OPTIMIZATIONS AND RECYCLING INDUSTRIAL WASTE (PALM OIL FLY ASH) AS A PIGMENT IN COATING TECHNOLOGY

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OPTIMIZATIONS AND RECYCLING INDUSTRIAL WASTE (PALM OIL FLY ASH) AS A PIGMENT IN COATING TECHNOLOGY

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Especially dedicated to my beloved parent and family members;
Mohd @ Mohd Nor Bin Jusoh and Hamsiah Binti Jaafar;
Syahian & Nurul Haslinda, Mohd Najib & Huahida, Mohd Shahril & Norfatiha,
Ahmad Kamilrul;
Muhammad Ilham Farhan, Irfan Mifzal, Muhammad Aiman Faris, Umar, Ainul
Mardhiah, Hannah Husna, Kauthar, Humaira;
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Co supervisor, Dr. Ong Pauline;
My love, Fatin Syahirah;
Dear lecturers, technicians, rowing coaches, UTHM Barracuda,
PSM students, SDPS team and friends;
May Allah S.W.T always bless us all.
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Last but not least, million thanks to those who had giving a hand, directly or indirectly in helping me to finish my project and thesis with successfully.
In this study, the feasibility of palm oil fly ash (POFA) as a pigment in paint technology is investigated. The utilization of POFA as paint pigment is favorable due to its stability and non-toxicity. On the other hand, producing colour pigment by using POFA as pigment element reduces the solid waste in palm oil industry, in addition to the advantages of inexpensiveness and ready access to POFA feedstock. The objective of this research are to identify the optimum composition of POFA in combination of binder, solvent and additives as pigment in paint, to examine the heat resistant behavior of POFA paint and investigating the POFA paint coating ability to withstand from the paint testing. In general, this research hopes to improve the technology of coating and also can be a significant contribution in the academic, research, development and field studies related to the paint technology. The optimum preparation and thermal conductivity of paint that prepared from the waste of palm oil fly ash (POFA) were investigated. For optimum pigment preparations, the aqueous precursor of 45 % wt POFA mixed with 25 % wt sulphur, 10 % wt graphite fine powder and 20 % wt calcium hydroxide was sintered at 700 – 800 °C to produce a complete crystalline product. The sintered product was milled and become grayish fine powder pigment. From the SEM images, the grain size approximately 0.404 µm. The pigment was mixed with binder, solvent, and additives and grinded to become a paint and tested (glossy test, hardness test, adhesion test, and thermal conductivity test). From the result, the optimum composition of a paint are 22 % wt POFA pigment, 29 % wt binder, 39 % wt solvent and 10 % wt additives (cobalt 10%, lead 32% and N.C Solution). It also discovers that the paint is good insulator with thermal conductivity approximately 78.95 W/m.°C.
Dalam kajian ini, abu terbang kelapa sawit atau POFA berkemungkinan dijadikan sebagai pigmen dalam teknologi cat disiasat. Penggunaan POFA sebagai pigmen cat adalah baik kerana kestabilan dan bukan ketoksikan bahan tersebut. Sebaliknya, menghasilkan pigmen warna dengan menggunakan POFA sebagai elemen didalam pigmen dapat mengurangkan sisa pepejal dalam industri minyak sawit, sebagai kelebihan tambahan bagi bahan mentah POFA. Objektif kajian ini adalah untuk mengenalpasti komposisi optimum POFA dengan mengabungkan bahan seperti pengikat, pelarut dan bahan tambahan sebagai pigmen dalam cat, untuk mengkaji cat POFA pada daya tahan haba dan menyiasat keupayaan lapisan cat POFA dalam standard ujian cat yang ditetapkan. Secara umum, kajian ini diharap dapat meningkatkan teknologi salutan dan juga boleh dijadikan sebagai sumbangan penting dalam bidang akademik, penyelidikan, pembangunan dan bidang kajian yang berkaitan dengan teknologi cat. Kajian tentang penyediaan optimum dan kekonduksian haba cat yang disediakan daripada sisa abu terbang kelapa sawit (POFA). Bagi penyediaan pigment yang optimum, 45% wt POFA dicampur dengan 25% wt sulphur, 10% wt serbuk grafik halus dan 20% wt kalsium hidroksida telah disinter pada 700-800 °C untuk menghasilkan produk kristal yang lengkap. Produk yang dibakar akan dikisar dan menjadi serbuk pigment kekelabuan yang halus. Dari keputusan imej SEM, saiz butiran adalah lebih kurang 0,404 mikron. Pigmen ini akan dicampurkan dengan pengikat, pelarut, dan bahan tambahan dan dikisar untuk menjadi cat dan diuji degan ujian kilatan, ujian kekerasan, ujian lekatan, dan ujian kekonduksian haba). Dari keputusan itu, komposisi cat yang optimum mempunyai 22% wt POFA pigmen, 29% wt pengikat, 39% wt pelarut dan 10% bahan penambah (cobalt 10%, lead 32% dan N.C Solution). Ia juga mendapati bahawa cat ini adalah penebat yang baik dengan kekonduksian haba kira-kira 78.95 W/m.°C.
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(Approximation)
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<tr>
<td>POFA</td>
<td>Palm Oil Fly Ash</td>
</tr>
<tr>
<td>SiO₂</td>
<td>Silicon Dioxide</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>Aluminium Oxide</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>Iron Oxide</td>
</tr>
<tr>
<td>SO₃</td>
<td>Sulphur Trioxide</td>
</tr>
<tr>
<td>min</td>
<td>Minimum</td>
</tr>
<tr>
<td>%</td>
<td>Percent</td>
</tr>
<tr>
<td>µ</td>
<td>Micron</td>
</tr>
<tr>
<td>% wt</td>
<td>Weight Percent</td>
</tr>
<tr>
<td>BC</td>
<td>Before Century</td>
</tr>
<tr>
<td>Ca(OH)₂</td>
<td>Calcium Hydroxide</td>
</tr>
<tr>
<td>Si</td>
<td>Silicon</td>
</tr>
<tr>
<td>Al</td>
<td>Aluminium</td>
</tr>
<tr>
<td>°C</td>
<td>Degree Celsius</td>
</tr>
<tr>
<td>°C/min</td>
<td>Degree Celsius Per Minute</td>
</tr>
<tr>
<td>h</td>
<td>Hour</td>
</tr>
<tr>
<td>(CH₃)₂CO</td>
<td>Acetone</td>
</tr>
<tr>
<td>g</td>
<td>Gram</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning Electron Microscope</td>
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$\theta$ - Theta

$^{\circ}$ - Degree

XRD - X-Ray Powder Diffraction

ASTM - America Society For Testing And Materials

H - Hardness

BS - British Standards

DIN EN - Deutsches Institut Für Normung (German Institute For Standardization)

ECCA - European Coil Coating Association

ISO - International Organization For Standardization

GU - Gloss Unit

W - Watt

W/m.°C - Watts Per Meter Degree Celsius

K - Kelvin

$Q'$ - Rate Of Heat Transfer

k - Thermal Conductivity

A - Cross-Sectional Area

T$_1$-T$_2$ - Temperature Different

L - Thickness

EDX - Energy-Dispersive X-Ray

Be - Beryllium

C - Carbon

O$_2$ - Oxygen

Mg - Magnesium
Sulfur

Potassium

Calcium

Molybdenum

Quartz

Muscovite

Kalsilite

Lazurite

Centipoise
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CHAPTER 1

INTRODUCTION

1.1. BACKGROUND OF THE STUDY

The manufacturing of paint dates back to the 1700s. It is mainly used for artwork initially due to its high production cost. The technology expands continuously for many years, with techniques being passed down from generation to generation by travelling craftsmen. A few centuries later, through further innovations and improvement, paints factories are opened in Europe and America. The mass production of paint in the nineteenth century marks the starting point of price down; to such an extent that houses start to be painted. Now, in the twentieth century, the chemical composition of paint has been unlocked, indicating that the manufacturing of paint has finally moved from being an art to a science (Waldie, 1983).

Paint is widely used to protect surfaces of objects and for colouring purposes. Its applications range from car paint coating to road marking. Different sorts of paint are used in different kinds of applications. The basics ingredients for most of the paint are pigments, binders, solvents and additives. The protective coating used for corrosion protection consists of pigment too. Despite the apparent purpose of pigmentation on the layer is to provide colour and opacity, correct formulation of pigment into the protective layer is far more than that. For corrosion protective coating, pigment works by providing inhibition or passivation of metal surfaces, preventing corrosion and strengthening the paint film.

The manufacturing of paint is rather simple. The pigment, binder and thinner are blended in the correct proportions, such that the final finished film is continuous, smooth and attractive to the eyes when the paint is applied to the surface. In the paint
technology, there are two types of pigment - the organic pigment and inorganic pigment. The organic pigment is for decorative purposes, while the inorganic pigment is for protective purposes (Talbert, 2008). Certain types of pigment increase the heat, abrasion, acid, or alkali resistance of the layers. The particle size and shape of pigment, ease of wettability by the binder, and attributes associated with a particular density contribute to the characteristics of viscosity and wet coating application significantly, in addition to dry lining and protection (Gedeon, 1995).

Various industrial wastes can be used as the pigment in paint technology, where one of them is fly ash. Fly ash, also known as flue-ash, is one of the residues that commonly obtained from the flue gases of furnaces at pulverised coal power plants. When coal is burnt in pulverised coal boilers, the minerals, entrained in the coal, are thermally transformed into chemical species that are reactive or could be chemically activated. On the other hand, bottom ash refers to ash which does not rise. Fly ash is generally captured from the chimneys of coal-fired power plants, whereas bottom ash is removed from the bottom of furnaces.

Palm oil fly ash (POFA) powder is residue obtained from the dust collectors in the boiler that uses palm oil dregs as fuel. The boiler here is usually found in power plant industries. The palm oil residue, palm fibre and shells are burnt in the boiler at about 800°C to 1000°C to produce steam for electricity generation in biomass thermal power plants (Science, 2012). Dregs of oil palm, combining with the kernel and its bunch are turned into fly ash after burning in a boiler. Fly ash is the result of burning, and has strong chemical bonding, high strength and low water absorption ability.

1.2. PROBLEM STATEMENT

The relatively high price of the paint in current market is mainly attributed to the cost of its raw material. To reduce the production cost of paint, further study into the use of alternative low cost materials forms the key component in the paint technology nowadays.

Biomass waste can be utilized as the pigment in paint technology through chemical processes. Silicon dioxide that exists in pigment composition, known as extender, is one of the components found in POFA. It thus indicates that POFA has high potential to be a low cost alternative to current raw material used in paint, since
it is a type of waste product. The POFA may be used to produce the multifunction paint that is water resistant and heat resistant.

In addition to cost efficiency, utilization of POFA as raw material in paint pigment helps to solve environmental problems, for instance, ground water contamination and spills of storage.

1.3. OBJECTIVES OF RESEARCH

The objectives of this study are summarized as follows:

1) To identify the optimum composition of POFA in a combination of binder, solvent and additives as the pigment in paint.
2) To evaluate the heat resistant characteristic of POFA paint.
3) To investigate the ability of POFA in paint coating using paint standard testing.

1.4. SCOPE OF RESEARCH

The scopes of this study are summarized as follows:

1) The study focused on the standard operation procedure of making paint by using POFA as a pigment.
2) The paint properties were examined according to the composition ratio of paint component and mixing ratio of raw materials in the pigment.
3) The POFA was used as the material in the manufacturing of pigments of paint.

1.5. SIGNIFICANT OF RESEARCH

In general, the research hopes to improve the technology of coating. It can also be a significant contribution to the academic, research, development and field studies related to the paint technology.

The research is important to produce a quality paint through the standard procedure in coating technology. Besides that, fly ash has a high potential to be developed as a pigment in paint application in order to improve the quality of paint. Furthermore, the data collected in this experiment are able to contribute to the
knowledge in regards to the uses of biomass waste as one type of pigment in the paint industry.

The research may be able to reduce environmental problems, such as ground water contamination and spills of bulk storage. Besides that, the added value of the industrial waste can be identified through this study.

1.6. AIM OF RESEARCH

The aim of the research is to exploit the potential of industry waste, specifically, POFA, as the pigment used in the paint technology, due to its abundance, inexpensiveness and ready accessibility. The making of paint with the POFA as the pigment is operated in accordance with the standard procedures, and the optimum mixing ratio of raw materials that gives better paint properties has been identified.

1.7. OVERVIEW OF THESIS

The thesis begins with Chapter 1, with an introduction to the history of paint. The needs of the study are discussed, in addition to the significance of the conducted study explained in this thesis. The objectives, scope, and overview of this thesis are then described.

Chapter 2 provides a review of industrial waste, palm oil fly ash (POFA). The review of paint components, including pigment, binder, additives and solvents are presented. The characteristics of the heat resistant paint are reviewed.

Chapter 3 discusses the methodology used to conduct this study. Specifically, the procedures of pigment preparation, paint preparation and characterization of paint properties were discussed in detail.

Chapter 4 presents the analysis results obtained during the paint pigment preparation initially. From the results, the pigment sample with the best quality was used in producing the POFA paint. The obtained optimal paint pigment and the POFA paint sample were discussed in detail in this chapter. Lastly, Chapter 5 discusses the conclusion and recommendation the entire work.
CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

A literature review is the first step towards considering the critical points of current knowledge (Baglione, 2012). In this chapter, the selected studies of paint and pigment, specifically, patents, research papers, journals, books and available products, were evaluated.

The literature review of composition, paint components and characterization was conducted as well. This is to explore and investigate the related applicable component with practical techniques on the existing paint that could assist in the paint research.

2.2 PALM OIL FLY ASH

Palm oil fly ash (POFA) is the industrial waste formed after the combustion of palm oil dreg. It is an amorphous solid, meaning that it is shapeless or non-crystalline solid. Ash can be classified into two types, namely, the bottom ash and fly ash. The term of “biomass” refers to a material that derived from living or recently living biological organisms. It is also often used to refer to plant material, food processing and preparation, and domestic organic waste that can be formed as the source of biomass. The temperature required to burn oil palm dregs in the boiler is between 700-1000 °C (Tangchirapat, 2007).

On the other hand, the term of “ash” is referring to the non-combustible mineral content of biomass. Fly ash consists of very small particles of ash, and will be carried
Various types of biomass produce ash having similar pozzolanic activity as coal fly ash, including palm, rice husk, wheat straw, sugar cane straw and wood. Generally, the fly ash is produced from palm oil, which has been blended and crushed and went through the burning tunnel (boiler). Here, palm oil dreg means the bunch and kernel of palm oil which have been pressed by a special press machine. The burning process of palm oil dreg in a furnace is the first step in order to produce fly ash (Tulus, 2011). The burning process makes dreg into small particles. Subsequently, a fan is used to pull out lighter fly ash and leave heavier fly ash in a special container. This process to isolated fly ash and bottom ash. During combustion, bottom ash is the ash left behind in or under the grate or combustion region, or at the bottom of a gasifier. The resultant fly ash in the first stage is produced in the lay bar of the system. This process will be repeated in the second stage but with different sizes of particles (Mustaffa Bin Hj Ibrahim, 2010). In the last stage, fine particles are stored below the chimney boiler. The fan is used to inhale lighter fly ash or granule from the chimney.

POFA is physically powder, fine amorphous and dark grey in colour. The colour of fly ash depends on the percentage of carbon in POFA. The more the carbon in fly ash, the darker the grey colour. In addition, POFA also has a variety of particle sizes and similar forms of spherical. One example of an electron micrograph of POFA is shown in Figure 2.1 (Awal, 2010).

Figure 2.1: Scanning Electron Micrograph from POFA (Awal, 2010)

POFA has its own benefit as compared to other new waste materials used in industries. In addition to its inexpensiveness and abundance, POFA can be utilised in
new paint technologies. POFA can be classified into two classes - F and C, based on the chemical composition of the fly ash (Tulus, 2011). The chemical requirements to classify any fly ash in accordance with ASTM C 618 standard are shown in Table 2.1.

Table 2.1: Chemical Requirement for Fly Ash Classification (Awal, 2010)

<table>
<thead>
<tr>
<th>Properties</th>
<th>Fly Ash Class</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicon Dioxide (SiO₂) + Aluminium Oxide (Al₂O₃) + Iron Oxide (Fe₂O₃), min, %</td>
<td>70.0 F, 50.0 C</td>
</tr>
<tr>
<td>Sulphur Trioxide (SO₃), min, %</td>
<td>5.0</td>
</tr>
<tr>
<td>Moisture content, min, %</td>
<td>3.0</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>6.0</td>
</tr>
</tbody>
</table>

The use of class F fly ash containing up to 12% lost ignition may be approved by the user if acceptable performance result are available.

The chemical compositions of POFA were determined by standard wet chemical analysis method as in (Tulus, 2011). The wet sieve analysis of POFA showed that 78% of the particles were below 4μm size. The chemical analysis of various oxide percent content, (wt %) is shown in Table 2.2 (Tulus, 2011).

Table 2.2: Chemical Composition and Oxide Percent Content (Awal, 2010)

<table>
<thead>
<tr>
<th>Chemical Compositions</th>
<th>Oxide Percent Content, (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>62.12</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>21.30</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>5.55</td>
</tr>
<tr>
<td>TiO₂</td>
<td>1.38</td>
</tr>
<tr>
<td>MgO</td>
<td>1.58</td>
</tr>
<tr>
<td>CaO</td>
<td>0.53</td>
</tr>
<tr>
<td>K₂O</td>
<td>4.24</td>
</tr>
<tr>
<td>Loss on ignition</td>
<td>3.3</td>
</tr>
</tbody>
</table>

According to existing studies, the characteristic of thermal insulation has been observed in construction materials contain POFA. The construction materials with a high percentage of POFA possess low thermal conductivity properties and high thermal resistance (Balo, 2013).
2.3 PAINT

Paint is one of the oldest synthetic substances. The first paint was made from clays and chalks with animal fats at more than 35000 years ago. Egyptians brought significant improvement to the paint technology in 2500BC. They developed paint with the combination of blue pigment (ground azurite), binders (animal fats such as gums, wax and maybe also albumen), and solvents. Many colours were available from both natural and synthetic sources. Interestingly, a purple pigment can be even made from heating the yellow earth (Waldie, 1983).

Paint is used for colouring and protecting the surface, including house, car, road sign and underground storage vessel. Each application requires different types of paint. The dependency of paint properties with different paint composition is the focus of this study. Paint, in general, consists of pigment, binder, additives and solvents (Waldie, 1983).

The paint production is determined by the overall cost of pigments, solvents, binders and additives. In order to reduce the cost of paint production, pigment preparation cost must be reduced. Material selection of the paint production is important in order to reduce the cost and produce high-quality paint (Talbert, 2008).

2.3.1 Heat Resistant Paint

The first heat resistance paint was produced in 1950. During the early stage, the production cost of heat resistance paint was very expensive. It was difficult to be applied to the object’s surface too. In addition, the heat resistance paint produced unhealthy smoke when in use. The smoke contained carcinogens that can be deadly and dangerous to humans. With the development of technology and innovation in the paint manufacturing, the original heat resistance paint was gradually replaced by intumescent paint, which was safer to handle. Intumescent paint is derived from products that are free of carcinogenic components content. Generally, the function of heat resistance paint is to withstand the high temperatures for a long time or to be used as thermal insulation. Silicon resin content increases the heat resistance of paint so that it has good heat resistance as compared with conventional paints. Paint heat resistance is used to minimize the fire damage. However, the heat resistance paint cannot protect flammable materials, such as wood (Automative Solution, 2008).
The heat resistance paint was produced in order to reduce the percentage of damages when fire. It controls the heat transfer rate and delays fire burning within a limited time. Therefore, the heat resistant paint was in-turnescent and swelled up when subjected to a certain temperature. Swelled up paint will produce a kind of protective material called "char" that prevents thermal conduction (Automative Solution, 2008).

2.4 PAINT COMPONENTS

Paint is an engineered product produced by mixing different ingredients. The formula usually includes the binder, pigments, additives, and solvents. The selection of the components is important as it decides the paint properties, quality and effect of the paint such as stability, handling, cleanup, and most importantly, the performance of the product (Talbert, 2008).

2.4.1 Pigment

Pigment is used for colouring of paint, ink, plastic, fabric, cosmetics, food and other materials. Pigment does not only provide the paint its colour, it also serves to protect the surface underneath from corrosion and weathering. In addition, pigment helps to hold the paint together (Waldie, 1983). Pigment is a material that can reflect or transmit light, and as a result, it shows different colours. Other than that, pigment gives mechanical strength, durability, opacity, tinting strength, bleeding characteristics, particle size and shape and corrosion protection for metallic substrates to the paint (Talbert, 2008). Some pigments are toxic, such as the lead pigments that are used in lead paint.

Size and shape of the pigments are important properties for paint components. It affects the form of the molecular of paints or packing between the other components. Most pigments are in crystalline form, which often affects the characteristics of the pigments. The particle size, shape and distribution of a pigment affect the paint properties, including rheological properties, shades, gloss, weathering characteristics and ease of dispersion (Talbert, 2008).

There are two major types of pigment in the earth, namely, the white pigment and colour pigment. White pigments are the major contributor in the paint formulation. White pigments are inorganic compounds of titanium, zinc, antimony or lead. The
white pigments are usually used to adjust the lightness or darkness of the final colour. On the other hand, the colour pigments are divided into inorganic and organic products. Organic pigments are usually used for decorative purposes, while inorganic pigments are used to add the protection properties to the paints. Some of them can be found naturally, while the others are synthesised through manufacturing process (Talbert, 2008). Both inorganic and organic substances are used in paint manufacturing, with the inorganic ones is cheaper but with less clear colours (Waldie, 1983). Special pigments can be used to give metallic finishes, for example for car bodies and to be used as hard wearing for road markings. Table 2.3 shows traditional properties of organic and inorganic pigments.

Table 2.3: Traditional Properties of Inorganic and Organic Pigments (Lambourne R., 1999)

<table>
<thead>
<tr>
<th>Pigment Properties</th>
<th>Inorganic</th>
<th>Organic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Colour</td>
<td>Often Dull</td>
<td>Bright</td>
</tr>
<tr>
<td>Opacity</td>
<td>High</td>
<td>Transparent</td>
</tr>
<tr>
<td>Colour Strength</td>
<td>Low</td>
<td>High</td>
</tr>
<tr>
<td>Insolubility</td>
<td>Good</td>
<td>Varies widely from good to poor</td>
</tr>
<tr>
<td>Chemical Resistant</td>
<td>Poor</td>
<td>Poor</td>
</tr>
<tr>
<td>Heat Resistant</td>
<td>Good</td>
<td>Poor</td>
</tr>
<tr>
<td>Durability</td>
<td>Good</td>
<td>Poor</td>
</tr>
<tr>
<td>Price</td>
<td>Cheap</td>
<td>Expensive</td>
</tr>
</tbody>
</table>

2.4.2 Binder

The binder holds the pigments to the surface. The binder is a polymeric substance. It is either dissolved in the paint or suspended in it by emulsifiers (Waldie, 1983). Binder, named as resin, functions as a vehicle (substance) in paint components. The resin is the main component to determine the paint properties, such as gloss, durability, flexibility and toughness.

There are two types of paint binder, namely, the convertible and non-convertible types. Convertible binders are materials that are used in an unpolymerised or partially polymerised state and undergo reaction to form a solid film after application to the substrate. Convertible binders include oils, oleoresinous varnishes, alkyds, amino resins, epoxy resins, phenolic resin, polyurethane resins, and thermosetting acrylics. Nonconvertible paints area based on polymerised binders
dispersed or dissolved in an evaporates medium to leave a coherent film on the substrate surface. Non-convertible binders include cellulose, nitrocellulose, chlorinated rubber, and vinyl resins (Talbert, 2008). Table 2.4 shows the types of major binder used in industries.

**Table 2.4: Types of Binder in Industries (Talbert, 2008)**

<table>
<thead>
<tr>
<th>Types of Binder</th>
<th>Purpose and Characteristic</th>
</tr>
</thead>
</table>
| **Drying Oils**         | 1. Used until the 1960's.  
2. Natural oils that polymerise as they dry.  
3. Take a long time to dry and have variable properties as the balance of oils varies from crop to crop. |
| **Alkyd Resins**        | 1. The most common resins to be used in solvent-based paints.  
2. Basically polyesters.  
3. Used for both air-drying and heat-cured paints. |
| **Vinyl and Acrylic Emulsions** | 1. Emulsions in water.  
2. Most common water-based binders for use in household paint. |
| **Epoxy Resins**        | 1. Based on polymers containing the simple organic compound 'oxirane' (ethylene oxide).  
2. A variety of other components are added to give a wide range of properties. |
| **Polyurethanes**       | 1. Polyurethanes are polymers of any ester of carbamic acid, H₂N—CO₂H.  
2. Tough.  
3. Durable films that retain their gloss.  
4. Easy to clean.  
5. Used for painting aircraft. |

### 2.4.3 Additives

Additives are chemicals added to paint in order to provide the special effects or properties to paints. Normally, the additives are used in small quantities to prevent the changes in other paint characteristics. Additives can affect the paint material finished film properties. Other than that, additives will also influence the chemical and physical properties of the paint. The effects of additives can be classified into six different types,
which are thickening agents, surface active agents, surface modifiers, levelling agents, coalescing agents and catalytically active additives (Talbert, 2008).

Additives as thickening agents increase the viscosity of the paint by influencing the rheological properties. Surface active agents function as dispersion, stabilisation and deformers in paints, while surface modifiers additives control the mechanical and optical properties of coated surface. Levelling agents and coalescing agents are used to controlling the flow and levelling of paint during and after the application, and before the film is formed. In addition, additives can be used as the catalyst to increase the rate of chemical reaction in paints. There are some additives which give special effects to paints, such as anti-skinning, light stabilisers, flame retardants, corrosion inhibitors, and biocides (Shin-Etsu Chemical Co. Ltd, 1996). Table 2.5 shows the additives used in paints manufacturing.

Table 2.5: Types of Additives in Paint Manufacturing (Lambourne R., 1999)

<table>
<thead>
<tr>
<th>Types of Additives</th>
<th>Purpose and Characteristic</th>
</tr>
</thead>
</table>
| Cobalt             | 1. Additives used as drying agents.  
                        2. Provides a dry surface after use. |
| Lead Plumbum       | 1. Traditional metal that is often used in the past.  
                        2. Serves to assist in drying, but requires combination with other metal sulfides except poison or colouring.  
                        3. Hazardous materials and materials are not environmentally friendly |
| Manganese          | 1. Among the best drying agents  
                        2. Serves as a drying agent.  
                        3. The lack of use in the production of manganese is in colour palette.  
                        4. Produced a dark or dark brown paints. |
| Calcium            | 1. Not the main additive.  
                        2. To affect substantially the production of paint.  
                        3. Serves as a supplement in the cobalt to increase the drying temperature. |
| Aluminum           | 1. An additive rather unusual  
                        2. A good agent in the production of paints.  
                        3. Drying additives that are not toxic because of the free lead content.  
                        4. The advantages of using aluminum in the production of paint are paint colour stability, quick-drying paint and create a hard surface layer. |
2.4.4 Solvent

The solvent evaporates when the paint is drying. Solvents are used in paint manufacturing to dissolve different compounds used in the paint formulation, such as pigment and binder, making the paint the correct consistency for application. For resin-based paints, various organic compounds are used with the mineral turpentine being the most common. Water is also a type of inorganic solvent. For emulsion paints, the solvent is simply water (water is a good solvent due to its polarity) (Waldie, 1983). The use of water is favoured, due to its advantages of availability, inexpensive, odourless, non-toxicity and non-flammability. The hydrocarbon solvent is an inorganic solvent, producing a more quality paint. In addition, the hydrocarbon solvents act as a carrier agent for the pigment and other basic components in the paint application. Examples of hydrocarbon solvents are toluene, xylene and mineral spirits. Besides the hydrocarbon solvent and water, the oxygenated solvent is also a type of solvent used in paint. Paints that use oxygenated solvents will have a strong odour and harmful to the human body. Examples of oxygenated solvents are butyl alcohol, ethyl alcohol, ethyl glycol monoethyl ether, acetone, methyl ethyl ketone, methyl isobutyl ketone, butyl acetate and ethyl acetate. (Talbert, 2008). In comparison, hydrocarbon solvents are more commonly used in the paint industry, compared to oxygenated solvents. However, the oxygenated solvents have better water solubility and evaporation rate (Clark, 1983.)
CHAPTER 3

METHODOLOGY

3.1 INTRODUCTION

There are three stages involved in this study, i.e. preparation of pigment using POFA, production of paint, and lastly, characterization of paint properties, as shown in Figure 1. POFA was the main material used in the preparation of pigment. The preparation of POFA as pigment involves five key processes - mixing process, reductive heating process, heating oxidative process, ball milling process and sieving process, and was explained in detail in this chapter.

After the paint pigment was produced from POFA in the first stage, the binders, solvents, and additives were mixed with other components, in producing a paint that meets the quality demanded by the market. Lastly, the characteristics of paints were identified.
3.2 PREPARATION OF PIGMENTS

POFA was the main material used in this study. Besides POFA, another three raw materials were added in the process, including sulphur, calcium hydroxide, and graphite fine powder. Distilled water was used as a medium that allowed a mixture of the raw materials into phase compounds.
3.2.1. Raw Material

In this study, POFA was suggested as a new low cost alternative pigment to be used in paint industry. This is attributed to POFA contains about 60% of silica, SiO$_2$, which is the common extender pigments used in the paint factory. It has been found that SiO$_2$ was weather resistance and can act as a flatting agent (Awal, 2010). In addition to the high content of SiO$_2$, POFA can be found in abundance in palm oil processing factory as biomass.

POFA was also used commonly as an additive for the cement content of concrete. Concrete with POFA has demonstrated low thermal conductivity (Balo, 2013). POFA used in this research was obtained from an oil palm processing plants located in Batu Pahat. Figure 3.2 shows the POFA used in this study.

The other raw materials used in pigments preparation were sulphur, calcium hydroxide and graphite fine powder. Sulphur powder is yellow in colour. It was used to produce paint pigment with chemical element symbol S and atomic number 16. During the preparation of pigment, sulphur powder acts as the pigment colouring agent.

Calcium hydroxide is an inorganic compound with the chemical formula of Ca(OH)$_2$. It is a white or colourless crystalline powder. In the pigment preparation process, calcium hydroxide was used as the starting reagents. Graphite fine powder, or better known as black lead or carbon, is widely used in heavy industry and motorsport.
Natural graphite is mostly consumed for refractories, batteries, steelmaking, expanded graphite, brake linings, foundry facings and lubricants and native element mineral (U.S. Geological Survey, 2009). Graphite fine powder was used as the starting reagents in the pigment preparation process.

### 3.2.2. Pigment Preparation Process

Five main processes were required in order to produce a paint pigment, specifically, the mixing process, reductive heating process, heating oxidative process, ball milling and sieving process. Figure 3.3 shows the process of preparing a pigment as disclosed in the flow chart of the methodology.

![Pigment Preparation Flow Chart](image)

**Figure 3.3:** Pigment Preparation Flow Chart

#### 3.2.2.1. Mixing Process

Mixing process - a process where all the raw materials used in pigment preparation process, *i.e.* POFA, sulphur, calcium hydroxide, graphite fine powder and distilled water were mixed in a compound. Prior to the mixing process, determining the optimal ratio of raw materials and heating conditions with best pigment characteristic was required.

Heating conditions were divided into two conditions – open condition and closed condition. Under different heating conditions, pigment appears in different colours. In order to produce pigment with similar characteristics as the product in the market, different mixing ratios were determined through calculation.
Based on the observations, high content of POFA and sulphur, with low content of graphite fine powder and calcium hydroxide, produced high quality pigment. Conventionally, the commonly used starting reagent in the industry is kaolin, which is bright blue in colour. In this research, kaolin was replaced by POFA as starting reagents. The ratio of POFA was maintained the same as kaolin. The kaolin was replaced by POFA, due to the main chemical components and the ratio of Si to Al in POFA were same as kaolin (Dutrow, 2013). In this regard, the cost of raw material may be decreased. The calculation step to determine the weights of four components used in the study are shown as follows:

Considering the total weight of compounds as $X_g$ and the mixing ratio of 40% POFA, 20% sulphur, 20% graphite fine powder and 20% calcium hydroxide was used. The weight of each component was:

\[
\text{Weight of 40 wt% POFA} = \frac{40}{100} \times X_g = 0.4X_g
\]

\[
\text{Weight of 20 wt% sulphur} = \frac{20}{100} \times X_g = 0.2X_g
\]

\[
\text{Weight of 20 wt% graphite fine powder} = \frac{20}{100} \times X_g = 0.2X_g
\]

\[
\text{Weight of 20 wt% calcium hydroxide} = \frac{20}{100} \times X_g = 0.2X_g
\]

After the weight of each ingredient was obtained, all ingredients with the appropriate ratio were added into the crucible. The ingredients were mixed before the distilled water was added to the mixing process to form a mixture. In this study, six samples with the different heating conditions and ratios of raw material were considered (Table 3.1). This is due to in addition to the heating condition, the colour of the produced pigment was affected by the mixing ratio of four ingredients too.
### Table 3.1: Ratio of Raw Material in Pigments

<table>
<thead>
<tr>
<th>Sample</th>
<th>Heating Condition</th>
<th>POFA (wt %)</th>
<th>Sulphur (wt %)</th>
<th>Graphite (wt %)</th>
<th>Calcium Hydroxide (wt %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Open</td>
<td>40</td>
<td>20</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>2</td>
<td>Close</td>
<td>40</td>
<td>20</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>Open</td>
<td>45</td>
<td>25</td>
<td>10</td>
<td>20</td>
</tr>
<tr>
<td>4</td>
<td>Close</td>
<td>45</td>
<td>25</td>
<td>10</td>
<td>20</td>
</tr>
<tr>
<td>5</td>
<td>Open</td>
<td>40</td>
<td>40</td>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>6</td>
<td>Close</td>
<td>40</td>
<td>40</td>
<td>10</td>
<td>10</td>
</tr>
</tbody>
</table>

#### 3.2.2.2. Reductive Heating Process

The mixing process was followed by the reductive heating process in high temperature furnace model *Lin High Therm VMK-1800* during the pigment preparation. The mixture was fed into the furnace and heated such that the electrons were doubled up and the oxygen resided in the mixture was removed.

The ideal temperature used to produce the pigment was between 700 °C to 800 °C. The carbon monoxide in atmosphere reduced the elemental sulphur at 750 °C (Al-Tawaha, 2011). In the temperature profile, the heating time of 10 hours was used. The time span can be divided into three main parts - increasing temperature, constant temperature and decreasing temperature. The temperature profile with increasing temperature was 1 hour 15 minutes, with the increasing rate of 10 °C/min. In the following 6 hours, the temperature of the heating process was maintained constantly at 750 °C. The temperature profile with decreasing temperature was 2 hours 45 minutes, with the decreasing rate of 4.545 °C/min. After the reductive heating process, the mixture formed a solid state, or also known as paint pigments. Figure 3.4 shows the temperature profile of heating process while Figure 3.5 shows the furnace used in the heating process.

![Figure 3.4: Temperature Profile for Heating Process](image-url)
3.2.2.3. Oxidative Heating Process

The reductive heating process was followed by the oxidative heating process in pigment preparation process. The mixture was exposed to the ambient air until its temperature was same as room temperature. This process increased the oxygen and removed the electrons in the pigment in order to form the desired colour.

3.2.2.4. Ball Milling Process

A ball mill is a type of grinder used to grind and blend materials, for the applications in mineral dressing processes, paints, pyrotechnics, ceramics and selective laser sintering. Grinding can be carried out either wet or dry, with the former was performed at low speed (Alban J. Linch, 2005). The ball milling machine model Planetary Micro Mill Pulverisette 7 Classic Line was used in this study, as shown in Figure 3.6.

Ball milling process produced a fine powder pigment from the pigment lumps. The speed, grinding time and the number of balls of the ball milling process was set in accordance with the desired particle size. In this study, the speed of the milling process was set to 250 rev/min for 15 minutes continuously. Ten balls were used to produce the pigment powder.
3.2.2.5. **Sieving Process**

The sieving process was the final step in the preparation of paint pigments. The particles were separated into the desired size using a device. This was to characterise the particle size distribution of a sample subsequently. The sieving process was typically done using a woven screen, such as a mesh or net.

The model of the sieving machine used in this study is *Vibratory Sieve Shake Analysette 3 Pro* (Figure 3.7). Prior to the sieving process, the size of sieve was set based on the desired particle size, which was chosen as 45 μm in this study. The coarsest pigment particles, which up to 50 μm, was an extender pigment (Lambourne R., 1999). The extender pigment was not suitable to be used for gloss paint making. The pigment particles with the size of 45 μm were easier to stick and dilute. In addition, the obtained pigment fine powder was more soluble in the paint components.
3.3 PAINT COMPONENTS

Four main components, namely, pigments, binder, solvent and additives, were used in preparing the paint. The paint components used in this research were supplied by the Ample Green Coating Enterprise Sdn. Bhd.

3.3.1 Pigment

The preparation of pigment from the raw materials of sulphur, calcium hydroxide, and graphite has been discussed in the previous section. The paint characteristics, such as colour, opacity, durability, and mechanical strength, corrosion protection for metallic substrates viscosity, gloss and adhesion relied on pigments. In addition, the coarsest pigment particle, which up to 50 µm will react as extender pigment, and was not suitable to be used for gloss paint (Awal, 2010). Figure 3.8 show the obtained POFA pigments after the sieving process.

![Figure 3.8: POFA Pigment](image)

3.3.2 Additive

Two types of additives, cobalt 10%, lead 32% and N.C solution were used in this study. The additives were added in order to adjust the special effects of paint, such as viscosity, foaming, skinning, pigment dispersion, stability, hardness, gloss, UV resistance, fire resistance, and bacteria resistance. The additives can act as algaecides, deodorizers, disinfectants, fungicides, preservatives, and sanitizers, too.
3.3.3 Solvent

The solvent was a medium where the binder, pigment and additives were dissolved in molecular form. The acetone was used as a solvent in this study. Acetone is an organic compound with the chemical formula \(((\text{CH}_3)_2\text{CO})\). It is colourless and miscible in all proportions. Acetone is non-toxic and known to be non-harmful to human beings.

3.3.4 Binder

Binder, or specifically, polymethyl methacrylate is currently used in paint industry. It acts as a vehicle (substance), combining all other components. Acrylic resin, having the advantages of water resistance, better adhesion, and resistance to alkali (Ryoko Co., 2014), was used in this study. The binders with different mixing ratio were discussed further in the mixing of paint components.

3.4 MIXING PAINT COMPONENTS

The main paint components, \textit{i.e.} the binder, pigment, solvent and additive were mixed together using magnetic stirrer hot plate. Different ratios of paint components were applied in the mixture. The manipulated ratios were the mass percentage of pigment and the ratio of mixture paint component, as suggested by the Ample Green Coating Enterprise Sdn. Bhd. The suggested mixing ratio producing quality gloss paint, namely, the alkyd paint, was 15-25\% of pigment, 25-30\% of the binder, 35-40\% of solvent and 5-10\% of additives (Science, 2012). The gloss paint was produced without extender pigment (Balo, 2013). The paint components were mixed for 15 minutes using magnetic stirrer hot plate, according to the different ratios as shown in Table 3.2. The paint components were added in the beaker with the sequence of solvent, binder, pigment and lastly additives.

<table>
<thead>
<tr>
<th>Ratio</th>
<th>Pigment, g</th>
<th>Binder, g</th>
<th>Solvent, g</th>
<th>Additive, g</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ratio 1</td>
<td>22</td>
<td>29</td>
<td>39</td>
<td>10</td>
</tr>
<tr>
<td>Ratio 2</td>
<td>20</td>
<td>30</td>
<td>40</td>
<td>10</td>
</tr>
<tr>
<td>Ratio 3</td>
<td>18</td>
<td>31</td>
<td>41</td>
<td>10</td>
</tr>
<tr>
<td>Ratio 4</td>
<td>16</td>
<td>32</td>
<td>42</td>
<td>10</td>
</tr>
</tbody>
</table>

Table 3.2: Ratio of Paint Components in Each Sample
3.5 EXPERIMENTAL METHODS

3.5.1 Characterization of Palm Oil Fly Ash

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The focused beam of high-energy electrons is used to generate various signals that can detect the sample's crystalline structure and chemical compositions, derive from electron-sample interactions. SEM is often used to generate high-resolution images for shapes of objects in order to show spatial variations in chemical compositions. Besides, SEM is also widely used to identify phases based on qualitative chemical analysis or crystalline structure (Swapp, 2013) (Analysis, 2013).

The particles morphology of pigment was observed using JEOL JSM 6390LA Scanning Electron Microscopy, from where the microstructure of pigment was determined. In the range of 2θ, the testing was performed from 20° to 80°. Figure 3.9 shows the microscopic structure of POFA after the downsizing process. The average size of the grain size was calculated using the ASTM E112 standard. Figure 3.10 shows the fracture surface of POFA under the SEM analysis (Zain, 2011).

X-ray powder diffraction (XRD) is a scientific technique used for characterization of crystalline materials, identification of fine-grained minerals such as clays and mixed layer clays that are difficult to determine optically, determination of unit cell dimensions and measurement of sample purity (Dutrow, 2013). With


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