

PERPUSTAKAAN UMP



0000073692

THE WASTE RECYCLING OF COPPER PHTHALOCYANINE (CuPC)

MOHD SHAZUFARIZAL ZUHRI BIN ZAINAL

**Report submitted in partial fulfilment of the requirements
for the award of Bachelor of Applied Science (Honours) in Industrial Chemistry**

**Faculty of Industrial Sciences & Technology
UNIVERSITI MALAYSIA PAHANG**

2012

ABSTRACT

Copper Phthalocyanine (CuPC) is one of the most stable pigments towards light, temperature and chemicals. CuPC is prepared by reacting copper chloride, urea, phthalic anhydride and catalyst with high boiling solvent. The objective of this method is to determine whether the 10% of the unreactive reactants can be recycled. There are 2 stage of work, which first stage is crude CuPC preparation and second stage is analyst 10% of unreactive reactants if can be recycled or not. Blue crude CuPC has been prepared by reaction of phthalic anhydrite with urea in presence of copper ions with a small amount of catalyst in inert organic solvent. An unreactive reagent has been collected from the crude and analyzed. Waste of crude CuPC can be recycled by adding some minimum amount of urea, phthalic anhydrite and catalyst or without catalyst and it can be used to make a new crude . The used of catalyst increase yeild and faster the reaction. Analysis of successful product has been done by using FTIR. By using this method, the cost can be reduced because the unreacted reagents can be reuse to produce pigment and this will also reduce damage for the environment.

ABSTRAK

Copper Phthalocyanine (CuPC) adalah salah satu pigmen yang stabil terhadap cahaya, suhu dan bahan kimia. CuPC dihasilkan melalui tindak balas di antara baja kuprum klorida, phthalic anhydride dan pemangkin dengan pelarut bertakat dididih tinggi. Objektif kajian ini adalah untuk mengkaji samaada 10 peratus bahan terbuang daripada CuPC boleh digunakan semula. Terdapat 2 peringkat kerja iaitu proses menghasilkan bahan mentah CuPC manakala peringkat kedua adalah proses menganalisis 10 peratus bahan terbuang sama ada boleh digunakan semula atau tidak. Bahan mentah CuPC telah dihasilkan melalui proses tindak balas phthalic anhydride dengan baja dan ditambah dengan ion kuprum dan sedikit pemangkin yang dilarutkan dalam pelarut organic. Bahan terbuang daripada CuPC mentah boleh digunakan semula dengan mencampurkan urea, phthalic anhydrite dan pemangkin atau tanpa pemangkin. Penggunaan pemangkin menanmbahkan lagi peratus produk dan mempercepatkan tindak balas. Bahan terbuang telah diambil dari bahan mentah CuPC dan dianalisis. Analisis telah dilakukan untuk produk yang berjaya digunakan semula menggunakan FTIR. Dengan menggunakan kaedah ini, kos bahan boleh dikurangkan kerana menggunakan reagen yang tidak bertindak balas dan boleh dikitar semula dan juga dapat mengurangkan pencemaran alam sekitar.

TABLE OF CONTENTS

	Page
SUPERVISOR'S DECLARATION	ii
STUDENT'S DECLARATION	iii
DEDICATION	iv
ACKNOWLEDGEMENTS	v
ABSTRACT	vi
ABSTRAK	vii
TABLE OF CONTENTS	viii
LIST OF TABLES	xi
LIST OF FIGURES	xii
LIST OF SYMBOLS	xiii
LIST OF ABBREVIATIONS	xiv
 CHAPTER 1 INTRODUCTION	
1.1 Background of Study	1
1.2 Problem statement	2
1.3 Project Objective	2
1.4 Scope of Works	3
 CHAPTER 2 LITERATURE REVIEW	
2.1 Phthalocyanine	4
2.1.1 Copper Phthalocyanine	5
2.2 Starting Material	6
2.2.1 Phthalic Anhydride	6

2.2.2	Urea	9
2.2.3	Copper (I) Chloride	11
2.2.4	Ammonium Molybdate	11
2.2.5	Alkyl Benzene	12
2.3	Instruments	12
2.3.1	Centrifuge Machine	12
2.3.2	FTIR	13

CHAPTER 3 METHODOLOGY

3.1	Chemicals and Materials	14
3.2	Preparing crude CuPC	14
3.3	Collecting unreacted reagents (waste)	16
3.3.1	Adjust the pH	16
3.2.1	Collecting waste	16
3.4	Analyze of unreacted reagents	17
3.4.1	Adding some amount of urea and catalyst	17
3.4.2	Adding some amount of phthalic anhydrite and catalyst	17
3.4.3	Adding some amount of urea, phthalic anhydrite and catalyst	18
3.4.4	Adding some amount of urea	18
3.4.5	Adding some amount of phthalic anhydrite	18
3.4.6	Adding some amount of urea, phthalic anhydrite	19
3.5	Analysis using FTIR	20
3.6	Flow Chart of Study	21
3.6.1	Crude formation and Waste collecting	21
3.6.1.a	pH6 waste	21
3.6.1.b	pH8 waste	22

3.6.2	Analyze unreactive reactants (waste)	23
3.6.2.a	pH6 waste	23
3.6.2.b	pH8 waste	23
CHAPTER 4 RESULTS AND DISCUSSION		
4.1	Crude CuPC formation and collecting unreacted reagents	24
4.2	Collecting unreacted reagents	24
4.3	Analyze unreacted reactants	27
4.3.1	Analysis of pH 6 waste	27
4.3.2	Analysis of pH 8 waste	29
4.4	Yield calculation	30
4.4.1	pH6 product (with catalyst)	31
4.4.2	pH6 product (without catalyst)	31
4.4.3	pH8 product (with catalyst)	31
4.4.4	pH8 product (without catalyst)	32
4.5	FTIR analysis	32
CHAPTER 5 CONCLUSION AND RECOMMENDATIONS		
5.1	Conclusion	36
5.2	Recommendations	37
REFERENCES		38

LIST OF TABLES

Table no.		Page
3.1	Data for crude CuPC formation	15
3.2	Data for crude CuPC after dry	16
3.3	Data for wastes after dry	17
3.4	pH 6 wastes analyzed	19
3.5	pH 8 wastes analyzed	20
4.1	Data for crude CuPC	24
4.2	Waste collected	26
4.3	Summarize data for pH 6 analyzed	28
4.4	Summarize data for pH 8 analyzed	30
4.5	Summary of product from waste	30
4.6	Peaks wavenumber of pH6 crude and products	33
4.7	Peaks wavenumber of pH8 crude and products	33

LIST OF FIGURES

Figure No.		Page
1.1	Molecular structure of copper phthalocyanine	1
2.1	Molecular structure of phthalic anhydride	7
2.2	Preparation of phthalic anhydride	7
2.3	Molecular structure of urea	10
4.1	The crude CuPC for pH 6 and pH	25
4.2	The unreactive reagents (waste) for pH 6 and pH 8	26
4.3	The successful product for pH 6	28
4.4	The successful product for pH 8	29
4.5	FTIR spectrum comparison between crude and product (pH6)	34
4.6	FTIR spectrum comparison between crude and product (pH8)	35

LIST OF SYMBOLS

Λ_{\max} Wavelength

LIST OF ABBREVIATIONS

CuPC	Copper phthalocyanine
H ₂ SO ₄	Sulphuric acid
NaOH	Natrium hydroxide
C ₆ H ₄ (CO) ₂ O	Phthalic anhydride
CO(NH ₂) ₂	Urea
g	Grams
mL	Mililitre
cm ⁻¹	Reciprocal centimeter
rpm	Revolution per minute
CuCl	Copper chloride
FTIR	Fourier Transform Infrared Spectroscopy
XRD	X-Ray Diffraction
AAS	Atomic Absorption Spectroscopy
GC-MS	Gas Chromatography–Mass spectrometry
NMR	Nuclear Magnetic Resonance

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Copper phthalocyanine is a complex of copper with phthalocyanine. Molecular mass is 576.08 and melting point 600 °C [1]. The color is due to a $\pi\text{-}\pi^*$ electronic transition, with $\lambda_{\text{max}} \sim 610$ nm [2]. It was first developed as a pigment in the mid-1930s. Copper phthalocyanine is a cool blue with a bias towards green. It has intense tinting strength and easily overpowers the mix when combined with other colors. Due to its stability, copper phthalocyanine is also used in inks, coatings, and many plastics. The pigment is insoluble and has no tendency to migrate in the material. The compound is non-biodegradable, but not toxic to fish or plants. There is some evidence that exposure to phthalocyanines can cause serious birth defects in developing embryos [3].

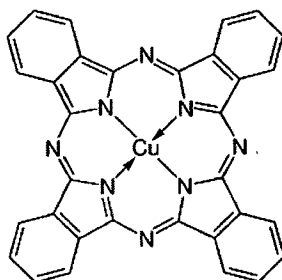


Figure 1.1: Molecular Structure of Copper Phthalocyanine

Two processes are commonly used for production of copper phthalocyanine. First is phthalic anhydrite – urea process patented by ICI [4] and second one is the I.G Farben dinitrile process. Both can be carried out continuously or batchwise in a solvent or by melting the starting material together. The type and amount of catalyst used are crucial for the yield. Especially effective as catalysts are molybdenum (IV) oxide and ammonium molybdate. Copper salt or copper powder is used as the copper source [5]. Use of copper (I) chloride result in a very smooth synthesis. Use of copper (I) chloride as starting material leads to the formation of small amount of chloro CuPC.

In this study, focuses was given in production of crude copper pthalocyanine, collecting waste from crude copper pthalocyanine and recycle of waste using minimum chemicals used in crude copper phthalocyanine production. Production of crude CuPC used the phthalic anhydrite – urea process patented by ICI and ammonium molybdate as a catalyst.

1.2 Problem Statement

The yield of CuPC and other metal PC is about 80-90% and the 10-20% or so is the unreacted reagents produce in industry. This unreacted reagent should be incinerated but many manufactures dispose this unreacted reagent to the environment. To reduce the damage for the environment, the unreacted reagents should be reused to produce pigment.

1.3 Project Objectives

To determine whether the 10% of the unreactive reactants can be recycled and used to make the same crude.

1.4 Scopes of Works

Based on the objectives, the work carried out in this project should be:

1. To prepare copper phthalocyanine.
2. To analyze 10% of unreactive reactants if can be recycled and used to make the same crude (Prepare new crude CuPC using the solids obtained from the washing of crude CuPC).

CHAPTER 2

LITERATURE REVIEW

2.1 Phthalocyanine

Phthalocyanine is an intensely blue-green coloured macrocyclic compound that is widely used in dyeing. Phthalocyanines form coordination complexes with most elements of the periodic table. These complexes are also intensely colored and also are used as dyes or pigments. An unidentified blue compound, which is now known to be metal-free phthalocyanine, was first reported in 1907. In 1927, Swiss researchers accidentally synthesized copper phthalocyanine, copper naphthalocyanine, and copper octamethylphthalocyanine in an attempted conversion of *o*-dibromobenzene into phthalonitrile. They remarked on the enormous stability of these complexes but did not further characterize these blue complexes. In the same year, copper phthalocyanine was also discovered at Scottish Dyes, Ltd., Grangemouth, Scotland (later ICI). ColorantHistory.org hosts an old documentary about the discovery of the pigment.

Unsubstituted phthalocyanine, abbreviated H₂Pc, and many of its complexes have very low solubility in organic solvents. Benzene at 40 °C dissolves less than a milligram of H₂Pc or CuPc per litre. H₂Pc or CuPc dissolve easily in sulfuric acid due to the protonation of the nitrogen atoms bridging the pyrrole rings. Many phthalocyanine compounds are thermally very stable, do not melt but can be sublimed, CuPc sublimes at >500 °C under inert gases (nitrogen, CO₂). Substituted phthalocyanine complexes often have much higher solubility. They are less thermally stable and often cannot be

sublimed. Unsubstituted phthalocyanines strongly absorb light between 600 to 700 nm, thus these materials are blue or green. Substitution can shift the absorption towards longer wavelengths, changing the color from pure blue to green to colorless (when the absorption is in the near infrared). Many derivatives of the parent phthalocyanine are known, where either carbon atoms of the macrocycle are exchanged for nitrogen atoms or where the hydrogen atoms of the ring are substituted by functional groups like halogens, hydroxy, amino, alkyl, aryl, thiol, alkoxy and nitro groups.

2.1.1 Copper Phthalocyanine

Two processes commonly used for the production of copper phthalocyanine are the phthalic anhydride-urea process patented by ICI and the I.G Farben dinitrile process. Both can be carried out continuously or batchwise in a solvent or by melting the starting materials together. The type and amount of catalyst used are crucial for the yield. Especially effective as catalysts are molybdenum (iv) oxide and ammonium molybdate. Copper salts or copper powder is used as the copper source. Used of copper (i) chloride results in a very smooth synthesis. Used of Copper (i) chloride as starting material leads to the formation of small amounts of chloro CuPC.

Phthalocyanine was the first new chromogenic type to be introduced into the field of the color chemistry of organic compounds concerned with pigments. Before this discovery, all the known organic pigments had been developed either by making dyes insoluble or by synthesising new insoluble azo compounds. This development is therefore interesting both technically and scientifically.

It began in 1928 when a blue impurity was found in phthalimide during its manufacture at Scottish Dyes (later part of ICI). The method of preparation being used was to pass ammonia through molten phthalic anhydride in an iron vessel. It is now known that the blue impurity was iron phthalocyanine. This was found to be exceptionally stable and steps were taken to determine its structure. It is of interest that another important blue pigment was also first synthesised as the result of investigating a

blue impurity. This was in an industry not directly related to colour making, namely, sodium carbonate manufacture at St Gobain in France.

With hindsight it can be guessed that other ways of solving the impurity problem in making phthalimide, such as the use of a ceramic vessel, might well have solved the initial difficulty but would have resulted in missing the discovery of what has turned out to be almost important chromogen. Two other preparations of phthalocyanine had already been reported in the chemical literature but did not lead to the recognition of its usefulness, the first in 1907 and the second in 1927. Linstead and his co-workers investigated the chemical structure of the blue impurity and its novel structure was finally established by Robertson using X-ray diffraction. Copper phthalocyanine first appeared commercially in 1935 under the ICI trade name of Monastral Blue B.

Copper phthalocyanine is a pigment that offers brightness, cleanliness, strength and economy with excellent fastness properties. The only drawback to this pigment is its tendency to change to a coarse, crystalline non pigmentary form when used in strong solvent systems if the crystal has not been adequately stabilized coupled with the pigment's tendency to flocculate. Another negative is the fact that copper phthalocyanine blues exhibit the phenomenon of surface bronzing when applied at mass tone levels and deep tints.

2.2 Starting Material

2.2.1 Phthalic Anhydride

Phthalic anhydride is the organic compound with the formula $C_6H_4(CO)_2O$. It is the anhydride of phthalic acid. This colourless solid is an important industrial chemical, especially for the large-scale production of plasticizers for plastics. In 2002, approximately 4.6 billion kilograms were produced [6].

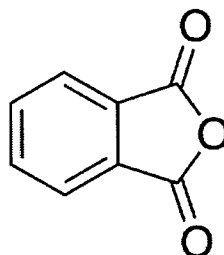
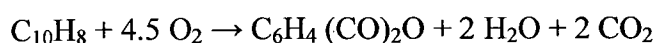
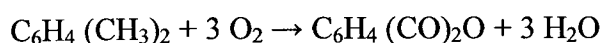


Figure 2.1: Molecular structure of Phthalic anhydride

Phthalic anhydride was first reported in 1836 by Laurent. It is presently obtained by catalytic oxidation of *ortho*-xylene and naphthalene (Gibbs phthalic anhydride process):



When separating the phthalic anhydride from byproducts such as *o*-xylene in water, or maleic anhydride, a series of “switch condensers” is required. Phthalic anhydride can also prepare from phthalic acid [6]:

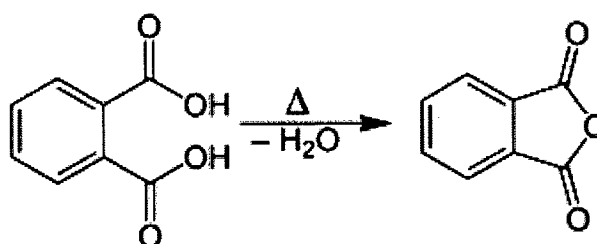


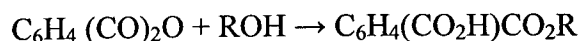
Figure 2.2: Preparation of phthalic anhydride

Phthalic anhydride is a versatile intermediate in organic chemistry, in part because it is bifunctional and cheaply available. It undergoes hydrolysis and alcoholysis. Hydrolysis by hot water forms *ortho*-phthalic acid. This process is reversible: Phthalic anhydride re-forms upon heating the acid above 180 °C [7]. Hydrolysis of anhydrides is not typically a reversible process. However, phthalic

acid is easily dehydrated to form phthalic anhydride due to the creation of a thermodynamically favorable 5-membered ring.

As with other anhydrides, the alcoholysis reaction is the basis of the manufacture of phthalate esters, which are widely used (and controversial - see endocrine disruptor) plasticizers [6].

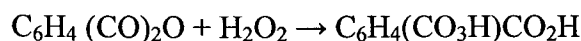
In the 1980s, approximately 6.5×10^9 kg of these esters was produced annually, and the scale of production was increasing each year, all from phthalic anhydride. The process begins with the reaction of phthalic anhydride with alcohols, giving the monoesters:



The second esterification is more difficult and requires removal of water:



The most important diester is bis(2-ethylhexyl) phthalate ("DEHP"), used in the manufacture of polyvinyl chloride. Phthalic anhydride is a precursor to a variety of reagents useful in organic synthesis. Important derivatives include phthalimide and its many derivatives. Chiral alcohols form half-esters (see above), and these derivatives are often resolvable because they form diastereomeric salts with chiral amines such as brucine [8]. A related ring-opening reaction involves peroxides to give the useful peroxy acid [9]:

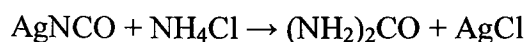


Phthalic anhydride is widely used in industry for the production of certain dyes. A well-known application of this reactivity is the anthraquinone dye quinizarin by reaction with para-chlorophenol followed by hydrolysis of the chloride [10].

2.2.2 Urea

Urea or carbamide is an organic compound with the chemical formula $\text{CO}(\text{NH}_2)_2$. The molecule has two —NH_2 groups joined by a carbonyl(C=O) functional group. Urea serves an important role in the metabolism of nitrogen-containing compounds by animals and is the main nitrogen-containing substance in the urine of mammals. It is solid, colourless, and odorless (although the ammonia that it gives off in the presence of water, including water vapor in the air, has a strong odor). It is highly soluble in water and non-toxic. Dissolved in water, it is neither acidic nor alkaline. The body uses it in many processes, the most notable one being nitrogen excretion. Urea is widely used in fertilizers as a convenient source of nitrogen. Urea is also an important raw material for the chemical industry. The synthesis of this organic compound by Friedrich Wöhler in 1828 from an inorganic precursor was an important milestone in the development of organic chemistry, as it showed for the first time that a molecule found in living organisms could be synthesized in the lab without biological starting materials.

Urea was first discovered in urine in 1727 by the Dutch scientist Herman Boerhaave, though this discovery is often attributed to the French chemist Hilaire Rouelle [11]. In 1828, the German chemist Friedrich Wöhler obtained urea by treating silver isocyanate with ammonium chloride [12].



Urea is produced on a scale of some 100,000,000 tons per year worldwide [13]. For use in industry, urea is produced from synthetic ammonia and carbon dioxide. Large quantities of carbon dioxide are produced during the manufacture of ammonia from coal or from hydrocarbons such as natural gas and petroleum-derived raw materials. Such point sources of CO_2 facilitate direct synthesis of urea.

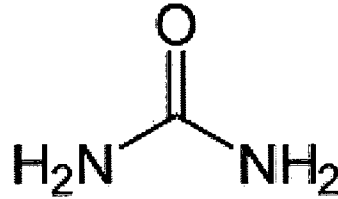
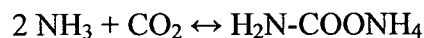
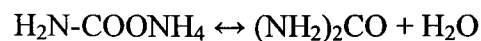


Figure 2.3: Molecular structure of urea

The basic process, developed in 1922, is also called the Bosch-Meiser urea process after its discoverers. The various urea processes are characterized by the conditions under which urea formation takes place and the way in which unconverted reactants are further processed. The process consists of two main equilibrium reactions, with incomplete conversion of the reactants. The first is an exothermic reaction of liquid ammonia with dry ice to form ammonium carbamate ($\text{H}_2\text{N-COONH}_4$) [14]:



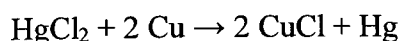
The second is an endothermic decomposition of ammonium carbamate into urea and water:



Unconverted reactants can be used for the manufacture of other products, for example ammonium nitrate or sulfate, or they can be recycled for complete conversion to urea in a total-recycle process. Urea can be produced as prills, granules, pellets, crystals, and solutions. Solid urea is marketed as prills or granules. The advantage of prills is that, in general, they can be produced more cheaply than granules. Properties such as impact strength, crushing strength, and free-flowing behaviour are, in particular, important in product handling, storage, and bulk transportation.

2.2.3 Copper (I) Chloride

Copper (I) chloride, commonly called cuprous chloride, is the lower chloride of copper, with the formula CuCl. The substance is a white solid sparingly soluble in water, but very soluble in concentrated hydrochloric acid. Impure samples appear green due to the presence of copper (II) chloride. Copper (I) chloride was first prepared by Robert Boyle in the mid-seventeenth century [15] from mercury (II) chloride ("Venetian sublimate") and copper metal:



In 1799, J.L. Proust characterized the two different chlorides of copper. He prepared CuCl by heating CuCl₂ at red heat in the absence of air, causing it to lose half of its combined chlorine followed by removing residual CuCl₂ by washing with water [16]. An acidic solution of CuCl was formerly used for analysis of carbon monoxide content in gases, for example in Hempel's gas apparatus [17]. This application was significant [18] during the time that coal gas was widely used for heating and lighting, during the nineteenth and early twentieth century's.

Ammoniacal solutions of CuCl react with acetylenes to form the explosive copper (I) acetylide. Complexes of CuCl with alkenes can be prepared by reduction of CuCl₂ by sulfur dioxide in the presence of the alkene in alcohol solution. In absence of other ligands, its aqueous solutions are unstable with respect to disproportionate into Cu and CuCl₂ [19]. In part for this reason samples in air assume a green coloration.

2.2.4 Ammonium molybdate

Ammonium heptamolybdate is an odourless crystalline compound ranging in colour from white to yellow-green. It is usually encountered as the tetrahydrate,

whose chemical formula is $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$. It is often referred to as ammonium paramolybdate or simply as ammonium molybdate, although ammonium molybdate can also refer to ammonium orthomolybdate, $(\text{NH}_4)_2\text{MoO}_4$, and several other species.

Ammonium heptamolybdate is easily prepared by dissolving molybdenum trioxide in an excess of aqueous ammonia and evaporating the solution at room temperature. While the solution evaporates, the excess of ammonia escapes. This method results in the formation of six-sided transparent prisms of the tetrahydrate of ammonium heptamolybdate [20]. Solutions of ammonium paramolybdate react with acids to form molybdic acid and an ammonium salt. The pH value of a concentrated solution will lie between 5 and 6.

2.2.5 Alkyl Benzene

Alkyl benzene solvent is an attractive solvent for large scale liquid scintillator detectors due to its low toxicity, high flash point, and good compatibility with acrylic. Very good optical purity is required to be used in large scale detectors [21]. Alkyl benzene solvent have higher boiling point so thus it will be heated up until temperature 200°C . Alkyl benzene also will make the reaction occur smoothly without give the dirty result.

2.3 Instruments

2.3.1 Centrifuge Machine

A centrifuge is a piece of equipment, generally driven by an electric motor (some older models were spun by hand), that puts an object in rotation around a fixed axis, applying a force perpendicular to the axis. The centrifuge works using the sedimentation principle, where the centripetal acceleration causes more dense substances to separate out along the radial direction (the bottom of the tube). By the same token, lighter objects

will tend to move to the top (of the tube; in the rotating picture, move to the centre). Simple centrifuges are used in chemistry, biology, and biochemistry for isolating and separating suspensions. They vary widely in speed and capacity. They usually comprise a rotor containing two, four, six, or many more numbered wells within which the samples, contained in centrifuge tubes, may be placed.

2.3.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectroscopy (FTIR) [22] is a technique which is used to obtain an infrared spectrum of absorption, emission, photoconductivity or Raman scattering of a solid, liquid or gas. An FTIR spectrometer simultaneously collects spectral data in a wide spectral range. This confers a significant advantage over a dispersive spectrometer which measures intensity over a narrow range of wavelengths at a time. FTIR has made dispersive infrared spectrometers all but obsolete (except sometimes in the near infrared), opening up new applications of infrared spectroscopy. Fourier transform spectroscopy is a measurement technique whereby spectra are collected based on measurements of the coherence of a radiative source, using time-domain or space-domain measurements of the electromagnetic radiation or other type of radiation.

The goal of any absorption spectroscopy is to measure how well a sample absorbs light at each wavelength. In infrared spectroscopy, IR radiation is passed through a sample. Some of the infrared radiation is absorbed by the sample and some of it is passed through (transmitted). The resulting spectrum represents the molecular absorption and transmission, creating a molecular fingerprint of the sample. Like a fingerprint no two unique molecular structures produce the same infrared spectrum. This makes infrared spectroscopy useful for several types of analysis. The information that FTIR can provide are can identify unknown materials, determine the quality or consistency of a sample and determine the amount of components in a mixture.

CHAPTER 3

METHODOLOGY

3.1 Chemicals and Materials

Production crude CuPC is involve urea ($\text{CH}_4\text{N}_2\text{O}$), phthalic anhydride ($\text{C}_8\text{H}_4\text{O}_3$) and copper (I) chloride (CuCl) as raw material, ammonium molybdate as a catalyst and alkyl benzene as a solvent.

3.2 Preparing crude CuPC

There are two standard crudes CuPC were prepared, one for pH 6 wastes and one for pH 8 wastes. Crude CuPC was prepared by mixing phthalic anhydrate, urea, copper (I) chloride, ammonium molybdate as a catalyst and alkyl benzene as a solvent. This entire chemical was added in round bottom flask with three necks using filter funnel. The experiment was running with speed of 80 rpm of homomixer and heating mental was heat up slowly until 200°C for one hour. Table 3.1 showed the data for each chemical used for preparing crude CuPC. All of this crude takes same time to stable during synthesis process. The crude will stable when the colour of mixture turn to blue colour. The reaction time for first crude (for pH6 crude) starts from 10.12 a.m and turn blue at 11.10 a.m. For the second crude (for pH8 crude), the reaction starts from 10.30 a.m and turn blue at 11.25 a.m. All of these two crudes take about one hour to stable. The reaction complete in 5 hours.