

**SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING
UNIVERSITI SAINS MALAYSIA**

**COMPARATIVE STUDY OF NANOPOROUS CARBON CONVERTED
FROM WASTE TYRE CHAR & MESOCARP FIBER**

By

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(Materials Engineering)

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled “**Comparative Study of Nanoporous Carbon Converted from Waste Tyre Char & Mesocarp Fiber**”. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or University.

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LIST OF ABBREVIATIONS

AC	Activated Carbon
ACF	Activated Carbon Fiber
BET	Brunauer-Emmett-Teller
CO ₂	Carbon Dioxide
FTIR	Fourier Transform Infrared
HNO ₃	Nitric Acid
KBr	Potassium Bromide
MF	Mesocarp Fiber
N ₂	Nitrogen
PAC	Powdered Activated Carbon
PSD	Pore Size Distribution
S	Sulfur
SEM	Scanning Electron Microscopy
SiO ₂	Silica
TGA	Thermogravimetric Analysis
WTC	Waste Tyre Char
XRD	X-ray Diffraction
ZnO	Zinc Oxide

LIST OF SYMBOLS

2θ	Angle of Diffraction
%	Percentage
wt %	Weight Percent
$^{\circ}\text{C}$	Unit of Temperature on Celsius Scale
K	Unit of Temperature on Kelvin Scale
g	Gram
kg	Kilogram
mg	Milligram
L	Liter
mL	Milliliter
mM	Milli Mol
nm	Nanometer

KAJIAN PERBANDINGAN NANOPOROUS KARBON TERAKTIF YANG DIHASILKAN DARIPADA SISA TAYAR DAN GENTIAN MESOKARPA

ABSTRAK

Sisa yang mempunyai nilai komersial yang kecil dan nilai karbon yang tinggi boleh digunakan untuk mensintesis nanoporous karbon teraktif dengan kos yang rendah. Karbon teraktif yang dihasilkan dari beberapa sisa mempunyai luas permukaan yang tinggi dan keliangan telah digunakan untuk tujuan penjerapan. Dalam kajian ini, serbuk karbon teraktif (PAC) dan serat karbon teraktif (ACF) telah disintesis daripada sisa tayar (WTC) dan gentian mesokarpa (MF) melalui suhu karbonisasi yang berbeza dalam suasana gas nitrogen dan diikuti oleh pengaktifan gas CO₂. Suhu karbonisasi digunakan adalah 500°C, 600°C dan 700°C. Sifat-sifat fizikal PAC dan ACF sampel telah dikaji dan dibandingkan. PAC dan ACF sampel yang dihasilkan kemudiannya digunakan untuk penyingkiran p-Cresol dari air sisa. Perbandingan menunjukkan bahawa ACF adalah penjerap yang berpotensi untuk p-cresol dengan kapasiti penjerapan yang lebih tinggi daripada PAC. Pengurangan suhu karbonisasi menyebabkan peningkatan dalam p-cresol penjerapan disebabkan oleh peningkatan dalam luas permukaan. ACF sampel yang teraktif dengan suhu karbonisasi 500°C mempunyai luas permukaan yang tertinggi iaitu 616 m²/g dan kapasiti penjerapan yang tertinggi iaitu 58.2 mg/g.

COMPARATIVE STUDY OF NANOPOROUS CARBON CONVERTED FROM WASTE TYRE CHAR & MESOCARP FIBER

ABSTRACT

Wastes with a little commercial value and have high carbonaceous value can be utilized to synthesize low cost nanoporous activated carbon (AC). AC produced from some wastes have high surface area and porosity can be used for various adsorption purposes. In this study, powdered activated carbon (PAC) and activated carbon fiber (ACF) were synthesized from waste tyre char (WTC) and mesocarp fiber (MF) at different carbonization temperatures in nitrogen gas atmosphere and followed by CO₂ gas activation at 900°C. The carbonization temperatures used were 500°C, 600°C and 700°C. The physical properties of the PAC and ACF samples were studied and compared. The PAC and ACF samples produced were then used for removal of p-Cresol. The comparison showed that ACF was a good potential adsorbent for p-Cresol with a higher adsorption capacity than that of PAC. Decrease of carbonization temperatures resulted in the increase in p-Cresol adsorption predominantly due to increase in surface area. The best ACF sample was found carbonized at 500°C having an excellent surface area of 616 m²/g and adsorption capacity of 58.2 mg/g.

CHAPTER 1

INTRODUCTION

1.1 General Introduction

Activated carbon (AC) is a porous carbonaceous solid material with high porosity and large surface area. It is influenced by precursor and activation processes. AC is used for adsorption purposes such as heavy metal removal, wastewater treatment, dye removal and air filtration. It is produced through carbonization and followed by activation. AC can be produced from various raw materials with high fixed carbon content such as coal, coconut husk, rice husk and sawdust. In this study, AC was produced from waste tyre char and mesocarp fiber.

Waste tyres are tyres discarded after they are unfit for further use in a vehicle they were made for. Waste tyres can be recycled into commodities and resources for concrete, asphalt and other tyres. In order to do that, it must first be reduced in size and then recycled. One of the by-products during waste tyre recycling is waste tyre char in powder form with high carbon content. A more value added application to explore would be to convert them into useful porous adsorbent such as AC for air-quality control application (Lopez et. al, 2010).

Another waste which could be converted into AC is mesocarp fiber. According to Malaysian Oil Palm Statistics in year 2005, Malaysia has processed 75.5 million tonnes of oil palm fresh fruit bunch (FFB) that created 11.9 million tonnes of palm-pressed mesocarp fiber; and the palm oil production was reported to rise annually. Conventionally, mixture of

mesocarp fiber and kernel shell was utilized as low energy fuel to generate electricity for palm oil mill. The excess mesocarp fiber and empty fruit bunch were also used as mulches in the plantation field (Lau, et al, 2008). In fact, mesocarp fiber is a good raw material for production of a new form of AC due to its naturally fibrous form.

Both waste tyre char and mesocarp fiber can be converted into nanoporous activated carbon through two-step physical activation. The physical activation includes carbonization to remove non-carbon impurities in biomass and followed by activation to further increase the porosity of carbonized biochar (carbonaceous material produced from carbonization process). Three main thermal carbonization methods are incineration, gasification and pyrolysis (Galvagno, et. al, 2002). Carbonization is a heat treatment or pyrolytic decomposition of heterogeneous precursor materials in the absence of oxygen at temperature below 1000 °C. Non-carbon impurities such as oxygen, hydrogen, nitrogen and sulfur are converted into volatile gaseous compounds and largely removed from carbonized material. Thus, the resulting char contains predominantly carbon element.

Carbonization did not fully developed the pore structure of carbonized char. Hence, carbonized char need to undergo activation; an oxidative treatment to promote its pore structure. During the activation process, the carbon structure of the carbonized material destructed and led to the formation of highly porous structure that are mostly micropores. The porous activated carbonized material owns an extremely high surface area which gives rise to an excellent adsorption capacity (Jankowska,et.al, 1991).

1.2 Research Background

Activated carbon (AC) is characterized by its extraordinary large specific surface area and well-developed porosity. AC is widely used as adsorbents for the removal of organic chemicals and metal ions from environmental concern air and wastewater. AC can exist in powder, granular and fiber forms. The shapes and properties of AC are greatly influenced by the types of raw materials used and the activation condition (El-Hendawy, 2003). AC can be produced from the proposed waste tyre char and mesocarp fiber by carbonization and later by activation process.

Waste tyres are difficult to reprocess since they contain complex mixture of various materials, e.g. rubber, carbon black, steel cord, organic and inorganic components. Mesocarp fibers from palm oil extraction are also a waste product that has very little commercial value. Therefore, conversion of these materials into AC has been considered to be a profitable and clean way to reutilize these materials (Edward, et. al, 2004). In short, the aim of this research is to identify the possibility of using AC derived from both waste tyre char and mesocarp fiber as effective adsorbents for p-Cresol removal in wastewater treatment plant.

p-Cresol is a positional isomer of phenol. Various physical, chemical and biological processes have been used for removal of p-Cresol from wastewater such as membrane filtration, advanced oxidation, electrolysis, activated sludge and adsorption (Das, et al, 2012). Adsorption is most versatile due to high efficiency. Kinetic analyses indicated that

p-Cresol was adsorbed mainly via chemisorptions and was limited mainly via intra-particle diffusion.

1.3 Problem Statement

The number of industrial wastes on earth continues to increase. Amongst them are waste tyres and mesocarp fiber due to their large production. The complexity of tyre recycle process caused most of the waste tyres went into uncontrolled dumps. On the other hand, mesocarp fiber is a major biomass generated by palm oil mills during palm oil extraction process. Biomass residues from palm oil mills are either burnt in the open or disposed off in waste ponds, which are the reasons to severe environmental pollution. Conventional disposals of these wastes are inappropriate and therefore converting waste tyre char and mesocarp fiber into nanoporous carbon adsorbents for other value added and ecofriendly applications, e.g. dyes and toxins removal from wastewater treatment plant, would be a better alternative.

1.4 Objectives

The overall objective of this study is to evaluate the physicochemical properties of AC derived from waste tyre char and mesocarp fiber, as stated below.

1. To convert AC from waste tyre char and mesocarp fiber through two-step physical activation.
2. To determine the physical and chemical properties of nanoporous AC produced.
3. To determine the p-Cresol adsorption of the nanoporous AC.

1.5 Scope of Research

In this study, powdered activated carbon (PAC) and activated carbon fiber (ACF) were synthesized from waste tyre char and mesocarp fiber respectively. AC was produced via pyrolysis and physical activation. Waste tyre char and mesocarp fiber were first characterized via thermogravimetric analysis (TGA) to determine their carbonization temperature. Consequently, the effect of several carbonization temperatures towards the prepared activated carbon samples was studied. Waste tyre char was characterized by carbon, hydrogen, nitrogen, oxygen and sulfur analysis (CHNOS) to determine its carbon, hydrogen, nitrogen, sulfur and oxygen content. PAC and ACF were analyzed using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), X-ray diffractometry (XRD), X-ray fluorescence (XRF) analysis, fourier transform infrared spectroscopy (FTIR), raman spectroscopy and nitrogen adsorption analysis. The p-Cresol adsorption tests were carried out on the activated carbon samples via UV-Vis spectrophotometry and the adsorption capacity of these samples were discussed.

CHAPTER 2

LITERATURE REVIEW

2.1 Activated Carbon

Nanoporous materials are defined as solid matters containing porous structure (pore size is about or less than 2 nm). They are usually used for energy storage, solar cells, supercapacitors, catalysis, adsorption applications, gas purification, separation technologies, drug delivery, cell biology, environmental remediation, water desalination, purification and separation process (Bhattacharyya et al., 2014). Activated carbon (AC) is an example of nanoporous materials used in many industrial applications.

AC is a carbonaceous material with a high degree of porosity and extended surface area. AC consists of more than 90% of carbon element. It is a very versatile adsorbent that has been used in the environmental and industrial fields for removal, separation and modification of a variety of species in liquid and gas-phase applications (Cecen, 2014).

There are three forms of AC which are powdered activated carbon (PAC), granular activated carbon (GAC) and activated carbon fibers (ACF). PAC is a pulverized carbon with particle size less than 0.18 mm. This results in the large external surface area with a small diffusion distance. GAC has irregular shapes of particle and sizes range from 0.2 to 5.0 mm. GAC is usually used in fixed bed adsorbers in continuous processes for the adsorption of gases and vapours (Nik et al., 2006). ACF is a light porous carbon in fiber

form with diameter ranged from 10-40 μm . ACF has several advantages compared to PAC and GAC. These advantages are large surface area, high adsorption capacity of liquid and gas phase of adsorbates (Ko et al., 2002).

2.2 Precursors

The selection of precursors affects the properties and adsorption capacity of AC. There are several important criteria to be considered in order to produce AC with outstanding properties cost-effectively. These criteria are the cost and availability of precursor, low inorganic material, low degradation upon storage and ease of activation (Marsh and Reinoso, 2006).

2.2.1 Conventional Starting Materials

Coal is a common conventional precursor used for the production of AC due to its availability and cost. Among all the type of coal-based materials, bituminous coal is preferable for AC production due to its better density, hardness, abrasion resistance and durability (Teng et al., 1997). However, the mining process for extracting coal has resulted in environmental issues such as land degradation, soil and water pollution (Yenilmez et al., 2011).

2.2.2 Biomass

Activated carbon is a useful adsorbent used in many adsorption and filtration applications. However, expensive and not eco-friendly conventional starting materials restrict the mass production of AC. Wastes or biomass from industry and agricultures could be used as starting materials for adsorbents production since they are available in huge amount, inexpensive and require little processing (Crini, 2006).

Empty fruit bunch fiber, palm kernel shell, sugar cane bagasse, coconut shell, rice husk, jute fiber, bamboo and sawdust are the common agricultural waste used to prepare low cost AC with excellent properties. All these agricultural waste are cheaper and easily available, with consistent quality and purity compared to the conventional starting materials such as coal (Abdullah et al., 2011).

2.2.3 Mesocarp Fiber

Mesocarp fiber (MF) is a part of solid biomass generated during the palm oil extraction process. MF is produced after the pressed oil palm fruits for oil extraction and separated from palm kernel. In the palm oil mill, fresh fruit bunches are sterilized after which the oil fruits can be removed from the branches. The empty fruit bunches are left as residues, and the fruits are pressed in oil mills. The palm oil fruits are then pressed, and the kernel is separated from the press cake which is mesocarp fibers. The palm kernels are then crushed and the kernels then transported and pressed in separate mills.

MF is elongated cellulose about 3-5 cm length which has been found to trap about 5–7 % of residue oil after screw-press extraction of crude palm oil. The amount of MF generated was about 13% of the total fresh fruit bunch (FFB) production. It is suggested that about 11.5 million tones of MF are produced from palm oil mills annually (Hoe, 2014). MF is normally disposed in plantation site for natural decomposition as to return organic matter to soil. However, this will cause an uncontrolled emission of green house gases such as methane and carbon dioxide which eventually resulted in global warming (Brebbia and Zubir, 2006).

2.2.4 Waste Tyre Char

Waste tyre char (WTC) is a solid waste generated from the recycling of waste tyre. It usually contains carbon black and some metals. 57 thousand tonnes of waste tyres were generated annually in Malaysia (Koh, et al., 2017). Cement industry uses waste tyre as a low cost supplementary fuel due to their high calorific value. This process can be acceptable from an environmental point of view only in the case of controlled combustion due to the toxic gas emission during the tyre combustion processes.

WTC can potentially be used as a precursor of activated carbon. Lopez and co-workers have successfully prepared char derived from WTC via pyrolysis. The WT-based char consists of mainly carbon (86.9 wt %) with BET surface area up to 65.2 m² g⁻¹. Such high amount of carbon indicates that waste tyre char is suitable precursor for the AC production.

2.2.5 Tyre Component

A tyre is a strong, flexible rubber casing attached to the rim of a wheel. Tyres provide a gripping surface for traction and serve as a cushion for the wheels of a moving vehicle. Tyre normally consists of rubber, steel wire, carbon black and some additives. Figure 2.1 shows the components of a tyre which includes inner liner, body ply, side wall, beads, apex, steel belt, tread and cushion gum.

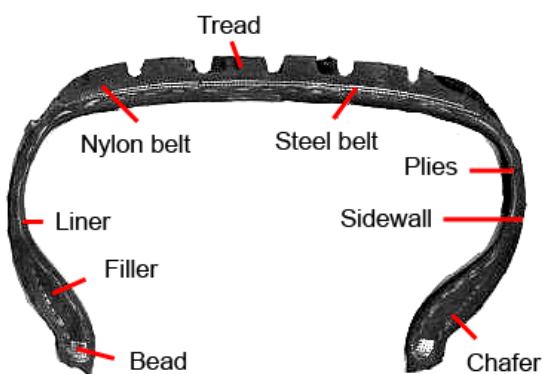


Figure 2.1: Tyre cross section showing components of a tyre (Arvind Boudha, 2011).

2.2.6 Recycling of Waste Tyre

Tyre is a complex mixture of different materials which include rubbers, carbon blacks, steel cord and other organic and inorganic minor components. Recycling of scrap tyres refers to the recovery of rubber granules, steel wires and carbon black from the waste tyre. Pyrolysis provides an environmentally attractive method to decompose waste tyre.

Current recycling process of waste tyre involves a lot of machinery to separate valuable material (e.g., steel wires) from waste tyres. The recycling process begins first by shredding waste tyres into small manageable chips, which are then cooled to cryogenic temperatures, causing the pieces to become brittle. These brittle pieces are then pulverized into fine material and screened to remove large chunks of rubber or polymer. Finally, the remaining fiber and magnetic material are separated from the pulverized material using a magnetic separator and vibrational separator. This recycling process is environmentally friendly and allows a valuable resource to be used again and again.

In the pyrolysis process, the organic volatile matter of tyre is decomposed to low molecular weight products, liquids or gases, which can be used as fuels or chemicals source. The non-volatile carbon black and the inorganic components remain as solid residues, which could be recycled for other applications. The liquid and gas products are used as source of chemicals and fuel in the pyrolysis process, respectively (Chemsain Konsultant Sdn Bhd, 2011). Presently, the recycled carbon black has very limited commercial values. It is only used as fuel and reinforcement in rubber industry (Moulin, et. al., 2017). The carbon black recovered could possibly be used as a precursor for AC production due to its high carbon content, availability and low cost.

2.3 Synthesis of Activated Carbon

AC is usually produced through carbonization of precursors at high temperature in an inert atmosphere and followed by an activation process. The type of precursors, carbonization and activation methods could significantly influence the properties of the AC.

2.3.1 Carbonization

Carbonization is defined as a heat treatment or pyrolytic decomposition of a heterogeneous precursor material like coal or wood in the absence of oxygen at temperatures from 400 to 600 °C. Along with tars, oils and gases, carbonization yields a carbonized material or simply called char. In char, non carbon impurities such as oxygen, hydrogen, nitrogen, and sulfur will be largely removed by conversion into volatile gaseous products. Thus, the carbon element predominates in the resulting char. The char exhibits considerable disorder structure since residual carbon atoms have arranged themselves into flat aromatic sheets that are cross-linked in a random manner. The interstices in these irregularly aromatic sheets give rise to the porosity (Cecen, 2014).

Pyrolysis is a thermal degradation process in which the material is heated indirectly in an oxygen-free atmosphere. Organic material that fed to the pyrolysis reactor undergoes a thermal cracking and cleaving itself into a volatile fraction and a solid residue. Volatile fraction can then be cooled and partially condensed to obtain a liquid and gaseous fraction. A gaseous fraction essentially composed of hydrogen, methane and carbon oxides while the liquid fraction composed of water, tar and oils. A solid residue containing fixed carbon and ashes of metals, oxides and inert matter are also produced during the pyrolysis (Galvagno, 2002).

2.3.2 Activation

Pore structure needs to be further enhanced by an oxidative treatment called activation. In this process, destruction of the carbon structure by the oxidation of carbonized material occurs and leading to the development of a highly porous structure consisting largely of micropores. Activation leads to a product with extremely high surface area which gives rise to an excellent adsorption capacity. There are two types of activation process which are known as physical and chemical activation (Cecen, 2014). The oxidizing agent reacts with carbon to produce oxides of carbon. These oxides diffuse out of the carbon resulting in opens pores that were previously closed and further expand the carbons internal porous structure.

Physical activation using carbon dioxide or steam as oxidizing agent is the commonly used processes in the production of activated carbons. The physical activation usually consists of two steps. At first, thermal pyrolysis occurs at a relatively low temperature typically 400–700 °C in the presence of nitrogen or helium to break down the cross-linkage between carbon atoms. Then, activation with activating gas at 800–1000 °C takes place for further development of the porosity of activated carbon (Edward, et. al, 2004).

For chemical activation, the carbon is reacted at high temperatures with a dehydrating agent that eliminates the majority of hydrogen and oxygen from the carbon structure. Chemical activation often combines the carbonization and activation steps. Precursor is first impregnated with activating agents in the form of concentrated solution to

degrade the cellulosic materials. The chemical impregnated precursor is then pyrolyzed in a furnace at temperature 400-600 °C in the absence of air (Jankowska, et.al, 1991).

2.4 Properties of Activated Carbon

AC is a carbonaceous, highly porous adsorptive medium that has a complex structure composed primarily of carbon atoms. AC is one of the mostly used and important microporous adsorbents due to its tremendous adsorptive capacity. It can adsorb different types of substances from gaseous or liquid phases. Therefore, AC is widely used in many fields such as water and air purification, separation of industrial gases and for discoloring in pharmaceutical (Patil Pragya, 2013)

2.4.1 Porosity in Activated Carbon

The porous structure of an AC consists of a wide range of pore size. The pores are classified into three categories which are micropores, mesopores and macropores according to their diameter. The diameter of the micropores is less than 2 nm, while 2 to 50 nm for mesopores and larger than 50 nm for macropores (Kral et. al, 1988).

Figure 2.2 shows the classifications of pores in AC. All of the pores have continuous channel of communications with external surface of porous materials. Micropores have pore volume of about 0.15 to 0.70 cm³ g⁻¹. About 95 % of total surface area of AC is contributed by the micropores. The pore volume for mesopores varied from 0.1 to 0.2 cm³ g⁻¹ and its surface area does not exceed 5% of total surface area of AC

(Bansal and Goyal, 2010). The macropores of an AC act as transport pores that enabling the molecules of the adsorptive to reach the smaller pores situated in the interior of the AC. Although the surface area of macropores is very low but they affect the rate of diffusion into the mesopores and micropores. Most of the adsorption takes places within the micropores, while the mesopores serve as passages for adsorbate to reach the micropores (Sevil Cetinkaya, 2003).

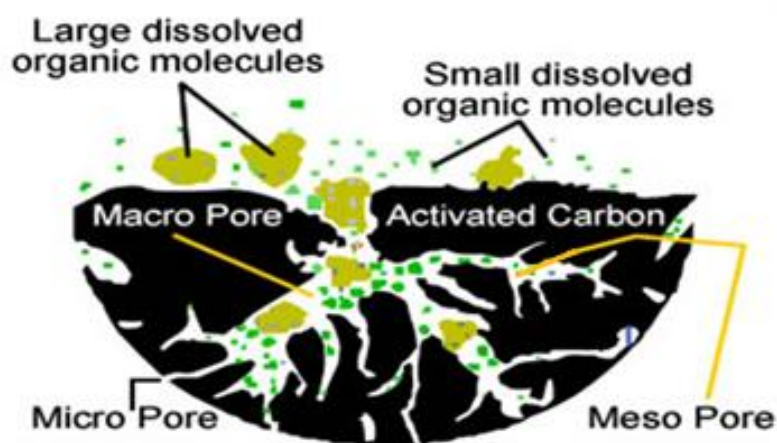


Figure 2.2: Classification of pores in AC (Kral et. al, 1988)

2.4.2 Structure of Activated Carbon

Activated carbon can be defined as a crude form of graphite with a random or amorphous structure, which is highly porous over a broad range of pore sizes. The graphite crystal is composed of layers of fused hexagons held by weak van de Waals forces (Figure 2.3). The layers are held by carbon-carbon bonds. Activated carbon is a disorganized form of graphite due to impurities and the carbonization and activation processes. The presence

of heteroatoms such as oxygen, hydrogen and defects such as vacant lattice sites prohibit the repetition of long range arrangement in AC (Bansal and Goyal, 2010).

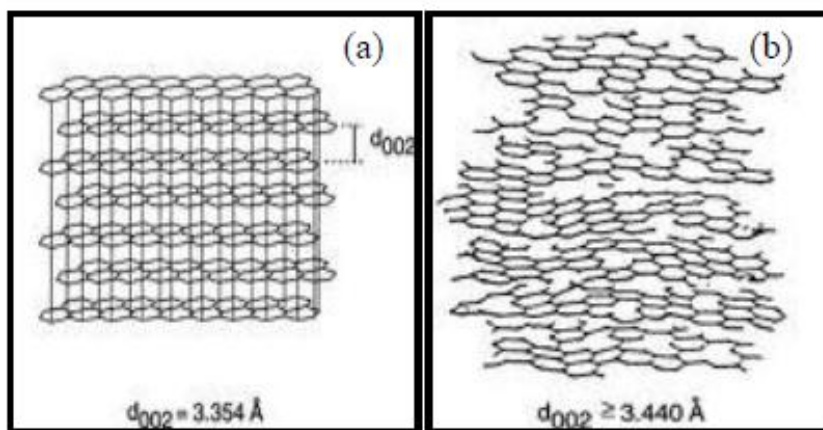


Figure 2.3: Three dimensional crystal lattice of (a) graphite and (b) activated carbon (Miremadi and Colbow, 1998)

2.4.3 Surface Morphology of Activated Carbon

The surface morphology of AC would affect the adsorption performance of AC. Scanning electron microscopy (SEM) is normally used to observe the surface morphology of AC. Macropores is common pores that could be observed on the surface of AC. The macropores would act as connecting channels to allow molecules of adsorbates to move into micropores and adsorbed onto the interior surface of AC. Figure 2.4 shows the SEM image of coal-based AC and it is observed that macropores are present on the surface.

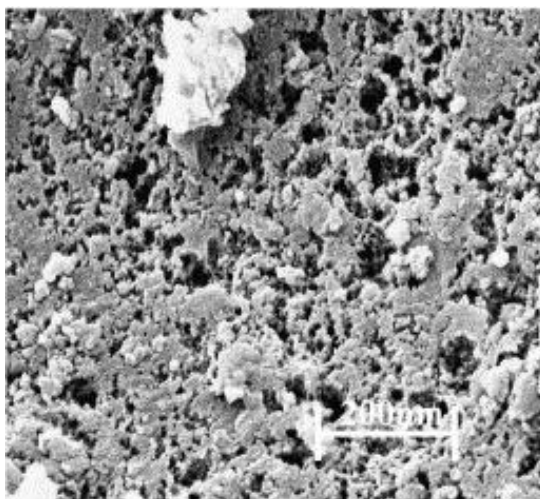


Figure 2.4: SEM image of coal-based AC (Chingombe et. al, 2005)

2.4.4 Surface Chemistry of Activated Carbon

It is well known that the principal component of AC is carbon element and surface of AC itself is non-polar, and hence it is concluded that as a whole, AC has hydrophobic surface. Functional groups of AC surface may be acidic and basic groups because the surface chemistry of AC is closely associated to the types of heteroatoms such as oxygen, hydrogen and nitrogen other than carbon atom within the carbon matrix.

Some common heteroatoms mainly including oxygen (O), nitrogen (N), phosphorus (P), hydrogen (H), chlorine (Cl) and sulfur (S) introduced in the activation process are bound to the edges of graphite-like layers. This will form organic functional groups such as carboxyl groups, lactonic groups, phenolic groups, carbonyl groups, aldehydes, ethers, amino groups and other N-containing groups and phosphates on the surface of AC. The

surface functional groups of ACs affect their moisture content, catalytic properties, acid/base character and adsorption capacity (Salame and Bandosz, 2001).

2.4.5 Liquid Phase Adsorption of Activated Carbon

AC is a useful adsorbent for the liquid phase adsorption applications. For liquid with homogeneous binary mixture, it contains two components which are solute and solvent. An adsorbent will change the concentration of the liquid by adsorbed the solute while the solvent is assumed to be non-adsorbed. Micropores are the major sites for the adsorption to occur in liquid phase adsorption. The pore size distribution of AC is greatly influenced by the percentage of burnt-off. The adsorption capacity is greater for higher surface area adsorbents due to increasing pore volumes (Mangun, 1998).

2.5 Application of Activated Carbon

2.5.1 Adsorption

AC was used in many adsorption applications due to their high surface area, microporous structure and high degree of surface reactivity (Bansal and Goyal, 2010). AC can be used in gas phase and liquid phase adsorption applications.

2.5.2 Introduction on p-Cresol

p-Cresol is also known as 4-methylphenol, is an organic compound with the formula $\text{CH}_3\text{C}_6\text{H}_4(\text{OH})$. It is a colourless solid that is widely used as an intermediate in the production of other chemicals such as p-hydroxybenzoic acid and 3,4,5-trimethoxybenzaldehyde (Greensboro, 2017). It is a derivative of phenol and is an isomer of o-Cresol and m-Cresol (Fiege, 2000). p-Cresol is an end product of aromatic amino acids and it affect immune system of uremic patients.. It is produced from food proteins by intestinal bacteria and is detectable in blood, urine and feces. p-Cresol may contribute to atherosclerosis and thrombosis in patients with uremia (National Center for Biotechnology Information) Figure 2.5 shows the structural formula of p-Cresol. It mainly consists of a benzene ring that attach with a hydroxyl group and a methyl group.

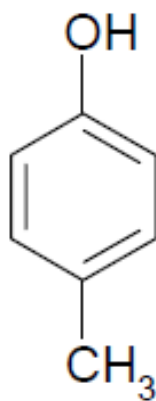


Figure 2.5: Structural formula of p-Cresol (National Center for Biotechnology Information)

2.5.3 p-Cresol Adsorption

In this study, the AC samples derived from agricultural waste were used for para-cresol (p-cresol) adsorption. p-Cresol is a protein-bound molecules found in body of patients suffer from kidney failure disease. This protein-bound molecule is not efficiently eliminated by present hemodialysis system. The retention of p-cresol molecules in body resulted in toxic effects to the patients (De Smet et. al, 1998, Dou et. al, 2002).

p-Cresol adsorption is important because it can reduce the effect of p-Cresol on human body and environment. p-Cresol is highly toxic. It can cause cancer and it has adverse effect on the nervous, cardiovascular and respiration systems if it enter human body. p-Cresol is also toxic to aquatic organisms and animals. p-Cresol is released to the environment through automobile exhaust, wood and trash burning and tobacco smoke. p-Cresol is found in many species of plants and occurs in coal, petroleum and as a constituent in wood.

CHAPTER 3

MATERIALS AND METHODOLOGY

3.1 Introduction

Experimental procedures involved in the synthesis and characterizations of powdered activated carbon (PAC) and activated carbon fiber (ACF) are discussed in this chapter (Figure 3.1). PAC and ACF samples were obtained from waste tyre char (WTC) and mesocarp fiber (MF) respectively, through a process of carbonization and activation. Cleaned raw WTC and MF were first characterized using CHNOS analysis and thermogravimetric analysis (TGA). The effect of carbonization temperature towards the PAC and ACF samples was evaluated. Characterizations such as scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), X-ray diffraction analysis (XRD), X-ray fluorescence analysis (XRF), fourier transform infrared spectroscopy (FTIR), raman spectroscopy and nitrogen adsorption analysis, were carried out on samples (WTC, MF, PAC, ACF). The adsorption behavior of PAC and ACF was identified with p-Cresol.

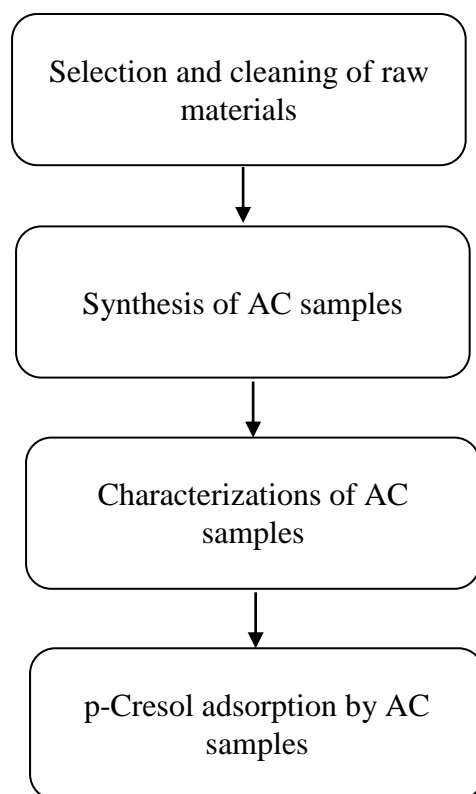


Figure 3.1: Experiments flowchart of this research

3.2 Synthesis of Powdered Activated Carbon from Waste Tyre Char

3.2.1 Raw Material

WTC powder was collected from a waste tyre recycle factory. It was gently washed with deionized water to remove impurities from its surface. Cleaned WTC was dried overnight in an oven at 100 °C.

3.2.2 Synthesis of Powdered Activated Carbon

10 g of clean WTC (Figure 3.2) placed in a crucible for carbonization using a horizontal tube furnace (OTF-1200X) with N_2 gas flowing at 100 ml min^{-1} . The WTC was heated to $500\text{--}700^\circ\text{C}$ for 1 hour at heating rate of $10^\circ\text{C min}^{-1}$ and then left to cool at room temperature. The weight of carbonized WTC was measured and then washed with deionized water to remove any volatile impurities that released during carbonization. Clean carbonized WTC was dried in an oven at 100°C overnight.



Figure 3.2: Raw WTC prior to carbonization

For activation, the carbonized WTC was further heated in the tube furnace for 1 hour at 900°C with heating rate of $10^\circ\text{C min}^{-1}$ under a constant N_2 gas flow. Once temperature reached 900°C , N_2 gas supply was turned off and dry CO_2 gas was purged into the furnace with 100 ml min^{-1} flow rate. After 1 hour, CO_2 gas supply was turned off and the activated sample was cooled down to 25°C in a N_2 gas atmosphere. The PAC sample obtained was weighed. The synthesis route of PAC sample from WTC was presented in Figure 3.3. From Table 3.1, various carbonization temperatures were carried out to study its effect upon the properties of PAC samples produced.

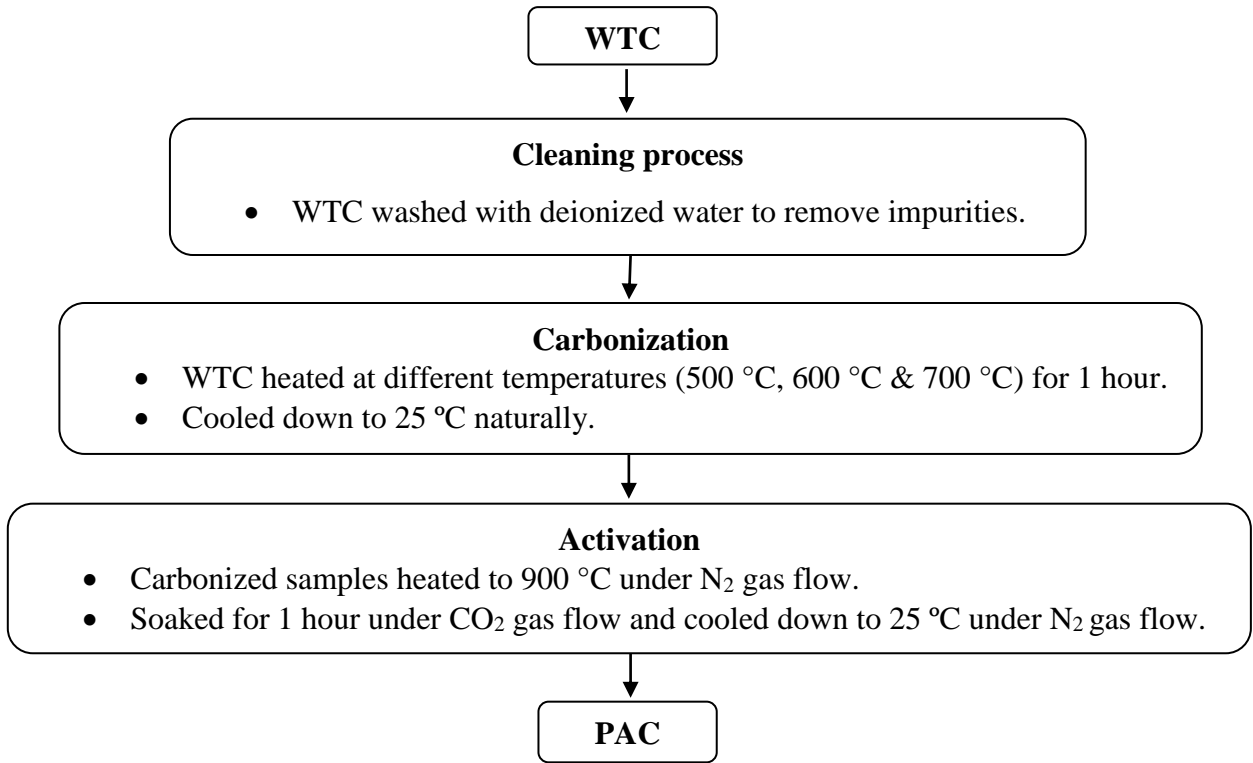


Figure 3.3: Synthesis route for the production of PAC samples from WTC

Table 3.1: Carbonization temperature and PAC samples identification

Sample	Carbonization Temperature (°C)
WT-CA500	500
WT-CA600	600
WT-CA700	700