



**STUDY OF *COCOS NUCIFERA L.* (COCONUT)
SHELL ACTIVATED CARBON FOR THE
REMOVAL OF METHYLENE BLUE IN
AQUEOUS SOLUTION**

by

IZZATY BINTI INU

A thesis submitted in fulfillment of the requirements for the degree of
Bachelor of Applied Science (Materials Technology) with honours

**FACULTY OF EARTH SCIENCE
UNIVERSITI MALAYSIA KELANTAN**

2016

ACKNOWLEDGEMENTS

In the name of ALLAH the most merciful, thank you for blessing and giving me strength and ability to complete this final year project.

Firstly, I would like to acknowledge my deepest gratitude to my great supervisor, Sir Nor hakim Bin Abdullah for his guidance, constant attention, valuable suggestion, enthusiastic support and personal concern during the this research and throughout the course of my study. Furthermore, appreciation also goes to my Co-supervisor, Dr Muhammad Azwadi Bin Sulaiman for his attention and guidance.

Special thanks to the Dean Earth Science Professor Madya Aweng A/L Ehrak, for his permission to let me use all the facilities and equipment in completing my final year project. I would also extend my sincere appreciations for all lecturers in the university that always gave me support and guidance. My deepest gratitude also goes to all technicians and administrative staffs of the Faculty of Earth Science especially to En. Nor Fadhli, En. Firdaus, En. Auzan, En. Shamsul Amri and En. Saiful for helping me throughout my research. Special appreciation goes to my loving parents especially my mom Mek Fisah Binti zulkifli and my father Inu Bin Daraman who were always on my side, riding along with me on my ups and downs as well as giving me the encouragement to pursue my dreams.

Enormous appreciation to my beloved friend, Siti Nur Aisyah Binti Zawawi and all of my dearest friends especially my teammates under the same supervision who always be there for me through my thick and thin and always love me.

IZZATY BINTI INU

Study Of *Cocos Nucifera L.* (Coconut) Shell Activated Carbon for The Removal of Methylene Blue in Aqueous Solution

ABSTRACT

Activated carbon (AC) was obtained from carbonaceous raw material that normally prepared through carbonization and activation process in certain conditions. The purpose of this study was to investigate the potential application of coconut shell activated carbon as methylene blue (MB) removal from aqueous solution. The carbonization process was a process converting the raw materials to char involving temperature 300 to 500°C. In the activation process, the pore size increased when impregnating with phosphoric acid, H₃PO₄ as acid activating agent and sodium hydroxide, NaOH as basic activating agent. The pore size of CSAC was characterized by using X-Ray Diffraction (XRD) for phase identification and Fourier transform infrared spectroscopy (FTIR) for detecting functional group that present in CSAC. After that, the kinetic study was investigated in order to determine the percentages removal of dyes (MB) once the adsorbent (CSAC) was introduced to the adsorbate (MB). It can be concluded that increasing the amount of CSAC also showed increment of dye removal. The results indicated that the coconut shell activated carbon could be employed as a low cost alternative in controlling wide range of sorption processes.

UNIVERSITI
MALAYSIA
KELANTAN

Kajian Tentang *Cocos Nucifera L.* (Tempurung Kelapa) Karbon Teraktif Untuk Penyingkiran Metilena Biru di dalam Larutan Cairan

ABSTRAK

Karbon teraktif (AC) diperoleh daripada bahan mentah karbon yang biasanya disediakan melalui karbonisasi dan pengaktifan proses dalam keadaan tertentu. Tujuan kajian ini adalah untuk mengkaji aplikasi potensi tempurung kelapa karbon diaktifkan sebagai metilena biru (MB) penyingkiran daripada larutan akueus. Proses karbonisasi adalah satu proses yang menukarkan bahan-bahan mentah ke arang melibatkan suhu 300°C hingga suhu 500°C. Dalam proses pengaktifan, saiz liang meningkat apabila direndam dengan asid fosforik, H₃PO₄ asid mengaktifkan ejen dan natrium hidroksida, NaOH sebagai ejen mengaktifkan alkali. Saiz liang CSAC dicirikan dengan menggunakan *X-Ray Diffraction* (XRD) untuk mengenal pasti fasa dan *Fourier transform infrared spectroscopy* (FTIR) untuk mengesan kumpulan berfungsi yang hadir dalam CSAC. Selepas itu, kajian kinetik telah disiasat untuk menentukan penyingkiran peratusan pewarna (MB) sebaik sahaja penjerap (CSAC) telah diperkenalkan kepada bahan terjerap yang (MB). Dapat disimpulkan bahawa peningkatan jumlah CSAC juga menunjukkan kenaikan penyingkiran pewarna. Keputusan menunjukkan bahawa tempurung kelapa karbon diaktifkan boleh digunakan sebagai alternatif kos rendah untuk mengawal pelbagai proses penyerapan.

UNIVERSITI
MALAYSIA
KELANTAN

TABLE OF CONTENT

	PAGE
DECLARATION	
ACKNOWLEDGEMENT	ii
ABSTRACT (ENGLISH)	iii
ABSTRAK (MALAY)	iv
TABLE OF CONTENTS	v-vii
LIST OF FIGURES	viii
LIST OF ABBREVIATIONS	ix
CHAPTER 1 : INTRODUCTION	
1.1 RESEARCH BACKGROUND	1
1.2 PROBLEM STATEMENT	5
1.3 OBJECTIVES	5
1.4 EXPECTED OUTCOMES	6
CHAPTER 2 : LITERATURE	
REVIEW	
2.1 Activated Carbon	7
2.2 Activated Carbon from Coconut Shell	12
2.3 Activation Method	16
2.3.1 Sodium Hydroxide, NaOH	19
2.3.2 Phosphoric Acid, H ₃ PO ₄	20
2.4 Kinetic study & Batch adsorption method	21

CHAPTER 3 : MATERIALS**METHODS**

3.0 Methodology	22
3.1 Study Area	23
3.2 Preparation Of Activated Carbon	23
Activation Method	24
3.3 Preparation Of Adsorbent Using Phosphoric Acid	25
3.4 Preparation Of Adsorbent Using Sodium Hydroxide	26
3.5 Preparation Of Methylene Blue	28
3.5.1 Adsorbate	28
3.6 Measurement of Decolorization of Dyes	30
3.6.1 Batch study	30
3.6.2 Dialysis Tube	20
3.7 Kinetic Study	33
3.8 Characterization of AC	35
3.8.2 X-Ray Diffraction (XRD)	35
3.8.3 Fourier transform infrared spectroscopy (FT-IR)	36
3.8.4 Ultraviolet–visible (Uv-Vis)	36

CHAPTER 4: RESULT & DISCUSSION

4.1 Overview	37
4.2 Phase identification (XRD)	37
4.3 FT-IR	40
4.4 Effect of Chemical Activation	43
	46
4.6 Kinetic study	49

CHAPTER 5 : CONCLUSION

Conclusion & Recommendation	51
References	53
Appendix	58

LIST OF FIGURE

Figure 2.1	Coconut shell activated carbon	12
Figure 2.2	Carbonized coconut shell morphology	18
Figure 2.3	Activated coconut shell morphology (Njoku <i>et al.</i> , 2014).	18
Figure 3.1	Chemical formula of methylene blue	28
Figure 4.1	XRD profile of CSAC	38
Figure 4.2	FT-IR profile of CSAC	42
Figure 4.3	Dye removal by CSAC	48
Figure 4.4	Percentages decolorization of MB (%)	50

LIST OF ABBREVIATIONS

CS	Coconut Shell
AC	Activated Carbon
MB	Methylene Blue
cm	Centimetre
ASTM	American Society For Testing And Materials
H ₃ PO ₄	Phosphoric Acid
NaOH	Sodium Hydroxide
KOH	Potassium Hydroxide
ZnCl ₂	Zinc Chloride
H ₂ SO ₄	Sulphuric Acid
CaCl ₂	Calcium Chloride
M	Meter
µm	Micrometer
g	Gram
ml	Millilitre
pH	Percentages of Hydrogen
nm	Nanometer
CSAC	Coconut Shell Activated Carbon
Rpm	Rotation per minute
XRD	X-Ray Diffraction
FT-IR	Fourier Transform Infrared Spectroscopy
UV-Vis	Ultraviolet Visible

MALAYSIA

KELANTAN

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The earth appears blue because large bodies of saline water known as ocean dominate the surface approximately 70.8 % and the land cover approximately 29.2 %. Water plays a vital role in developing of communities hence a reliable supply of water is essential and needs to be maintained all the time for human and industrial use. The factor that contributes to the increasing of water crises are rapidly continued due to the increasing in population's growth and urbanization (Adedayo *et al.*, 2012).

A large amount of freshwater and synthetic dyes involve in textile dyeing. In the dyeing process, the amount of colour effluents of high polluting degree are generated Asghar *et al.* (2015) this situation will not only influence the environment but also affect the human health if it is release to environment without being treated properly (Hu *et al.*, 2015).

In recent year, pollution from dye wastewater becomes a serious environmental problem due to the high demand and increasing of using varieties of dye. Dye has been an indispensable tool for a variety of industries such as textile, pulp and paper industries and other fabric finishing. More than 100,000 types of dyes were produced exceeding 150 metrics tons per year (Selvanathan & Subki, 2015).

According to Akar *et al.* (2009) contamination of the industrial waters with the organic based chemicals has created a serious environmental problem. Dyes can cause major environmental problems due to its toxicity and carcinogenicity properties. The carcinogenicity is the substances that can cause cancer from benzidine, naphthalene and other aromatic components (Selvanathan & Subki, 2015).

The high level of dyes production worldwide plus their extensive use in many applications in general has produced coloured wastewater which cause in severe water pollution. Water pollution is any contamination or change in the quality of water through contact with chemical, physical and biological elements obviously would affect the originality of water (Adedayo *et al.*, 2012).

The coloured dyes are usually very toxic to the flora and fauna of life of the particular region and habitat (Abdulraheem *et al.*, 2015). Reactive dyes pose the greatest problem in terms of colour, which is released by the cotton fashion industry that dominance in today's industry (Santhy & Selvapathy, 2006). The cotton textile industry residues are generally composed of the microfibers lost in the industrial processes of spinning and weaving (Avelar *et al.*, 2016).

Due to their chemical structures, dyes are resistant to fade on exposure to light, water and many chemicals. Therefore, the dyes are difficult to treat once they are released into aquatic environment (Sharma *et al.*, 2008). Synthetic dye is a group of organic pollutants that are extensively used in several industries that can cause hazardous effect once introduced to aqueous solution due to its carcinogenic, toxicity, mutagenic and allergenic to nature (Akar *et al.*, 2009).

The presence of very small amount of dyes in water eventhough it is less than 1mg dm^{-3} for some dyes are highly visible and undesirable. Synthetic dyes also have a complex aromatic molecular structure, which makes them stable and resistant to biological degradation (Akar *et al.*, 2009). The released of coloured wastewater may present an eco-toxic hazard and introduce the potential danger of bioaccumulation which may eventually affect human being through food chain (Demiral, 2008).

There are many approaches to remove the organic pollutants, such as dyes from wastewaters such as chemical oxidation, physicochemical and biological processes. Some of these processes include coagulation-flocculation, filtration, adsorption, advanced oxidation processes, ion-exchange, biological treatment and magnetic separation. Among these, adsorption is considered one of the most promising alternatives for the removal of organic pollutants from wastewater because of its simplicity, ease of operation, high removal efficiency and regeneration capacity (Martinez *et al.*, 2003). Among several adsorbents listed in the literature, activated carbon is a well-known adsorbent that has been used effectively for the removal of organic pollutants, because it is a highly porous material and possesses an extensive surface area (Yorgun & Yildiz, 2014; Akar *et al.*, 2009).

Activated carbons (AC) are porous materials of great importance for several processes. Among the main applications of AC, it is also can be used in the adsorption of pollutants in gaseous phase or liquid phase, gases storage, and as catalysts support. Porous materials such as AC are usually characterized by several physical parameters such as surface area and pore volume (Nunes & Guerreiro, 2011).

AC has large specific surface area and superior adsorption characteristic (Zhang *et al.*, 2012). Activated carbons are used worldwide in a many great industrial separation processes because of their ability to preferentially adsorb particular chemicals when introduced to solution containing those chemicals (Mozammel *et al.*, 2002). Therefore, the development of a porous structure is improved with chemical activation process.

AC is an important component of filter material for the removal of hazardous components in exhaust gases for the purification of drinking water and for waste water treatment (Asma *et al.*, 2011). Liquid phase adsorption has been proven to be an effective way for removing suspended solids, odours, organic matters, and oil from aqueous solutions (Latinwo *et al.*, 2015).

Natural materials that are available in large quantities or certain waste from agricultural operations may have potential as low cost adsorbents, as they represent unused resources (Babel & Kurniawan, 2004). The characteristics of agricultural plant materials, such as polar and aromatic components, impose a significant influence on their sorptive behaviour (Latinwo *et al.*, 2015).

Biomass has become an important source of carbon for the production of AC. By reusing and recycling the low cost materials to produce multifunction of activated carbon, it provides another eco-friendly alternatives to dispose the waste and by product (Asma *et al.*, 2011).

Coconut shell (CS) is an agricultural solid waste, available throughout the year. Conversion of CS into AC which can be used as adsorbents in water purifications as well as the treatment of industrial and municipal effluents would have value added of these agricultural commodities.

1.2 Problem Statement

Generally, the dyes\ from wastewater in textile application contain residue of dyes and chemical substances. There are many various treatment methods for the dyes removal that have been proposed such as coagulation, filtration, sedimentation and adsorption. The previous method such as coagulation will eventually causing the production of sludge and ion exchange which could result in higher cost maintenance. Besides, the previous method also less efficient and result in phase transfer of pollutants and leaving the problem unsolved.

Adsorption method is the efficient way to remove dye due to good properties such as high surface area and high pore volume that will increase the rate of adsorption whether been introduced into liquid or gaseous phase. The adsorption method can be prepared by numerous of number of carbonaceous raw material. The adsorption of dye using AC from CS has high potential since it economically viable, easy to obtain, good adsorption ability and good in controlling the pollution.

1.3 Objectives

- i. To characterize the activated carbon from coconut shell.
- ii. To study the potential application of CS activated carbon as methylene blue (MB) removal from aqueous solution.
- iii. To identify the effects of chemical treatment on dye removal efficiency

1.4 Expected Outcomes

From this study, the effect of CS as AC on dye adsorber can be improved. The properties of CS as AC can increase the rate of adsorption of dye removal. It is expected that activated carbon from CS could remove the MB from aqueous solution. The AC derived from CS has been widely used in various number of applications especially in applications that involve textile production.

The CSAC was expected to show better efficiency in dye removal due to the properties such as high pore size and extensive surface area. The wastewater pollution able to be controlled by using low cost starting material but can give a huge positive feedback in many applications whether in air or water pollution. The CSAC able to remove high percentages concentration of dyes once applied in industry.

The AC will be an outstanding way to be applied in numerous applications and able to compete with other reliable methods. It will also contribute many benefits compared to other previous methods. In today's applications, we also been exposed on how to create or value added the abundant material to be something that very useful in daily routine. The abundant of coconut shell can be controlled if we can commercialize it in widely application.

CHAPTER 2

2.0 LITERATURE REVIEW

2.1 Activated Carbon

AC has received much attention in recent years due to its versatile applications in material science (Chen *et al.*, 2012). A variety of biomaterials waste with high carbon content are widely used as initial materials for obtaining AC such as coconut shell, walnut shell and pine sawdust (Üner *et al.*, 2015). AC is the generic term used to describe a family of carbonaceous adsorbents with a highly crystalline form and extensively developed internal pore structure.

AC are made from materials enrich in carbon element through carbonization and activation process (Shamsuddin *et al.*, 2016). AC is produced in most develop areas around the world (Chandra *et al.*, 2008). Carbonaceous adsorbents have been found to be particularly useful due to their good kinetic properties and high adsorption capacities (milligrams of adsorbate retained per gram of carbon) (Boualem *et al.*, 2014).

The activated carbon has a large number of very fine pores (micropores) that gives the AC larger inner surface, which is the basis of its remarkable adsorption properties (Ansari, 2009). Activated carbon with high micropores volume are widely used for the adsorption of small size molecules, but adsorbents with high mesopores volume are required for the adsorption of large molecules such as dyes and compound with higher molecular weight (Üner *et al.*, 2015).

AC are able to adsorb pollutants due to high surface area and high pore volume that can be produced from different raw carbon resources like lignite, peat, coal, and biomass resources such as wood, sawdust, bagasse, and coconut shells as show in Figure 2.1(Grauto *et al.*, 2008). But it also has several disadvantages such as high operating cost and regeneration problems (Akar *et al.*, 2009).

There are several decontamination methods have been developed and established over recent decades to remove organic and inorganic pollutants from water and wastewater, including floatation, precipitation, membrane separation, biosorption, and activated carbon adsorption (Muhamad, 2016).

According to Selvanathan & Subki (2015), the previous method such as activated carbon, sorption, chemical coagulation, ion exchange, electrolysis and chemical treatment have been developed before dyes are released to environment however, adsorption of activated carbon is the better option in terms of practicality and costing compared to other methods.

Among the previous methods, adsorption is considered one of the most promising alternatives for the removal of organic pollutants from waste because it is simple, conventional technology operation, good efficiency and regeneration capacity (Georgin *et al.*, 2016). According to Chang *et al.* (2000), any inexpensive material with high carbon content but low inorganic can be used as raw material for the production of activated carbon.

Activated carbon should be low in ash but rich in carbon and volatiles. The spherical AC have a more regular and smoother surface and a higher control over the pore size distribution (Laginhas *et al.*, 2016). The ash consists of mainly minerals such as silica, alumina, iron, magnesium, and calcium (Asma *et al.*, 2011). The primary raw material used for activated carbon is any organic material with high carbon content.

The adsorptive capacities of AC are associates with the internal porosity and are related to its properties such as surface area, pore volume, and pore size distribution (Yorgun & Yildiz, 2014). In addition, the internal pore structure, surface characteristics, degree of surface reactivity and presence of functional group on pore structure play an important role in adsorption processes (Yorgun & Yildiz, 2014). According to Byamba-Ochir *et al.* (2016), the AC is widely known due to high adsorption capacity because their internal structure which consist of a large number of the interconnecting fines pores.

AC is well known adsorbent that has been used for high efficiency for the removal of organic pollutants, because it is highly porous material and have extensive surface area (Georgin *et al.*, 2016). Activated carbon is an inert porous carrier substances dominantly used in air and wastewater decontamination since its capability of distributing chemical on its large hydrophobic internal surface (Li *et al.*, 2016).

The carbon-based material is converted to AC through physical modification and thermal decomposition in a furnace, under controlled atmosphere and temperature. The pore structure and pore size distributions of activated carbon commonly varies based on the nature of raw materials and activation method (Chandra *et al.*, 2009).

CS was carbonized in an old-age process commonly known as open pit method. In this process earth is used as an insulator and to heat the shells in the absence of oxygen (Shankar, 2008). Traditionally activated carbons possess a wide range of pore size typically from 2 to 2000 Å. Although, they have a larger surface area, the adsorption selectivity is limited because it is based on the shape and size of molecules. An increasing in selectivity and hence a narrower pore size distribution is highly desirable for separation purpose (Boualem *et al.*, 2014).

Adsorption on activated carbon is one of the most efficient techniques used in water treatment process for the removal of organics and micro pollutants from wastes and drinking waters (Latinwo *et al.*, 2015). According to Gao *et al.* (2016), amongst the numerous techniques, adsorption with activated carbon is an attractive and effective alternatives way to eliminate organic dyes from wastewater.

Adsorption with AC is a well-studied technique to remove amount of adsorbate from aqueous solution (Azmi *et al.*, 2015). AC is extremely porous with a very large surface area, which makes it an effective adsorbent material. This large surface area relative to the size of the actual carbon particle makes it easy to remove the large amount of impurities in a relatively smaller closed space (Shankar, 2008).

AC has a tremendous adsorbing capacity for a wide variety of dissolved organics and chlorine and can be custom-tailored to suit specific application (I *et al.*, 2012). The higher the filter content the good the activated carbon produced. The surface of the carbon is in square meters per gram and the pore structure in distribution of macropores (>50 nm), mesopores (>2-50 nm) and micropores (<2 nm) (Adedayo *et al.*, 2012).

AC adsorptive properties are used to remove organics. AC adsorbs organic material because the attractive forces between the carbon surface (non-polar) and the contaminant (non-polar) are stronger than the forces keeping the contaminant dissolved in water (polar) (Inegbenebor *et al.*, 2012).

The properties of activated carbon make it possible to trap poisons, insecticides, bad smell, tastes, heavy metal and undesirable element in both liquid and gas and there are more potential raw material for the production of AC to meet local industrial needs (Inegbenebor *et al.*, 2012). According to Adedayo *et al.*, (2012), The specific properties of an activated carbon are results from the type of raw carbonaceous material used to produce it and the activation process which can boosts up its adsorbent quality.

The application of activated carbon are widely used in many fields , for example in water and air purification, separation of industrial gases, off-gas cleaning in pharmaceutical and food industry, discolouring pharmaceuticals, as well as in making oral carbon tablets, anti antidotes raw materials (Boualem *et al.*, 2014). AC can acts as a filter material in air cleaning filters for removal of gaseous and vapours in the industrial environment (Ansari, 2009). According to Ünner *et al.*, (2015), high mesopores activated carbon are widely used in removal dye in aqueous solution, electro-catalysis, biocatalysts, lithium batteries and electrochemical capacities.

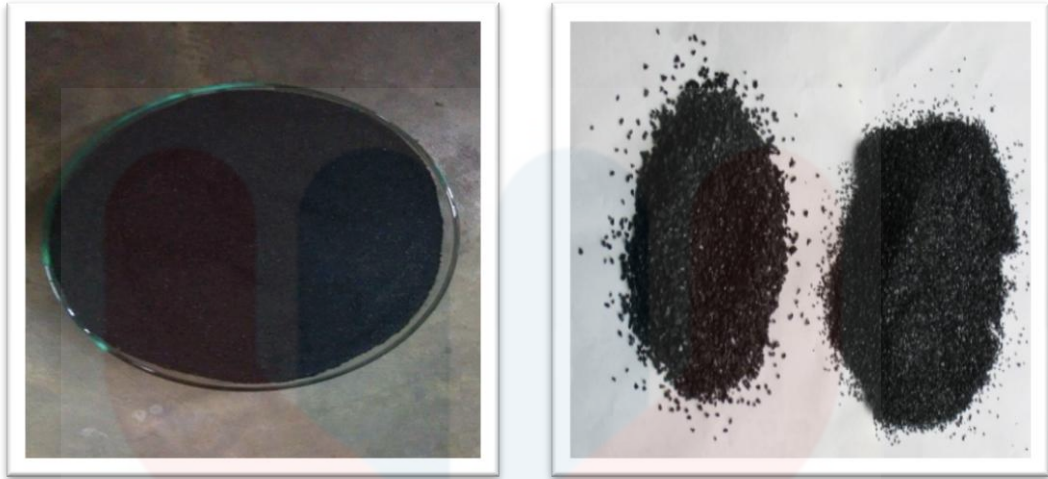


Figure 2.1: Coconut shell activated carbon

2.2 Activated Carbon from Coconut Shell

Coconut palm is a member of the *Areaceae* family (palm family) which is grown throughout the tropics. It sustains the livelihood of millions of people in coastal regions of the tropical world (Latinwo *et al.*, 2015). Coconut Shell (CS) contain cellulose, hemicelluloses and lignin, with an average composition of carbohydrate $C_6H_{10}O_5$ (Shankar, 2008). The CS is a hard and thick bony endocarp material, which often present serious disposal problem for local environment. It is also an abundantly available agricultural waste from the local coconut industry (Babel & Kurniawan, 2004). In the present study CSAC is used as an adsorbent (Kulkarni *et al.*, 2013).

The CS is an agriculture crops that widely found in tropics region. Coconut coir dust is one of the agricultural waste products often used as adsorbent in waste water treatment (Okafor *et al*, 2012). The CS is the species that have been cultivated for over than 400 years ago. The coconut tree is originated from the Southeast Asia but this species has been cultivated widely and spread vigorously all over the world.

Cocos Nucifera can be distinguished based on their stem height, tall and dwarf varieties. Presently over 100 varieties are known about half of them are tall. The maximum tall of adult coconut palm is above 20 m and can reach 50-60 years old (Adedayo *et al.*, 2012).

The coconut tree can be easily obtained from tropical beaches. The country such as Caribbean, West Africa, South Asia, Philippines and Indonesia produced a large crops plantation for coconut tree. CS trees have a smooth, columnar, light grey-brown trunk, with a mean diameter at 30 - 40cm at breast height, and topped with a terminal crown of leaves (Inegbenebor *et al.*, 2012).

Coconut is the third most important industrial crop plantation in Malaysia in terms of cultivated area after oil palm and rubber. Coconut is known due to its versatility plant species. Coconut is not only applied in food based but also can be a main sources in obtaining an activated carbon (Njoku *et al.*, 2014). In Malaysia, in year 2001 the number of land being used for coconut plantation is around 151,000 ha but this gradually decreasing every year due to highly competition with oil palm plantation (Njoku *et al.*, 2014).

The remarkable properties of coir dust that enhances its effectiveness as adsorptive/ion exchange capacities include good structural stability, high water absorptivity and high porous nature. Its constituents of coconut shell include lignin-36.15 %, cellulose-33.61 %, pentosana-29.27 %, ash-0.61 % and other dry basis-0.37 % (Okafor *et al.*, 2012).

CS is an inexpensive and readily available biomass material that has high mechanical strength, good abrasion resistance and an inherent granular structure as well as high surface area and high pore volume (Zhang *et al.*, 2015). Furthermore, AC from coconut and pine tree have been studied as bioadsorbents for the removal of numerous type of pollutants from liquid phase due to their great availability (Tonucci *et al.*, 2015).

According to Zhu & Kolar, (2014), suggested that activated charcoal derived from CS has an excellent physical strength. Microstructure of pyrolyzed carbon is known to depend critically on the carbon source and hence a porous carbon with high surface area and efficiency sorption properties on pyrolysis (Raj & Alia 2015).

CS is also an abundant low cost agricultural biomass, which contains high amounts of natural polymer such as cellulose, hemicellulose and lignin are suitable for alternative materials to produce low cost and green adsorbent with necessary modification to improve efficiency (Shamsuddin *et al.*, 2016).

In addition, CS contains oxygen, hydrogen, and other elements whereas carbon as its main components. The carbonaceous adsorbents were characterized for iodine number in accordance with ASTM (1995) (Latinwo *et al.*, 2015). The iodine number gives an indication of the activated carbon in microspores or indication of porosity (Latinwo *et al.*, 2015).

According to Asma *et al.* (2011), increasing activation temperature gave higher iodine values and subsequently increased the adsorption capacity of the activated carbon. Iodine number is a relative indicator of porosity in an activated carbon sample. The iodine value is a milligram of iodine adsorbed by 1 gram of activated carbon determined according to ASTM 607-86 (ASTM 2011) and MMS 873:1984 (MS 2004) (Asma *et al.*, 2011).

Conversion of CS into AC which can be used in water purification would add its economic value will reduced the cost of waste disposal and most importantly provide a potentially inexpensive alternative to the existing coconut shell activated carbon (Babel & Kurniawan, 2004).

The CS is more applicable in producing AC compared to coconut husk because the husk generated carbon was not very durable and physically broke down in the treatment system. In filtration application, the coconut husk is less used compared to coconut shell due to fine charcoal residues leaching into the water during filtration. But the coconut husk good in flammability (Cobb, 2012) and high in filter content (Adedayo *et al.*, 2012). However, the coconut husk proved to be a useful combustion medium to improve the carbonization of the coconut shell (Cobb, 2012).

CSAC have several advantages over another sources of AC such as high density, high purity and virtually dust-free, since they are hardener and more resistant to attrition. The pore structure of the activated carbon from coconut shell is also uniform with the micropore range particularly effectives in gaseous and liquid phase adsorption (Mozammel *et al.*, 2002).

2.3 Activation method

Activation method is the method to enlarge the diameter of fine pores and also create new pores that the adsorptive power of the carbonization products is enhanced (Chandra *et al.*, 2009). In general, two different processes of physical and chemical activations are used to prepare the activated carbon (Muhamad, 2016; Shamsuddin *et al.*, 2016; Yakout & El-Deen, 2012; Njoku *et al.*, 2014). Physical activation involves carbonization of raw materials at high temperature in the presence of suitable oxidizing gaseous such as carbon dioxide, steam and air at temperature 800-1000°C to produce charcoal (Shamsuddin *et al.*, 2016) as shown in figure 2.3.

Chemical activation involves the impregnation of the precursor material with a chemical activity agent followed by activation at temperature of 400-500°C. Chemical activation is simultaneous process that combine both carbonization and activation method in single step (Njoku *et al.*, 2014).

In activated carbon, elements such as hydrogen, nitrogen, sulphur, and oxygen contents have been decreased because during pyrolysis and activation process, the coconut shell has been decomposed. During the decomposition, the volatile compounds containing mainly H, O and N leave the carbonaceous product and the CS become rich in carbon percentages (Das *et al.*, 2015).

The activation process plays an important role on the amount of yield activated carbon. Activation method can be separated into physical and chemical activation. Physical activation includes carbonization process or pyrolysis process under temperature 500°C to 800°C. The chemical activation has shown several advantages over physical activation, including lower temperature, shorter time, higher yield and larger surface area. Chemical activation method was divided into

two ways that were preparation of adsorbent using phosphoric acid and preparation of adsorbent using sodium hydroxide. Recently, AC with very high surface area has been obtained chemical activation and extensively used for pollutants removal (Üner *et al.*, 2015).

The widely used of chemical activating agents are zinc chloride, $ZnCl_2$, phosphoric acid, H_3PO_4 and potassium hydroxide, KOH (Shamsuddin *et al.*, 2016)). Chemical activation is more popular since its combination of carbonization with activation, lower operating temperature, higher yield, easy recovery of the activated reagents and better production with higher surface area and better developed porous structure (Li *et al.*, 2016). The chemical activating agents also improved the porosity of activated carbon by means of dehydration and degradation (Yorgun & Yildiz, 2014) as shown in Figure 2.2 and Figure 2.3.

The carbonization process is the process to increase the carbon content and produce initial porosity whereas the activation process helps in improving the pore structures as show in figure. The carbonization process was fully completed when the emitted gas turned in bluish colour (Asma *et al.*, 2011). However, the physical activation is more fulfilled compared to chemical activation.

The research found that surface area recorded by physical activation was slightly higher compared to chemical activation but lower in term of yield production (Shamsuddin *et al.*, 2016).The chemical activation involves impregnation of raw materials with dehydrating agents and oxidants such as sulphuric acid, H_2SO_4 , phosphoric acid, H_3PO_4 , sodium hydroxide, NaOH, Potassium hydroxide, KOH and Zinc chloride, $ZnCl_2$ (Muhamad, 2016). Previously, the highly porous carbon is produced by traditional steam activation process. However in order to produce high quality activated carbon it is involves the pyrolysis temperature at 800 to 1000 °C

which is not suitable technologies in developing country on a small scale production. The activated carbon can be produced through chemical activation method which is prepared through soaking the charcoal in the strong dehydrating agent such as zinc chloride, $ZnCl_2$ and calcium chloride, $CaCl_2$ after being carbonized at temperature range from 500 to 800 °C (Cobb, 2012).

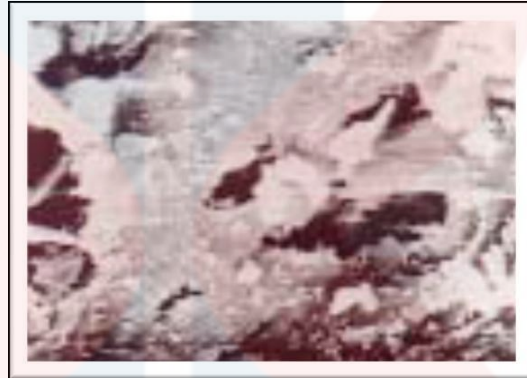


Figure 2.2: Carbonized coconut shell morphology

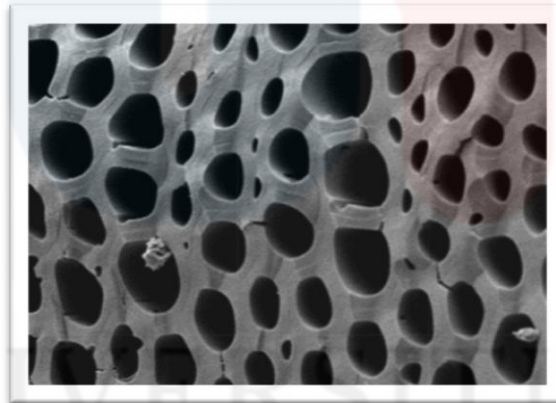


Figure 2.3: Activated coconut shell morphology

(Njoku *et al.*, 2014)

UNIVERSITI
MALAYSIA

KELANTAN

2.3.1 Sodium Hydroxide, NaOH

Generally NaOH are widely used as activating agents in carbon activation process. NaOH is an etching material that promotes defects in the structure of the carbon material in which this causes and improves porosity of carbon materials. According to Islam *et al.* (2015) NaOH activation enhanced the porosity and surface functionality of the hydrochar. NaOH is a reducing agent that has a nucleophilic effect because of a free electron pair in its hydroxide group ($-OH$). Thus, NaOH may decompose residual hemicellulose and to a particular extent, cellulose and lignin. In addition, by adjusting the activation temperature and impregnation ratio, the pore structure of carbon can be tailored (Chandra *et al.*, 2009).

According to Selvanathan & Subki (2015), the size of pore of AC depends on cation in the hydroxide which is sodium ion. When NaOH compared to KOH, the NaOH show excellent performances compared to KOH due to smaller size lies in periodic table. The NaOH can also penetrate more deeply into the activated carbon during activation method that produces high number of smaller pores. The iodine number increased as the activation temperature increased and particle size decreased (Adedayo *et al.*, 2012).

Besides, potassium hydroxide, KOH also can be used as an activating agent to the prepared activated carbon of durian shell by ratio of 0.5 and activation temperature of 500°C was found as the optimum conditions to acquire high surface area AC (Chandra *et al.*, 2009).

2.3.2 Phosphoric acid, H_3PO_4 ,

In chemical activation, phosphoric acid has been known for several years and applied to different cellulosic and lignocellulosic material to prepare activated carbon (Chen *et al.*, 2012)(Yakout & Sharaf El-Deen, 2012).The H_3PO_4 is well-known in preparation of AC. Among the numerous dehydrating agents used for chemical activation, phosphoric acid has been preferred recently due to environmental and economic concerns. Moreover phosphoric acid allows the developing both micropores and mesopores in the resulting activated carbon.

The H_3PO_4 , is a precursor material that used to retard the formation of chars, reduce the evolution of volatile matter and can enhance the yield of the resulting AC (Chang *et al.*, 2000). Phosphoric acid activation has been applied to different lignocellulosic materials such as wood and woody materials, fruit shell stones, and agricultural wastes (Yorgun & Yildiz, 2014). Compared to Zinc chloride, $ZnCl_2$ and hydroxides, H_3PO_4 shows advantages for producing activated carbon used for wastewater treatment (Sun *et al.*, 2016).

H_3PO_4 has low corrosivity to the equipment and metal residues, as well as environmental friendly (Sun *et al.*, 2016). Activated carbon activated from H_3PO_4 shows larger particles and good sedimentation performance, which is very suitable for water treatment. The H_3PO_4 , could increase the bond cleavage, hydrolysis, dehydration and condensation followed by cross-linking reactions between H_3PO_4 and AC of CS (Valle, 2015).

According to Selvanathan & Subki (2015), the adsorption of methylene blue for AC produced from NaOH activation shows lower adsorption compared to AC produced from acid activation H_3PO_4 . H_3PO_4 activation is beneficial for the development of mesoporous, which is beneficial for larger molecules adsorption (Sun *et al.*, 2016).

2.4 Kinetic Study and batch adsorption method

Batch adsorption study plays important roles in obtaining parameters for designing adsorption experiments and to obtain key parameter. Adsorption using column is one of the most common and efficiency way for removal of pollutants from water. However, it is worth to note that it may be helpful that before evaluating that feat of adsorbent in fixed, fixed-bed adsorber, adsorption isotherm studies such as batch experiments were conducted to calculate the maximum adsorption capacity of the adsorbent.

The activated carbon was characterized for its adsorption capacity using MB. The solution was kept in shaking incubator at $27^{\circ}C$ with rotation speed 150 rpm for 3 hours. The samples were withdrawn at appropriate time interval using glass syringe to determine the residue concentration of the solutions. Spectrophotometer was used to determine the residual concentration of the methylene blue at 668 nm wave length (Lam & Zakaria, 2008).

2.5 Characterization of activated carbon

AC is mainly characterized by its degree of adsorptive ability. Total surface is one of the main parameters that is often used to market activated carbon products (Mozammel *et al.*, 2002). The AC was characterized due to its surface physical and chemical properties (Zhu & Kolar, 2014).

Chemical activation can be both acid and base activation are widely used to introduced different types of oxygen functionalities on the carbon surface by partial oxidation of carbon. According to Chen *et al.* (2012), the adsorptive performance of AC characterized by iodine number and methylene blue. The methylene blue number is defined as the maximum amount of dyes adsorbed by 1 g of adsorbent. The iodine number is defined as the milligrams of iodine adsorbed by 1 g of carbon (Nunes & Guerreiro, 2011).

CHAPTER 3

3.0 METHODOLOGY

3.1 Study Area

The study area for this final year project was located around the University Malaysia Kelantan (UMK). The coconut shells (CS) were obtained from residential area in Gemang, Jeli. This experiment was carried out mostly in Material & Science Laboratory in UMK Jeli Campus and several laboratories for activation process and the rest. Preparation of raw coconut shell was conducted in laboratory in Agropark UMK Jeli.

3.2 Preparation of Activated Carbon

The CS was washed repeatedly with tap water to remove all the dirt and dust that adhere to it. The CS were crushed into smaller pieces with jaw crusher in order to obtain uniform the size of the coconut shell about 10 mm or the CS ground in micro hammer into smaller pieces (40/60 meshes). The CS was dried in oven for 2 to 3 hours at 105 °C to remove all the moisture content and other volatile impurities so that it may reduce in weight. Then the CS was grinded by using the grinder machine. The CS was sieved by using 300µm siever about 30 minutes and repeated for twice in order to obtain uniform size of sieved CS as shown in Figure 3.1.

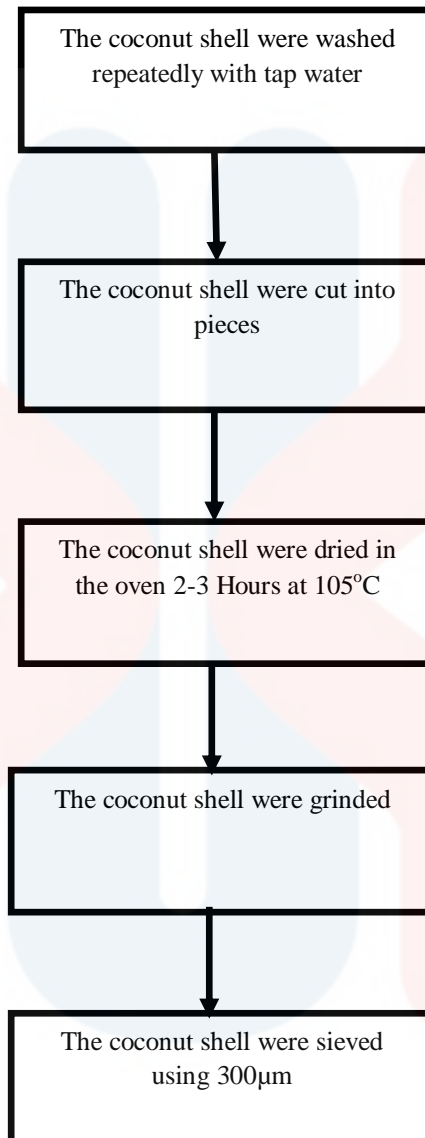


Figure 3.1: Preparation of Activated Carbon

3.3 Preparation of Adsorbent Using Phosphoric Acid

For activation process based on Phosphoric acid H_3PO_4 , the 85% of orthophosphoric acid was diluted with distilled water by using dilution formula $M_1V_1 = M_2V_2$ until 40% of phosphoric acid was obtained. Then, the CS was soaked in a boiling solution of 40 % phosphoric acid for 1 hour. Later, the CS was placed in the oven at 105 °C for 24 hours for further activation process. The CS was kept at room temperature for 24 hours then placed again in furnace at 300°C for 1 hour. Then, CS was washed and filtered with hot and cold deionized water until the pH dropped to 5-7 for 17 hours by using pH meter. Next, the CS was dried in oven at 120 °C for 2 hours to eliminate the moisture content. Finally, the CS was stored in the airtight lid container such as glass petri dish to prevent moisture build up and fungi infection for prior to analysis as shown in Figure 3.2.

3.4 Preparation of Adsorbent Using Sodium Hydroxide

For activation process based on Sodium Hydroxide, the pellets form of NaOH were stirred and diluted with distilled water until dissolved homogenously. The CS was soaked in 4 % sodium hydroxide for 1 hour in the ratio 1:20 to homogenous the solution. Next, the CS was kept overnight before dried in oven at 105 °C for 3 hours. Then, the CS was carbonized in furnace at 200 °C for 60 minutes for further activation process. After that, the CS was washed by 0.5 M hydrochloric acid, HCl solution to stabilize the pH of the based sodium hydroxide and remove ashes that blocked the pores of AC. Subsequently, the CS was washed with hot deionized water to remove different residual organic and mineral matter until pH reaches from 7 to 8 by using pH meter. Later, the CS was rewashed with cold deionized water. The CS was dried in oven at 105 °C for 2 hours for fully dried. Finally, the CS was stored in

airtight lid container such as glass Petri dish to overcome the moisture growth and fungal build up as shown in Figure 3.3.

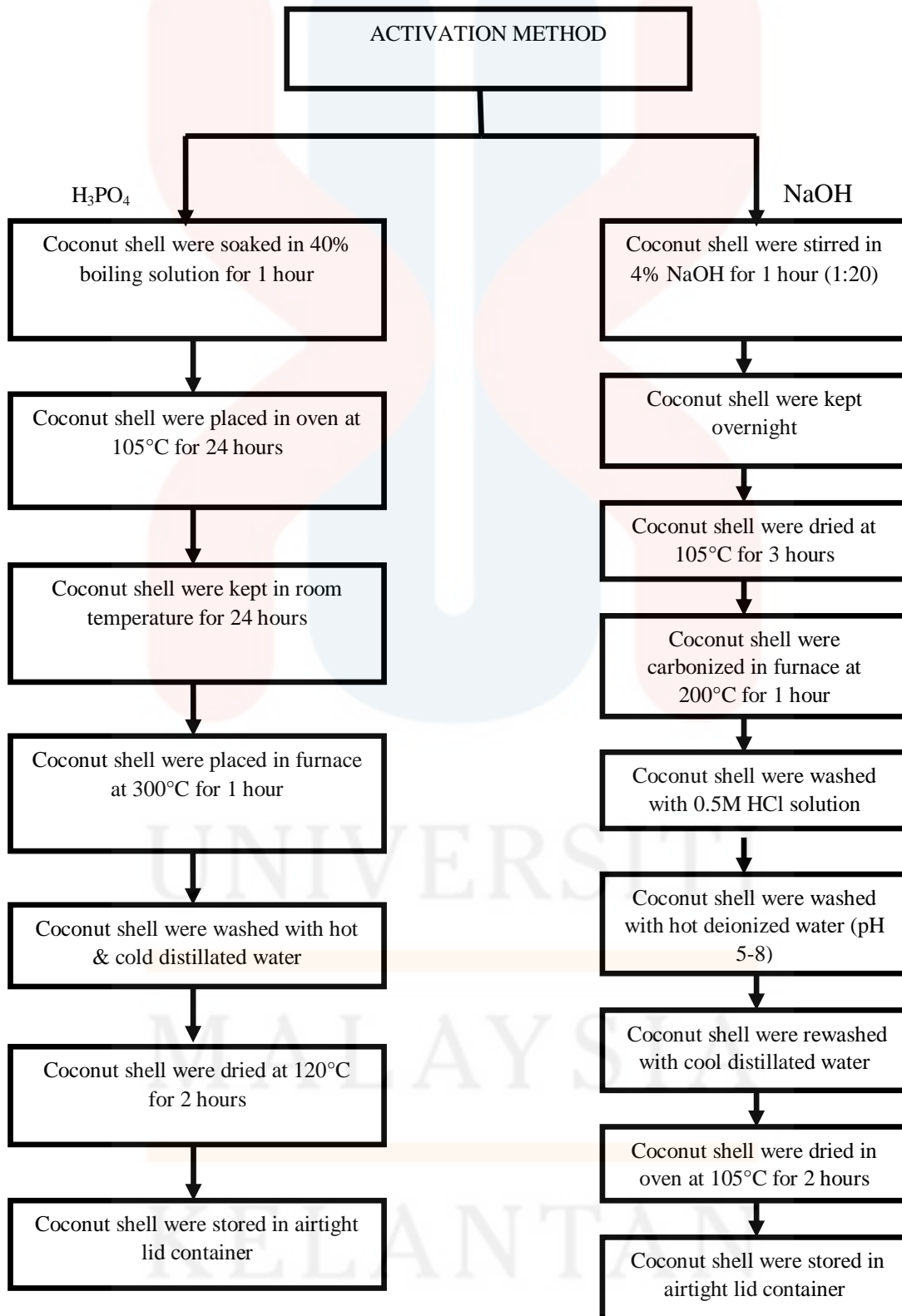


Figure 3.2 and 3.3: Activation Method by using H₃PO₄ and NaOH

3.5 Preparation of Adsorbate

3.5.1 Adsorbate

Methylene blue was supplied by Sigma- Aldrich (M) Sdn Bhd, Malaysia and use as received. According to Reddy & Nirmala, (2013), MB has a chemical formula of $C_{16}H_{18}ClN_3S_1$ as shown in Figure 3.4 with the molecular weight 319.85 g/mol and the wavelength of maximum MB is 668 nm (Geçgel *et al.*, 2013). Stock solution of 100 mg / L was prepared by dissolving accurately weighed quantity of sample of dye in distilled water. The experimental solution was obtained by diluting the stock solution to the designed initial dye concentration (Reddy & Nirmala, 2013).

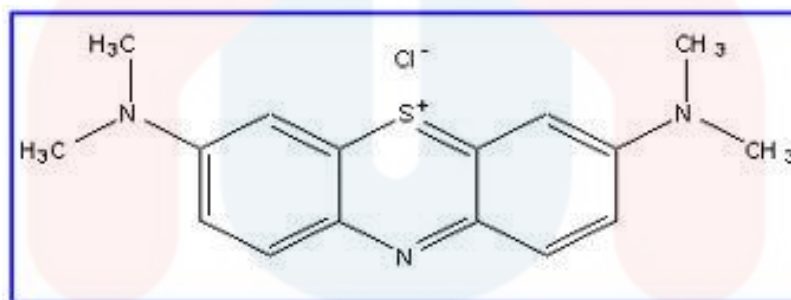


Figure 3.4: Chemical formula of MB

The MB powder was weighed by using a weighing balance for 0.15 g .The 0.15 g of MB and 20 ml of distilled water was poured into the 250 ml beaker. Then, the solution was stirred immediately by using glass rod to ensure the dyes are fully dissolved in the distilled water. Afterward, the solution of MB mixed with distilled water was poured again into the 100 ml volumetric flask and keep on adding distilled water to the volumetric flask until reached the mark. Next, the dye solution was filtered by using 0.45 μ m Whatman filter paper in order to filter the pure solution.

Later, the 5 ml of dye solution was pipette in 50 ml volumetric flask together mixing with 1500 ml of distilled water. Lastly, the prepared dye was stored in the 1500 ml polypropylene bottle and labeled as shown in Figure 3.5.

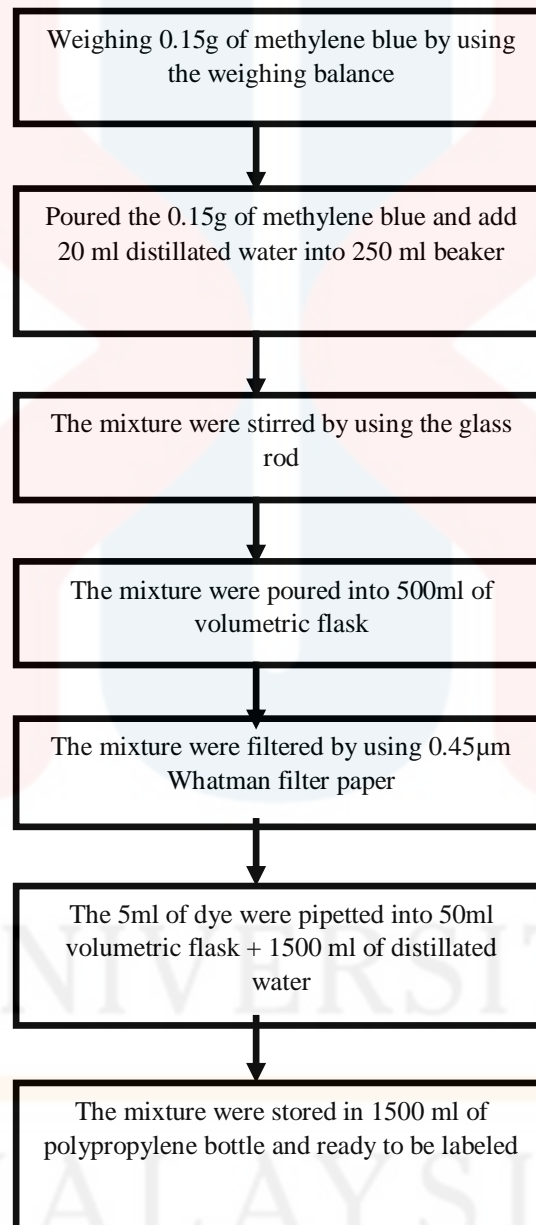


Figure 3.5: Preparation of Methylene Blue

3.6 Measurement of Decolorization of Dyes

In this experiment, the measurement of decolorization of dyes separated into two techniques that were by batch study method and dialysis tube. The different ways of decolorization method influences the adsorption rate of dyes. The decolorization of dyes can be influenced by many factors such as dosage of adsorbent, activation time, activation temperature, pH of activation and impregnation ratio.

3.6.1 Batch Study

The 1, 2, 3, 4 and 5g of AC was weighed by using weighing balance to get the precise mass. The dosages of AC were distributed into 50 ml of 10 centrifuge tubes contain of 50mg/ml of MB. Then, the mixture of MB and AC were put in 250 ml beaker for easy use when placed in shaker. The mixture were stirred for 2 hours at 150 rpm (Selvanathan & Subki, 2015) by using the shaker. Then, the mixture was left in room temperature for 24 hours for complete Adsorbance. After that, the treated mixture was filtered with 0.45 μ m by using the Whatman filter paper to separate between the AC and MB solution. The filtered mixture was dropped about 1.5 ml into cuvette and ready to be tested with spectrophotometer UV-VIS Thermoscientific Genesys 20 for wavelength 668 nm (Hua *et al.*, 2016).

3.6.2 Dialysis Tube

Another method to observe the adsorption of MB in aqueous solution is by using the dialysis tube. The dialysis tube was given about 10 meters and was cut into 10 parts and every part was divided in range 8-10 cm. The AC was weighed for 1, 2, 3, 4, and 5g and ready to be filled into the dialysis tube. The AC were filled into the dialysis tube and tied with the thread to ensure that the AC did not flow out from the dialysis tube. The 1, 2, 3, 4 and 5g of AC were immersed in 50 mg/ml of MB with different concentration and continued stirring. Then, the dialysis tubes were left in room temperature for 24 hours and ready to be observed. The filtered mixture was dropped about 1.5 ml into cuvette and ready was tested with spectrophotometer UV VIS Thermoscientific Genesys 20 as shown in Figure 3.6.

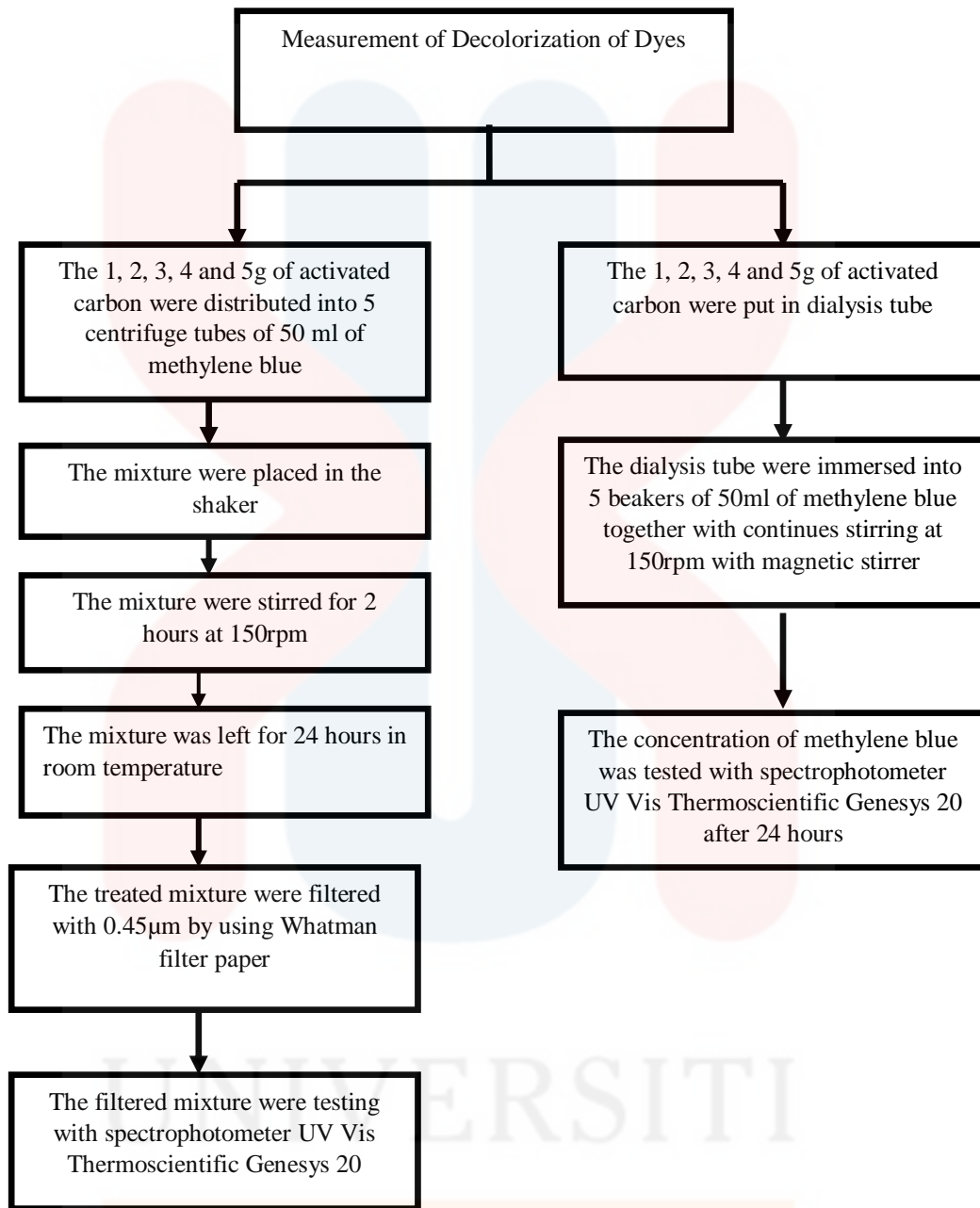


Figure 3.6: Measurement of Decolorization of Dyes

3.7 Kinetic Study

The CSAC was weighed about 5.0 g by using the weighing balance for both 50 and 100 ml of 100 mg/L of MB. Then, the 5.0 g of CSAC were filled into the dialysis tube and tied up both end of dialysis. The MB solution for 100 mg /L was immersed into two 250 ml beakers for 50 and 100 ml of 100 ml/L of MB. The MB solution and 5.0 g of CSAC were placed on the hot plate stirring together with magnetic stirrer. The decolorized MB about 1.5 ml was dropped into the cuvette. Lastly, the decolorization of MB was observed by using UV-VIS Spectrophotometer for every one hour for 6 hours as shown in Figure 3.7. The general processes of obtaining the percentages of MB shown in Figure 3.8 and 3.9.

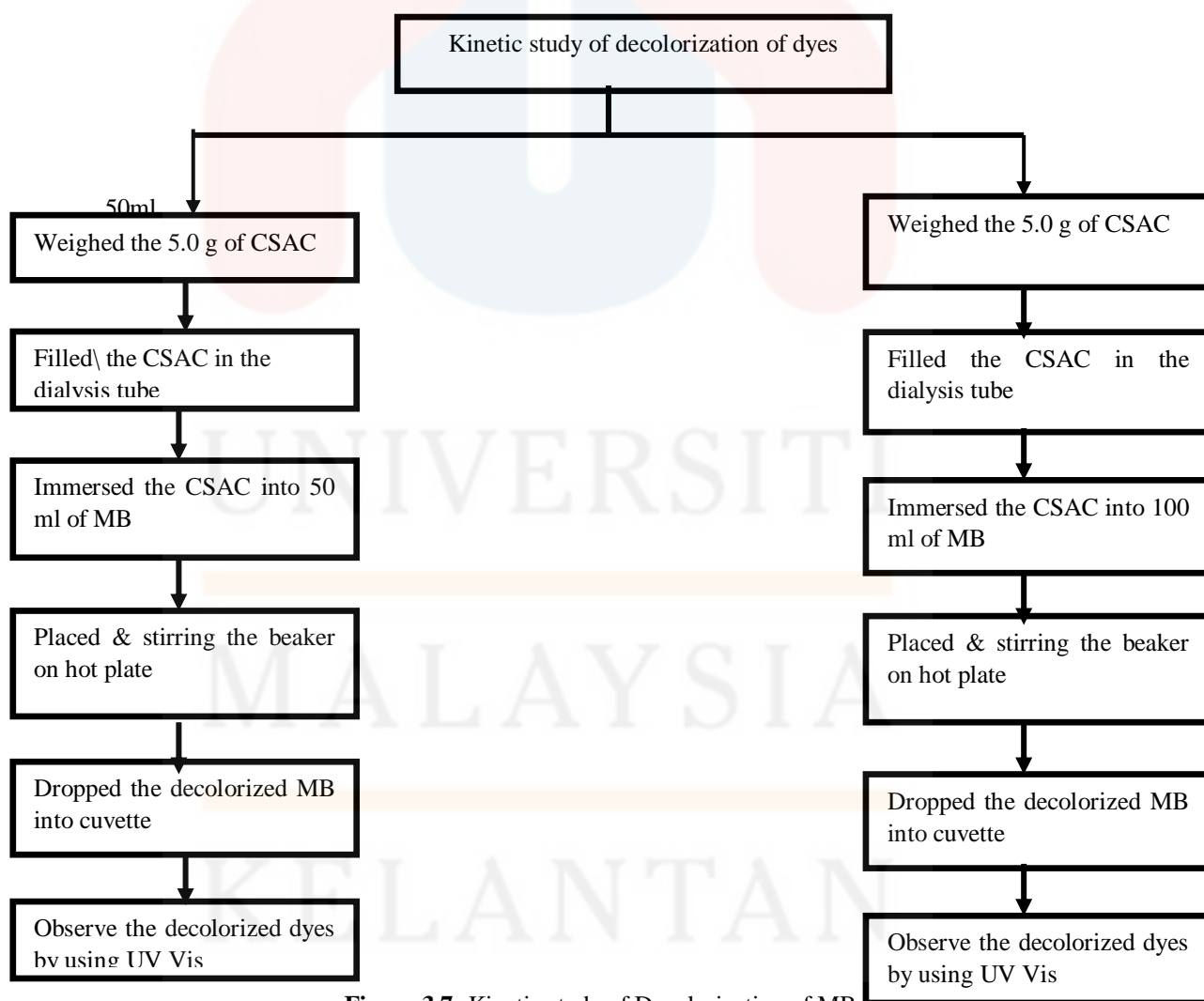


Figure 3.7: Kinetic study of Decolorization of MB

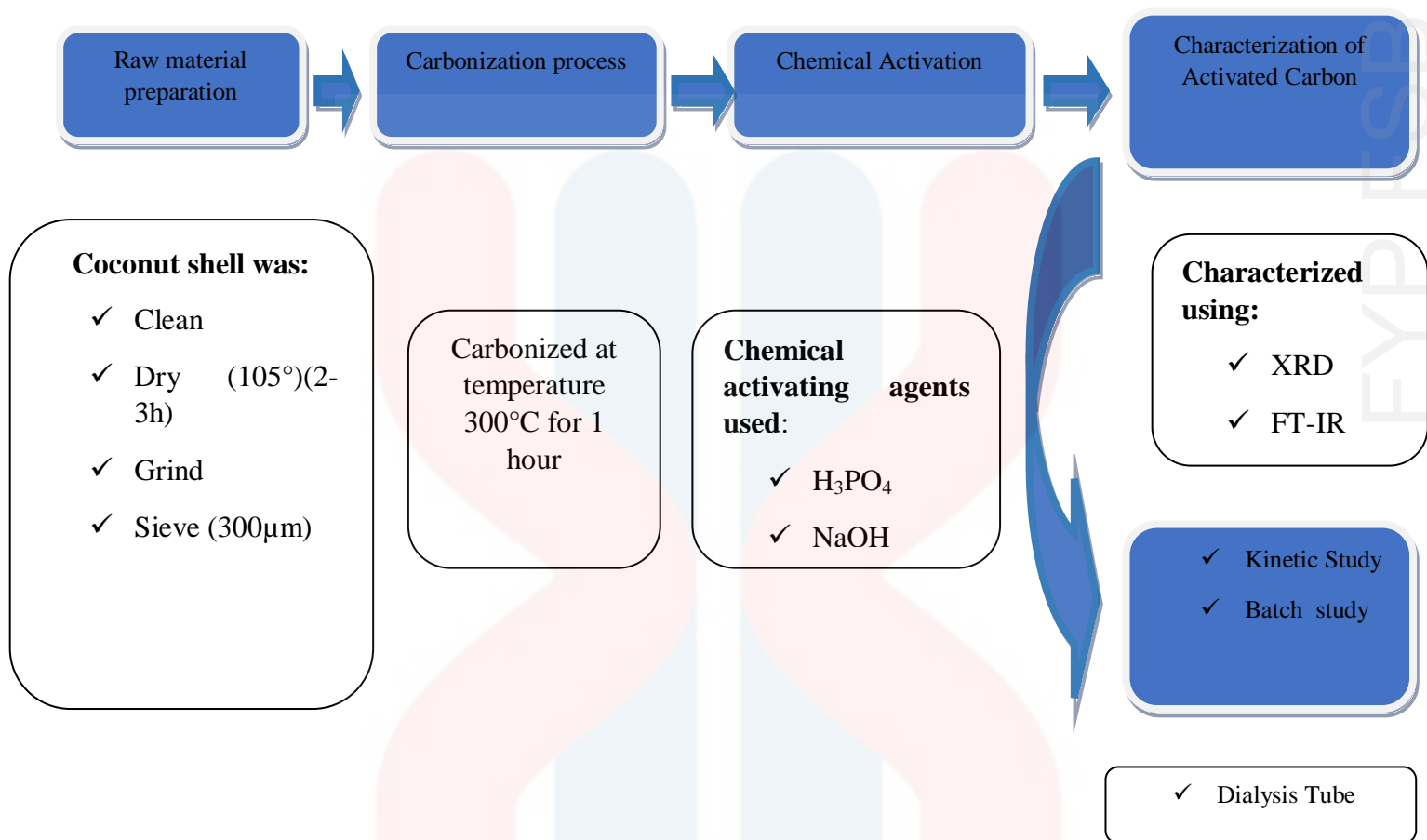


Figure 3.8: General process of obtaining the percentage removal of methylene blue

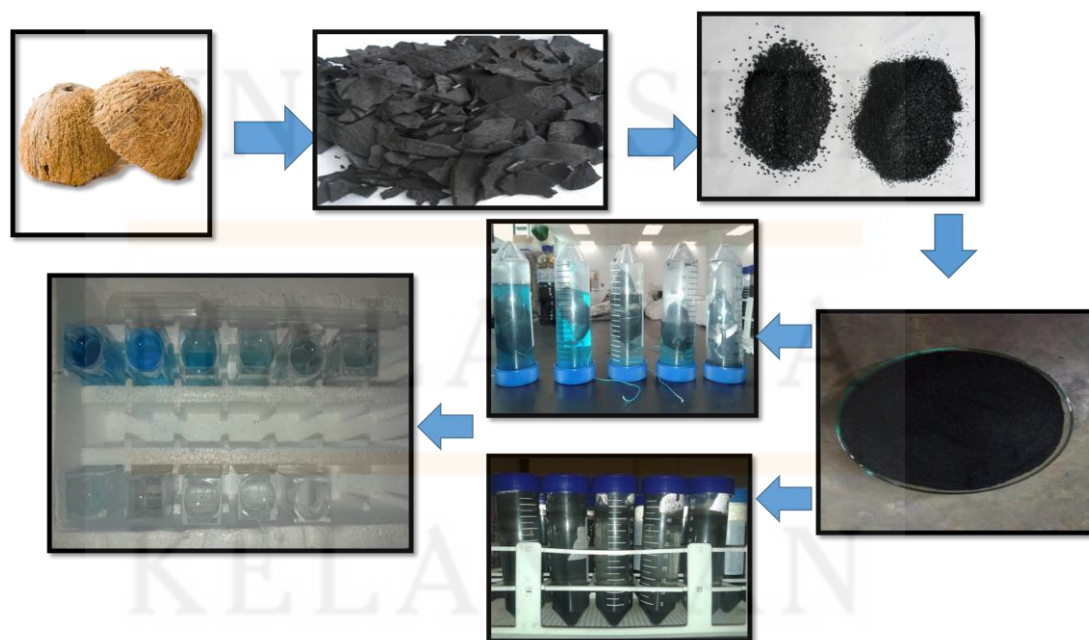


Figure 3.9: Schematic diagram of obtaining the percentage removal of methylene blue

3.8 Characterization of Activated Carbon

The characterization of AC was determined using several machines composed of X-ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR) and spectrophotometer UV VIS Thermoscientific Genesys 20.

3.8.1 X-ray Diffraction

The X-ray diffraction (XRD) was used to determine natural structure of AC either crystallinity or amorphous nature. The XRD analysis was performed by Bruker D2 Phase using Cu-K α ($\lambda=0.154060\text{\AA}$) radiation source operating under a voltage of 40 kV and a current of 25mA. The diffraction angle (2θ) was varied from 10° to 90° . The X-Ray diffraction patterns were collected with a scan rate of 5.000 $1/\text{min}$ in 25° at room temperature. DIFFRAC.EVA was used for qualitative analysis.

3.8.2 Fourier Transformation Infra-Red

The infrared spectroscopy (FT-IR) is a conventional method for structural analyses of organic compound. FT-IR is a technique which is used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. The FT-IR used for detecting functional groups, to identify the structure of crystallized phases and to observe possible structural changes after adsorption. FT-IR can also give useful information on the types and intensities of surface functional group (Gao *et al.*, 2016). The FT-IR spectra were recorded from 4000 to 400 cm^{-1} at a resolution of 4 cm^{-1} using diffuse reflectance infrared Fourier transform spectroscopy (DRIFTS). The three samples that have been prepared were Raw CS, CSC1, and CSAC2.

3.8.3 UV-Vis Spectrophotometer

The UV-Vis Spectrophotometer obeys the Beer-Lambert law which states that, when a beam of monochromatic light is passed through a solution of an absorbing substance the rate of decrease of intensity of radiation with thickness of the absorbing solution is proportional to the incident radiation as well as the concentration of the solution. The maximum wavelength of adsorbance for methylene blue was 668 nm (Selvanathan & Subki, 2015). The UV-Vis spectrophotometer is thermoscientific Genesys 20.

CHAPTER 4

4.0 RESULT & DISCUSSION

4.1 Overview

In this chapter, the CSAC was characterized by using X-Ray Diffraction (XRD) for phase identification and Fourier transform infrared spectroscopy (FTIR) for determine the functional group of CSAC. The UV-Vis was applied to determine the absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region.

4.2 Phase Identification of Activated Carbon

The phase identification that was X-Ray Diffraction patterns of the CSAC including 3 samples that were Raw CS, CSAC1 and CSAC2. The samples were analyzed by XRD and the results are shown in Figure 4.1. The diffraction pattern of CSAC with or without chemical activating agents such as H_3PO_4 and NaOH were also presented for comparison.

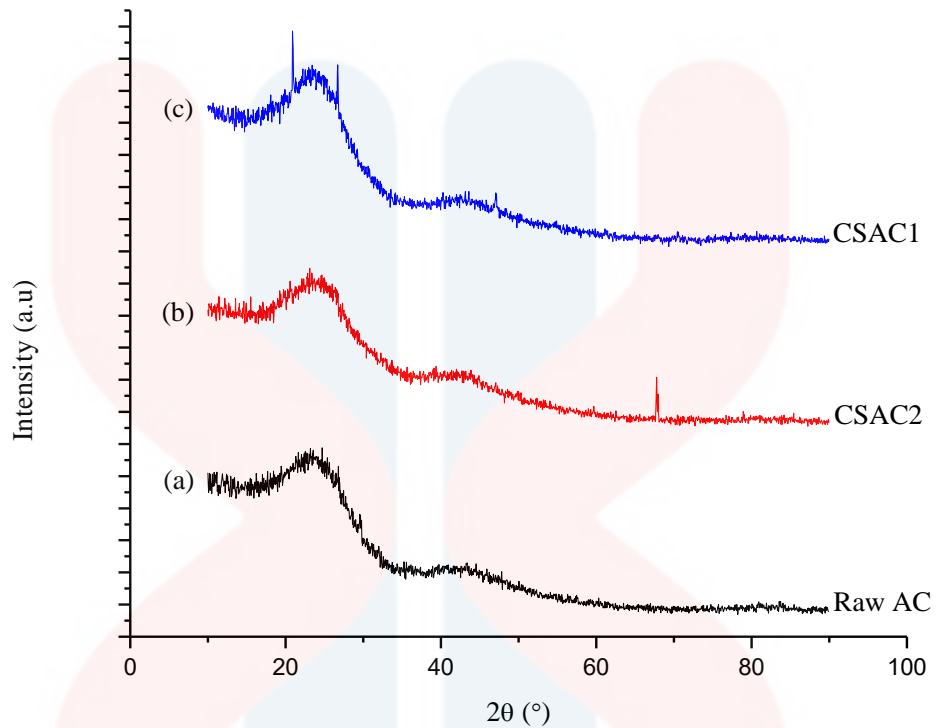


Figure 4.1 XRD profile of CSAC

● CSAC1 ● CSAC2 ● Raw AC

Figure 4.2 Raw AC (a), CSAC2 (b) and CSAC1 (c) illustrated the X-Ray Diffraction (XRD) pattern of CSAC without activated with a chemical activation. Appearance of broad diffraction background and an absence of sharp peak indicating a predominantly amorphous structure which is a typical characteristic of activated carbon (Shamsuddin *et al.*, 2016).

From the above graph, there were three sharp peaks that have been obtained at angle 25° , 45° and 55° respectively revealed crystalline carbonaceous structure. The percentage of crystallinity for raw CS is 32.6 % with percentages of amorphous is 67.4 %. The sharp peak is produced due to better layer alignment which is the characteristics of a crystalline structure. According to Das *et al.*, (2015), absence of

sharp peak is activated carbon suggests it is predominantly amorphous structure which is advantageous property for well defined adsorbents.

The CSAC2 activated with NaOH (b) the crystalline peak started at 65° followed by three sharp peaks at angle 25° , 45° and 55° for amorphous peak. The percentage of crystallinity is 29.4 % with percentages of amorphous is 70.6 %.

While, the CSAC1 activated with H_3PO_4 (c) revealed the highest crystalline peak started at 20° , 30° and 50° followed by three sharp peak that are 17° , 22° and 18° at angle for amorphous peak. The percentage of crystallinity is 35.2 % with amorphous peak is 64.8 %. According to Shamsuddin et al., (2016), the crystallinity recorded by CSAC is 8.19 % with 91.81 % amorphous. The previous study applied that oil palm empty fruit bunch (EFBAC), bamboo stem (BSAC) and coconut shell (CSAC) as activated carbon and the X- Ray Diffraction (XRD) analyzed revealed all the synthesized AC in the form of high amorphous state with low crystallinity (Shamsuddin *et al.*, 2016).

4.3 Functional group analysis of the AC

The infrared spectroscopy (FT-IR) spectrum of coconut shell activated carbon were obtained in order to identify the functional groups like -OH, -CO, -CHO, N-H, -CONH, -C = C and -COOH presented in them that can be involved in bonding with MB during adsorption rate uptake (Latinwo *et al.*, 2015). According to Njoku *et al.*, (2014), The spectroscopy revealed the active functional groups on the surface of the prepared CSAC before and after activation process.

The FT-IR study indicates that the interaction of MB and CSAC surface is via the nitrogen atoms of the adsorbate (MB) and oxygen groups in the adsorbent (CSAC). Figure 4.2 (a) Raw AC, (b) CSAC1, (c) CSAC2 and (d) CSAC3 were analyzed by using the FT-IR. The spectra's vibrations of Raw CS (a) characteristics indicates the aliphatic amino acids salts at band 2855 to 2955 cm^{-1} that assigned to methyl (-CH₃), at band 2970 to 2950 cm^{-1} and also at band 1500 to 1600 cm^{-1} . It is also indicates the inorganic nitrate ions at band 1300 to 1450 cm^{-1} that revealed the present of nitrate ion. The chemical properties of aliphatic amino acid are containing amino group and carboxylic acid group and the physical properties are they are non volatile crystalline solids which decompose on melting.

For the CSAC1 (b) characteristics indicates the aliphatic carboxylic acid salts assigned to methyl C-H bend between band 1450 to 1500 cm^{-1} and 1590 to 1600 cm^{-1} . It is also indicates the present of inorganic nitrate at band 1750 to 1800 cm^{-1} and 1300 to 1450 cm^{-1} for nitrate and carbonate ion. The physical properties of aliphatic amino acid are made up of non- volatile crystalline solids which decompose on melting, insoluble in non-polar solvents but soluble in water and show highly soluble dipole behaviour in aqueous solutions.

For CSAC2 (c) characteristics indicates the aliphatic carboxylic acid salts assigned to carboxylate or carboxylic acid salts at band 1560 to 1600 cm^{-1} and at band 1450 to 1500 cm^{-1} that show the suspected functional group is methyl C-H bend ($-\text{CH}_3$). Carboxylic acid salts show a strong, characteristic asymmetric stretching absorption from the CO_2^- group in the 1650 to 1550 cm^{-1} region. For acid salts with a strongly electronegativity group the asymmetric stretching adsorption will shift to higher frequencies (around 1690 cm^{-1}). The chemical properties of carboxylic acid salts are more acidic than the very weak organic acids (alcohols).

For the CSAC3 (CSAC after adsorption) characteristics indicates the aliphatic amino hydrocarbons for band 2850 to 2950 cm^{-1} assigned for $-\text{CH}_3$ and $-\text{CH}_2$ stretching absorptions (C-H stretch). The CH_3 asymmetric stretching vibration occurs at 2975 to 2950 cm^{-1} while the CH_2 adsorption occurs at about 2930 cm^{-1} . The symmetric CH_3 vibration occurs at 2885 to 2865 cm^{-1} while the CH_2 absorption occurs at about 2870 to 2840 cm^{-1} . The band is at 1580 to 1650 cm^{-1} for secondary amine that is NH deformations ($>\text{N-H}$ bend). Amines are an important class of organic compounds because they show appreciable basicity and 1500 to 1550 cm^{-1} for aromatic nitro compounds. The spectral expectation for aromatic nitro compounds is when a hydroxyl group is situated in the ortho or para position. It is also indicates the present of inorganic nitrate at band 1300 to 1450 cm^{-1} for phenol or tertiary alcohol (O-H bend). There are three characteristic bands of inorganic nitrates for spectrum's band at 1780 cm^{-1} , 1380 cm^{-1} and 830 cm^{-1} . The 1380 cm^{-1} band is the strongest and is relatively broad while the other two absorptions are weaker and narrow.

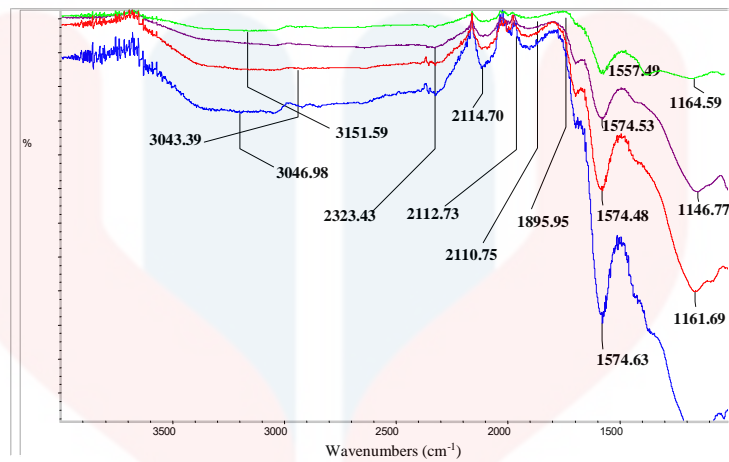


Figure 4.3 FT-IR profile of CSAC

● Raw CS ● CSAC1 ● CSAC2 ● CSAC3

4.4 The Effect of Chemical Activation on Surface Area and Pore Volume

Nowadays, AC with very high surface area has been obtained by chemical activation and extensively used for liquid or gas pollutants removal. An activation method by chemical activation resulting in high surface area and appropriate porous structure. According to Ünner *et al.* (2015), activation based on ZnCl_2 was the highest because efficient in producing greater pore size but it also had a side effect that may be dangerous to the ecosystem.

Generally, chemical such as potassium hydroxide (KOH), sodium hydroxide (NaOH), phosphoric acid (H_3PO_4) were chosen as an activating agent in carbon activation process. However in this research sodium hydroxide (NaOH) was selected as an activating agent.

According to Selvanathan & Subki, (2015), sodium hydroxide acts as etching materials that promotes defects in the structure of the carbon materials in which this causes and improves the porosity of carbon materials. According to Sperlich et al., (2016) chemical activation of biomass with KOH or NaOH produce carbon with high surface area ($>1000 \text{ m}^2/\text{g}$) and the activation costs could be reduced by using NaOH rather than KOH.

The NaOH activation shows better adsorption efficiency compared to KOH activation because the cation in the hydroxide in this case which is sodium ion (Na^+) is responsible to determine the size and number of pores in activated carbon. Sodium lies above potassium in the periodic table which also revealed the size of sodium ion (Na^+) is far smaller compared to potassium ion (K^+). Due to the size of Na^+ is smaller that potassium ions enable the Na^+ to penetrate more deeply into the carbon structure and develop more higher number of smaller and uniformly (Selvanathan & Subki, 2015).

In this research, the activation based on phosphoric acid showed the greater efficiency compared to base activation. When the AC activated with acid activation immersed in the methylene blue, the adsorption rate was higher compared to the base activation. According to Akar et al. (2009), at strongly acid condition, the surface of the biosorbent will get positively charge thus provide attractive forces for negatively charged dyes molecules. When the pH of alkaline solution was increase, the number of negatively charged sites also increased. The lower biosorption capacity of biomass recorded at alkaline pH was due to the competition between excess hydroxyl ions (OH^-) and negatively charged dyes ions in filling the binding sites.

According to Selvanathan & Subki, (2015), revealed that NaOH are strong base in nature dissociates completely when dissolves in water to produce hydroxide ions (-OH). This will also increase the actives sites in activated carbon produced by base activation. Despite the facts discussed before, the adsorption of methylene blue for activated carbon activated from H_3PO_4 show great adsorption compared to NaOH activation process.

According to Shamsuddin *et al.* (2016), pore development in AC during pyrolysis was also essential thus it would improves the surface area and pore volume of the AC by promoting the diffusion of H_3PO_4 molecules into the pores and thereby increasing the H_3PO_4 carbon reaction via acid hydrolysis processes which would create more pores. The glycosidic bond in hemicellulose and cellulose will be hydrolyzed by H_3PO_4 continued with the cleavage of aryl bonding in lignin.

According to Yorgun & Yildiz, (2014), H_3PO_4 was found to be more effective for developing microporous structured in the activated carbon. The main function of H_3PO_4 as activating agents are promoting the pyrolytic decomposition of the initial material, developing the formation of the cross-linked structures and allowing the development of both micropores and mesopores in the activated carbon.

4.5 Effect on Amount of Activated Carbon (g)

Removal efficiency of CSAC generally improved with increasing dose up to certain value and then there is no further increase of adsorption rate (Babel & Kurniawan, 2004). According to Taylor *et al.* (2012) the increasing in dye adsorption was due to the increased in availability of dye binding sites resulting from an increase in adsorbent. At some point the dye are more removed from the solution which is the amount of dyes adsorbed onto the adsorbent was in the state of dynamic equilibrium with the amount of dye desorbed from the adsorbent.

Generally adsorption takes place because all molecules exert forces to bind to each other. Activated carbon adsorbs organic material because the attractive forces between the CSAC surface (non-polar) and the contaminant or adsorbate (MB) which is also non-polar are stronger than attraction forces that preventing the contaminant dissolved in water that is polar (Adedayo *et al.*, 2012). The adsorption rate increase with the increase of adsorbent usage for 1, 2, 3, 4 and 5 g of CSAC. When the active sites on the adsorbent are fully occupied with adsorbate (MB) it is difficult for other adsorbate to bind to the active site (Azmi *et al.*, 2015). The adsorptive forces are weaker and cannot occur unless the organic molecules are closed to the carbon surface.

The effect of different amount of coconut shell activated carbon (CSAC) on adsorption of methylene blue was conducted and understood by plotting the graph between Adsorbance versus amount of CSAC. Initial methylene blue concentration was set to be 100 mg / L and different amount of CSAC as adsorbent were set up for 1, 2, 3, 4 and 5 g / L as shown in Figure 4.2. Figure 4.2 showed that the adsorption rate of methylene blue was directly proportional to the amount of CSAC.

According to Seey. (2012) the removal of dyes increased with the increase of amount of CSAC up to maximum ($> 90\%$), after which an increase in adsorbent dosage does not further improve the dyes removal, implying that a complete dyes removal could not be achieved even though using large amount of CSAC. Lowest adsorption rate uptake is by $1.0\text{g} / \text{L}$ of CSAC while the highest adsorption rate uptake is by using $5.0\text{g} / \text{L}$ of CSAC. It is due to the too much adsorbent was added into the dyes solution, the transportation of dyes ions to the active adsorption sites will be limited as well, hence reduced the adsorption efficiency (Seey, 2012).

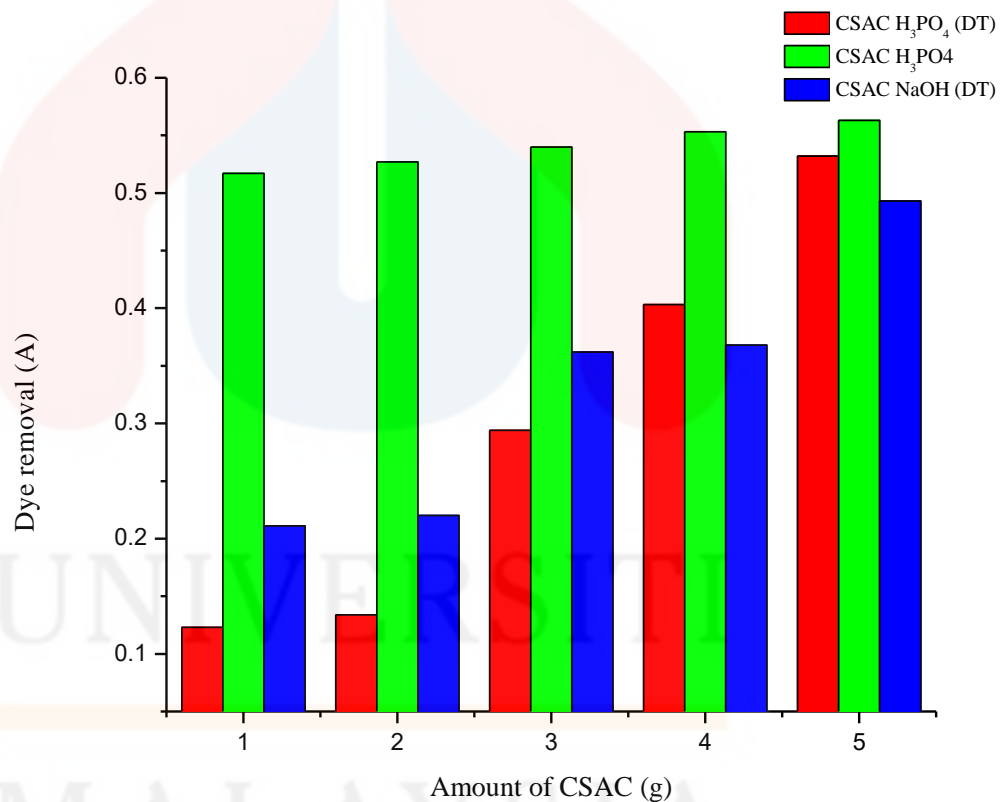


Figure 4.2: Dye removal by CSAC

4.6 Kinetic Study

The experiment to investigate the percentages of removal of dye was conducted for 5 g / L of CSAC in 100 ml of 100 mg/L of MB and 100 ml of 100 mg/L of MB after 6 hours using magnetic stirrer and hot plate at 25° in room temperature. According to the experiment that conducted, it is postulated that the 5 g of CSAC show the highest adsorption rate. According to Arau, (2005), the percentages of dye removal was increase with the increment of adsorbance dosage.

The dialysis tube filled with 5 g of CSAC show far better adsorption rate uptake compare to the normally released CSAC method. The reading was taken every 1 hour for 6 hours. The percentages of decolorization of MB increase with the increment of time taken. The percentages of decolorization of 5 g in 100 ml of 100 mg/L of MB increase from 27.58 % to 60.05 % as the adsorbent dosage increased from 1 g to 5 g after reached equilibrium time as shown in figure 4.3. While the percentages of decolorization of 5.0 g in 100 ml of 100 mg/L of MB show increment from 43.32 % to 71.08 % which show that the adsorption rate was better compare to 100 ml of 100 mg/L MB for the same increment of adsorbent dosage. This is due to increased in adsorbent dosage attributed to increase in surface area and availability of adsorption binding site (Arau, 2005). The 5g of CSAC was investigated in this kinetic study because that amount shows the excellent rate of dye removal. The concentration of dye that was recorded was positively decreased and this also revealed that this can be the best method to control wastewater pollution.

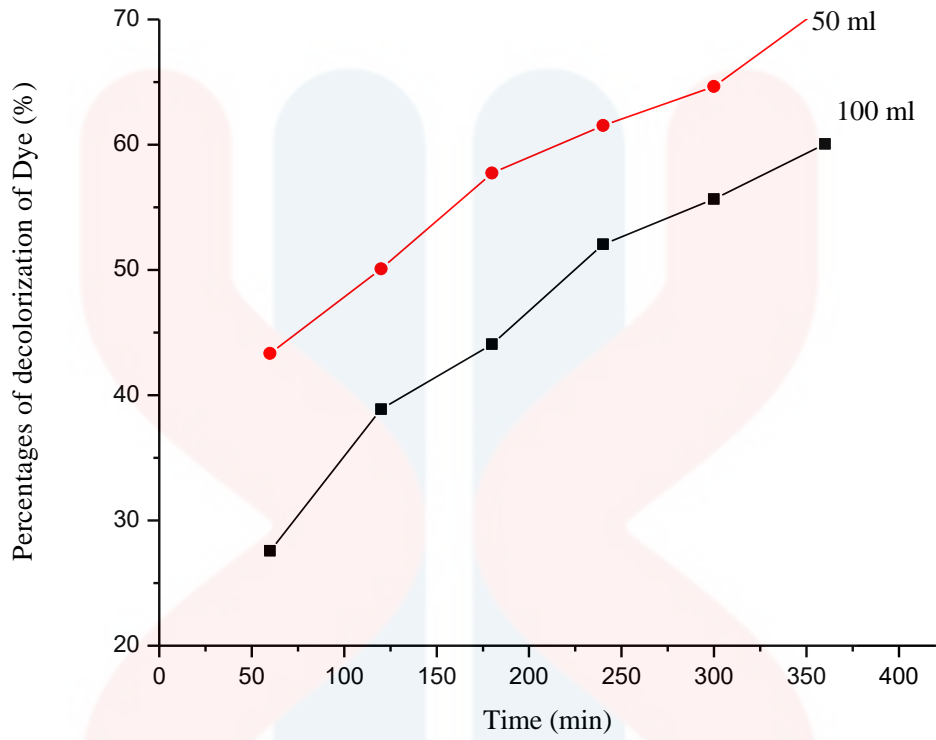


Figure 4.3: Comparison of percentages of decolorization of MB (%)

CHAPTER 5

5.1 CONCLUSION

This study showed that *COCOS NUCIFERA L.* (coconut) could be used as an adsorbent to remove MB dye from aqueous solution because it is inexpensive raw materials and performing a suitable production method may be useful for producing low cost activated carbon. Moreover, the removal of MB by adsorption utilizing activated carbon derived from coconut shell waste to be useful in controlling the water pollution. In this study, coconut shell, CS undergoes acid (H_3PO_4) and base (NaOH) chemical activation.

CS activated with H_3PO_4 show better adsorption compared to activation process with NaOH. It is because the phosphoric acid acts as modifying agents that increasing the porosity and giving rise to surface groups of acidic character. Pore structure and surface chemistry of the raw CS are altered after being carbonized and activated with chemical activating agents. Chemical activation can also produces highly active carbon, which presents a wider mesopores and macropores. Coconut shell carbons have several advantages over carbons made from other materials

The XRD show the presence of significantly different peak frequency of CSAC before and after activation process. The XRD results confirmed that CSAC showed less crystallinity with broadness peak due to amorphous structure. The FT-IR also showed the presence of different peak frequencies before and after chemical activation process.

The adsorption process depends on some factors and one of them is the adsorbent dosage. The percentage of decolorization of MB increase with the increment of adsorbent dosage. When 5.0 g of CSAC released to the MB the percentages of dye removal increase compare to lower amount of CSAC. But, the adsorption rate will reach at saturation point where the dyes molecule will completely bind to the pore of CSAC and the dye cannot longer be adsorbed by CSAC.

5.2 Recommendation

A) The raw material used in this experiment was coconut shell. In wastewater treatment the coconut husk is more preferred compared to coconut shell because it showed an excellent in flammability and high in filter content. However, the coconut husk proved to be a useful combustion medium to improve the carbonization of the coconut shells (Cobb, 2012). So, it is better to use coconut husk rather than coconut shell especially in removing dyes.

B) For the kinetic study, the time taken was important in determine the adsorption rate of methylene blue removal. The higher the time taken, the excellent the adsorption uptake undergoing until the equilibrium state was reached. The time taken for kinetic study in this experiment was only 6 hours and it is proved that to the last 6 hours the percentages removal of dyes at maximum peak compared to lower time taken.

C) In the activation process the coconut shell was activated using furnace at 300°C. It was revealed that activated using microwave show far better result in pore size opening. Therefore, microwave treatment heats the coal in a very short time. On

the other hand, when the water was suddenly evaporated by the microwave, the crack occurred on the activated carbon structure causing the increase in the surface area.

REFERENCES

- A, U. I., Abduraheem, G., Bala, S., & Muhammad, S. (2015). International Biodeterioration & Biodegradation Kinetics , equilibrium and thermodynamics studies of C . I . Reactive Blue 19 dye adsorption on coconut shell based activated carbon. *International Biodeterioration & Biodegradation*, 1–9.
- Adedayo, I., Anthony, I., & Henry, B. (2012). Comparison of the Adsorptive Capacity of Raw Materials in Making Activated Carbon Filter for Purification of Polluted Water for Drinking, 2(9), 754–760.
- Akar, S. T., Özcan, A. S., Akar, T., Özcan, A., & Kaynak, Z. (2009). Biosorption of a reactive textile dye from aqueous solutions utilizing an agro-waste. *Desalination*, 249(2), 757–761.
- Ansari, R. (2009). Activated Charcoal: Preparation , characterization and Applications : A review article, 1(4), 859–864.
- Arau, J. (2005). Dye Removal from Aqueous Solution by using Adsorption on Treated Sugarcane Bagasse S . Saiful Azhar , 2 A . Ghaniey Liew , 1 D . Suhardy , 1 K . Farizul Hafiz , 1 M . D . Irfan Hatim School of Materials Engineering , Northern Malaysia University College of Engineering (KUKUM) Chemical and Environmental Engineering Department , Faculty of Engineering, 2(11), 1499–1503.
- Asma, I. W., Rafidah, J., Puad, E., & Shaharuddin, H. (2011). Production of activated carbon from industrial bamboo wastes, 23(4), 417–424.
- Avelar, N. V., Rezende, A. A. P., Carneiro, A. de C. O., & Silva, C. M. (2016). Evaluation of briquettes made from textile industry solid waste. *Renewable Energy*, 91, 417–424.
- Azmi, N. A. I., Zainudin, N. F., Ali, U. F., & Senusi, F. (2015). Adsorption kinetics on basic red 46 removal using cerbera odollam activated carbon, 82–91.
- Babel, S., & Kurniawan, T. A. (2004). Cr (VI) removal from synthetic wastewater using coconut shell charcoal and commercial activated carbon modified with oxidizing agents and / or chitosan, 54, 951–967.
- Boualem, T., Debab, A., Yuso, A. M. De, & Izquierdo, M. T. (2014). Activated carbons obtained from sewage sludge by chemical activation: Gas-phase environmental applications. *Journal of Environmental Management*, 140, 145–151.

- Byamba-Ochir, N., Shim, W. G., Balathanigaimani, M. S., & Moona, H. (2016). Highly Porous Activated Carbons Prepared from Carbon Rich Mongolian Anthracite by Direct NaOH Activation. *Applied Surface Science*.
- Carlos J. Duran-Valle. (2015). Waste water treatment activated carbon and other methods. In A. Brooks (Ed.), (p. 92). Callisto References.
- Chandra, T. C., Mirna, M. M., Sunarso, J., Sudaryanto, Y., & Ismadji, S. (2009). Activated carbon from durian shell: Preparation and characterization. *Journal of the Taiwan Institute of Chemical Engineers*, 40, 457–462.
- Chang, C., Chang, C., & Tsai, W. (2000). Effects of Burn-off and Activation Temperature on Preparation of Activated Carbon from Corn Cob Agrowaste by CO₂ and Steam. *Journal of Colloid and Interface Science*, 232(1), 45–49.
- Chen, C. X., Huang, B., Li, T., & Wu, G. F. (2012). Preparation of phosphoric acid activated carbon from sugarcane bagasse by mechanochemical processing. *BioResources*, 7(4), 5109–5116.
- Cobb, A. (2012). Low-Tech Coconut Shell Activated Charcoal Production, 7(1), 93–104.
- Das, D., Samal, D. P., & Bc, M. (2015). Preparation of Activated Carbon from Green Coconut Shell and its Chemical Engineering & Process Technology, 6(5). <http://doi.org/10.4172/2157-7048.1000248>
- Demiral, H. (2008). Adsorption of Textile Dye onto Activated Carbon Prepared from Industrial, 3(5), 381–389.
- Gao, Y., Xu, S., Yue, Q., Wu, Y., & Gao, B. (2016). Chemical preparation of crab shell-based activated carbon with superior adsorption performance for dye removal from wastewater. *Journal of the Taiwan Institute of Chemical Engineers*, 0, 1–9.
- Geçgel, Ü., Özcan, G., & Gürpınar, G. Ç. (2013). Removal of Methylene Blue from Aqueous Solution by Activated Carbon Prepared from Pea Shells (*Pisum sativum*), 2013.
- Georgin, J., Dotto, G. L., Mazutti, M. A., & Foletto, E. L. (2016). Preparation of activated carbon from peanut shell by conventional pyrolysis and microwave irradiation-pyrolysis to remove organic dyes from aqueous solutions. *Journal of Environmental Chemical Engineering*, 4(1), 266–275.
- Govind Raj, K., & Alias Joy, P. (2015). Coconut shell based activated carbon–iron oxide magnetic nanocomposite for fast and efficient removal of oil spills. *Journal of Environmental Chemical Engineering*, 3(3), 2068–2075.
- Gratuito, M. K. B., Panyathanmaporn, T., & Chumnanklang, R. (2008). Production of activated carbon from coconut shell : Optimization using response surface

methodology, 99, 4887–4895.

- Hu, E., Xinbo, W., Shang, S., Tao, X., & Jiang, S. (2015). AC SC. *Journal of Cleaner Production*.
- Hua, Y., Fengzhen, Y., & Xiaozhen, F. (2016). CODEN (USA): PCHHAX Catalytic degradation of methyl orange under visible light by, 8(1), 188–191.
- I, I. A., O, I. A., & I, B. H. (2012). Comparison of the Adsorptive Capacity of Raw Materials in Making Activated Carbon Filter for Purification of Polluted Water for Drinking, 2(9), 754–760.
- Islam, M. A., Benhouria, A., Asif, M., & Hameed, B. H. (2015). Methylene blue adsorption on factory-rejected tea activated carbon prepared by conjunction of hydrothermal carbonization and sodium hydroxide activation processes. *Journal of the Taiwan Institute of Chemical Engineers*, 52, 57–64.
- Kulkarni, S. J., Tapre, R. W., Patil, S. V., & Sawarkar, M. B. (2013). Adsorption of Phenol from Wastewater in Fluidized Bed Using Coconut Shell Activated Carbon. *Procedia Engineering*, 51(NUiCONE 2012), 300–307.
- Laginhas, C., Nabais, J. M. V., & Titirici, M. M. (2016). Activated carbons with high nitrogen content by a combination of hydrothermal carbonization with activation. *Microporous and Mesoporous Materials*, 226, 125–132. <http://doi.org/10.1016/j.micromeso.2015.12.047>
- Lam, M. A. N. K. E. E., & Zakaria, R. (2008). Production of activated carbon from sawdust using fluidized bed reactor, 2008(Icenv).
- Latinwo, G. K., Agarry, S. E., Engineering, B., & Biotechnology, C. E. (2015). Removal of Phenol from Paint Wastewater by Adsorption onto Phosphoric Acid Activated Carbon Produced from Coconut Shell: Isothermal and Kinetic Modelling Studies, 7(5), 123–138.
- Li, Y., Li, Y., Li, L., Shi, X., & Wang, Z. (2016). Preparation and analysis of activated carbon from sewage sludge and corn stalk. *Advanced Powder Technology*, 27(2), 684–691.
- Martinez, M. L., Moiraghi, L., Agnese, M., & Guzman, C. (2003). Making and some properties of activated carbon produced from agricultural residues from Argentinian. *The Journal of the Argentine Chemical Society*, 91(4), 103–108.
- Mozammel, H. M., Masahiro, O., & Sc, B. (2002). Activated charcoal from coconut shell using ZnCl₂ activation, 22, 397–400.
- Muhamad, I. (2016). Treatment of lead-contaminated water using activated carbon adsorbent from locally available papaya peel biowaste. *Journal of Cleaner*
- Njoku, V. O., Islam, M. A., Asif, M., & Hameed, B. H. (2014). Preparation of mesoporous activated carbon from coconut frond for the adsorption of

carbofuran insecticide. *Journal of Analytical and Applied Pyrolysis*, 110(1), 172–180.




- Nunes, C. a., & Guerreiro, M. C. (2011). Estimation of Surface Area and Pore Volume of Activates Carbons by Methylene Blue and Iodine Numbers. *Química Nova*, 34(3), 472–476.
- Okafor, P. C., Okon, P. U., Daniel, E. F., & Ebenso, E. E. (2012a). Adsorption Capacity of Coconut (Cocos nucifera L.) Shell for Lead, Copper, Cadmium and Arsenic from Aqueous Solutions. *International Journal of Electrochemical Science*, 7, 12354–12369.
- Okafor, P. C., Okon, P. U., Daniel, E. F., & Ebenso, E. E. (2012b). Adsorption Capacity of Coconut (Cocos nucifera L.) Shell for Lead, Copper, Cadmium and Arsenic from Aqueous Solutions, 7, 12354–12369.
- Reddy, M. C. S., & Nirmala, V. (2013). Bengal gram seed husk as an adsorbent for the removal of dyes from aqueous solutions – Equilibrium studies. *Arabian journal of chemistry*.
- Santhy, K., & Selvapathy, P. (2006). Removal of reactive dyes from wastewater by adsorption on coir pith activated carbon, 97, 1329–1336.
- Seey, T. L. (2012). Acidic and Basic Dyes Removal by Adsorption on Chemically Treated Mangrove Barks, 2(3), 270–276.
- Selvanathan, N., & Subki, N. S. (2015). Dye adsorbent by pineapple activated carbon : H₃PO₄ and NaOH activation, 10(20), 9476–9480.
- Shamsuddin, M. S., Yusoff, N. R. N., & Sulaiman, M. A. (2016). Synthesis and Characterization of Activated Carbon Produced from Kenaf Core Fiber Using H₃PO₄ Activation. *Procedia Chemistry*, 19, 558–565.
- Shankar, D. P. A. (2008). *Water Conditioning & Purification*, 1–4.
- Sharma, G. D., Tripathi, A. K., Nilsson, J. O., & Salaneck, W. R. (n.d.). Adsorption of Crystal Violet on Activated Carbon Prepared from Coal Flotation Concentrate.
- Sperlich, A., Jekel, M., Altmann, J., Rehfeld, D., & Tr, K. (2016). Combination of granular activated carbon adsorption and deep-bed filtration as a single advanced wastewater treatment step for organic micropollutant and phosphorus removal, 92, 131–139.
- Sun, Y., Li, H., Li, G., Gao, B., Yue, Q., & Li, X. (2016). Characterization and ciprofloxacin adsorption properties of activated carbons prepared from biomass wastes by H₃PO₄ activation. *Bioresource Technology*.

- Taylor, P., Bello, O. S., Ahmad, M. A., Bello, O. S., & Ahmad, M. A. (n.d.). Separation Science and Technology Coconut (*Cocos nucifera*) Shell Based Activated Carbon for the Removal of Malachite Green Dye from Aqueous Solutions Coconut (*Cocos nucifera*) Shell Based Activated Carbon for the Removal of Malachite Green Dye from Aqu, (September 2014), 37–41.
- Tonucci, M. C., Gurgel, L. V. A., & Aquino, S. F. de. (2015). Activated carbons from agricultural byproducts (pine tree and coconut shell), coal, and carbon nanotubes as adsorbents for removal of sulfamethoxazole from spiked aqueous solutions: Kinetic and thermodynamic studies. *Industrial Crops and Products*, 74, 111–121.
- Üner, O., Geçgel, Ü., & Bayrak, Y. (2015). Preparation and characterization of mesoporous activated carbons from waste watermelon rind by using the chemical activation method with zinc chloride. *Arabian Journal of Chemistry*.
- Yakout, S. M., & Sharaf El-Deen, G. (2012). Characterization of activated carbon prepared by phosphoric acid activation of olive stones. *Arabian Journal of Chemistry*.
- Yorgun, S., & Yildiz, D. (2014). Preparation and characterization of activated carbons from Paulownia wood by chemical activation with H₃PO₄. *Journal of the Taiwan Institute of Chemical Engineers*, 0, 1–10.
- Zhang, Y., Tan, Z., Liu, B., Mao, D., & Xiong, C. (2015). Coconut shell activated carbon tethered ionic liquids for continuous cycloaddition of CO₂ to epichlorohydrin in packed bed reactor. *Catalysis Communications*, 68, 73–76.
- Zhu, Y., & Kolar, P. (2014). Journal of Environmental Chemical Engineering Adsorptive removal of p -cresol using coconut shell-activated char. *Biochemical Pharmacology*, 2(4), 2050–2058. <http://doi.org/10.1016/j.jece.2014.08.022>

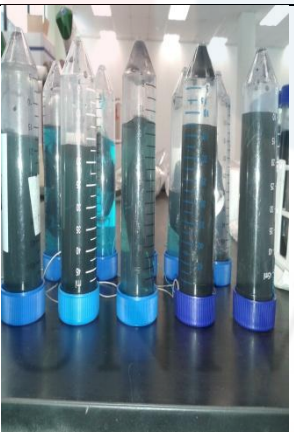


APPENDIX A

Observation of Dye Removal

(A) Acid Activation, H_3PO_4

Dialysis Tube		Normal	
Initial	Final	Initial	Final
			

(A) Base Activation, NaOH



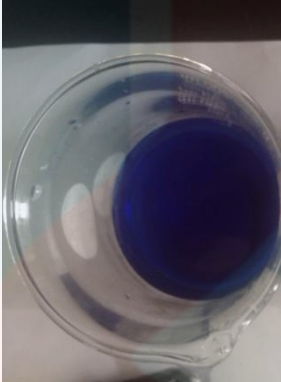
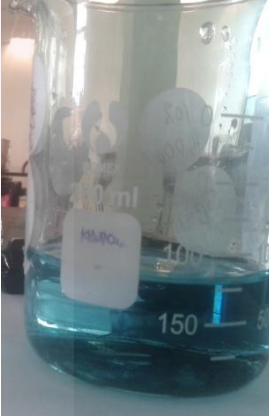
Dialysis Tube		Normal	
Initial	Final	Initial	Final
			

MALAYSIA

KELANTAN

Kinetic Study

Comparison between 50 & 100 ml of MB of 5g Dialysis Tube

50ml		100ml	
Initial	Final	Initial	Final
			

**APPENDIX B
CALCULATIONS**

Kinetic Study

Percentages (%) of decolorization

The percentage removal of dye is defined as the difference in dye concentration before and after adsorption, and was calculated by using the Equation:

$$\% \text{ Removal} = \frac{C_o - C_t}{C_o} \times 100$$

Where C_o is the adsorbance value before the dyes are treated (pure treated) with different activated carbon while C_t is the adsorbance value after the dyes are treated (treated dye).

A) 100 ml of 100 mg/L of MB

Time (h)	Percentages of decolorization (%)
1	27.575
2	38.866
3	44.084
4	52.047
5	55.645
6	60.054

B) 50 ml of 100 mg/L of MB

Time (h)	Percentages of decolorization (%)
1	43.319
2	50.068
3	57.715
4	61.539
5	64.642
6	71.076

Percentages of 50ml of MB after 24h (%)

Amount of CSAC (g)	Acid activation (H_3PO_4) (%)		Base activation (NaOH) (%)
	Dialysis Tube	Normal	Dialysis Tube
1	76.069	77.823	74.674
2	81.871	83.446	75.124
3	86.775	83.716	75.709
4	93.972	90.103	76.293
5	94.467	90.508	76.743