# Optimisation of Malachite Green Removal Using Activated Carbon Derived from Coconut Shell as Low Cost Adsorbent

By

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**DECLARATION**

I declare that this thesis entitled Optimisation of Malachite Green Removal Using Activated Carbon Derived from Coconut Shell as Low Cost Adsorbent is the result of my own research except as cited in the references. The thesis has not been accepted by any degree and is not concurrently submitted in any candidature of any degree.

Signature : _____

Name : _____

Date : _____

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| ppm | Part per million |
| DO | Dissolved oxygen |
| GAC | Granular activated carbon |
| PAC | Powder activated carbon |
| ACF | Activated carbon fibbers |
| ACC | Activated carbon cloths |
| TSS | Total suspended solid |
| COD | Chemical oxygen demand |
| BOD | Biological oxygen demand |
| Zn | Zinc |
| Cr | Chromium |
| MG | Malachite Green |
| $ZnCl_2$ | Zinc chloride |
| $H_3PO_4$ | Phosphoric acid |
| ANOVA | Analysis of variance |

## LIST OF SYMBOLS

| | |
|---|---|
| ºC | Degree celcius |
| mm | Milimeter |
| % | Percent |
| g | Gram |
| ml | Mililiter |

## ABSTRACT

Textile industry is a sub industry that contributes to economy of our country hence the industry keep growing from day to day. The processes that involved in textile industry are fibre production, spinning, twisting, weaving, knitting, scorching and dyeing which involved colours or dye. The combination of the processes and products make the waste water from textile plant contains many types of pollutants. The presence of smallest amount of dye in water (less than 1ppm) is highly visible and will affect the water transparency and gas (carbon dioxide, oxygen) solubility in the water body where the waste water discharged. Dyes are usually resistant to conventional biodegradation therefore adsorptions is an effective alternative for dyes removal treatment. Therefore a study has been conducted to find a cheap alternative method of treating textile waste water. In this study, the capability of activated carbon derived from coconut shell to adsorp malachite green dye was investigated. Three parameters were studied ie activated carbon with different chemicals impregnation and carbonization time, contact time and initial concentrations of dye. From the results acquired, 99.9% of malachite green dye was removed by the activated carbon impregnated with phosphoric acid solution of 5 minutes carbonization time. The optimum time contact and initial concentration of dye was 1 hour and 10 ppm respectively. Therefore, this result can contribute some knowledges using low cost raw material impregnation with some chemicals to remediate textile waste water.

## ABSTRAK

Industri tekstil merupakan industri sub yang menyumbang kepada ekonomi negara kita dengan itu industri terus berkembang dari hari ke hari. Proses-proses yang terlibat dalam industri tekstil adalah pengeluaran serat, berputar, berpusing, tenunan, mengait, terik dan pencelupan yang melibatkan warna atau pewarna. Gabungan proses dan produk menyebabkan air sisa dari kilang tekstil mengandungi pelbagai jenis bahan pencemar. . Kehadiran jumlah kecil pewarna dalam air (kurang daripada 1ppm) adalah amat ketara dan akan memberi kesan kepada kelarutan ketelusan air dan gas (karbon dioksida, oksigen) dalam badan air di mana air sisa dilepaskan. Pewarna biasanya sukar disingkirkan oleh rawatan konvensional oleh itu penjerapan adalah satu alternatif yang berkesan untuk rawatan pewarna penyingkiran. Demi mencari kaedah alternatif merawat tekstil sisa air terutama disebabkan pewarna yang jauh lebih murah, satu kajian telah dijalankan. Kajian ini dijalankan untuk mengkaji keupayaan tempurung kelapa untuk menjerap molekul pewarna hijau malachite daripada air sisa sintetik. Tiga jenis parameter yang dikaji; penjerap terbaik dengan kandungan bahan kimia dan masa karbonisasi, masa sentuhan dan kepekatan awal pewarna. Daripada keputusan yang diperolehi, 99.9% daripada pewarna hijau malachite telah berjaya disingkirkan oleh karbon diaktifkan dengan asid fosforik dengan masa karbonisasi selama 5 minit. Masa optimum dan kepekatan awal pewarna yang digunakan adalah 1 jam dan 10 ppm. Oleh itu, keputusan ini boleh menyumbang beberapa ilmu dalam menggunakan bahan mentah kos rendah yang diaktifkan dengan bahan kimia untuk merawat air sisa tekstil.

# CHAPTER 1

# INTRODUCTIONS

## 1.1 Background of study

Waste water from textile industry can contain variety of pollutants for example dyes. The main component used to produce textile is colour which is easily recognized in the waste water and has to be removed or treated from the waste water before discharging into water body. The effluents generated from industrial activities contribute as most sources of natural water pollution. This will cause huge problems in term of waste water management or treatment and can continuously lead to direct source pollution problem which not only consume high cost of treatment, but also introduce variety of chemical pollutants and microbial contaminants to water sources. The presence of smallest amount of dye in water (less than 1ppm) is highly visible and will affect the water transparency and gas (carbon dioxide, oxygen) solubility in the water body where the waste water discharged. Once the gas solubility affected, it will affect dissolved oxygen (DO) too. The amount of dissolved oxygen in water body is dependent on the temperature, amount of sediment, the amount of oxygen taken out of the water ecosystem by respiring and decaying organisms, and the amount of oxygen intake by photosynthesizing plants and aeration.

Nowadays, there are many types of technologies are used to remove contaminants from water sources such as filtration, chemical precipitation and ion-exchange. These methods can be either high cost or not efficient. It may need less time compared to use of activated carbon, which it have to be carbonized

conventionally but in term of performance to remove contaminants, it shows equal or even better result. The nature of microwave that heats the material completely makes it an ideal initiative for faster sample carbonizing. The electrical usage and size of material shown inversely proportional, giving this method more advantage over conventional carbonization (Wahi & Senghie, 2007).

The most commonly used adsorbent for dye removal is activated carbon which is also known as activated charcoal that contains small, low-volume pores that increase the surface area which can allow efficient adsorption process and chemical reactions. Activated carbon has the capability to adsorb broad range of adsorbents efficiently. The main material needed to produce activated carbon is any organic material with high carbon content such as coal, wood, peat and coconut shell. The raw material which is carbon-based is transformed to activated carbon by thermal decomposition in a furnace using certain heat and duration. Basically, there are two different processes for the preparation of activated carbon, the so-called physical or thermal and chemical activation (Yusufu et al., 2012). Activated carbons are widely used as adsorbents for the removal of organic chemicals and metal ions of environmental or economic concern from air, gases, potable water and wastewater (Al-Qodah *et al*., 2009).

The preparation of activated carbon using raw material or waste material divided into two kinds which are physical activation and chemical activation. In physical activation, there are two phase that start with carbonization, a chemical activation by saturating it with the strong acid or strong base such as sodium hydroxide, zinc chloride, phosphoric acid or phosphoric hydroxide depending on

what kind of material used to produce activated carbon and followed by activation using activating agent at temperature range of 500ºC to 1000ºC. The chemical activation acts as a dehydration reagent to lower the carbonization temperature during chemical activation, minimizing tar formation and facilitate in carbonization of carbon (Shaarani, 2010).

There are four types of physical form of activated carbon as for now, which are granular activated carbon (GAC), powdered activated carbon (PAC), activated carbon fibbers (ACF) and activated carbon cloths (ACC) (Tennant, 2004). Recent research found that few material to produce cheap activated carbons are from agriculture waste such as nutshells, fruit stones, coir pith, baggase, bamboo, rice husk and cotton stalks (Song *et al*., 2013). Transformation of coconut shell into activated carbon which play the role as adsorbents of textile effluents added value to this waste and at the same time reduce the cost of waste disposal (Song *et al*., 2013). By using this agriculture waste, we also can provide an efficient and cheap activated carbon alternative to expensive-existing activated carbon (Song *et al*., 2013).

## 1.2 Problem Statement

Textile industry is one of the industry that have very high risk in affecting the water quality by disposing the contaminants, products from the activity into water bodies, where organisms that live in there or organisms that utilising it as resources may be affected as well. The pollutants that frequent to be found in textile waste water are called recalcitrant, organics, colours and toxicants (Varsha *et al*., 2013). The textile effluents contain many types of chemical from the various phases of

process which include printing, scouring, bleaching and dyeing (Varsha *et al*., 2013). Toxicity, long-persistant, bioaccumulation and bio magnification presence in food chain could indicate that the water was heavily polluted. Dyes are usually resistant to conventional biodegradation therefore adsorptions is an effective alternative for dyes removal treatment.

Adsorptions using activated carbons obtained from agricultural waste for waste water treatment is way more effective to remove dye. Activated carbons also is widely used in water treatment because of its extended surface area, microporous structure, high adsorption capacity and highly reactive. The major problem occurred is the existing activated carbons in the market are very expensive (Nachiyunde, 2013). This issue make people tried to use activated carbon that originated from low cost material such as bamboo and coconut shell.

### 1.3 Objective

1. To prepare activated carbon that originated from coconut shell.
2. To determine the efficiency of activated carbon to remove malachite green in synthetic textile waste water.

### 1.4 Significance of study

This study will use coconut shells as a sources of cheap activated carbon to remove dye (malachite green) from synthetic textile waste water hence this research will be an alternative cheap treatment of textile waste water.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 Industrial waste water

Industrial effluents (including agricultural-based industrial waste water) are waste that result from anthropogenic activities which are related with raw-material processing and manufacturing. Industrial effluents is the liquid discard that aftermath from the appliance of water in an industrial manufacturing process or the cleaning stages that come along with the process . As mentioned by (Woodard & Curran Inc., 2011) , industrial waste water is the result from the process that have been dissolved or suspended in water. The waste water can originates from washing, cooking, cooling, heating, extracting, by product of a reaction, separation, conveyance and quality control that lead to product rejection (Ng et al., 2006). Industrial sewerage have very diverse combinations relying on the types of industry and materials processed (Ng et al., 2006). He also stated that part of the waste water can be essentially very strong, easily biodegradable, extensively inorganic or potentially inhibitory. This indicate total suspended solid (TSS), BOD and COD values may be in tens of thousands $mgL^{-1}$ (Ng et al., 2006). There are also characteristics of industrial waste water that need consideration; biodegradability, strengths, volumes, variations and special characteristics which may lead to operational difficulties (Ng et al., 2006).

### 2.1.1 Types of industrial effluents and its elements

There are several types of substances present in the waste water which depends on the type of industrial activities carry out (Table 2.1).

**Table 2.1**: Substances present in industrial effluents (Tennant, 2004)

| Substances | Present in waste water from: |
|---|---|
| Acetic acid | Acetate rayon, beet root manufacturer |
| Acids | Chemical manufacture, mines, textiles manufacturer |
| Alkalies | Cotton and straw kiering, wool scouring |
| Ammonia | Gas and coke and chemical manufacturer |
| Arsenic | Sheep dipping |
| Cadmium | Plating |
| Chromium | Plating, chrome tanning, alum anodizing |
| Citric acid | Soft drinks and citrus fruit processing |
| Copper | Copper plating, copper pickling |
| Cyanides | Gas manufacturer, plating, metal cleaning |
| Fats, oils, grease | Wool scouring, laundries, textile industries |
| Fluorides | Scrubbing of flue gases, glass etching |
| Formaldehyde | Synthetic resins and penicillin manufacturer |
| Free chlorine | Laundries, paper mills, textile bleaching |
| Hydrocarbons | Petrochemical and rubber factories |
| Mercaptans mills | Oil refining, pulp |
| Nickel | Plating |
| Nitro compounds | Explosives and chemical works |
| Organic acids | Distilleries and fermentation plants |
| Phenols | Gas and coke manufacturer, chemical plants |
| Starch | Food processing, textile industry |
| Sugars | Dairies, breweries, sweet industry |
| Sulphides | Textile industry, tanneries, gas manufacturer |
| Sulphites | Pulp processing, viscose film manufacturer |
| Tannic acid | Tanning, sawmills |
| Tartaric acid | Dyeing, wine, leather, chemical manufacturer |
| Zinc | Galvanizing, zinc plating, rubber process |

### 2.1.2 Textile waste water

Textile industry is one of the main industry that cause in degrading the water quality by disposing their effluents in water bodies that cause negative impacts to our environment (Walakira *et al*., 2005). The main sources of waste water come from cleaning water, pre-treatment, dyeing and round off process water non – contact cooling water (Arumai Dhas, 2008). The combination of the processes and products make the waste water from textile plant contains many types of pollutants. The dyeing and finishing operations are such that the dyestuffs, chemicals and textile auxiliaries used can change from day to day and sometimes even within several times a day (Arumai Dhas, 2008).Colour in the water results from various organic chemicals that prevent the sunlight to penetrate affecting the aquatic system. These pollutants contributes to high suspended solids (SS), chemical oxygen demand (COD), biochemical oxygen demand (BOD), heat, color, acidity, basicity and other soluble substances (Arumai Dhas, 2008). The parameters to conduct chemical analysis to determine water quality are conductivity, alkalinity, hardness, pH, total dissolved oxygen (COD and BOD) and turbidity.

## 2.2 Effect of industrial waste water to water body

Industrial contamination is broadening and constitute high environmental risk that will become greater causes that leading to degradation of water quality (Walakira, 2011). Such water contamination will disturbs the balance of ecosystem, resulting mortality of the organisms present in the water (Walakira, 2011). The pollution of metals is big environmental issue (Ho *et al*., 2012). Some metals are possibly carcinogenic or hazardous to human if contact or enter through food chain

even at low concentration (Ho *et al*., 2012). Metals in the debris is afflicted by mineralogical and chemical composition of suspended material, human activities influences by deposition, sorption and enrichment in living organisms or aquatic plant. Metals, nitrates and ammonia which found in the water body might be responsible for causing metabolic destruction in the living organisms (Ho *et al*., 2012). In a term, the harmful elements could even cause mortality in aquatic living organisms. Heavy metals such as zinc (Zn), chromium (Cr), copper (Cu), and lead (Pb) in the fertilizer industry effluents can adheres with certain proteins in fish and agitate membrane system, cellular metabolism and ion-transports that will cause damage to the maintenance of homeostasis (Ho *et al*., 2012). When metals affiliates with other chemical compound in the fertilizer waste, it could lead to distortion in the cell organelles and constrain the activity of enzymes. Studies also proved that metals will cause human kidney and liver failure even at low concentration (Ho *et al*., 2012). In another study, it showed that exposure of C.striatus towards fertilizer industry waste resulted in an extravagant reduction in the enzyme activity. Besides that, metals can also can change the taste of water and contribute to a severe corrosion in pipe lines (Ho *et al*., 2012). PCBs and PAHs are insistent in the ambience, hard to degrade and piled up in food chain will cause jeopardy to human and other living organisms including mutagenicity and carcinogenicity in future.

## 2.3 Synthetic dye (malachite green)

A basic, cationic dye, malachite green (MG), tri-phenyl methane, dye has been widely used for the dyeing of leather, wool, jute and silk and in distilleries. It also functions, fungicide and antiseptic in aquaculture industry (Sartape *et al*, 2013). Malachite green is highly cytotoxic to mammalian cells and also acts as a liver tumor

enhancing agent (Baskaran *et al*, 2011). It decreases food intake, growth and fertility rates; causes damage to liver, spleen, kidney and heart; inflicts lesions on skin, eyes, lungs and bones and produces teratogenic effects. Decrease in red blood cell count (dyscrasia), haemoglobin (anaemia) and HTC (%) increase in white blood cell count (leucocytosis) and delay in blood coagulation were observed after exposure to MG (Sartape *et al*, 2013). The concentration and effective utilization of activated carbon generated from natural plant material have attracted worldwide attention in a view of the large disposal problem without detriment to environment (Baskaran *et al*, 2011).

## 2.4 Activated carbon

Activated carbon is a unique material because of the way it is filled with "holes" (voids, spaces, sites, pores, whatever) and the size of its molecule (Virginia Hernandez Montoya et al., 2015).Activated carbon is a special kind of carbonaceous substances (Das, 2014). Among all of the technology and alternatives used to treat effluents in waste water, adsorption by activated carbon is the well-established alternative to deal with dye removal due to its characteristics which are high surface area (porosity) and has many functional groups. Substance with high carbon content such as hard woods, coconut shell, fruit stones, coals and synthetic macromolecular systems can be used in the production of activated carbon. Activated carbon is also black solid substance and has a highly developed internal surface area as such as 1000 square meter per gram, which is about 10 million times the surface area of 1 gram of water in an open air container. Hence, it has a large adsorbent capacity. Activate carbon is usually used in adsorption, especially in adsorbing substances that are not or are only slightly adsorbed by other method (Mohammad-Khah et al., 2009).

Activated carbon has a porous carbonaceous structure that has been processed to develop its internal porosity, characterized by their extraordinary large specific surface areas and tune able surface-containing functional groups. Therefore, activated carbon is good in absorbing the organic compound, metal ions from environment or the air pollution, gases, potable water and waste water (Sivakumar et al., 2009). General process to produce activated carbon is based on carbonizing and activating the starting carbonaceous material. The activation method can be physical or chemical. Coconut shell is one of the potential waste for producing activated carbon due to its excellent original structure and low ash content (Song *et al*., 2014).

### 2.4.1 Characterization and Properties of Activated Carbon

Characterization for activated carbon is important in order to classify it for specific uses. Basically, activated carbon characterized by physical properties and chemical properties. These activated carbon materials are characterized by their large surface areas and better porosity which was well developed. Wahi (2010) mentioned that the characteristic of activated carbon depends on the physical and chemical properties of the raw materials as well activation method used. The process of activated carbon starts with the selection and classification of a raw carbon source. These sources are selected based on design specification with different properties of activated carbon.

Activated carbon, also widely known as activated charcoal or activated coal is a form of carbon which has been processed to make it extremely porous and thus to have a very large surface area available for adsorption or chemical reactions (Sivakumar et al., 2009).. Activated carbon has high degree of porosity, an extensive surface area, and a high degree of surface reactivity. Its large specific surface area of

(500-2000 $m^2g/g$) is in fact the most important physical properties of activated carbon which allow the physical adsorption of gases from liquids (Mohammad-Khah *et al*., 2009). It has a large number of very fine pores (microspores) gives the activated carbon a large inner surface, which is the basic of its remarkable adsorption properties (Aziz *et al*., 2015).

Physical properties of activated carbon, such as ash content and moisture content can affect the use of a granular activated carbon and render them either suitable or unsuitable for specific application (Jabit, 2007). While the specific surface area of activated carbon and surface chemistry is classified as chemical properties. Furthermore, the porous structure of activated carbon also can be characterized by various techniques such as adsorption of gases or vapors, scanning electron microscopy and so on.

## 2.5 Preparation of Activated Carbon

### 2.5.1 Activation

There are two types of activation methods which can be used to activate the carbon namely physical and chemical activation. These different types of activation will be produced different types of activated carbon. The production of activated carbon by physical activation can be completed using two methods. These methods are called one-step activation and two-step activation liable on whether the carbonization and activation method occur instantaneously, but in distinct steps. (Virginia Hernandez-Montoya et al., 2015). In physical activation, the raw material is first subjected to a carbonization process under an inert atmosphere shadowed by activation of the resulting char in the presence of activating agent such as steam and $CO_2$ (Gratuito et al., 2008).

In chemical activation, both carbonization and activation take place as one, in which the raw precursor is saturated with activating agents and heated at preferred temperature (Nwabenne *et al*., 2014). Chemical activation includes mixing an acidic or basic solution with the carbonaceous material to impact the pyrolytic decomposition of the starting materials, conquer tar formation and lower the pyrolysis temperature (Hesas et al., 2013)

### 2.5.2 Carbonization

Carbonization phase is the process that growths the content of the element carbon that can originate in the agriculture waste and frequently pyrolysis in an inactive atmosphere with gases like nitrogen. In this study, microwave processing will be used as the carbonization.

### 2.5.3 Microwave Processing

Microwave heating has been successfully applied for the preparation and regeneration of activated carbon (Foo et al., 2011). Microwaves supply energy to the carbon particles and this energy is converted into heat within the particles themselves by dipole rotation and ionic conduction. Microwave heating has the benefits of rapid temperature rise, uniform temperature dispersal and saving of energy over conventional heating method. In the microwave method, microwave irradiation relates directly with the particles inside the forced compact material and changes electromagnetic energy into heat transfer inside the dielectric materials (Hesas et al., 2013)

Microwave processing can help to solve the problem in conventional or furnace heating management method such as the sample will fast firing and become

ash (Hesas et al., 2013). Microwave processing method has the capability to produce higher temperature and enhance the chemical reactivity (Woodard, 2001).The basis of the heat is quickly remove when the microwave is turn off and this method are more accurate and more effective. This method easy to control hence made the process become faster thus control the pollution.

### 2.5.4 Types of activating agents

Activated carbon is widely used as adsorbent because it is versatile and efficient for the removal of organic and inorganic pollutant from waste in the liquid stage. Generally, the price range for commercial activated carbon is fairly expensive as the production of the activated carbon is energy and time consuming so prepared activated carbon can reduce the cost of remediation. Activated carbon can be prepared from variety of raw materials from biomass resources. In this experiment, chemical activation process was used to prepare the activated carbon from coconut shell (Yusufu *et al*., 2012)

Chemical activation was used as this process will enhanced the porosity of the carbon in shorter time compared to physical activation. Other advantages of chemical activation includes higher yield of the product, lower temperature used during activation (reduced energy cost) and allow to obtain very high surface areas. Chemical activation can be performed using different types of chemical activating agents. Zinc chloride and phosphoric acid were used as the activating agent in this experiment as these chemicals is most widely used (Gratuito et al., 2008).

## 2.6 Introduction to coconut shell

Coconut shell is a waste that originate from agriculture activity and is abundant throughout the tropical countries of the world (Madakson *et al*., 2012). Coconut also is important as the agricultural product that function as new source of energy-biofuel in the tropical countries (Madakson *et al*., 2012). The coconut besides being used as edible oil, it also has variety industrial usage. The coconut is used in the production of soaps, laundry soaps, surface-active agents and detergents, hair tonics, cosmetics and many more. Coconut shell is a potential precursor for the production of ACs due to its excellent natural structure and low ash content (Song *et al*, 2013). Waste coconut shell material is a carbonaceous material that has high composition of carbon and high potential to become activated carbon (Iqbaldin *et al*., 2013). The percentage of carbon significantly increased after carbonisation and activation process to become coconut shell activated carbon (Mohd Iqbaldin *et al*., 2013). The activation process has successfully increased the surface area and porosity of char derivative from carbonised organic precursor. Habitual characteristics of the coconut carbon shell composites relies on several factors such as stress-strain behaviours of carbon and matric phases, the phase volume fractions, the carbon concentration, the distribution and orientation of the carbon or filler relative to each other (Salleh Z. *et al*., 2013). This is due to the fact that coconut carbon filler particles strengthen the interface of resin matrix and filler materials (Salleh Z *et al*., 2013).

# CHAPTER 3

# MATERIALS AND METHOD

## 3.1 Materials

The materials used for this study is agriculture waste which is coconut shell. The raw materials was collected from small stall located in Gemang, Jeli. The part of the coconut that been used is coconut shell. Next, zinc chloride ($ZnCl_2$) and phosphoric acid ($H_3PO_4$) were used as activating agent during chemical activation process. The malachite green powder was used to prepare 1000ppm stock solution. Malachite green solutions were act as synthetic textile waste water.

### 3.1.1 Raw material preparation

The coconut shell was smashed into small pieces using hammer and rinsed with tap water. Then it was dried in oven for 2 hours at 150ºC. Next, the dried coconut shell was ground using blender. After that, the coconut shell was sieved using siever with size 2.00 mm. Then it was stored in a room temperature and covered.

## 3.2 Method

### 3.2.1 Chemical Activation process

Chemical activation process using zinc chloride and phosphoric acid as activating agent will lead to the production of activated carbon with well-developed porosity in a single step. The activation process was conducted with the impregnation ratio 2:1 (dry weight of activating agent in ml /dry weight of precursor in gram). 8

conical flask were prepared with the granulated coconut shell. 4 of the conical flask were poured with 25% zinc chloride and the remaining conical flask were poured with 25% phosphoric acid. The granulated coconut shell was stirred until it mix well and soaked overnight so that the activating agent can be fully absorbed. The mixture was fully covered using aluminum foil.

### 3.2.2 Carbonization

The mixture was carbonized under an inert atmosphere in muffle furnace. This method is used to remove moisture and volatile impurities. The mixtures were heated with different set time which are 5 minutes, 10 minutes, 30 minutes and 1 hour. The temperature used to heat the mixtures was fixed to 400°C. These parameters were used for both type of mixtures, which are zinc chloride and phosphoric acid. The parameter of activation is normally referred as “burn-off”.

### 3.2.3 Preparation of synthetic textile waste water (malachite green)

Malachite green powder (1g) was mixed with 1000ml of distilled water to prepare 1000ppm stock solution. The stock solution then was kept in the reagent bottle.

### 3.2.4 Analysis of synthetic dye removal (malachite green) using activated carbon

#### 3.2.4.1 Type of activated carbon

To test the intensity of sample, UV-VIS Spectrometer (-Spec-20; thermos scientific Genesys) was used. Dye solution (25ml, 10ppm) was mixed with prepared activated carbon labelled as (AC5) in a conical flask. The initial reading of dye solution was recorded using UV-Vis Spectrometer. The conical flask was covered with parafilm and was put into the mechanical shaker for an hour at 100 rpm. These steps were repeated using activated carbon prepared at 10 minutes, 30 minutes and 1 hour activation time for both activating agent (zinc chloride and phosphoric acid). The activated carbon with the highest rate of removal acquired was used for the subsequent experiment.

#### 3.2.4.2 Effect of contact time

Dye solution (25ml, 10ppm) was mixed with 10g of the activated carbon that has the highest rate of removal from previous parameter in a conical flask. The initial reading of dye solution was recorded. The conical flask was covered with parafilm and was put into the mechanical shaker for half an hour at 100rpm. These steps were repeated using 2 hour and 3 hour of contact time. The most optimum contact time acquired was used for the next parameter.

#### 3.2.4.3 Effect of concentration of dye solution

Dye solution (25ml, 25ppm) was mixed with 10g of the activated carbon that has the highest rate of removal in a conical flask. The conical flask was covered with parafilm and was put into the mechanical shaker within the best time contact

acquired from previous parameter. The mechanical shaker was set with 100rpm. Solution was then filtered and final reading was taken. These steps were repeated using 50ppm dye solution.

### 3.3 Determination of dye removal

The percentage of dye removal was determined using formula;

$$\frac{\text{Initial concentration of dye} - \text{Final concentration of dye}}{\text{Initial}} \times 100$$

This equation was used to determine the percentage of dye molecules that have been adsorbed from the synthetic waste water by allocated activated carbon and contact time.

# CHAPTER 4

# RESULTS AND DISCUSSIONS

## 4.1 Characterization of activated carbon derived from coconut shell

In this study, there were 2 types of activating agents which are zinc chloride and phosphoric acid and different sets of activating times which were 5 minutes, 10 minutes, 30 minutes and 1 hour. Figure 4.1 and 4.2 shows the effect of different types of activated (zinc chloride and phosphoric acid) respectively.

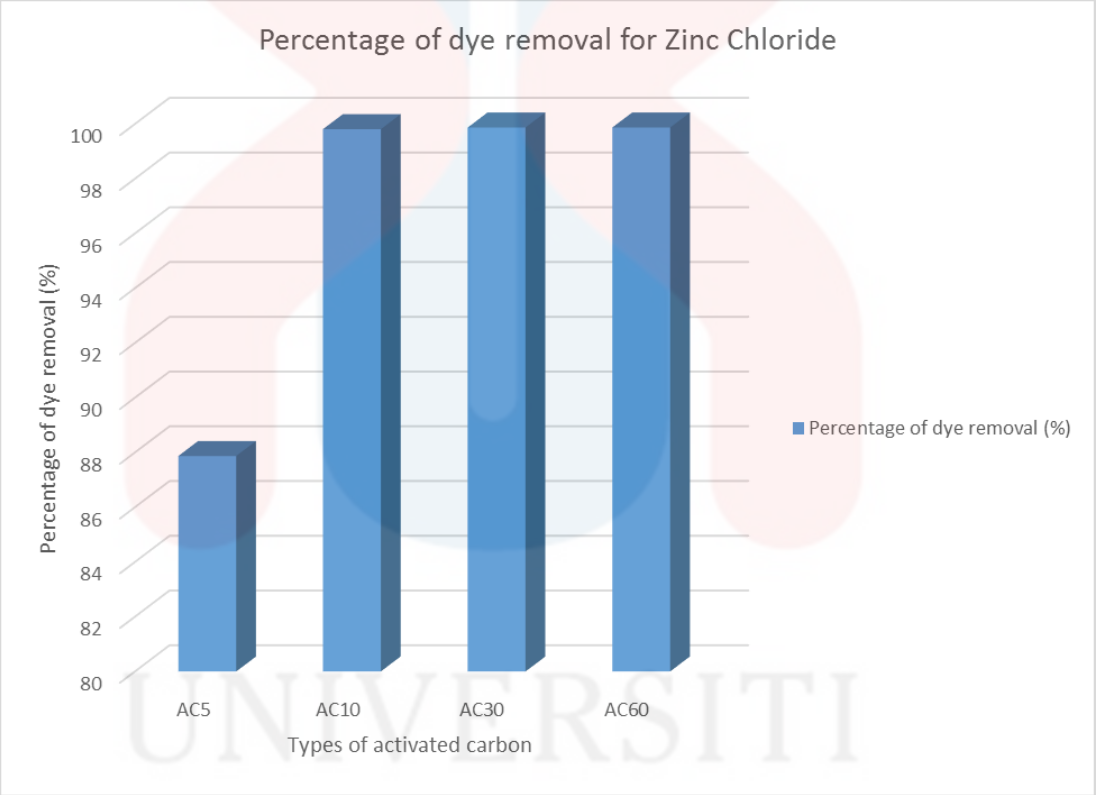


Figure 4.1 Effect of different types of activated carbons ($ZnCl_2$)

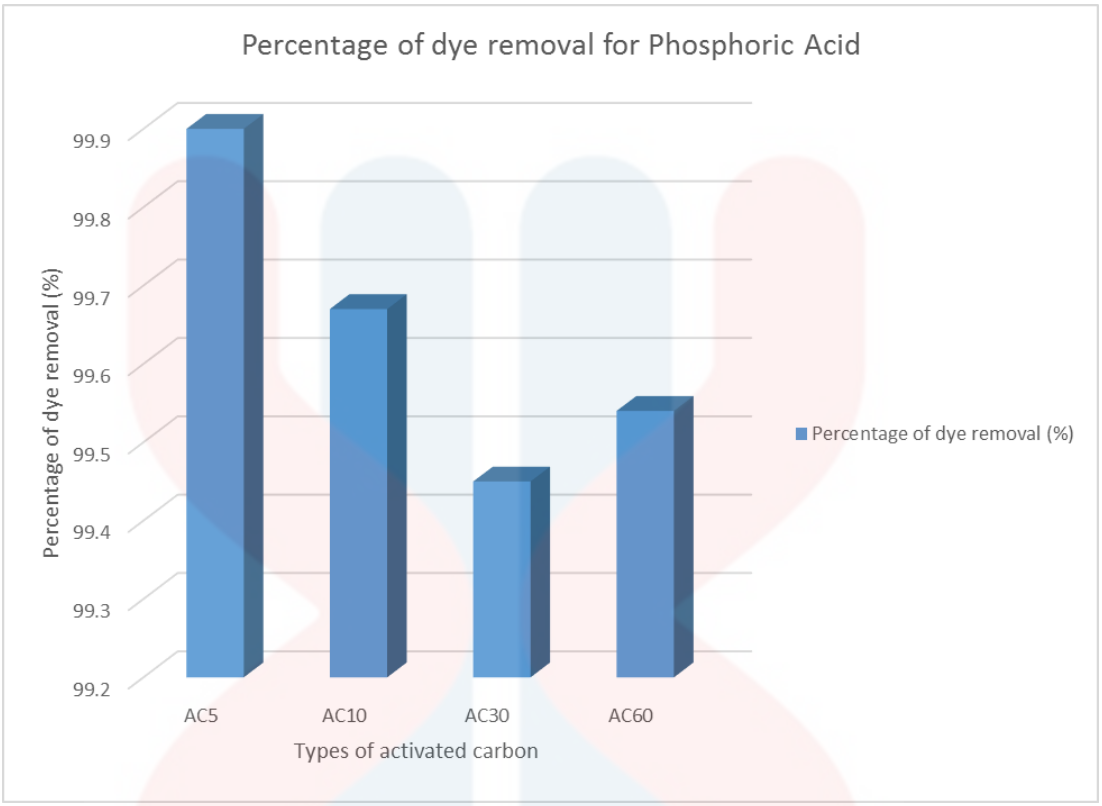


Figure 4.2 Effect of different types of activated carbons ($H_3PO_4$)

Two types of activating agents (zinc chloride and phosphoric acid) were used in the experiment to compare which activating agent works the best for coconut shell to produce activated carbon. From the results acquired, phosphoric acid is the best activating agent for coconut shell derived activated carbon. This has been proven as the activated carbon using phosphoric acid as activating agent only needs 5 minutes activation time to remove 99.9% of 10ppm dye solutions. The other batch of activated carbon activated by phosphoric acid but with different activation time (10 minutes,30 minutes and 1 hour) also achieved 99% and above. So the fact that using chemical activation can reduced energy cost as the temperature and time needed is lesser was proven in this experiment.

The use of zinc chloride as activating agent is also possible for producing activated carbon from coconut shell but it was not as efficient as phosphoric acid. The activated carbon of zinc chloride with 5 minutes activation time can only

achieved 87.87% removal of dye from the 10ppm solutions. The other batch of activated carbon by zinc chloride but with different activation time (10 minutes, 30 minutes and 1 hour) achieved 99% but still not as efficient as activated carbon by phosphoric acid which achieved 99.9%. So that is mean that the activated carbon which activated by zinc chloride might needs more activation time to remove dye from the solutions.

The coconut shell is a very hard material so it required strong activating agent to decompose its structure. Phosphoric acid is stronger activating agent so it can weakened the structure of coconut shell as well as produced carbon with higher surface area and higher microporous. These characteristics is important for activated carbon to achieve higher rate of dye removal.

The results obtained was analysed by using Minitab 17 statistical analysis of variance (ANOVA). The data was tabulated in Table 4.1.

| Source | Degree of freedom | Sum square | Mean square | F-value | P-value |
|---|---|---|---|---|---|
| Activated carbon | 1 | 15.51 | 15.51 | 0.86 | 0.389 |
| Error | 6 | 107.67 | 17.96 | | |
| Total | 7 | 123.27 | | | |

Table 4.1 Statistical analysis of variance

The one-way ANOVA of percentage of dye removal versus types of activating agents shows that the p-value is greater than 0.05. This means that we accepted the null hypothesis as all means are assumed equal. This determined that the means are not significantly different.

Based on the Tukey test conducted, the mean grouping of both types activated carbons share a letter. This shows that the difference are not significant. Based on the

Tukey test, it also shows that activated carbon activated by phosphoric acid has significantly higher mean compared to activated carbon activated by zinc chloride. Details of ANOVA was shown in Appendix A.

The activation duration has a significant effect on the development of the network of carbon's porous. The activation time should provide sufficient time to eliminate remaining moisture content in the raw material to produce excellent activated carbons. The activation time also was the time to eliminate most of the volatile components that will interrupt the developments of porous in the precursor. Longer durations of activation cause enlargement of pores at the expense of the surface area. These traits increase the rate of removal of dye from the synthetic waste water as the adsorption rate also increased.

The control of activation time is of economic interest since shorter time needed will contribute to lesser energy consumption. It is also reduce cost of energy used (electricity). Yield also appeared to be dependent on the activation duration as it dropped upon reaching an optimum point. Low activation time resulted to an incomplete burn-off thus resulting in higher yields.

There were different sets of activation time which were 5 minutes, 10 minutes, 30 minutes and 1 hour used to produce the activated carbons derived from coconut shell. These different set of activation time used to determine the most optimum activation time to produce the activated carbons. The temperature of the activation process was fixed to 400°C as this temperature was found to be the optimum temperature for coconut shell precursor.

The results recorded show that the best activation time was dependent on the type of activating agents. As for the phosphoric acid activating agent, the most optimum activation time required was 5 minutes. This batch of activated carbon has

the highest percentage of dye removal which was 99.9%. The other batch of activated carbon with 10 minutes, 30 minutes and 1 hour activation time also achieved high percentage of dye removal, 99.67%, 99.45% and 99.54% respectively.

The results also show that for activated carbon with zinc chloride as activating agent, the activation time needed was higher to produce the optimum activated carbon. The most optimum activation time required was 30 minutes and 1 hour. This batch of the activated carbon achieve 99.87% of dye removal. The other batch of activated carbon with 5 minutes and 10 minutes activation time achieved 87.87% and 99.81% respectively.

The results obtained was analysed by using Minitab 17 statistical analysis of variance (ANOVA). The data was tabulated in Table 4.2.

| Source | Degree of freedom | Sum square | Mean square | F-value | P-value |
|---|---|---|---|---|---|
| Activated carbon | 3 | 50.76 | 16.92 | 0.93 | 0.503 |
| Error | 4 | 72.51 | 18.13 | | |
| Total | 7 | 123.27 | | | |

Table 4.2 Statistical analysis of variance

The one-way ANOVA; percentage of dye removal versus activation time shows that the p-value is greater than 0.05. This means that we accepted the null hypothesis as all means are assumed equal as the means are not significantly different. The A, B, C and D represented 5 minutes, 10 minutes, 30 minutes and 1 hour respectively.

Based on the Tukey test conducted, the mean grouping of all types of activated carbons with different activating time share a letter. This shows that the difference are not significant. Based on the Tukey test, it also shows that activated

carbon with 10 minutes activating time has significantly higher mean compared to others. This is because 10 minutes activating time is effective for both types of activating agents. Activated carbon with 5 minutes activating time has significantly lower means because it only effective for activated carbon with zinc chloride as activating agents.

### 4.2 Effect of contact time

The results recorded in Figure 4.3 shows that the percentage of dye removal is the highest when the contact time (dye solution mixed with activated carbon in the shaker) was 1 hour. The colour of dye solution changed from light blue to colourless. The result also shows that the percentage of dye removal is the lowest when the contact time (dye solution mixed with activated carbon in the shaker) was 3 hours but the dye solution still changed from light blue to colourless.

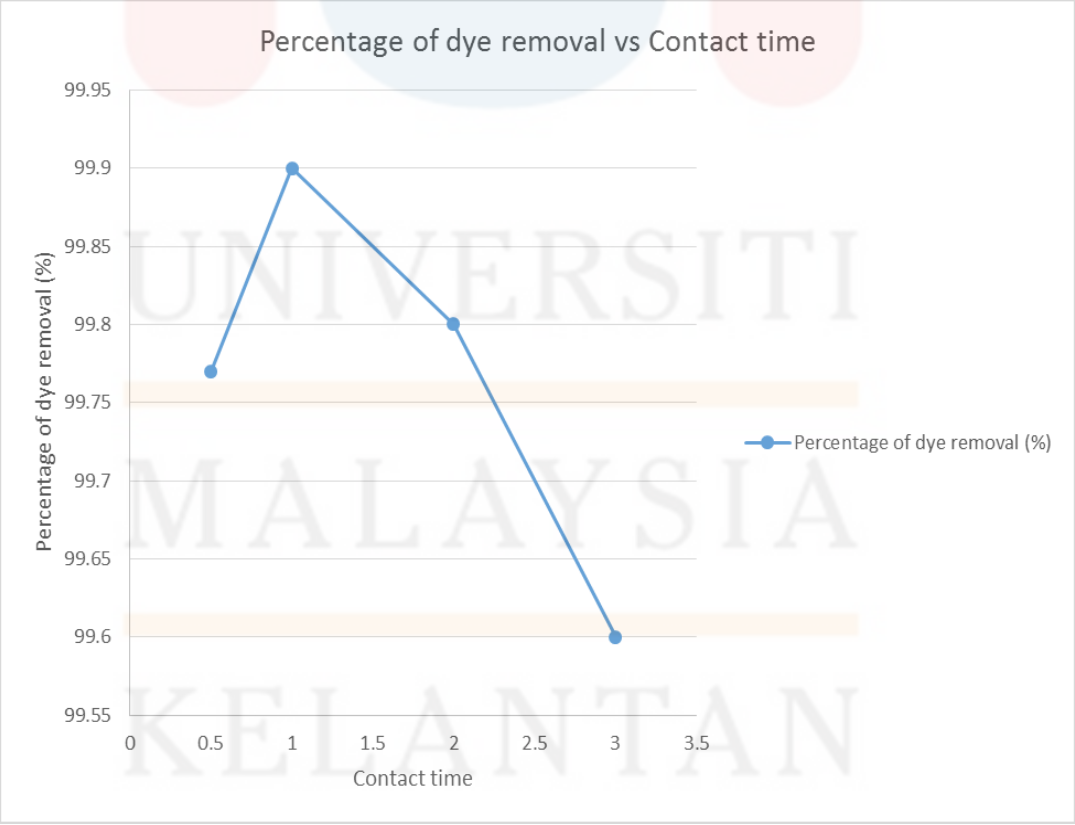


Figure 4.3 Effect of contact time

The adsorption of dye is highly dependent to the initial concentration (ppm). At lower concentrations, the ratio of the initial number of dye molecules to the available surface area subsequently the fractional adsorption becomes independent of initial concentrations. However, at high concentrations, the available sites of adsorption become fewer hence the percentage of dye removal is dependent to the initial concentrations (Gratuito *et al*., 2008). So the contact time required was varied.

The lesser contact time needed, the higher the efficiency of the activated carbon. This means that the activated carbon is excellent in adsorbing the dye to its surface area in a short time. The activated carbon with high surface area and high pores development needed shorter time to remove dye from the synthetic waste water.

The results from this experiment shows that the most optimum contact time needed was 1 hour. The percentage of dye removal achieved 99.9%. This is because the time allocated was sufficient enough for the adsorption process for dye solutions with initial concentrations of 10ppm.

The remaining contact time which were 30 minutes, 2 hours and 3 hours achieved 99.77%, 99.8% and 99.6% respectively. The batch with 30 minutes contact time achieved lower percentage of dye removal because the time allocated was not sufficient for the dye molecules to adsorb to the activated carbon surface. The remaining batch with higher contact time achieved low percentage of dye removal because the activated carbon had become saturated and did not capable to adsorb the dye molecules anymore.

The results obtained was analysed by using Minitab 17 statistical analysis of variance (ANOVA). The data was tabulated in Table 4.3.

| Source | Degree of freedom | Sum square | Mean square | F-value | P-value |
|---|---|---|---|---|---|
| Activated carbon | 3 | 0.11594 | 0.038646 | 13.50 | 0.0.15 |
| Error | 4 | 0.01145 | 0.002862 | | |
| Total | 7 | 0.12739 | | | |

Table 4.3 Statistical analysis of variance

The one-way ANOVA; percentage of dye removal versus time contact shows that the p-value is lower than 0.05. This means that we rejected the null hypothesis and accepted the alternatives hypothesis as the means are significantly different. The A, B, C and D represented 30 minutes, 1 hour, 2 hours and 3 hours respectively.

Based on the Tukey test conducted, the mean grouping divided into two which is C and A that shared a letter and B and D that does not share a letter. Difference of means that shared a letter determined that it is not statistically significant while B and D that does not share a letter determined that B has significantly higher mean compared to D. Details of ANOVA was shown in Appendix A.

The confidence intervals for the remaining pairs of means all include zero which indicates that the differences are not statistically significant. Each individual confidence interval has a confidence level of 98.48%. This result indicates that the results has 98.48% confident that each individual interval contains the true difference between a specific pair of group means. The individual confidence levels for each comparison produce the 95% simultaneous confidence level for all four comparisons.

### 4.3 Effect of concentration

The results recorded in Figure 4.4 shows that the percentage of dye removal is the highest when the concentrations of dye used in the analysis was 10ppm. The colour also changed from light blue to colourless. The result also shows that the percentage of dye removal is the lowest when the concentrations of dye used in the analysis was 25 ppm but the colour still changed from light blue to colourless.

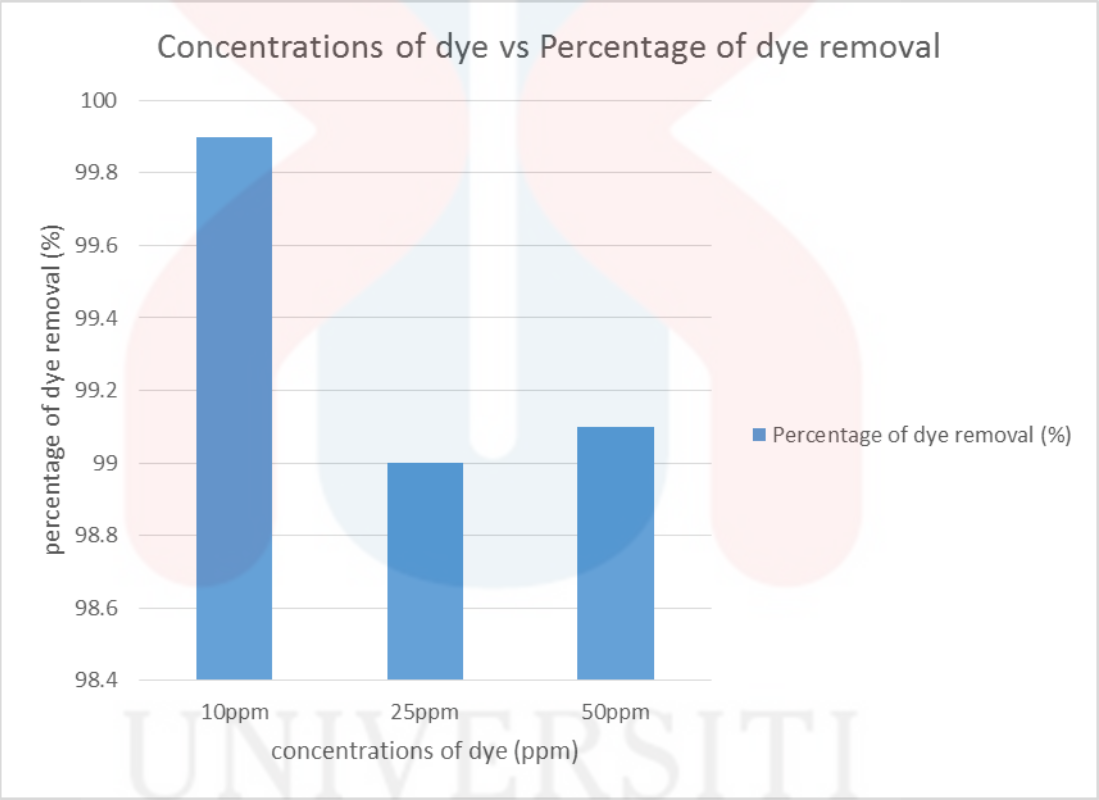


Figure 4.4 Effect of dye concentrations

The initial dye concentration of an effluent is crucial since a given mass of activated carbon's pores can only adsorb fixed amount of dye. The effect of initial concentration was carried out by preparing adsorbent-adsorbate solution with fixed adsorbent dose and different initial concentration. The effect of the of the initial concentration factors depend on the immediate relation between the concentration of the dye and the available binding sites on the activated carbon derived from coconut shell (Salleh *et al*., 2011).

The percentage of dye removal normally decreased when the initial concentration increased. This is because for a given mass of activated carbon, the amount of dye that can be adsorbed was fixed. The time taken to reach equilibrium also increased with the increasing of initial concentrations. This may be due to the high driving force for mass transfer at a high initial concentrations of dye (Bharati et al., 2013).

The results acquired from this experiment shows that the most optimum initial concentrations that can be removed by 0.5g of activated carbon derived from coconut shell was 10 ppm. The percentage of dye removal achieved was 99.9%. This is because there is sufficient binding sites on the activated carbon to adsorb the malachite green molecules.

The remaining initial concentrations of dye which were 25ppm and 50ppm achieved 99.0% and 99.10% respectively. These batch of adsorbent-adsorbate achieved lower percentage of dye removal compared to 10ppm initial concentrations because the binding sites or pores was not sufficient enough to adsorb the dye molecules. For a higher initial concentrations, higher dosage of activated carbon are required so that there will be sufficient binding sites on the activated carbon to adsorb the dye molecules.

The results obtained was analysed by using Minitab 17 statistical analysis of variance (ANOVA). The data was tabulated in Table 4.4.

| Source | Degree of freedom | Sum square | Mean square | F-value | P-value |
|---|---|---|---|---|---|
| Activated carbon | 2 | 0.986033 | 0.493017 | 1173.73 | 0.000 |
| Error | 3 | 0.001300 | 0.000433 | | |
| Total | 5 | 0.987333 | | | |

Table 4.4 Statistical analysis of variance

The one-way ANOVA; percentage of dye removal versus dye concentrations shows that the p-value is lower than 0.05. This means that we rejected the null hypothesis and accepted the alternatives hypothesis as the means are significantly different. The A, B and C represented 10 ppm, 25ppm and 50 ppm respectively.

Based on the Tukey test conducted, the mean grouping divided into two which is B and C that shared a letter and A that does not share a letter. Difference of means that shared a letter determined that it is not statistically significant while A that does not share a letter determined that A has significantly higher mean compared to both B and C. Details of ANOVA result was shown in Appendix A.

The confidence intervals for the remaining pairs of means all include zero, which indicates that the differences are not statistically significant. Each individual confidence interval has a confidence level of 97.50%. This result indicates that the results has 97.50% confident that each individual interval contains the true difference between a specific pair of group means. The individual confidence levels for each comparison produce the 95% simultaneous confidence level for all three comparisons.

# CHAPTER 5

# CONCLUSION AND RECOMMENDATIONS

## 5.1 Conclusion

This research was done to produce activated carbon derived from agricultural waste that is cheap and recyclable. These kind of activated carbon can replace the commercial activated carbon which is very expensive. The coconut shell is abundant in Malaysia and also other countries hence it is convenient to use coconut shell to produce activated carbon. The activating agents used, zinc chloride and phosphoric acid enhanced the porosity of the activated carbon hence increase the surface of binding sites. Based on the result acquired, the best activating agent to produce activated carbon derive from coconut shell was phosphoric acid which required only 5 minutes activating time to remove 99.9% malachite green dye from synthetic waste water. The highest percentage was obtained by using activated carbon that used phosphoric acid and activated for 5 minutes. Based on the results acquired, 5 minutes was already sufficient for activated carbon by phosphoric acid. From the results stated earlier, 1 hour of contact time is the most optimum to adsorb the dye molecules from the synthetic waste water. As the dosage of activated carbon was fixed in the research, the most optimum dye concentrations for 0.5g of activated carbon was 10ppm.

## 5.2 Recommendations

As mentioned earlier, factors that may affect the optimum removal of malachite green from synthetic waste water are activating agents, activating times, contact time and initial concentrations of dye. So as recommendations, the factors mentioned above should be control. Moreover if these factors affected the experiment, it will cause inaccuracy of the percentage of dye removal.

For the activating agents, the volume of the activating agents should be increased to enhance the porosity of the activated carbon. Next, to remove dye molecules from higher initial concentrations, the dosage of activated carbon should be increased so that the activated carbon will be capable to remove the malachite green molecules from the synthetic waste water.

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## APPENDIX A

### One-way ANOVA: Percentage versus Activated carbon

```
Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.


Factor Information

Factor            Levels  Values
Activated carbon       2  ACPH, ACZN


Analysis of Variance

Source            DF  Adj SS  Adj MS  F-Value  P-Value
Activated carbon   1   15.51   15.51     0.86    0.389
Error              6  107.76   17.96
Total              7  123.27


Model Summary

      S    R-sq  R-sq(adj)  R-sq(pred)
4.23787  12.58%      0.00%       0.00%


Means

Activated
carbon     N     Mean   StDev         95% CI
ACPH       4  99.6400  0.1954  (94.4552, 104.8248)
ACZN       4    96.86    5.99  (  91.67,   102.04)

Pooled StDev = 4.23787
```

### Tukey Pairwise Comparisons

```
Grouping Information Using the Tukey Method and 95% Confidence

Activated
carbon     N     Mean  Grouping
ACPH       4  99.6400  A
ACZN       4    96.86  A

Means that do not share a letter are significantly different.
```

### Tukey Simultaneous 95% CIs

### Interval Plot of Percentage vs Activated carbon

## One-way ANOVA: Percentage versus Time

```
Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.


Factor Information

Factor  Levels  Values
Time         4  A, B, C, D


Analysis of Variance

Source  DF  Adj SS  Adj MS  F-Value  P-Value
Time     3   50.76   16.92     0.93    0.503
Error    4   72.51   18.13
Total    7  123.27


Model Summary

      S    R-sq  R-sq(adj)  R-sq(pred)
4.25773  41.18%      0.00%       0.00%


Means

Time  N     Mean   StDev         95% CI
A     2    93.89    8.51  (  85.53,   102.24)
B     2  99.7400  0.0990  (91.3810, 108.0990)
C     2   99.660   0.297  ( 91.301,  108.019)
D     2   99.705   0.233  ( 91.346,  108.064)

Pooled StDev = 4.25773
```

## Tukey Pairwise Comparisons

```
Grouping Information Using the Tukey Method and 95% Confidence

Time  N     Mean  Grouping
B     2  99.7400  A
D     2   99.705  A
C     2   99.660  A
A     2    93.89  A

Means that do not share a letter are significantly different.
```

## Tukey Simultaneous 95% CIs

## Interval Plot of Percentage vs Time

## One-way ANOVA: Percentage of dye removal versus Time contact

```
Method

Null hypothesis         All means are equal
Alternative hypothesis  At least one mean is different
Significance level      α = 0.05

Equal variances were assumed for the analysis.


Factor Information

Factor        Levels  Values
Time contact       4  A, B, C, D


Analysis of Variance

Source        DF   Adj SS    Adj MS  F-Value  P-Value
Time contact   3  0.11594  0.038646    13.50    0.015
Error          4  0.01145  0.002862
Total          7  0.12739


Model Summary

        S    R-sq  R-sq(adj)  R-sq(pred)
0.0535023  91.01%     84.27%      64.05%


Means

Time
contact  N     Mean   StDev        95% CI
A        2  99.7100  0.0849  (99.6050, 99.8150)
B        2    99.90    0.00  (  99.79,  100.01)
C        2  99.7700  0.0424  (99.6650, 99.8750)
D        2  99.5650  0.0495  (99.4600, 99.6700)

Pooled StDev = 0.0535023
```

## Tukey Pairwise Comparisons

```
Grouping Information Using the Tukey Method and 95% Confidence

Time
contact  N     Mean  Grouping
B        2    99.90  A
C        2  99.7700  A B
A        2  99.7100  A B
D        2  99.5650    B

Means that do not share a letter are significantly different.


Tukey Simultaneous Tests for Differences of Means

Difference  Difference       SE of                                 Adjusted
```

| of Levels | of Means | Difference | 95% CI | T-Value | P-Value |
|---|---|---|---|---|---|
| B - A | 0.1900 | 0.0535 | (-0.0279, 0.4079) | 3.55 | 0.077 |
| C - A | 0.0600 | 0.0535 | (-0.1579, 0.2779) | 1.12 | 0.698 |
| D - A | -0.1450 | 0.0535 | (-0.3629, 0.0729) | -2.71 | 0.163 |
| C - B | -0.1300 | 0.0535 | (-0.3479, 0.0879) | -2.43 | 0.213 |
| D - B | -0.3350 | 0.0535 | (-0.5529, -0.1171) | -6.26 | 0.011 |
| D - C | -0.2050 | 0.0535 | (-0.4229, 0.0129) | -3.83 | 0.061 |

Individual confidence level = 98.48%

## Tukey Simultaneous 95% CIs

## Interval Plot of Percentage of dye removal vs Time contact

## One-way ANOVA: Percentage of dye removal versus Concentration of dye

Method

| | |
|---|---|
| Null hypothesis | All means are equal |
| Alternative hypothesis | At least one mean is different |
| Significance level | α = 0.05 |

Equal variances were assumed for the analysis.

Factor Information

| Factor | Levels | Values |
|---|---|---|
| Concentration of dye | 3 | A, B, C |

Analysis of Variance

| Source | DF | Adj SS | Adj MS | F-Value | P-Value |
|---|---|---|---|---|---|
| Concentration of dye | 2 | 0.986033 | 0.493017 | 1137.73 | 0.000 |
| Error | 3 | 0.001300 | 0.000433 | | |
| Total | 5 | 0.987333 | | | |

Model Summary

| S | R-sq | R-sq(adj) | R-sq(pred) |
|---|---|---|---|
| 0.0208167 | 99.87% | 99.78% | 99.47% |

Means

| Concentration of dye | N | Mean | StDev | 95% CI |
|---|---|---|---|---|
| A | 2 | 99.8950 | 0.0071 | (99.8482, 99.9418) |
| B | 2 | 99.00 | 0.00 | ( 98.95, 99.05) |
| C | 2 | 99.0750 | 0.0354 | (99.0282, 99.1218) |

Pooled StDev = 0.0208167

## Tukey Pairwise Comparisons

Grouping Information Using the Tukey Method and 95% Confidence

| Concentration of dye | N | Mean | Grouping |
|---|---|---|---|
| A | 2 | 99.8950 | A |
| C | 2 | 99.0750 | B |
| B | 2 | 99.00 | B |

Means that do not share a letter are significantly different.

Tukey Simultaneous Tests for Differences of Means

| Difference of Levels | Difference of Means | SE of Difference | 95% CI | T-Value | Adjusted P-Value |
|---|---|---|---|---|---|
| B - A | -0.8950 | 0.0208 | (-0.9820, -0.8080) | -42.99 | 0.000 |
| C - A | -0.8200 | 0.0208 | (-0.9070, -0.7330) | -39.39 | 0.000 |
| C - B | 0.0750 | 0.0208 | (-0.0120, 0.1620) | 3.60 | 0.073 |

Individual confidence level = 97.50%

## Tukey Simultaneous 95% CIs

## Interval Plot of Percentage of dye removal vs Concentration of dye