DEGRADATION OF METHYLENE BLUE USING CuO PREPARED USING CONVENTIONAL SOLID STATE METHOD

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2017

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by

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Thesis submitted in partial fulfilment of the requirement

for the degree of Bachelor of Chemical Engineering

May 2017

ACKNOWLEDGEMENT

First and foremost, I would like to convey my sincere gratitude to my supervisor, Dr.Noorashrina A.Hamid for her precious encouragement, guidance and generous support throughout this work.

I would also extend my gratitude towards all the MSc and PhD students for their kindness cooperation and helping hands in guiding me carrying out the lab experiment. They are willing to sacrifice their time in guiding and helping me throughout the experiment besides sharing their valuable knowledge.

Apart from that, I would also like to thank all staff members of School of Chemical Engineering, Universiti Sains Malaysia for their kindness cooperation and helping hands. Indeed their willingness in sharing ideas, knowledge and skills are deeply appreciated.

Once again, I would like to thank all the people, including those whom I might have missed out and my friends who have helped me directly or indirectly. Their contributions are very much appreciated. Thank you very much.

Tai Ming Li

May 2017

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LIST OF SYMBOL

Symbol		Unit
C_e	Equilibrium concentration of adsorbate	mg/dm^3
C_o	Highest initial adsorbate concentration	mg/L
C_t	Dye concentration at time, t	mg/L
K	Freundlich isotherm constant	$mg/g\ (dm^3/mg)^{1/n}$
k	Langmuir adsorption constant	dm^3/mg
m	Mass of adsorbent	g
n	Estimation of adsorption intensity	-
R	Universal gas constant	8.314 J/mol K
T	Time	Min
T	Absolute temperature	K
Vm	Monolayer adsorption capacity of the adsorbent	mol/g

LIST OF ABBREVIATIONS

CAS	Chemical Abstracts Service
CuO	Copper Oxide
FESEM	Field Emission Scanning Electron Microscope
IUPAC	International Union of Pure and Applied Chemistry
MB	Methylene blue
NO_3	Nitrate
SEM	Scanning electron microscopy
TEM	Transmission Electron Microscope
UV	Ultraviolet
XRD	X-Ray Powder Diffraction

PENURUNAN METILENA BIRU MENGGUNAKAN CuO YANG DIHASILKAN MENGGUNAKAN KAEDAH KEADAAN PEPEJAL BIASA

ABSTRAK

Oksida Kuprum (CuO) telah dihasilkan melalui kaedah keadaan pepejal biasa dan digunakan bagi mengaji aktiviti degradasi dalam proses kelompok. Dalam sintesis keadaan pepejal ibasa yang telah dijalankan, suhu yang tinggi daripada 400^{o}C kepada 600^{o}C dan etanol telah digunakan untuk menghasilkan CuO. Suhu yang optimum dikena 600^{o}C kerana ia boleh menghasilkan CuO yang boleh menurunkan 8.5% MB. Analisis XRD menunjukan bahawa sistem kristal CuO adalah sistem monoklinik. Parameter yang diperolehi daripada XRD adalah a = 4.6860Å, b = 3.4280Å, c = 5.1330Å dengan jumlah sel 81.84 Å^{3}. Analisis SEM menunjukkan bahawa CuO mempunyai saiz partikel yang standard dalam lingkungan 37.0μm. Analisis SEM juga menunjukkan bahawa CuO mempunyai permukaan yang tidak teratur. Kesan kepekatan awal pewarna (100ppm-600ppm), masa sentuhan (24-48 jam) dan suhu (30^{o}C) turut dikaji. Peratusan yang tertinggi direkodkan untuk penyingkiran MB pada kepekatan sebambah 600ppm MB kepekatan. Masa sentuhan 48 jam telah didapat lebih berkesan daripada 24 jam dalam proces penurunan MB. Kesimpulannya, penjerapan MB ke CuO mempunyai kecekapan lebih tinggi pada perbezaan kepekatan yang lebih besar, masa sentuhan yang lebih panjang dan suhu yang lebih tinggi digunakan semasa sintesis CuO.

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ABSTRACT

The degradation of methylene blue (MB) dye onto copper oxide (CuO) synthesized using solid state method was investigated in a batch adsorption process. Copper Oxide (CuO) was successfully synthesized using solid state synthesis which involves heat treatment with temperatures from 400^{o}C to 600^{o}C. The optimum preparation temperature was 600^{o}C, which have resulted in 8.5% MB removal. XRD analysis shows that the crystal system of CuO is a monoclinic system and therefore all the diffraction peaks has been indexed using the CuO monoclinic phase. The obtained parameters were a = 4.6860Å, b = 3.4280Å, c = 5.1330Å with a volume cell of 81.84 Å^{3}. SEM analysis indicates that a standard particle size for CuO is 37.0μm and that it has irregular surface. The effect of initial dye concentration (100ppm-600ppm), contact time (24-48 hours) and solution temperature (30^{o}C) were also evaluated through. Highest percentage of removal of MB was observed in MB concentration of 600ppm. A longer contact time of 48 hours was shown to be more effective than 24 hours. As a conclusion, the adsorption of MB onto CuO has higher efficiency at larger concentration difference, longer contact time and higher temperature used for CuO synthesis.

CHAPTER ONE

INTRODUCTION

1.1 Introduction

Wastewater treatment is a wide area that contained many different fields of knowledge. This paper aims to focus on the treatment of one of the most concerning pollutants in wastewater that is dye. Dye is widely used in multiple industries but it is most commonly found in the textile industry. Dyes are colour substance that are able to transfer their colour to other substances (Soniya et al., 2015). Discharge from the wastewater in this industry has exceptionally high amounts of dye contaminants. These dyes are also highly resistant to natural decomposition and biodegradation. The discharge of the dye from the industry to the environment will contribute to environmental pollution (Hamidreza Sadegh, 2016).

Methylene blue (MB) was chosen as a model for the source of colouring and pollution in the industry (Farrouji et al., 2015). This because methylene blue contains harmful effects to health and it is also found commonly in the wastewater of the textile industry.

Copper (II) oxide (CuO) is known as a material with high efficiency with low cost. It is also known to be non-toxic in nature (Farrouji et al., 2015). Due to this, CuO has large number application and uses in various fields. Therefore, this research aims to utilize the properties of CuO for the adsorption of MB.

1.2 Problem statement

Over the past years, there have been many research in different fields for treating wastewater. Some of these examples are the utilization of high gravity technologies (Jiaoa et al, 2017), crystallization techniques (Lu et al., 2017) and even development of pathways for prevention of micro-plastics to micro plastics to enter the aquatic environment (Ziajahromi et al., 2017).

However when utilizing these new and emerging technologies may prove to be high cost. Besides that, utilizing these new technologies may induce a lot of maintenance.

An effective method used to treat dye in wastewater over the years is acknowledge and accepted in the industry is adsorption (Orthmana et al., 2003).

CuO as adsorbent will be more cost efficient as it is largely available in different countries. There have been different research on synthesizing CuO such as catalytic oxidation and adopting aqueous precipitation method (Farrouji et al., 2015; Mustafa et al., 2013). However, catalytic oxidation and aqueous precipitation method uses catalyst and solvent that may lead to high cost.

This investigation employs solid state synthesis method for the production of CuO and finding the optimum condition in doing so.

1.3 Scope of study

In this work, the copper (II) nitrate ($Cu(NO_3)_2 \cdot 3H_2O$) was used to prepare copper oxide (CuO) for Methylene Blue (MB) dye removal. The preparation of CuO was carried out via solid state synthesis which utilizes an agate and mortar to crush the $Cu(NO_3)_2 \cdot 3H_2O$ while adding small traces of ethanol. Heat treatment is applied for the the conversion of $Cu(NO_3)_2 \cdot 3H_2O$ to CuO. Study was carried out for finding out the effects of using different temperatures for synthesis.

The CuO was then characterized in terms of surface area, surface morphology, proximate content, and elemental content and surface chemistry by using surface area analyzer, SEM and XRD respectively.

The CuO was then used to investigate the adsorption behaviour of MB dye onto CuO. In order to carry out the analysis, batch adsorption study was carried out by examined the effect of adsorbent initial concentration (100-600 ppm), contact time (24-48 hour), solution temperature (30°C) for adsorption of dye onto CuO prepared.

1.4 Research Objectives

The main objectives of this study are:

i. To prepare and evaluate the optimum parameter for the fabrication of Cooper Oxide (CuO) using solid state reaction method.

ii. To evaluate the degradation of Methylene Blue (MB) using in-house prepared absorbents.

1.5 Organization of thesis

This thesis consists of five main chapters and each chapter contributes to the sequence of this study. The following are the contents for each chapter in this study :

Chapter 1 introduces the usage of adsorbents on dyes in textile industries, problem statement, research objectives and organization of thesis.

Chapter 2 discusses the literature review of this study. An insight into wastewater, methylene blue as a substitute for wastewater, discussion on adsorption process, CuO and raw material used in preparing CuO are discussed in this chapter. Moreover, the isotherm models are included as well.

Chapter 3 report on the materials of the experiment and the details of methodology. It examines on the description of equipment and materials used, batch adsorption experiment, experimental procedure and description of factors affecting the adsorption process.

Chapter 4 refers to the experimental results and discussions of the data obtained. Further elaboration on the effect of different factors on batch system adsorption, the results on equilibrium, kinetic and thermodynamic properties are provided in this chapter.

Chapter 5 concludes all the findings obtained in this study. Recommendations are also included as well.

CHAPTER TWO

LITERATURE REVIEW

2.1 Wastewater

A dye is commonly referred to a substance that is us to intensify or to alter colour of a certain material. There are many industries that utilizes dye in their daily operations. The industry known to be as the largest consumer of dye is the textile industry (Mustafa et al., 2013). Due to this, the discharged by the textile industry contains large amounts of colour contaminants. Therefore, this industry is one of the major source contributors to water contamination (Prashanth et al., 2017). Where compared to other types of colouring agents in wastewater, dyes are the primary source of contamination (Chiang et al, 2013).

The dye that has been discharged to the environment are mostly stable organic pollutants that are able to persist in the natural environment. This is because these organic pollutant contain high immunity to light, temperature and even towards detergents and some forms of soaps (Prashanth et al., 2017).

The ecosystem is highly affected by the colour water that has been produced by the industry though the wastewater (Farrouji et al., 2015). It is also known to be an international concern to the public and the world that the toxic materials may produce harmful effect to the human health such as poisoning and carcinogenic effect and to the environment where animals and plants that have been exposed by the wastewater have been affected as well.

Based on the reason above, it is imperative to find a cost effective and advance methods for treating dyes from wastewater.

2.2 Methylene Blue

Melthylthioninium Chloride or it is otherwise known as methylene blue is a dye and a type of medication. It has the molecular formula of $C_{16}H_{18}CIN_3SCl$ and it is a heterocyclic aromatic chemical compound (Raizada et al., 2014). It is a cationic character that has a lot of industrial application (Soniya et al., 2015). Methylene Blue is usually used as a dyeing and colouring agent for various industries such a paper, pencil and the textile industry. The textile industry utilizes methylene blue as a colorant.

It is a toxic substance that causes bladder irritation, anemia and gastrointestinal problems (Bhattacharjee et al., 2016). Due to its hazardous properties, methylene blue must be extracted properly from the industry waste to prevent any adverse health effect to the public and to prevent harm to the environment.

In this investigation, commercial Methylene Blue, $C_{16}H_{18}CIN_3S$, was chosen as a model source of pollution(Farrouji et al., 2015). This was required for the evaluation of the catalytic activity of the solid state synthesise catalyst. Evaluation of the methylene blue colour removal was completed by measuring the absorbance decrease by using an UV-Visible spectrometer apparatus.

Research on adsorption shows that maximum adsorption capacities were obtained at the initial concentrations of MB concentration of 250 ml L^{-1}. A powerful driving force will be provided to overcome the mass transfer resistance between the aqueous phase and solid phase when there is an increase in the initial concentration of MB (Karagoz et al., 2008).

2.3 Copper Oxide

Copper Oxide is a compound that contains two elements which is copper and oxygen. There are 4 different components that may be refer to cooper oxide which are copper (I) oxide, copper (II) oxide, copper peroxide, and copper (III) oxide.

Copper (I) oxide (Cu_2O) is a widely used material for colour pigmentation and an anti–fouling agent for marine paints. It is also known for to be a type of fungicide as well. Depending on the size of the particles, (Cu_2O) can be either yellow or red in colour. Copper peroxide (CuO_2) is a dark olive green solid and it is an unstable component that decomposes to other copper oxides and oxygen. Copper (III) oxide (Cu_2O_3) is used as a component for high-temperature superconductors.

Copper (II) oxide (CuO) can be used as wood preservatives and colour pigmentation. The colour that may be produced during the pigmentation process varies with black, grey, and sometimes blue, red, and green. It is also sometimes used in wielding with copper alloys.

The reason why CuO is used in many fields of various industry is because of its unique properties and features. CuO is capable of becoming a photocatalyst due to it being a p–type semiconductor (Raizada et al, 2014). Some of these features include chemical stability, high electron communication features, large interfacial areas, homogeneity, highly reactive surfaces, unusual optical and catalytic properties, etc (Hong et al., 2002).

There have been previous research on different methods of synthesising CuO such as using solution combustion method, aqueous precipitation method and catalytic oxidation (Raizada et al., 2014 ; Mustafa et al., 2013; Farrouji et al, 2015).

In the solution combustion method, copper nitrate $Cu(NO_3)_2$ was chosen as the precursor. The sample was placed on a petri dish then introduced into a furnace for a period of time at 350^{o}C.Thermal hydration occur followed by ignition at one spot with release of gaseous products such as nitrogen and oxygen (Raizada et al, 2014).

XRD analysis shows that the diffraction peaks of CuO was assigned to be (110), (002), (200), (-113), (-311) and (220) and that CuO has a monoclinic structure (Raizada et al, 2014).

The study on adsorption from this method is by varying the CuO amount from 1mg to 5mg at constant room temperature. It was seen that optimum removal was seen at 1mg.This might be due to the driving force caused by concentration difference. From the results, it is seen that 80% of MB is achieved with 20 mg/l CuO for 2 mins (Raizada et al, 2014).

In the catalytic oxidation, copper (II) sulphate pentahydrate ($CuSO_4 \cdot H_2O$) was used as the precursor. The sample was dissolved in 30ml of distilled water with 0.2g of sodium dodecyl sulphate (SDS) then mixed and stirred for 5 minutes with aqueous NaOCl solution to get a bleu suspension. When another bath of 20 ml of NaOH was added and stirred for 10 minutes, a black suspension was obtained. The black suspension was filtered with water several times and was dried in air at 100 ^oC for 24h (Farrouji et al., 2015).

XRD analysis shows all diffraction peaks of CuO was indexed in the monoclinic phase where the obtained parameters were a = 4.653Å, b = 3.410Å, c = 5.108Å with a volume cell of 79.94 $Å^3$ (Farrouji et al., 2015).

The results on MB adsorption shows that degradation does not significantly depend initial dye concentration. A trend is shown where when the MB concentration increases, dye degradation efficiency decreases. The decrease in catalytic activity was explained by the saturation of the catalyst (Farrouji et al., 2015).

The product from the synthesized CuO product contains highly ordered nanowires.

For the aqueous precipitation method, copper sulphate 5-hydrate was used as a precursor and NaOH as its stabilizing agent.

The mixing was done in 125ml distilled water with copper sulphate 5-hydrate (Merk) and hydrocyl ammonium chloride. In a cool water bath, the mixture was allowed to cool with well swirling. Then sodium hydroxide was added in 750ml of distilled water. The precipitate was then cooled for a while before removing the supernatant liquid. The left over oxide was then transferred to a 250 ml flask were the volume was mixed. Decantation was repeated for washing of the content until the sample was free of chlorine. Suction filtration was applied and the residue was washed with 95% alcohol and ether. The residue was then dried to get Cu_2O in air oxidized conditions at 200^oC to 250^oC .The temperature was increased to 300 ^oC for the formation of CuO.

XRD analysis shows that the CuO produced contained monoclinic structure with lattice parameters of a = 4.688Å, b = 3.422Å, c = 5.131Å with a volume cell of 82.31 $Å^3$ (Mustafa et al., 2013).

In this investigation, CuO is synthesize by using the solid state method to be used as an effective adsorbent for the adsorption of methylene blue. Solid state synthesis

2.4 Solid State Synthesis

There are a few methods of solid state synthesis which are solid state reactions, film deposition, and crystal growth. This research primarily focus on the first method which is in the solid state reaction route which prepares the sample of polycrystalline solids starting from a mixture of solid components (Smart et al., 2005).

It is required to heat up the solid to high temperatures, sometimes to a range of $2300K$, for the reaction to occur at an acceptable rate (Smart et al., 2005). However, in the case of this research the temperatures of range from 400°C to 600°C was chosen. The usage of high temperatures was required to induce heat diffusion of the reactants.

Due to the need for the production of copper oxide, copper (II) nitrate ($Cu(NO_3)_2 \cdot 3H_2O$) was chosen used as a reagent. This is due its availability and ease of obtain it. After sufficient amounts have been weight out, the reagent must then be crush and grinded. This is usually done with a mortar and a pestle. Little amount of volatile organic liquid must be added to ease the mixing of the reagents. After that the organic liquid was allowed to vaporize before placing it for heat treatment.

After that, the sample can be sent for characterization process. This can be done by sending the sample to X-Ray Powder Diffraction (XRD), Scanning Electron Microscope (SEM) or even Transmission Electron Microscope (TEM) for analysis.

2.5 Adsorption isotherm

Under normal circumstances such as vapour-liquid and liquid-liquid equilibrium, theories such as Raoults Law, Daltons Law and Henry's Law can be applied to estimate the phase distribution. However, in the case of vapour-solid equilibrium and liquid-solid equilibrium, conventional theories mentioned cannot be used. Therefore, it is required to collect data of the samples and analyse it using a different method. This method has come to be known as the analysis of the adsorption isotherm.

Adsorption isotherm is the plot of the solute loading on the adsorbent against the concentration or partial pressure made by the fluid. The data from the plot is taken at a constant temperature over a range of concentrations of the fluid.

For the distribution of the solute between the fluid and the solid phase, a dynamic phase equilibrium must established. This is same for all the cases whether the fluid is either gas, vapour or liquid. There are many different ways of expressing the equilibrium mentioned above, such as in terms of concentrations or partial pressure for the adsorbate in the fluid and the solute loading time on the adsorbent. For the adsorbate, partial pressure will be used when the adsorbate is in a gaseous state while concentration will be used when the adsorbate is in a liquid state. For the adsorbent, it will be expressed in terms of mass, mole, or even volume per unit mass, etc.

This investigation primarily focus on 2 types of isotherms which are the Freundlich Isotherm and Langmiur Isotherm.

2..5.1 Freundlich isotherm

An empirical equation was created by Freundlich to represent the isothermal variation of adsorption on the quality of gas adsorbed by unit mas of solid adsorbent with concentration (Mustafa et al., 2013).The following equation is known to be empirical equation by Freundlich:

$$\frac{x}{m} = KC_e^{\frac{1}{n}} \tag{2.1}$$

The equation above can then be further rearranged to:

$$\ln \frac{x}{m} = \frac{1}{\mathrm{n}} \ln C_e + \ln K \tag{2.2}$$

where,

$\frac{x}{m}$ = Amount of adsorbate adsorbed per unit mass of adsorbent (mol/g),

C_e = Equilibrium concentration of the adsorbate (mol/dm^3),

K = Freundlich isotherm constant,

n = Estimation of adsorption intensity.

As the temperature rises, the increase in K will indicate that the affinity to methylene blue is much more favourable in high temperatures. If the K value decreases as the temperature increase, this is a sign that the adsorption affinity of methylene blue on copper oxide is less approving at higher temperatures (Mustafa et al., 2013).

2.5.2 Langmuir isotherm

Another adsorption isotherm is known as the Langmuir isotherm was founded with a few assumptions (Mustafa et al., 2013). The different assumptions that exist are:

1. Phase transitions does not exist.
2. Absorptions occur at specific homogeneous sites.
3. Equivalent adsorption sites where each site can only accommodate 1 molecule.
4. The monolayer absorption with a finite number of adsorption sites

Langmuir model can be expressed by the following linear equation:

$$\frac{C_e}{x/m} = \frac{1}{kV_m} + \frac{C_e}{V_m} \tag{2.3}$$

where,

C_e = Equilibrium concentration of adsorbate (mol/dm^3),

$\frac{x}{m}$ = Amount of adsorbate adsorbed at equilibrium (mol/g),

V_m = Monolayer adsorption capacity of the adsorbent (mol/g),

k = Langmuir adsorption constant (L/mg).

Consequently, a graph plot of $\frac{C_e}{x/m}$ against C_e produces a straight line with the slope of $\frac{1}{V_m}$ and intercept of $\frac{1}{kV_m}$. The adsorption coefficient can be determined from the slope and intercept of the straight line.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials and equipment

In this study, copper (II) nitrate trihydrate, were used for the production of copper oxides. It was supplied by Ever Gainful Enterprise Sdn Bhd under a company named R&S Chemicals. The table below shows the properties of copper (II) nitrate.

Table 3.1 Properties of copper (II) nitrate (PubChem, 2017)

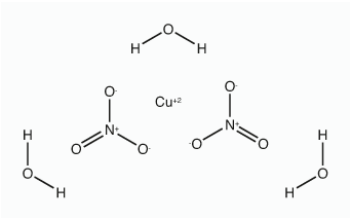
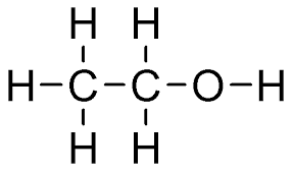
Properties	
Common name	Copper (II) nitrate trihydrate
IUPAC name	Copper (II) nitrate trihydrate
Other name	Copper Nitrate Trihydrate**,** Cupric Nitrate Trihydrate**,** Copper(II) Nitrate 3-hydrate
Molecular formula	$Cu(NO_3)_2 \cdot 3H_2O$
Molecular weight	241.599 g/mol
CAS number	10031-43-3
Chemical structure	

Figure 3.1 Copper (II) nitrate trihydrate used in this investigation

The volatile organic liquid chosen used to aid the homogenization in this research is ethanol. Like copper (II) nitrate trihydrate, the ethanol was also obtained from the company call R&M Chemicals. The table properties of ethanol.

Table 3.2 Properties of Ethanol (PubChem, 2017)

Properties	
Common name	Ethanol
IUPAC name	Ethanol
Other name	Ethyl alcohol; Alcohol; Methylcarbinol; Grain alcohol; Ethyl hydroxide
Molecular formula	CH_3CH_2OH
Molecular weight	46.069 g/mol
CAS number	64-17-5
Chemical structure	

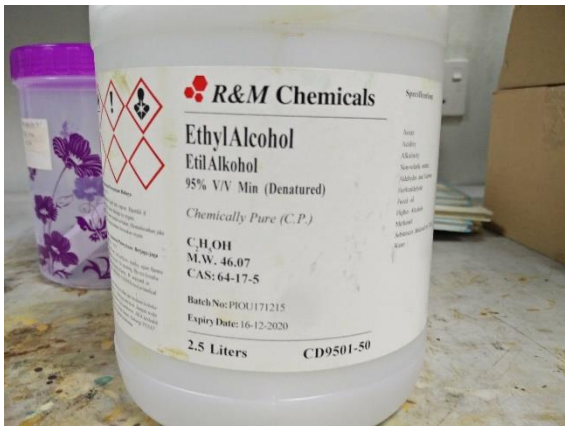
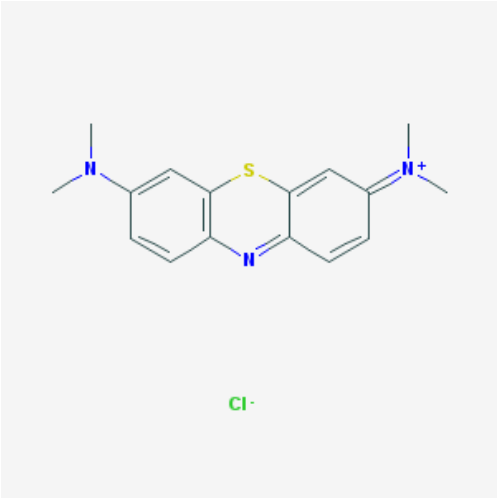


Figure 3.2 Ethanol used in this investigation

Methylene blue (MB) as an adsorbate in this investigation. The table properties of methylene blue.

Table 3.3 Properties of Methylene Blue (PubChem, 2017)

Properties	
Common name	Methylene Blue
IUPAC name	[7-(dimethylamino)phenothiazin-3-ylidene]-dimethylazanium chloride
Other name	Blue N Methylene , Blue Methylene, Basic blue 9 , Swiss Blue,
Molecular formula	$C_{16}H_{18}ClN_3S$
Molecular weight	319.851 g/mol
CAS number	61-73-4
Chemical structure	

3.2 Solid state synthesis

Copper (II) nitrate ($Cu(NO_3)_2 \cdot 3H_20$) was placed in the agate mortar. Small traces of ethanol was then added on the $Cu(NO_3)_2 \cdot 3H_20$ crystals. A pestle was then used to grind the $Cu(NO_3)_2 \cdot 3H_20$. The grinding process was continued for 10 minutes before it was scooped out and place in a crucible.

Figure 3.3 The agate mortar and pestle used in this investigation

The grinded sample was placed in a crucible before placing it in the furnace.

Figure 3.4 The crucible that was used in this investigation

The crucible was then placed in the middle of the furnace. The furnace was programmed to meet the suitable temperature, rate of heating and the duration of the heating process. The furnace used was a Carbolite CWF 1200 furnace.

Figure 3.5 The furnace used for heating process

3.2.1 Characterization of the synthesize materials

X-Ray Diffraction (XRD) was conducted in order to examine the structure of the synthesized sample. The scanning range was set to be 20 to 70° (2Ɵ) CuKα was used as a radiation source with wavelengths, λ, of 1.54056 A.

SEM was used to investigate the morphology of synthesized sample. Field Emission Scanning Electron Microscope (FESEM) Zeiss Supra 35VP was utilized for the analysis of the sample.

Figure 3.6 The FESEM Zeiss Supra 35VP with its computer systems used SEM analysis

Figure 3.7 The CuO sample was placed on a stage before SEM analysis

3.2.2 Degradation of Methylene Blue

The batch adsorption analysis was conducted using a set of Erlenmeyer flasks (250ml). 0.20 g of the prepared CuO was added into each flask filled with 100 ml of the prepared dye solutions. Six different dye concentrations of 100,200,300,400,500 and 600ppm were prepared by mixing a known amount of dye with deionized water. After that, the Erlenmeyer flasks were sealed and placed in the isothermal water bath shaker at a speed of 60 rpm and 30^{o}C for 24 hours and 48 hours.

All the samples that contain methylene blue was analysed using Cary 60 UV-Vis by Agilent Technologies as shown in the picture below. UV-Vis is also known as ultraviolet-visible spectrophotometer. This type of particular spectrometer is able to adsorb spectroscopy or reflectance spectroscopy in the ultraviolet visible spectral region. Methylene blue (MB) was scanned between 660 to 670 nm for different concentrations to obtain the optimum wavelength (Raizada et al, 2014).

Figure 3.5 Ultraviolet-visible spectrophotometer used in this research

3.3 Experimental procedures

3.3.1 Solid State Synthesis

Copper (II) nitrate and ethanol were used to prepare a copper oxide. Copper (II) nitrate was chosen as a reactant due to versatile nature and it is easily obtained. The increase in surface area leads to increase of the reaction rate. The mass of the copper (II) nitrate were weighed out to be the mass equivalent of 1 mole of copper oxide. An agate mortar and pestle were used to manually grind the copper (II) nitrate in small quantities.

In this process, a small amount of volatile organic liquid was added to smoothen the synthesis. Ethanol was added to aid the homogenation process. After continuous crushing and adding small amounts of ethanol until a paste is formed. The paste was then grinded and mixed thoroughly. This then leads to the ethanol been evaporated usually around 15 to 20 minutes.

Due to the high temperatures around 400°C to 600°C , a small crucible made of clay was use to contain the samples.

After carefully placing the sample in the crucible, the crucible was then place for heating in the furnace. Different samples was to heated to different temperatures of 400°C, 450°C, 500°C and 550°C.

The products from the furnace was removed and characterized using x-ray powder diffraction (XRD) and SEM analysis for structure and morphology.