Evaluation of Activated Carbon from Waste Tyre for the Adsorption of 2-chlorophenol

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Evaluation of Activated Carbon from Waste Tyre for the Adsorption of 2-chlorophenol

KANCHANA MANIRAJAH

Thesis submitted in partial fulfilment of the requirements
For the award of the degree of
Bachelor of Chemical Engineering

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SUPERVISOR'S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Bachelor of Chemical Engineering.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

Signature :
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Date : 15 DECEMBER 2016

Dedication

To my parents, lecturers, and God.

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ABSTRACT

A mesoporous carbon developed from waste rubber tire, characterized by chemical analysis where firstly reported with X-ray diffraction (XRD) indicating the presence of activated carbon and revealed a predominantly amorphous structure of the tire-derived activated carbon. Field Emission Scanning Electron Microscope (FESEM) images resulted in surface structure of the activated carbon. Fourier transform Infrared spectroscopy (FTIR) showed the presence of functional group. Hence, in this research activated carbon was used as an adsorbent for the removal of 2-chlorophenol. Surface area, porosity, and density were determined. The adsorption of the 2-chlorophenol over the prepared adsorbent was achieved under different adsorbate concentration, pH and adsorbent dosage. By percolating the 2-chlorophenol solution through UV–vis diffuse reflectance spectroscopy, the bulk removal of the chlorine was carried out and necessary parameters were determined to find out the percentage removal. The synthesized activated carbon as an effective adsorbent dosage for the removal of 2-chlorophenol due to its excellent stability and chemical inertness and is known to be biocompatible.

ABSTRAK

Karbon dibuat daripada sisa tayar getah, ciri-ciri analisis kimia di mana pertama dilaporkan dengan sinar-X pembelauan (XRD) menunjukkan kehadiran karbon diaktifkan dan mendedahkan struktur terutamanya amorfus karbon diaktifkan tayar yang diperolehi. Field Pelepasan Scanning Electron Microscope imej (FESEM) menyebabkan struktur permukaan karbon yang diaktifkan. Fourier spektroskopi inframerah (FTIR) menunjukkan kehadiran kumpulan berfungsi. Oleh itu, dalam kajian ini karbon diaktifkan digunakan sebagai penjerap untuk penyingkiran 2-klorofenol. kawasan permukaan, keliangan dan ketumpatan ditentukan. Penjerapan 2-klorofenol lebih penjerap disediakan telah dicapai di bawah kepekatan bahan terjerap yang berbeza, pH dan penjerap dos. Oleh penelusan penyelesaian 2-klorofenol melalui UV-vis meresap pantulan spektroskopi, penyingkiran sebahagian besar daripada klorin telah dijalankan dan parameter yang perlu ditentukan untuk mengetahui penyingkiran peratusan. Karbon diaktifkan disintesis sebagai dos penjerap berkesan untuk penyingkiran 2-klorofenol kerana kestabilan.

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LIST OF ABBREVIATIONS

AC	Activated Carbon
2-CP	2- Chlorophenol
DCM	Dichloromethane
Zr	Zirconia
VOC	Volatile organic compound
PAC	Powder Activated Carbon
GAC	Granulated Activated Carbon
KOH	Potassium Hydroxide
HCL	Hydrochloric Acid
H_2O_2	Hydrogen Peroxide
N_2	Nitrogen Gas
CO_2	Carbon Dioxide
HNO_3	Nitric Acid
BET	Brunauer-Emmet-Teller
EDX	Energy dispersive X-ray spectroscope
SEM	Scanning electron microscopy micrographs
FTIR	Fourier Transform Infrared
XRD	X-ray diffraction
UV-Spectrometer	Ultraviolet Spectrometer
EI	Electron Impact

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Generally, almost every ground transportation need is met through vehicles of any kind. Subsequently, a gigantic measure of utilized tires is produced each year all inclusive. Recent statistical data show a 5% increase of global tyres production in 2011, compared to 2010, reaching 14.68 million tons (Jatma, 2012). Asia and Oceania represent very nearly 60% of the worldwide production (Fig. 1).

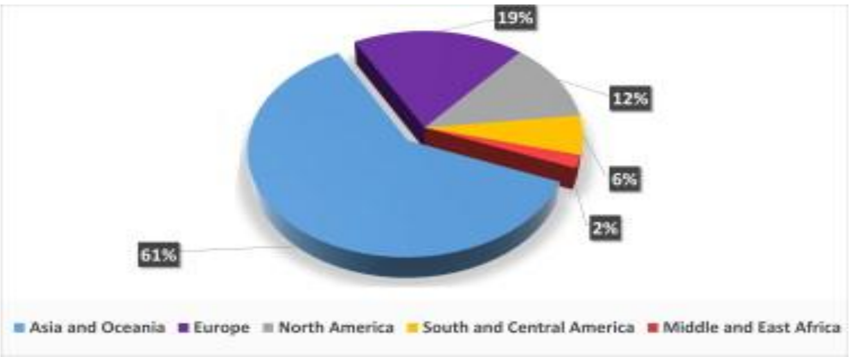

Figure 1: World tyre global production (European Tyre & Rubber Industry, 2011)

The ever growing transportation industry induced serious pollution issues to the environment. The disposal of tyres represents a major environmental issue throughout the world, since the same properties that make them desirable as tyres, most notably durability, also make their disposal and reprocessing difficult. Hazards associated with scrap tires causes both health and environmental problems since most of the waste rubber tires are stockpiled (O.S. Chan, 2011).

Fire hazards in large stockpiles could consequently cause uncontrollable burning and air pollution where it will be emitting large amounts of thick black smoke and noxious gases including carcinogens. Besides that, due to the high cost of legal disposal for tyres, illegal dumping may increase and disposal of tyres is becoming more expensive, while this trend is likely to continue as landfill space becomes scarcer (Edward, 2004).

Although they constitute a problem, at the same time they provide valid opportunities for resources conservation, since they represent waste materials with great potentials as for recovering of valuable materials and fuels (Vermeulen, 2011). As waste rubber tires embodied with high amount of carbon, they were recycled likely to convert them to valuable products such as carbonaceous adsorbents. These are very extensively used in the adsorption of organic and inorganic solutes from aqueous solution. The properties of an adsorbent that are important to adsorption processes depend on the starting material and on the method used in their preparation.

Accordingly, the use of inexpensive materials such as wastes with such an aim will certainly lower the production cost of the adsorbent. At present, in fact, low- cost adsorbents are being frequently tested for heavy metal removal from contaminated water. Recycling waste tyres is definitely a challenge due to both their highly complex structure and their diverse composition (Sienkiewicz, 2012). Waste tyres can be employed as additives in road pavement or in other applications such as playground surfaces, rubber roofs, drainage systems and energy generation (Manchón-Vizuete, 2005).

Therefore, waste tyres will be used as a source for activated carbon. Some studies showed the activation of the tyre char could actually result in a product with reasonable desirable properties such as high surface area and porosity as the commercially available activated carbon (Gonzalez, 2006). It is widely accepted that the tyre chars possess higher reactivity with steam activation than carbon dioxide and the steam activated carbon had a higher BET surface area (Guillermo, 2003). The extraction of activated carbon from waste rubber tires lower the production cost and it increase the demand of activated carbon in waste water treatment industry (Gusain, 2014).

1.2 Motivation

In this study, activated carbon chosen as an efficient adsorbent for removal of 2-chlorophenol due to its high surface area, large pore volume and high adsorption efficiency. Activated carbon will be extracted from waste rubber tire as it was considered an attractive source for generating activated carbon as the process was doubly effective solution for the environmental pollution where it reducing waste in a green way. Moreover, due to high content of carbon AC from waste tires effectively removes the chlorine content in 2-chlorophenol. Besides, lowering the production cost of the activated carbon, extracting activated carbon from waste gives a chance to add values to waste rubber tires and developed attractive, economical alternative to existing treatment methods.

1.3 Problem Statement

Among the highest priority pollutants, chlorophenols represent an important class of very common water pollutants. Most of those recalcitrant pollutants are considered highly toxic to pose a human health hazard, due to their estrogenic, mutagenic or carcinogenic effects on mammalian as well as aquatic life. Hence for the removal of chlorophenol, activated carbon will be used as an adsorbent.

Instead of dispose the waste rubber tires which lead to ozone layer depletion, it was recycled to be used as a source for activated carbon due to high cost of activated carbon. Meanwhile, waste tires will be used as a source for activated carbon in order to protect the environment. (Dobrzyńska, 2010). One of the fundamental difficulties in extracting activated carbon from waste tires is high amount of energy will be utilized in this process.

The main purpose of the present work is to prepare highly mesoporous activated carbon from waste tyres which is suitable for adsorbing relatively large molecules. Therefore, effects of activated carbon from waste tyre for the removal of 2-chlorophenol will be investigated in this study.

1. 4 Objectives

- To evaluate the performance of activated carbon from waste tyre for the removal of chlorine from 2-chlorophenol.

1.5. Scopes of study

The following are the scopes of this research:

- To investigate the performance of activated for removal of chlorine from 2-chlorophenol in order to establish the optimum conditions for producing effective adsorbent.

- The performance of the activated carbon in producing the best adsorbent was investigated by manipulating the concentration of the solution at 10ppm, 30ppm, 50ppm and 100ppm and adsorbent dosage at 0.05g, 0.1g, 0.3g and 0.5g.

- In order to remove chlorine from 2-chlorophenol by using activated carbon, pH of the solution manipulated at pH 3, pH 5, pH 7 and pH 10.

- Adsorbent characterization is crucial prior experiment. Equipment such as BET Surface Area Analyzer, X-Ray Diffraction (XRD), Field Emission Scanning Microscopy (FESEM) and Fourier Transform Infrared (FTIR) will be used.

CHAPTER 2

LITERATURE REVIEW

2.1 2-chlorophenol

Many phenolic compounds are regulated as priority pollutants for environmental control because of their high toxicity to human beings even at low concentration exposure (Yagu, 2014). Chlorophenols (CPs) constitute an important class of aquatic pollutants that are present in aquatic ecosystems at concentration levels of ng/L and mg/L. Due to their toxicity and adverse effects upon human and biota, the United States Environmental Protection Agency (US EPA) has classified them as toxics or hazardous pollutants (WHO, 1989).

2-Chlorophenol (2-CP), a low chlorine-substituted phenol and the precursor of the higher-substituted CPs, has been introduced into the environment *via* anthropogenic activities. Chlorophenols have been extensively used in the production of fungicides, herbicides, insecticides, pharmaceuticals, preservatives, glue, paint, fibers, leather, and as intermediates in chemical synthesis (Huong, 2016). Exposure to 2-CP may cause carcinogenic and teratogenic properties in humans and the environment.

Additionally, high amounts of chlorinated solvents were found to cause dizziness, reduce the ability to concentrate and remember, damage the nervous system, and produce an irregular heartbeat. Various treatment strategies have been applied for the removal of 2-CP from wastewater. Some of the treatment processes engaged for the removal of chlorophenols from waste water include adsorption (Gao, 2015), catalytic wet oxidation (Chaliha, 2008), biodegradation (Steinle, 2000), ozonation and electrochemical degradation (Lim, 2013). Adsorption process has been proven globally as one of the best and most effective water treatment technologies.

2-chlorophenol (2-CP) were chosen as the adsorbates in this work because of their persistence in environment as well as being characterized as carcinogenic, having strong odour and inability to biodegrade easily (Ren, 2011). Therefore, removing them from the water is very crucial.

2.1.1 Adsorption

Techniques engaged for the removal of chlorophenols from wastewater include adsorption (Gao, 2015), catalytic wet oxidation (Chaliha, 2008), biodegradation (Steinle, 2000), ozonation and electrochemical degradation (Lim, 2013). Among them, adsorption technology has become an efficient and universal method of water purifications. The removal of impurities from gases and liquids by activated carbon takes place by adsorption. Adsorption is a term which describes the existence of a higher concentration of a substance at the interface between a fluid and a solid than is present in the fluid. The cost effectiveness of this technology is due to the use of effective adsorbents by converting solid waste to value-added products (T.Saleh, 2013). Moreover, adsorption technique also one of the potential method used for evaluate the performance of activated carbon on removal of 2-CP due to its high efficiency and ability to separate wide range of chlorinated compounds (Mittal, 2005).

2.2 Definition of Activated carbon

Increasing demand for adsorption processes in the water treatment industry enhance more researches in the production of activated carbon from alternative precursors including industrial wastes and agricultural by-products. Activated carbons with developed porous texture and high surface area have been widely used as pollution control adsorbents to adsorb a range of pollutant gases from flue gas streams (Amal, 2016).

Activated carbon was selected as effective adsorbent for removal of 2-CP due to its high adsorption capability and high porosity which can help to enrich the organic substrate around the adsorbent, promoting the pollutant transfer process and hence increasing the efficiency (Youji Li M. M., 2008). Besides their high relative abundance and easy processing costs, such precursors have been identified to have high carbon content, are insoluble in nature, have good chemical stability, high mechanical strength and also have a favorable surface chemistry (V.Gupta, 2014). Pore structure of activated carbon (from TEM) is shown in Figure 2.1

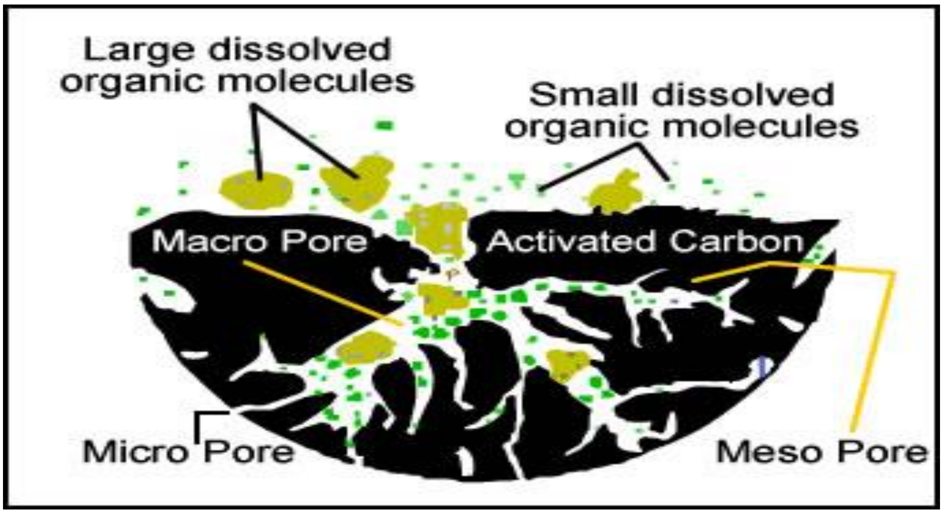

Figure 2.1 : Pore structure of activated carbon

Table 2.1: Pore Sizes of Activated Carbon (Reinose, 1985)

Macro pores	**Pores with diameters larger than 50 nm**
Mesopores	Pores with diameters between 2 nm and 50 nm
Micropores	Pores with diameters less than 2 nm

2.2.1 Types of activated carbon and its applications

Activated carbons are complex products that are difficult to classify on the basis of their behaviour, surface characteristics, and preparation methods. However, some broad classification is made for general purpose based on their physical characteristics. The types of activated carbon are powder, granular and pellet. It is classified according to its particle sizes and shape, and each type has its specific application.

The size of the powder activated carbon (PAC) is less than 1.0 mm with an average diameter between 15 and.25 mm. Thus they present a large surface-to-volume ratio with a small diffusion distance. PAC is made up of crushed or ground carbon particles. PAC is not commonly used in a dedicated vessel, owing to the high head loss that would occur. PAC is generally added directly to other process units, such as raw water intakes, rapid mix basins, clarifiers, and gravity filters. Powdered activated carbons are mainly used in liquid phase adsorption and flue gas treatment (New World Encyclopedia, 2009).

Granulated activated carbon (GAC) has a relatively larger particle size compared to powdered activated carbon and consequently, presents a smaller external surface. The size range is from 0.5 to 4.0 mm. This type of activated carbons is commonly used in water treatment facilities where it removes organic constituents and residual disinfectants in water supplies. This not only improves taste and minimizes health hazards. GAC is a favoured water treatment technique because of its multifunctional nature and the fact that it adds nothing detrimental to the treated water (DeSilva, 2000).

Meanwhile, pellet activated carbon consists of extruded and cylindrical shaped activated carbon with diameters in the range of 4-7 mm and 8-15 mm length. Pellets activated carbon is mainly used for gas phase applications because of their low pressure drop, high mechanical strength and low dust content (New World Encyclopedia, 2009).

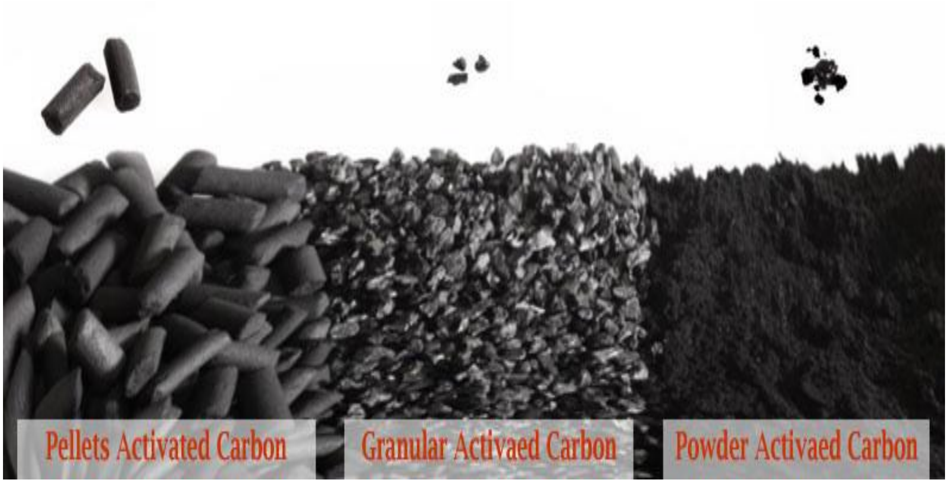

Figure 2.2: Types of activated carbon

2.2.2 Various sources of activated carbon

Activated carbons consist of interconnected cavities between defective graphene layers, which result in a high degree of porosity and an extended internal surface area. It is well known that the porosity and the surface chemistry of the activated carbons depend on the raw material from which they are made and the manufacture process. Various sources for production of activated carbon-like material such as peanut shell, bagasse, sawdust, bamboo, cherry stones, and waste tire rubber have been developed by researchers for various applications (O.S. Chan, 2011).

Table 2.2: List of local producer commercial activated carbons in Malaysia

Name of producer	**Raw material used**	**Type of activated carbon**	**Surface area (m^2/g)**
Century Chemical Works, Penang	Sawdust charcoal	Sawdust charcoal	800-1000
Effigen Sdn Bhd	Coconut shell	Granular and powder form	-
Laju Carbon Product Sdn. Bhd (LCP), Selangor.	Coconut shell	Granular, pellets and powder form	500-2500

2.2.3 Waste tires as source of activated carbon

An interesting environmental and sustainable concept is to convert waste materials to activated carbons which are then used in pollution control applications. In this study, activated carbon will be extracted from waste rubber tires. Tire rubber is a mixture of different elastomers such as natural rubber, butadiene rubber, and styrene butadiene rubber plus other additives like carbon black, sulphur, and zinc oxide. Approximately 32% by weight of the waste tire is mainly constituted of carbon black in which the carbon content is as high as 70–75 wt. % (Saleh T. A., 2013). As waste rubber tires consists high amount of carbon, they were recycled. Recycling waste tires is definitely a challenge because of both their profoundly complex structure and their diverse composition (Sienkiewicz, 2012).

Some studies showed the activation of the tire char could actually result in a product with reasonable desirable properties such as high surface area and porosity as the commercially available activated carbon (Gonzalez, 2006). It is widely accepted that the tire chars possess higher reactivity with steam activation than carbon dioxide and the steam activated carbon had a higher BET surface area in excess of 1000m2/g (Miguel, 2002). The use of inexpensive materials such as wastes with certainly lower the production cost of the activated carbon and it increase the demand for activated carbon in waste water treatment industry (Gupta, 2012). This can add great value to this low-cost material and also have dual-environmental benefit.

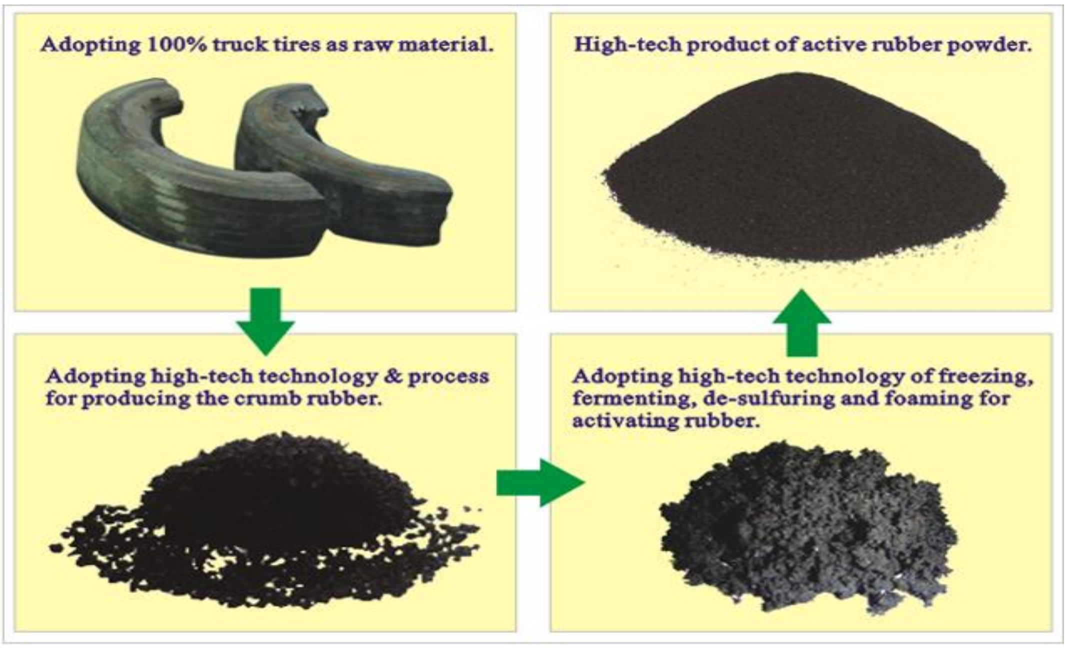

Figure 2.3: An example of production of activated carbon from waste tire.

2.3 Pyrolysis

The terms pyrolysis means to convert organic matter to elemental carbon at high temperature in the absence of oxygen. The thermal decomposition of the organic matrix in the pyrolysis yields three types of products as chars, tars and gases. The char obtained from pyrolysis has a high carbon content that can be suitable to use in the activated carbon production. This process drives off the volatiles matter to form char. The char obtained normally has low surface area and adsorption capacity since the porous structure is not well developed. This pyrolysis process must take place at 400–700°C (Ahmad, 2006).

2.4 Activation

Previous studies suggested that the activation process consisted of micropore formation, followed by pore enlargement in which the resulting carbons will have high BET surface area. At 40% burn-off, the micropore volume reached the maximum, and beyond this point mesopores grew at the expense of micro- pores but exhibits an increasing trend in the BET surface area. A similar phenomenon, reported by different researchers, stated that pores developed on highly activated samples, with a burn-off of 80–90%, will be mainly mesoporous and macro porous since both activation will be widened the existing pores and created new porosity. There are two main activation process; physical activation and chemical activation (Edward, 2004). The activation process creates or increases porosity on the activated carbon surface as illustrated in Figure 2.1.

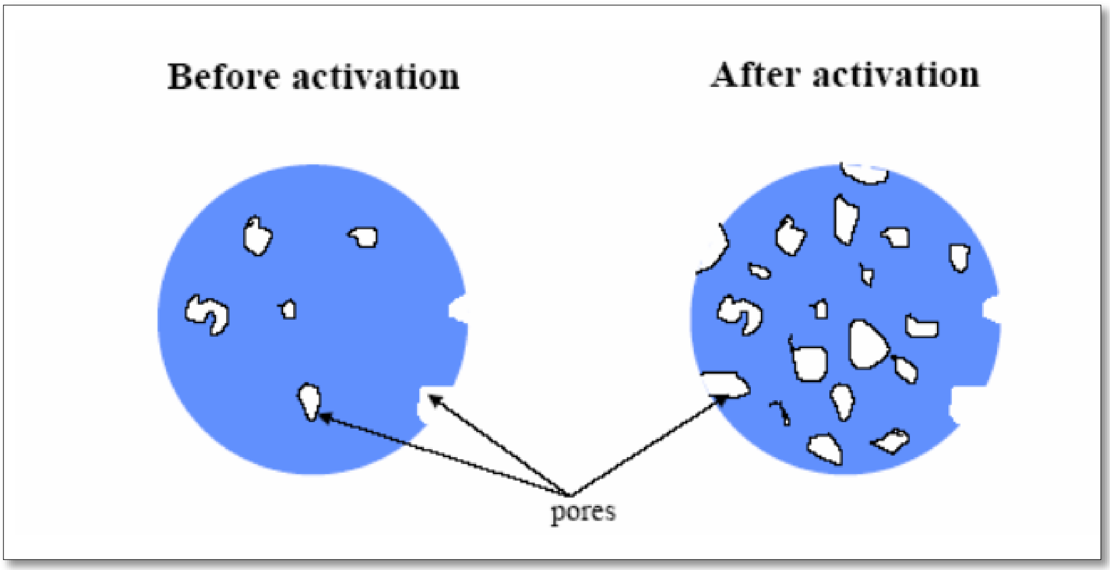

Figure 2.4: Two-dimensional representation of carbon activation (Lehmann, 1998)

2.4.1 Physical Activation

Physical activation using carbon dioxide or steam as oxidizing agents are the most commonly used processes in the production of tire carbons. It is a conventional manufacturing process of activated carbon. The overall process usually consists of two steps: thermal pyrolysis at a relatively low temperature (typically 400-600C) in the presence of nitrogen or helium to break down the cross-linkage between carbon atoms, and activation with activating gas at 800-1000 ^{o}C for further development of the porosity of carbon.

Initially, the raw material undergoes a size- reduction process and then is fed to the pyrolysis reactor for its de-volatilization. Subsequently, the received product is activated in a second reactor with steam (or CO_2); the produced activated carbon is cooled and further treated, according to the physical form requested, either granular (Granular Activated Carbon – GAC) or powdered (Powdered Activated Carbon – PAC). If a pelletized activated carbon is proposed to be produced, modifications (additional mechanical equipment) on the process are required. The process is presented in a simplified flow sheet (Antoniou, 2014).

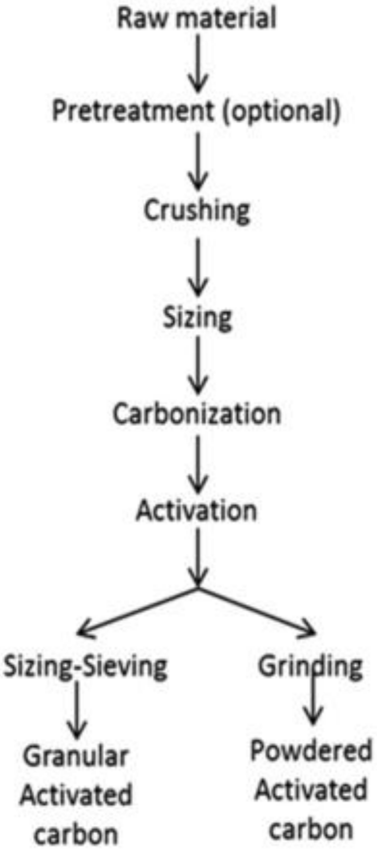

Figure 2.5: Flow sheet of activated carbon production by physical activation (Antoniou, 2014)

2.4.2 Chemical Activation

During chemical activation, the raw material is impregnated with the activating agent, mostly KOH and H3PO4; ZnCl2 is not used in current practice, due to environmental concerns regarding Zn emissions to environment. Chemical activation presents several advantages and disadvantages compared to physical activation. The main advantages are the higher yield, lower temperature of activation, less activation time and generally, higher development of porosity. Among the disadvantages, the activating agents are more expensive (KOH and NaOH vs. CO_2 and H_2O) and it is also necessary an additional washing stage (Macia, 2014). The process is presented in a simplified flow sheet (Antoniou, 2014).

Figure 2.6: Flow sheet of activated carbon production by physical activation (Antoniou, 2014)

2.5 Activated carbon as adsorbent

From findings, waste tires have been a agricultural waste materials with plausible chance of being a good precursor material for activated carbon productions. Activated carbon will be resulted in increasing the surface properties which might be attributed to the increase in microporosity (as confirmed by the reduction in average pore diameter) (Khayoon, 2013). To the best of our knowledge no study has been done on the adsorption applications of pure activated carbon from waste tires using potassium hydroxide as chemical activating agent for the removal of 2-cp.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

Table 3.1: Properties of materials used and their applications

Reagents	Molecular Weight, g/mol	State of material	Application
Potassium Hydroxide	56.1056	Liquid	Carbon Activation
Hydrochloric Acid	36.4609	Liquid	Pre-treatment of adsorbent
Hydrogen Peroxide	34.0147	Liquid	Pre-treatment of activated carbon
Activated carbon	-	Solid	Preparation of adsorbent
Nitrogen Gas	14.007	Gas	Carbon Activation
2-Chlorophenol	128.56	Liquid	Adsorption process

3.2 Method

Figure 3.1 summarises the overall methodology in order to extract activated carbon from waste rubber tires and preparation of adsorbent

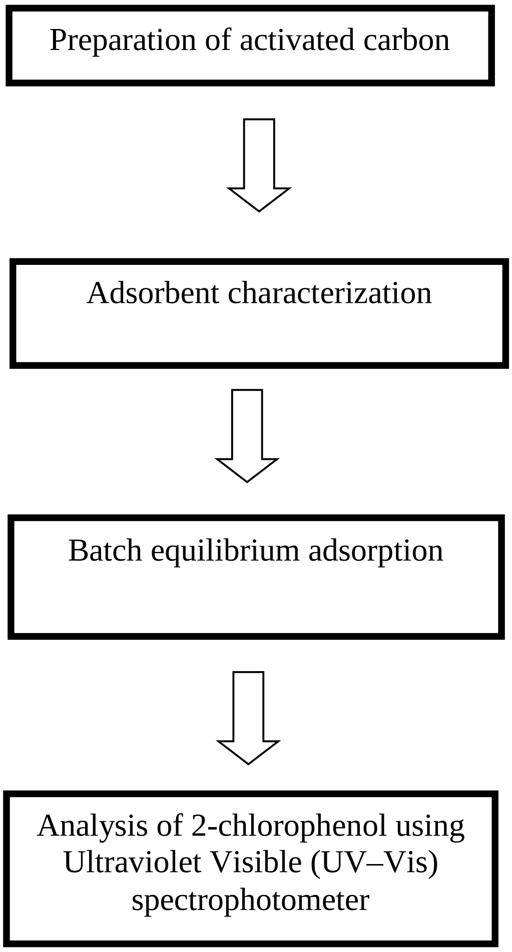

Figure 3.1: Flowchart of methodology

3.2.1 Preparation of activated carbon

In this work, the activated carbons produced from the waste materials were prepared by pyrolysis followed by chemical activation with alkali metals. Pyrolysis was carried out in an electrical furnace. The reactor will be fully instrumented in terms of gas flow control, furnace temperature control and temperature monitoring throughout. Briefly, waste rubber tires will be cleaned by removing the iron wires from the tire. Then, the waste rubber tires cut into small pieces and washed thoroughly with deionized water, and then dried in an oven at 100 °C for 2h (Tawfik, 2016). Then, the granules heated up to 300 °C in furnace for 3 h to separate the black tire crude oil, distilled diesel oil and produced oil.

As shown in figure 3.2, the waste tyre held in a sample crucible within the furnace and heated at a temperature of 500^{o}C for 5 h in the presence of nitrogen gas to break down the cross-linkage between carbon atoms (200 ml min$^-$1). Typical characteristics of the derived pyrolysis char have been reported previously with the char consisting of 78.8 wt.% fixed carbon, 2.0 wt.% volatiles and a high ash content of 18.9 wt. %. Elemental analysis of the char will be 70.1 wt. % carbon, 0.3 wt. % hydrogen, 0.83 wt. % nitrogen and 4.78 wt. % sulphur (Al-Rahbi, 2016).

Then, the chars recovered from the pyrolysis reactor oxidized all adhering impurities on the surface of the adsorbent upon treatment with a hydrogen peroxide solution. For this purpose, the product treated with H_2O_2 (6% concentration) with a ratio of 1 g/20 mL carbon/H_2O_2. The mixture (carbon and acid solution) stirred and heated at 60^{o}C for 24 h in oven. Then, the chars washed thoroughly with deionized water and dried at 110^{o}C for 2h.

The chars ground and sieved to 1.5–2mm particle size and dried at a temperature of 105^{o}C overnight. Although a wide variety of activating agents are known, using KOH in making carbons has become popular in recent studies (Sun, 1997). The char physically soaked with KOH at 30^{o}C for 30 minutes with a weight ratio of 1:3. Chars dried in oven at 60^{o}C for 2h. The activations was take place at an activation temperature of 700^{o}C in the presence of nitrogen gas (200 ml min$^-$1). In this case, a higher activation temperature will be chosen for treating samples so that a shorter activation period could be applied. The activation period was set at 2h 30 minutes. The authors also reported that a smaller particle size (0.4 mm) of the tyre rubber will be used and favourable as it would allow good surface contact with KOH (Edward, 2004).

The impregnation ratio = weight (KOH) (g) / weight (char) (g)

Further modification was accomplished via HCl treatment in order to develop oxygen surface groups on the rubber tire carbon. For this purpose, the product treated with HCl (37M concentration). The mixture (carbon and acid solution) stirred and heated at 100°C for 24 h. Then, it washed thoroughly with deionized water and dried at 100°C (T.Saleh, 2013).

Figure 3.2: Activated carbon extracted from waste tyres

3.2.2 Adsorbent characterization

Analysis done to evaluate carbon bond formed in adsorbent using certain instruments for reaction study. Adsorbent characterisation is important as it enables the study of the adsorbent's chemical & physical properties and their effects in reaction kinetics under various conditions. The following are the instruments used in the catalyst characterisation part.

3.2.2.1 Brunauer-Emmet-Teller (BET)

The textural properties such as surface area, pore volume and average pore diameter of the developed adsorbent characterized by nitrogen adsorption–desorption isotherms at −197◦C using Micrometrics ASAP 2020 surface area and porosity analyser (Micrometrics Instruments Corporation, USA) (Khayoon, 2013). Prior to nitrogen adsorption, samples were prepared on the degassing unit of the device for removal of water from the surface of the carbon. All the products were outgassed at 120^{o}C for 4h, which would not result any structural changes on the samples.

3.2.2.1 Field Emission Scanning Electron Microscope (FESEM)

FESEM is used to produce images of a sample by scanning it with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that can be detected provides information about the sample's surface topography and composition. Activated carbon sent to the Central Lab of University Malaysia Pahang to be scanned. The analysis instrument that will be used there is the JEOL/JSM-7800F Thermal FESEM instrument with accelerating voltage employed to be in the range of 5-15 kV. Figure 3.9 shows an example of an image taken by the FESEM unit.

Figure 3.3: Field Emission Scanning Electron Microscope

3.2.3.4 Fourier Transform Infrared (FTIR)

The performance of surface functional groups will be identified. The IR spectra for the synthesized c will be collected on Perkin- Elmer System 2000 spectrometer using the KBr disk method. The spectra will be recorded in the range of 2000–400 cm−1 (Khayoon, 2013)

Figure 3.4: Fourier transform infra-red spectrometry

3.2.3.5 X-ray diffraction XRD

X-ray diffractometer (XRD) is an instrumental technique which involves phase identification, and quantitative and qualitative analysis of materials. It is based on the interference of monochromatic x-rays and crystalline sample (Cullity & Stock, 2001). Figure 3.8 shows the schematic of XRD. The generated rays were initially generated by a cathode ray tube (F), and the x-rays will be filtered to produce monochromatic radiation (SS1) aligned and concentrated towards the sample (S). The interference produced (SS2) were corresponded to Bragg's Law (Equation 3.1) (Lachance & Claisse, 1995). After detecting (AS) the diffracted x-rays, the sample was scanned through a range of 2θ angles to identify all possible diffraction directions of the sample's crystal lattice structure.

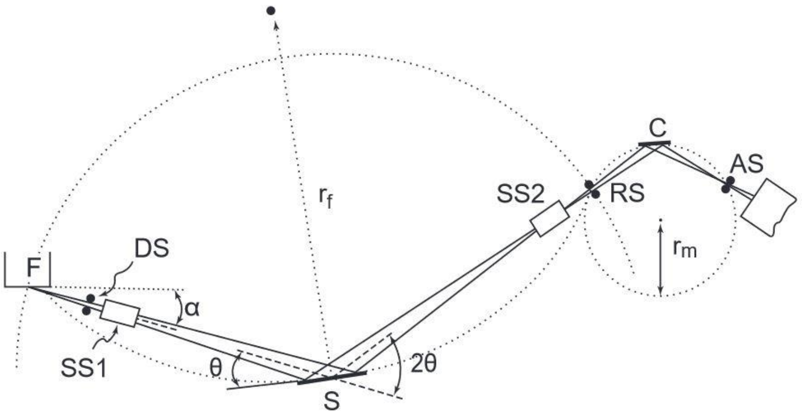

Figure 3.5: Bragg-Brentano parafocusing geometry (Jenkins, 2000)

The mean crystallite size can be calculated via the Scherrer equation (Equation 3.2)

$$D = \frac{k_{Sch}\lambda}{\beta_d cos\theta} \tag{3.2}$$

Where D is the crystallite size (Å), k_{Sch} is the Scherrer constant, λ is the wavelength is x-ray, β_d is the angular width if half-maximum intensity (degree) and θ is Bragg's angle (degree). In current research, the samples were irridated by Ni-filtered CuKα with a wavelength of 1.542 Å at 40mA and 45kV and scanning was done from 10^{o}C (283K) to 80^{o}C (353K) at 4 min^{-1}. The XRD equipment that was used was Rigaku Miniflex II.

The crystalline structure of the catalyst may be determined by the following method suggested by (Cullity & Stock, 2001). Each of the four common cubic lattices types is recognizable by their characteristic sequence of diffraction lines, and these in turn are explained by their sequential s values.

Simple cubic: 1, 2, 3, 4, 5, 6, 8, 9, 10, 11, 12, 13, 14, 16, ...
Body-centered cubic: 2, 4, 6, 8, 10, 12, 14, 16, ...
Face-centered cubic: 3, 4, 8, 11, 12, 16, ...
Diamond cubic: 3, 8, 11, 16, ...
For simplicity, the sequences are explained in the Figure 3.8

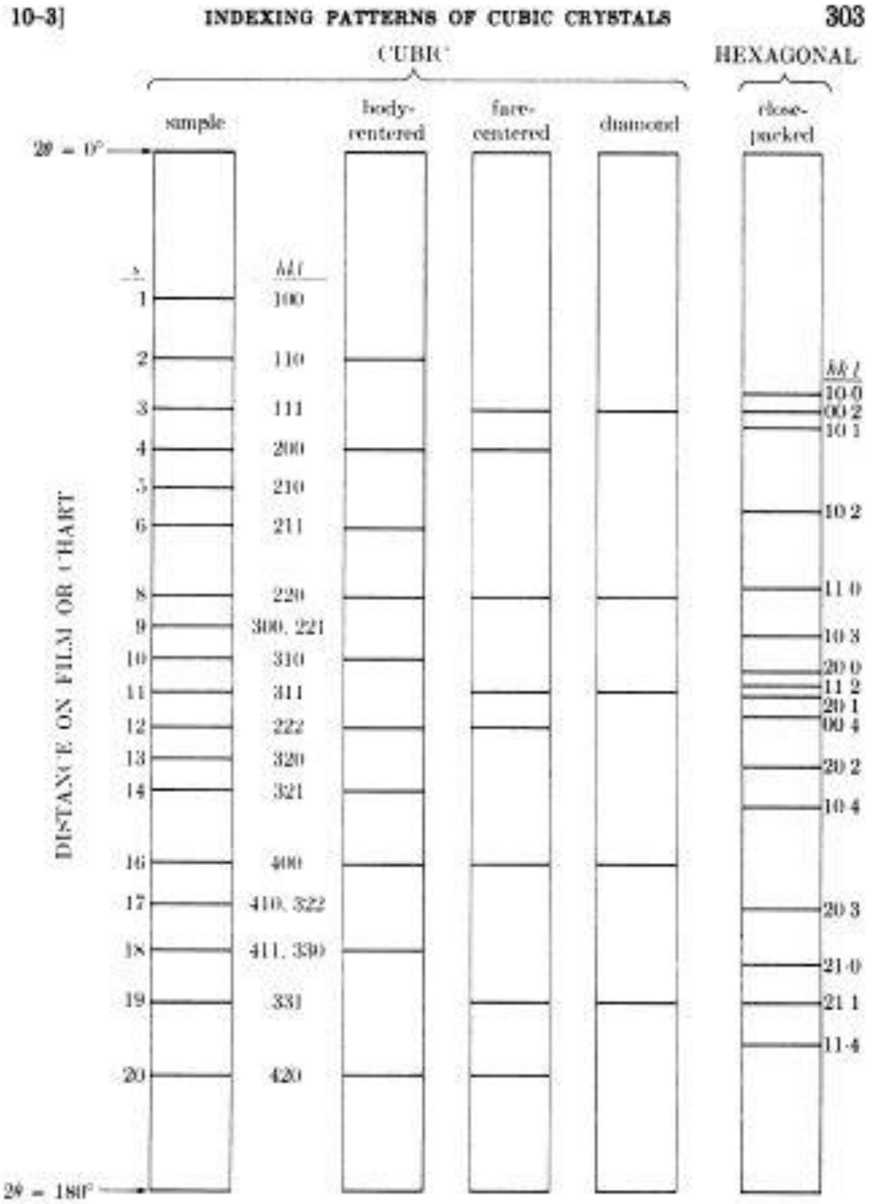

Figure 3.6: Calculated diffraction patterns for various lattices (Cullity & Stock, 2001)

Table 3.2: Summary of analytical instruments used

NO	Application	Purpose	Location
1	Brunauer-Emmet-Teller (BET)	• Surface area, pore volume, average pore diameter will be characterized	FIST Lab
2	Field Emission Scanning Electron Microscope (FESEM)	• Surface morphology of the developed adsorbent	Central Lab
3	Fourier Transform Infrared (FTIR)	• Performance of surface functional groups identified • Change in modified surface of adsorbent	FKKSA Lab
4	X-Ray Diffraction (XRD)	• Crystalline structure of adsorbent investigated	FIST Lab
5	UV–vis diffuse reflectance spectroscopy	• Concentration removal of 2-chlorophenol	FKKSA LAB

3.2.4 Adsorption of 2-chlorophenol

The equilibrium adsorption studied by using batch mode adsorption. A constant mass of adsorbent (0.1 g) was contacted with 50 mL of 2-CP solutions of different initial concentration from 10 to 100 mg/L. The bottles were sealed and placed in a shaker until equilibrium was reached. The time required for reaching an equilibrium 2-chlorophenol concentration is the equilibrium time for adsorption. Samples were then withdrawn by syringe, filtered through 0.22m syringe filter, diluted to the appropriate level.

The effect of initial pH (3–10) on the adsorption of the 2-CPs by AC was conducted by adjusting the solution pH with 0.1M HCl and 0.1M KOH solutions and measured using a pH metre (Martini instrument, Mi 150). The 2-CPs initial concentration was 10mg/L with AC dosage of 0.1g at temperature of 30◦C. Experiment repeated by varying the adsorbent dosage at (0.05g, 0.1 g, 0.3g and 0.5g) with constant concentration of 10mg/L at pH 5. The percentage removal of CPs (R%) calculated using Eq 1.

3.2.5 Analysis of 2-Chlorophenol

Samples were analysed using a Varian Cary 1E UV/Vis spectrophotometer at their maximum wavelength of 284nm. The percentage removals (% R) is to determine the residual equilibrium liquid-phase 2-CP concentration. The equilibrium adsorption capacity, qe (mg/g), at different 2-CP concentrations. The percentage removals (% R) of the adsorbates (CPs) were calculated using Eq. (1).

$$\mathrm{CPsremoval}\ (\%) = \frac{\mathrm{Co} - \mathrm{Ce}}{\mathrm{Co}} \qquad Equation\ 1$$

where Co and Ce are the liquid-phase concentrations at initial and equilibrium states (mg/L), respectively. The equilibrium amount of CPs adsorbed per unit mass of adsorbent, qe (mg/g), was calculated by Eq. (2):

$$qe = \frac{(\mathrm{Co} - \mathrm{Ce})\mathrm{V}}{W} \qquad Equation\ 2$$

where qe (mg/g) is the equilibrium amount of adsorbate (CPs) adsorbed per unit mass of adsorbent (AC) ; V (L) is the volume of the solution and W(g) is the mass of the adsorbent used.

Figure 3.7: UV-Visible spectrophotometer

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Overview

This chapter details out the results and discussions of the adsorption of 2-CP using activated carbon from waste tires as adsorbent. Adsorbent characterization will be discussed using established techniques such as, X-Ray Diffraction (XRD), Field Emission Scanning Microscopy (FESEM), Fourier Transform Infrared (FTIR) and BET Surface Area Analysis. The reaction study will be outlined the optimal conditions for the adsorption process to take place.

4.2 Adsorbent Characterization

4.2.1 X-Ray Diffraction (XRD)

X-Ray Diffraction was employed for phase identification in terms of qualitative and quantitative analysis on the fresh adsorbent. The XRD analysis was done using the Rigaku Mniflex II with the notion of determining the adsorbent's crystalline phase. Figure 4.1 shows the complete pattern of adsorbent.

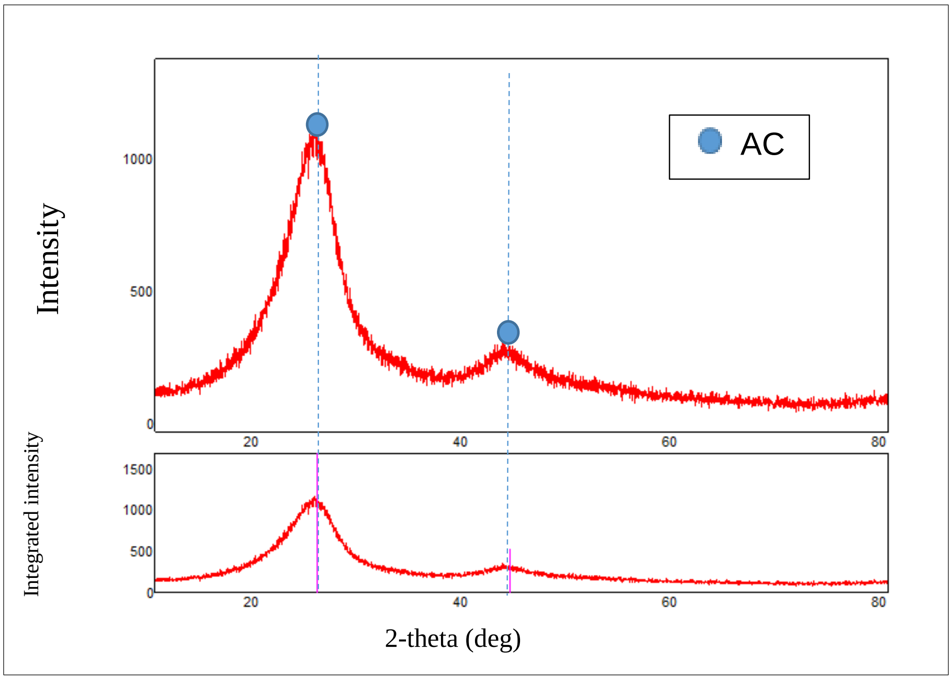

Figure 4.1: Complete XRD patterns for Activated Carbon

Based on Figure 4.1, pattern exhibits peaks similar to crystalline structures. Peaks at 20-30^{o} and 40-50^{o} indicated presence of AC which is disorderly stacked up by carbon rings. Thus, the activated carbon obtained can be identified as the carbon with amorphous structure, there are massive microcrystal with turbostratic graphite structure in the activated carbon prepared, and the two obvious diffraction peaks represent, respectively.

It can also be seen from figure, in the diffraction spectrum of the activated carbon prepared, its microcrystalline crystal face appears at 2θ = 25.43 and 43.89. From Bragg equation $2dhkl\sin\theta = n\lambda$, we can see that plane spacing d of diffraction crystal face for activated carbon is relatively larger, indicating that the turbostratic degree of the activated carbon prepared is relatively apparente. The (1 0 0) crystal surface peak of activated carbon prepared is more gentle and stronger.

As analyzed above, the microcrystalline structure of activated carbon tended to mess stratification and carbon structure appears disordering, thus more developed pore structure of the activated carbon was formed, at the same time, the adsorptive ability was enhanced (Tang, 2012).

4.2.2 Field Emission Scanning Microscopy (FESEM)

The morphology of the activated carbon produced from waste rubber tires was characterized by FESEM, Fig. 4.2.

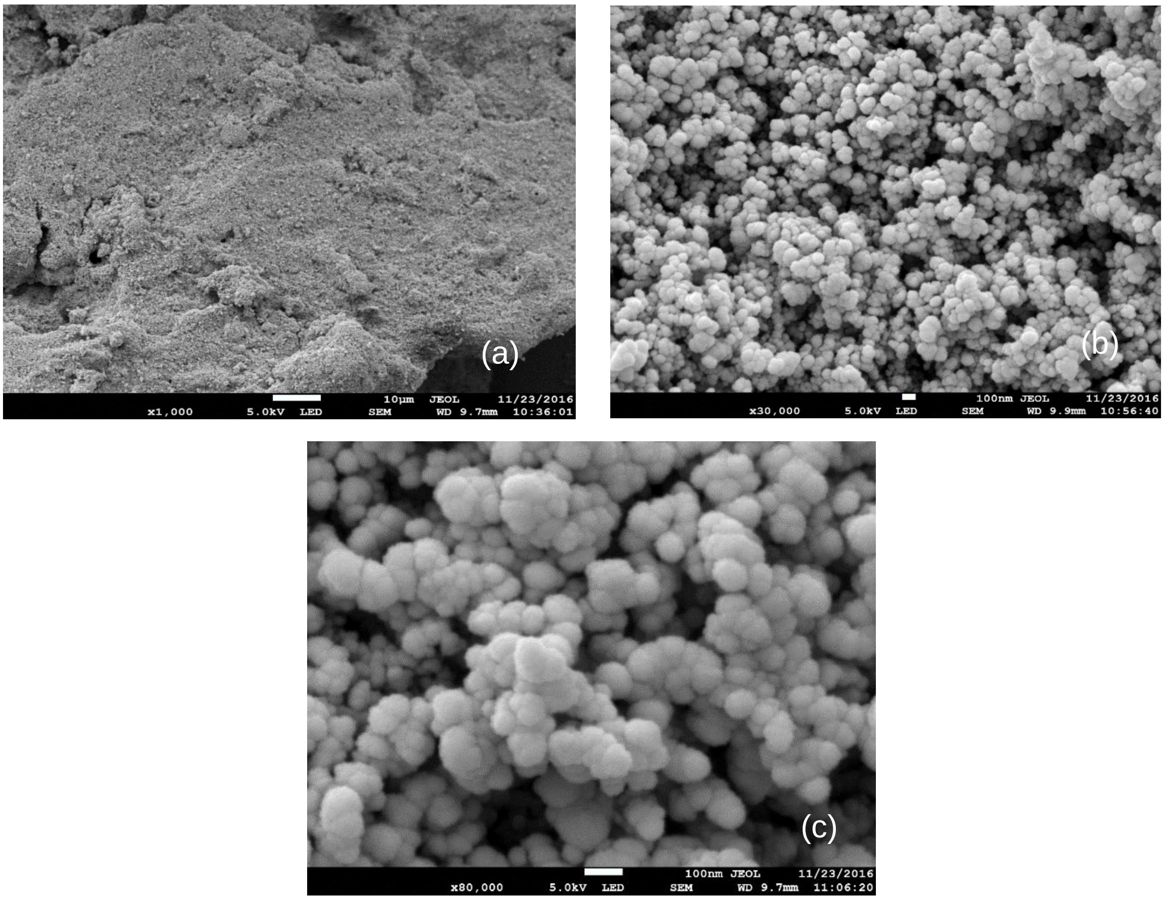

Figure 4.2: FESEM images under magnification (a) 1000x (b) 30000x and (c) 80000x

The FESEM image shows the porous structure of the activated carbon. The image exhibits three distinct morphologies. First morphological region is granular (the granules are 0.5 μm diameter), and second and third morphological region can be observed in the image (the granules are 100nm diameter respectively). High resolution of the image of the developed activated carbon indicates that there are different pore structures in the adsorbent. The basic parameters for an effective adsorbent are high surface area and pore structure. When the porosity increases, the surface area also increases (T.Saleh, 2013).

4.2.3 Fourier Transform Infrared (FTIR)

FTIR spectroscopy is an analytical tool for qualitatively determine the chemical changes and functional group in the adsorbent. The FT-IR spectrum of AC adsorbent is illustrated in Fig. 4.3

Table 4.1: Functional group according to wavenumber (Li et al., 2008)

Wavenumber (cm^{-1})	Functional Group
1030 – 1155	C- O
1550 - 1650	NH_2
600 - 400	Aromatic ring
2500 – 3500	O-H
2880 – 2500	C- H
3300 - 3500	N-H

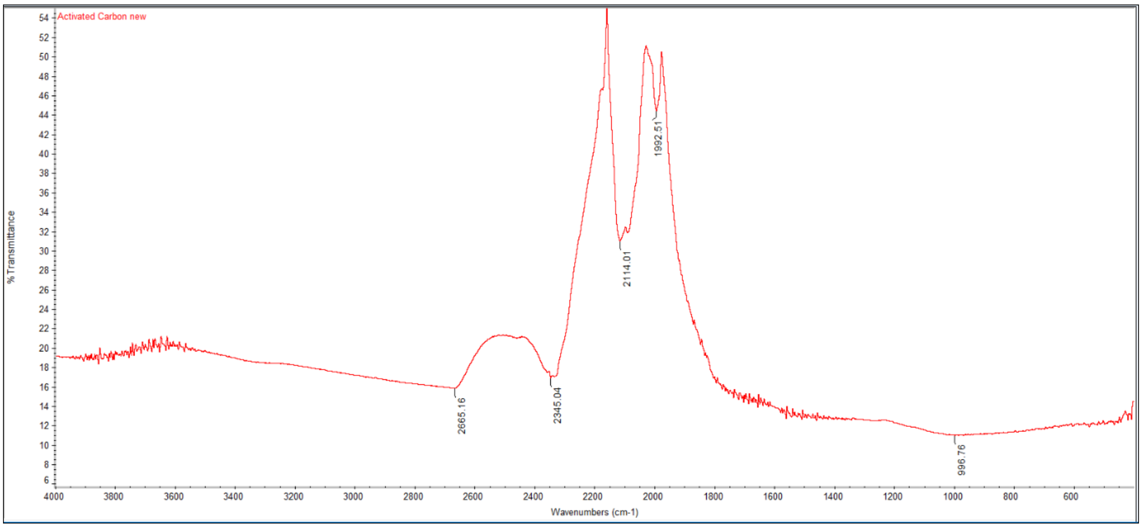

Figure 4.3: FTIR spectrum of AC

Figure 4.3 shows FTIR spectrum of AC. The first peak at 2665.16 cm^{-1} indicates the strong bonded functional group of O-H and C-H stretching absorption. Next, the band centered on 1992.51 cm−1 indicates enhancement in the aromatic C=C groups (carbonization). Then the peak at 996.76 cm^{-1} represents C-O stretching functional group. Fingerprint region occurs towards the end. Fingerprint region is the region consists of absorptions due to all other single bonds (except H-Z), making it often a complex region that is very difficult to analyze.

4.2.4 Brunauer–Emmett–Teller (BET) surface area measurements

The Brunauer-Emmett-Teller (BET) specific surface area of AC was obtained from the results of N_2 adsorption/desorption measurements at 77 K using Thermo-Scientific Surfer. Figure 4.4 shows the adsorption/desorption isotherms for the researched adsorbent. Based on the isotherm data, the surface area, pore volume and pore diameter were determined with the equations covered in Chapter 3.

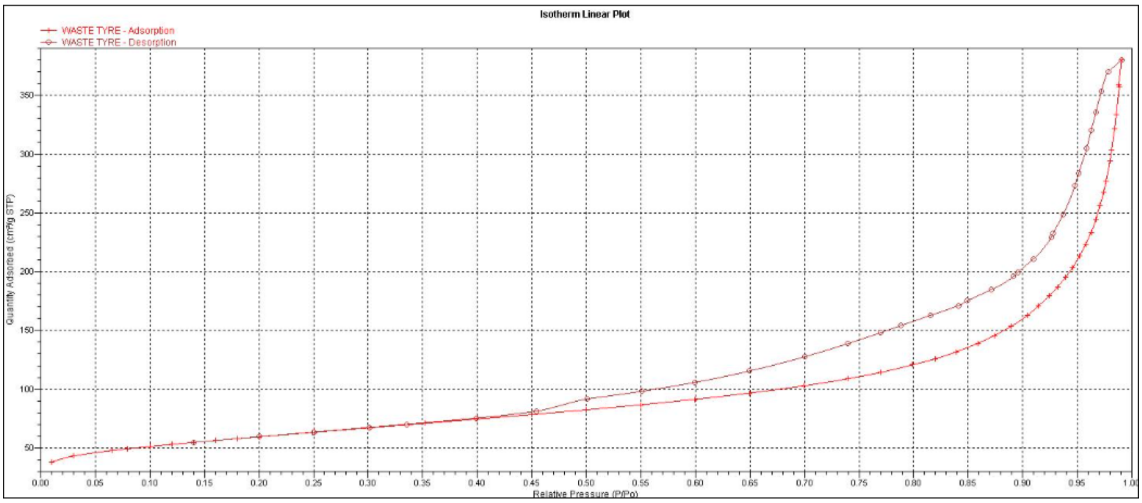

Figure 4.4: Typical adsorption/desorption isotherm for AC

The isotherms produced by AC showed type IV adsorption characteristics according to IUPAC classification (Gao, 2015). The hysteresis loop was determined to be type H3 due to the type of capillary condensation taking place in its mesopores. Type IV isotherm is usually attributed to monolayer-multilayer adsorption since it follows a similar part of the corresponding part of a Type II isotherm obtained with the given adsorptive on the similar surface area as the adsorbent in a non-porous form (Sing, 1982). Table 4.2 summarizes the BET surface area and pore volume and pore diameter of the researched adsorbent. The BET specific surface area was analyzed using the BET method while the pore volumes and average pore diameters were computed by BJH method which analysis result was available along with the BET test results. According to the results, AC possesses high specific surface area and pore volume compared the other adsorbents. However, the average pore diameter for the current researched adsorbent is slightly lower than other adsorbents.

Table 4.2: Estimates Of BET Specific Surface Area, Pore Volume % Average Pore Diameter for Adsorbents Researched

Adsorbents	BET specific surface area (m^2/g)	Pore volume (cm^3/g)	Average pore diameter (Å)	Researchers
AC	62.3489	0.9592	615.383	(T.Saleh, 2013)
ACZnO	15.1786	0.3041	801.6115	(T.Saleh, 2013)
ZrSAC4	115.07	0.08	N/A	(P.Suresh, 2014)
AC	207.8876	0.581670	126.476	Current Research

4.3 Screening of the Best Range of Concentration, pH and Adsorbent dosage on removal of 2-chlorophenol

This study is done to determine the best range for all parameters for further optimization process. Basically, the low and high levels of every parameter were examined. The parameters involved in this study were Concentration, pH and enzyme concentration.

4.3.1 *Effect of Concentration*

The effect of concentration on the adsorption capacity of chlorinated hydrocarbons by using AC at different concentrations is illustrated in Figure 4.5. The adsorption of chlorinated hydrocarbons rapidly increased with contact time and then proceeded at a slower rate until the equilibrium was achieved. The rapid adsorption was achieved at 10 mg/L. The adsorption process takes place rapidly at the initial step on the external surface of the adsorbent followed by a slower internal diffusion process (Gialamouidis, 2010). The rapid adsorption at the initial step is due to the presence of a large number of vacant active sites observed after a certain time indicated that the uptake of the solute molecules was made difficult due to the repulsive forces between the solute molecules on the surface of the AC as well as in the bulk phases. Thus it takes some time to reach equilibrium (Mall, 2005).

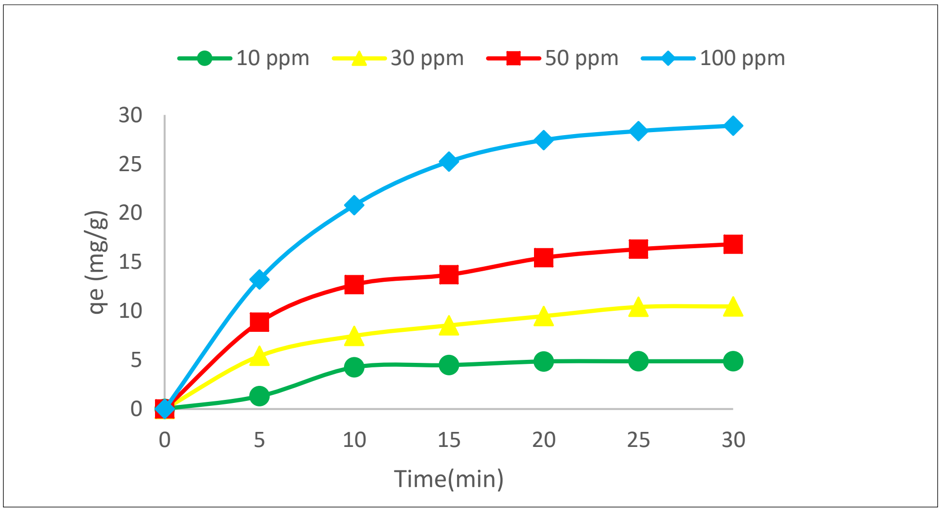

Figure 4.5: Effect of concentrations

4.3.2 *Effect of pH on adsorption*

Figure 4.6 indicates the influences of pH on adsorption of 2-CP. Removal of chlorinated hydrocarbons were examined in pH range between 3 and 10. Adsorption was attributed to the acidic character of the solute molecules. In terms of pH, it signifying that endothermic nature of the process with acidic solution pH being the most favorable for the adsorption where its capacity increase at pH 5 because 2-CP is a weak acid and it associated with the electron withdrawal effect of the chlorine substitution on the aromatic ring thus reducing the overall electron density of the aromatic ring of the adsorbate (László, 2004).

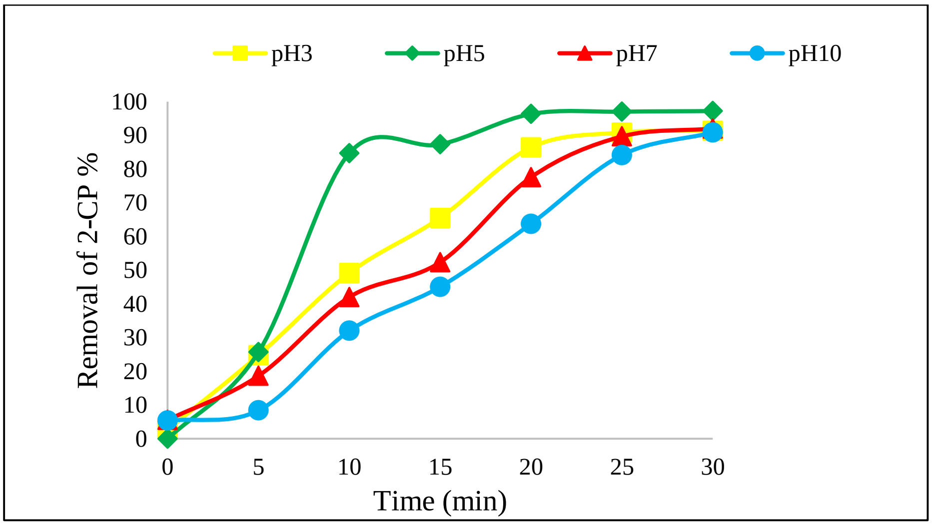

Figure 4.6: Effect of pH on removal efficiency of 2-CP

4.3.3 *Effect of adsorbent dosage*

The amount of the adsorbent is considered a significant factor because it can identify the capacity of the adsorption for a given initial concentration of the adsorbate. The effect of AC dosage on the adsorption of chlorinated hydrocarbons illustrated in Figure 4.7. Various adsorbent dosage utilized for adsorption of 2-CP at initial concentration 10 mg/L, temperature 25 °C and shaking time 30 min. Figure 4.8 shows that as the adsorbent dosage increased, the percent removal of chlorinated hydrocarbons increased from 96.98% to 99.43%. As seen in figure below, increase in adsorption sites for a constant concentration relatively increase the performance on AC on adsorption of 2-CP.

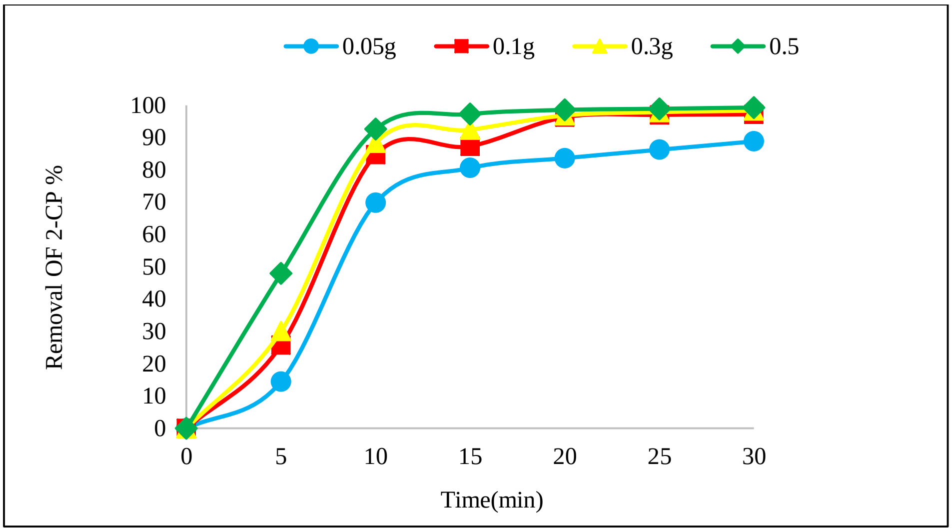

Figure 4.7: Effect of adsorbent dosage on removal efficiency of 2-CP

5 CONCLUSION AND RECOMMENDATIONS

5.1 Overview

This chapter provides the overall conclusions to the current research based on significant results obtained from adsorbent physicochemical studies and reaction studies of AC. Besides that, recommendations for future research were also included.

5.2 Conclusions

This research has notably demonstrated that wet impregnation-synthesized AC adsorbent was effective for removal of chlorine from 2-chlorophenol.

XRD patterns exhibited peaks which indicated presence of AC and through further analysis it was deduced that the crystalline structure of the adsorbent is face-centered cubic (FCC). Furthermore, N_2 physisorption results showed that the pore volume and average pore diameter of the researched adsorbent is relatively small compared to other researched adsorbents which confirms that the type of alumina used consisted mainly of alpha typed particles. The BET surface area was the highest. The adsorbent was found to show type IV adsorption FESEM images also supported the BET surface area results and at the same time illustrated the amorphous crystal structure of the researched adsorbent. In addition, FTIR analysis indicates the strong bonded functional group of O-H and C-H stretching absorption.

Adsorption studies showed that rapid adsorption was achieved at 10 ppm (mg/L) whereby it removed high amount of 2-CP. In terms of pH, it signifying that endothermic nature of the process with acidic solution pH being the most favourable for the adsorption where its capacity increase at pH 5 because 2-CP is a weak acid and it associated with the electron withdrawal effect of the chlorine substitution on the aromatic ring. Performance of AC effective at 0.5g of adsorbent dosage where it AC removed larger amount of chlorine from 2-CP. The studies reveal that AC derived from waste rubber tires possess high adsorption capacity hence it could be employed effectively as low-cost adsorbents for removal of chlorophenols.

5.3 Recommendations

Based on the findings from this research, some suggestions are proposed in order to explore and strengthen the prospects of adsorption of 2-CP. The suggestions are as follows;

1. In the current study AC was used as the adsorbent. It is suggested that different types of promoters to be researched for this process as well such as other group metals such as Zn, Zr and cerium in order to obtain the best yields. Similar work has been done for sorption of DCM using AC/ZnO by (Tawfik, 2015) and it is suggested that similar methods to be tried for adsorption.

2. In the current study, adsorbent characterization and reaction studies were researched. Nevertheless, more research could have been done on the adsorbent studied. It is suggested that used adsorbent characterization and longevity tests to be explored to reinforce this adsorbent’s credibility.

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7.1 APPENDICES

Activation of Carbon

The char was physically mixed with the potassium hydroxide, KOH with a weight ratio of 1:3.

The impregnation ratio = weight (KOH) (g) / weight (char) (g)

$$Chars\ = 7.5368g$$

$$KOH = 7.5368\ \times 3 = 22.6104g$$

$$H2O = \frac{22.6104}{0.428}\ -22.6104 = 30.2176ml$$

$$\approx 31ml$$

- 31 ml of H_2O diluted with KOH for the activation of carbon at 700^{o}C for 2h 30 minutes.

HCl treatment

HCl treatment in order to develop oxygen surface groups on the rubber tire carbon. For this purpose, the product treated with HCl (37M concentration).

Specific Gravity,SG of HCl = 1.18

Moleculat weight of HCl = 36.5 g/mol

Assume 300ml of HCl,

$$\frac{37}{100}\times\frac{1.18}{L}\times\frac{mol}{36.5g} = 11.96 \approx 12\ \frac{mol}{L}$$

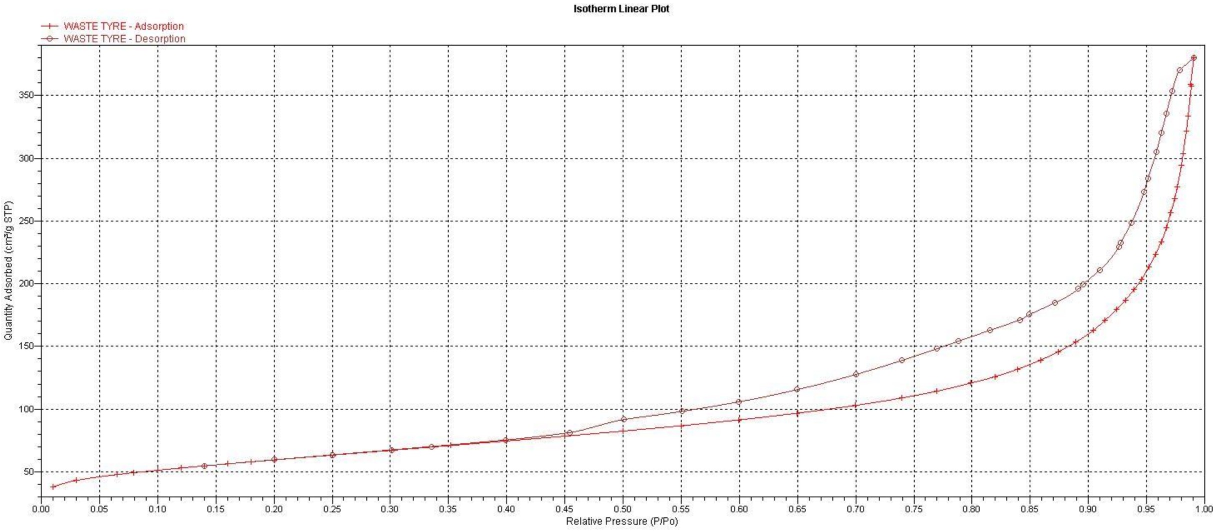
$$M1V1 = M2V2$$

$$12MV1 = 1M(300ml)$$

$V1 = 25ml$ (25ml of water needed for the dilution of HCl)

7.2 Adsorbent Physicochemical Data

Brunauer-Emmet-Teller (BET)



Full Report Set

ASAP 2020 V3.04 H Unit 1 Serial #: 1255 Page 1

Sample: WASTE TYRE

Operator: HALIM

Submitter: UMP

File: C:\2020\DATA\001-745.SMP

Started: 12/2/2016 5:43:33PM Analysis Adsorptive: N2

Completed: 12/3/2016 12:25:19AM Analysis Bath Temp.: -195.798 °C

Report Time: 12/5/2016 8:54:02AM Thermal Correction: No

Sample Mass: 0.1104 g Warm Free Space: 26.9547 cm³ Measured

Cold Free Space: 81.4976 cm³ Equilibration Interval: 5 s

Low Pressure Dose: None Automatic Degas: Yes

Summary Report

Surface Area

Single point surface area at P/Po = 0.301047861: 204.2270 m²/g

BET Surface Area: 207.8876 m²/g

Langmuir Surface Area: 327.2321 m²/g

t-Plot Micropore Area: 23.9874 m²/g

t-Plot External Surface Area: 183.9002 m²/g

BJH Adsorption cumulative surface area of pore between 17.000 Å and 3000.000 Å diameter: 183.962 m²/g

BJH Desorption cumulative surface area of pores between 17.000 Å and 3000.000 Å diameter: 225.5099 m²/g

Pore Volume

Single point adsorption total pore volume of pores less than 1248.743 Å diameter at P/Po = 0.984250780: 0.497927 cm³/g

Single point desorption total pore volume of pores less than 922.562 Å diameter at P/Po = 0.978555023: 0.572724 cm³/g

t-Plot micropore volume: 0.011641 cm³/g

BJH Adsorption cumulative volume of pores between 17.000 Å and 3000.000 Å diameter: 0.581670 cm³/g

BJH Desorption cumulative volume of pores between 17.000 Å and 3000.000 Å diameter: 0.597867 cm³/g

Pore Size

Adsorption average pore width (4V/A by BET): 95.8070 Å

Desorption average pore width (4V/A by BET): 110.1987 Å

BJH Adsorption average pore diameter (4V/A): 126.476 Å

BJH Desorption average pore diameter (4V/A): 106.047 Å

Full Report Set

```
ASAP 2020 V3.04 H    Unit 1    Serial #: 1255    Page  1

     Sample:    WASTE TYRE
   Operator:    HALIM
  Submitter:    UMP
       File:    C:\2020\DATA\001-745.SMP

     Started:    12/2/2016 5:43:33PM           Analysis Adsorptive:    N2
     Completed:    12/3/2016 12:25:19AM    Analysis Bath Temp.:    -195.798 °C
     Report Time:    12/5/2016 8:54:02AM   Thermal Correction:    No
     Sample Mass:    0.1104 g                  Warm Free Space:    26.9547 cm³   Measured
    Cold Free Space:    81.4976 cm³            Equilibration Interval:    5 s
     Low Pressure Dose:    None                Automatic Degas:    Yes
```

Isotherm Tabular Report

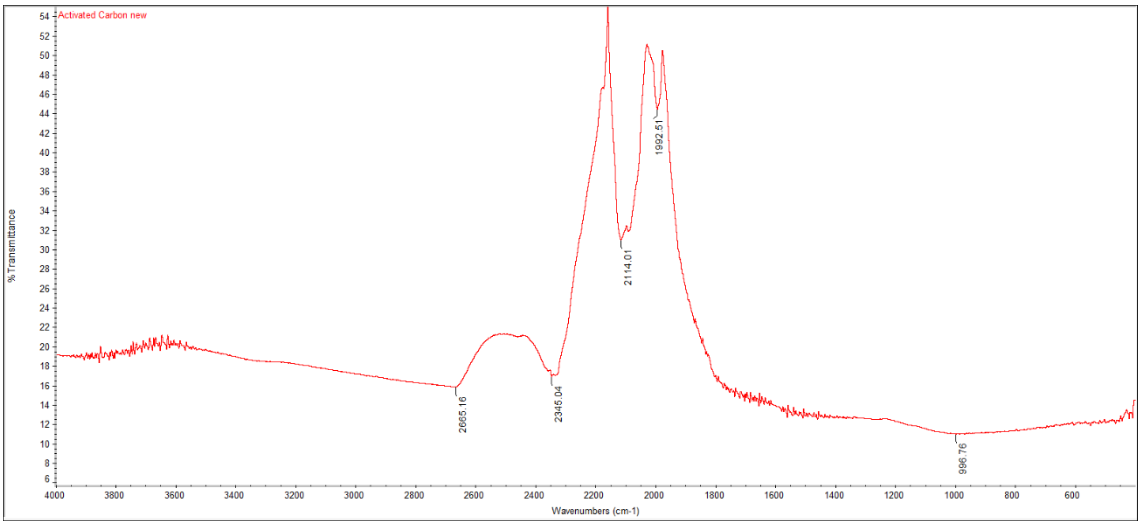
```
                              Quantity
   Relative      Absolute     Adsorbed    Elapsed    Saturation
   Pressure      Pressure      (cm³/g      Time       Pressure
    (P/Po)        (mmHg)        STP)      (h:min)      (mmHg)
-----
  1.#QNAN0000     1.#QNAN0      1.#QNB      01:08    759.266785
  0.009916231      7.530039     37.8989      01:22      1.#QNAN0
  0.029986144     22.771063     43.1255      01:25      1.#QNAN0
  0.064825307     49.228806     47.7882      01:28      1.#QNAN0
  0.079458225     60.342823     49.2946      01:31      1.#QNAN0
```

0.099968974	75.920670	51.2421	01:33	1.#QNAN0
0.120000836	91.135399	53.0316	01:35	1.#QNAN0
0.140002936	106.328087	54.7279	01:37	1.#QNAN0
0.160005709	121.522972	56.3478	01:40	1.#QNAN0
0.180083581	136.773209	57.9214	01:41	1.#QNAN0
0.200128884	152.000397	59.4747	01:43	1.#QNAN0
0.248755071	188.937836	63.1894	01:46	1.#QNAN0
0.301047861	228.662308	67.1208	01:49	1.#QNAN0
0.351759107	267.185211	70.9492	01:51	1.#QNAN0
0.399892725	303.751617	74.5803	01:53	1.#QNAN0
0.449787347	341.660187	78.4203	01:56	1.#QNAN0
0.499718975	379.595459	82.4623	01:58	1.#QNAN0
0.549740695	417.604431	86.7263	02:01	1.#QNAN0
0.599537162	455.440186	91.3861	02:03	1.#QNAN0
0.649486274	493.397858	96.7056	02:06	1.#QNAN0
0.699489979	531.399109	102.8875	02:09	1.#QNAN0
0.739469252	561.786743	108.8585	02:12	1.#QNAN0
0.769635011	584.714966	114.2554	02:14	1.#QNAN0
0.799350015	607.307190	120.6863	02:17	1.#QNAN0
0.819776740	622.843689	125.8443	02:20	1.#QNAN0
0.839230727	637.641968	131.8000	02:23	1.#QNAN0
0.859301285	652.909546	139.1215	02:26	1.#QNAN0
0.874441508	664.437866	145.7629	02:30	1.#QNAN0
0.889324968	675.765686	153.3897	02:33	1.#QNAN0
0.903868889	686.842468	162.6189	02:37	1.#QNAN0
0.914410457	694.878601	170.7648	02:41	1.#QNAN0
0.924495941	702.568726	179.3932	02:45	1.#QNAN0

0.931984774	708.279480	187.0028	02:48	1.#QNAN0
0.939488685	714.008606	195.0598	02:52	1.#QNAN0
0.945580231	718.658081	203.0941	02:55	1.#QNAN0
0.952092395	723.640869	213.1440	03:00	1.#QNAN0
0.958070541	728.211487	223.5061	03:04	1.#QNAN0
0.962650741	731.719849	233.4506	03:08	1.#QNAN0
1.#QNAN0000	1.#QNAN0	1.#QNB	03:10	760.123413
0.967018014	735.065674	244.5262	03:14	1.#QNAN0
0.970803288	737.955688	256.2840	03:18	1.#QNAN0
0.974400535	740.706055	267.8124	03:23	1.#QNAN0
0.976302584	742.161499	276.9927	03:26	1.#QNAN0
0.979932149	744.939819	294.2594	03:32	1.#QNAN0
0.981469342	746.121216	303.8680	03:36	1.#QNAN0
0.984250780	748.251770	321.9078	03:41	1.#QNAN0
0.985509272	749.224609	333.4781	03:46	1.#QNAN0
0.988440558	751.472473	357.3195	03:52	1.#QNAN0
0.987628998	750.858704	358.9161	03:53	1.#QNAN0
0.990378064	752.971375	380.0972	04:00	1.#QNAN0
0.978555023	743.992065	370.2636	04:03	1.#QNAN0
0.972370856	739.306152	353.5433	04:08	1.#QNAN0
0.967295548	735.463135	335.7095	04:13	1.#QNAN0
0.962877091	732.119385	320.0136	04:18	1.#QNAN0
0.958739987	728.992554	305.0226	04:24	1.#QNAN0
0.951523825	723.524292	283.9226	04:30	1.#QNAN0
0.948092431	720.930603	273.1993	04:35	1.#QNAN0
0.936780068	712.350098	248.2934	04:42	1.#QNAN0
0.927636322	705.415161	232.2846	04:48	1.#QNAN0

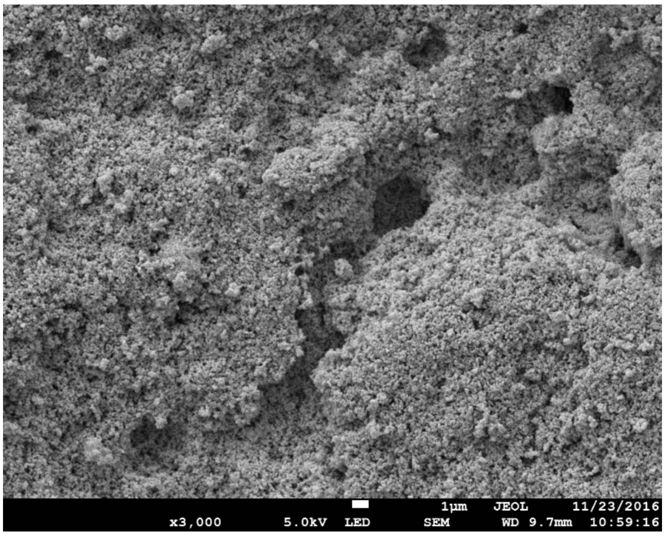
0.926222001	704.345703	229.2606	04:50	1.#QNAN0
0.909682121	691.788757	210.9675	04:57	1.#QNAN0
0.895764422	681.216431	199.3674	05:01	1.#QNAN0
0.891573730	678.038208	195.7396	05:04	1.#QNAN0
0.870899394	662.326843	184.7568	05:08	1.#QNAN0
0.848967061	645.658203	175.1834	05:12	1.#QNAN0
1.#QNAN0000	1.#QNAN0	1.#QNB	05:14	760.528625
0.841363521	639.881042	171.0631	05:16	1.#QNAN0
0.815252453	620.022827	162.8098	05:20	1.#QNAN0
0.788300005	599.524719	154.1117	05:23	1.#QNAN0
0.769972411	585.586060	148.0919	05:27	1.#QNAN0
0.739509752	562.418335	138.7328	05:31	1.#QNAN0
0.699980763	532.355408	127.6449	05:35	1.#QNAN0
0.649765848	494.165527	115.4705	05:40	1.#QNAN0
0.599701970	456.090515	105.7617	05:44	1.#QNAN0
0.550441672	418.626648	98.1431	05:47	1.#QNAN0
0.500424180	380.586914	91.6307	05:51	1.#QNAN0
0.454083508	345.343506	81.3400	05:56	1.#QNAN0
0.399164608	303.576111	75.3714	05:59	1.#QNAN0
0.335176371	254.911224	70.1672	06:02	1.#QNAN0
0.301379756	229.207932	67.5028	06:04	1.#QNAN0
0.250170539	190.261856	63.5017	06:07	1.#QNAN0
0.200230512	152.281036	59.5767	06:09	1.#QNAN0
0.140149180	106.587463	54.7250	06:14	1.#QNAN0

Fourier Transform Infrared (FTIR)

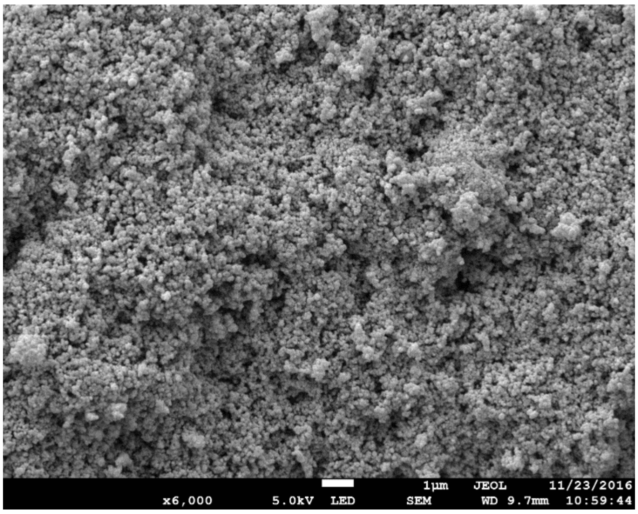


FESEM

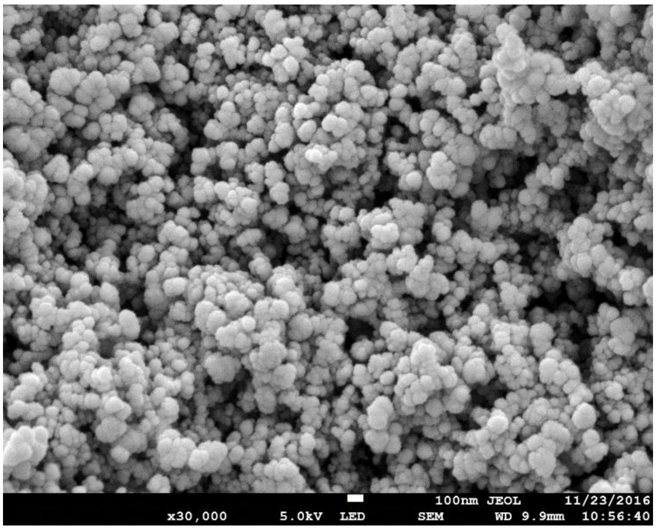
At 3,000 magnification



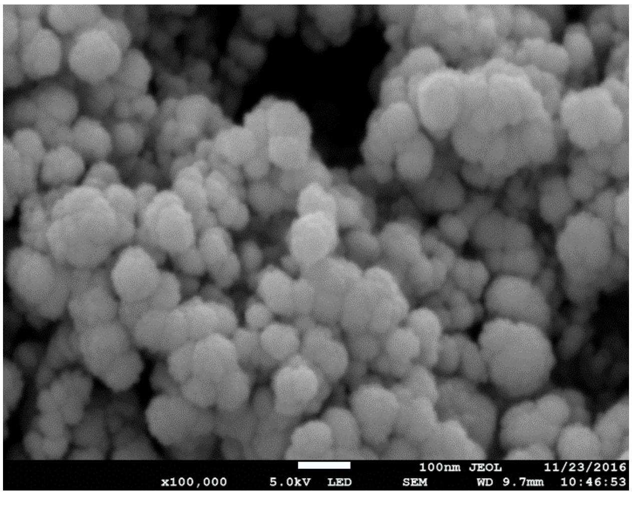
At 6,000 magnification



At 30,000 magnification



At 100,000 magnification



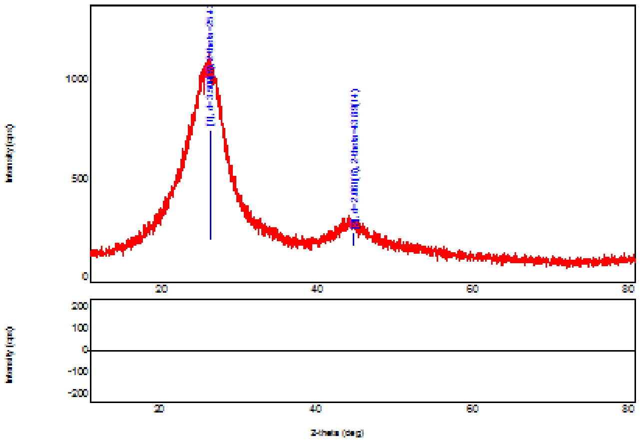
XRD

Analysis Results

General Information

Analysis date	11/5/2016 9:53:22 AM		
Sample name	XRD Analysis	Measurement date	2016/11/05 08:43:02
File name	AC (waste tyre).raw	Operator	Admin
Comment	UMP		

Measurement profile



Measurement conditions

X-Ray	30 kV , 15 mA	Scan speed / Duration time	1.0000 sec
Goniometer		Step width	0.0200 deg.
Attachment	-	Scan axis	2theta/theta
Filter		Scan range	10.0000 - 80.0000 deg.
CBO selection slit	-	Incident slit	0.625 deg.
Diffrected beam mono.	Fixed Monochromator	Length limiting slit	-
Detector	MiniFlex2 counter	Receiving slit #1	1.25 deg.
Scan mode	STEP	Receiving slit #2	0.3mm

Qualitative analysis results

Phase name	Formula	Figure of merit	Phase reg. detail	DB card number

Phase name	Formula	Space group	Phase reg. detail	DB card number