



Universiti Malaysia
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**REMOVAL OF DYE FROM TEXTILE
EFFLUENT USING ACTIVATED CARBON
FROM KENAF FIBER AND RUBBER WOOD
SAWDUST PRODUCED BY MICROWAVE
PROCESSING**

by

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A report submitted in fulfillment of the requirement for the degree
of Bachelor of Applied Science (Sustainable Science) with Honours

**FACULTY OF EARTH SCIENCE
UNIVERSITI MALAYSIA KELANTAN**

2017

DECLARATION

I declare that this thesis entitled “Investigation of Sesbania grandiflora leave extracts as eco-friendly anti-termite agent for wood panel” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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ACKNOWLEDGEMENT

First of all, a great thankful to Allah S.W.T. upon the completion of this thesis. A special gratitude to my beloved parents, Wong Fook Seng and Jamaliah binti Ishak for their love, endless support and prayers. I would like to express my heartiest appreciation to my supervisor, Dr. Nik Raihan binti Nik Yusoff for her guidance and continuous support and supervision of the research project until the completion of the thesis. I am really honored for the opportunity to work under her supervision.

I would also like to extend my gratitude to the Faculty of Earth Science Universiti Malaysia Kelantan, which giving me golden opportunities to learn and utilized the instrument and facilities had in UMK in order to complete my research. Besides, I would like to thank Mr. Mohamad Rohanif bin Mohamed Ali, Mr. Mohammed Firdaus Bin Mohd Ridzuan and Mr. Mohamad Khidzir Bin Mohd Ibrahim act as lab assistance provided throughout the entire study period.

Last but not least thanks to my senior, Mohd Shafiq bin Shamsuddin for all the valuable help, moral supports throughout this entire process this study. The blessing, help and guidance given by everybody mentioned or not mentioned here shall carry me a long way in the journey of life on which I am about to embark.

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LIST OF SYMBOLS

N₂	Nitrogen
Ar	Argon
CO₂	Carbon dioxide
H₃PO₄	Phosphoric acid
CuO	Copper Oxide
HCl	Hydrochloric Acid
HNO₃	Nitric Acid
Cd	Cadmium
Pb	Lead

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LIST OF ABBREVIATIONS

PAC	Powdered Activated Carbon
GAC	Granular Activated Carbon
EAC	Extruded Activated Carbon
SS	Suspended Solid
BOD	Biochemical Oxygen Demand
COD	Chemical Oxygen Demand
C_i	Initial Concentration
C_o	Final Concentration
RWS	Rubber Wood Sawdust
RWAC	Rubber Wood Sawdust Activated Carbon
KCF	Kenaf Core Fiber
KFAC	Kenaf Fiber Activated Carbon

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ABSTRACT

Textile wastewater is hard to treat because they are originate from multi processes which produce multi segments of wastewater. Expansive measure of suspended solids, high color and high chemical oxygen demand of the wastewater can cause serious ecological issues. Adsorption is a compelling technique for bringing down the convergence of dissolved dyes in the effluent resulting in colour removal. Thus, there is a developing enthusiasm for utilizing material such as agricultural by-product for instance, rubber wood sawdust since they are abundantly available and kenaf fibre. The crude materials, kenaf core fibre (KCF) and rubber wood sawdust (RWS) were carbonized for an hour at 500°C, soaked in H₃PO₄ and activated by microwave processing with input power of 500 W and irradiation time of 6 min. Impregnation of RWAC and KFAC with CuO powder was carried out through solid state reaction of mechanochemical activation processing followed by carbothermal reduction. 0.4 g of activated carbon produced from KFAC and RWAC of 60% acid loading were used to treat 50ml of effluent textile wastewater with the pH of 11.2 with 4 different contact time. The optimum contact time was 90 minutes then used to test the best dosage of activated carbon for adsorption of dye in effluent wastewater. 2.0g of KFAC and RWAC then used to treat 50 ml of effluent wastewater for 90 minutes to study the best pH for dye adsorption. The best pH was pH 3. Then the best parameters from the treatment then used to treat wastewater with CuO assisted KFAC and RWAC. CuO assisted activated carbon proven can enhance reaction activity. The study shows that microwave heating is an effective and facilitate method which can be used for preparation of activated carbon and the effectiveness of activated carbon produced by microwave processing proven can be use to remove dye from textile wastewater.

PENYINGKIRAN PEWARNAAN MENGGUNAKAN KARBON TERAKTIF DARI GENTIAN KENAF DAN HABUK KAYU GETAH TERHASIL MELALUI PEMROSESAN KETUHAR GELOMBANG MIKRO

ABSTRAK

Sisa air buangan tekstil sukar dirawat kerana mereka terhasil daripada pelbagai proses yang menghasilkan pelbagai segmen sisa air buangan. Pepejal terampai, pewarna yang tinggi dan oksigen kimia yang tinggi dalam sisa buangan boleh menyebabkan isu-isu ekologi yang serius. Penjerapan adalah satu teknik yang menarik untuk menghapuskan pewarna dalam effluen. Oleh itu, kajian dijalankan menggunakan bahan buangan pertanian seperti habuk kayu getah dan gentian kenaf kerana bahan-bahan ini mudah didapati di Malaysia. Gentian kenaf (KCF) dan habuk kayu getah (RWS) dibakar selama satu jam pada suhu 500°C. Selepas itu, arang yang terhasil direndam dalam H_3PO_4 dan diaktifkan dengan pemprosesan gelombang mikro dengan kuasa 500W selama 6 minit. Karbon teraktif yang terhasil kemudian dicampurkan dengan CuO melalui tindak balas keadaan pepejal pemprosesan pengaktifan mechanochemical diikuti oleh pengurangan carbothermal. 0.4 g karbon teraktif dengan kepekatan acid 60% yang dihasilkan dari KFAC dan RWAC digunakan untuk merawat 50ml efluen sisa buangan tekstil ber-pH 11.2 dengan 4 masa rawatan yang berbeza. Kadar rawatan optimum bagi rawatan pewarnaan ini adalah 90 minit; kemudian hasil kajian ini digunakan untuk menguji dos yang terbaik untuk penjerapan pewarna dalam air sisa efluen. 2.0g KFAC dan RWAC kemudian digunakan untuk merawat 50 ml air sisa efluen selama 90 minit untuk mengkaji pH terbaik untuk penjerapan pewarna. Hasil kajian menunjukkan pH terbaik adalah pH 3. Kemudian parameter yang terbaik daripada rawatan yang kemudiannya digunakan untuk merawat sisa buangan tekstil dengan KFAC-CuO dan RWAC-CuO. Kombinasi CuO dan karbon teraktif terbukti boleh meningkatkan aktiviti tindak balas. Kajian ini menunjukkan bahawa pemanasan gelombang mikro berkesan dan mempercepatkan penghasilan karbon teraktif dan produk penghasilan ini berkesan untuk menyingkirkan pewarna dalam sisa buangan tekstil.

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Released wastewater by commercial enterprises under uncontrolled and unacceptable conditions is bringing about noteworthy natural issues. There are strict requirements for the discharge of the wastewater because wastewater is destructive to the environment and individuals. The key component towards a sustainable future is the importance of the contamination control and treatment. The regular bodies and land in the encompassing region will be seriously affected if a textile factory releases the wastewater into the nearby environment with no treatment. Oil in the gushing, vicinity of particulate matter and residue, and high estimations of COD and BOD₅ are the signs of depletion of dissolved oxygen, which adversely affects the aquatic ecological system. Gushing from textile wastewater additionally contains chromium, which can accumulate and higher conceivable outcomes for going into the food chain. Utilization of colours and chemicals causes the effluents are dim in shading which adversely expands the turbidity of the water body. This thus hampers the photosynthesis process, bringing on adjustment in the natural surroundings.

Activated carbon is a type of carbon handled to have low-volume pores that build the surface territory accessible for compound responses or adsorption. Activated carbon has an incredibly vast surface area per unit volume, and a network of submicroscopic pores where adsorption happens. Activated carbon is a material that is delivered from carbonaceous source materials, for example, coal, coconuts,

nutshells, peat, wood, and lignite. The essential crude material utilized for activated carbon is any natural material with high carbon content (Sherbondy and Mickler, 2015). The carbon-based material is changed over to activated carbon through physical alteration and thermal disintegration in a furnace, under a controlled air and temperature. The completed item has a huge surface area for every unit volume and a network of submicroscopic pores where adsorption happens. Due to its high level of microporosity, only one gram of activated carbon has a surface range in overabundance of 500 m^2 (5,400 sq ft), as determined by gas adsorption (Jhadhav, 2015). An activation level adequate for useful application might be achieved exclusively from the high surface region; however, further substance treatment regularly upgrades adsorption properties.

At present, textile industry produces floating solid materials and high levels of dye. It is estimated that 5000 tons of dyeing materials are discharged into the environment every year (Patil *et al.*, 2011). These poisonous materials absorb the oxygen in the water. This has risen much as it threatens human life and the environment. Industrial wastewaters contain various kinds of toxic substances such as cyanides, alkaline cleaning agents, degreasing solvents, oil, fat, and metals (Pirkarami *et al.*, 2013). This adsorption of activated carbon just used low preparation cost and simple free design make this an advantage over other method. Basically, the activated carbon is conventionally carbonized, where sample are heated eternally. Thus, it leads to inefficient energy use. The nature of microwave that heats the material internally makes it an ideal solution for carbonizing sample faster. The electrical consumption and size of the material shown inversely proportional, giving this technique more advantage over conventional carbonization (Wahi & Senghie, 2007). In the present study, agricultural biomass have been

applied as a precursor for the preparation of activated carbon using microwave heating followed by metal oxide impregnation of CuO through mechanochemical processing for surface modification towards industrial textile wastewater treatment.

1.2 Problem Statement

Textile wastewater is hard to treat because they originate from multi processes which produce multi segments of wastewater. Expansive measure of suspended solids, high color and high chemical oxygen demand of the wastewater can cause serious ecological issues (Dhas, 2008). Industries have problems in complying with the standard discharge limits of textile effluent because they are becoming more stringent these days. Azo dyes can show toxic effects, especially carcinogenic and mutagenic events (Pineiro *et al.*, 2004). The azo dyes entering the body by ingestion can be metabolized to aromatic amines by the azoreductases of intestinal microorganisms. Mammalian liver enzymes and other organizations may also catalyze the reductive cleavage of the azo bond and the nitroreduction of the nitro group. In both cases, if N-hydroxylamines are formed, these compounds are capable of causing DNA damage (Arlt, 2002). At present, textile wastewater can be treated by a few techniques like coagulation and membrane bioreactor and iron anode but they can not be utilized independently because textile wastewater has non-biodegradable organics, color and high salinity (Ahn *et al.*, 1999).

Adsorption is a compelling technique for bringing down the convergence of dissolved dyes in the effluent resulting in colour removal. High adsorption capacity, extended surface area, high level of reactivity and microporous structure are the reasons why activated carbon is the most broadly utilized adsorbent. However,

commercially accessible activated carbons are exceptionally costly. Thus, there is a developing enthusiasm for utilizing material such as agricultural by-product for instance, rubber wood sawdust since they are abundantly available (Couto *et al.* 2012) and kenaf fibre (Ishak, 2007).

1.3 Objective

The main objectives of this study are:

- To produce activated carbon from rubber wood sawdust and kenaf fibre using microwave processing
- To synthesize CuO doped activated carbon produced by mechanochemical processing.
- To analyse the effectiveness of the produced activated carbon in the removal of dye from the textile wastewater.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to Kenaf

Kenaf (*Hibiscus cannabinus L.*) is a fibre plant of the local east-focal Africa. It has been developed for a few thousand years for fiber and nourishment. It is a typical wild plant of tropical and subtropical Africa and Asia. It has been source of textile fiber for such items as rope, bagging, twines and floor coverings. Kenaf is a promising source of crude material fibre for pulp, paper and other fibre items, and has been presented since world war II in China, Thailand, Egypt, South Africa, Mexico and Cuba. Kenaf has a one of a kind blend of long bast and short central elements which makes it suitable for a range of paper and cardboard items. Researchers at the ARS have tried a few kenaf pulping procedures, with the pulps being utilized to make a few evaluations of paper including newsprint, bond, covering crude stock and surfaced sized. Results have been certain, especially as far as paper quality, toughness, print quality and ink retention (Gowda, 2011).

2.1.1 Stalk

Kenaf stalks are for the most part round and relying upon assortment, thorns of the stalks of kenaf range from very modest to as vast as blackberry shrub. Kenaf stem consist of two sorts of fibre, including one long strand arranged in the cortical layer, and another containing short filaments situated in the ligneous zone. The focal territory, corresponding to pith, consists of sponge-like tissue. Hemp stalks are four sided with no thorns (Rymsza, 2010). They are depicted similar to a rigid herbaceous

stalk with well marked nodes at intervals of 4 to 20 inches that are obtusely four cornered and are fluted or channelled as shown in Figure 2.1 (Webber, 2011).



Figure 2.1: Stalk and core (Webber, 2002)

2.1.2 Core Fibre

The fibers in kenaf are found in the bast (bark) and core (wood). The bast constitutes 40% of the plant. "Unrefined fibre" isolated from the bast is multi-cellular, comprising of a few individual cells stuck together (Paridah et al, 2011). The individual fibre cells are around 2–6 mm long and slim. The cell divider is thick (6.3 μm). The center is around 60% of the plant and has thick ($\approx 38 \mu\text{m}$) however short (0.5 mm) and slim walled (3 μm) fiber cells (Nanko et al, 2005). Paper pulp is created from the entire stem, and along these lines contains two sorts of fibers, from the bast and from the center. The pulp quality is like hardwood.

2.2 Introduction of Rubber Wood Sawdust

Rubber wood sawdust was picked as the antecedent for activated carbons due to its accessibility as one of the fundamental plants in Southern East Asia. In Malaysia, the evaluated manor region of rubber trees is 1.82 million hectares, which adds to roughly 20% of the worldwide estate. Sawdust are refers to the tiny sized and powdery wood waste produced by the sawing of wood shown in Figure 2.2. In addition, rubber wood sawdust has a high carbon and low fiery debris content (Hameed, 2007).

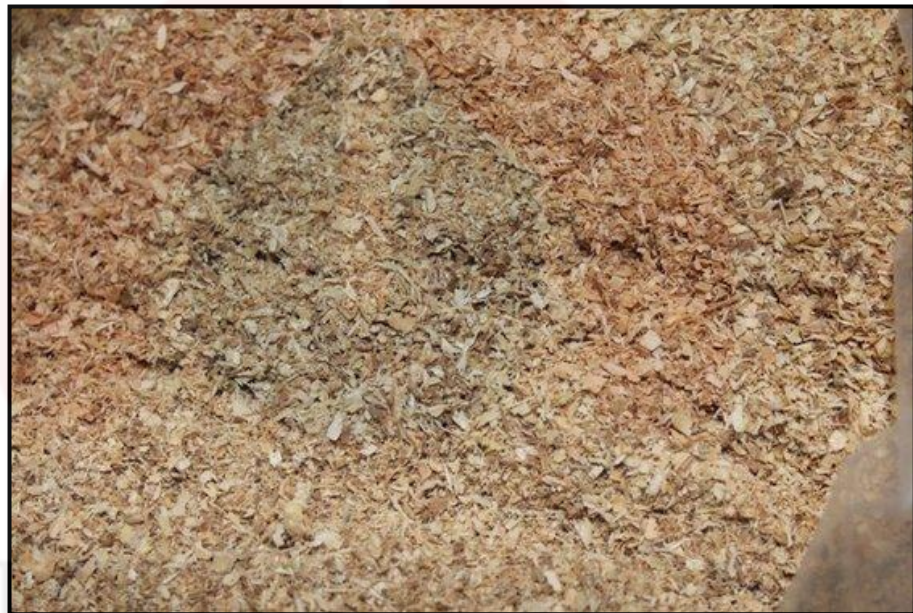


Figure 2.2: Rubber wood sawdust chip particles

2.3 Activated Carbon

Activated carbon has recently been utilized as an absorbent because of its ability for adsorption of the gas and fluid stage (Daifullah *et al.* 2003). Activated carbons are utilized as a part of wastewater treatment, drinking water purification (Heijman and Hopman, 1999) and fluid stage adsorption. The high applicability of

activated carbon is identified with its high porosity, quick absorption and thermal steadiness (Hesas *et al.* 2013). Activated carbon can be made with an extensive variety of structures, compositions and properties, depending upon the way of the organic precursor and procedure parameters.

2.3.1 Activated Carbon from Kenaf

Recent studies have demonstrated the capability of kenaf core and bast fibres to enhance bioremediation and adsorption process. There were earlier studies have been made aimed to prepare, characterize and evaluate the application of activated carbon derived from kenaf fiber (KFAC) to remove copper (II) from synthetic wastewater. Results obtained indicate that a very fast and efficient removal of copper ions can be achieved by using KFAC. The research indicates that the findings will be obliging up to a great extent for treating Cu (II) contaminated effluents, and at the same time it is economically feasible and environmentally friendly material which can be employed successfully for separation of Cu (II) on industrial scale (Chowdhury *et al.*, 2011). Whereas the other study were to synthesis the AC from kenaf core fiber using H_3PO_4 as activating agent and the results shows the increase of surface area and pore development after carbonize and acid treatment, presence of significantly different peak frequencies of functional group before and after activation which are the exact characteristics for a good activated carbon (Shamsuddin *et al.*, 2015).

2.3.2 Activated Carbon from Rubber Wood Sawdust

There were studies produce activated carbon from sawdust and the results indicates that compared to the commercial activated carbon, the sawdust carbon could be employed as a low cost alternative to produce commercial activated carbon

(Lam and Zakaria, 2008). The other study shows that the characteristic of the activated carbon that were produced from a biowaste product like rubber wood sawdust was found comparable to the commercial activated carbon (Prakash *et al.*, 2006).

2.3.3 Classification of Activated Carbon

Activated carbons are complex items which are hard to characterize on the premise of their behaviour, surface characteristics and other basic criteria. However, the general grouping of activated carbons based on molecule size divides them into powdered activated carbon (PAC), granular activated carbon (GAC), and extruded activated carbon (EAC) (Babel and Tonni, 2003).

2.3.3.1 Powdered Activated Carbon (PAC)

Typically, activated carbons are made in particulate structure as powders or fine granules under 1.0 mm in size with a normal width somewhere around 0.15 and 0.25 mm. Consequently, they exhibit a vast surface to volume proportion with a little diffusion distance. Activated carbon is characterized as the activated carbon particles held on a 50-network sifter (0.297 mm) (Salih, 2016).

PAC material is finer material. PAC is comprised of pounded or ground carbon particles, 95–100% of which will go through an assigned cross section sifter. The ASTM groups particles going through a 80-network strainer (0.177 mm) and smaller as PAC. It is not common to utilize PAC in a dedicated vessel, because of the high head loss that would happen. Rather, PAC is generally added directly to different procedure units, for example, crude water admissions, rapid blend basins, clarifiers, and gravity filters (Mohamad, 2015).

2.3.3.2 Granular Activated Carbon (GAC)

Granular activated carbon has a moderately bigger molecule size compared to powdered activated carbon and hence, exhibits a smaller outer surface. Dispersion of the adsorbent is consequently a vital variable. These carbons are suitable for retention of gasses and vapors, since they diffuse quickly. Granulated carbons are utilized for water treatment, deodorization and separation of components of the flow system and are also used in rapid mix basins (Ashish S. Saksule, 2012).

2.3.3.3 Extruded Activated Carbon (EAC)

Extruded activated carbon joins powdered activated carbon with a binder, which are intertwined and extruded into a cylindrical moulded activated carbon block with measurements from 0.8 to 130 mm. These are for the most part utilized for gas stage applications because of their low pressure drop, high mechanical strength and low dust substance (Mohamad, 2015).

2.4 Preparation of Activated Carbon

2.4.1 Chemical Activation

Impregnation with chemicals, for example, acids like phosphoric acid or bases like potassium hydroxide, sodium hydroxide or salts like zinc chloride, trailed via carbonization at temperatures in the scope of 450-900°C. Phosphoric acid and zinc chloride are used for the activation of lignocellulosic materials, when compared to zinc chloride, phosphoric acid is the most preferred because of the environmental

disadvantages associated with zinc chloride. Problems of corrosion and inefficient chemical recovery are also associated with it (Prahas *et al.*, 2008).

2.4.2 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).

Energy is supplied by an electromagnetic field directly to the material in microwave processing. This results in rapid heating throughout the material thickness with reduced thermal gradients. Volumetric heating can also save energy and reduce processing times. In general, carbon materials are very good absorbers of microwaves because they are easily heated by microwave radiation. This characteristic allows them to be transformed by microwave heating, giving rise to new carbons with tailored properties, then to be used as microwave receptors, in order to heat other materials indirectly or to act as a catalyst and microwave receptor in different heterogeneous reactions.

Microwave radiation will transfer electromagnetic energy into heat energy and heat up the core part of sample as shown in Figure 2.3. 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 (Jones *et al.*, 2001). The basis of the heat is quickly remove when the microwave is turn off and this method are more accurate and more effective (Dehdashti *et al.*, 2011). This method easy to control hence made the process become faster thus control the pollution (Jones *et al.*, 2001).

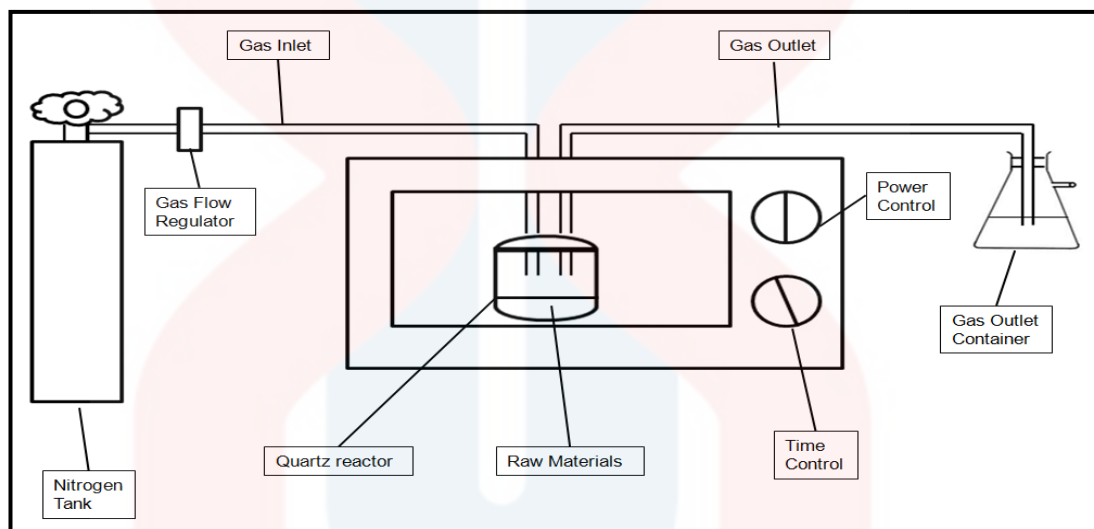


Figure 2.3: Apparatus set up for microwave processing (Kit, 2013)

2.4.2.1 Microwave Processing of Kenaf

Microwave processing is superior due to the energy savings compared to conventional ovens. Composites require 60 – 65 W to cure in the microwave where oven cures composites require 1600 W. With microwave processing the composites reach the cure temperature in approximately 20 minutes. Oven processing required approximately 95 minutes. Silane treated kenaf shows an improvement of 27% in modulus. This fundamental observation shows that the silane surface treatment improves adhesion of kenaf fibers.

2.4.2.2 Microwave Processing of Rubber Wood Sawdust

Locally available rubber wood sawdust was used as a low-cost alternative precursor for chemically activated carbons with $ZnCl_2$ as an activating agent. The results indicate that the high specific surface area activated carbon derived from rubber wood sawdust has great potential to serve as filler of dielectric and microwave absorbing materials (Shaaban *et al.*, 2015).

2.5 Textile Wastewater

Textile industry causes considerable higher effects to water contamination by releasing their effluents into various receiving bodies incorporates lakes, waterways and other open sewer. Significant toxins load from the textile industries are from the few of their wet-handling operations like scouring, bleaching, mercerizing and dyeing. Among these different procedures, dyeing process ordinarily utilizes an expansive measure of water for dyeing, altering and washing processes (Hauser, 2011). Along these lines, textile wastewater has a high COD concentration, a substantial measure of suspended solids, comprehensively fluctuating pH, solid shading, high temperature and low biodegradability caused by varying contaminants within the water environment (Patel and Vashi, 2015). There are many stages in water treatment in the textile industry. River water that are contaminated because of textile industry go through screening/ straining, oil removal from the wax, homogenization, neutralization, biological purification then lastly final color removal from dyes.

2.5.1 Dyes in Textile Wastewater

Dyes may be defined as substances that, when applied to a substrate provide color by a process that alters, at least temporarily, any crystal structure of the colored substances (Bafana *et al.*, 2011). The dyes can adhere to compatible surfaces by solution, by forming covalent bond or complexes with salts or metals, by physical adsorption or by mechanical retention (Kirk *et al.*, 1991). Dyes are classified according to their application and chemical structure, and are composed of a group of atoms known as chromophores, responsible for the dye color. In the textile industry, up to 200,000 tons of these dyes are lost to effluents every year during the dyeing and finishing operations, due to the inefficiency of the dyeing process (Ogugbue and Sawidis, 2011). Unfortunately, most of these dyes escape conventional wastewater treatment processes and persist in the environment as a result of their high stability to light, temperature, water, detergents, chemicals, soap and other parameters such as bleach and perspiration (Couto, 2009). However, environmental legislation obliges industries to eliminate color from their dye-containing effluents, before disposal into water bodies (Ogugbue and Sawidis, 2011).

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

The materials utilized for this study are kenaf core fibre (KCF) and rubber wood sawdust (RWS). These crude materials are gathered from the Saw Mill, Kota Bharu and Lembaga Kenaf Tembakau Negara, Kota Bharu respectively. The carbonized materials experience for chemical activation by utilizing phosphoric acid (H_3PO_4) as the activating agents. Textile dye wastewater was obtained from local batik factory in Kota Bharu (Razali Batik Sdn. Bhd.) to test the effectiveness of activated carbon produced.

3.1.1 Raw Materials Preparation

The crude materials, kenaf core fibre (KCF) and rubber wood sawdust (RWS) are washed with distilled water and the specimens will be cut down to small pieces and dried in the oven at the ($60^{\circ}C - 120^{\circ}C$) for 24 hours. Sieve the material in the range of 0.5-1.0mm. Then, wash the sieved crude material with distilled water to evacuate any buildups or impurities such as ash and dust. Then keep it at room temperature. The same methods above are rehearse for the rubber wood sawdust.

3.2 Production of Activated Carbon

3.2.1 Carbonization

Carbonize the raw material using furnace for an hour at 500°C to turn the raw material into char denoted as KF-Char and RW-Char from KCF and RWSD respectively.

3.2.2 Activation of Char

The resulting chars were subsequently impregnated with different concentrations of H₃PO₄ (v/v) (30%, 40%, 50%, and 60%) with impregnation ratios (IR) of 1:4 (w/w) for 24 hours of soaking duration. The dimensionless impregnation ratio was calculated in Equation 3.1.

$$IR = \frac{W_{H_3PO_4}}{W_{char}} \quad (3.1)$$

where, $W_{H_3PO_4}$ is the weight (g) of phosphoric acid and W_{char} is the dry weight (g) of char.

A specific amount of char for each precursor was soaked with H₃PO₄ and the mixtures were stirred to form a paste or slurry. The impregnation ratio of 1:4 (w/w) was based on the dry weight of the sample to H₃PO₄. The soaked samples with a different concentration of H₃PO₄ were filtered using a vacuum pump and dehydrated overnight in an oven at 105°C. After chemical soaking, the mixtures were activate by microwave processing. The radiation times of the specimen is 6 minutes with the 500 W input powers and a nitrogen stream of 20 ml/min. Therefore, the ideal mix for development of carbon yield and adsorption execution is gather in the microwave input power of 500 W and activation time of 6 min (Foo *et al.*, 2012). After the

activation period of 1 hour, the samples were cooled to room temperature under an inert atmosphere and washed with water followed by 0.1 M Sodium Hydroxide (NaOH) to remove the residual chemical agents until the pH value of the rinsed water was neutral. The samples were then dried in an oven at 105°C for 24 hours. The produced activated carbons were further crushed and sieved to get the particles size of 200-300 µm and were kept in an airtight container prior to analysis. The activated carbon produced for KCF and RWSD were denoted as KFAC and RWAC, respectively throughout the work. Record the weight reduction and rate yield.

3.2.3 Copper Oxide Assisted Activated Carbon

Mixing of RWAC and KFAC with CuO powder was carried out through solid state reaction of mechanochemical activation processing followed by carbothermal reduction. In order to obtain mechanically mixed activated carbon and CuO, RWAC and KFAC were blended with CuO powder according to the stoichiometric ratio of 1 mol % powder mole ratio. Alumina balls were then weighed to get the required ball to powder ratio (BPR). The mixtures were milled at room temperature with a ratio of grinder alumina ball to powder mass is 10:1 and a rotational speed at 30 rpm for 3 hours. The obtained impregnated samples were denoted as RW-CuO and KF-CuO. RW-CuO and KF-CuO were then undergone carbothermal reduction where both samples were introduced to firing process using electric muffle furnace (Model: JSMF-30T) at 650 °C for 1 hour for further CuO reduction process.

3.3 Characterization of Adsorbents

3.3.1 Thermal Behavior Analysis

Thermal behaviour and stability of raw and activated carbons was evaluated using thermogravimetric analyzer, TGA/DSC (Mettler Toledo). The temperature range of the analysis was room temperature to 900 °C with the heating rate of 10°C/min under nitrogen atmosphere.

3.3.2 Surface Area Analysis

The pore structure characterization of the carbon materials was carried out using a Micromeritics ASAP 2020 V3.04H (Micromeritics®) with N₂ adsorption isotherm measured at -196°C. Before performing the nitrogen gas adsorption at 77 K, the prepared adsorbent was outgassed under vacuum at 350°C for 6 hours to remove any moisture content from the solid surface. The micromeritics ASAP system uses the gas sorption technique to generate high quality data for applications that require high performance/high sample throughput. The BET surface areas of the activated carbon samples were calculated from the N₂ adsorption isotherms using the BET equation. The micropore volumes (V_{mi}) of the AC samples have been calculated by applying t-plot method.

3.4 Textile Wastewater

Collect the textile wastewater and keep it in a plastic container. Rinse the plastic container with hydrochloric acid, HCl to a pH under 2.0. After that, keep the plastic container in cooler. Next, boil the textile wastewater using a hot plate and cool it in room temperature. At that point, uproot the wax in the textile waste using

filtration technique. Then, keep the textile wastewater in a freezer to avert the development of microorganism.

3.4.1 Adsorption by Activated Carbon

Determine the concentration of dye in textile wastewater before and after treatment with UV-VIS. Four parameters will be studied which are the effect of acid loading, effect of activated carbon dosage, the impact of contact time of activated carbon with textile wastewater and effect of pH on the treatment.

3.4.1.1 Effect of Contact Time

Investigate the impact of contact time for 30 min, 60 min, 90 min and 120 min. Keep the amount of absorbent dose consistent at 0.4 g and shake on an orbital shaker operated at 150 rpm and 25°C. Filter the specimens using filter paper and examine the specimens using UV-VIS. Table 3.1 and 3.2 shows the effect of contact time using KFAC and RWAC.

Table 3.1: Effect different of contact time on textile dye removal by KCF

Samples	Acid loading (%)	Contact time of activated carbon with textile waste water (min)	Amount of activated used (g)	volume of textile waste water (ml)
KFAC	60	30	0.4	50
		60		
		90		
		120		

Table 3.2: Effect of different contact time on textile dye removal by RWS

Samples	Acid loading (%)	Contact time of activated carbon with textile waste water (min)	Amount of activated used (g)	volume of textile waste water (ml)
RWAC	60	30	0.4	50
		60		
		90		
		120		

3.4.1.2 Effect of Dosage of Activated Carbon Used

The initial volume of textile wastewater is 50 ml utilized with activation carbon varying in weight dosage, 0.4, 0.6, 0.8, 1.0, 1.4, 1.6, 1.8 and 2.0 g. Keep the contact time consistent at 90 min and shake on an orbital shaker operated at 150 rpm and 25°C. Specimens are then filter with filter paper and prepare to be analyzed using UV-VIS. Table 3.3 and 3.4 shows the effect of amount of activated carbon.

Table 3.3: Effect of dosage of KFAC on textile dye removal

Samples	Acid loading (%)	Amount of activated carbon used (g)	Contact time of activated carbon with textile waste water (min)	Volume of textile waste water (ml)
KFAC	60	0.4	90	50
		0.6		
		0.8		
		1.0		
		1.4		
		1.6		
		1.8		
		2.0		

Table 3.4: Effect of amount of RWAC on textile dye removal

Samples	Acid loading (%)	Amount of activated carbon used (g)	Contact time of activated carbon with textile waste water (min)	Volume of textile waste water (ml)
RWAC	60	0.4	90	50
		0.6		
		0.8		
		1.0		
		1.4		
		1.6		
		1.8		
		2.0		

3.4.2 Sample Analysis

Once the absorption with activated carbon was prepared, analyze the textile wastewater by UV-VIS using the dhitozone technique to check the absorbance of the effluent of textile wastewater. Calculate the percentage of dye removal due to bio adsorption using the following equation:

$$\text{Percentage of heavy metals removal (\%)} = \frac{[C_o - C_i]}{C_o} \times 100 \%$$

Where C_i and C_o was the initial and the final dye absorbance ($\mu\text{g/L}$) respectively.

CHAPTER 4

RESULT AND DISCUSSION

4.1 Physical Properties of Raw Material

Figure 4.1 shows the raw material of both kenaf and rubber wood. Then these raw materials went through the preliminary washing of the fibers with warm distilled water to remove dust-like impurities and inorganic matters on their surfaces. They were then dried at 105 °C for 24 hours to remove all moisture. In the drying process, it showed that KCF needs longer time to dry compared to RWS. Linked glucose residues which were highly hydrophilic in nature and permeable in water that was found in kenaf cause the KCF took more time to dry due to the high moisture content in fiber (Wong, 2003).

According to the visual interpretation, KCF was a strand of fiber stuck together while RWS was in wood chips particle form and RWS was darker in color than KCF. Wood particles of rubber wood sawdust were denser than kenaf fiber strand which directly shows that RWS is heavier than KCF. Hence, the amount of RWS was less than KCF although the weight of both KCF and RWS are the same.

The dried raw materials were cut into small pieces, pounded and sieved into 350µm to obtain a desirable size fraction. Figure 4.1 show the raw material rubber wood sawdust and kenaf core fiber before and after the raw material preparation.



Raw material of RWS



Raw material of RWS after pounded, ground and sieved



Raw material of KCF



Raw material of KCF after pounded, ground and sieved

Figure 4.1: Raw material RWS and KCF

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4.2 Activated Carbon Produced By Microwave Heating

4.2.1 Effect of H₃PO₄ Activation

After the raw materials went through all the preparation processes, samples were then soaked with four different concentrations, 30%, 40%, 50% and 60% of phosphoric acid (H₃PO₄) and undergo heating process in a microwave with 500W radiation power for 6 minutes each sample.

Figure 4.2 and 4.3 show the activated carbon produced from both KCF and RWS through microwave processing. Results obtained indicate that microwave processing can reduce the production time of activated carbon. Microwave radiation covers both internal and volumetric heating where the huge thermal gradient from the interior of the char particles to the cool surface which allows the microwave induced reaction to proceed faster and effectively at a lower bulk temperature, providing shorter processing time and saves energy (Deng, 2010).

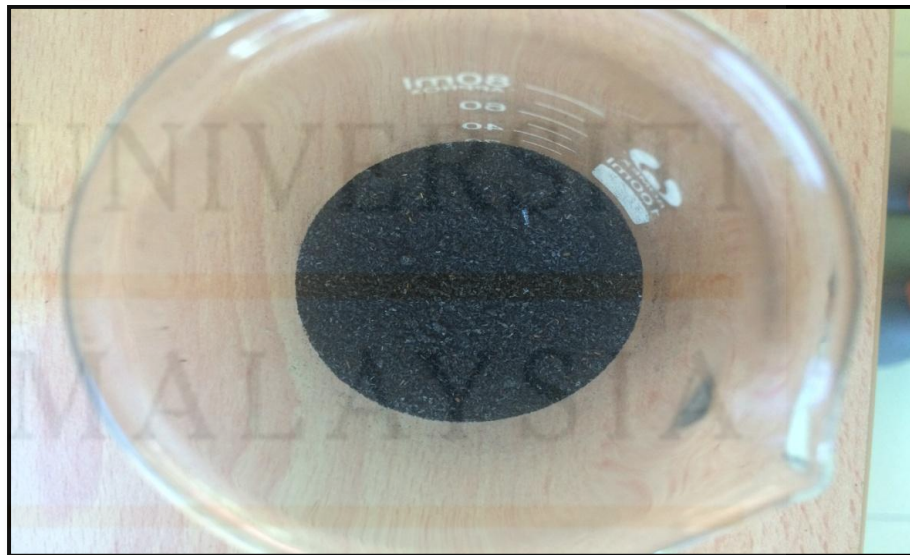


Figure 4.2: Activated carbon produced from kenaf core fiber (KFAC)



Figure 4.3: Activated carbon of rubber wood sawdust (RWAC)

20 gram of each KCF and RWS was used in activated carbon production. Replication has been done for every percentage of acid loading to attain the average reading. Table 4.1 shows the average weight after soak, average weight loss, average final weight and average percentage yield.

The nomenclature, operation conditions, density and yield of the different samples prepared by the method of chemical activation with phosphoric acid are summarized in Table 4.1. According to Table 4.1, both samples KFAC and RWAC were increased in weight after soaking with phosphoric acid, H_3PO_4 . The weight of carbonized KCF after soaking with 30% acid loading was increased from 15 g to 17.3 g while the weight of 30% acid loading of RWAC increase from 20 g to 24.1 g. The weight continues to increase for other acid loading (40% - 60%) for each sample. The weight of after soak KFAC and RWAC 60% acid loading increase the least compared to others which were only 0.8g for KCF and 1.2g for RWAC. The

results obtained show that, as expected, as H_3PO_4 concentration increases the weight will also increase gradually. This means that the weight of residue after phosphoric acid impregnation shows a great influence of the H_3PO_4 concentration on the amount of H_3PO_4 loaded on both samples. Hence, the more concentrated can create a more synergic effect and increase the weight of the sample.

Table 4.1: Percentage weight loss and percentage yield of KFAC and RWAC sample activated carbon

Samples	Acid concentration (%)	Initial weight (g)	Average weight after carbonization (g)	Average weight after soak (g)	Average final weight after washing and drying (g)	Average percentage of weight loss (%)	Average percentage yield (%)
KFAC	30	50.0	15.0	17.3	14.90	70.20	29.80
	40	50.0	15.0	16.8	13.15	73.70	26.30
	50	50.0	15.0	16.5	13.92	72.16	27.84
	60	50.0	15.0	15.8	12.90	74.20	25.80
RWAC	30	80.0	20.0	24.1	19.38	75.78	24.23
	40	80.0	20.0	23.9	17.82	77.73	22.28
	50	80.0	20.0	23.0	17.99	77.51	22.49
	60	80.0	20.0	21.2	16.92	78.85	21.15

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4.3 Weight Loss of Activated Carbon

The results for the weight loss of KFAC and RWAC after the microwave processing were shown in Table 4.1. From the result obtained, the percentage weight loss for KFAC 30% acid loading was 70.20% while RWAC 30% acid loading was 75.78%. It shows that 30% acid loading for both KFAC and RWAC have the least percentage of weight loss. The average percentage weight loss of both KFAC and RWAC increased gradually from 30% - 60% acid loading concentration. This indicates that as phosphoric acid, H_3PO_4 concentration increases the percentage of weight loss will increase.

The increase of concentration will stimulate gasification of char and increase total weight loss of the activated carbon. As the phosphoric acid dosage increased, more potential sites could be penetrated and occupied by the activating agent, which benefited the following pore-opening and widening processes. However, an insulating layer might be formed if excessive phosphoric acid could not promote further activation of the char (Yakout *et al.*, 2011).

Figure 4.4 below shows percentage weight loss of KFAC is lower compared to RWAC because there was less space between molecules and KFAC had more compact fiber structure, therefore, the weight loss was less than RWAC. The excess phosphoric acid will promote gasification of char and increased total weight loss of carbon. Hence the concentration of acid will increase with the percentage of weight loss (Yakout *et al.*, 2011).

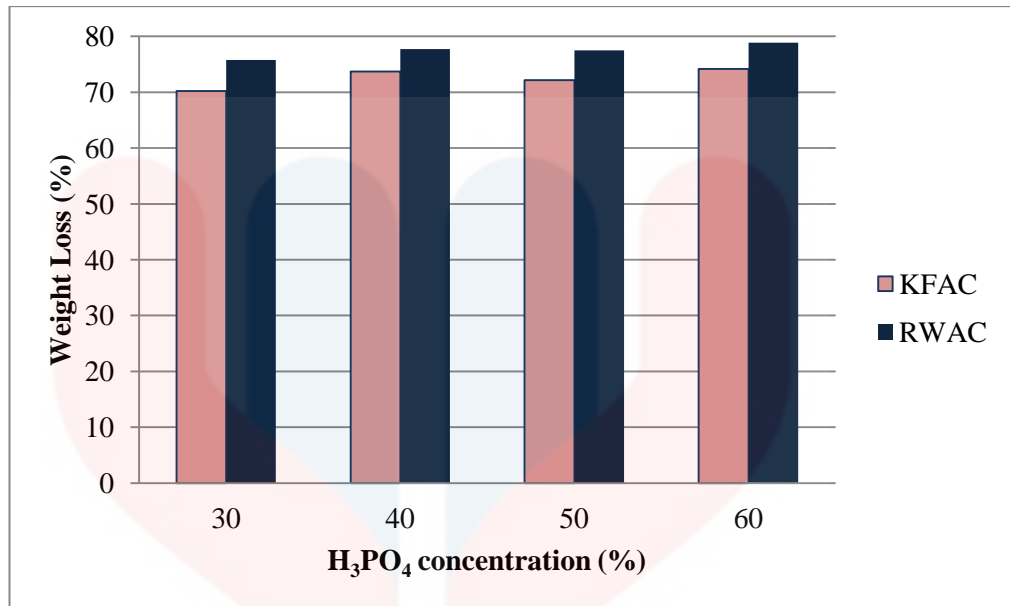


Figure 4.4: Percentage weight loss of activated carbon sample

4.3.1 Percentage Yield of Activated Carbon

Figure 4.5 shows the percentage yield of both KFAC and RWAC. The percentage yield of KFAC at 30% acid loading was 29.80% while RWAC was 24.23%. The percentage yield of KFAC and RWAC both gradually decrease with acid concentration (30% - 60%). The results obtained clearly show that, as expected, as H₃PO₄ concentration increases the total yield of the process decreases. The main components of KFAC and RWAC both were lignin, lignocelluloses material with hemicollulose and cellulose. The reaction of lignocellulose with phosphoric acid begins as soon as the components are mixed, the acid first attacks hemicellulose and lignin because cellulose is known to be more resistant to acid hydrolysis (Jagtuyen and Derbyshire, 1998). As the acid concentration increases, the aromatic condensation reactions also take place among the adjacent molecules, which result in

the evolution of gaseous products from the hydroaromatic structure of carbonized char leading to decreased the yield of carbon (Timur *et al.*, 2006).

The percentage yield of KFAC generally higher than RWAC, due to RWAC pore structure is more open and wider than KFAC. Hence, increased acid concentration results in development of the total surface area, pore volume and mesoporous area, therefore, reduce the percentage yield.

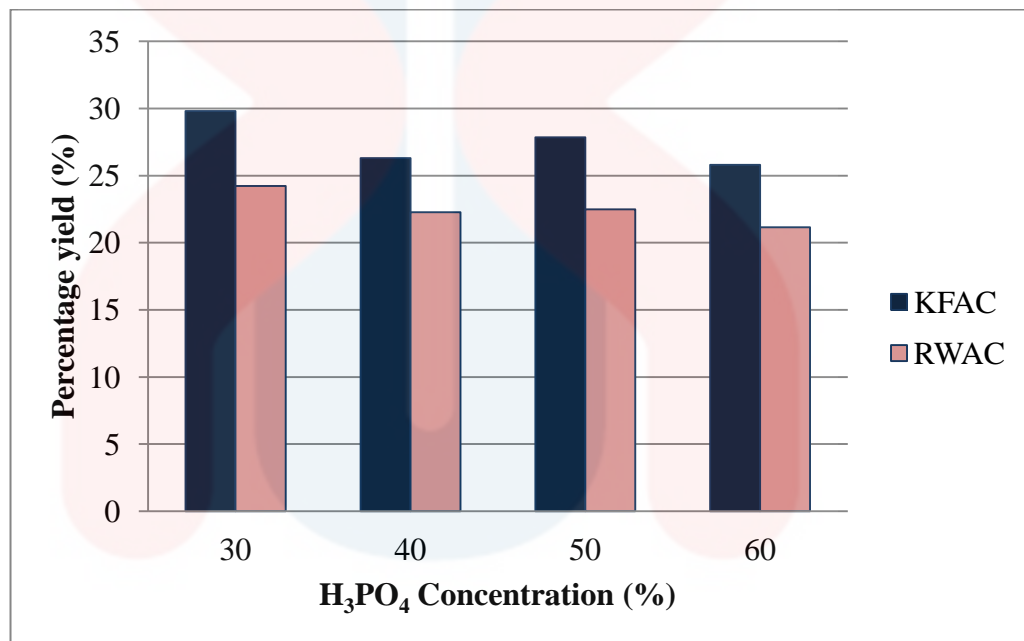


Figure 4.5: Percentage yield of activated carbon sample

4.4 Textile Wastewater

Figure 4.6 showed the color of the wastewater after treatment with activated carbon, the color was the first contaminant and need to be removed. This showed that the KFAC and RWAC activated carbon act as a good adsorbent by reducing the color of the wastewater. RWAC can be used as an effective adsorbent material which can be used successfully for removal of color. The KFAC and RWAC had large

surface area for the removal of the color thus reduces the contaminant (Zainul *et al.*, 2008).



Figure 4.6: Textile wastewater after treatment and digestion

4.4.1 Effect of Contact Time on Removal of Dye in Textile Wastewater

Table 4.2 show the removal of dye in textile wastewater by RWAC activated carbon with four different contact times. 0.4 g of activated carbon produced from KFAC and RWAC of 60% acid loading were used to treat 50ml of effluent textile wastewater with the pH of 11.2 (industrial) in 30 minutes, 60 minutes, 90 minutes and 120 minutes. Figure 4.7 shows the removal of dye process by RWAC and KFAC activated carbon using orbital shaker at 150rpm to study the effect of contact time.



Figure 4.7: The removal of dye process by RWAC and KFAC activated carbon

Table 4.2: Effect of contact time on the removal of dye by RWAC activated carbon.

Variables	RWAC			Absorbance
	Time (min)	Weight (g)	pH	
Contact Time	30	0.4	11.2	0.296
	60			0.282
	90			0.276
	120			0.291

Table 4.3: Effect of contact time on the removal of dye by KFAC activated carbon.

Variables	KFAC			Absorbance
	Time (min)	Weight (g)	pH	
Contact Time	30	0.4	11.2	0.304
	60			0.274
	90			0.267
	120			0.284

According to Table 4.2, the removal of dye by adsorption of RWAC activated carbon at 30 minutes was 0.296. The absorbance of treated wastewater continues to decrease for 60 minutes and 90 minutes. At 120 minutes, the absorbance value increased to 0.291. This shows that the maximum removal of dye by RWAC activated carbon was at 90 minutes. The same pattern happened to KFAC. Table 4.3 indicates the removal of dye by adsorption of KFAC activated carbon at 30 minutes was 0.304. The absorbance value of contact time 60 minutes and 90 minutes continues to decreased just like RWAC. The removal then decrease after 90 minutes. At 120 minutes, the value of absorbance of treated textile wastewater was 0.284 which is higher than the previous contact time (90 minutes).

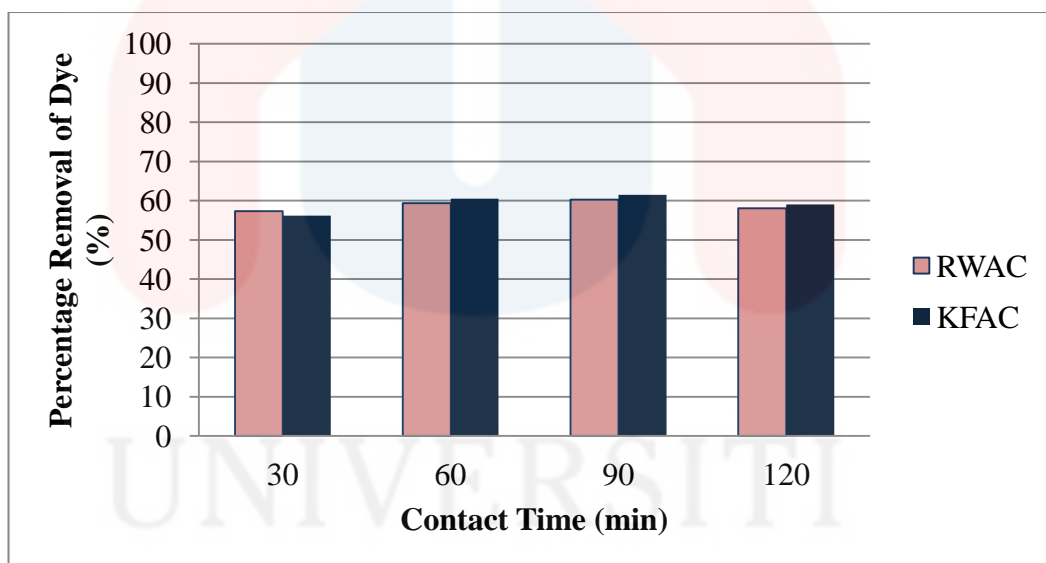


Figure 4.8: Effect of contact time on percentage removal of dye by RWAC and KFAC with 0.4 g of activated carbon.

The percentage removal of dye for both RWAC and KFAC activated carbon are shown in Figure 4.8. The figure shows that at 30 minutes, RWAC activated carbon treated 57.34% of the dye in the textile wastewater while KFAC activated

carbon treated 56.19% of dye in the same industrial wastewater. The percentage of dye removal of textile wastewater by both RWAC activated carbon and KFAC activated carbon slightly increased gradually from 30 minutes to 90 minutes, then the percentage of dye removal decrease after 90 minutes. The maximum percentage removal of dye in textile wastewater by the effect of contact time was 90 minutes for both RWAC and KFAC activated carbon.

From the result obtained, the removal percentage of dye for both RWAC and KFAC were gradually increased from 30 minutes to 90 minutes. It showed that as the contact time increases the percentages of removal also increase. The removal of dye in industrial textile wastewater by RWAC and KFAC activated carbon adsorption maintained then decreased after 90 minutes of the treatment process. The removal percentage maintained then decreased after 90 minutes because it has reached its optimum contact time and attained equilibrium. The fast adsorption at initial stage may be due to the availability of uncovered surface area and the remaining active sites on the adsorbents. The removal percentage of dye by RWAC and KFAC activated carbon attained about 90 minutes of shaking time. The attainment of equilibrium adsorption might have been due to reduction in the available active adsorption sites on the adsorbent with time resulting in the limited mass transfer of the adsorbent molecules from the bulk liquid to the external surface of adsorbent (Sofia, 2014).

4.4.2 Effect of Dosage of Activated Carbon Used On Removal of Dye in Textile Wastewater

The amount of activated carbon used was 0.4, 0.6, 0.8, 1.0, 1.4, 1.6, 1.8 and 2.0 g. The contact time of the activated carbon with the textile wastewater and textile

wastewater used pH was fixed at 90 minutes and 11.2 (industrial). It is important to fix the amount of the activated carbon to design the optimum treatment systems and for a quick response to the analysis. This result was to show the best amount of activated carbon used for the removal of dye in textile wastewater at the optimum contact time.

Table 4.4 and 4.5 showed the absorbance value of 50ml effluent textile wastewater treated with 0.4 g to 2.0 g of RWAC and KFAC activated carbon in 90 minutes time.

Table 4.4: Effect of dosage on the removal of dye by RWAC activated carbon.

Variables	RWAC			Absorbance
	Time (min)	Weight (g)	pH	
Dosage	90	0.4	11.2	0.300
		0.6		0.266
		0.8		0.263
		1.0		0.240
		1.4		0.237
		1.6		0.236
		1.8		0.221
		2.0		0.202

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Table 4.5: Effect of dosage on the removal of dye by KFAC activated carbon

Variables	KFAC			Absorbance
	Time (min)	Weight (g)	pH	
Dosage	90	0.4	11.2	0.268
		0.6		0.246
		0.8		0.237
		1.0		0.229
		1.4		0.228
		1.6		0.225
		1.8		0.226
		2.0		0.218

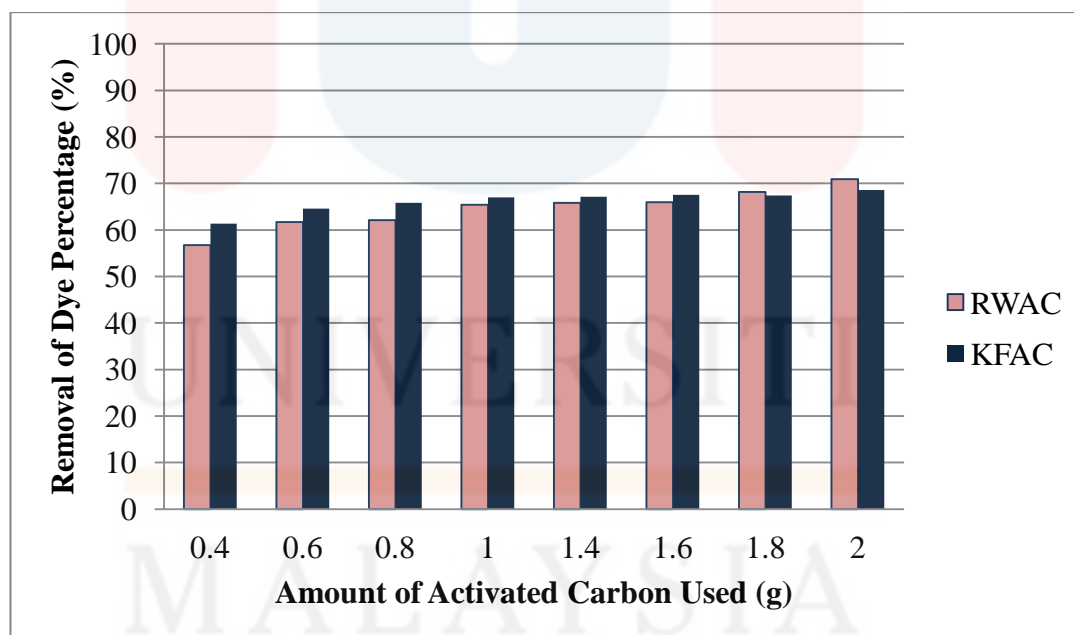


Figure 4.9: Effect of dosage of activated carbon used for removal of dye in textile wastewater at 90 minute

The effect of dosage on the removal of dye by RWAC activated carbon is shown in Table 4.4. The contact time was fixed to 90 minutes because the previous parameter showed that 90 minutes was the optimum duration for this treatment and 50 ml of effluent textile wastewater were used to study the effect of RWAC activated carbon dosage. In the table stated that the absorbance was the highest when 0.4 g of RWAC activated carbon used in the dye treatment. The absorbance values show an overall decrease as the dosage of activated carbon increase. 2.0 g RWAC activated carbon have the lowest absorbance value in Table 4.4. This indicates that more dye in the effluent textile wastewater treated.

Table 4.5 above shows the absorbance values of treated wastewater by various amount of KFAC activated carbon. The same patterns occur to KFAC treatment. From the table, carbon used amount that have the highest absorbance value was 0.4 g. Then the absorbance value continues to decrease as the dosage of KFAC activated carbon increase. The maximum removal of dye by KFAC was with 2.0 g of activated carbon (0.218 absorbance value).

Figure 4.9 above shows the removal of dye in textile wastewater percentage by 0.4, 0.6, 0.8, 1.0, 1.4, 1.6, 1.8 and 2.0 g of both RWAC and KFAC activated carbon for 90 minutes contact time. Removal of dye in effluent textile wastewater percentage for 0.4 g of RWAC was 56.77% while KFAC was 61.38%. They were both have the lowest percentage of removal. According to the figure, the percentage of removal of dye increased as the amount of activated carbon used increase. This applied to both RWAC and KFAC. For KFAC activated carbon, the percentage of removal maintained at 0.8g to 1.4g then at 1.8g it continues to increase. The same pattern occurs to RWAC too where at 0.6g to 1.4g the percentage of removal maintained then at 1.8g the percentage increase. The maximum removal of dye in

textile wastewater percentage was 70.89% for RWAC and 68.58% for KFAC; both used 2.0g of activated carbon for the treatment. This indicates that 2.0g of both RWAC and KFAC is the best amount in for study.

From the result obtained, the removal of dye in effluent textile wastewater increased from 0.4 g to 2.0 g for both RWAC and KFAC activated carbon. It is apparent that the percentage removal of dye increases rapidly with increase in the dose of the adsorbents due to the greater availability of the exchangeable sites or surface area. The figure also has shown an increase in the bio sorption percentage as amount of activated carbon of increases to a certain level and then decreases; this may be due to overlapping of adsorption sites as a result of overcrowding of adsorbent particles (Kumar *et al.*, 2009).

From the result shown in Figure 4.9, the highest removal of dye was with 2.0 g of RWAC activated carbon which was 70.89%. This indicates that RWAC has high absorption ability than KFAC. Thus, RWAC could have a higher surface area, complex pore structure and high porosity (Vijayakumar, 2013).

4.4.3 Effect of pH on Removal of Dye in Textile Wastewater

Four different pH (3, 5, 7 and 9) of textile wastewater were prepared in order to determine which pH leads to the highest efficiency of dye removal. The effect of pH was studied by altering the pH of the textile wastewater which was initially 11.2 to 3, 5, 7 and 9. The pH was adjusted with 0.5M HCl. Figure 4.10 shows the set up of the pH altering process. The study of the effect of pH in dye removal of textile wastewater by RWAC and KFAC activated carbon were projected in tables and figure below.



Figure 4.10: The set up for pH altering process

Table 4.6: Effect of pH on the removal of dye by RWAC activated carbon

Variables	RWAC			Absorbance
	Time (min)	Weight (g)	pH	
pH	90	2	3	0.02
			5	0.084
			7	0.098
			9	0.148

Table 4.7: Effect of pH on the removal of dye by KFAC activated carbon

Variables	KFAC			Absorbance
	Time (min)	Weight (g)	pH	
pH	90	2	3	0.075
			5	0.109
			7	0.179
			9	0.238

Table 4.6 shows the effect of pH on the removal of dye by RWAC activated carbon. The treatment process took 90 minutes and used 2.0g of RWAC activated carbon. From the table, we can see that the absorbance of treated wastewater was the highest when we use pH 9 textile wastewater which was 0.148. The absorbance value decreased as the pH decrease. pH 3 had the least absorbance value which was 0.02. The same pattern occurs when KFAC activated carbon used for the treatment. From Table 4.7, the highest value of absorbance was 0.238 which was pH 9 while the least was pH 3 (0.075). The absorbance value of treated wastewater increased as the pH increased. This indicates that the lower the pH, the better the adsorption of both RWAC and KFAC.

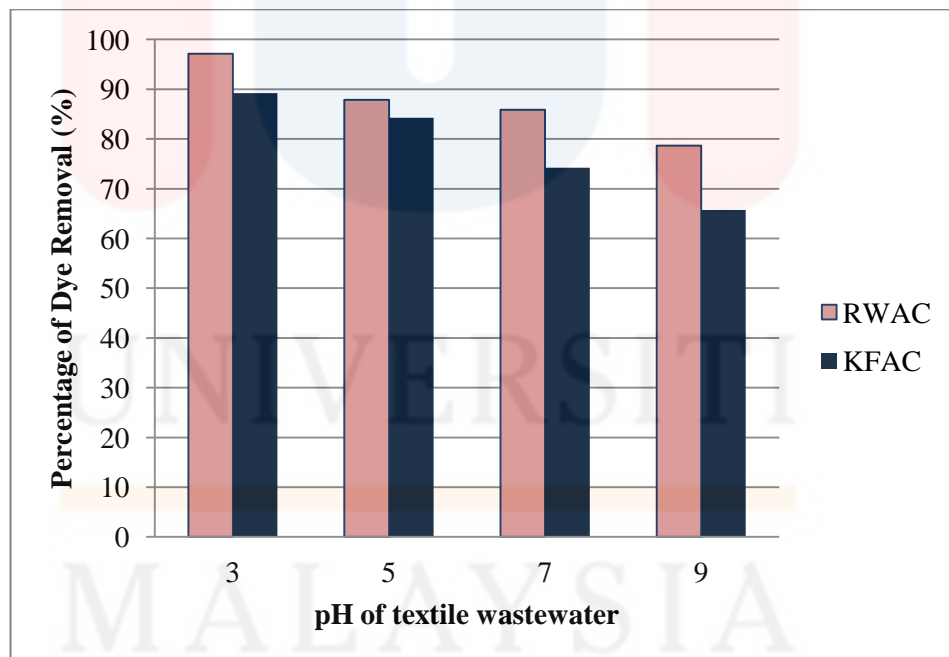


Figure 4.11: Effect of pH on removal of dye in textile wastewater with 2.0g activated carbon at 90 minutes

From the figure above, percentage of dye removal for pH 9 with 2.0g of RWAC and KFAC activated carbon was 78.67% and 65.7%. The figure shows that there is significant increase of dye removal percentage from pH 9 to pH 3. The highest percentage of dye removal for KFAC was pH 3 (89.19%). The same form occurs for RWAC, the maximum percentage of dye removal was 97.11% (pH 3 textile wastewater). It can be seen in Figure 4.11 that the dye in textile wastewater was treated by adsorption of different effluent pH.

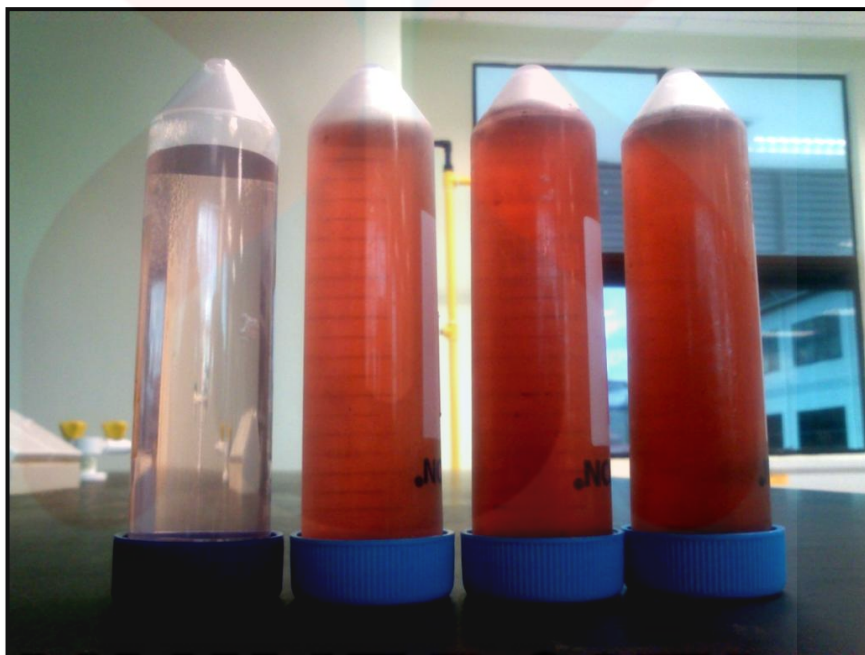


Figure 4.12: Different gradient of wastewater that were treated with

pH 3, 4, 5 and 9 wastewater

The result obtained indicates that the percentage of dye removal in textile wastewater that was treated with both RWAC and KFAC activated carbon for 90 minutes increase as the pH decrease. One of the main variables affecting the adsorption process is pH, influencing not only the surface charge of adsorbent, the

degree of ionization of the material present in the solution and the dissociation of functional groups on the active sites of the adsorbent, but also the solution dye chemistry. In this fact, pH control the adsorption of dyes onto suspended particles because both adsorbed molecules and adsorbent particles may have functional groups which are affected by the concentration of H^+ ions in the solution and which are involved in the molecular adsorption process at the active sites of adsorbent (Haddad *et al.*, 2013).

4.5 Effect of CuO Asisted Activated Carbon

Figure 4.13 below shows how the activated carbon for both RWAC and KFAC with the best acid loading (60%) were impregnated with CuO by milling process with 30 rpm for 3 hours. The prepared RWAC-CuO and KFAC-CuO then went through treatment process based on best parameters from the previous RWAC and KFAC treatment which are 90 minutes contact time and 2.0 g of activated carbon. The effluent textile wastewater used are wastewater that already been altered to pH 3, pH 5 and industrial wastewater with pH 11.2. The study of the effect of CuO assisted activated carbon were projected in tables and figures below.



Figure 4.13: Milling process of RWAC-CuO and KFAC-CuO

Table 4.8: Absorbance of RWAC impregnated with CuO and without CuO based on best parameters.

Variables	RWAC-CuO			RWAC		
Contact Time (min)	90			90		
Dosage (g)	2.0			2.0		
pH	3	5	11.2	3	5	11.2
Absorbance	0.019	0.059	0.225	0.020	0.084	0.255

Table 4.9: Absorbance of KFAC impregnated with CuO and without CuO based on best parameters.

Variables	KFAC-CuO			KFAC		
Contact Time (min)	90			90		
Dosage (g)	2.0			2.0		
pH	3	5	11.2	3	5	11.2
Absorbance	0.001	0.031	0.225	0.075	0.109	0.255

Table 4.8 above shows the effect of CuO assisted RWAC on the removal of dye of the textile wastewater. The treatment process took 90 minutes and used 2.0 g of RWAC and RWAC-CuO. From the table, we can see that the absorbance of treated wastewater was the highest when we use the industrial wastewater which was 0.225 for RWAC-CuO and 0.255 for RWAC without CuO. The value of absorbance seems to decrease as the pH decrease. The pH that had the least absorbance value is pH 3 which was 0.019 for RWAC-CuO and 0.020 for RWAC respectively. The same trend occurs when KFAC and KFAC-CuO used for the treatment. From Table 4.9, the highest value of absorbance for KFAC-CuO was 0.225 which was the pH 11.2

wastewater while for KFAC was 11.2 (industry) too which was 0.255. pH 3 of KFAC-CuO had the lowest absorbance which was 0.001 and KFAC was 0.075. The absorbance value of treated wastewater decreased as the pH decreased. This indicates that the lower the pH, the better the adsorption of both RWAC and KFAC especially when assisted with CuO.

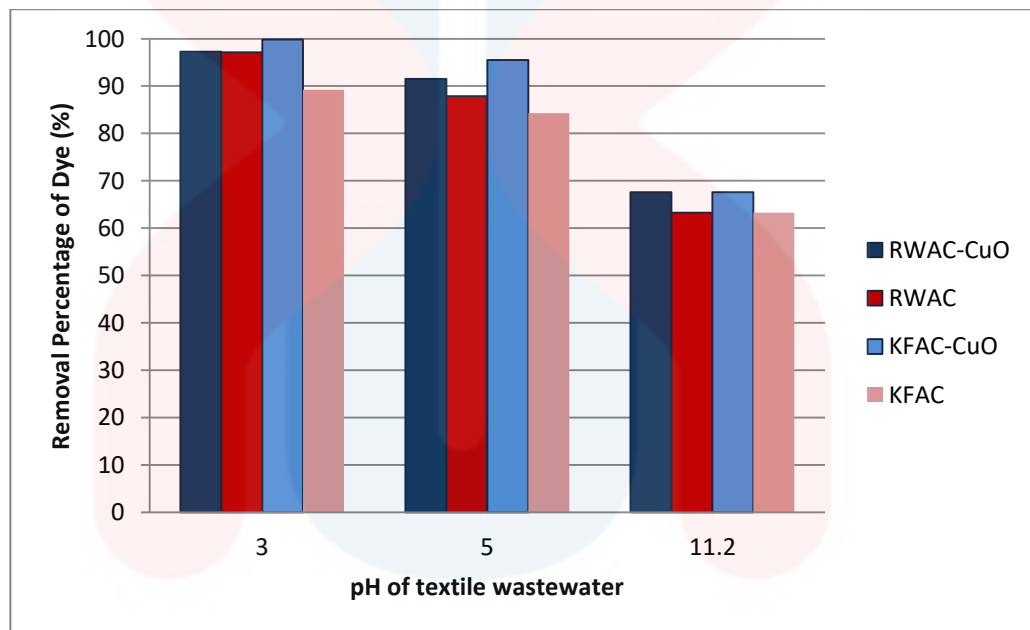


Figure 4.14: Removal percentage of dye in textile wastewater treated with 2.0 g of RWAC and KFAC impregnated with CuO and without CuO for 90 minutes

The dye removal percentage between CuO assisted activated carbon and without CuO are shown in Figure 4.14. Percentage of dye removal for industrial textile wastewater with the pH of 11.2 for both RWAC-CuO and RWAC were 67.57% and 63.25% while KFAC-CuO and KFAC was 67.57% and 63.25%. The figure shows that pH 11.2 have the least percentage of removal compared to others.

Dye removal percentage of pH 11.2 to pH 3 shows significant increase. The maximum percentage of dye removal for KFAC-CuO was pH 3 which was 99.85%. KFAC, RWAC-CuO and RWAC have the highest percentage of removal of dye in textile wastewater which are 89.19%, 97.26% and 97.11%. All three of them are pH 3 textile wastewater.



Figure 4.15: Different pH (pH 11.2, 5, 3; from left to right)

wastewater treated with KFAC- CuO

The result obtained indicates that the percentage of dye removal in effluent textile wastewater with both RWAC and KFAC assisted with CuO and without for 90 minutes increases as the pH decrease. One of the main variables affecting the adsorption process is pH. In this fact, pH control the adsorption of dyes onto suspended particles because both adsorbed molecules and adsorbent particles may have functional groups which are affected by the concentration of H^+ ions in the

solution and which are involved in the molecular adsorption process at the active sites of adsorbent (Haddad *et al.*, 2013). Figure 4.15 shows the different gradient of wastewater treated with 2.0 g of KFAC-CuO for 90 minutes with 3 different pH (industrial; 11.2, pH 5 and pH 3). Both CuO assisted RWAC and KFAC shown significant removal of dye in textile wastewater compared to the normal RWAC and KFAC. CuO was selected to be used in this study due to its high efficiency in removal of several pollutants and the enhancement of reaction activity (Tseng *et al.*, 2003).

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

The study showed the suitability of kenaf core fiber and rubber wood sawdust for the dye removal using activated carbon from kenaf fiber and rubber wood sawdust produced by microwave induced H_3PO_4 activation. The activated carbon was produced in a short activation time by microwave heating. Microwave heating used in this study shortened the processing period, save energy and produced a high-quality activated carbon by opening of previously inaccessible pores and creation of new pores, presumably due to the interior and volumetric heating of microwave irradiation. In chemical activation, increasing concentration could strengthen the activation stage, thus higher amount and beyond the optimum impregnation value, the pores could be blocked by excess agent and burning, leading to a reduction of the accessible area resulting adsorption reduction.

The removal of dye from textile waste water was significantly influenced by contact time, amount of activated carbon used and pH. The result showed that rubber wood sawdust was much better adsorbent for removal of dye compared to the kenaf core. This was shown at the percentage removal of dye by the effect of contact time, amount of activated carbon used and effect of pH. From the result, it shows that, for RWS the best percentage removal of dye with effect of contact time was 59.79 %, percentage of removal of dye for the effect amount of adsorbent dosage was 70.89 %, percentage of removal of dye for effect of pH was 97.11 %, therefore the removal percentage of dye with CuO assisted RWAC was 97.29 %. For KCF, the best removal percentage of dye with effect of contact time was 61.52 %, percentage of removal of dye for the effect amount of adsorbent dosage was 68.59 %, percentage

removal of dye for effect of pH was 87.75 %. Hence, the removal percentage of dye with CuO assisted KFAC was 98.55 %.

Presented data show that microwave heating is an effective, and method which can be used for preparation of activated carbon with high surface area and pore volume and the effectiveness of activated carbon produced by microwave processing on the removal of dye from textile wastewater.

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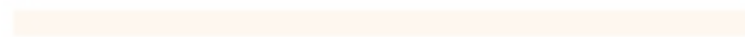
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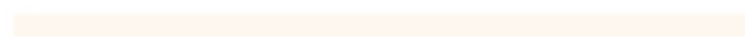
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APPENDIX A



Figure 1: Raw material carbonization



Figure 2: Char was soaked with H_3PO_4



Figure 3: Wash process after soaked with H_3PO_4



Figure 4: N_2 gas was used in activated carbon activation

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Figure 5: pH of activated carbon after wash

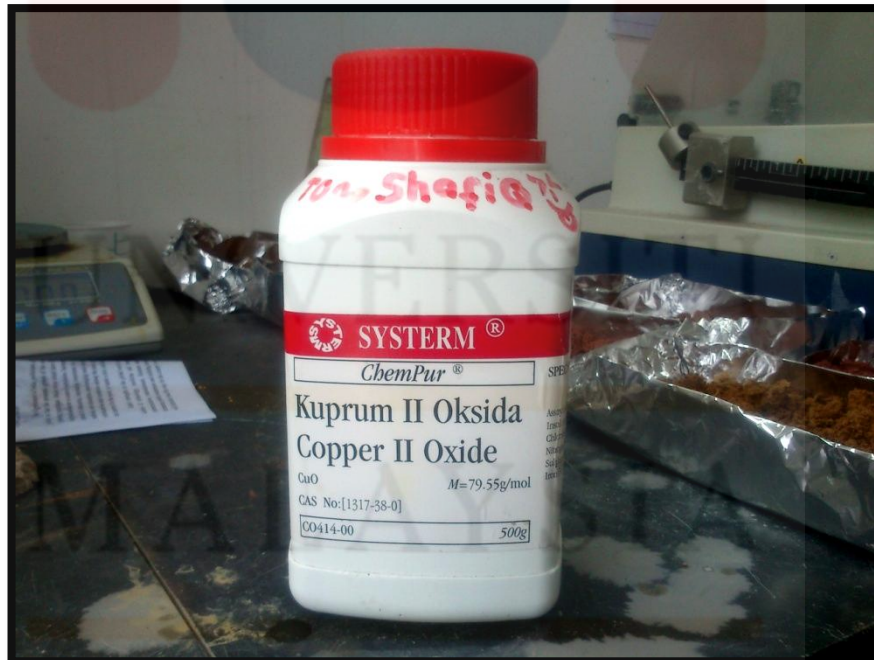


Figure 6: Copper II oxide for impregnate with KFAC and RWAC