

PERPUSTAKAAN UMP



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NOVEL METHOD OF PREPARATION OF BLUE CRUDE COPPER
PHTHALOCYANINE HAVING THE REDDISH SHADE

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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 novel method is to obtain reddish blue shade on blue crude CuPc. For the preparation of α -pigment CuPc, there are two stages which the first stage is crude CuPc synthesis while second stage is the α -pigment synthesis. Blue crude CuPc has been prepared by condensation of phthalic anhydride with urea in presence of copper ions with a small amount of catalyst in inert organic solvent. Concentrated sulphuric acid has been added to the blue crude CuPc and produce α -pigment. The presence of sulphuric acid make crude and α -pigment more reddish blue shade while the presence of hydrochloric acid make crude and α -pigment more greenish blue shade. FTIR and chromometer had been used to analyze blue crude and α -pigment obtained and be compared to the standard preparation without the addition of acids. By using this novel method, the cost of material has been reduced.

ABSTRAK

Kuprum phthalocyanine (CuPc) adalah salah satu pigmen yang stabil terhadap cahaya, suhu dan bahan kimia. CuPc dihasilkan melalui proses di antara baja kuprum klorida, phthalic anhydride dan pemangkin dengan suhu pelarut yang tinggi. Objektif kajian ini adalah untuk mendapatkan bahan mentah CuPc dalam menghasilkan warna biru kemerahan. Terdapat dua peringkat dalam menghasilkan pigmen CuPc iaitu proses menghasilkan bahan mentah CuPc manakala peringkat kedua adalah proses menghasilkan α -pigmen CuPc. Bahan mentah CuPc telah dihasilkan melalui proses kondensasi phthalic anhydride dengan baja dan ditambah dengan kuprum ion dan sedikit pemangkin yang dilarutkan dalam pelarut organik. Asid sulfurik pekat telah ditambah dalam bahan mentah CuPc dan menghasilkan α -pigmen. Bahan mentah dan α -pigmen menjadi warna biru kemerahan hasil kehadiran asid sulfurik manakala dengan kehadiran asid hidroklorik menjadi warna biru kehijauan. Kromometer dan FTIR telah digunakan untuk menganalisa bahan mentah biru dan α -pigmen dan dibandingkan dengan standard. Dengan menggunakan kaedah baru ini, kos bahan telah dikurangkan.

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LIST OF ABBREVIATIONS

$C_{12}H_{24}O_2$	Lauric acid
$C_6H_4(CO)_2O$	Phthalic anhydride
cm^{-1}	Reciprocal centimeter
$CO(NH_2)_2$	Urea
CuCl	Copper chloride
CuPC	Copper phthalocyanine
ECD	Electron Capture Detector
FTIR	Fourier Transform Infrared Spectroscopy
g	Gram
H_2SO_4	Sulphuric acid
HCl	Hydrochloric acid
mL	Mililitre
rpm	Revolution per minute
XRD	X-Ray Diffraction
ΔA	Green-on-Red value
ΔL	Colour brightness

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND STUDY

Blue pigment copper phthalocyanine, CuPc is one of the most stable pigment towards light, temperature and chemicals and hence, combined with its attractive colors, this pigment is widely used in many applications such as in paints water & solvent based paints including automotive paints, in water & solvent based inks, in textiles and in plastics with temperature of operations up to 350 °C. CuPc have two types of pigment where blue and green. The blue copper phthalocyanine have 3 types which are α -CuPc, β -CuPc, and ϵ -CuPc. In industry, this pigment is produced in two stages. The first stage is the preparation of the blue crude copper phthalocyanine while the second is the pigmentation of crude copper phthalocyanine. Copper phthalocyanine also known as phthalocyanine blue ($C_{32}H_{16}N_8Cu$) a complex of copper with phthalocyanine. Molecular mass is 576.08 g/mol, melting point 600 °C (with decomposition), the substance is practically insoluble in water (<0.1 g / 100 ml @ 20 °C), but soluble in concentrated sulphuric acid. Density of the solid is ~1.6 g/cc. The color is due to a π - π^* electronic transition, with λ_{max} ~ 610 nm.

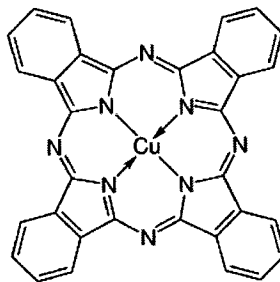


Figure 1.1 : Molecular structure of copper phthalocyanine

Copper phthalocyanine pigments, by virtue of their exceptionally brilliant shade, high tinctorial strength, low cost and outstanding fastness properties, have been ruling the colorant industry ever since their commercial introduction in 1935 [1]. They are about to complete 70 years of meritorious service as an unchallenged leader of various types of blue and green pigments. In this context it is appropriate to present a brief review on some aspects related to their origin, development, mechanisms of formation and other finer details pertaining to their various properties. Phthalocyanine pigments constitute a broad class of pigments. Phthalocyanine blue and phthalocyanine green are among the most important organic pigments.

In the beginning of the 20th century, commercially available blue pigments consisted mainly of Ultramarine blue, Prussian blue, Indigo derivatives and some azo derivatives. Unfortunately none of these had the desired qualities demanded by the colour industry. Most of these pigments had poor chemical resistance, unsatisfactory acid and/or alkali resistance, insufficient light fastness and poor sublimation fastness. [2]. The colour chemists in those days were in need of a cheap and durable pigment that could fill the bluish region of the colour space. The discovery of copper phthalocyanine blue provided them with a satisfactory solution to their problems.

Copper phthalocyanine crude have high market demand over the world. World production of this product is over 110,000 tons/year while the gap between the world demand and the supply according to Business Analyst for Asahi Songwon Colors Ltd for 2011 is 19,000 tons /year [3]. Copper phthalocyanine crude was used

to be produced in Japan, Europe and America but now it has been shifted to mainly China and India due to lower labor cost and also mainly as the waste regulations of these countries are not strictly followed.

Copper phthalocyanine pigments proved to be outstanding organic pigments because of their incredible resistance to chemical attack, good fastness to heat and light, combined with brightness and cleanliness of shade all the qualities sought by the colour industry. The pigment is insoluble and has no tendency to migrate in the material. It is a standard pigment used in printing ink and the packaging industry [4].

1.2 PROBLEM STATEMENT

In this experiment, we have to face many problems which how to conduct the synthesis of blue crude copper phthalocyanine without need higher cost and lower environmental effect.

1.3 OBJECTIVES

The objective of this research is to obtain the reddish shade of blue crude copper phthalocyanine which is required in the industries.

1.4 PROJECT SCOPES

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

1. To conduct the synthesis of blue crude copper phthalocyanine in the presence of different acid at different concentration.
2. To conduct the synthesis of α -copper phthalocyanine pigment from blue crude copper phthalocyanine.
3. To analyse the shade colour of blue crude copper phthalocyanine and α -copper phthalocyanine pigment.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

This chapter is discussed about the type of pigment, starting materials to produce blue crude bopper phthalocyanine and the instruments used to analyze the crude.

2.2 PIGMENT

A pigment is a material that changes the color of reflected or transmitted light as the result of wavelength-selective absorption. This physical process differs from fluorescence, phosphorescence, and other forms of luminescence, in which a material emits light. Pigments are coloured, black, white or fluorescent particulate organic and inorganic solids which are insoluble in, and essentially physically and chemically unaffected by the vehicle or substrate in which they are incorporated. They alter appearance by selectivity absorption and/or by scattering of light [5].

Many materials selectively absorb certain wavelengths of light. Materials that humans have chosen and developed for use as pigments usually have special properties that make them ideal for coloring other materials. A pigment must have a high tinting strength relative to the materials it colors. It must be stable in solid form at ambient temperatures.

Entire technologies are involved in each of the three largest applications of pigments, which are as colorants in paints, plastics and printing inks [6]. Each of

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 [8]. 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 a most 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 [9] and the second in 1927 [10]. Instead and his co-workers investigated the chemical structure of the blue impurity and its novel structure was finally established by Robertson [11] 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 nonpigmentary 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 levels and deep tints.

Copper phthalocyanine is available as two major commercial forms, the alpha and the beta crystal. The alpha crystal is described as CI pigment blue 15:1 and 15:2 and is a clean, bright-red-shade. The beta crystal is described as CI pigment blue 15:3 and 15:4 and is a clean green-shade-blue. The beta form is the most stable crystal form and readily resists crystallization. The alpha form, conversely, is the

least stable, or meta, form and readily converts to the more stable, green-shade beta crystal. As such, the alpha crystal requires special, proprietary treatments to produce a red-shade product offering stability to both crystallization treatment to produce a red-shade product offering stability to both crystallization and flocculation.

The manufacture of copper phthalocyanine is a two stage process where a crude, nonpigmentary product is first isolated and then the product is conditioned using a variety techniques to render the crystal pigmentary. Production of CuPC patented by ICI [12]. Manufacture of the crude proceeds at high temperature, sometimes under increased pressure and typically using organic solvents as the medium in which the condensation takes place. A phthalic anhydride acid derivative is condensed with a source of nitrogen such as urea and a copper salt such as copper chloride in the presence of a metal catalyst such as molybdenum or vanadium.

2.3 STARTING MATERIALS

2.3.1 Phthalic Anhydride

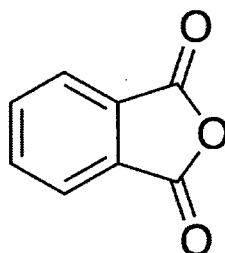


Figure 2.1 : Molecular structure of Phthalic anhydride

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 [13]. 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). Phthalic anhydride can also prepared from phthalic acid:

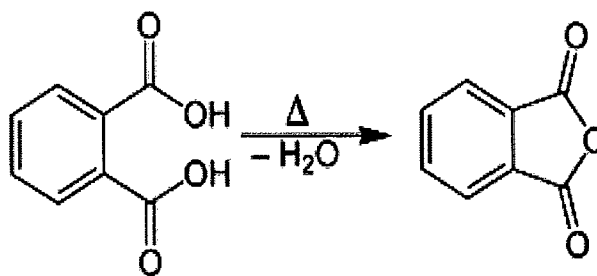


Figure 2.2 : Preparation of phthalic anhydride

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 [14]. 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. Phthalic anhydride also widely used in industry for the production of certain dyes.

2.3.2 Urea

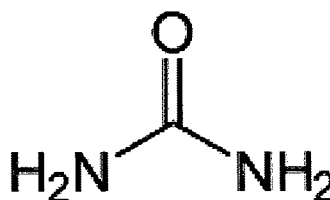


Figure 2.3 : Molecular structure of urea

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 ($\text{C}=\text{O}$) functional group. 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. 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 [15].

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 CO₂ facilitate direct synthesis of urea.

The urea molecule is planar in the crystal structure, but the geometry around the nitrogens is pyramidal in the gas-phase minimum-energy structure [16]. In solid urea, the oxygen center is engaged in two N-H-O hydrogen bonds. The resulting dense and energetically favourable hydrogen-bond network is probably established at the cost of efficient molecular packing: The structure is quite open, the ribbons forming tunnels with square cross-section. The carbon in urea is described as sp² hybridized, the C-N bonds have significant double bond character, and the carbonyl oxygen is basic compared to, say, formaldehyde. Urea's high aqueous solubility reflects its ability to engage in extensive hydrogen bonding with water. Urea is produced on a scale of some 100,000,000 tons per year worldwide [17].

2.3.3 Ammonium Molybdate

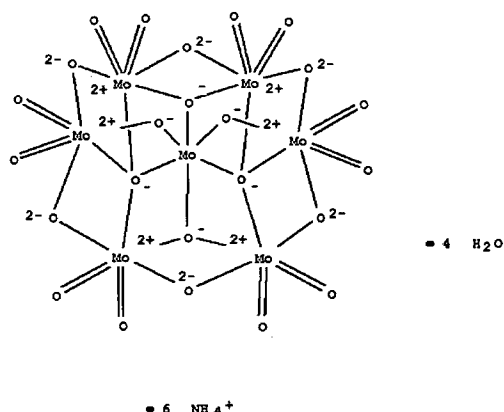


Figure 2.4 : Molecular structure of ammonium molybdate

The chemical formula for ammonium molybdate is (NH₄)₆Mo₇O₂₄·4H₂O. Ammonium molybdate is an odorless crystalline compound ranging in color from white to yellow-green. Also called molybdic acid hexammonium salt tetrahydrate, ammonium molybdate tetrahydrate and ammonium heptamolybdate tetrahydrate. In

chemistry a ammonium molybdate is a compound containing an oxoanion with molybdenum in its highest oxidation state of 6.

Ammonium molybdate can form a very large range of such oxoanions which can be discrete structures or polymeric extended structures, although the latter are only found in the solid state. The discrete ammonium molybdate oxoanions range in size from the simplest MoO_4^{2-} , found in potassium molybdate up to extremely large structures found in isopoly-molybdenum blues that contain for example 154 Mo atoms.

2.3.4 Copper Chloride

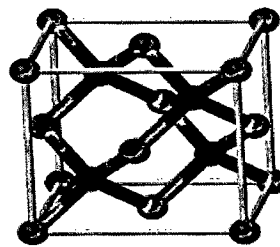
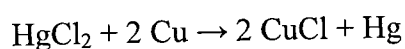


Figure 2.5 : Molecular structure of copper chloride

Copper 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 [18].

Copper chloride was first prepared by Robert Boyle in the mid-seventeenth century 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 CuCl_2 at red heat in the absence of air, causing it to lose

half of its combined chlorine followed by removing residual CuCl_2 by washing with water [19].

An acidic solution of copper chloride was formerly used for analysis of carbon monoxide content in gases, for example in Hempel's gas apparatus [20]. This application was significant during the time that coal gas was widely used for heating and lighting, during the nineteenth and early twentieth centuries.

Copper chloride is a Lewis acid, which is classified as soft according to the Hard-Soft and Acid-Base concept. Thus, it tends to form stable complexes with soft Lewis bases such as triphenylphosphine. Although copper chloride is insoluble in water, it dissolves in aqueous solutions containing suitable donor molecules.

2.3.5 Sulphuric Acid

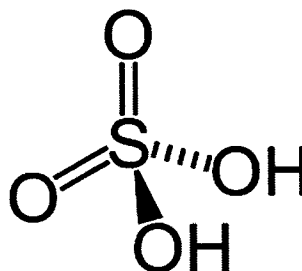
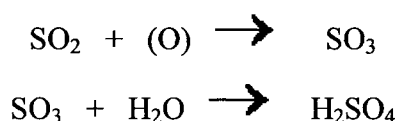


Figure 2.6 : Molecular structure of sulphuric acid

Sulfuric acid is a clear, colorless, odorless, viscous liquid that is very corrosive. Sulfuric acid is a strong mineral acid with the molecular formula H_2SO_4 . Its historical name is oil of vitriol. Pure sulfuric acid is a highly corrosive, colorless, viscous liquid. The salts of sulfuric acid are called sulfates. Sulfuric acid is soluble in water at all concentrations. Sulfuric acid is a very important commodity chemical, and indeed, a nation's sulfuric acid production is a good indicator of its industrial strength [21].

Sulfuric acid is often prepared as a byproduct from mining operations. Many metal ores are sulfides and smelting operations eliminate sulfur in the form of sulfur dioxide gas. Early in the 20th century, sulfur dioxide just went up the smokestack but today smelters must recover the gas. The sulfur dioxide gas is cleaned and converted into sulfuric acid.

The extremely hot sulfur dioxide gas from the furnace gets sprayed with water, cooled, cleaned, dried, run through a catalytic converter, converted to sulfur trioxide (SO₃), and combined with water to make sulfuric acid:



Sulfuric acid is a very important commodity chemical, and indeed, a nation's sulfuric acid production is a good indicator of its industrial strength [22]. As the largest-volume industrial chemical produced in the world, consumption of sulfuric acid is often used to monitor a country's degree of industrialization. Agricultural fertilizers represent the largest single application for sulfuric acid (65 %). Other uses include production of dyes, alcohols, plastics, rubber, ether, glue, film, explosives, drugs, paints, food containers, wood preservatives, soaps and detergents, pharmaceutical products, petroleum products, pulp and paper. The common lead-acid storage battery is one of the few consumer products that actually contains sulfuric acid is the common lead-acid storage battery.

2.3.6 Hydrochloric Acid



Figure 2.7 : Molecular structure of hydrochloric acid

Hydrochloric acid is a solution of hydrogen chloride (HCl) in water, that is a highly corrosive, strong mineral acid with many industrial uses. It is found naturally in gastric acid. The physical properties of hydrochloric acid such as boiling and melting points, density, and pH - depend on the concentration or molarity of HCl in the acid solution. They range from those of water at very low concentrations approaching 0 % HCl to values for fuming hydrochloric acid at over 40 % HCl [23].

Hydrochloric acid is prepared by dissolving hydrogen chloride in water. Hydrogen chloride can be generated in many ways, and thus several precursors to hydrochloric acid exist. The large-scale production of hydrochloric acid is almost always integrated with the industrial scale production of other chemicals.

Hydrochloric acid is a strong inorganic acid that is used in many industrial processes. The application often determines the required product quality [24]. Concentrated hydrochloric acid (fuming hydrochloric acid) forms acidic mists. Both the mist and the solution have a corrosive effect on human tissue, with the potential to damage respiratory organs, eyes, skin, and intestines. Personal protective equipment such as rubber or PVC gloves, protective eye goggles, and chemical-resistant clothing and shoes are used to minimize risks when handling hydrochloric acid.

2.3.7 Lauric Acid

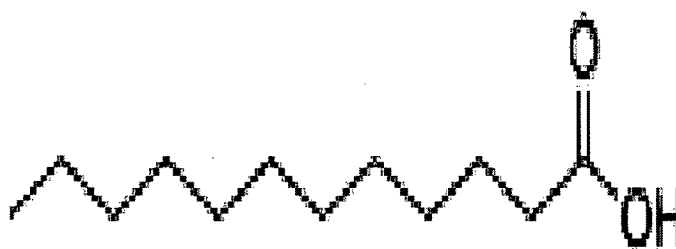


Figure 2.8 : Molecular structure of lauric acid

Lauric acid ($C_{12}H_{24}O_2$), the saturated fatty acid with a 12-carbon atom chain, is a white, powdery solid with a faint odor of bay oil or soap. Like many other fatty

acids, lauric acid is inexpensive, has a long shelf-life, and is non-toxic and safe to handle. It is mainly used for the production of soaps and cosmetics. For these purposes, lauric acid is neutralized with sodium hydroxide to give sodium laurate, which is a soap. Most commonly, sodium laurate is obtained by saponification of various oils, such as coconut oil. These precursors give mixtures of sodium laurate and other soaps [25].

In the laboratory, lauric acid is often used to investigate the molar mass of an unknown substance via the freezing-point depression. Lauric acid is convenient because its melting point when pure is relatively high (43.9 °C). Its cryoscopic constant is 3.9 K/kg/mol. By melting lauric acid with the unknown substance, allowing it to cool, and recording the temperature at which the mixture freezes, the molar mass of the unknown compound may be determined [26]. Other uses for lauric acid include treatment of bronchitis, gonorrhoea, yeast infections, chlamydia, intestinal infections caused by a parasite called *Giardia lamblia*, and ringworm. In foods, lauric acid is used as a vegetable shortening. In manufacturing, lauric acid is used to make soap and shampoo.

2.3.8 Alkyl Benzene Solvent

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 [27]. 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.

2.3.9 NP-10



Figure 2.9 : NP-10

NP-10 is the type of detergent and surfactant. The copper phthalocyanine not soluble easily in the solvent, thus NP-10 added to the reaction to disperse the copper phthalocyanine. When the copper phthalocyanine disperse easily, impurities also can wash easily.

2.4 INSTRUMENTS

2.4.1 Homomixer

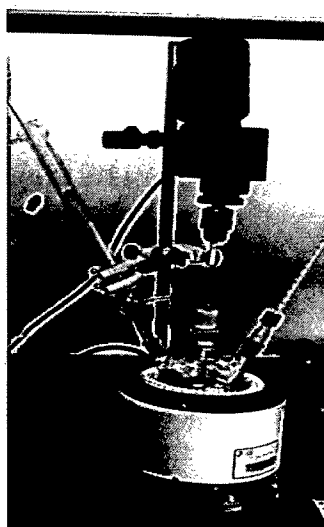


Figure 2.10 : Homomixer