* sp., and *Zooglea* sp. are used and reported to be highly prevalent and enormously contributed to the development of a well-flocculated sludge or solid biomass (Orhon, 2014). The wastewater proceeds to filtration and disinfection before it is safe to be released into the environment (Ahansazan et al., 2014). Figure 2.4 shows the basic model of an ASP treatment plant and the production of biomass.

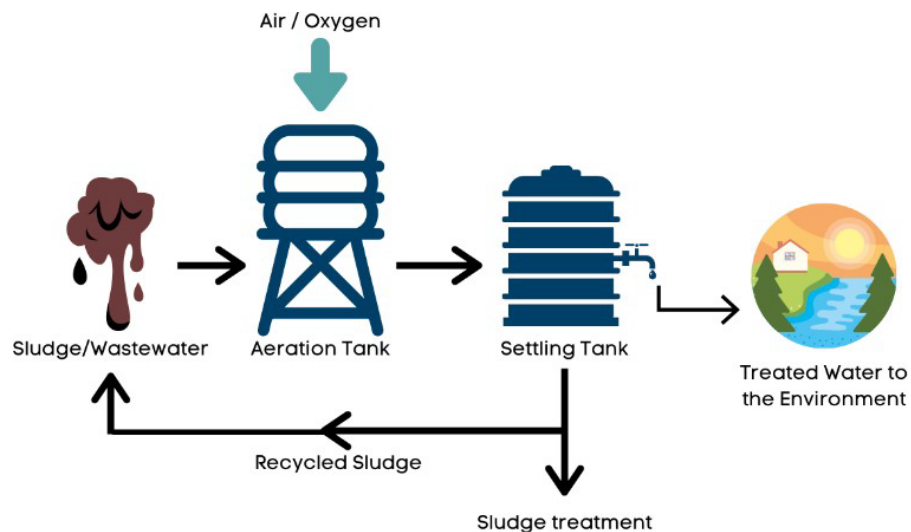


Figure 2.4: ASP basic model interpretation for biomass production

2.6 Biomass from Agricultural Wastes

Biomass is a biological organic or non-organic solid product derived from living organisms (Tripathi et al., 2016). Biomass is often associated with plant-based materials or wastes generated from agricultural activities (Fahmy, 2020). Biomass is known as a sustainable source of renewable energy as well as helping the agriculture industries in terms of waste management (Kumar et al., 2018). Biomass from plants such as corn cob (Reddy et al., 2016), coffee residue (Chon, 2021), sago bark (Ngaini et al., 2017; Amin et al., 2019), and sago *hampas* (Hammado & Utomo, 2018; Rajan et al., 2018) are composed of lignocellulosic materials such as cellulose, hemicellulose, and lignin.

Cellulose is the largest fraction contributing about 38 – 50% by weight of lignocellulosic biomass (Hensley et al., 2011a). Hemicellulose consists of 20 – 40% by weight of lignocellulosic biomass, the second-largest fraction in the compound. Lignin is an amorphous and high molecular weight compound that contributes to the third-largest fraction (15 – 25%) (Hensley et al., 2011b). Other trace materials such as nitrogen, phosphorus, potassium, silicon, chlorine, and sulphur might also be found in minutes quantity (Lehmann & Joseph, 2009).

Biomass is not limited to plant-based solid wastes and may extend to any biologically formed matter such as wastewater from agricultural, sewage, and municipal (Fahmy, 2020). Many studies reported on biomass from municipal and sewage sludge *via* ASP and employed it as fertilizers, soil amender, and drought tolerance in vegetation (Tao et al., 2015; Hassard et al., 2018). Besides, biomass also supplies nutrients such as nitrogen and phosphorus and micronutrients such as nickel, copper, and zinc for plant growth (Amuda et al., 2008).

Further biomass carbonization into biochar would produce a more versatile material for wider applications (Singh & Mishra, 2020). Biomass from ASP of sewage sludge (Tao

et al., 2015), treatment plant sludge biomass (Singh et al., 2020), olive mill effluent biomass (Haddad et al., 2017, 2021) has been utilized as the precursors for the production of biochar. The generated sludge or biosolids derived from ASP is usually rich in nitrogen and phosphorus and contain valuable micronutrients for gardening, commercial agriculture, greenways, and silviculture (Amuda et al., 2008; Tao et al., 2015).

2.7 Pyrolysis of Biomass

Biomass pyrolysis has been extensively studied at various reaction conditions to produce biochar (Duman et al., 2010). Biochar is a product from oxygen-starved combustion or pyrolysis, which is rich in carbon materials (Wang et al., 2020). Pyrolysis is a thermal decomposition of organic matter under high temperatures without the presence of oxygen to obtain an array of solid, liquid, and gas products (Li et al., 2020). Pyrolysis of biomass with high lignocellulosic content usually produces carbon-rich biochar (Roy & Dias, 2017).

The pyrolysis of biomass into biochar involves three stages; Stage 1 (Pre-pyrolysis), Stage 2 (Main Pyrolysis), and Stage 3 (Formation of Carbonaceous Compound) (McBeath et al., 2014). Pre-pyrolysis occurs at the earliest stage of pyrolysis whereby the evaporation of moisture, volatile matter, and non-condensable gases such as CO₂, CO, H₂, and methane gas occurs at ambient temperature to 200 °C (McBeath et al., 2014; Tomczyk, 2020). The moisture evaporation causes the breakage of bonds and forms hydroperoxide, -COOH, and -CO groups (Tomczyk, 2020).

Stage 2 (Main pyrolysis) occurred at 100 – 500 °C, where the devolatilization and decomposition of hemicellulose and cellulose took place. Lastly, stage 3 causes the degradation of lignin and other organic matter with stronger chemical bonds producing

carbon-rich residual solid (Demirbas, 2009; Tomczyk, 2020). The summary of pyrolysis stages is tabulated in Table 2.2.

Table 2.2: Pyrolysis stages

Stages	Details
Stage 1 (Pre-pyrolysis)	Removal of moisture and volatile matter. Formation of hydroperoxide, -COOH, and -CO groups 0 – 200 °C
Stage 2 (Main pyrolysis)	Degradation of hemicellulose and cellulose 200 – 500 °C
Stage 3 (Degradation)	Degradation of lignin and other organic matter with stronger chemical bonds >500 °C

The pyrolysis of biomass occurs at different rates and pathways depending on the composition of the biomass. Lignin decomposed at a wider temperature range than hemicellulose and cellulose, which decomposed at a narrow temperature range (Bridgwater, 2012). Pyrolysis of biomass is performed either at a fast or slow rate to produce the desired product depending on its application.

Conventional or slow pyrolysis has been used ever since to produce biochar (Aliakbari et al., 2017). Slow pyrolysis requires heating at 500 °C with vapour residence time between 5 – 30 minutes (Mohan et al., 2008; Zhou et al., 2020). The heating rate of slow pyrolysis is 5 – 7 °C/min and produces up to 50% less liquid and 35% or more char than fast pyrolysis (Bridgwater, 2012). However, the yield of char produced greatly depends on the feedstock properties, processing temperature, heating rate, and heating environment, such as the presence of nitrogen or carbon dioxide as well as different bed materials (Roy & Dias, 2017). In contrast, fast pyrolysis is conducted at a very high temperature with low residence time for vapour (approximately less than 2 sec) (Bridgwater, 2018). Fast pyrolysis

produces up to 60% bio-oil and 20% biochar at a heating rate of 300 °C/min (Duman et al., 2010).

The characteristics of biochar produced at different pyrolysis rates are tabulated in Table 2.3. Different parameters produce different percentages of the desired yield of liquid, solid, and gas. Recent technology executes new pyrolysis technology using tube furnace, which is likely to produce large surface area biochar that is ideal for heavy metals (HM) and dyes adsorbent (Mohan et al., 2006; Divyashree & Hegde, 2015).

The temperature of pyrolysis plays a vital role in determining the characteristic of biochar produced. Pyrolysis at high temperatures produces biochar with high surface area, microporous, and hydrophobic. Meanwhile, pyrolysis at a low temperature produces biochar which is suitable for removing inorganic contaminants by oxygen-containing functional group, electrostatic attraction, and precipitation (Hensley et al., 2011a).

Table 2.3: Typical product weight yields obtained by different modes of pyrolysis of wood (Bridgwater, 2012)

Mode	Conditions	Liquid (%)	Solid (Biochar) (%)	Gas (%)
Fast	~ 500 °C ~ 1 s	75	12	13
Intermediate	~500 °C, ~30 s	50 (2 phases)	25	25
Slow	~400 °C, ~1 d	30	35	35
Gasification	~750 – 900 °C	5	10	85
Torrefaction	~290 – 60 °C	0 (unless condensed – 5)	80	20

2.8 Biochar

Biochar is a natural occurring material found in soils as the consequence of wildfire and/or after a long natural process (McBeath et al., 2014). Biochar is a carbon-rich compound comprised of carbon, hydrogen, oxygen, nitrogen, sulfur, and ash (Sohi et al.,

2010). Nowadays, biochar from agriculture is used as the precursor to produce activated carbon due to its low-cost, easily produced, and environmentally friendly (Sizmur et al., 2016). A wide range of biomass is used to produce biochar such as seaweed (*Gelidiella accrosa*) (Ahmed et al., 2019), Foxtail palm trunk (*Wodyetia bifurcate*) (Jean et al., 2019), coconut shell (Atikah et al., 2020), and Bermuda grass (Zeng et al., 2021). The biomass went through pyrolysis at different rates producing biochar with the desired properties for specific usage and study.

The characteristics, such as the size of biochar produced after pyrolysis, are highly dependent on the type of feedstock and pyrolysis rate (Fahmy, 2020). The size of biochar is usually smaller than the particle size of its precursor due to shrinkage and attrition during carbonization (Roy & Dias, 2017). The physical properties of biochar depend on the composition of biomass which determines the pyrolytic yield. Biomass with high lignin content produces a high yield of biochar. This is due to the high stability of lignin to thermal degradation (Danish & Ahmad, 2018). Lower lignin content in biomass such as maize and rye produces low yield biochar, less robust, and low mechanical (Hensley et al., 2011b).

The porosity of biochar determines the capacity of adsorption (Kharrazi et al., 2020). Biochar can be divided into three different categories, namely micropores, mesopores, and macropores. Micropores biochar have a pore size of less than 2 nm, while mesopores biochar have an internal pore size between 2 – 50 nm. Pore diameters more than 50 nm are classified as macropores (Lehmann & Joseph, 2009; McBeath et al., 2014). The excellence of biochar as an adsorbent mainly depending on the type of pore (micro, meso, or macro) and the size of pollutants. Table 2.4 Summarised the biochar pore properties with a different mode of pyrolysis.

Table 2.4: Biochar Pore Sizes at Different Mode of Pyrolysis

Precursor	Pyrolysis Mode	Pore Size	Citation
Sewage sludge	Slow pyrolysis 500 – 700 °C 5 h	Micro – mesopores	(Zielinska et al., 2015)
Chipping chunks - <i>Abies Altar</i> M - <i>Larix decidua</i> Mill - <i>Picea excelsa</i> L - <i>Pinus nigra</i> A - <i>Pinus sylvestris</i> L	Slow Pyrolysis 450 °C 4 h	Macropores	(Baiamonte et al., 2019)
<i>Miscanthus giganteus</i> grass	Fast pyrolysis 450 °C 0.5 – 1 h	Micropores	(Duarte & Glaser, 2019)
Seaweed (<i>Gelidiella</i> <i>acrosa</i>)	Fast pyrolysis 800 °C 1.5 h	Mesopores	(Ahmed et al., 2019)
Endocarp (<i>Wodyetia</i> <i>bifurcate</i>)	Intermediate – fast pyrolysis 400 – 700 °C 2 h	Micro- mesopores	(Jean et al., 2019)
<i>Opuntia Ficus Indica</i>	Intermediate pyrolysis 400 °C 1 h	Micropores	(Choudhary et al., 2020)
Coconut shell	Fast pyrolysis 700 °C 2 h	Mesopores	(Atikah et al., 2020)
Bermuda grass	Fast pyrolysis 800 °C 2 h	Micropores	(Zeng et al., 2021)

Biochar has received enormous attention among scientists around the globe for its multi-functionality. Biochar from sewage sludge (Aliakbari et al., 2017) and other sludge-based biochar (Bian et al., 2018) pulled attention among researchers due to its high adsorbent function. In waste management, biochar is widely used in four different areas, mainly soil improvement and remediation (Pan, 2020), waste management, climate change mitigation (Sangon et al., 2017), and energy production (biofuels) (Thangavelu et al., 2020). Biochar from agricultural waste can increase soil fertility and is widely used to replace fertilizer (Inyang & Dickenson, 2015). Biochar can adsorb nitrate nitrogen, reduce contamination in farmland, to reduce contamination of groundwater with nitrate-nitrogen (Ch'ng et al., 2014; Jindo et al., 2014). Biochar with further activation to become activated carbon might enhance its features and versatility as well as porosity.

2.9 Activated Carbon from Biochar

Activated carbon (AC) is a carbon-rich material with high porosity and surface area (Makshut et al., 2020). Biochar activation to form activated carbon is divided into 2 types of activations, namely chemical activation, and physical activation. Physical activation involves thermal treatment on biochar which consists of carbonization and a controlled gasification process. For example, hydrothermal, steam, and pyrolysis (Daifullah et al., 2007; Li et al., 2013; Mor et al., 2017).

Chemical activation involves impregnating biochar with an oxidizing agent such as sodium hydroxide (NaOH) (Saad et al., 2020; Chon, 2021), potassium hydroxide (KOH) (Liu et al., 2020), zinc chloride (ZnCl₂) (Ukanwa et al., 2019), sulphuric acid (H₂SO₄) (Jain & Gogate, 2017), phosphoric acid (H₃PO₄) (Liu et al., 2018) and aluminium chloride (AlCl₃)

(Bhatnagar et al., 2013) prior to introduction to heat. The activations conducted on biochar enhanced its characteristics to be used as a more versatile value-added product.

AC emits interesting and unique characteristics, making it more ideal to be used as an adsorbent for gas and aqueous solutions. It has high porosity and a significantly large surface area which makes them suitable to be used in air and gas filtration systems (Holkar et al., 2016) and removal of toxic components in wastewater (Islam et al., 2017). AC has been reported as an excellent adsorbent for HM such as lead (Pb(II)), chromium (Cr(VI)) (Kharrazi et al., 2020), and cadmium (Cd(II)) (Kavand et al., 2020) as well as dyes such as methylene blue (Yadav et al., 2021), and congo red (Lafi et al., 2018).

2.10 Adsorption

Adsorption is the activity of transferring adsorbate particles or ions from solution to adsorbent surfaces (Burakov et al., 2018; Thomas et al., 2019) *via* physical or chemical adsorption. Physical adsorptions involve non-specific *van der Waals* forces between adsorbent and adsorbate and are not dependent on the nature of substances, it is also reversible (desorption) when the concentration of adsorbate is high (Burakov et al., 2018). In contrast, chemical adsorption or chemisorption involves chemical reactions between adsorbent and adsorbate, which creates an ionic or covalent bond. Chemisorption involves thermal, very selective on the choice of substrate, and irreversible (Kan et al., 2017; Burakov et al., 2018). AC also is the cheapest and efficient adsorbent for excellent removal performance due to its large surface area (Thomas et al., 2019), porous structure, and high chemical stability (Xu et al., 2019; Utsev et al., 2020).

2.10.1 AC for Heavy Metals Adsorption

Heavy metals (HM) such as cadmium (Cd(II)), nickel (Ni(II)), lead (Pb(II)), and chromium (Cr(IV)) occur naturally in the Earth's crust (Karnib et al., 2014). They are high-density metallic elements with an atomic weight between 63.5 – 200.6 g/mol (Ghasemi et al., 2014). Exposure to HM in a trace amount causes toxicity effects on humans and other living organisms (Bashir et al., 2019). The development of industries such as metal plating facilities (Xie et al., 2018), fertilizer industries (Wilson et al., 2018), batteries, and tanneries increased the tendency of polluting the nearby streams with HM (Alguacil et al., 2018).

Dumping waste containing HM into the environment affects the ecosystem, such as flora, fauna, and other biotic and abiotic components (Alipour et al., 2020; Zhang & Duan, 2020). Since HM are stable and non-degradable, accumulation in the environment will contaminate the ecosystem (Kamari et al., 2019). Lead (Pb(II)) is a type of metal that is commonly found in industrial wastewater. According to the Agency for Toxic Substances and Diseases Registry, Pb(II) is in the second place after arsenic to be the highest priority based on exposure frequency and intrinsic toxicity (Gebrati et al., 2018).

Untreated wastewater containing Pb(II) is released into the streams causing pollutions and accumulations of Pb(II) in the ecosystem. The polluted water from the streams flows to the sea and caused more serious problems to the aquatic organism as well as affecting humans' health (Chaduka et al., 2019). It has been reported that a high concentration of Pb has been discovered in seafood and molluscs (Arnich et al., 2012). Pb(II) accumulates in seafood and is transferred to humans *via* the food chain, lead to serious disease and central nervous system damage (Fu & Wang, 2011). The toxicity of Pb(II) leads to anaemia, insomnia, dizziness, irritation (Abdelhafez & Li, 2016), weakening of muscles, hallucination, and renal damages (Arcibar-Orozco et al., 2015). Besides, Pb(II) is teratogenic

and able to disrupt enzyme activity to develop red blood cells and nerve in the human body (Kamaraj et al., 2020).

Other metals such as zinc (Zn(II)) are needed in a permissible amount in human daily life, however, excess exposure to Zn(II) cause serious health issues such as stomach cramps, vomiting, nausea, and anaemia (Am et al., 2019). Mercury (Hg) is another harmful metal that causes acute and immediate effects. It is a neurotoxic chemical that affects the central nervous system of humans and living organisms (Wang et al., 2017). The most well-known example of mercury poisoning happened in Japan and caused Minamata disease. For so many years, the effect of mercury poisoning can still be observed in the people living in Hiroshima (Zahir et al., 2005).

Several technologies have been developed for the removal of HM from wastewater namely chemical precipitation (Zhang & Duan, 2020), ion exchange, membrane filtration (Song et al., 2018), electrochemical treatment, and adsorption (Fu & Wang, 2011). In chemical precipitation, chemicals are added into the treatment chamber to react with HM to form a precipitate (Ibrahim et al., 2016). Adsorption is one of the most simplified techniques with excellent removal of HM (Chon, 2021). Adsorption employing AC has been reported to be excellent on HM such as lead (Pb(II)) (Kavand et al., 2020; Liu et al., 2020), chromium (Cr(IV)) (Kan et al., 2017), copper (Cu(II)), cadmium (Cd(II)) (Hodgson et al., 2016), and others such as nickel (Ni(II)) (Bouhamed et al., 2016) and mercury (Hg) (Wahi et al., 2009).

AC from different precursors is employed for the removal of heavy metal. The removal efficiency and adsorption capacity of AC onto HM depend greatly on the types of precursors used and the activating conditions such as pyrolysis temperature and activating agent. Less sturdy precursor that has high hemicellulose, starch, and extracts composition generally requires lower pyrolysis temperature (<500 °C) to produce activated carbon

(Danish & Ahmad, 2018). Winemaking wastes (Alguacil et al., 2018) and marine microalgae (Ibrahim et al., 2016) for instance are rich in hemicellulose and starch because of the abundance of parenchyma cells. Wine wastes are activated with KOH at 250 °C to give 58.32 mg/g adsorption capacity of Pb(II) (Table 2.5). Marine microalgae showed slightly higher Pb(II) adsorption capacity (83.3 mg/g) as the pyrolysis temperature was higher (600 °C). Sugar molasses are thick viscous sugar production residue that is rich in starch and give significantly high adsorption capacity on Pb(II) (303.03 mg/g) when activated with H₃PO₄ at 500 °C (Legrouri et al., 2017). The adsorption capacity of the mentioned ACs is considerably high considering the low amount of adsorbent dosage used (1.0×10^{-4} to 8.0×10^{-4} g/mL).

The adsorption capacity of AC from palm shell (333.00 mg/g) (Gaya et al., 2015) and wood apple shell (151.51 mg/g) (Doke & Khan, 2017) are higher compared to a date stone (18.68 mg/g) (Bouhamed et al., 2016) due to the higher pyrolysis temperature (500 – 600 °C). The date stone AC is pyrolyzed at 350 °C which is quite low since higher temperature is needed to break down the high lignin and cellulose content in a sturdier precursor. The removal of Cr(VI) using cotton textile AC showed high maximum adsorption capacity (267.12 mg/g) (Xu et al., 2019) compared to macadamia shell AC (17.51 mg/g) (Lesaoana et al., 2019) due to the activation of cotton textile biomass with FeCl₃ which creates AC with higher total surface area, thus improving the adsorption.

Table 2.5: The adsorption capacity on heavy metals adsorption of different types of AC with different activation conditions

No.	Precursor	Treatments and Adsorptions	Pollutants	Citation
1.	Palm shell	AC Treatment: KOH, NaOH, ZnCl ₂ Pyrolysis temperature: 500 °C Concentration: 2000 mg/L Adsorption time: 1 h 40 min Adsorbent dosage: 0.02 g/mL Mixing rate: 350 rpm pH: 6.0 Qmax: 1000 mg/g (Cd(II)-KOH), 333.00 mg/g (Pb(II)-KOH), 100 mg/g (Cd(II)-NaOH), 500.00 mg/g (Pb(II)-NaOH), 125.00 mg/g (Cd(II)-ZnCl ₂), 250.00 mg/g (Pb(II)-ZnCl ₂)	Pb(II), Cd(II)	(Gaya et al., 2015)
2.	Olive stones	AC Treatment: H ₃ PO ₄ Pyrolysis temperature: 380 °C Concentration: 5.0 mmol/L Adsorption time: 10 h Adsorbent dosage: 0.006 g/mL Mixing rate: 400 rpm pH: 5 Qmax: 0.268 mmol/g (Cu(II)), 0.559 mmol/g (Cd(II)), 0.654 mmol/g (Pb(II))	Cu(II), Cd(II), Pb(II)	(Bohli et al., 2015)
3.	Date stones	AC Treatment: H ₃ PO ₄ Pyrolysis temperature: 350 °C Concentration: 100 mg/L Adsorption time: 2 h Adsorbent dosage: 0.0025 g/mL Mixing rate: 350 rpm pH: 5.5 Qmax: 18.68 mg/g (Cu), 16.12 mg/g (Ni), 12.19 mg/g (Zn)	Cu(II), Ni(II), Zn(II)	(Bouhamed et al., 2016)
4.	Marine microalgae	AC Treatment: KOH Concentration: 60 mg/L Pyrolysis temperature: 600 °C Adsorption time: 1 h Adsorbent dosage: 8.0 x 10 ⁻⁴ g/mL pH: 5.0 Qmax: 84.7 mg/g (Cu(II)), 81.9 mg/g (Cr(II)), 84.6 mg/g (Cd(II)), 83.3 mg/g (Pb(II))	Cu(II), Cr(II), Cd(II), Pb(II)	(Ibrahim et al., 2016)

Table 2.5 continued

5.	Papaya peel	AC Treatment: H ₃ PO ₄ Pyrolysis temperature: 450 °C Concentration: 200 mg/L Adsorption time: 2 h Adsorbent dosage: 0.05 g/mL Mixing rate: 150 rpm pH: 5.0 Q_{max}: 38.31 mg/g	Pb(II)	(Abbaszadeh et al., 2016)
6.	Rice Husk	AC Treatment: H ₃ PO ₄ Pyrolysis temperature: 105 °C (oven) Concentration: 200 mg/L Adsorption time: 3 h Adsorbent dosage: 0.02 g/mL pH: 6 Q_{max}: 77.519 mg/g	Pb(II)	(Dada et al., 2017)
7.	Molasses	AC Treatment: H ₃ PO ₄ Pyrolysis temperature: 500 °C Concentration: 50 mg/L (Pb(II) and Cr(VI)), 70 mg/L (Cu(II)) Adsorption time: 8 h Adsorbent dosage: 1.5 x 10 ⁻⁴ g/mL Mixing rate: 150 rpm pH: 4 Q_{max}: 144.93 mg/g (Cr(VI)), 303.03 mg/g (Pb(II)), 526.32 mg/g (Cu(II))	Cr(VI), Pb(II), Cu(II)	(Legrouiri et al., 2017)
8.	Wood Apple shell	AC Treatment: H ₂ SO ₄ Pyrolysis temperature: 600 °C Concentration: 75 mg/L Adsorption time: 2 h Adsorbent dosage: 1.25 x 10 ⁻⁶ g/mL pH: 1.8 Q_{max}: 151.51 mg/g	Cr(VI)	(Doke & Khan, 2017)
9.	Winemaking wastes	AC Treatment: KOH Pyrolysis temperature: 250 °C Concentration: 500 mg/L Adsorption time: 3 h Adsorbent dosage: 1.0 x 10 ⁻⁴ g/mL Mixing rate: 750 rpm pH: 4 Q_{max}: 58.31 mg/g	Pb(II)	(Alguacil et al., 2018)

Table 2.5 continued

10. Shaddock peel	AC Treatment: H ₃ PO ₄ Pyrolysis temperature: 450 °C Concentration: 50 mg/L Adsorption time: 3 h Adsorbent dosage: 0.001 g/mL Mixing rate: 140 rpm pH: 3 Q_{max}: 50.76 mg/g	Cr(VI)	(Tao et al., 2019)
11. Macadamia nuts	AC Treatment: H ₃ PO ₄ , H ₂ SO ₄ Pyrolysis temperature: 500 °C Concentration: 100 mg/L Adsorption time: 2 h Adsorbent dosage: 0.003 g/mL Mixing rate: 150 rpm pH: 9.0 (H ₂ SO ₄), 4.0 (H ₃ PO ₄) Q_{max}: 25.75 mg/g (H ₃ PO ₄), 17.51 mg/g (H ₂ SO ₄)	Cr(VI)	(Lesaoana et al., 2019)
12. Cotton textiles wastes	AC Treatment: FeCl ₃ Pyrolysis temperature: 700 °C Concentration: 200 mg/L Adsorption time: 24 h Adsorbent dosage: 0.002 g/mL Mixing rate: 150 rpm pH: 2 Q_{max}: 267.12 mg/g	Cr(VI)	(Xu et al., 2019)
13. Corn cob	AC Treatment: KOH Pyrolysis temperature: 800 °C Concentration: 0.06 mg/L Adsorption time: 2 h Adsorbent dosage: 2.0 x 10 ⁻⁵ g/mL Mixing rate: 200 rpm pH: 11.0 Q_{max}: 2.39 mg/g	Hg(II)	(Liu et al., 2020)
14. Olive branches	AC Treatment: H ₃ PO ₄ Concentration: 100 mg/L Adsorption time: 24 h Adsorbent dosage: 0.02 g/mL Mixing rate: 150 rpm pH: 5.0 (Pb(II), Cu(II), Cd(II)), 3.0 (Zn(II)) Q_{max}: 41.32 mg/g (Pb(II)), 43.10 mg/g (Cu(II)), 38.17 mg/g (Cd(II)), 34.97 (Zn(II))	Pb(II), Zn(II), Cu(II), Cd(II)	(Alkherraz et al., 2020)

2.10.2 AC for Dyes Adsorption

Dyes contaminants in the wastewater are usually coming from textile industries that being released to the nearby stream. (Cheng et al., 2017). The industry would discharge the wastewater directly into the nearby river before treatment causes pollutions and damage to the aqueous environment. The effluent from the textile industry has high pH, temperature (Fazal et al., 2019), COD, BOD, vibrant colour, and low biodegradability (Shu et al., 2017). Apart from textile industries, other industries such as cosmetic (Sangon et al., 2017), printing (Danish et al., 2018), food, and stationaries industries are also contributing dye-containing wastewater as much as the textile industry (Li et al., 2021).

It is reported that approximately 60,000 – 90,000 tons of dyes were used in 2016 (Bedin et al., 2016) and increased to 178, 000 tons of dyes in 2021 (Araujo et al., 2021). Some industries conducted water treatment before the effluent is discharged into the environment however most of the dye runoff managed to escape the wastewater treatment and stays in the environment (Shu et al., 2017). Some dyes are refractory which means it is resistant to biological treatment, stable to light, temperature, water, and other chemicals (Herrera-gonzález et al., 2019)

Among all, MB is one of the most commonly used dyes in industries. Reddy et al. (2016) stated that 10 – 20% of MB was found in the dye bath and released together with other deposits. MB is hazardous, toxic, and carcinogenic to humans and the environment. Besides, it is dangerous to aquatic life and might be transferred along the way through the food chain although in low concentration due to its low biodegradability (dos Santos et al., 2019). Thus, it is necessary to remove MB from the environment. Many studies reported on the removal of MB from an aqueous environment through coagulation, flocculation, magnetic separation (Nayara et al., 2018), electrooxidation in filter press electrochemical

flow cells (Gharibian et al., 2020), solar irradiation (Salem et al., 2020) and electrolysis (Liu et al., 2020). However, the treatment methods have a huge downturn in terms of cost-effectiveness, practicality, and efficiency

The adsorption employed AC has raised the attention of many researchers for its' effectiveness, environmentally friendly and cheap approach (Teimouri et al., 2019). Besides, the unique physicochemical series of activated carbon as well as its mechanical and electrical properties with large surface areas make an ideal sorbent for the removal of dyes (Aliakbari et al., 2017). Table 2.6 reported the recent literature precedent of the employment of AC and the adsorption capacity on many types of dyes including MB.

Acid activation for AC using various types of acids such as zinc chloride (ZnCl_2), phosphoric acid (H_3PO_4), and sulphuric acid (H_2SO_4) reported to give good adsorption capacity for dyes in wastewater. Teimouri et al. (2019), utilizes ZnCl_2 -activated walnut shell (WS) and almond shell (AS) for the removal of MB with a maximum adsorption capacity of 98.10 mg/g and 113.90 mg/g respectively. A year after, Li et al. (2020) suggested that the pyrolysis of WS at higher temperature (800 °C) resulted in higher adsorption capacity of MB (381.88 mg/g) as the high-temperature pyrolysis leads to the production of larger AC pore structures. The activation of peanut shells with Iron chloride (FeCl_3) gave a significantly high removal of malachite green (1106.40 mg/g) due to the improvement of adsorbent surface area and porosity (Feiqiang et al., 2018). However, the drawbacks of acid-activation AC are highly corrosive, non-environmentally friendly, expensive and lack of practicality causes the attention to shift to base-activation over the years (Pan, 2020).

Moreover, the activation of AC with NaOH and KOH resulted in a promisingly high surface area and adsorption capacity on various dyes especially MB. Sunflower pith AC shows high surface area when activated with NaOH (2690 m^2/g) and KOH (2090 m^2/g)

which resulted in high removal efficiency of MB 965 mg/g and 580 mg/g respectively (Baysal et al., 2018). Vinasses wastes AC is reported to have significantly high removal efficiency on MB (2000 mg/g). Li et al. (2021) mentioned that the vinasses wastes is activated with NaOH and pyrolyzed at different temperatures to give AC with high surface area when analysed with BET (2015 m²/g: 700 °C) (1999 m²/g: 800 °C).

Table 2.6: The adsorption capacity on dyes adsorption of different types of AC with different activation conditions

No.	Precursor	Treatments and Adsorptions	Pollutants	Citations
1.	Tomato Wastes	AC Treatment: ZnCl ₂ Concentration: 550 mg/L Adsorption time: 24 h Adsorbent dosage: 6.0 g/L Mixing rate: 120 rpm pH: 6.33 (MB), 6.23 (MY) Qmax: 400 mg/g (MB), 385 mg/g (MY)	MB, Metanil Yellow (MY)	(Saygılı & Güzel, 2016)
2.	Coconut shell	AC Treatment: NaOH Concentration: 1000 mg/L Adsorption time: 150 min Adsorbent dosage: 0.1 g/L pH: 6.5 Qmax: 916.26 mg/g	MB	(Cazetta et al., 2016)
3.	<i>Elaeagnus</i> stone	AC Treatment: ZnCl ₂ Concentration: 200 mg/L Adsorption time: 180 min Adsorbent dosage: 1 g/L Mixing rate: 150 rpm pH: 7 Qmax: 288.18 mg/g (MB), 281.69 mg/g (RB), 432.90 mg/g (MG)	MB, Rhodamine B (RB), Malachite Green (MG)	(Gecgel et al., 2016)
4.	Denim fabric wastes	AC Treatment: H ₃ PO ₄ Concentration: 200 mg/L Adsorption time: 240 min Adsorbent dosage: 0.8 g/L Mixing rate: 200 rpm pH: 2.1 Qmax: 292 mg/g	Remazol Brilliant Blue R (RBBR)	(Silva et al., 2017)

Table 2.6 continued

5.	Rice straw wastes	AC Treatment: H_3PO_4 Concentration: 6 mg/L (MB), 22 mg/L (CR) Adsorption time: 24 h Adsorbent dosage: 0.01 g/L Mixing rate: 250 rpm pH: 7.0 Qmax: 167.50 mg/g (MB), 390.62 mg/g (CR)	MB, Congo Red (CR)	(Sangon et al., 2017)
6.	Banana trunk	AC Treatment: H_3PO_4 Concentration: 150 ppm Adsorption time: 51 min Adsorbent dosage: 4.0 g/L pH: 7.0 Qmax: 64.66 mg/g	MB	(Danish et al., 2018)
7.	Peanut shells	AC Treatment: FeCl_3 Concentration: 800 mg/L Adsorption time: 24 h Adsorbent dosage: 1.0 g/L Mixing rate: 150 rpm pH: 8.0 Qmax: 1106.40 mg/g	Malachite Green (MG)	(Feiqiang et al., 2018)
8.	Coffee wastes	AC Treatment: CH_3COOK Concentration: 50 mg/L Adsorption time: 120 min Adsorbent dosage: 2 g/L Mixing rate: 220 rpm pH: 3 Qmax: 90.90 mg/g	Congo Red (CR)	(Lafi et al., 2018)
9.	Empty Cotton Flower	AC Treatment: KOH Concentration: 200 mg/L Adsorption time: 30 min Adsorbent dosage: 93.5 g/L pH: 5.9 Qmax: 720 mg/4.67 g	Reactive Orange 84 (RO 84)	(Charola et al., 2018)
10.	Sunflower pith	AC Treatment: KOH, NaOH Concentration: 250 mg/L Adsorption time: 3 h Adsorbent dosage: 1 g/L Mixing rate: 200 rpm Qmax: 965 mg/g (NaOH), 580 mg/g (KOH)	MB	(Baysal et al., 2018)

Table 2.6 continued

11.	Acai seeds (AS), Brazil nutshell (BS)	AC Treatment: H ₃ PO ₄ Concentration: 250 mg/L Adsorption time: 24 h Adsorbent dosage: 3 g/L pH: 5.8 Qmax: BB26 (AS: 73 mg/g, BS: 71 mg/g), BG1 (AS: 83 mg/g, BS: 82 mg/g), BY2 (AS: 31 mg/g, BS: 58 mg/g), BR1 (AS: 34 mg/g, BS: 39 mg/g)	Basic blue 26 (BB26), Basic green 1 (BG1), Basic yellow 2 (BY2), Basic red 1 (BR1)	(Nayara et al., 2018)
12.	Walnut and (WS) Almond shell (AS)	AC Treatment: ZnCl ₂ Concentration: 25 mg/L Adsorption time: 24 h Adsorbent dosage: 1 g/L Mixing rate: 3500 rpm Qmax: 98.1 mg/g (WS), 113.9 mg/g (AS)	MB	(Teimouri et al., 2019)
13.	<i>Ziziphus</i> lotus stone	AC Treatment: H ₃ PO ₄ Concentration: 100 mg/L Adsorption time: 180 min Adsorbent dosage: 0.5 g/L Mixing rate: 300 rpm pH: 5.8 Qmax: 423 mg/g (BY28), 306 mg/g (BR46)	Basic yellow 28 (BY28), Basic red 46 (BR46)	(Boudechiche et al., 2019)
14.	Corn cob	AC Treatment: H ₂ SO ₄ Concentration: 100 mg/L Adsorption time: 250 min Adsorbent dosage: 1.2 g/L Mixing rate: 110 rpm pH: 5.6 Qmax: 216.2 mg/g	MB	(Jawad & Mohammed, 2019)
15.	Pineapple crown leaves	AC Treatment: KOH Concentration: 20 mg/L Adsorption time: 180 min Adsorbent dosage: 6 g/L Mixing rate: 120 rpm pH: 5 Qmax: 31.24 mg/g	Methyl violet (MV)	(Astuti et al., 2019)

Table 2.6 continued

16.	Walnut shell (WS) and ashitaba wastes (AW)	AC Treatment: ZnCl ₂ Concentration: 200 mg/L (CR), 100 mg/L (MB) Adsorption time: 9 h Adsorbent dosage: 0.1 g/L Mixing rate: 300 rpm pH: 7.0 Q_{max}: 632 mg/g (AW-CR), 381.88 mg/g (AW-MB), 442.45 mg/g (WS-CR), 400.11 mg/g (WS-MB)	Congo Red (CR), MB	(Li et al., 2020)
17.	Vinasses wastes	AC Treatment: NaOH Concentration: 500 mg/L Adsorption time: 300 min Adsorbent dosage: 0.25 g/L Mixing rate: 150 rpm pH: 8.0 Q_{max}: 2000 mg/g	MB	(Li et al., 2021)

2.11 Chapter Summary

In Summary, many studies reported on the utilisation of sago *hampas* and bark wastes as well as other lignocellulosic biomass as the precursor for AC. Various carbonization method and pyrolysis rate were reported that produces AC with enhanced pore structure, surface morphology and particle size. The application of lignocellulosic AC for the adsorbent for pollutants such as heavy metals and dyes were also reported (Bashir et al., 2019; Fazal et al., 2019). However, this review revealed a lack of significant extant literature on the reutilization of SE as the adsorbent for pollutants in aqueous solutions. Previous studies conducted on SE are inconclusive and limited to its employment as medium of cultivations for various microorganisms. Thus, this review led to the development of experimental framework on the repurposing of SE into a value-added product. The high C:N:P ratio content in SE indicated that it is a suitable medium for biological treatment such as ASP to generate solid biomass (SE-B) which was further pyrolysed and activated to become SE-AC. Furthermore, the reutilisation of SE in this study provides a sustainable,

practical, and low-cost alternatives for sago industries' environmental management.

CHAPTER 3

METHODOLOGY

3.1 Materials

Sago effluent was collected from Herdsen Sago Industries Sdn Bhd located in Pusa, Sarawak. The sago mill effluent was filtered with gauge netting and kept in gallons as raw sago effluent and brought back to the laboratory for further analysis.

Chemicals such as sodium hydroxide (NaOH), methylene blue (MB) powder, nitric acid, lead (Pb) solutions (1000 mg/L) were purchased from Merck. Hydrochloric acid (HCl) was obtained from Fischer Scientific and ethanol from HmBG.

3.2 Preparation of Adsorbent

3.2.1 Activated Sludge Process (ASP)

Sago effluent was placed in three different tanks. Unoptimized tank (Tank C) (20 mL) was aerated for 7 days, filtered, and sun-dried for 2 days to give 52.2 g of sago effluent biomass (SE-B). Tank A: Optimization in Tank A was conducted following (Slade et al., 2011) with C:N:P ratio 100:10:1. A mixture of urea (24 g) in 1000 mL distilled water and phosphoric acid (200 mL) in 1000 mL distilled water was added into SE (20 L) in a 25 L tank.

Optimization in Tank B was conducted following (Slade et al., 2011) with C:N:P ratio 100:10:3. A mixture of urea (24 g) in 1000 mL distilled water and phosphoric acid (200 mL) in 1000 mL distilled water was added into SE (20 L) in a 25 L tank.

3.2.2 Pyrolysis of SE-B

The SE-B was sieved using a 45-micron sieve and samples with size <45-micron were collected for the pyrolysis experiment. Pyrolysis was conducted in a tube furnace (TI-01200-50SL) at 400 °C for 30 min with nitrogen gas flow to create an inert atmosphere. The solid product of pyrolysis, sago effluent biochar (SE-BC) was cooled to room temperature before being collected (Eum et al., 2012; Ahmad et al., 2014).

3.2.3 Activations of SE-BC

SE-BC (5 g) was chemically activated by soaking in NaOH (20 mL, 5 M) for 2 hr. The SE-BC was filtered and washed with distilled water until pH 7 was achieved. The solid obtained was oven-dried (100 °C) for 24 h. The dried solid was pyrolyzed again (500 °C) for 90 min (Cao et al., 2017) followed by treatment with HCl (5 M) to remove any impregnating salts. The solid was washed with distilled water till it reached pH 4 (Wahi et al., 2009). The solid was oven-dried (100 °C) for 1 hr to obtain sago effluent activated carbon (SE-AC) (3 g). Figure 3.1 summarizes the flow of preparing SE-AC.

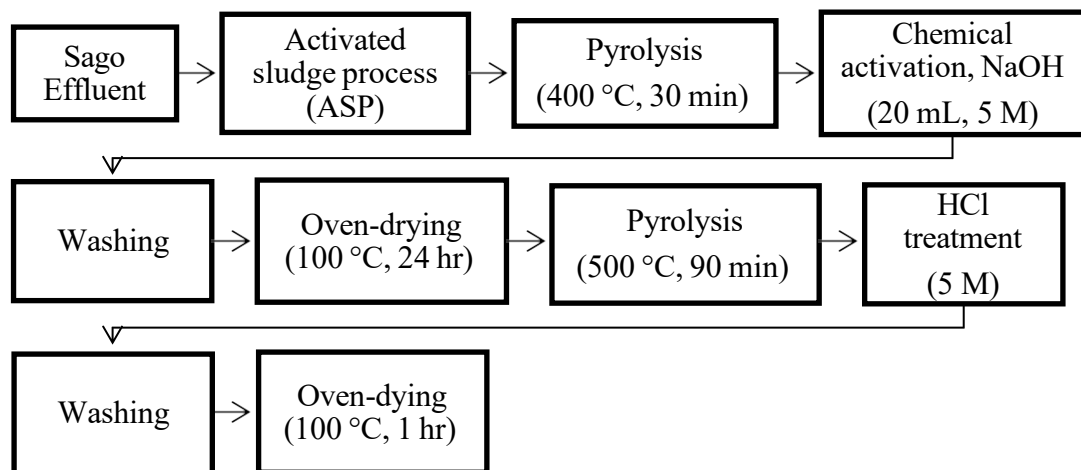


Figure 3.1: Flow chart of SE-AC preparation

3.3 Characterization of Adsorbent

3.3.1 Proximate Analysis

The procedure for obtaining the moisture content, volatile matter content, and ash content of SE-B, SE-BC, and SE-AC was adapted from the American Society for Testing Materials (ASTM) ASTM D-3173, D-3175, and D-3174 respectively.

Moisture Content

Empty dried crucible (W_e) was weighed. A certain amount of samples was added into the crucible, weighed, and labelled as W_s . The sample was dried at 105 °C for 24 h. The sample was dried until a constant weight was obtained. The dried sample was weighed again and denoted as W_d . The moisture content of samples was calculated by using Equation 3.1,

$$\text{Moisture Content (\%)} = \frac{W_s - W_d}{W_s - W_e} \times 100 \% \dots\dots\dots (\text{Eq 3.1})$$

Volatile Matter Content

Empty dried crucible (W_e) was weighed. A certain quantity of samples was placed into the crucibles, weighed, and labeled (W_s). The sample was dried for 7 min in a furnace at 500 °C. The dried sample was then weighed and denoted as W_v . The volatile matter content of samples was calculated by using Equation 3.2,

$$\text{Volatile Matter Content (\%)} = \frac{W_s - W_v}{W_s - W_e} \times 100 \% \dots\dots\dots (\text{Eq 3.2})$$

Ash Content

An empty dried crucible was weighed (W_e). The sample was kept dry for 24 hr in a desiccator before the ashing process. The dried sample was placed into the crucible and weighed (W_s). The sample was burnt in the furnace for 3 hr with the first 1 hr at 500 °C and the subsequent 2 hr at 700 °C. The crucible with the dried sample was labeled (W_a). The ash content was calculated by using Equation 3.3,

$$\text{Ash Content (\%)} = \frac{W_s - W_a}{W_s - W_e} \times 100 \% \dots\dots\dots(\text{Eq 3.3})$$

Total Carbon Content

Total carbon content in samples was calculated by using Equation 3.4,

$$\text{Total Carbon Content (\%)} = 100 \% - (\% \text{ Moisture} + \% \text{ Volatile} + \% \text{ Ash}) \dots\dots\dots(\text{Eq 3.4})$$

3.3.2 Instrumental Analysis

The characterization of the functional groups' presence in SE-B, SE-BC, and SE_AC was conducted by using Fourier transform infrared spectroscopy (FTIR) (Thermo Scientific Nicolet iS10) at the Faculty of Resource Science and Technology, UNIMAS. The samples (2 mg) were ground and compressed with dried KBr powder into a pellet. The pellets with samples were analyzed and IR spectra were plotted over a frequency range from 400 – 4000 cm^{-1} .

The surface morphology and pore structure were analyzed using a scanning electron microscope (SEM) (JEOL JSM06390LA). The samples (SE-B, SE-BC, and SE-AC) were coated with think film of gold or platinum using Auto Fine Coater (JEOL JFC-1600). The beam was scanned across the samples' surface at 1000x magnification and images of samples were collected.

The total surface area of SE-B, SE-BC, and SE-AC was analyzed using Brunauer, Emmett, and Teller (Quantachrome Instruments Nova 3200e). The particle size of SE-BC and SE-AC was analyzed using Transmission Electron Microscope (JEOL 1230 Electron Microscope).

3.4 Batch Adsorption

3.4.1 Adsorption of Pb(II)

3.4.1.1 Preparation of Pb (II) Stock Solution

The stock of Pb solution (100 mg/L) was prepared by diluting the 1000 mg/L standard in a 1 L volumetric flask to make a 100 mg/L stock solution. 5 mL of 65% of nitric acid was added into the solution to preserve the stock solution.

3.4.1.2 Factors Affecting the Adsorption of Pb(II)

Adsorbent Dosage

Pb (II) solution (5 mg/L) was prepared in 100 mL distilled water in a 250 mL conical flask. The procedure was carried out at room temperature. Various weights of adsorbent ranging from 0.1 – 0.5 g were added into the conical flask and agitated (260 rpm) for 30 min

(Bishnoi et al., 2004). The adsorbent was filtered and analyzed using ICP-OES (Kumar et al., 2009).

Concentration of Pb (II) solution

Various initial concentrations of Pb (II) were prepared (1 – 5 mg/L) in 100 mL distilled water in a 250 mL conical flask. The procedure was carried out at room temperature. Adsorbent dosage (0.5 g) was added into the conical flask and agitated (260 rpm) for 30 mins.

pH Value of Pb (II) Solution

Pb (II) solution (5 mg/L) was prepared in 100 mL distilled water in a 250 mL conical flask. The procedure was carried out at room temperature. The solution was adjusted to a variety of pH values (2, 4, 6, 8, 10). Adsorbent (0.5 g) was added into the conical flask and agitated at 260 rpm for 30 min.

3.4.2 Adsorption of MB

3.4.2.1 Preparation of MB Stock Solution

Stock solution 1000 mg/L was prepared by dissolving 0.1 g of MB powder in 1 L of distilled water/ The stock solution was wrapped with an aluminium sheet and stored in a dark area to avoid UV degradation. The stock solution was diluted into desired concentration (2 – 10 mg/L).

3.4.2.2 Factors Affecting the Adsorption of MB

Initial Concentration

MB solutions (100 mL) of different concentration (2 – 10 mg/L) was placed in a 250 mL conical flask. Adsorbent (0.5 g) was added into the solution and the mixture was agitated (260 rpm) at room temperature for 30 mins. The final MB concentration in the flask was calculated based on the calibration curve obtained from the absorbance value noted using UV-Vis Spectrophotometer.

Contact Time

Adsorbent dosage (0.5 g) was agitated (260 rpm) in 100 mL of MB solution (10 mg/L) in different contact times (5 - 30 min) respectively. The efficiency of removal was calculated and a graph of percentage removal against contact times was plotted.

3.4.3 The Removal of Pb(II) and MB

Final Concentration of MB

A calibration graph of standard solution (2 – 10 mg/L) was plotted to obtain the graph linear expression whereby y is the final concentration and x is the absorbance value denoted from UV-Vis.

Adsorption Capacity

The adsorption capacity of Pb(II) and MB was calculated using Equation 3.5;

$$q_e = \frac{C_o - C_e}{m} \times V \dots\dots\dots \text{(Eq 3.5)}$$

Where, C_o = Initial concentration of MB, C_e = Final concentration of MB, V = Volume of solution (mL), and m = Adsorbent dosage (mg)

Percentage of Removal

The percentage removal of Pb(II) and MB was calculated using Equation 3.6;

$$\% \text{ Removal} = \frac{C_o - C_e}{C_o} \times 100 \% \dots\dots\dots (\text{Eq 3.6})$$

Where, C_o = Initial concentration of Pb(II)/MB, and C_e = Final concentration of Pb(II)/MB respectively.

3.4.4 Isotherm Study

Adsorption isotherm indicates how the adsorption of molecules is distributed between the liquid phase and solid phase when the adsorption process reaches an equilibrium state (Üner et al., 2016). The adsorption isotherm is commonly divided into two types of namely Langmuir Isotherm and Freundlich Isotherm.

Langmuir Isotherm

In this study, the Langmuir sorption study is applied to equilibrium sorption assuming there is monolayer sorption onto a surface with a finite number of identical sites (Pathania et al., 2017). The Langmuir isotherm can be expressed in linear form (Equation 3.7);

$$\text{Langmuir isotherm: } \frac{C_e}{q_e} = \frac{1}{Q_{ob}} + \frac{C_e}{Q_o} \dots\dots\dots(\text{Eq 3.7})$$

Where, $Q_{ob} = C_e/q_e$ vs C_e graph slopes and linear intercepts

Langmuir Isotherm describes the monolayer adsorption of solute on a finite number of localized sites over the homogenous adsorbent surface (Foo & Hameed, 2012; Pathania et al., 2017) In Langmuir, the value for Q_o and b are obtained from the slopes and intercepts of the linear plot of (C_e/q_e) vs C_e . The Q_o and b are constants related to adsorption capacity and energy of adsorption respectively (Pathania et al., 2017).

The separation factor (R_L) was calculated to predict the affinity between sorbate and sorbent as a supporting value if only the adsorption is best fitted Langmuir Isotherm following Equation 3.8,

$$\text{Equilibrium parameter, } R_L = \frac{1}{(1+bC_e)} \dots\dots\dots(\text{Eq 3.7})$$

Where, b = Langmuir constant (L/mg), and C_e = Initial concentration (mg/L)

Freundlich Isotherm

Freundlich isotherm is adsorption that occurs on a heterogeneous layer and interacts between adsorbed molecules with various kinds of energies (Gao et al., 2013). Freundlich isotherm can be written in Equation 3.8;

$$\text{Freundlich isotherm: } \log q_e = \log K_f + \frac{1}{n} \log C_e \dots\dots\dots(\text{Eq 3.8})$$

Where, Q_e = Amount of adsorbed particles per unit mass, and K_f = Constant (Adsorption capacity) – graph of $\log q_e$ against $\log C_e$

Freundlich Isotherm was applied for the heterogeneous surface where q_e is the amount of adsorbate adsorbed per unit mass of adsorbent at equilibrium. The values of K_f and $1/n$ are correlated to the sorption intensity and the adsorption capacity of the system (Alghamdi et al., 2019).

3.5 Response Surface Methodology (RSM)

RSM is a statistical method which one of the alternative methods to optimize the certain process by relating the mutual interactions among all variables and provides an estimation of the joined impact of these variables on results (Chaudan et al., 2006). A second-order design called Central Composite Design (CCD) based RSM was used in this study to determine and evaluate the optimum variables on the adsorption efficiency of Pb(II) and methylene blue. The operating variables chosen were adsorbent dosage, initial concentration, and contact time was chosen for the adsorption of Pb(II) (Table 3.1) while the operating variables chosen for the adsorption of MB are pH, initial concentration, and contact time (Table 3.2). Design Expert software 7.1.6 (trial version) was used for experimental design, ANOVA analysis, and generation of RSM plots.

Table 3.1: Experimental Factors and Response for the Adsorption of Pb(II)

Standard	Parameters		
	Initial Concentration (mg/L)	Adsorbent Dosage (g)	Contact Time (min)
1	1.00	0.10	30.00
2	5.00	0.10	30.00
3	1.00	0.50	30.00
4	5.00	0.50	30.00
5	1.00	0.10	90.00
6	5.00	0.10	90.00
7	1.00	0.50	90.00
8	5.00	0.50	90.00
9	3.00	0.30	60.00
10	3.00	0.30	60.00
11	1.00	0.30	60.00
12	5.00	0.30	60.00
13	3.00	0.10	60.00
14	3.00	0.50	60.00
15	3.00	0.30	30.00
16	3.00	0.30	90.00
17	3.00	0.30	60.00

Table 3.2: Experimental Factors and Response for the Adsorption of MB

Standard	Parameters		
	pH	Initial Concentration (mg/L)	Contact Time (min)
1	2.00	2.00	30.00
2	10.00	2.00	30.00
3	2.00	10.00	30.00
4	10.00	10.00	30.00
5	2.00	2.00	90.00
6	10.00	2.00	90.00
7	2.00	10.00	90.00
8	10.00	10.00	90.00
9	6.00	6.00	60.000
10	6.00	6.00	60.00
11	6.00	6.00	60.00
12	6.00	6.00	60.00
13	2.00	6.00	60.00
14	10.00	6.00	60.00
15	6.00	2.00	60.00
16	6.00	10.00	60.00
17	6.00	6.00	30.00

Table 3.2 continued

18	6.00	6.00	90.00
19	6.00	6.00	60.00
20	6.00	6.00	60.00

CHAPTER 4

RESULTS AND DISCUSSION

4.1 The Yield of Sago Effluent Activated Sludge Process (ASP)

The ASP was conducted in a 25 L tank at three different conditions, namely sago effluent modified at C:N:P ratio 100:10:1 (Tank A), C:N:P ratio 100:10:3 (Tank B), and unmodified sago effluent as a control experiment (Tank C). Seven-day aeration during ASP yielded biomass (SE-B) of 93.2 g in Tank A, 40.1 g (Tank B) and 52.2 g (Tank C).

The aeration process conducted on the three tanks provides oxygen for the aerobic bacteria to degrade carbonaceous and nitrogenous matters in SE for energy and resulted in the growth of the bacterial population (Kristensen et al., 2018). Nitrifying bacteria available in Tank A utilises the N from urea and C in SE to carry out the nitrification process and produce energy. However, due to the relatively small quantity of energy produced during nitrification, the bacteria started to degenerate and form flocs (Khan et al., 2017). Filamentous bacteria utilizes P for growth and aid in the flocculation of the degenerated nitrifying bacteria (Gebrati et al., 2018). For this reason, dense and stable sediments are produced, which results in an improved yield of biomass (Ahansazan et al., 2014) in Tank A.

In contrast, an excessive amount of P in Tank B reduced the efficiency of organic carbon removal during ASP as it disturb the growth of filamentous bacteria thus reducing the flocculating and coagulating ability to form stable sediments (Slade et al., 2011). Tank C had a lower yield compared to Tank A, which can be explained by the lack of nutrients that are readily available in SE thus the degeneration of bacteria took part before bacterial growth as the result of lack of energy production (Anbalagan et al., 2016).

Figure 4.1 shows the colour and physical appearance of effluent in all tanks after seven days of ASP. Tank A was observed to have significant layers of clear water on top and sediments settled at the bottom of the tank. This proves that the SE in Tank A underwent complete ASP at optimum nutrients adjustment and the clear water indicated that the BOD level was reduced throughout the process (Sepehri & Sarrafzadeh, 2019). On the other hands, Tank B shows dark coloured sludge with no visible separation between clear water and sediments. Slimy and squishy lumps were observed due to the poor settling. The observation proved that excess P disrupt the growth of filamentous bacteria that supposed to help the settling process of flocs (Grau, 2018). Meanwhile, in Tank C, there was sediments form at the bottom of the tank, however the layer of water on top was still very cloudy indicated an incomplete ASP due to the lack of important nutrients for excellent floc formation and settlement (Sepehri & Sarrafzadeh, 2019).

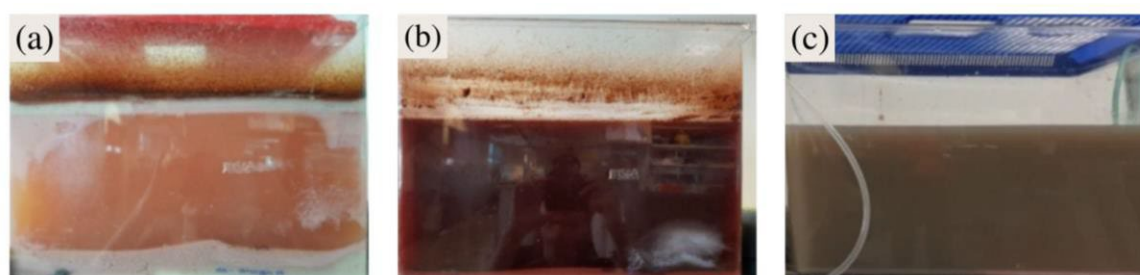


Figure 4.1: ASP of SE with different C: N: P ratio of tank (a) 100:10:1; (b) 100:10:3; and tank (c) unmodified SE, at Day 7 of aeration and 5 days settling.

4.2 Characterization of Sago Effluent Activated Carbon (SE-AC)

The physicochemical characterization of SE-AC was conducted and compared to SE-B and SE-BC and described below. Results showed that the activated carbon exhibited a good adsorptive property.

4.2.1 Proximate Analysis

Table 4.1 showed the proximate analysis of SE-B and SE-AC. The moisture content of SE-B (0.51%) was very low compared to other sago wastes such as sago bark (4.90%) and sago *hampas* (7.20%) (Siruru et al., 2019). This is due to the drying process of SE-B under the sun for two days that causes water evaporation. The low moisture content of an AC precursor is advisable because high moisture content (>30%) in biomass causes longer pyrolysis time, which subsequently increases the energy consumption. This is because more energy is required for the removal of moisture from biomass and for increasing the carbonization temperature for carbonization (Jankovi et al., 2019). The results also showed that SE-AC had very low moisture content, which could be due to the long pyrolysis period (Wahi & Senghie, 2011).

The volatile matter content in SE-AC was also low, probably due to the dehydration and decarboxylation of SE-B during pyrolysis and chemical activation. The afore-mentioned processes released elements such as hydrogen, carbon and oxygen to produce carbonyl and carboxylic groups in the activated carbon (Menya, 2019; Siruru et al., 2020). The complete removal of volatile matter in SE-AC also indicated that there is very little possibility of clogged pores on its surface (Wahi et al., 2009; Zhang et al., 2015). The release of volatile fractions during the conversion of SE-B to SE-AC has broken the weak bonds in SE-B matrices (Park et al., 2013; Jankovi et al., 2019), resulting in well-developed porous structures (Zbair et al., 2018).

The ash content of SE-B was also high compared to other agricultural precursors and was increased from 99.10% to 99.90% in SE-AC. High ash content is usually attributed to the high mineral content in the sample, such as calcium and potassium (Siruru et al., 2019; Pedicini et al., 2020). High ash content in SE-AC could clog the pore structure of AC,

causing low surface area and reduced ability of adsorption (Lam et al., 2017). The total carbon content in SE-B decreases from 0.22% to 0.08% in SE-AC. The low fixed carbon content in SE-AC was due to its high ash content.

Table 4.1: Proximate analysis results for SE-B and SE-AC

Tanks	SE-B	SE-AC
Moisture Content (%)	0.51 ± 0.02	0.00 ± 0.00
Volatile Matter Content (%)	0.08 ± 0.02	0.00 ± 0.00
Ash Content (%)	99.19 ± 0.03	99.92 ± 0.02
Total Carbon Content (%)	0.22 ± 0.01	0.08 ± 0.01

4.2.2 Fourier Transform Infrared (FTIR) Analysis

The FTIR spectra of SE-B, SE-BC, and SE-AC are shown in Figure 4.2. The adsorption peak at $3408.43 - 3426.45 \text{ cm}^{-1}$ in SE-B, SE-BC samples indicated the presence of $\nu\text{O-H}$ attributed to the vibration of cellulose, lignin, carboxylic acid, phenols, and alcohol. The broad O-H peak was reduced significantly in SE-AC due to the dehydration of cellulose and ligneous compound during pyrolysis and chemical activation (Rafiq et al., 2016). The weakening of the peaks was also due to the decomposition of the O-H functional group and the liberation of volatile matters during pyrolysis (Barkauskas & Dervinyte, 2004; Foo & Hameed, 2012b).

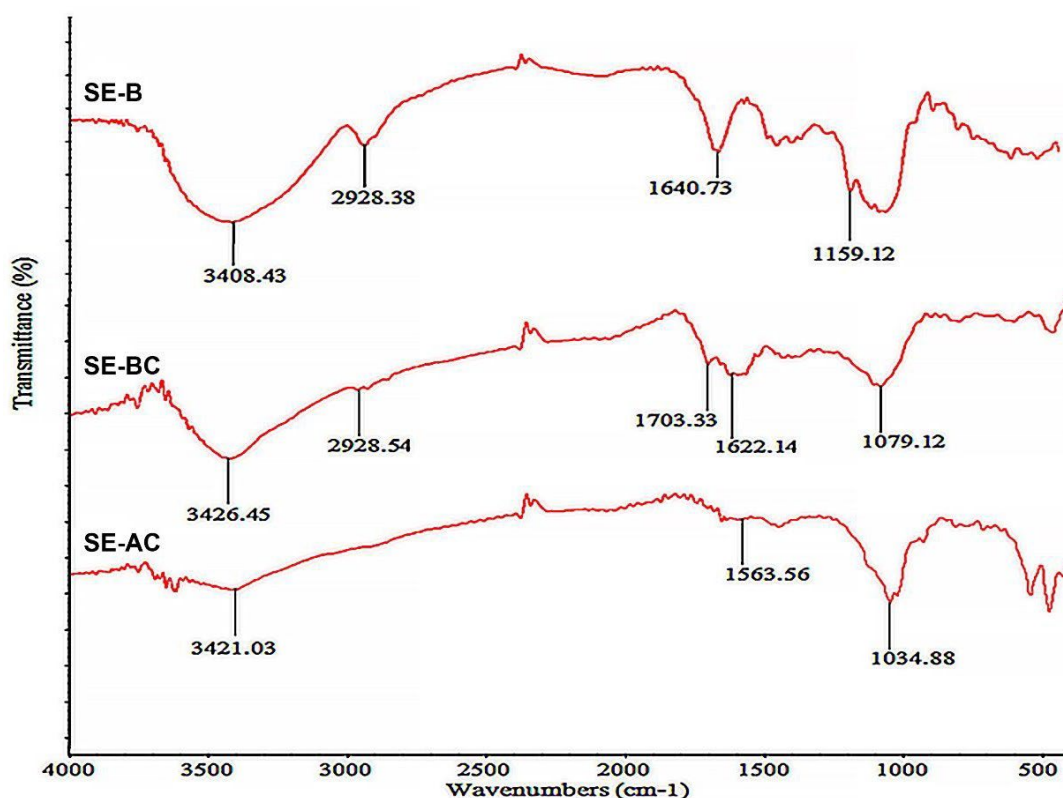


Figure 4.2: FTIR Spectrum of SE-B, SE-BC, and SE-AC

The weak peak at 2928.38 cm^{-1} in SE-B can be attributed to the presence of $\nu\text{C-H}$ ($-\text{CH}_2$, $-\text{CH}_3$) from alkenes and alkanes (Syamsu et al., 2020). The peak disappears in SE-AC due to the complete removal of the hydrogen-containing aliphatic group after pyrolysis and activation (Baysal et al., 2018). The peaks at 1622.14 and 1703.33 cm^{-1} in SE-B and SE-BC respectively, were attributed to $\nu\text{C}=\text{C}$ and $\nu\text{C}=\text{O}$ from lignin, hemicellulose, phenols, ester, and ketone. These functional groups were reduced in SE-AC due to the carbonization process and the reaction with NaOH during activations (Baysal et al., 2018; Syamsu et al., 2020).

The $\nu\text{C-N}$, $\nu\text{N-H}$ and $\nu\text{N-O}$ stretching observed in SE-AC ($1034.88 - 1563.56\text{ cm}^{-1}$) were due to the activation with NaOH at high molarity (Mary et al., 2016). The corresponding $-\text{OH}$ and oxygen functional groups present on SE-AC is important for

adsorption. It is also capable to acts as the active sites and capture the adsorbate molecules (Norouzi et al., 2018). The main functional groups found in SE-AC was similar to other AC from different precursors such as rattan wastes AC (Islam et al., 2017) and date pressed cake AC (Norouzi et al., 2018) shows strong -OH peak, reduced C-O groups and eliminates aromatic functional groups upon activation with NaOH.

4.2.3 Transmission Electron Microscope (TEM) Analysis

Figure 4.3a and b show the TEM image of SE-BC and SE-AC particles respectively. The particle size of SE-BC was in the range of 45 – 75 nm while the particle size of SE-AC was as small as 37 nm with a wider range. Pyrolysis in tube furnace can produce nano-size particles compared to the pyrolysis in a microwave which produced micro-diameter biochar ranging from 0.47 – 1000 μm (Fu et al., 2015; dos Santos et al., 2019). Tube furnaces is operating in high temperature during carbonization and able to reduce the particle size of biochar from micro-diameter to nano-diameter (Hedge et al., 2015).

During pyrolysis, the high temperature in the vacuum N_2 environment creates high pressure which leads to the agglomeration of char particles to form fine carbon fragments that can be in nano-diameters (Deng et al., 2019). A similar study conducted by Zhang et al. (2018) on the pyrolysis of bituminous coal in a tube furnace has also reported a nano-sized diameter due to the rapid heating rate and pressure of the condition. Smaller sized biochar particles (<250 nm) plays an important role in creating high-quality biochar or activated carbon for adsorption (Ricordel et al., 2001; dos Santos et al., 2019). Smaller particle diameters have greater uptake and accessibility to pores as well as large surface areas for mass adsorption (Senthilkumar et al., 2015). Moreover, the activation with NaOH also contributes to the reduction of particle size. The evaporation of NaOH-derived compounds

during carbonization leaves space that was previously occupied by the reagents creating small holes and brittle linings that collapsed and creates smaller particles (Shokry et al., 2019). Gaya et al. (2015) reported that the NaOH-AC have a smaller particle diameter (43.83 nm) compared to KOH-AC (190.10 nm).

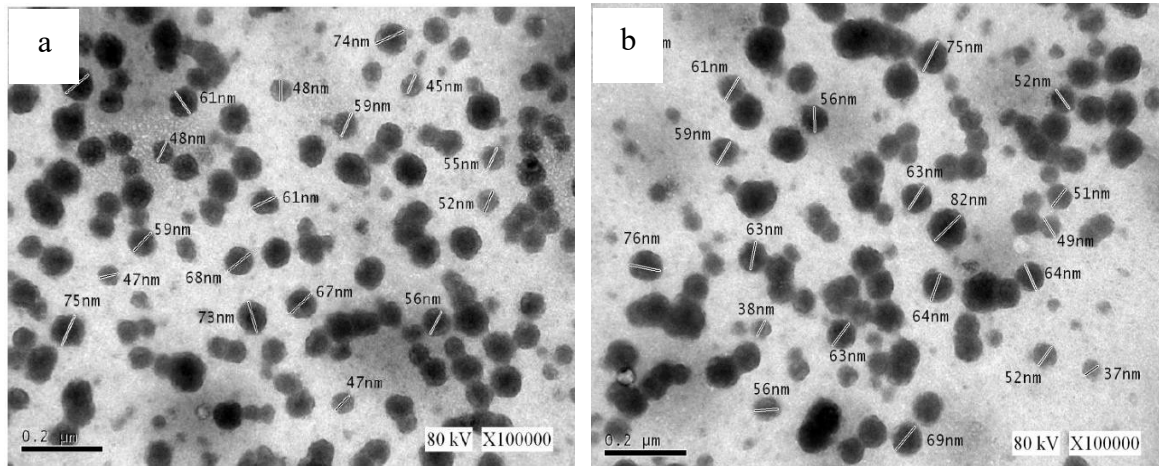


Figure 4.3: TEM image of (a) SE-BC and (b) SE-AC (Magnification: 100,000X)

4.2.4 Scanning Electron Microscope (SEM) Analysis

The surface morphology and pore structure of SE-B, SE-BC and SE-AC were observed using SEM analysis. Figure 4.4 shows the surface morphology of SE-B, SE-BC and SE-AC. There was no evidence of porous structure observed on the surface of SE-B (Figure 4.4(a)). Starch granules are attached onto the SE-B surfaces, which is commonly observed on the surface of raw biomass (Min et al., 2018). The surface of raw biomass consists of solid cells that are strongly bonded together with no visible pore structure (Tan et al., 2018).

Figure 4.4(b) shows that micropores (0.71 – 1.2 nm) were formed in SE-BC, and a relatively larger porous structure was observed in SE-AC (1.2 – 6.9 nm). Results indicated that the first pyrolysis at 400 °C had successfully generated pore structure *via* destruction of

starch granules on biomass surface (Siruru et al., 2020). Thermal degradation during pyrolysis has also caused the rigid SE-B structure to break and create pores in the afforded SE-BC (Shokry et al., 2019). Pyrolysis at a temperature of 400 °C resulted in the decomposition of hemicellulose at 220 – 315 °C, cellulose (at 300 – 400 °C) and a fraction of lignin which decomposed at a temperature above 190 °C. In addition, SEM image of SE-BC (Figure 4.5(b)) also showed the presence of particle debris on its surface. It is envisaged that the debris was resulted from the destruction of SE-B surfaces during pyrolysis. The debris must be removed in order to expose the porous network on SE-BC, increase its surface area and subsequently enhance the availability of adsorption sites on SE-BC surface.

Figure 4.4(c) reveals that the particle debris which was previously attached to the surface of SE-BC disappeared. Chemical activation with NaOH has successfully removed the surface debris, at the same time unclogged the pores which were previously occupied by starch granules (Tan et al., 2018). The second pyrolysis of the chemically modified SE-BC at 500 °C further developed the porous surfaces on the afforded SE-AC through the release of volatile matters (Lam et al., 2018). The SEM image also shows that the SE-AC had irregular pore diameters. The observed non-uniformity of pores could be due to incomplete pyrolysis at medium pyrolysis temperature (400 - 500 °C) (Tan et al., 2018). Higher temperature (up to 800 °C) produces biochar with larger and more regular pores (Liew et al., 2018; Siruru et al., 2019).

The types of activating agents during chemical activation of SE-BC play a vital role in determining the porosity, surface area, and quality of SE-AC produced. In SE-AC, the activation with NaOH created additional adsorption sites due to the enlargement of pores and interactions between specific functional groups (Min et al., 2018). The release of gaseous components during NaOH treatment knocked down the weakening bonds and walls

thus, aid the production of pores (Norouzi et al., 2018; Liu et al., 2020). The chemical activation process had also successfully enhanced the surface area and adsorptive properties of SE-AC compared to SE-BC, as discussed in Section 4.2.5, Section 4.3 and Section 4.4.

Second pyrolysis is an important step during the preparation of SE-AC. The second pyrolysis resulted in degradation of organic matrices which further weakens the precursor pore structure and creates larger pores as observed in SE-AC (dos Santos et al., 2019). The second pyrolysis also promoted the further decomposition of lignocellulosic materials in SE-AC (Singh, 2020), particularly a fraction of lignin that has not been fully degraded during the first pyrolysis. In fact, lignin degradation occurs at a broad range of temperatures (150–900 °C) (Siruru et al., 2019).

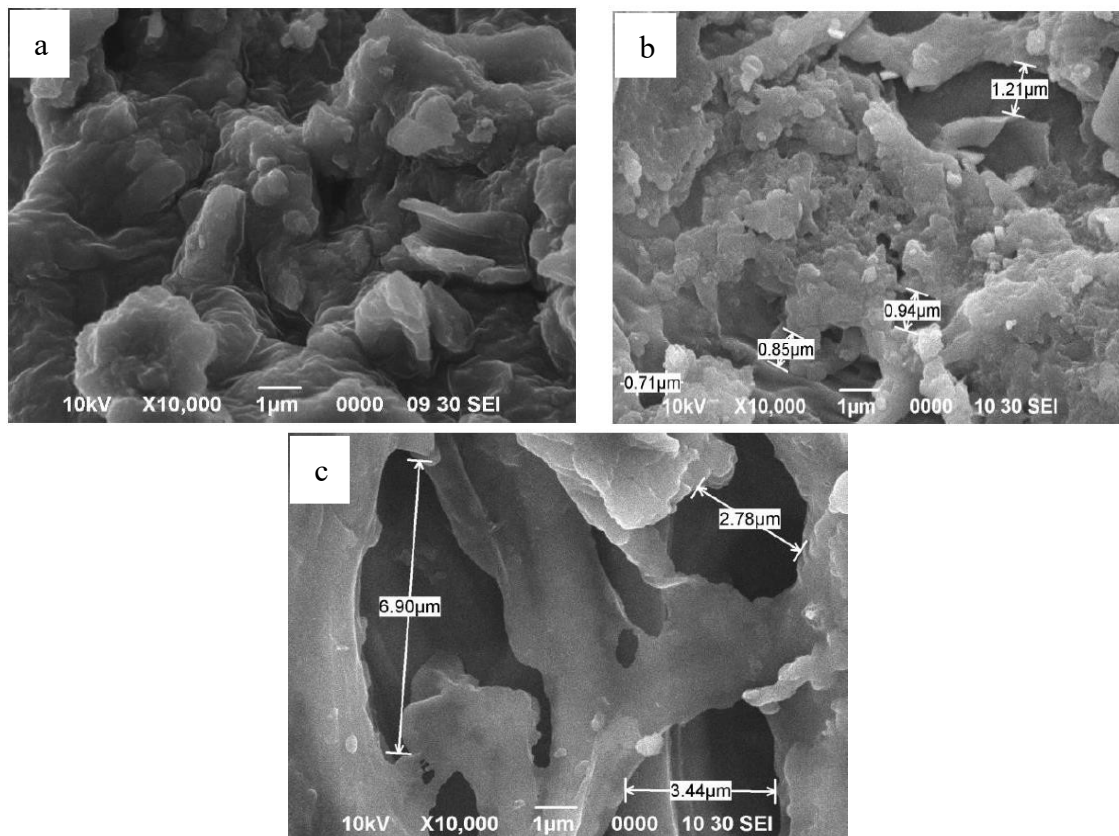


Figure 4.4: The SEM images for (a) SE-B, (b) SE-BC, and (c) SE-AC at magnification x10,000

4.2.5 Brunauer Emmett and Tellet (BET) Analysis

The surface area of SE-B, SE-BC, and SE-AC was characterized by using Brunauer, Emmet, and Teller (BET) analysis and the result is tabulated in Table 4.2. The surface area increased from 1.303 m²/g (SE-B) to 8.044 m²/g (SE-BC) and 78.863 m²/g (SE-AC). Results indicated that pyrolysis and chemical activation by NaOH has successfully enhanced the surface area in SE-AC. The increase of surface area after the first pyrolysis of SE-B can be explained by the volatilization of cellulose and hemicellulose during the first pyrolysis (Siruru et al., 2020). Further activations with NaOH unclogged pores and enhanced the formation of pores in the internal carbon structure *via* NaOH-C reaction (Liu et al., 2020). The second pyrolysis further improved the total surface area due to the degradation of lignin, cellulose, and hemicellulose (Jawad et al., 2017).

Table 4.2: The BET surface area of SE-B, SE-BC and SE-AC

Samples	Surface Area (m ² /g)
SE-B	1.303
SE-BC	8.044
SE-AC	78.863

The surface area of the adsorbent was in direct relationship with the efficiency of adsorption as mentioned by Nasrullah et al., (2018), higher surface area provides more availability of active binding sites on the adsorbent. This is agreeable with Baysal et al. (2018), whereby AC that was activated with NaOH have 29% higher total surface area and removal efficiency compared with AC activated with KOH.

4.3 Adsorption of Pb (II) on SE-AC

Batch adsorption was conducted to investigate the relationship of individual parameters on the adsorption of Pb(II).

4.3.1 The Effect of Adsorbent Dosage

Adsorbent dosage is an important factor affecting the rate of adsorption of particles. Figure 4.5 shows the effect of SE-AC adsorbent dosage (0.0 – 0.5 g) on the adsorption of Pb(II). In general, the adsorption of Pb(II) increased as the adsorbent dosage increased. At 0.5 g of adsorption dosage, the percentage of removal was at the maximum (99.78%) while at 0.1 g adsorbent dosage, the percentage removal was the least (27.40%). When the adsorption dosage is high, the availability of active sites also increases, which provides more binding sites of Pb(II) ions (Choudhary et al., 2020). At lower adsorbent dosage, limited active binding sites are available for the abundance of Pb(II) ions available in the solution.

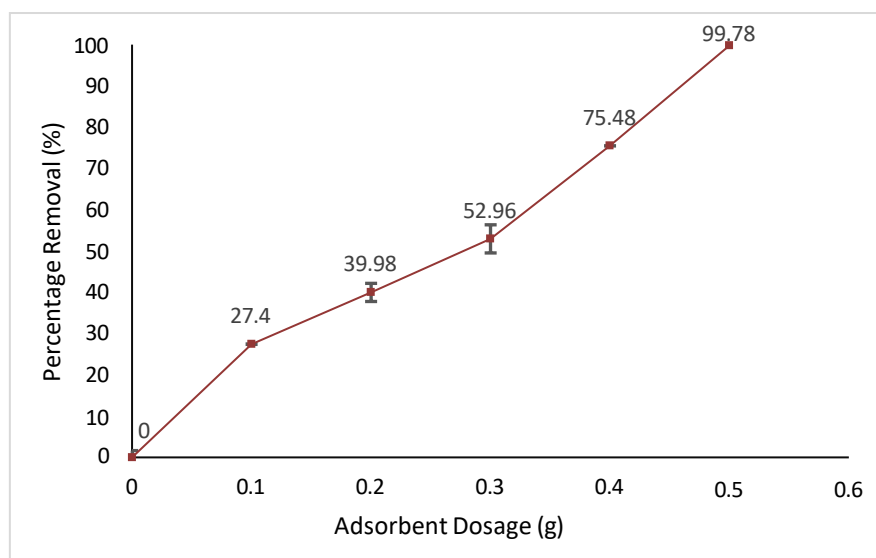


Figure 4.5: The graph of the effect of adsorbent dosage on the adsorption of Pb(II)

Many studies reported the same adsorption trend on increasing adsorbent dosage. Findings from this study are comparable to Krishnamoorthy et al. (2019) on the adsorption of Pb(II), whereby the adsorption showed a sharp increase when the adsorbent dosage

increased from 0.0005 to 0.002 g/mL. A similar study reported by Aboli et al. (2020) that the adsorption of Pb(II) increases with the adsorbent dosage where the adsorption increases from 83.43 to 98.32%. However, further increase in adsorbent dosage had no obvious effect on the adsorption due to the attainment of equilibrium and limited availability of Pb²⁺ ions to bind onto the surface of SE-AC (Gedam & Dongre, 2019).

4.3.2 The Effect of Initial Concentration

The effect of Pb(II) initial concentration on the removal of Pb(II) by SE-AC is shown in Figure 4.6. In general, the adsorption of Pb(II) decreased with increasing Pb(II) initial concentration. The highest Pb(II) removal (84.18%) was observed at the initial concentration of 1 mg/L, while the lowest removal (27.18%) was at a 5 mg/L initial concentration. The decreasing trend of Pb(II) removal as the initial concentration increase resulted from low availability of binding sites in SE-AC which limit the mass transfer of Pb(II) molecules at higher concentrations (Mehdizadeh et al., 2014; Anisuzzaman et al., 2015). At lower concentrations, the ratio of adsorbent dosage to the number of Pb²⁺ ions was higher, resulting in higher uptake of Pb²⁺ ions on SE-AC (Krishnamoorthy et al., 2019).

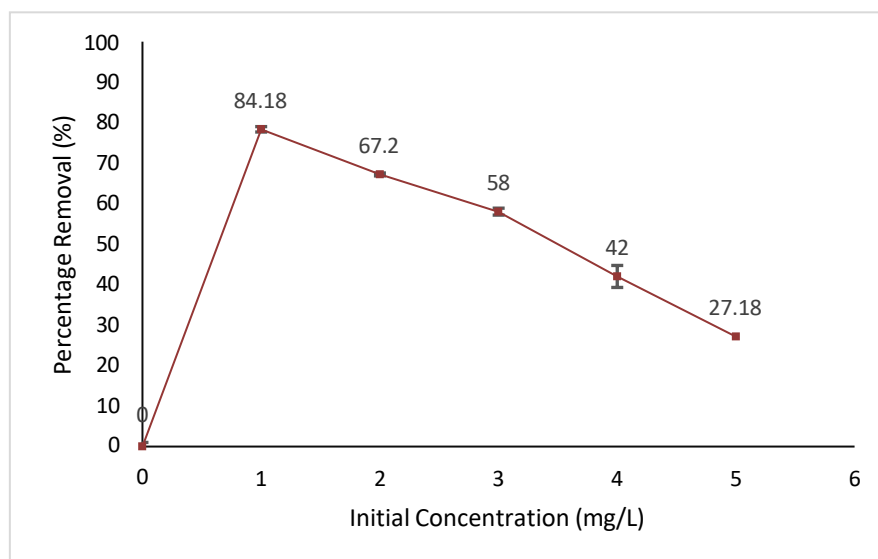


Figure 4.6: The graph of the effect of initial concentration on the adsorption of Pb(II)

The results are in agreement with a previous study by Aboli et al. (2020) whereby the Pb(II) adsorption was higher at low initial concentration and decreases as the initial concentration increase. Similar finding by Krishnamoorthy et al. (2019) where high removal was achieved when the Pb(II) initial concentration was 5 mg/L and significantly reduced with the increase of initial concentration. However, most studies reported that adsorption increases with concentration until it reaches equilibrium. High Pb^{2+} concentration provides the enhancement in the motivating gradient and increases the tendency for effective collision between adsorbent and adsorbate (Alkherraz et al., 2020). Jawad et al. (2017) reported that the adsorption of Pb(II) increased rapidly from 50 – 350 mg/L however, more time is required to achieve higher removal efficiency.

4.3.3 The Effect of pH

The effect of pH in Pb(II) adsorption was studied at different pH values of the Pb(II) solution (pH 2, 4, 6, 8, 10). Figure 4.7 shows that the highest adsorption occurred at pH 8 (95.06%) while the lowest was at pH 2 (12.00%). The pH of a solution affects both chemistries in the solution and the surface charge of the adsorbent (Dada et al., 2017). At

lower pH (<pH8), the abundance of H^+ competes with Pb(II) ions that exist as Pb^{2+} and $Pb(OH)^+$ in the solution. The presence of excess H^+ ions reduces the interaction of adsorbate with predominant Pb^+ species (Krishnamoorthy et al., 2019).

Protons occupy most of the active sites on the adsorbent and fewer Pb^{2+} ions could be adsorbed due to the repulsion with protons on SE-AC (Dada et al., 2017). At higher pH (pH 4 – 7), it is evident that the adsorption of Pb(II) ions increased due to the reducing electrostatic repulsion, low competition between intercalating cations, and the availability of negative ligands for Pb(II) ions binding (Tao et al., 2015; Dada et al., 2017).

The highest removal at pH 8 (95.05%) was possibly due to the simultaneous precipitation of $Pb(OH)^+$ and sorption of Pb^{2+} . At pH 7 – 11, different Pb species predominate the aqueous solutions, such as $Pb(OH)^+$ and $Pb(OH)_2$ that contribute to the decrease in Pb^{2+} concentration (Sajjadi et al., 2019). The decreased adsorption at pH 10 was due to the negatively charged surface of SE-AC. Soluble hydroxylated complexes were also formed at higher pH causing competition between the complexes and Pb(II) ions for the binding sites (Choudhary et al., 2020).

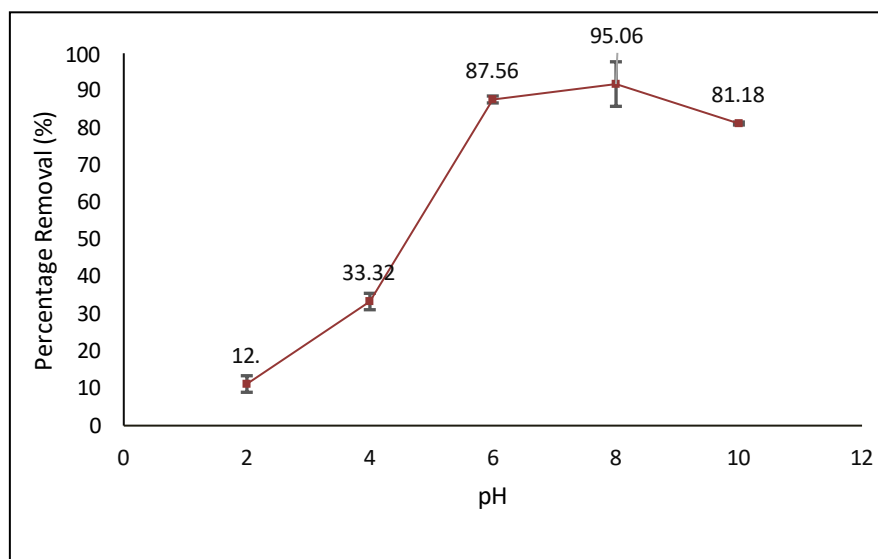


Figure 4.7: The graph of the effect of pH on the adsorption of Pb(II)

Many studies, such as Sajjadi et al. (2019), Gedam & Dongre (2019), and Fauzia et al. (2018), obtained similar finding was reported whereby the maximum adsorption of cationic pollutants (Pb^{2+}) was at pH between 6 – 8 . Further increase in pH resulted to the precipitation of $\text{Pb}(\text{OH})^+$ and $\text{Pb}(\text{OH})_2$.

4.3.4 Isotherm Study on the Adsorption of Pb(II)

Langmuir Isotherm

Langmuir isotherm is based on the hypothesis on the ability of the heavy metal to be adsorbed at fixed sites where each site holds only one metal ion and actively equivalent sites with no interaction between metal ions (Nejadshafiee & Islami, 2019). Table 4.3 showed isotherm parameters that represent specific information. Figure 4.8a shows the graph of (C_e/q_e) against C_e showed good linearity and fitted well with R^2 (0.9308). The Q_{max} value, which represents a maximum monolayer adsorption capacity of SE-AC for Pb(II) removal, was 3.202×10^{-3} mg/g. The constant b was 0.5108 L/mg showed initial concentration with high adsorption energy. High adsorption energy describes an increase in adsorption at a low concentration of adsorbate (Alguacil et al., 2018).

The separation factor, R_L was calculated to classify the adsorption shape of the Langmuir isotherm (Chon, 2021). When the value of $R_L > 1$, the adsorption following Langmuir isotherm is not favourable while the value of $R_L < 1$ indicates that the adsorption is irreversible (Doke & Khan, 2017). The R_L value obtained was 0.999 which indicates that the adsorption onto SE-AC was favourable according to Langmuir adsorption isotherm ($0 < R_L < 1$).

Table 4.3: Parameters and Values of Langmuir and Freundlich Isotherms for Pb(II) adsorption by SE-AC

Isotherm	Parameters	Values
Langmuir	$Q_{\max}$ (mg/g)	3.202×10^{-3}
	b (L/mg)	0.5108
	R^2	0.9308
	R_L	0.999
Freundlich	n	2.5109
	$1/n$	0.3999
	K_f	1.131×10^{-3}
	R^2	0.9084

Freundlich Isotherm

Table 4.3 shows the equilibrium constant of Pb(II) adsorption on SE-AC of Freundlich isotherm. The value of n showed the degree of non-linearity between solution concentration and adsorption (Desta, 2013). When $n=1$, the adsorption is assumed as linear. When $n < 1$ the adsorption is a chemical process while $n > 1$ shows that the adsorption is a physical process (Alguacil et al., 2018). The n value was 2.5109 ($n > 1$), indicating that the adsorption of Pb(II) by SE-AC was a physical process. Physical adsorption occurs as a result of hydrogen bonding, *van der Waals* forces, electrostatic forces, and hydrophobic interactions (Zhang et al., 2016). Results also revealed that the adsorption intensity, $1/n$ was 0.3999, indicating the heterolayer Pb(II) adsorption by SE-AC (dos Santos et al., 2019).

However, the R^2 value was 0.9084 which was slightly lower than R^2 obtained from the Langmuir isotherm. Figure 4.8b shows the Freundlich linear plot of Pb(II) adsorption.

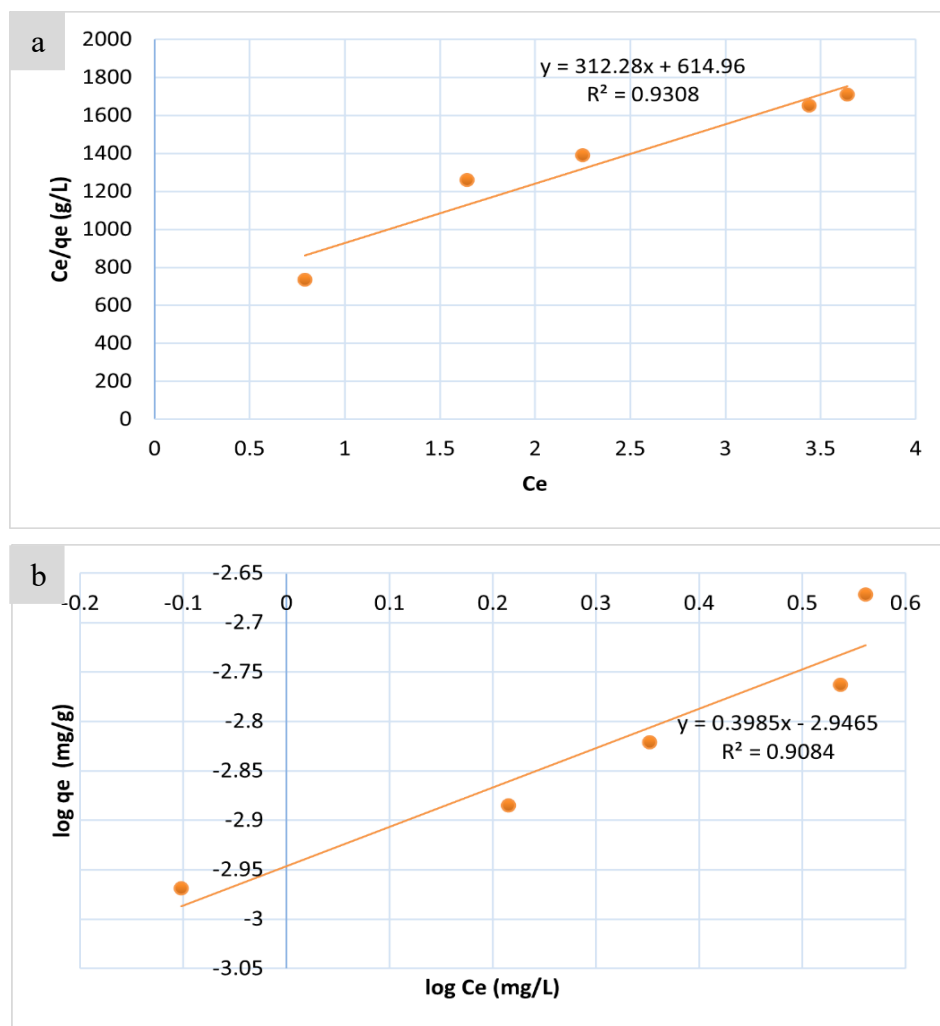


Figure 4.8: Linear plot of (a) Langmuir isotherm and (b) Freundlich isotherm for the adsorption of Pb(II)

Comparison on the removal of Pb(II) by SE-AC with adsorbents from previous studies

The SE-AC has a relatively low maximum adsorption capacity (3.202×10^{-3} mg/g) compared to other adsorbents from previous studies (Table 4.4). One of the plausible reasons for the low adsorption capacity is the low adsorbent dosage used in this study (0.005 g/mL). Based on the table, high adsorption capacity was observed when the adsorbent dosage at

0.02 g/mL as more adsorbent sites area available for the binding with Pb(II) in the solution (Gaya et al., 2015). Another reason for the low adsorption capacity are the adsorption was conducted at a basic pH (pH 8) while most of the other studies were conducted at lower pH (pH 5 – 6). At a higher pH, Pb(II) ions tend to bind with the excess -OH ions available in the solution to produce Pb(OH)₂ precipitate (Aboli et al., 2020). Even so, the removal of Pb(II) in the present study was considerably high (99.78%) despite the low Q_{max} value.

Table 4.4: Comparison of maximum adsorption capacity, Q_{max} obtained from Langmuir isotherm for the removal of Pb(II) by different adsorbent

No.	Activated Carbon Precursor	Q _{max} (mg/g)	Treatments and Adsorptions	Reference
1.	Palm shell	500.00	Concentration: 50 mg/L Adsorption time: 1 h 40 min Adsorbent dosage: 0.02 g/mL Mixing rate: 350 rpm pH: 6.0	(Gaya et al., 2015)
2.	Marine microalgae	83.30	Concentration: 60 mg/L Adsorption time: 1 h Adsorbent dosage: 8.0 x 10 ⁻⁴ g/mL pH: 5.0	(Ibrahim et al., 2016)
3.	Papaya peel	38.31	Concentration: 200 mg/L Adsorption time: 2 h Adsorbent dosage: 0.05 g/mL Mixing rate: 150 rpm pH: 5.0	(Abbaszadeh et al., 2016)
4.	Wood	77.52	Concentration: 200 mg/L Adsorption time: 3 h Adsorbent dosage: 0.02 g/mL pH: 6	(Dada et al., 2017)
5.	Molasses	303.03	Concentration: 50 mg/L Adsorption time: 8 h Adsorbent dosage: 1.5 x 10 ⁻⁴ g/mL Mixing rate: 150 rpm pH: 4	(Legrouiri et al., 2017)

Table 4.4 continued

6.	Winemaking wastes	58.31	Concentration: 0.5 g/L Adsorption time: 3 h Adsorbent dosage: 1.0×10^{-4} g/mL Mixing rate: 750 rpm pH: 4	(Alguacil et al., 2018)
7.	Olive branches	41.32	Concentration: 100 mg/L Adsorption time: 24 h Adsorbent dosage: 1.0×10^{-4} g/mL Mixing rate: 150 rpm pH: 5.0	(Alkherraz et al., 2020)
8.	<i>Citrus limetta</i> leaves	62.89	Concentration: 5 - 100 mg/L Adsorption time: 1 h Adsorbent dosage: 0.02 g/mL Mixing rate: 200 rpm pH: 6.0	(Aboli et al., 2020)
9.	SE-AC	3.202×10^{-1}	Concentration: 1 - 5 mg/L Adsorption time: 30 min Adsorbent dosage: 0.005 g/mL Mixing rate: 260 rpm pH: 8.0	Present study

4.4 Adsorption of MB on SE-AC

Batch adsorption was conducted to investigate the relationship of individual parameters on the adsorption of MB.

4.4.1 The Effect of Initial Concentration

Figure 4.9 shows the result obtained by manipulating the initial concentration of MB (0 - 10 mg/L), in which the highest removal occurred at 2 mg/L (99.96%). The adsorption decreases as the concentration increases. However, the percentage removal increased slightly at 8 mg/L (98.18%) and decreased again at 10 mg/L (90.70%). The concentration of a solution is an important factor in determining the effectiveness of adsorption as it provides a driving force to improve mass transfer resistance from the aqueous phase to the adsorbent (Mahmuda et al., 2021).

As the concentration of MB increases, the adsorption of MB decreases. This is due to the limited availability of active sites for the binding of MB on the adsorbent surface (Tharaneedhar et al., 2017). The aggregation and overlying of MB particles on the adsorption sites caused a decrease in effective adsorption at an increasing concentration (Tharaneedhar et al., 2017; Somsesta et al., 2020).

Since the initial concentration of MB solution provides a driving force for MB attachment, there might be some chances that the adsorption increases at a specific concentration. High concentration enhanced the interaction between adsorbent and adsorbate (Ghosh et al., 2020). Thus, the slight increment that occurred at 8 mg/L was due to higher effective collision that happened during agitation as more particles are colliding to the adsorbent causing more MB to bind on the surface of SE-AC (Pathania et al., 2017).

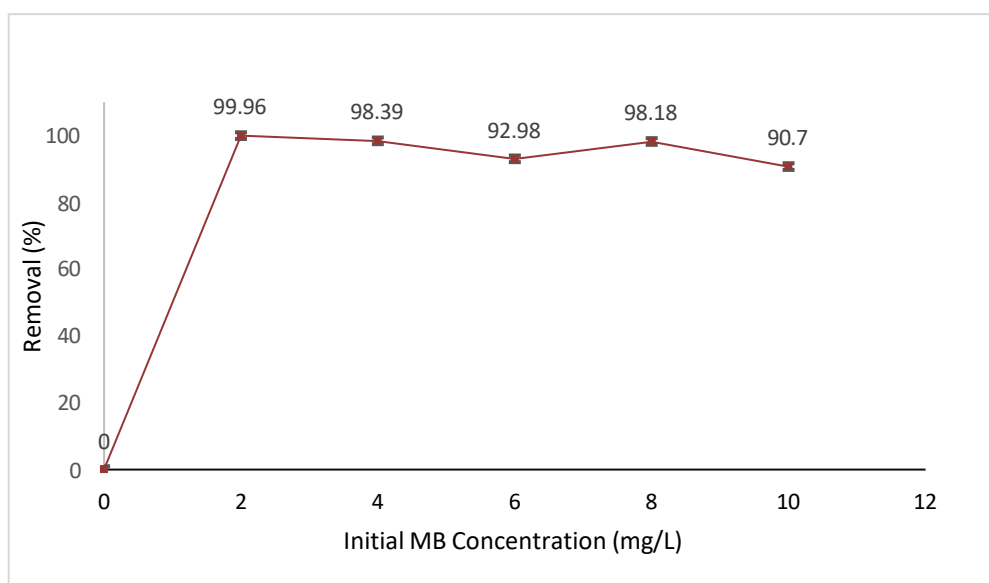


Figure 4.9: The graph of the effect of initial concentration on the adsorption of MB

Most studies are contradictory to the findings in this study. Generally, the adsorption removal efficiency of MB increases with initial concentration until an equilibrium is reached. Aljeboree et al. (2019) mentioned that the adsorption of MB increased rapidly at low concentrations and gradually slowed down as the reaction reaches equilibrium and decreases

at a further increase of concentration. This is due to the limited availability of active binding sites on the SE-AC surface (Fito et al., 2020).

4.4.2 The Effect of Contact Time

Figure 4.10 shows the effect of contact time on the removal of MB by SE-AC. Based on the result obtained at different contact times (0 - 30 min), the removal of MB increased as the contact time increase. The maximum adsorption occurred at 30 min where the removal was almost completed (98.55%). Longer contact time during adsorption on MB increased the tendency of effective collision between MB and SE-AC until it reaches equilibrium (Kausar et al., 2020). At a shorter contact time, less collision occurs compared to a longer time, thus reducing the efficiency of removal of MB (Somsesta et al., 2020). The adsorption of MB onto SE-AC is high considering the short contact time (0 – 30 min).

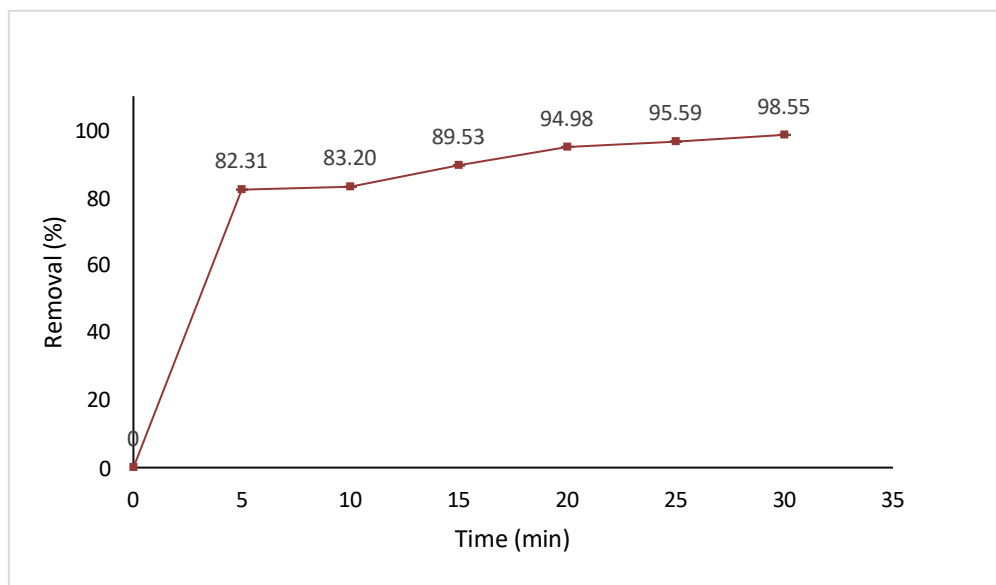


Figure 4.10: The graph of the effect of contact time on the adsorption of MB

Results from this study are comparable with previous studies on adsorption of MB by activated carbon whereby the adsorption increased rapidly at the first 20 – 30 min and

gradually as time increases until it reaches equilibrium. Similar findings by Nibret et al. (2019) stated that the adsorption of MB increased speedily in the first 20 min contact time and decreased after equilibrium at 60 min. Similar finding by Hu et al. (2019) reported that rapid adsorption of MB on the Oolong tea wastes biomass occurred in the first 30 mins and slightly decrease at higher contact time. The decrease in adsorption after equilibrium was due to the increase in desorption rate at a saturated active site (Araujo et al., 2021).

4.4.3 Isotherm Study on the Adsorption of MB

Langmuir Isotherm

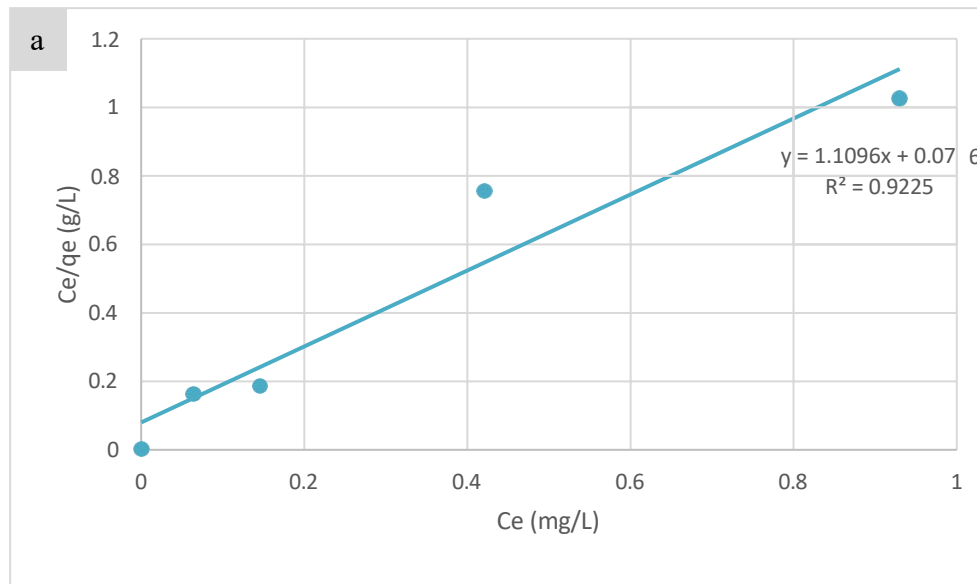
The plotted graph of C_e/q_e vs C_e (Figure 4.11a) showed a high R^2 value (0.9012) which indicates that the Langmuir model is the best model to describe the sorption of MB on SE-AC (Amode et al., 2016). The value of the correlation coefficient indicates the applicability of Langmuir isotherm, assuming a monolayer coverage and uniform activity distribution on the sorbent surface (Pathania et al., 2017). The constant b obtained from the Langmuir linear graph was 13.940 L/mg indicated high adsorption energy that contributed to the fast increment of MB adsorption at various concentrations. The maximum removal of MB was quite high (98.55%) considering the low adsorption capacity, $Q_{\max}$ (0.9012 mg/g). The separation factor, R_L was 0.034 showed that the adsorption favourable with Langmuir isotherm. Table 4.5 shows the equilibrium constant for Langmuir and Freundlich isotherm.

Table 4.5: Parameters and Values of Langmuir and Freundlich for MB adsorption by SE-AC

Isotherm	Parameters	Values
Langmuir	$Q_{\max}$ (mg/g)	0.9012
	b (L/mg)	13.940
	R^2	0.9225
	R_L	0.0340
Freundlich	N	1.5100
	$1/n$	0.7191
	K_f	0.8140
	R^2	0.8527

Freundlich Isotherm

The plotted graph of $\log q_e$ vs $\log C_e$ (Figure 4.11b) where the R^2 was 0.8527. However, the R^2 value was lower than the R^2 of the Langmuir isotherm. This suggested that the model is not suitable to describe the adsorption nature and process of MB onto sludge-activated carbon (Ahmed et al., 2019). The n value was 1.510 indicates that the adsorption of MB onto SE-AC is a physical process ($n > 1$).



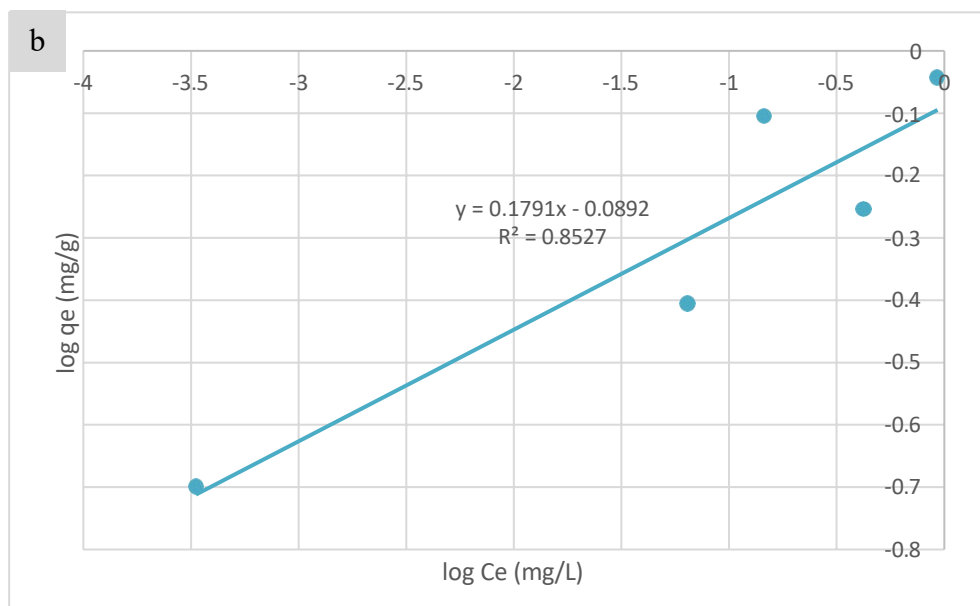


Figure 4.11: The linear plot of (a) Langmuir isotherm and (b) Freundlich isotherm on the adsorption of MB

Comparison on the removal of Pb(II) by SE-AC with adsorbents from previous studies

The maximum adsorption capacity in this study is considerably lower (0.9012 mg/g) than previous studies as tabulated in Table 4.6. One of the reasons for this is that the reaction time was shorter (30 min) thus reducing the contact of SE-AC and MB. Most studies were conducted at a longer reacting time (60 – 180 min) and slightly acidic pH compared to this present study. Longer contact time increase the chances of effective collisions between the adsorbent and adsorbate (Araujo et al., 2021). Nevertheless, the highest was 98.55% which was high considering the low $Q_{\max}$ value.

Table 4.6: Comparison of maximum adsorption capacity, $Q_{\max}$ obtained from Langmuir isotherm for the removal of MB by different adsorbent

No.	Activated Carbon Precursor	$Q_{\max}$ (mg/g)	Adsorption condition	Reference
1.	Palm oil residue	48.84	Concentration: 750 mg/L Adsorption time: 180 min Adsorbent dosage: 0.002 g/mL Mixing rate: 120 rpm pH: 7.0	(Araujo et al., 2021)
2.	Eucalyptus residue	977.00	Concentration: 220 mg/L Adsorption time: 24 h Adsorbent dosage: 1.0×10^{-4} g/mL Mixing rate: 150 rpm pH: 7.0	(Han et al., 2020)
3.	Corn cob	198.15	Concentration: 200 mg/L Adsorption time: 180 min Adsorbent dosage: 0.005 g/mL Mixing rate: 110 rpm pH: 5.6	(Jawad & Mohammed, 2019)
4.	Pumpkin peels	198.15	Concentration: 100 – 400 mg/L Adsorption time: 120 min Adsorbent dosage: 1.6×10^{-4} g/mL pH: 5.45	(Rashid et al., 2019)
5.	SE-AC	6.85	Concentration: 5 – 10 mg/L Adsorption time: 30 min Adsorbent dosage: 0.005 g/mL Mixing rate: 260 rpm pH: 7.0	Present study

4.5 Response Surface Methodology (RSM)

4.5.1 The Utilisation of RSM for Optimization of Pb(II) Adsorption by SE-AC

The RSM approach was employed to find the optimized conditions for adsorption Pb(II) by SE-AC. The experimental factors and response for the adsorption of Pb(II) by SE-AC are shown in Table 4.7. Results showed that maximum adsorption (99.87%) was achieved at Run 4, with Pb(II) initial concentration of 5 mg/L, adsorbent dosage (0.5 g), and contact time (30 min). Meanwhile, Run 6 afforded the lowest adsorption (22.55%), whereby the Pb(II) initial concentration was 5 mg/L, adsorbent dosage (0.1 g), and contact time (90 min). The results indicated that lower adsorption dosage leads to low Pb(II) removal even though the adsorption time is prolonged.

Table 4.7: Experimental Factors and Response for the Adsorption of Pb(II) by SE-AC

Run	Parameters			Response
	Initial Concentration (mg/L)	Adsorbent Dosage (g)	Contact Time (min)	Adsorption (%)*
1	1.00	0.10	30.00	57.58 (57.81)
2	5.00	0.10	30.00	36.89 (38.16)
3	1.00	0.50	30.00	98.55 (98.46)
4	5.00	0.50	30.00	99.87 (101.12)
5	1.00	0.10	90.00	64.58 (64.52)
6	5.00	0.10	90.00	22.55 (23.83)
7	1.00	0.50	90.00	83.96 (83.88)
8	5.00	0.50	90.00	64.56 (65.52)
9	3.00	0.30	60.00	52.73 (58.66)
10	3.00	0.30	60.00	52.09 (58.66)
11	1.00	0.30	60.00	60.88 (58.66)
12	5.00	0.30	60.00	41.55 (36.79)
13	3.00	0.10	60.00	30.05 (27.33)
14	3.00	0.50	60.00	70.54 (68.50)
15	3.00	0.30	30.00	99.65 (97.00)
16	3.00	0.30	90.00	84.65 (82.55)
17	3.00	0.30	60.00	59.43 (58.66)

*Note: Predicted values (%) are shown in brackets

Surface graphs (3D) for percent adsorption of Pb(II) are represented in Figure 4.12(a-c), showing the combined effect of initial concentration, adsorbent dosage, and contact time on the response of percent adsorption of Pb(II) using SE-AC. The combined effect of the 2 variables was varied in the experimental runs while other variables were fixed at the centre (Kafshgari et al., 2017). The pH was adjusted to pH 2 to mimic the real acidic wastewater containing Pb(II) that comes from metal plating and battery manufacturing industries (Alghamdi et al., 2019).

Figure 4.12(a) displays the dependency of Pb(II) adsorption on both Pb(II) initial concentration and adsorbent dosage. It can be seen that the adsorption is greatly affected by the adsorbent dosage. Based on the results shown in Figure 4.12a, the adsorption of Pb(II) increases when the adsorbent dosage increased from 0.1 g (36.89%) to 0.5 g (99.87%) with a fixed initial concentration (5 mg/L) and 30 mins contact time.

Adsorbent dosage plays an important role in determining the effectiveness of adsorption and strongly affects the adsorption capacity (Aboli et al., 2020). As the adsorbent dosage increases, the number of adsorbent particles also increases which led to the availability of metal attachment (Acharya et al., 2009; Aboli et al., 2020). When the adsorbent dosage was low, the availability of active sites on the SE-AC was also low, which make it unavailable for excess Pb(II) particles to bind (Suhada et al., 2013; Dada et al., 2017). Adsorbent dosage (0.5 g) was the optimum dose for optimum adsorption which proves that the equilibrium between Pb(II) and SE-AC was achieved (Tao et al., 2015).

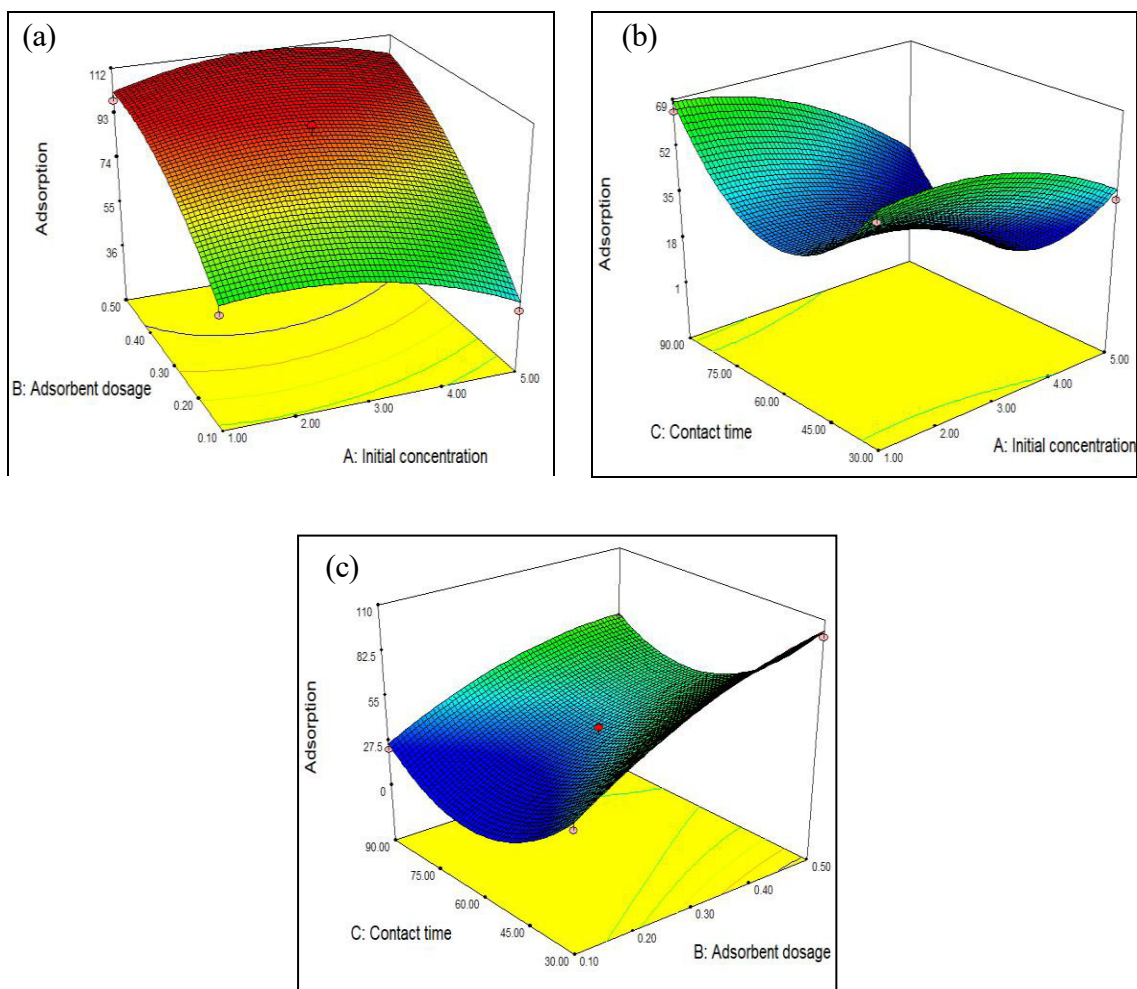


Figure 4.12: The combined effects of (a) adsorbent dosage-initial concentration, (b) contact time-initial concentration, and (c) contact time-adsorbent dosage during Pb(II) adsorption by SE-AC

Figure 4.12(b) shows the combined effect of contact time and initial concentration on the percent adsorption of Pb(II) using SAC at adsorbent dosage 0.1 g. Based on the data shown, the highest Pb(II) adsorption (64.58%) occurred when the initial concentration was 1 mg/L while the lowest occurred at 5 mg/L (22.55%) when the contact time was fixed at 90 min (Table 4.7). Lower adsorption occurred when the contact time was 30 mins (36.89%) compared to when the contact time was 60 min (41.55%) at a constant initial concentration (5 mg/L).

It can be deduced that a longer time is required to remove a higher concentration of Pb(II). The initial concentration of Pb(II) solution promotes a driving force to overcome the mass transfer resistance between the aqueous and solid phases (Hema & Srinivasan, 2010; Aboli et al., 2020). At lower concentrations, Pb(II) could interact with the binding site of adsorbents, increasing the adsorption rate (Anisuzzaman et al., 2015; Dada et al., 2017). However, at higher concentrations, the availability of the active sites is very low before the adsorbent with time. This has caused a limited mass transfer of Pb molecules to the outer surface of the adsorbent (Mehdizadeh et al., 2014; Aboli et al., 2020).

Figure 4.12(c) shows the combined effect of the contact time and adsorbent dosage on the percent removal of Pb(II). At consistent initial Pb(II) concentration (5 mg/L), the highest adsorption (98.55%) was observed at 30 mins contact time and 0.5 g adsorbent dosage. The least adsorption (22.55%) was observed at 90 mins contact time and 0.1 g adsorbent dosage. The adsorption at 60 mins increased at a higher adsorbent dosage (0.5 g). The adsorption at 60 mins was 41.55% when the adsorbent dosage was 0.3 g. The adsorption at 30 mins was higher than at 90 mins contact time with the same adsorbent dosage (0.1 g) and initial concentration (5 mg/L). Generally, the longer the contact time, the higher the adsorption (Pap et al., 2016). Longer contact time increased the chances of effective collisions occurring between adsorbent and adsorbate (Song et al., 2018). However, there is no prominent adsorption increment as the time increases further, indicating that the adsorption reaches equilibrium (Sajjadi et al., 2019). Based on the result obtained, 30 mins contact time was chosen to be the best time for the adsorption of Pb(II).

4.5.1 Development of Regression Model of RSM

The model developed for the response percentage adsorption was depending on the highest order polynomials where additional terms were significant and the model was not aliased according to the sequential sum of squares (Arami-niya et al., 2012). In this study, a quadratic model was developed and the final empirical model in terms of coded factors are represented in the following equation:

$$\text{Percentage adsorption of Pb(II)} = 56.11 - 10.01A + 20.58B - 7.22C + 5.58AB - 5.26AC - 5.32BC - 8.75A^2 - 9.67B^2 + 32.18C^2 \dots\dots\dots (\text{Eq 4.1})$$

where A = initial concentration, B = adsorbent dosage, and C = contact time.

A positive symbol in front of the terms shows there are synergistic effects, while negative symbols mean antagonistic effects (Chowdhury et al., 2012). The coefficients multiplied between two (2) variables with the others showed the correlation between the two (2) variables and quadratic effect. The quality of the model developed was determined by the R^2 , adjusted R^2 , and standard deviation of R^2 values. The R^2 shows the ratio between the sum of squares (SSR) together with the total sum of the squares (SST), and determine how well the model fitted the experimental data points (Kumar et al., 2019). The performance of the model is depicted by observing the plots of predicted against experimental percentage adsorption (Appendix E1) respectively. Table 4.8 shows that the predicted values for percentage adsorption of Pb(II) were close to their experimental values, reflecting the suitability of the models depicted by the equation written. Appendix E1 shows the R^2 data

from the result obtained. The predicted R^2 of 0.9989 was reasonable with the adjusted R^2 of 0.9996.

The model's signal-to-noise ratio of 205.459 indicated an adequate signal of the model. Any ratio above 4 is preferable to show that this design is suitable to navigate the design space as the desired signal power is higher than the undesired background noise. The determination of Coefficient of Variance (CV) value is important as it shows the ratio between the standard error of estimate with the mean value of the observed response (0.73%) as a percentage where it measures the reproducibility of the model (Chowdhury et al., 2012; Zbair et al., 2018).

Appendix E2 shows the outlier t plot for percentage adsorption of Pb(II) by SE-AC. The outlier was observed thoroughly for the successful resolution of designed experiments. Outlier t plots indicated a simple data recording error. It was observed from the data, most of the residuals fall between the interval of ± 3.00 , which showed the data approximation for a fitted model to the response surface was a good and null error (Chowdhury et al., 2012).

The competence and significance of the model were proven by analysis of variances (ANOVA). The ANOVA for response surface quadratic model for Pb (II) adsorption using sago activated carbon is shown in Appendix E3. The model F-value of 4604.84 indicated that the model is significant. Values of Probability > F less than 0.0500 indicates model terms are significant. In this case, A , B , C , AB , AC , BC , A^2 , B^2 and C^2 are significant model terms. Values greater than 0.100 indicate the model terms are not significant. If there are insignificant model terms, model reduction may improve the model.

4.5.2 The Utilisation of RSM for Optimization of MB Adsorption by SE-AC

RSM was also employed to determine the optimized condition for the adsorption of MB by SE-AC. Results in Table 4.8 showed that maximum adsorption of MB (92.69%) was achieved at Run 16, with pH 6, initial concentration (10 mg/L), and contact time (60 min). Meanwhile, Run 2 afforded the lowest adsorption (58.15%), whereby the pH 10, initial concentration (2 mg/L), and contact time (30 min).

Table 4.8: Experimental Factors and Response for the Adsorption of MB

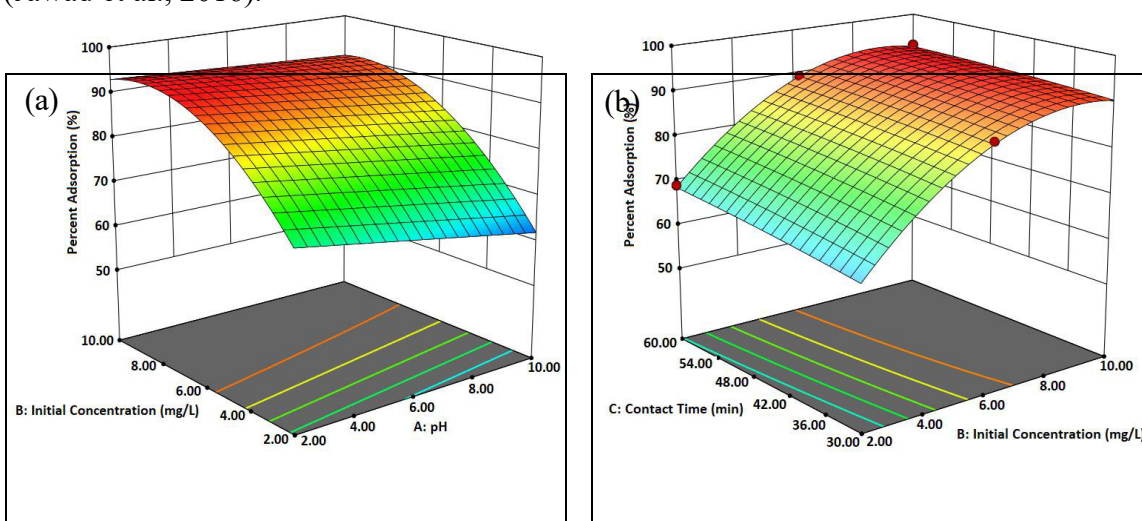
Run	Parameters			Response
	pH	Initial Concentration (mg/L)	Contact Time (min)	Adsorption (%)*
1	2.00	2.00	30.00	69.30 (69.27)
2	10.00	2.00	30.00	58.15 (58.35)
3	2.00	10.00	30.00	92.17 (92.19)
4	10.00	10.00	30.00	88.11 (88.25)
5	2.00	2.00	90.00	72.35 (72.30)
6	10.00	2.00	90.00	67.15 (67.22)
7	2.00	10.00	90.00	89.78 (89.68)
8	10.00	10.00	90.00	91.45 (91.58)
9	6.00	6.00	60.00	88.73 (88.87)
10	6.00	6.00	60.00	89.25 (88.87)
11	6.00	6.00	60.00	88.95 (88.87)
12	6.00	6.00	60.00	88.95 (88.87)
13	2.00	6.00	60.00	91.51 (91.66)
14	10.00	6.00	60.00	87.70 (87.15)
15	6.00	2.00	60.00	69.05 (68.85)
16	6.00	10.00	60.00	92.69 (92.49)
17	6.00	6.00	30.00	86.67 (86.33)
18	6.00	6.00	90.00	89.57 (89.51)
19	6.00	6.00	60.00	88.73 (89.43)
20	6.00	6.00	60.00	88.95 (89.43)

*Note: Predicted values (%) are shown in brackets

Surface graphs (3D) for percent adsorption of MB are represented in Figure 4.13 (a-c), which show the effect of pH, contact time, and initial concentration on the response of percent adsorption of MB. The adsorbent dosage of 0.5 g and condition temperature at 27 °C is maintained. Figure 4.13(a) shows the effect of pH on the adsorption of MB. The pH of the adsorbent surface and solution plays an important role in the adsorption of MB. The

adsorption of MB appears to be higher when the pH is higher. The highest adsorption (92.69%) occurred at pH 6 when the initial concentration was 10 mg/L and 60 min reaction time. Lower adsorption (89.78%) occurred at pH 2 when the initial concentration was 10 mg/L and contact time 90 min.

At lower pH, a high concentration of H^+ ions interfere with the adsorption sites on SE-AC. At higher pH values, more OH^- are available creating a negatively charged adsorbent surface (Cheenmatchaya & Kungwankunakorn, 2013). MB is a cationic dye that requires a negatively charged adsorbent surface for high adsorption capacity (Liu et al., 2020). A similar result was reported by Jawad et al. (2016) where the removal of MB increased as the pH increased from 3 to 6. The surface of the adsorbent adopts a negative surface at high pH which enhanced the uptake of positive dyes via electrostatic attraction (Jawad et al., 2016).



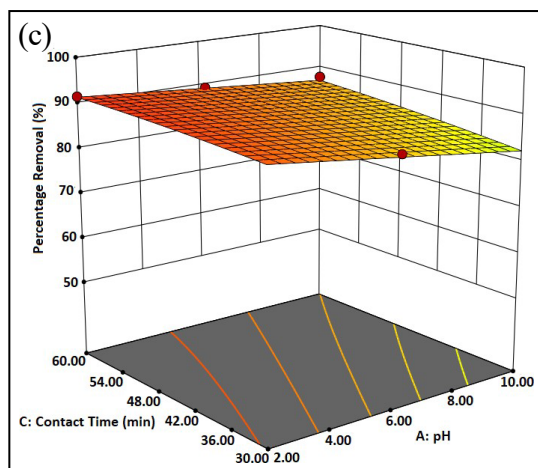


Figure 4.13: The Combined Effect of (a) Initial Concentration-pH, (b) Contact Time-Initial Concentration, and (c) Contact Time-pH during MB adsorption by SE-AC

Figure 4.13 (b) and (c) shows the effect of MB initial concentration and contact time on the removal of MB. Based on the observation in Table 4.8, 88.11% MB removal occurred when the initial concentration was 10 mg/L with a contact time of 30 min. Higher removal was observed (91.45%) when the time was increased to 90 min. An additional time is needed to remove a higher concentration of MB. This is because, longer time is required for MB to penetrated deeper into the SE-AC internal pore structure to be adsorbed (Jawad et al., 2016). Furthermore, a high concentration of MB increased the mass transfer driving force that causes an increment in adsorption (Nasrullah et al., 2018).

The removal of MB decreased to 69.30% when the initial MB concentration was 2 mg/L and 60 min contact time. The removal decreased further to 67.15% when the time increased to 90 min. At low concentrations, almost all MB ions can attach to the adsorbent surface in a shorter time. However, a long contact time causes MB ions to detach the adsorbent. In this situation, desorption occurs after the adsorption reached equilibrium (Nasrullah et al., 2018).

4.5.1 The Development of Regression Model and The Analysis of Variance (ANOVA)

A quadratic model was developed with final empirical coded factors represented in the following equations:

$$\text{Percentage adsorption of MB} = 87.98 - 2.98A + 12.51B + 1.55C + 1.74AB + 0.730AC - 0.6938BC - 0.0155A^2 - 8.75B^2 - 0.3751C^2 \dots\dots\dots(\text{Eq 4.2})$$

where, A = pH, B = initial concentration, and C = Contact time.

Appendix F1 shows the R^2 data from the result obtained. The predicted R^2 of 0.9967 was reasonable with the adjusted R^2 of 0.9984. The adequate precision was 113.048, which was greater than 4 and is suitable to navigate the design space (Ghorbani et al., 2020). The model showed Coefficient of Variance values of 0.49% which proves the reproducibility of the model (Asfaram et al., 2015). Appendix F2 shows the outlier t plot percentage adsorption of MB. The outlier fall between ± 2.00 which showed the data approximation fitted the model and the response was good (Zbair et al., 2018). The model competency was proven by the analysis of variances (ANOVA). The ANOVA for the response surface quadratic model is shown in Appendix F3. The model F-value was 1283.44, indicated that the model is significant.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

This study investigated the production of activated carbon derived from sago effluent activated sludge process (SE-AC) via pyrolysis and chemical modification. Subsequently, the physicochemical properties of afforded SE-AC were determined, and the efficiency of the prepared SE-AC for the removal of Pb(II) and MB from an aqueous solution was explored. Physicochemical characterization indicated that SE-AC is consisted of nano-sized particles (37 - 82 nm) with relatively larger surface area (78.863 m²/g) and pore sizes (1.20 – 6.9 µm) compared to the initial sample (sago effluent biomass, SE-B).

SE-AC was successfully employed as an adsorbent for batch removal of Pb(II) in an aqueous solution. The maximum removal efficiency of Pb(II) by SE-AC was 99.78%. Higher adsorbent dosage and lower initial concentration resulted in higher Pb(II) removal efficiency, with ideal pH 8. The isotherm study showed that the adsorption of Pb(II) by SE-AC worked best under the Langmuir isotherm, with R² value of 0.9308 and maximum adsorption capacity, Q_{max} of 3.202 x 10⁻³ mg/g. Finally, the response surface methodology (RSM) optimization study concluded that the optimum conditions for maximum adsorption of Pb(II) (99.87%) were at pH 2, 30 mins contact time, 0.005 g/L g adsorbent dosage, and 5 mg/L initial concentration.

SE-AC was also successfully applied for the removal of MB in aqueous solution. Results indicated that higher MB initial concentration led to low removal efficiency, whereby the highest was at 2 mg/L (99.96%). When the contact time is 30 mins, the adsorption was also high (98.55%). The isotherm study showed that the adsorption of MB

was best explained with Langmuir isotherm, with R^2 value of 0.9225 with $Q_{\max}$ 0.9012 mg/g. The RSM optimization study concluded that the optimum condition for maximum adsorption of MB by SE-AC (92.69%) was at pH 6, 0.005 g/L adsorbent dosage, 10 mg/L initial concentration, and 60 min contact time.

5.2 Recommendations

This study has shown the potential of ASP as an effective way to treat SE to obtain solid SE-B for employment as the adsorbent for Pb(II) and MB in an aqueous solution. This study also suggested a low cost, practical and sustainable practise for the sago industries' wastes management. However, for further recommendations, a study could be carried out to determine the properties of SE-B in terms of lignocellulosic compositions, extracts, and elemental components. Furthermore, a more concluded analysis should be conducted, such as pore size and particle size distributions, and the total surface area should be carried out in SE-B, SE-BC, and SE-AC.

A different activating agent such as H_3PO_4 and KOH could be used to compare the effectiveness of adsorption on heavy metals and dyes and the enhancement of surface properties of samples. Furthermore, a kinetic study should also be conducted in the adsorption study. Finally, RSM with more optimized parameters is suggested to determine the nature of adsorption and the ideal conditions for the optimum removal of pollutants.

REFERENCES

- AbdulRahman, F., Akter, M., & Abedin, M. Z. (2013). Dyes removal from textile wastewater using orange peels. *International Journal of Scientific & Technology Reasearch*, 2(9), 47–50.
- Abbaszadeh, S., Wan Alwi, S. R., Webb, C., Ghasemi, N., & Muhamad, I. I. (2016). Treatment of lead-contaminated water using activated carbon adsorbent from locally available papaya peel biowaste. *Journal of Cleaner Production*, 118, 210–222.
- Abdelhafez, A. A., & Li, J. (2016). Removal of Pb (II) from aqueous solution by using biochars derived from sugar cane bagasse and orange peel. *Journal of the Taiwan Institute of Chemical Engineers*, 61, 367-375.
- Aboli, E., Jafari, D., & Esmacili, H. (2020). Heavy metal ions (lead , cobalt , and nickel) biosorption from aqueous solution onto activated carbon prepared from *Citrus limetta* leaves. *Carbon Letters*, 30(6), 683-698.
- Acharya, J., Sahu, J. N., Mohanty, C. R., & Meikap, B. C. (2009). Removal of lead (II) from wastewater by activated carbon developed from tamarind wood by zinc chloride activation. *Chemical Engineering Journal*, 149, 249–262.
- Adeni, D. S. A., Aziz, S. A., Bujang, K., & Hassan, M. A. (2010). Bioconversion of sago residue into value added products. *African Journal of Biotechnology*, 9(14), 2016–2021.
- Ahansazan, B., Afrashteh, H., Ahansazan, N., & Ahansazan, Z. (2014). Activated sludge process overview. *International Journal of Environmental Science and Development*, 5(1), 81–85.
- Ahmad, M., Upamali, A., Eun, J., Zhang, M., & Bolan, N. (2014). Biochar as a sorbent for contaminant management in soil and water : A review. *Chemosphere*, 99, 19–33.

- Ahmed, M. J., Okoye, P. U., Hummadi, E. H., & Hameed, B. H. (2019). High-performance porous biochar from the pyrolysis of natural and renewable seaweed (*Gelidiella acerosa*) and its application for the adsorption of methylene blue. *Bioresource Technology*, 278, 159–164.
- Alghamdi, A. A., Al-Odayni, A. B., Saeed, W. S., Al-Kahtani, A., Alharthi, F. A., & Aouak, T. (2019). Efficient adsorption of lead (II) from aqueous phase solutions using polypyrrole-based activated carbon. *Materials*, 12(12), 2020.
- Alguacil, F. J., Alcaraz, L., García-Díaz, I., & López, F. A. (2018). Removal of Pb²⁺ in wastewater *via* adsorption onto an activated carbon produced from winemaking waste. *Metals*, 8(9), 697.
- Aliakbari, Z., Younesi, H., & Asghar, A. (2017). Production and characterization of sewage-sludge based activated carbons under different post-activation conditions. *Waste and Biomass Valorization*, 9(3), 451-463.
- Alipour, A., Zarinabadi, S., Azimi, A., & Mirzaei, M. (2020). Adsorptive removal of Pb (II) ions from aqueous solutions by thiourea- functionalized magnetic ZnO / nanocellulose composite : Optimization by response surface methodology (RSM). *International Journal of Biological Macromolecules*, 151, 124–135.
- Aljeboree, A. M., Hussein, F. H., & Alkaim, A. F. (2019). Removal of textile dye (methylene blue mb) from aqueous solution by activated carbon as a model (corn-cob source waste of plant). *Plant Archives*, 19(2), 906–909.
- Alkherraz, A. M., Ali, A. K., & Elsherif, K. M. (2020). Removal of Pb (II), Zn (II), Cu (II) and Cd (II) from aqueous solutions by adsorption onto olive branches activated carbon: Equilibrium and thermodynamic studies. *Journal of Chemistry International*, 6(1), 11–20.
- Am, N., Kannan, S., & Sb, A. (2019). Identification and characterization of bacteria isolated from sago industrial effluent. *Indian Journal of Experimental Biotechnology*, 57, 427–

- Amin, N., Sabli, N., Izhar, S., & Yoshida, H. (2019). Sago wastes and its application. *Pertanika Science and Technology*, 27(4), 1841-1862.
- Amode, J.O., Santos, J.S., Alam, Z.M., Mirza, A.H., Mei, C. C. (2016). Adsorption of methylene blue from aqueous solution using untreated and treated (*Metroxylon* spp .) waste adsorbent : Equilibrium and kinetics studies. *International Journal of Industrial Chemistry*, 7(3), 333–345.
- Amuda, O. S., Deng, A., Alade, A. O., & Hung, Y. (2008). Conversion of sewage sludge biosolids. *Biosolids Engineering and Management*, (pp. 65-119). Humana Press.
- Anbalagan, A., Schwede, S., Lindberg, C., & Nehrenheim, E. (2016). Influence of hydraulic retention time on indigenous microalgae and activated sludge process. *Water Research*. 91, 277-284.
- Anisuzzaman, S. M., Joseph, C. G., Daud, W. M. A. B. W., Krishnaiah, D., & Yee, H. S. (2015). Preparation and characterization of activated carbon from *Typha orientalis* leaves. *International Journal of Industrial Chemistry*, 6(1), 9–21.
- Arami-niya, A., Mohd, W., Wan, A., Mjalli, F. S., & Abnisa, F. (2012). Production of microporous palm shell based activated carbon for methane adsorption : Modeling and optimization using response surface methodology. *Chemical Engineering Research and Design*, 90(6), 776–784.
- Araujo, L. K. F., Albuquerque, A. A., Ramos, W. C. O., Santos, A. T., Carvalho, S. H. V., Soletti, J. I., Bispo, M. D. (2021). *Elaeis guineensis*-activated carbon for methylene blue removal: Adsorption capacity and optimization using CCD-RSM. *Environment, Development and Sustainability*, 23, 11732-11750.
- Arcibar-Orozco, J. A., Rangel-Mendez, J. R., & Diaz-Flores, P. E. (2015). Simultaneous adsorption of Pb(II)-Cd(II), Pb(II)-phenol, and Cd(II)-phenol by activated carbon cloth

- in aqueous solution. *Water, Air, and Soil Pollution*, 226(1), 1–10.
- Arnich, N., Sirot, V., Rivière, G., Jean, J., Noël, L., & Guérin, T. (2012). Dietary exposure to trace elements and health risk assessment in the 2nd French Total Diet Study. *Food and Chemical Toxicology*, 50, 2432–2449.
- Asfaram, A., Ghaedi, M., Hajati, S., Rezaeinejad, M., Goudarzi, A., & Purkait, M. K. (2015). Rapid removal of auramine-O and methylene blue by ZnS: Cu nanoparticles loaded on activated carbon: A response surface methodology approach. *Journal of the Taiwan Institute of Chemical Engineers*, 53, 80–91.
- Astuti, W., Sulistyaningsih, T., Kusumastuti, E., Yanny, G., & Sari, R. (2019). Thermal conversion of pineapple crown leaf waste to magnetized activated carbon for dye removal. *Bioresource Technology*, 287, 121426.
- Asuquo, E., Martin, A., Nzerem, P., Siperstein, F., & Fan, X. (2017). Adsorption of Cd(II) and Pb(II) ions from aqueous solutions using mesoporous activated carbon adsorbent: Equilibrium, kinetics and characterisation studies. *Journal of Environmental Chemical Engineering*, 5(1), 679–698.
- Atikah, N., Mohmad, H., Yusoff, M., Mohd, N., Ali, M., & Zaharin, A. (2020). Highly efficient removal of diazinon pesticide from aqueous solutions by using coconut shell-modified biochar. *Arabian Journal of Chemistry*, 13(7), 6106–6121.
- Awg-Adeni, D. S., Bujang, K. B., Hassan, M. A., & Abd-Aziz, S. (2013). Recovery of glucose from residual starch of sago hampas for bioethanol production. *BioMed Research International*, 2013.
- Azhar, A., Makihara, D., Naito, H., & Ehara, H. (2018). Photosynthesis of sago palm (*Metroxylon sagu* rottb.) seedling at different air temperatures. *Agriculture*, 8(1), 1–10.
- Baiamonte, G., Crescimanno, G., Parrino, F., & Pasquale, C. De. (2019). Catena Effect of biochar on the physical and structural properties of a sandy soil. *Catena*, 175, 294–303.

- Barkauskas, J., & Dervinyte, M. (2004). An investigation of the functional groups on the surface of activated carbons. *Journal of the Serbian Chemical Society*, 69(5), 363–375.
- Bashir, S. M., Niyi, A. J., Ngozi, I. J., Evaristus, O. E., & Esther, O. (2019). Trace metals content of soil around a municipal solid waste dumpsite in Gombe , Nigeria : Assessing the ecological and human health impact. *Journal of Chemical Health Risk*, 9, 173–190.
- Baysal, M., Bilge, K., Yılmaz, B., Papila, M., & Yürüm, Y. (2018). Preparation of high surface area activated carbon from waste-biomass of sunflower piths: Kinetics and equilibrium studies on the dye removal. *Journal of Environmental Chemical Engineering*, 6(2), 1702-1713.
- Bedin, K. C., Martins, A. C., Cazetta, A. L., Pezoti, O., & Almeida, V. C. (2016). KOH-activated carbon prepared from sucrose spherical carbon: Adsorption equilibrium, kinetic and thermodynamic studies for methylene blue removal. *Chemical Engineering Journal*, 286, 476–484.
- Bhatnagar, A., Hogland, W., Marques, M., & Sillanpää, M. (2013). An overview of the modification methods of activated carbon for its water treatment applications. *Chemical Engineering Journal*, 219, 499–511.
- Bian, Y., Yuan, Q., Zhu, G., Ren, B., Hursthouse, A., & Zhang, P. (2018). Review article recycling of waste sludge : Preparation and Application of sludge-based activated carbon. *Journal of Polymer Science*, 2018.
- Bohli, T., Ouederni, A., Fiol, N., & Villaescusa, I. (2015). Evaluation of an activated carbon from olive stones used as an adsorbent for heavy metal removal from aqueous phases. *Comptes Rendus Chimie*, 18(1), 88–99.
- Boudechiche, N., Fares, M., Ouyahia, S., Yazid, H., & Trari, M. (2019). Comparative study on removal of two basic dyes in aqueous medium by adsorption using activated carbon from *Ziziphus lotus* stones. *Microchemical Journal*, 146, 1010–1018.

- Bouhamed, F., Elouear, Z., Bouzid, J., & Ouddane, B. (2016). Multi-component adsorption of copper, nickel and zinc from aqueous solutions onto activated carbon prepared from date stones. *Environmental Science and Pollution Research*, 23(16), 15801–15806.
- Bridgwater, B. T. (2018). Challenges and opportunities in fast pyrolysis of biomass : Part I. *Johnson Matthew Technology Review*, 1(1), 118–130.
- Bridgwater, A. V. (2012). Review of fast pyrolysis of biomass and product upgrading. *Biomass and Bioenergy*, 38, 68–94.
- Bujang, K. B. (2008). Potentials of bioenergy from the sago industries in Malaysia. *Biotechnology*, 517.
- Bujang, K. B. (2014). Sago : A food and fuel alternative. *Proceedings of BioBorneo*, 141-145.
- Burakov, A. E., Galunin, E. V, Burakova, I. V, Kucheroval, A. E., Agarwal, S., Tkachev, A. G., & Gupta, V. K. (2018). Adsorption of heavy metals on conventional and nanostructured materials for wastewater treatment purposes : A review. *Ecotoxicology and Environmental Safety*, 148, 702–712.
- Cao, X., Sun, S., & Sun, R. (2017). Application of biochar-based catalysts in biomass upgrading: A review. *RSC Advances*, 7(77), 48793–48805.
- Cazetta, A. L., Pezoti, O., Bedin, K. C., Silva, T. L., Junior, A. P., Asefa, T., & Almeida, V. C. (2016). Magnetic activated carbon derived from biomass waste by concurrent synthesis : Efficient adsorbent for toxic dyes. *ACS Sustainable Chemistry and Engineering*, 4(3), 1058-1068.
- Ch'ng, H. Y., Ahmed, O. H., Kassim, S., & Majid, N. M. A. (2014). Recycling of sago (*Metroxylon sagu*) bagasse with chicken manure slurry through co-composting. *Journal of Agricultural Science and Technology*, 16(6), 1441–1454.

- Chaduka, M., Guyo, U., Zinyama, N. P., & Matsinha, L. C. (2019). Modeling and Optimization of lead (II) Adsorption by a novel peanut hull-g-methyl methacrylate biopolymer using response surface methodology (RSM). *Analytical Letters*, 53(8), 1294-1311.
- Charola, S., Yadav, R., Das, P., & Maiti, S. (2018). Fixed-bed adsorption of reactive orange 84 dye onto activated carbon prepared from empty cotton flower agro-waste. *Sustainable Environment Research*, 28(6), 298–308.
- Chaudan., Kishor., Trivedi, U., & Patel, K. C. (2006). Application of response surface methodology for optimization of lactic acid production using date juice. *Journal of Microbial and Technology*, 16, 1410–1415.
- Cheenmatchaya, A., & Kungwankunakorn, S. (2013). Preparation of activated carbon derived from rice husk by simple carbonization and chemical activation for using as gasoline adsorbent. *International Journal of Environmental Science and Development*, 5(2), 171–175.
- Cheng, S., Zhang, L., Ma, A., Xia, H., Peng, J., Li, C., & Shu, J. (2017). Comparison of activated carbon and iron/cerium modified activated carbon to remove methylene blue from wastewater. *Journal of Environmental Sciences*, 65, 92-102.
- Chon, K. (2021). Effects of NaOH activation on adsorptive removal of herbicides by biochars prepared from ground coffee residues. *Energies*, 14, 1-12.
- Choudhary, M., Kumar, R., & Neogi, S. (2020). Activated biochar derived from *Opuntia ficus-indica* for the efficient adsorption of malachite green dye , Cu^{+2} and Ni^{+2} from water. *Journal of Hazardous Materials*, 392, 122441.
- Chowdhury Z.Z., Zain S.M., Khan R.A., Arami-Niya A., K. K. (2012). Process variables optimization for preparation and characterisation of novel adsorbent from lignocellulosic waste. *Bioresources*, 7, 3732–3754.

- Dada, A. O., Adekola, F. A., Odebunmi, E. O., & Shon, Y.-S. (2017). Kinetics, mechanism, isotherm and thermodynamic studies of liquid phase adsorption of Pb^{2+} onto wood activated carbon supported zerovalent iron (WAC-ZVI) nanocomposite. *Cogent Chemistry*, 3(1), 1351653.
- Daifullah, A. A. M., Yakout, S. M., & Elreefy, S. A. (2007). Adsorption of fluoride in aqueous solutions using KMnO_4 -modified activated carbon derived from steam pyrolysis of rice straw. *Journal of Hazardous Materials*, 147(1–2), 633–643.
- Danish, M., & Ahmad, T. (2018). A review on utilization of wood biomass as a sustainable precursor for activated carbon production and application. *Renewable and Sustainable Energy Reviews*, 87, 1–21.
- Danish, M., Ahmad, T., Mehraj, W. N. A. W. N., Waheed, A., Khanday, A., Ziyang, L., & Pin, Z. (2018). Optimization of banana trunk - activated carbon production for methylene blue - contaminated water treatment. *Applied Water Science*, 8(1), 1–11.
- de Souza, T. N. V., de Carvalho, S. M. L., Vieira, M. G. A., da Silva, M. G. C., & Brasil, D. D. S. B. (2018). Adsorption of basic dyes onto activated carbon: experimental and theoretical investigation of chemical reactivity of basic dyes using dft-based descriptors. *Applied Surface Science*, 448, 662-670.
- Demirbas, A. (2009). Agricultural based activated carbons for the removal of dyes from aqueous solutions : A review. *Journal of Hazardous Materials*, 167, 1–9.
- Deng, C., Liaw, S. B., & Wu, H. (2019). Characterization of size-segregated soot from pine wood pyrolysis in a drop tube furnace at 1300 ° C. *Energy & Fuels*, 33(2), 2293–2300.
- Desta, M. B. (2013). Batch sorption experiments : Langmuir and Freundlich isotherm studies for the adsorption of textile metal ions onto teff straw (*Eragrostis tef*) Agricultural Waste. *Journal of Thermodynamics*, 2013, 375830.
- Divyashree, A., & Hegde, G. (2015). Activated carbon nanosphere from biowastes materials

- for supercapacitor applications: A review. *RSC Advances*, 5, 88339–88352.
- Doke, K. M., & Khan, E. M. (2017). Equilibrium, kinetic and diffusion mechanism of Cr(VI) adsorption onto activated carbon derived from wood apple shell. *Arabian Journal of Chemistry*, 10, S252–S260.
- dos Santos, K. J. L., de Souza dos Santos, G. E., de Sá, Í. M. G. L., de Carvalho, S. H. V., Soletti, J. I., Meili, L., Duarte, J. L. S., Bispo, M. D., & Dotto, G. L. (2019). *Syagrus oleracea*–activated carbon prepared by vacuum pyrolysis for methylene blue adsorption. *Environmental Science and Pollution Research*, 26(16), 16470–16481.
- Duarte, S. D. J., & Glaser, B. (2019). Effect of biochar particle size on physical, hydrological and chemical properties of loamy and sandy tropical soils. *Agronomy*, 9, 165.
- Duman, G., Okutucu, C., Ucar, Suat., Stahl, R., & Yanik, J. (2010). The slow and fast pyrolysis of cherry seed. *Bioresource Technology*, 102(2), 1869–1878.
- Ehara, H., Toyoda, Y., & Johnson, D. V. (2018). *Sago palm: Multiple contributions to food security and sustainable livelihoods*. [e-book] Singapore:Springer Nature. 1-330.
- Eum, P. R. B., Ryu, C., Hyun, S., Lee, Y., Jung, J. H., & Park, Y. K. (2012). Characteristics of biochar produced from slow pyrolysis of Geodae-Uksae 1. *Bioresource Technology*, 130, 345–350.
- Fahmy, T. A. (2020). Biomass pyrolysis: Past, present, and future. *Environmental Development and Sustainability*, 22, 17–32.
- Fauzia, S., Aziz, H., Dahlan, D., & Zein, R. (2018). Study of equilibrium, kinetic and thermodynamic for removal of Pb (II) in aqueous solution using Sago bark (*Metroxylon sago*). In *Proceedings of the 3rd International Symposium on Current Progree in Mathematics and Science 2017*, (pp. 020081-1-020081-7).
- Fazal, T., Faisal, A., Mushtaq, A., Hafeez, A., Javed, F., Din, A. A., & Rashid, N. (2019).

- Macroalgae and coal-based biochar as a sustainable bioresource reuse for treatment of textile wastewater. *Biomass Conversion and Biorefinery*, 11, 1491-1506.
- Feiqiang, G., Xiaolei, L., Xiaochen, J., Xingmin, Z., Chenglong, G., & Zhonghao, R. (2018). Characteristics and toxic dye adsorption of magnetic activated carbon prepared from biomass waste by modified one-step synthesis. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 555, 43-54.
- Fergala, A., Alsayed, A., & Eldyasti, A. (2018). Behavior of type II methanotrophic bacteria enriched from activated sludge process while utilizing ammonium as a nitrogen source. *International Biodeterioration and Biodegradation*, 130, 8-16.
- Fito, J., Abrham, S., & Angassa, K. (2020). Adsorption of methylene blue from textile industrial wastewater onto activated carbon of *Parthenium hysterophorus*. *International Journal of Environmental Research*, 14(5), 501-511.
- Flach, M. (2011). Sago Palm: *Metroxylon sagu Rottb.*-Promoting the conversion and used of underutilised and neglected crops, *The International Plant Genetic Resources Institute (IPGRI)*, 18, Rome, Italy.
- Foo, K. Y., & Hameed, B. H. (2012a). Preparation, characterization and evaluation of adsorptive properties of orange peel based activated carbon via microwave induced K_2CO_3 activation. *Bioresource Technology*, 104, 679–686.
- Foo, K. Y., & Hameed, B. H. (2012b). Textural porosity , surface chemistry and adsorptive properties of durian shell derived activated carbon prepared by microwave assisted NaOH activation. *Chemical Engineering Journal*, 187, 53–62.
- Fu, F., & Wang, Q. (2011). Removal of heavy metal ions from wastewaters: A review. *Journal of Environmental Management*, 92(3), 407–418.
- Fu, J., Chen, Z., Wang, M., Liu, S., Zhang, J., Zhang, J., Han, R., & Xu, Q. (2015). Adsorption of methylene blue by a high-efficiency adsorbent (polydopamine

- microspheres): Kinetics, isotherm, thermodynamics and mechanism analysis. *Chemical Engineering Journal*, 259, 53–61.
- Gao, H., Sun, Y., Zhou, J., Xu, R., & Duan, H. (2013). Mussel-inspired synthesis of polydopamine-functionalized graphene hydrogel as reusable adsorbents for water purification. *ACS Applied Materials and Interfaces*, 5, 425-432.
- Gaya, U. I., Otene, E., & Abdullah, A. H. (2015). Adsorption of aqueous Cd (II) and Pb (II) on activated carbon nanopores prepared by chemical activation of doum palm shell. *SpringerPlus*, 4, 458.
- Gebrati, L., El Achaby, M., Chatoui, H., Laqbaqbi, M., El Kharraz, J., & Aziz, F. (2018). Inhibiting effect of textile wastewater on the activity of sludge from the biological treatment process of the activated sludge plant. *Saudi Journal of Biological Sciences*, 26(7), 1753-1757.
- Gecgel, U., Uner, O., Gokara, G., Bayrak, Y. (2016). Adsorption of cationic dyes on activated carbon obtained from waste *Elaeagnus* stone. *Adsorption Science and Technology*, 34(9-10), 512-525.
- Gedam, A. H., & Dongre, R. S. (2019). Comparative study of sponge gourd derived biochar and activated carbon for bio-sorption and desorption of Pb (II) ions. *Materials Today: Proceedings*, 18, 887–900.
- Gharibian, S., Hazrati, H., & Rostamizadeh, M. (2020). Continuous electrooxidation of methylene blue in filter press electrochemical flowcell : CFD simulation and RTD validation. *Chemical Engineering and Processing: Process Intensification*, 150(2020), 107880.
- Ghasemi, M., Bavand, A., Ghasemi, N., Javadian, H., & Fattahi, M. (2014). Microwave-assisted functionalization of *Rosa Canina*-Lfruits activated carbon with tetraethylenepentamine and its adsorption behavior toward Ni (II) in aqueous solution: Kinetic, equilibrium and thermodynamic studies. *Powder Technology*, 274, 362-371.

- Ghorbani, F., Kamari, S., Zamani, S., Akbari, S., & Salehi, M. (2020). Optimization and modeling of aqueous Cr (VI) adsorption onto activated carbon prepared from sugar beet bagasse agricultural waste by application of response surface methodology. *Surfaces and Interfaces*, 18, 100444.
- Ghosh, I., Kar, S., Chatterjee, T., Bar, N., & Das, S. K. (2020). Removal of methylene blue from aqueous solution using *Lathyrus sativus*: Adsorption study MPL and AAN modelling. *Process Safety and Environmental Protection*. 149, 345-361.
- Grau, P. (2018). Criteria for nutrient - balanced operation of activated sludge process. *Water Science and Technology*, 24(3), 251–258.
- Haddad, K., Jeguirim, M., Jellali, S., Thevenin, N., Ruidavets, L., & Limousy, L. (2021). Biochar production from *Cypress* sawdust and olive mill wastewater : Agronomic approach. *Science of the Total Environment*, 752, 141713.
- Haddad, K., Jeguirim, M., Jerbi, B., Chouchene, A., Dutournie, P., Thevenin, N., Ruidavets, L., Jellali, S., & Limousy, L. (2017). Olive mill wastewater: From a pollutant to green fuels, agricultural water source and biofertilizer. *ACS Sustainable Chemistry and Engineering*, 5, 8988-8996.
- Hammado, N., & Utomo, S. (2018). Physicochemical characteristic of sago hampas and sago wastewater in Luwu Regency. *ICENIS 2018*, 7, 4–6.
- Han, Q., Wang, J., Goodman, B. A., Xie, J., & Liu, Z. (2020). High adsorption of methylene blue by activated carbon prepared from phosphoric acid treated eucalyptus residue. *Powder Technology*, 366, 239–248.
- Hassard, F., Biddle, J., Harnett, R., & Stephenson, T. (2018). Microbial extracellular enzyme activity affects performance in a full-scale modified activated sludge process. *Science of the Total Environment*, 625, 1527–1534.
- Hassimi, A. H., Hafiz, R. E., & Muhamad, M. H. (2020). Biofloculant production using

- palm oil mill and sago mill effluent as a fermentation feedstock : Characterization and mechanism of flocculation. *Journal of Environmental Management*, 260(2019), 110046.
- Hedge, G., manaf, A.A.A., Kumar, A., Ali, G.A.M., Chong, K.F., Ngaini, Z., Sharma, K. . (2015). Biowaste sago bark based catalyst free carbon nanospheres : Waste to wealth approach. *ACS Sustainable Chemistry and Engineering*, 3(9), 2247-2253.
- Hema, M., & Srinivasan, K. (2010). Uptake of toxic metals from wastewater by activated carbon from agro industrial by-product. *Indian Journal of Engineering and Material Science*, 17, 373–381.
- Hensley, M., Gu, S., & Ben, E. (2011a). A comprehensive review of biomass resources and biofuels potential in Ghana. *Renewable and Sustainable Energy Reviews*, 15(1), 404–415.
- Hensley, M., Gu, S., & Ben, E. (2011b). Biochar production potential in Ghana — A review, *Renewable and Sustainable Energy Review*, 15, 3539–3551.
- Herrera-gonzález, A. M., Caldera-villalobos, M., & Peláez-cid, A. (2019). Adsorption of textile dyes using an activated carbon and crosslinked polyvinyl phosphonic acid composite. *Journal of Environmental Management*, 234, 237–244.
- Hodgson, E., Lewys-James, A., Rao Ravella, S., Thomas-Jones, S., Perkins, W., & Gallagher, J. (2016). Optimisation of slow-pyrolysis process conditions to maximise char yield and heavy metal adsorption of biochar produced from different feedstocks. *Bioresource Technology*, 214, 574–581.
- Holkar, C. R., Jadhav, A. J., Pinjari, D. V., Mahamuni, N. M., & Pandit, A. B. (2016). A critical review on textile wastewater treatments: Possible approaches. *Journal of Environmental Management*, 182, 351–366.
- Hu, Y., Zhang, Y., Hu, Y., Chu, C., Lin, J., Gao, S., Lin, D., Lu, J., Xiang, P., & Ko, T.

- (2019). Application of wasted oolong tea as a biosorbent for the adsorption of methylene blue. *Journal of Chemistry*, 2019, 4980965.
- Husin, H., Ibrahim, M. F., Kamal Bahrin, E., & Abd-Aziz, S. (2019). Simultaneous saccharification and fermentation of sago hampas into biobutanol by *Clostridium acetobutylicum* ATCC 824. *Energy Science and Engineering*, 7(1), 66–75.
- Ibrahim, W. M., Hassan, A. F., & Azab, Y. A. (2016). Biosorption of toxic heavy metals from aqueous solution by *Ulva lactuca* activated carbon. *Egyptian Journal of Basic and Applied Sciences*, 3(3), 241-249.
- Illi, N., Puad, M., Madihah, U., Rashidi, M., & Suhaida, A. (2020). Microwave-assisted acid pretreatment of sago wastewater for fermentable sugar production. *Material Science and Engineering*, 778, 012051.
- Inyang, M., & Dickenson, E. (2015). The potential role of biochar in the removal of organic and microbial contaminants from potable and reuse water : A review. *Chemosphere*, 134, 232–240.
- Islam, A., Sabar, S., Benhouria, A., Khanday, W. A., Asif, M., & Hameed, B. H. (2017). Nanoporous activated carbon prepared from karanj (*Pongamia pinnata*) fruit hulls for methylene blue adsorption. *Journal of the Taiwan Institute of Chemical Engineers*, 74, 96-104.
- Islam, M. A., Ahmed, M. J., Khanday, W. A., Asif, M., & Hameed, B. H. (2017). Mesoporous activated coconut shell-derived hydrochar prepared via hydrothermal carbonization-NaOH activation for methylene blue adsorption. *Journal of Environmental Management*, 203, 237–244.
- Jain, S. N., & Gogate, P. R. (2017). Adsorptive removal of Acid Violet 17 dye from wastewater using biosorbent obtained from NaOH and H₂SO₄ activation of fallen leaves of *Ficus racemosa*. *Journal of Molecular Liquids*, 243, 132-143.

- Jankovi, B., Dodevski, V., Radovi, I., & Pijovi, M. (2019). Physico-chemical characterization of carbonized apricot kernel shell as precursor for activated carbon preparation in clean technology utilization. *Journal of Cleaner Production*, 236, 117614.
- Jawad, A. H., & Mohammed, S. A. (2019). Carbonization of corn (*Zea mays*) cob agricultural residue by one-step activation with sulfuric acid for methylene blue adsorption. *Desalination and Water Treatment*, 118, 342-351.
- Jawad, A. H., Rashid, R. A., Azlan, M., Ishak, M., & Wilson, L. D. (2016). Adsorption of methylene blue onto activated carbon developed from biomass waste by H₂SO₄ activation : kinetic , equilibrium and thermodynamic studies. *Desalination and Water Treatment*, 57(52), 25194-25206.
- Jawad, A. H., Sabar, S., Ishak, M. A. M., Wilson, L. D., Ahmad Norrahma, S. S., Talari, M. K., & Farhan, A. M. (2017). Microwave-assisted preparation of mesoporous-activated carbon from coconut (*Cocos nucifera*) leaf by H₃PO₄ activation for methylene blue adsorption. *Chemical Engineering Communications*, 204(10), 1143–1156.
- Jean, K. D. L. S., Emanuella, G. D. S. D. S., Mychel, I. G. L., Ide, A. H., DUarte, J. L. S., Carvalho, S. H. V., Soletti, J. I., & Meili, L. (2019). *Wodyetia bifurcata* biochar for methylene blue removal from aqueous matrix. *Bioresource Technology*, 293, 122093.
- Jindo, K., Mizumoto, H., Sawada, Y., Sanchez-Monedero, M. A., & Sonoki, T. (2014). Physical and chemical characterization of biochars derived from different agricultural residues. *Biogeosciences*, 11(23), 6613–6621.
- Kafshgari, L. A., Ghorbani, M., Azizi, A., Agarwal, S., & Gupta, V. K. (2017). Modeling and optimization of Direct Red 16 adsorption from aqueous solutions using nanocomposite of MnFe₂O₄/MWCNTs: RSM-CCRD model. *Journal of Molecular Liquids*, 233, 370-377.
- Kamaraj, M., Srinivasan, N. R., Assefa, G., & Adugna, A. T. (2020). Facile development of

- sunlit ZnO nanoparticles-activated carbon hybrid from pernicious weed as an operative nano-adsorbent for removal of methylene blue and chromium from aqueous solution : Extended application in tannery. *Environmental Technology & Innovation*, 17, 100540.
- Kamari, S., Ghorbani, F., & Mohammad, A. (2019). Adsorptive removal of lead from aqueous solutions by amine – functionalized magMCM-41 as a low – cost nanocomposite prepared from rice husk : Modeling and optimization by response surface methodology. *Sustainable Chemistry and Pharmacy*, 13(2018), 100153.
- Kan, C. C., Ibe, A. H., Rivera, K. K. P., Arazo, R. O., & de Luna, M. D. G. (2017). Hexavalent chromium removal from aqueous solution by adsorbents synthesized from groundwater treatment residuals. *Sustainable Environment Research*, 27(4), 163–171.
- Kannan, T. S., Ahmed, A. S., & Ani, F. N. (2013). Energy efficient microwave irradiation of sago bark waste (SBW) for bioethanol production. *Advance Materials Research*, 701, 249-253.
- Karim, A. A., Tie, A. P. L., Manan, D. M. A., & Zaidul, I. S. M. (2008). Starch from the sago (*Metroxylon sagu*) palm tree - Properties, prospects, and challenges as a new industrial source for food and other uses. *Comprehensive Reviews in Food Science and Food Safety*, 7(3), 215–228.
- Karnib, M., Kabbani, A., Holail, H., & Olama, Z. (2014). Heavy metals removal using activated carbon, silica and silica activated carbon composite. *Energy Procedia*, 50, 113–120.
- Karri, R. R., & Sahu, J. N. (2018). Process optimization and adsorption modeling using activated carbon derived from palm oil kernel shell for Zn (II) disposal from the aqueous environment using differential evolution embedded neural network. *Journal of Molecular Liquids*, 265, 592-602.
- Karthika, C., Vennilamani, N., Pattabhi, S., & Sekar, M. (2010). Utilization of sago waste as an adsorption. *International Journal of Engineering Science and Technology*, 2(6),

1867-1879.

- Kausar, R.A., Buhani & Suharso (2020). Methylene blue adsorption isotherm on *Spirulina* sp . microalgae biomass coated by silica-magnetite. *Materials Science and Engineering*, 857, 012019.
- Kavand, M., Eslami, P., & Razeh, L. (2020). The adsorption of cadmium and lead ions from the synthesis wastewater with the activated carbon : Optimization of the single and binary systems. *Journal of Water Process Engineering*, 34(2019), 101151.
- Khan, M. B., Nisar, H., Ng, C. A., & Lo, P. K. (2017). Generalized classification modeling of activated sludge process based on microscopic image analysis. *Environmental Technology*, 39(1), 24-34.
- Kharrazi, S. M., Mirghaffari, N., Dastgerdi, M. M., & Soleimani, M. (2020). A novel post-modification of powdered activated carbon prepared from lignocellulosic waste through thermal tension treatment to enhance the porosity and heavy metals adsorption. *Powder Technology*, 366, 358–368.
- Krishnamoorthy, R., Govindan, B., Banat, F., Sagadevan, V., Purushothaman, M., & Show, P. L. (2019). Date pits activated carbon for divalent lead ions removal. *Journal of Bioscience and Bioengineering*, 128(1), 88–97.
- Kristensen, G. H., Elberg, P. J., & Henze, M. (2018). Characterization of functional microorganism groups and substrate in activated sludge and wastewater by aur , nur and our. *Water Science and Tehnology*, 25(6), 43–57.
- Kuang, Y., Zhang, X., & Zhou, S. (2020). Adsorption of methylene blue in water onto activated carbon by surfactant modification. *Water*, 12(2), 587.
- Kumar, A., Saw, R. K., & Mandal, A. (2019). RSM optimization of oil-in-water microemulsion stabilized by synthesized zwitterionic surfactant and its properties evaluation for application in enhanced oil recovery. *Chemical Engineering Research*

and Design, 147, 399–411.

Kumar, S., Pattnaik, D., Bhuyan, S. K., & Mishra, S. C. (2018). Influence of temperature on pyrolysis of biomass. In *IOP Conference Series: Materials Science and Engineering*, 338(1), p. 012061. IOP Publishing.

Kumar, V., & Chopra, A. K. (2017). Phytoremediation potential of water caltrop (*Trapa natans* L .) using municipal wastewater of activated sludge process based municipal wastewater treatment plant. *Environmental Technology*, 39(1), 12-23.

Lafi, Ridha., Montasser, I., Hafiane, A. (2018). Adsorption of congo red dye from aqueous solutions by prepared activated carbon with oxygen-containing functional groups and its regeneration. *Adsorption Science and Technology*, 39(1-2), 160-181.

Lam, S. S., Liew, R. K., Wong, Y. M., Azwar, E., Jusoh, A., & Wahi, R. (2017). Activated carbon for catalyst support from microwave pyrolysis of orange peel. *Waste and Biomass Valorization*, 8(6), 2109–2119.

Lam, S. S., Su, M. H., Nam, W. L., Thoo, D. S., Min, C., Liew, R. K., Yek, P. N. Y., Ma, N. L., & Vo, N. (2018). Microwave pyrolysis with steam activation in producing activated carbon for removal of herbicides in agricultural surface water. *Industrial and Engineering Chemistry Research*, 158(2), 695-703.

Legrouri, K., Khouya, E., Hannache, H., El Hartti, M., Ezzine, M., & Naslain, R. (2017). Activated carbon from molasses efficiency for Cr(Vi), Pb(II) and Cu(II) adsorption: A mechanistic study. *Chemistry International*, 3(3), 301–310.

Lehmann, J., & Joseph, S. (2009). A future for biochar in vermont. *Biochar for Environmental Management: Science and Technology*, 416.

Lesaoana, M., Mlaba, R. P. V, Mtunzi, F. M., Klink, M. J., Ejidike, P., & Pakade, V. E. (2019). Influence of inorganic acid modification on Cr (VI) adsorption performance and the physicochemical properties of activated carbon. *South African Journal of*

Chemical Engineering, 28(2018), 8–18.

- Li, L., Wu, M., Song, C., Liu, L., Gong, W., Ding, Y., & Yao, J. (2021). Efficient removal of cationic dyes via activated carbon with ultrahigh specific surface derived from vinasse wastes. *Bioresource Technology*, 322(2020), 124540.
- Li, S., Han, K., Si, P., Li, J., & Lu, C. (2018). High – performance activated carbons prepared by KOH activation of gulfweed for supercapacitors, 13, 1728–1743.
- Li, X. F., Zuo, Y., Zhang, Y., Fu, Y., & Guo, Q. X. (2013). In situ preparation of K₂CO₃ supported kraft lignin activated carbon as solid base catalyst for biodiesel production. *Fuel*, 113, 435–442.
- Li, X., Yin, C., Kær, S. K., & Condra, T. (2020). A detailed pyrolysis model for a thermally large biomass particle. *Fuel*, 278, 118397.
- Li, Z., Hanafy, H., Zhang, L., Sellaoui, L., Schadeck, M., Oliveira, M. L. S., Seliem, M. K., Dotto, G. L., & Bonilla-petriciolet, A. (2020). Adsorption of congo red and methylene blue dyes on an ashitaba waste and a walnut shell-based activated carbon from aqueous solutions : Experiments , characterization and physical interpretations. *Chemical Engineering Journal*, 388, 124263.
- Liew, R. K., Chai, C., Nai, P., Yek, Y., Phang, X. Y., Chong, M. Y., Wai, L. N., Si, M. H., Lam, W. H. Ma. N. L., & Lam, S. S. (2018). Innovative production of highly porous carbon for industrial effluent remediation via microwave vacuum pyrolysis plus sodium-potassium hydroxide mixture activation. *Journal of Cleaner Production*, 208, 1436-1445.
- Lim, L. W. K., Chung, H. H., & Hussain, H. (2020a). Complete chloroplast genome sequencing of sago palm (*Metroxylon sagu* Rottb.): Molecular structures, comparative analysis and evolutionary significance. *Gene Reports*, 19, 100662.
- Lim, L. W. K., Chung, H. H., & Hussain, H. (2020b). Organellar genome copy number

- variations and integrity across different organs, growth stages, phenotypes and main localities of sago palm (*Metroxylon sagu* Rottboll) in Sarawak, Malaysia. *Gene Reports*, 21, 100808.
- Liu, J., Yang, X., Liu, H., Cheng, W., & Bao, Y. (2020). Modification of calcium-rich biochar by loading Si / Mn binary oxide after NaOH activation and its adsorption mechanisms for removal of Cu (II) from aqueous solution. *Colloids and Surfaces A*, 601(March), 124960.
- Liu, L., He, D., Pan, F., Huang, R., & Lin, H. (2020). Comparative study on treatment of methylene blue dye wastewater by different internal electrolysis systems and COD removal kinetics , thermodynamics and mechanism. *Chemosphere*, 238, 124671.
- Liu, X., Xu, X., Dong, X., & Park, J. (2020). Competitive adsorption of heavy metal ions from aqueous solutions onto activated carbon and agricultural waste materials. *Polish Journal of Environmental Studies*, 29(1), 749–761.
- Liu, Xin, He, C., Yu, X., Bai, Y., Ye, L., Wang, B., & Zhang, L. (2018). Net-like porous activated carbon materials from shrimp shell by solution-processed carbonization and H₃PO₄ activation for methylene blue adsorption. *Powder Technology*, 326, 181–189.
- Liu, Z., Sun, Y., Xu, X., Meng, X., Qu, J., & Wang, Z. (2020). Preparation , characterization and application of activated carbon from corn cob by KOH activation for removal of Hg (II) from aqueous solution. *Bioresource Technology*, 306(600), 123154.
- Mahmuda, K. N., Wen, T. H., & Zakaria, Z. A. (2021). Activated carbon and biochar from pineapple waste biomass for the removal of methylene blue. *Environmental and Toxicology Management*, 1, 30-36.
- Makshut, N. A., Ngaini, Z., Wahi, R., Hussain, H., Mahmut, N. I., & Bahrin, N. Q. (2020). Nano-sized adsorbent from pyrolysed sago activated sludge for removal of pb(II) from aqueous solution. *Pertanika Journal of Science and Technology*, 28(3), 893–916.

- Mary, G. S., Niveditha, P. S. S., & Mary, G. S. (2016). Production , characterization and evaluation of biochar from pod (*Pisum sativum*) , leaf (*Brassica oleracea*) and peel (*Citrus sinensis*) wastes. *International Journal of Recycling of Organic Waste in Agriculture*, 5(1), 43–53.
- McBeath, A. V., Smernik, R. J., Krull, E. S., & Lehmann, J. (2014). The influence of feedstock and production temperature on biochar carbon chemistry: A solid-state ¹³C NMR study. *Biomass and Bioenergy*, 60, 121–129.
- Mehdizadeh, S., Sadjadi, S., Ahmadi, S. J., & Outokesh, M. (2014). Removal of heavy metals from aqueous solution using platinum nanoparticles / Zeolite-4A. *Journal of Environmental Health Science and Engineering*, 14(7), 1–7.
- Menya, E. (2019). Optimization of pyrolysis conditions for char production from rice husks and its characterization as a precursor for production of activated carbon. *Biomass Conversion and Biorefinery*, 10(1), 57-72.
- Metaragakusuma, AP., Katsuya, O., Bai, H. (2016). An overview of the traditional use of sago for sago-based food industry in Indonesia. *ICaO Conference Proceedings*, 3(2015), 119–124.
- Min, H., Yoo, S., Choi, Y., Park, S., & Kan, E. (2018). Adsorption isotherm , kinetic modeling and mechanism of tetracycline on *Pinus taeda*-derived activated biochar. *Bioresource Technology*, 259, 24–31.
- Mofu, S. S., & Abbas, B. (2018). Development of sago palm research and agroindustry in University of Papua. In *Proceedings of the 12th International Sago Symposium*, (15-16 September 2015), 1–12.
- Mohan, D., Pittman, C. U., & Steele, P. H. (2006). Pyrolysis of wood/biomass for bio-oil: A critical review. *Energy and Fuels*, 20(3), 848–889.
- Mohan, D., Shi, J., Nicholas, D. D., Pittman, C. U., Steele, P. H., & Cooper, J. E. (2008).

Fungicidal values of bio-oils and their lignin-rich fractions obtained from wood / bark fast pyrolysis, *71*, 456–465.

Mookiah, V. P., Kasimani, R., Pandian, S., & Asokan, T. (2020). Study on the effects of initial pH , temperature and agitation speed on lipid production by *Yarrowia lipolytica* and *Chlorella vulgaris* using sago wastewater as a substrate. *Tierarztlieche Praxis*, *40*, 692–703.

Mor, S. (2007). Adsorption of chromium from aqueous solution by activated alumina and activated charcoal. *Bioresource Technology*, *98*, 954–957.

Mor, S., Chhavi, M. K., & Sushil, K. K. (2017). Assessment of hydrothermally modified fly ash for the treatment of methylene blue dye in the textile industry wastewater. *Environment, Development and Sustainability*, *20*(2), 625-639.

Mukome, F. N. D., Zhang, X., Silva, L. C. R., Six, J., & Parikh, S. J. (2013). Use of Chemical and Physical Characteristics To Investigate Trends in Biochar Feedstocks.

Nallapan, M., Hari, M., & Suhaila, N. (2019). Enhanced methylene blue decolourization using banana peel agricultural waste by *Rhodococcus strain* Ucc 0003 through response surface methodology. *Biocatalysis and Agricultural Biotechnology*, *23*, 101486.

Nasrullah, A., Bhat, A. H., Naeem, A., Isa, M. H., & Danish, M. (2018). High surface area mesoporous activated carbon-alginate beads for efficient removal of methylene blue. *International Journal of Biological Macromolecules*, *107*, 1792–1799.

Neethu, A., & Murugan, A. (2018). Bioconversion of sago effluent and oil cakes for bio-butanol production using environmental isolates. *Biofuels*, *12*(1), 35-42.

Nejadshafiee, V., & Islami, M. R. (2019). Adsorption capacity of heavy metal ions using sultone-modified magnetic activated carbon as a bio-adsorbent. *Materials Science and Engineering C*, *101*(2018), 42–52.

- Ngaini, Z., Noh, F., & Wahi, R. (2014). Esterified sago waste for engine oil removal in aqueous environment. *Environmental Technology*, 35(22), 2761-2766.
- Ngaini, Z. (2017). Facile sorbent from esterified cellulosic sago waste for engine oil removal in marine environment. *International Journal of Environmental Science and Technology*, 15(2), 341-348.
- Nibret, G., Ahmad, S., Govardhana, D., Ahmad, I., & Mohamed, S. (2019). Removal of methylene blue dye from textile wastewater using water *Hyacinth* activated carbon as adsorbent : Synthesis , characterization and kinetic studies, (2013), 1959–1969.
- Norouzi, S., Heidari, M., Alipour, V., Rahmanian, O., Fazlzadeh, M., Mohammadi-moghadam, F., Nourmoradi, H., Goudarzi, B., Dindarloo, K. (2018). Preparation, characterization and Cr(VI) adsorption evaluation of NaOH-activated carbon produced from Date Press Cake: An agro-industrial waste. *Bioresource Technology*, 258. 48-56.
- Orhon, D. (2014). Evolution of the activated sludge process : The first 50 years. *Society of Chemical Industry*, 90(4), 608-640.
- Pan, M. (2020). Biochar adsorption of antibiotics and its implications to remediation of contaminated soil. *Water, Air and Soil Pollution*, 231(221).
- Pap, S., Radonić, J., Trifunović, S., Adamović, D., Mihajlović, I., Vojinović Miloradov, M., & Turk Sekulić, M. (2016). Evaluation of the adsorption potential of eco-friendly activated carbon prepared from cherry kernels for the removal of Pb^{2+} , Cd^{2+} and Ni^{2+} from aqueous wastes. *Journal of Environmental Management*, 184, 297–306.
- Park, J., Hung, I., Gan, Z., Rojas, O. J., Lim, K. H., & Park, S. (2013). Activated carbon from biochar: Influence of its physicochemical properties on the sorption characteristics of phenanthrene. *Bioresource Technology*, 149, 383–389.
- Pathania, D., Sharma, S., & Singh, P. (2017). Removal of methylene blue by adsorption onto activated carbon developed from *Ficus carica* bast. *Arabian Journal of Chemistry*, 10,

S1445–S1451.

- Pedicini, R., Maisano, S., Chiodo, V., Conte, G., Policichio, A., & Agostino, R. G. (2020). *Posidonia Oceanica* and Wood chips activated carbon as interesting materials for hydrogen storage. *International Journal of Hydrogen Energy*, 45(27), 14038-14047.
- Phang, S., Miah, M., & Yeoh, B. (2000). Spirulina cultivation in digested sago starch factory wastewater. *Journal of Applied Phycology*, 12, 395–400.
- Priya, M. V., & Ramesh, K. (2021). Optimization of process parameters of the growth of *Chlorella vulgaris* for the production of biodiesel feedstock (microbial lipid) using sago wastewater as substrate. *Biomass Conversion and Biorefinery*, Doi 10.1007/s13399-021-01401-1
- Rafiq, M. K., Bachmann, R. T., Rafiq, M. T., Shang, Z., Joseph, S., & Long, R. (2016). Influence of pyrolysis temperature on physico-chemical properties of corn stover (*Zea mays* L .) biochar and feasibility for carbon capture and energy balance. *PLOS*, 11(6), e0156894.
- Rajan, Y., Ngaini, Z., & Wahi, R. (2018). Novel adsorbent from sago-grafted silica for removal of methylene blue. *International Journal of Environmental Science and Technology*, 16(8), 4531-4542.
- Rambli, J., Ghani, W.A.A.K., Salleh, M.A.M., & Khezri, R. (2019). com Evaluation of Biochar from Sago (*Metroxylon* Spp.) as a Potential Solid Fuel, 14(1), 1928–1940.
- Ramesh, F., Nagarajan, K., & Gracelyn Portia, A. (2013). Comparative account of untreated and treated sago effluent analysis by investigating different physical and chemical parameters. *International Journal of Pure Applied Science and Technology*, 17(2), 17–20.
- Rashid, J., Tehreem, F., Rehman, A., & Kumar, R. (2019). Synthesis using natural functionalization of activated carbon from pumpkin peels for decolourization of

- aqueous methylene blue. *Science of the Total Environment*, 671, 369–376.
- Reddy, P. M. K., Verma, P., & Subrahmanyam, C. (2016). Bio-waste derived adsorbent material for methylene blue adsorption. *Journal of the Taiwan Institute of Chemical Engineers*, 58, 500–508.
- Ricordel, S., Taha, S., Cisse, I., & Dorange, G. (2001). Heavy metals removal by adsorption onto peanut husks carbon : characterization , kinetic study and modeling. *Separation and Purification Technology*, 24, 389–401.
- Riyadi, I., Efendi, D., Purwoko, B. S., & Santoso, D. (2018). Somatic embryogenesis of sago palm (*Metroxylon sagu* Rottb.) through suspension culture technique. *IOP Conference Series: Earth and Environmental Science*, 183(1).
- Roy, P., & Dias, G. (2017). Prospects for pyrolysis technologies in the bioenergy sector: A review. *Renewable and Sustainable Energy Reviews*, 77, 59–69.
- Saad, M. J., Sajab, M. S., & Misran, S. (2020). Comparative adsorption mechanism of rice straw activated carbon activated with NaOH and KOH. *Sains Malaysiana*, 49(11), 2721-2734.
- Sajjadi, S., Meknati, A., Claudio, E., Dotto, G. L., Mendoza-castillo, D. I., Anastopoulos, I., Alakhras, F., Unuabonah, E. I., Singh, P., & Hosseini-bandegharaei, A. (2019). A novel route for preparation of chemically activated carbon from pistachio wood for highly efficient Pb (II) sorption. *Journal of Environmental Management*, 236(2018), 34–44.
- Saleh, T. A. (2016). Nanocomposite of carbon nanotubes/silica nanoparticles and their use for adsorption of Pb(II): from surface properties to sorption mechanism. *Desalination and Water Treatment*, 57(23), 10730–10744.
- Salem, S., Nouri, B., & Ghadiri, M. (2020). Photoactivity of magnesium aluminate under solar irradiation for treatment of wastewater contaminated by methylene blue : Effect of self-combustion factors on spinel characteristics. *Solar Energy Materials and Solar*

Cells, 218, 110773.

Sangon, S., Hunt, A. J., Attard, T. M., & Mengchang, P. (2017). Valorisation of waste rice straw for the production of highly effective carbon based adsorbents for dyes removal. *Journal of Cleaner Production*, 172, 1128-1139.

Saygılı, H., & Güzel, F. (2016). High surface area mesoporous activated carbon from tomato processing solid waste by zinc chloride activation: Process optimization, characterization and dyes adsorption. *Journal of Cleaner Production*, 113, 995-1004.

Senghie, H., Bolhassan, M. H., & Awg-adeni, D. S. (2021). The effects of sago (*Metroxylon sagu*) bark and frond waste as substrates on the growth and yield of grey oyster mushrooms. *Pertanika Journal of Tropical and Agricultural Science*, 44(2), 307–316.

Senthilkumar, P., Kirtika, K. (2015). Removal of Nickel (II) from aqueous solutions by ceralite IR 120 cationic exchange resins. *Journal of Engineering Science and Technology*, 4(4), 351-363.

Sepehri, A., & Sarrafzadeh, M. H. (2019). Activity enhancement of ammonia - oxidizing bacteria and nitrite - oxidizing bacteria in activated sludge process : metabolite reduction and CO₂ mitigation intensification process. *Applied Water Science*, 9(5), 1–12.

Shchegolkova, N. M., Krasnov, G. S., & Belova, A. A. (2016). Microbial Community Structure of Activated Sludge in Treatment Plants with Different Wastewater Compositions. *Frontiers in Microbiology*, 7, 1–15.

Shokry, H., Elkady, M., & Hamad, H. (2019). Nano activated carbon from industrial mine coal as adsorbents for removal of dye from simulated textile wastewater : operational parameters and mechanism study. *Integrative Medicine Research*, 8(5), 4477–4488.

Shu, J., Cheng, S., Xia, H., Zhang, L., Peng, J., Li, C., & Zhang, S. (2017). Copper loaded on activated carbon as an efficient adsorbent for removal of methylene blue. *RSC*

Advances, 7(24), 14395–14405.

- Silva, T. L., Cazette, A. L., Souza, P. S. C., Zhang, T., Asefa, T., Almeida, V. C. (2017). Mesoporous activated carbon fibers synthesized from denim fabric waste: Efficient adsorbents for removal of textile dye from aqueous solutions. *Journal of Cleaner Production*, 171, 482-490.
- Singh, J., & Mishra, V. (2020). Synthesis and characterization of activated carbon derived from *Tectona grandis* sawdust via green route. *Environmental Progress and Sustainable Energy*, 40(2), e13525.
- Singh, V., Chakravarthi, M. H., & Srivastava, V. C. (2020). Chemically modified biochar derived from effluent treatment plant sludge of a distillery for the removal of an emerging pollutant , tetracycline , from aqueous solution. *Biomass Conversion and Biorefinery*, 1-12.
- Singhal, R. S., Kennedy, J. F., Gopalakrishnan, S. M., Kaczmarek, A., Knill, C. J., & Faridatul, P. (2008). Industrial production , processing , and utilization of sago palm-derived products. *Carbohydrates Polymers*, 72, 1–20.
- Siruru, H., Syafii, W., Wistara, I. N. J., & Pari, G. (2019). Characteristics of *Metroxylon rumphii* (pith and bark waste) from Seram Island, Maluku, Indonesia. *Biodiversitas*, 20(12), 3517–3526.
- Siruru, H., Syafii, W., Wistara, I. N. J., Pari, G., & Budiman, I. (2020). Properties of sago waste charcoal using hydrothermal and pyrolysis carbonization. *Biomass Conversion and Biorefinery*, 4, 1-12.
- Sizmur, T., Quilliam, R., Puga, A. P., Moreno-jiménez, E., Beesley, L., & Gomez-eyles, J. L. (2016). Application of biochar for soil remediation. *Agrilcultural and Environmental Applications of Biochar: Advances and Barriers*, 98104, 295–324.
- Slade, A. H., Thorn, G. J. S., & Dennis, M. A. (2011). The relationship between BOD : N

- ratio and wastewater treatability in a nitrogen-fixing wastewater treatment system. *Water Science and Technology*, 63(4), 627-632.
- Sohi, S. P., Krull, E., Lopez-Capel, E., & Bol, R. (2010). A review of biochar and its use and function in soil. *Advances in Agronomy*, 105(1), 47-82.
- Somsesta, N., Sricharoenchaikul, V., & Aht-Ong, D. (2020). Adsorption removal of methylene blue onto activated carbon/cellulose biocomposite films: Equilibrium and kinetic studies. *Materials Chemistry and Physics*, 240(2019), 122221.
- Song, M., Wei, Y., Cai, S., Yu, L., Zhong, Z., & Jin, B. (2018). Study on adsorption properties and mechanism of Pb^{2+} with different carbon based adsorbents. *Science of the Total Environment*, 618, 1416–1422.
- Suhada, N., Tahiruddin, M., Zubaidah, S., & Rahman, A. (2013). Adsorption of lead in aqueous solution by a mixture of activated charcoal and peanut shell. *World Journal of Science and Technology Research*, 1(5), 102–109.
- Srinivasan, N., Thangavelu, K., Sekar, A., & Uthandi, S. (2020). Characteristics of sago processing wastewater effluents released from different sago factories in Salem and Namakkal District of Tamil Nadu, India. *Madras Agricultural Journal*, 107(special),1.
- Syamsu, K., Fahma, F., & Pari, G. (2020). Characterization of ball-milled sago pith waste-based activated carbon treated with KOH and $KMnO_4$ as activating agent. *Material Science and Engineering*, 935(2020).
- Tan, Z., Zou, J., Zhang, L., & Huang, Q. (2018). Morphology , pore size distribution , and nutrient characteristics in biochars under different pyrolysis temperatures and atmospheres. *Journal of Material Cycles and Waste Management*, 20(2), 1036–1049.
- Tao, H. C., Zhang, H. R., Li, J. B., & Ding, W. Y. (2015). Biomass based activated carbon obtained from sludge and sugarcane bagasse for removing lead ion from wastewater. *Bioresource Technology*, 192, 611–617.

- Tao, X., Wu, Y., & Cha, L. (2019). Shaddock peels-based activated carbon as cost-saving adsorbents for efficient removal of Cr (VI) and methyl orange. *Environmental Science and Pollution Research*, 26, 19828–19842.
- Teimouri, Z., Salem, A., & Salem, S. (2019). Regeneration of wastewater contaminated by cationic dye by nanoporous activated carbon produced from agriculture waste shells. *Environmental Science and Pollution Research*, 26, 7718–7729.
- Thangavelu, K., Sundararaju, P., Srinivasan, N., & Muniraj, I. (2020). Biotechnology for biofuels simultaneous lipid production for biodiesel feedstock and decontamination of sago processing wastewater using *Candida tropicalis*. *Biotechnology for Biofuels*, 1–14.
- Tharaneedhar, V., Senthil Kumar, P., Saravanan, A., Ravikumar, C., & Jaikumar, V. (2017). Prediction and interpretation of adsorption parameters for the sequestration of methylene blue dye from aqueous solution using microwave assisted corncob activated carbon. *Sustainable Materials and Technologies*, 11, 1–11.
- Thomas, T., Loganath, R., & Meenambal, T. (2019). Removal of copper from bioleachate of e-waste using orange activated carbon (OAC) and comparison with commercial activated carbon (CAC). *Springer Singapore*, 373-383.
- Tomczyk, A. (2020). Biochar physicochemical properties : Pyrolysis temperature and feedstock kind effects. *Reviews in Environmental Science and Bio/Technology*, 19(1), 191–215.
- Tong, D. S., Wu, C. W., Adebajo, M. O., Jin, G. C., Yu, W. H., Ji, S. F., & Zhou, C. H. (2018). Adsorption of methylene blue from aqueous solution onto porous cellulose-derived carbon/montmorillonite nanocomposites. *Applied Clay Science*, 161(2017), 256–264.
- Tripathi, M., Sahu, J. N., & Ganesan, P. (2016). Effect of process parameters on production of biochar from biomass waste through pyrolysis: A review. *Renewable and*

Sustainable Energy Reviews, 55, 467–481.

Ukanwa, K. S., Patchigolla, K., Sakrabani, R., Anthony, E., & Mandavgane, S. (2019). A Review of Chemicals to Produce Activated Carbon from Agricultural Waste Biomass. *Sustainability*, 11, 1–35.

Ulhiza, T. A., Illi, N., Puad, M., & Azmi, A. S. (2018). Optimization of culture conditions for biohydrogen production from sago wastewater by *Enterobacter aerogenes* using response surface methodology. *International Journal of Hydrogen Energy*, 1–11.

Üner, O., Geçgel, Ü., & Bayrak, Y. (2016). Adsorption of methylene blue by an efficient activated carbon prepared from *Citrullus lanatus* Rind : Kinetic , isotherm , thermodynamic , and mechanism analysis. *Water, Air, & Soil Pollution*, 227(7), 1-15.

Utsev, J. T., Iwar, R. T., & Ifyale, K. J. (2020). Adsorption of methylene blue from aqueous solution onto *Delonix regia* pod activated carbon : Batch equilibrium isotherm , kinetic and thermodynamic studies, 11(7), 1058–1078.

Wahi, R., Abdullah, L. C., Mobarekeh, M. N., Ngaini, Z., Choong, T., & Yaw, S. (2016). Utilization of esterified sago bark fibre waste for removal of oil from palm oil mill effluent. *Journal of Environmental Engineering*, 5(1), 170-177.

Wahi, R., Aimi, S., Su, Y., Lam, S., Mona, S., Aziz, A., & Ngaini, Z. (2019). Esterification of microwave induced pyrolytic oil from sago bark waste. *Waste and Biomass Valorization*, 11(7), 3653-3663.

Wahi, R., Bidin, E. R., Muntaj, N., Asif, M., Athirah, N., Hamizat, N., & Ngaini, Z. (2019). Nutrient availability in sago bark and empty fruit bunch composts for the growth of water spinach and green mustard. *Environmental Science and Pollution Research*, 26, 22246–22253.

Wahi, R., Ngaini, Z., & Jok, V. U. (2009). Removal of mercury , lead and copper from aqueous solution by activated carbon of palm oil empty fruit bunch. *World Applied*

Science Journal (Special Issue for Environment), 5, 84–91.

Wahi, R., & Senghie, H. (2011). The use of microwave derived activated carbon for removal of heavy metal in aqueous solution. *Journal of Science and Technology*, 97–108.

Wang, G., Dai, Y., Yang, H., Xiong, Q., Wang, K., Zhou, J., Li, Y., & Wang, S. (2020). A review of recent advances in biomass pyrolysis. *Energy and Fuels*, 35(12), 15557-15578

Wang, T., Liu, J., Zhang, Y., Zhang, H., Chen, W., & Pan, W. (2017). Use of a non-thermal plasma technique to increase the number of chlorine active sites on biochar for improved mercury removal. *Chemical Engineering Journal*, 331, 536-544.

Wilson, F., Tremain, P., & Moghtaderi, B. (2018). Characterization of biochars derived from pyrolysis of biomass and calcium oxide mixtures. *Energy and Fuels*, 32(4), 4167-4177.

Xie, S., Wen, Z., Zhan, H., & Jin, M. (2018). An experimental study on the adsorption and desorption of Cu (II) in silty clay. *Geofluids*, 2018, 1-12.

Xu, Z., Sun, Z., Zhou, Y., Chen, W., Zhang, T., Huang, Y., & Zhang, D. (2019). Insights into the pyrolysis behavior and adsorption properties of activated carbon from waste cotton textiles by FeCl₃-activation. *Colloids and Surfaces A*, 582, 123934.

Yadav, V., Ali, J., Asce, A. M., & Garg, M. C. (2021). Biosorption of methylene blue dye from textile-industry wastewater onto sugarcane bagasse : Response surface modeling , isotherms , kinetic and thermodynamic modeling. *Journal of Hazard, Toxic and Radioactive Waste*, 25(1), 1–9.

Yanti, N. A., Ahmad, S. W., & Muhiddin, N. H. (2019). Characteristics of biocellulose from sago liquid waste with different ammonium sulfate concentration. *International Journal of Ecophysiology*, 01(01), 56–64.

Zahir, F., Rizwi, S. J., Haq, S. K., & Khan, R. H. (2005). Low dose mercury toxicity and

- human health. *Environmental Technology and Pharmacology*, 20, 351–360.
- Zbair, M., Anfar, Z., Khallok, H., & Ahsaine, H. A. (2018). Adsorption kinetics and surface modeling of aqueous methylene blue onto activated carbonaceous wood sawdust. *Fullerenes, Nanotubes and Carbon Nanostructures*, 26(7), 433–442.
- Zeng, S., Choi, Y., & Kan, E. (2021). Iron-activated bermudagrass-derived biochar for adsorption of aqueous sulfamethoxazole : Effects of iron impregnation ratio on biochar properties , adsorption , and regeneration. *Science of the Total Environment*, 750, 141691.
- Zhang, J., Liu, J., & Liu, R. (2015). Effects of pyrolysis temperature and heating time on biochar obtained from the pyrolysis of straw and lignosulfonate. *Bioresource Technology*, 176, 288–291.
- Zhang, K., Wang, Z., Fang, W., He, Y., Hsu, E., Li, Q., & Gul-e-rana, J. (2018). High-temperature pyrolysis behavior of a bituminous coal in a drop tube furnace and further characterization of the resultant char. *Journal of Analytical and Applied Pyrolysis*, 137, 163-170.
- Zhang, P., Chen, Y., Wang, W., Shen, Y., & Guo, J. (2016). Surface plasmon resonance for water pollutant detection and water process analysis. *Trends in Analytical Chemistry*, 85, 153–165.
- Zhang, Y., & Duan, X. (2020). Chemical precipitation of heavy metals from wastewater by using the synthetical magnecium hydroxy carbonate. *Water, Science and Technology*, 81(6), 1130-1136.
- Zhou, R., Zhang, M., Li, J., & Zhao, W. (2020). Optimization of preparation conditions for biochar derived from water hyacinth by using response surface methodology (RSM) and its application in Pb²⁺ removal. *Journal of Environmental Chemical Engineering*, 8(5), 104198.

Zielinska, A., Oleszczuk, P., Charmas, B., Skubiszewska-Zieba, J., & Pasięcsna-Patkowska, S. (2015). Effect of Sewage Sludge Properties on the Biochar Characteristic. *Journal of Analytical and Applied Pyrolysis*, 112, 201 – 213.

APPENDICES

Appendix A: Activated Sludge Process

Appendix A1: Total Carbon Content

Weight of empty crucible = 46.694 g

Weight of crucible + SE = 146.694 g

Weight of crucible + dried SE = 146.814 g

Weight of Carbon = 146.814 g – 146.694 g = 0.12 g

In every 100 mL of SE contain 0.12 g of Carbon.

Appendix A2: C:N:P Optimisation

Optimisation C₁₀₀: N₁₀: P₁

C: 100 mL = 0.12 g C

N: 10 mL = 0.012 g N

P: 1 mL = 0.0012 g P

Since 10 mL of SE require 0.012 g of N, 25 L tank of SE required 24 g of N.

Since 1 mL of P is required in every 100 mL of SE, thus 20 L required 200 mL of P.

Optimisation C₁₀₀: N₁₀: P₃,

Since 3 mL of P is required in every 100 mL of SE, thus 20 L required 600 mL of P.

Appendix B: Proximate Analysis

Appendix B1: Proximate Analysis of SE-B

Moisture Content			
Analysis	Crucible 1	Crucible 2	Crucible 3
Initial weight of crucible + sample (g)	53.168	57.635	56.306
Final weight of crucible + sample (g)	52.953	57.395	56.031
Weight loss (g)	0.215	0.240	0.275
Moisture content (%)	0.404	0.416	0.488
Average (%)	0.452		
Standard deviation	0.051		
Volatile Matter Content			
Analysis	Crucible 1	Crucible 2	Crucible 3
Initial weight of crucible + sample (g)	52.953	57.395	56.031
Final weight of crucible + sample (g)	53.902	57.354	55.996
Weight loss (g)	0.051	0.041	0.035
Volatile matter content (%)	0.096	0.071	0.062
Average (%)	0.080		
Standard deviation	0.018		
Ash Content			
Analysis	Crucible 1	Crucible 2	Crucible 3
Initial weight of crucible + sample (g)	53.165	57.633	56.311
Final weight of crucible + sample (g)	52.718	57.180	55.863
Weight loss (g)	0.447	0.453	0.448
Ash content (%)	99.159	99.214	99.204
Average (%)	99.190		
Standard deviation	0.026		
Fixed Carbon Content			
Analysis	Crucible 1	Crucible 2	Crucible 3
Fixed carbon content (%)	0.224	0.229	0.218
Average (%)	0.224		
Standard deviation	0.005		

Appendix A2: Proximate Analysis of SE-BC

Moisture Content			
Analysis	Crucible 1	Crucible 2	Crucible 3
Initial weight of crucible + sample (g)	53.243	54.122	51.299
Final weight of crucible + sample (g)	53.243	54.122	51.299
Weight loss (g)	0.000	0.000	0.000
Moisture content (%)	0.000	0.000	0.000
Average (%)	0.000		
Standard deviation	0.000		
Volatile Matter Content			
Analysis	Crucible 1	Crucible 2	Crucible 3
Initial weight of crucible + sample (g)	53.243	54.122	51.299
Final weight of crucible + sample (g)	53.243	54.121	51.299
Weight loss (g)	0.000	0.001	0.000
Volatile matter content (%)	0.000	0.002	0.000
Average (%)	0.001		
Standard deviation	0.001		
Ash Content			
Analysis	Crucible 1	Crucible 2	Crucible 3
Initial weight of crucible + sample (g)	57.583	53.292	56.101
Final weight of crucible + sample (g)	57.535	53.259	56.046
Weight loss (g)	0.048	0.033	0.055
Ash content (%)	99.917	99.938	99.902
Average (%)	99.919		
Standard deviation	0.018		
Fixed Carbon Content			
Analysis	Crucible 1	Crucible 2	Crucible 3
Fixed carbon content (%)	0.080	0.070	0.090
Average (%)	0.080		
Standard deviation	0.010		

Appendix C: Batch Adsorption of Pb(II)

Appendix C1: The Effect of Adsorbent Dosage

Dose (g)	MB (mg/L) n=1	MB (mg/L) n=2	MB (mg/L) n=3	Ce	SD	Removal (%)	qe	Ce/qe	Log Ce	Log qe
0.1	3.661	3.692	3.537	3.630	0.082	27.40	1.370	2.650	0.560	0.137
0.2	2.997	3.002	3.001	3.000	0.003	40.00	1.00	3.00	0.477	0.000
0.3	2.243	2.351	2.462	2.340	0.110	53.20	0.887	2.638	0.369	-0.052
0.4	1.007	1.101	1.420	1.176	0.216	76.48	0.956	1.230	0.070	-0.019
0.5	0.011	0.012	0.010	0.011	0.001	99.78	0.998	0.011	-1.959	-0.001

Appendix C2: The Effect of Initial Concentration

Conc (mg/L)	MB (mg/L) n=1	MB (mg/L) n=2	MB (mg/L) n=3	Ce	SD	Removal (%)	qe	Ce/qe	Log Ce	Log qe
1	0.221	0.235	0.224	0.227	0.015	78.33	0.773	0.294	-0.644	-0.112
2	0.671	0.652	0.645	0.656	0.013	67.18	1.344	0.488	-0.183	0.128
3	1.256	1.272	1.252	1.260	0.011	58.00	1.740	0.724	0.100	0.241
4	2.259	2.261	2.320	2.280	0.035	43.00	1.720	1.326	0.358	0.236
5	3.771	3.652	3.500	3.641	0.136	27.18	1.359	2.679	0.561	0.133

Appendix C3: The Effect of pH

pH	MB (mg/L) n=1	MB (mg/L) n=2	MB (mg/L) n=3	Ce	SD	Removal (%)	qe	Ce/qe	Log Ce	Log qe
2	4.423	4.340	4.560	4.441	0.111	11.20	0.559	7.945	0.647	-0.253
4	3.231	3.450	3.321	3.334	0.110	33.32	1.666	2.001	0.523	0.222
6	0.568	0.652	0.646	0.622	0.047	87.56	4.378	0.142	-0.206	0.641
8	0.251	0.259	0.231	0.247	0.014	95.06	4.753	0.052	-0.607	0.677
10	0.920	0.951	0.952	0.941	0.018	81.18	4.059	0.232	-0.026	0.608

Appendix D: Batch Adsorption of MB

Appendix D1: The Effect of Initial Concentration

Conc (mg/L)	MB (mg/L) n=1	MB (mg/L) n=2	MB (mg/L) n=3	Ce	SD	Removal (%)	qe	Ce/qe	Log Ce	Log qe
2	0.0004	0.0004	0.0002	3.3×10^{-4}	0.0058	99.98	0.1999	0.0017	-3.4771	-0.6990
4	0.0488	0.0528	0.0912	0.0643	0.5853	98.39	0.3936	0.1633	-1.1920	-0.4049
6	0.3960	0.3840	0.4830	0.4210	0.9005	92.98	0.5579	0.7546	-0.3757	-0.2534
8	0.1944	0.1192	0.1232	0.1456	0.5289	98.18	0.7854	0.1854	-0.8368	-0.1049
10	0.9080	0.9650	0.9170	0.9300	0.3064	90.70	0.9070	1.0253	-0.0315	-0.0424

Appendix D2: The Effect of Contact Time

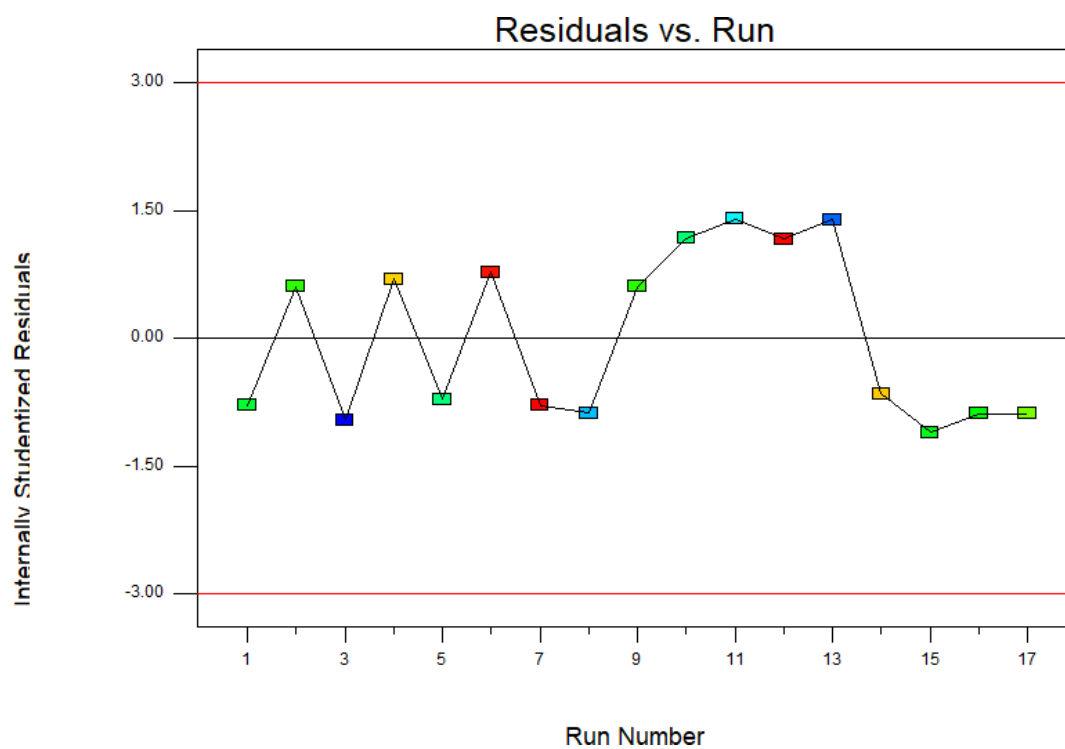
Time (min)	MB (mg/L) n=1	MB (mg/L) n=2	MB (mg/L) n=3	Ce	SD	Removal (%)	qe	Ce/qe	Log Ce	Log qe
5	1.8782	1.8642	1.8645	1.8690	1.771	82.31	0.8131	2.2986	0.2716	-0.0899
10	1.6845	1.6753	1.6805	1.6801	0.0451	83.20	0.8319	2.0194	0.2253	-0.0799
15	1.0339	1.0551	1.0530	1.0473	0.1159	89.53	0.8953	1.1698	0.0201	-0.0481
20	0.4883	0.5088	0.6019	0.4997	0.1185	94.98	0.9500	0.5259	-0.3013	-0.0223
25	0.3850	0.3249	0.3123	0.3407	0.3894	96.59	0.9659	0.3538	-0.4676	-0.0151
30	0.1942	0.1190	0.1212	0.1448	0.4246	98.55	0.9855	0.1469	-0.8392	-0.0063s

Appendix E: RSM of Pb(II)

Appendix E1: Statistical parameters obtained from the Analysis of Variance (ANOVA) for the Model of Pb(II) adsorption

Variables	Adsorption (%)
Std. Dev.	0.460
Mean	63.540
Coefficient of Variation (%)	0.730
PRESS	9.910
R-squared	0.999
Adj R-squared	0.999
Pred R-squared	0.999
Adeq Precision	206.796

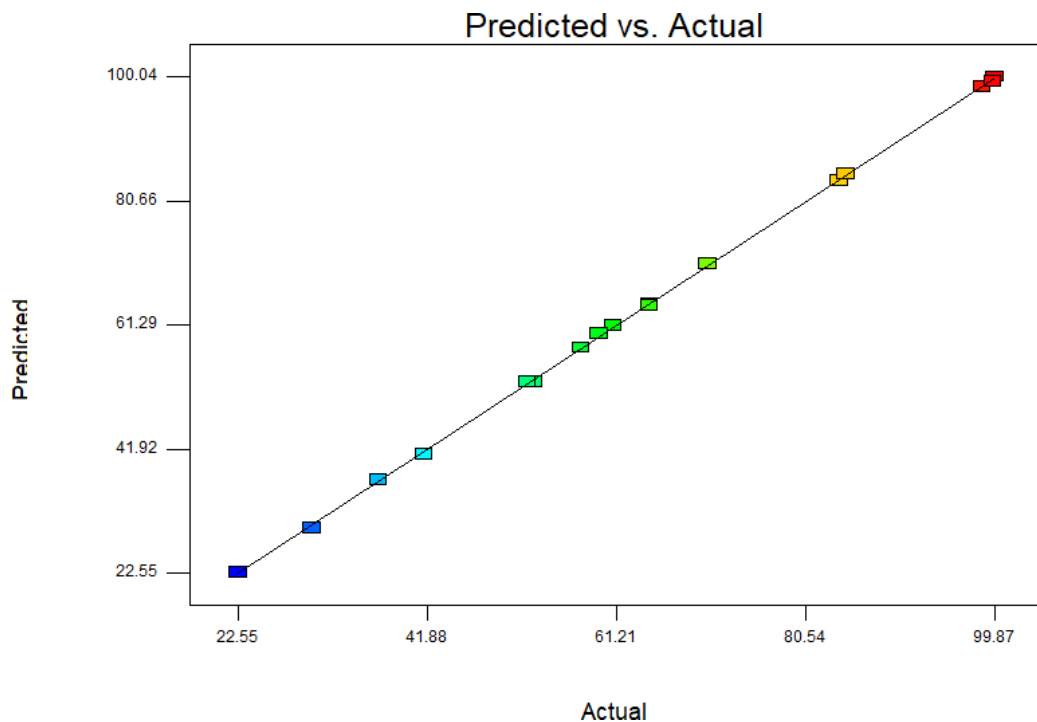
Appendix E2: Outlier Plot of Residuals vs. Run



Appendix E3: Analysis of Variances (ANOVA) and lack-of-fit

Source	SOS	df	Mean square	F value	P-Prob
Block	0.98	1	0.98		
Model <0.0001 (significant)	9062.59	9	1006.95	4604.84	
A (0.0001)	1002.60	1	1002.60	4584.94	<
B (0.0001)	4235.78	1	4235.78	19370.38	<
C (0.0001)	521.86	1	521.86	2386.50	<
AB (0.0001)	249.09	1	249.09	1139.10	<
AC (0.0001)	221.13	1	221.13	1011.24	<
BC (0.0001)	226.42	1	226.42	1035.42	<
A^2 (0.0001)	192.25	1	192.25	879.16	<
B^2 (0.0001)	234.28	1	234.28	1071.38	<
C^2 (0.0001)	2595.87	1	2595.87	11871.00	<
Residual	1.31	6	0.22		
Lack of Fit (not Significant)	1.11	5	0.22	1.08	0.6196
Pure Error	0.20	1	0.20		
Cor Total	9064.88	16			

Appendix E4: Graph of Predicted vs. Actual

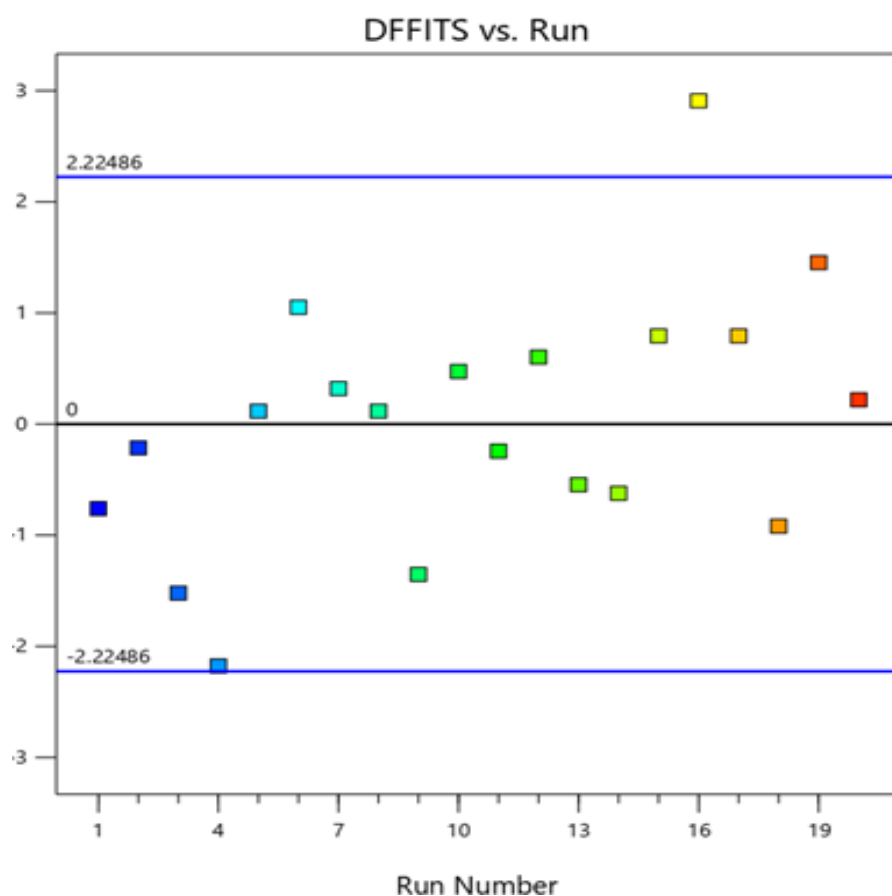


Appendix F: RSM of MB

Appendix F1: Statistical Parameters obtained from the Analysis of Variance (ANOVA) for the model of MB adsorption

Variables	Adsorption (%)
Std. Dev.	0.407
Mean	83.96
Coefficient of Variation (%)	0.485
PRESS	6.28
R-squared	0.999
Adj R-squared	0.998
Pred R-squared	0.997
Adeq Precision	113.0483

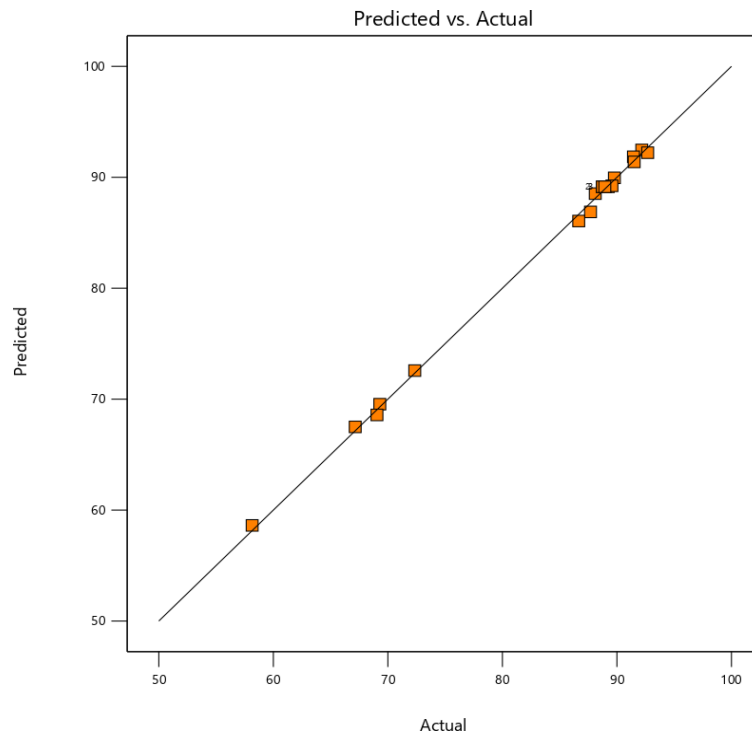
Appendix F2: Outlier Plot of DFFITS vs. Run



Appendix F3: Analysis of Variance (ANOVA) and lack-of-fit

Source	SOS	df	Mean square	F value	P-Prob
Block	112.00	1	112.00		
Model <0.0001 (significant)	1916.07	9	212.90	1283.44	<
A (0.0001)	67.89	1	67.89	409.26	<
B (0.0001)	1193.10	1	1193.10	7192.56	<
C (0.0001)	20.21	1	20.21	121.84	<
AB (0.0001)	24.36	1	24.36	146.86	<
AC (0.0001)	17.05	1	17.05	102.80	<
BC (0.0001)	15.40	1	15.40	92.85	<
A^2 (0.9520)	6.359E-004	1	6.359E-004	3.834E-003	
B^2 (0.0001)	205.53	1	205.53	1239.02	<
C^2 (0.0002)	6.04	1	6.04	36.43	
Residual	1.49	9	0.17		
Lack of Fit (not Significant)	1.33	5	0.27	6.62	0.054
Pure Error	0.16	4	0.040		
Cor Total	2029.59	19			

Appendix F4: Graph of Predicted vs Actual



Appendix H: BET Analysis

Appendix H1: BET analysis of SE-B



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version 2.01



Analysis	Report	
Operator: 1	Operator: 1	Date: 2018/05/25
Sample ID: J28-SA	Filename: J28-SA.qps	

Multi-Point BET

Data Reduction Parameters Data

Adsorbate	Thermal Transpiration: on	Eff. mol. diameter (D): 3.54 Å
	Nitrogen	Temperature 77.350K
	Molec. Wt.: 28.013	Cross Section: 16.200 Å²
		Liquid Density: 0.808 g/cc

Multi-Point BET Data

Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]	Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]
9.98052e-02	0.2336	3.7969e+02	2.50755e-01	0.3635	7.3663e+02
1.50640e-01	0.2944	4.8205e+02	3.00728e-01	0.3833	8.9783e+02
2.00703e-01	0.3399	5.9116e+02			

BET summary

Slope =	2570.936
Intercept =	1.019e+02
Correlation coefficient, r =	0.994542
C constant =	26.223
Surface Area =	1.303 m²/g

Appendix H2: BET analysis of SE-BC

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Analysis			Report	
Operator:	1	Date: 2018/05/03	Operator:	1
Sample ID:	J24-SA	Filename:	J24-SA(1).qps	Date: 2018/05/03

Multi-Point BET

Data Reduction Parameters Data

<u>Adsorbate</u>	Thermal Transpiration: on	Eff. mol. diameter (D): 3.54 Å	Eff. cell stem diam. (d): 4.0000 mm
	Nitrogen	Temperature 77.350K	
	Molec. Wt.: 28.013	Cross Section: 16.200 Å²	Liquid Density: 0.808 g/cc

Multi-Point BET Data

Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]	Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]
1.00426e-01	1.5792	5.6564e+01	2.49809e-01	2.2398	1.1895e+02
1.49926e-01	1.8138	7.7799e+01	2.99732e-01	2.4425	1.4021e+02
1.99988e-01	2.0305	9.8505e+01			

BET summary

Slope = 418.159
 Intercept = 1.479e+01
 Correlation coefficient, r = 0.999971
 C constant = 29.282
 Surface Area = 8.044 m²/g

Appendix H3: BET analysis of SE-AC

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Analysis			Report	
Operator:	1	Date: 2018/05/25	Operator:	1
Sample ID:	J28-SB	Filename:	J28-SB.qps	Date: 2018/05/25

Multi-Point BET

Data Reduction Parameters Data

Adsorbate	Thermal Transpiration: on	Eff. mol. diameter (D): 3.54 Å	Eff. cell stem diam. (d): 4.0000 mm
Nitrogen		Temperature 77.350K	
Molec. Wt.: 28.013		Cross Section: 16.200 Å²	Liquid Density: 0.808 g/cc

Multi-Point BET Data

Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]	Relative Pressure [P/Po]	Volume @ STP [cc/g]	1 / [W((Po/P) - 1)]
9.98035e-02	16.6677	5.3222e+00	2.49390e-01	22.9878	1.1564e+01
1.50034e-01	19.7774	7.1413e+00	2.99263e-01	24.5546	1.3916e+01
1.99181e-01	21.4781	9.2656e+00			

BET summary

Slope =	43.372
Intercept =	7.877e-01
Correlation coefficient, r =	0.998764
C constant =	56.063
Surface Area =	78.863 m²/g

Journal Publications

1. **Makshut, N.**, Ngaini, Z., Wahi, R., H, Hussain., Mahmut, N. I. & Bahrin, N. Q. (2020). Nano-sized adsorbent from pyrolysed sago activated sludge for removal of Pb(II) from Aqueous Solution. *Pertanika Journal of Science and Technology*, 28(3), 893-916.