CHARACTERISATION OF CRAYONS BASED NATURAL WAXES AND WCO AS AN ADDITIVE

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BACHELOR OF MECHANICAL ENGINEERING TECHNOLOGY WITH HONOURS

2024

Faculty of Mechanical Technology and Engineering

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**A thesis submitted
in fulfillment of the requirements for the degree of
Bachelor of Mechanical Engineering Technology with Honours**

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

Faculty of Mechanical Technology and Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2024

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DEDICATION

Dedicated to

My beloved mother, Naimah Binti Mohd Yusof

My appreciated father, Ahmad Shapawi Bin Teh

My lovely brothers, Ahmad Faiz Firdaus and Ahmad Fadhil

My sweet sisters, Nurhanani and Nurhanisah

My inspiration, Iffa Sabrina

for giving me moral support, cooperation, encouragement and understandings.

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ABSTRACT

Cooking oil is widely used in household and food industry for cooking and frying purpose. It caused the amount of waste cooking oil increasing gradually over the years. Despite the waste cooking oil to be disposed, it can be used in making many new products such as candles, soap, and biodiesel. Thus, the idea of producing organic crayons from a combination of waxes with WCO as an additive is emerged. Crayons is an art medium that well-known among the children for drawing and painting. Generally, crayons are made by using paraffin wax as its main ingredient. This wax has a big potential on bringing harm to environment and human health. Many chemical substances found in commercial crayon such as arsenic, lead, and cadmium. Nowadays, many manufacturers of crayons has produced the organic crayon by using natural waxes such as beeswax, soy wax and vegetable wax. However, these waxes is low in durability and strength. The combination of natural waxes which are carnauba wax and beeswax were chosen in this project. Initially, the waste cooking oil need to undergo purification process by adding the alcohol which is ethanol into waste cooking oil. The ratio of ethanol to waste cooking oil is 15:1. This is the best ratio according to previous study conducted. In production of organic crayons, the treated waste cooking oil is mixed with the melted beeswax and carnauba wax in ratio 3:7, respectively. The ratio of these natural waxes promotes a better strength and durability. This ratio is obtained from several tests such as melting test in prior study. The mixture is then heated up and stirring continously before adding colour pigment. The mixture is poured into the mould to let it harden. The organic crayons produced are used as a sample to identify chemical substances present by using XRF analysis. These chemical substances are required to meet ASTM D4236 standard consumer safety specification for toy safety. From XRF analysis result, there are no harmful chemical detected in crayon and met ASTM D4236 regulations.

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ABSTRAK

Minyak masak digunakan secara meluas dalam isi rumah dan industri makanan untuk tujuan memasak dan menggoreng. Ia menyebabkan jumlah sisa minyak masak meningkat secara beransur-ansur dari tahun ke tahun. Selain sisa minyak masak dilupuskan, ia boleh digunakan untuk membuat banyak produk baharu seperti lilin, sabun, dan biodiesel. Oleh itu, idea untuk menghasilkan krayon organik daripada kombinasi lilin dan minyak masak terpakai telah tercetus. Krayon adalah sejenis medium seni yang terkenal dalam kalangan kanak-kanak untuk melukis dan mewarna. Pada asasnya, krayon diperbuat daripada lilin parafin sebagai bahan utama. Lilin ini mempunyai potensi yang besar untuk memberi ancaman kepada alam sekitar dan kesihatan manusia. Banyak bahan kimia telah dijumpai dalam komersial krayon seperti arsenik, timah dan kadmium. Pada masa kini, banyak pengeluar krayon telah menghasilkan krayon organik menggunakan lilin semula jadi seperti lilin lebah, lilin soya dan lilin sayur. Walaubagaimanapun, lilin-lilin ini mempunyai kekuatan dan ketahanan yang rendah. Kombinasi lilin semula jadi iaitu lilin lebah dan lilin carnauba telah dipilih dalam projek ini. Pada awalnya, sisa minyak masak perlu menjalani proses penulenan dengan menambah alkohol iaitu etanol ke dalam sisa minyak masak. Nisbah yang digunakan untuk etanol kepada sisa minyak masak terpakai ialah 15:1. Nisbah ini adalah nisbah terbaik berdasarkan kajian lepas. Dalam pembuatan krayon organik, sisa minyak masak terawat dicampurkan dengan lilin lebah dan lilin carnauba yang telah dicairkan menggunakan nisbah 3:7. Nisbah lilin semulajadi ini memberikan kekuatan dan ketahanan yang lebih baik. Nisbah ini diperolehi daripada beberapa ujian seperti ujian pencairan daripada kajian lepas. Campuran ini seterusnya dipanaskan dan dikacau secara berterusan sebelum memasukkan pigmen pewarna. Campuran ini dituang ke dalam acuan dan dibiarkan untuk mengeras. Krayon organik yang telah dihasilkan ini digunakan sebagai sampel untuk mengenalpasti bahan kimia yang terdapat dalam krayon organik menggunakan analisis XRF.Bahan-bahan kimia ini mestilah memenuhi standard ASTM D4236 spesifikasi keselamatan pengguna untuk keselamatan mainan. Daripada keputusan analisis XRF, tiada bahan kimia berbahaya yang dikesan dalam krayon dan memenuhi peraturan ASTM D4236.

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ACKNOWLEDGEMENTS

In the Name of Allah, the Most Gracious, the Most Merciful

First and foremost, I would like to thank and praise Allah the Almighty, my Creator, my Sustainer, for everything I received since the beginning of my life. I would like to extend my appreciation to the Universiti Teknikal Malaysia Melaka (UTeM) for providing the research platform.

My utmost appreciation goes to my main supervisor, Puan Nor Faizah Binti Haminudin, Faculty of Mechanical Technology and Engineering, Universiti Teknikal Malaysia Melaka (UTeM) for all her support, advice and inspiration. Her constant patience for guiding and providing priceless insights will forever be remembered. Also, to my co-supervisor, Dr. Mahanum binti Mohd Zamberi, Universiti Teknikal Malaysia Melaka (UTeM) who constantly supported my journey by giving an information and advice in writing this thesis.

Last but not least, from the bottom of my heart a gratitude to my parents for their endless support, love and prayers. My eternal love also to all my supportive friends who have been the pillar of strength in all my endeavors. Finally, I would like to express my gratitude to everyone who provided me with the assistance, support and inspiration to embark on my study.

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LIST OF SYMBOLS AND ABBREVIATIONS

ATSDR	-	Agency for Toxic Substances and Disease Registry
As	-	Arsenic
Cd	-	Cadmium
Cr	-	Chromium
EPA	-	Environmental Protection Agency
FDA	-	Food and Drugs Administration
IARC	-	International Agency for Research on Cancer
KOH	-	Potassium Hydroxide
LiF	-	Lithium Fluoride
LR	-	Lithol Rubine
NaOH	-	Potassium Hydroxide
Ni	-	Nickel
Pb	-	Plumbum
PIB	-	Polyisobutylene
PPM	-	Parts per million
PSA	-	Pressure-sensitive Adhesives
PTWI	-	Provisional Tolerable Weekly Intake
SEM	-	Scanning Electron Microscopy
TiO_2	-	Titanium Dioxide
UL	-	Tolerable Under Intake Level
VOC	-	Volatile Organic Compunds
WHO	-	World Health Organization
XRD	-	X-ray Diffraction
XRF	-	X-ray Fluorescene
°C	-	Degree celcius

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CHAPTER 1

INTRODUCTION

1.1 Background study

Crayon or also known as wax pastel is a substance that is widely used for drawing and writing purposes, especially among children. Originally, crayons are made by using paraffin wax as a hardening agent and non-toxic pigments. The first crayon produced was invented in 1903 by Edwin Binney and C. Harold Smith with a brand namely Crayola Crayons as shown in Figure 1.1. The characteristics of this crayon make it one of the best arts mediums among children. It is because these crayons are easy to use and less messy compared with other art mediums (Ahmad G.R et al., 2017). Moreover, it comes in a blunt state that can avoid the children from harming themselves or hurting their surroundings.

Figure 1.1 Crayola Crayons

Nowadays, most crayon companies or manufacturers take an alternative by changing the natural wax to the paraffin wax due to the cost and availability. However, paraffin wax was found to have hazardous substance that contains carcinogens which can lead to cancer since this wax was derived from petroleum or coal (Ben Beldman, 2020).

Nevertheless, after conducting experiments on some crayons, researchers found that there are still chemical substances such as lead and asbestos from a few manufacturers that claim their products are non-toxic crayons. Consequently, this will indirectly result in an awful environment in terms of health and community for the future generation. According to Dogo et al. (2022), interrupted cognitive development and poor health issues may occur even if the concentration of lead in blood is less than 10 mg dm^{-3}. Asbestos is still a major industry and used extensively in shipbuilding, house construction and insulation even though it has been banned by many countries. Long term asbestos exposure can increase the risk of mesothelioma and asbestosis that can be a factor of lung cancer (Robinson, 2012; O'Reilly et al, 2007). A study on the impacts of oil on environment done by Dahlmann et al (1994) found that the great number of cases of crude oil (paraffin wax) that been illegally discharge from the ship at the coasts of Netherlands, Denmark, and Germany cause water pollution and the deaths of seabirds.

Aside from that, waste cooking oil (WCO) which is a well-known waste that can cause pollution to the environment. This waste is acquired from domestic or food industries. The wrong way of disposing of this waste can result in clogging in drainpipes because the oil will solidify due to degradation and can lead to corrosion in the pipelines. WCO needs special disposal procedure since it is not diluted with water which can cause water pollution. It can be worsened if the oil reaches sewer streams that need extra cost for maintenance (Panadare D.C. & Rathod V. K., 2015). However, the usage of WCO as an additive to create new products such as cosmetics, organic soap and even organic crayons is growing rapidly considering it comes with low cost and easy to attain.

In this project, the treated WCO as an additive will be mix together with a combination of waxes (beeswax and carnauba wax) to produce organic crayons. Then, these

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crayons will undergo an analysis using X-Ray Fluorescence (XRF) to determine the elemental composition of organic crayon and do not exceed the standard limit.

1.2 Problem Statement

Presently, many chemical compounds such as arsenic, asbestos and lead are found in commercial crayons even though the manufacturers claimed that their products as non-toxic crayons. It may cause by the usage of paraffin wax as hardening agent which potentially bring hazardous to human health since it contains carcinogens. To overbear this issue, many manufacturers have produced organic crayons at the marketplace by substitute the use of paraffin wax to natural wax such as solely beeswax or additional with vegetable wax. However, the properties of these natural waxes which is low in strength and durability become an issue of organic crayons production.

In this project, an alternative way has been used by combining natural waxes which are carnauba wax and beeswax to promote a stronger durability and much friendly of crayon to consumers. Moreover, the increasing amount of WCO has been impacted the environment badly. Therefore, many initiatives has been proposed to recycle this waste such as converting the WCO into soaps, candles and even biodiesel. However, this waste needs proper treatment before ready to be used. Hence, the idea of using recycle WCO into a new product which is crayon has emerged.

Furthermore, children have a high likelihood of being exposed to dangerous chemicals found in common products, especially those intended for their use and enjoyment. Exposed to possibly toxic substances may lead to adverse effect on their health and well-being. Crayon is one of the tools for children to develop their cognitive developement and creativity. Even though the produced organic crayons claim to be safe due to the use of natural sources, they need to undergo an analysis to determine the possible harmful elements

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present in the organic crayon. The main goal of this project is to analyse the elemental composition present in organic crayon that produced from a combination of natural waxes and treated WCO as an additive.

1.3 Objective

The objectives of this research are as follows:

a) To compare the acid value of treated WCO using denatured ethanol with food-grade ethanol.

b) To conduct chemical testing using X-Ray Fluorescence to identify any toxic element and compare to ASTM D4236.

1.4 Scope of Research

Following is an overview of the research's scope:

- List the probable chemical components, such as arsenic, lead, chromium, cadmium, nickel, and antimony that were present in the crayon according to prior studies.
- Study on the maximum and minimum value for each chemical substance supposed to be in organic crayon.
- Purify the WCO by adding alcohol, denatured ethanol with ratio 15:1, which is 15 for denatured ethanol and 1 for WCO.
- Conduct a titration process by mixing 5 g of treated WCO with 25 ml of propanol and add 4 drops of phenolphthalein for titration process with Potassium Hydroxide (KOH) solution until the colour of mixture turn into red colour permanently.
- Calculate the acid value from the value of the titrant.

- To produce the organic crayon, two main ingredients are important; (1) hardening agent consists of combination of waxes which are beeswax and carnauba wax with ratio 3:7 (gram), respectively. (2) WCO as an additive to be added to the waxes combination (10 ml of WCO to 10 g waxes).
- Test the sample using XRF to identify existing chemical substances in organic crayon.
- Analyse the result of chemical substances in organic crayon such as arsenic, lead, chromium, cadmium, nickel, and antimony and compare to the toy safety standard ASTM D4236.

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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, study begins with introduction to variety form of art mediums including coloured pencil, watercolour paint, oil paint, crayon and many more. In addition, the study on crayon ingredients also has been discussed continues with different types of waxes such as beeswax, carnauba wax and soy wax. Next, a study on WCO and its application to produce a new product such as cosmetic, candles and soap were discussed. In addition, the chemical substances that may be contained in crayons such as lead, cadmium and arsenic also has been discussed at the end of this chapter.

2.2 Art Mediums

Art can be a great channel for humans to express their emotions, to strengthen their sense of community, to convey spiritual beliefs, to solidify their ideology and to illustrate knowledge. Artists turned their visions of life into art from various mediums that were easy for them to attain such as charcoal, clay, and wood. The development of art mediums evolves the perceptive of artists to improve their artworks to be more authentic. During the Renaissance era, art conveyed concepts about its viewers. Paintings, prints, sculpture and architectural structures all featured status, money, social and religious identities. Some of the famous Renaissance period artists like Leonardo Da Vinci who created the painting of Mona Lisa as shown in Figure 2.1. This painting is known as the most valuable painting in the world due to its uniqueness and very realistic portrait (Zelazko, 2018).

Figure 2.1 Portrait of Mona Lisa

Nowadays, many art mediums enhanced come with various types of colours and materials such as paint and coloured pencil. Art can be the best medium to enhance children's development, provide a safe outlet for emotions and promote self-esteem. Children enjoy using their artistic abilities to express themselves without worrying about right and wrong answers. In this way, they learn to trust themselves and express their feelings. They can explore the unknown with art mediums. It can fulfil cognitive development and optimize their creativity in the growth phase. However, parents should be alert about the substances in their child's art mediums because it may contain harmful substances even though the manufacturer claimed that their products are safe.

2.2.1 Coloured Pencil

Coloured pencil as in Figure 2.2 is a thin and colourful that encases in the wood as its grip. Wax or oil, water, binders, and pigments are all combined to create the core of a coloured pencil. The pigments can be either natural or synthetic coloured pigment. This art medium is an enhancement from the standard pencil that is made from charcoal and graphite**.**

Some factors that affect a brand's quality and its market price include the concentration of pigments in the core, the lightfastness of the pigments, the longevity of the coloured pencil, and the softness of the core.

Figure 2.2 Coloured pencil

Despite coloured pencil is the easiest way to create art, especially in preliminary levels, it could come with several challenges with this art medium. According to Brandon F. (2023), coloured pencils have difficulty getting the perfect blend. Getting a desirable colour and intensity that is not included in the box of coloured pencil will take a lot of experience for this art medium. It depends on the pencil's quality, the original colour choice and the substance used for the core. Besides, this art medium has difficulty maintaining the sharp point to colouring the complicated areas. The core of coloured pencil is easy to break and becomes blunt. Artists have to go back to the sharpener if they applied even the tiniest amount of pressure since it might easily shatter the tip.

2.2.2 Oil Paint

Oil painting has been a significant artwork since ancient times. But this art medium is not prominent in Europe until it was enhanced in the 7^{th} century by Flemish painter namely Jack Van Eyck. Before the evolution of oil paint, artists were more likely to use tempera paint. However, the characteristics of tempera paint is not friendly to the artists since it dries fast, did not dilute in water and make the artwork become dim and dull. Fundamentally, the binder's material of tempera paint is either vegetable gum, animal glue and egg yolk. Artists changed the material of binders to vegetable oil or linseed oil that created oil paint which is used nowadays (Serifaki et al., 2008).

The improvement of this art medium changes its behavior. The drying time becomes longer that can bring both benefits and disadvantages to artists. The benefit to the artists is they can make the changes to their artwork before the paint dries completely. It also can bring a disadvantage which is delayed the process of layering the paint to get the wanted colour. According to Viguerie et al. (2009), before adding further layers, it was required to wait for the previous one to dry for at least two days. Figure 2.3 shows the artwork done by using oil paint at Taxiarhis Church. Furthermore, oil paint poses a number of hazardous health risks that need a ventilated area to use it. It is because the solvents such as mineral and turpentine contain poisonous hydrocarbons and high level of volatile organic compounds (VOC) will fill the air with fumes when it dries.

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Figure 2.3 Michael and Gabriel paintings from Taxiarhis Church (Serifaki et al., 2008)

2.2.3 Acrylic Paint

Acrylic paint as in Figure 2.4 is an art medium that is improvised from oil paint. The material structure is totally changed which makes the acrylic paint dry faster, much safer, and easier to use since it is water-based. This art medium is commonly applied to building paintings to create murals and words of wisdom. However, the exposure of this painting to the sunlight might fade its colour due to chroma loss from the paint film. Even though acrylic paint is impenetrable to the environment, this phenomenon can lead to photodegradation (Jos´e S. et al., 2023). The cause of this phenomenon attributable to the photodegradation will form crystallized salt deposits known as efflorescence on the walls, stone and concrete of the paintings (Aline et al., 2020). This crystallized salt is formed either from rainwater, internal moisture, or from leached subsoil through the building's material. However, this phenomenon is not hazardous to human health, but it has a great potential for damaging building materials.

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Figure 2.4 Acrylic paint

2.2.4 Watercolour Paint

Watercolour paint can be said to be sone of the most common art mediums used for lessons at school. The substances of this art medium usually only colour pigment that is mixed with arabic glue. In general, this art medium is water-based and needs to be diluted with water before it is ready to use. Water acts as a vehicle to transport this art medium to paper or canvas. In addition, the quantity of water determines the thickness and brightness of the colour. Overwater can make the colour fade and dreary.

Transparent is a relevant term to describe watercolour painting. It can reflect the light to the artwork when the water is completely evaporated. Resistance of colour to light can have a detrimental effect on the strength of the paper or canvas that make watercolour paint degrade more rapidly compared to other art mediums even though using the same pigments. At present, Nalintip et al., (2021) found that this issue could be resolved by adding nanocellulose as a novel stabilising compound to the paper since its structure resembles the paper matrix. Figure 2.5 shows the molecular structure of nanocellulose.

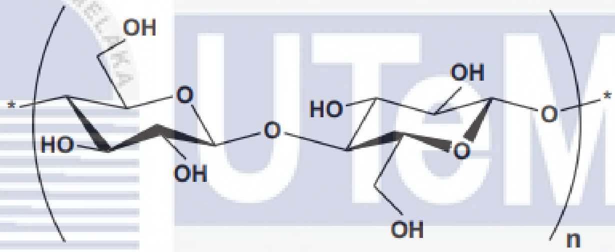

Figure 2.5 Molecular structure of nanocellulose (Phanthong et al., 2018)

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2.2.5 Crayon

Crayon is a stick-shaped art medium that is used for drawing and colouring purposes. This art medium can be said to be the greatest choice in art for children because it is the least messy compared with other art mediums. This is because they cannot scribble the crayon on their surroundings when they are playing with it. Crayons are made from a combination of paraffin wax and pigments. Yet the usage of paraffin wax in crayons may bring a great impact to the environment, especially toddlers since it was acquired from petroleum. After some research, many toxic elements were found like lead and cadmium in commercial crayons. Besides, it may take a very long period to decompose since the crayon wax is not biodegradable.

Nowadays, there are a lot of crayons in the market replacing paraffin wax with natural or organic waxes such as beeswax, soy wax and carnauba wax instead of paraffin wax as a primary ingredient. Many brands already produced their organic crayons with various shapes and colours to give the best fit and comfort use for children as in Figure 2.6. However, according to Angelica (2023), organic crayons have its limitation that is quite expensive and inclined the crayons to melt and break since the durability of organic waxes is not great as paraffin wax. Table 2.1 shows the pros and cons of comparison of organic crayons for children in the market. This project is highlighting crayons as an art medium. The next subtopic will be discussed on the ingredients, application and chemical substances contained in crayons.

Figure 2.6 The various of size and colours of crayons (Emma, 2023)

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Table 2.1 Pros and cons of organic crayons in the market (Corrita, 2022)

Pros	Cons
Washable	Expensive
Fit to hands	Small
Durable	Colours not vibrant
Made with natural ingredients	Not smooth
Long-lasting	Limited colours

2.3 Crayon Ingredients

Crayons are traditionally made using a combination of wax, pigment, and a binding agent. Crayons' solid and smooth texture comes from the wax, which is typically made from petroleum-based materials. However, more sustainable and environmentally friendly alternatives have been explored recently, including beeswax and carnauba wax. In this context, WCO becomes an appealing component for making crayons. WCO can be used as an additive to improve the properties of the wax and improves the overall quality of crayons. WCO is a renewable resource that would otherwise contribute to environmental pollution. WCO contributes to a more sustainable and environmentally friendly method of making crayons by not only reducing waste but also by adding a special and useful component. Not only this combination enhances crayons' texture and colour vibrancy but the durability and melting resistance are also improved. Furthermore, by using WCO and natural waxes, these eco-friendly crayons support a greener and more sustainable art supply sector.

This study highlights the utilization of WCO as an additive, along with a combination of carnauba wax and beeswax in crayons production that promotes many benefits compared to commercial crayons.

2.3.1 Waxes

Waxes are commonly used in paint formulation and coating to intensify and preserve its looks, strength, and resistance. It became famous among manufacturers and the agriculture sector due to its behavior which is insoluble in water. Nowadays, natural wax such as beeswax, carnauba wax and soy wax are widely used in industry due to their versatility. It can be useful in the food industry, cosmetics and agriculture to enhance their productivity. Basically, natural wax was derived from animals and plants. This factor makes the use of natural wax increase due to its low pricing and superior gelling capabilities (Xinya Gu et al., 2023). However, each natural wax has different properties such as durability, melting point and chemical reactions to ensure it is suitable for manufacturing process.

2.3.1.1 Paraffin wax

Paraffin wax (also known as crude oil) is a colourless and soft solid that derives from petroleum. The structure is then combined with a long-straight chain of hydrocarbons which consists of atom hydrogen and atom carbon as in Figure 2.7. Paraffin wax formed in a white waxy solid, odourless and tasteless with a heat combustion of 42 MJ/kg. Normally, paraffin wax is used for therapeutical treatment, candles, and crayons. It is because paraffin wax has a low melting point which is between 46°C to 68°C that easily turns the solid wax into liquid and vice versa.

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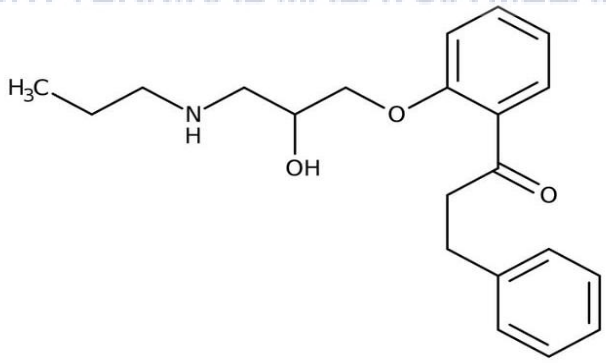

Figure 2.7 Molecular structure of paraffin wax

On the other hand, paraffin wax now can be said to be one of the important materials involved in boosting the efficiency of solar thermal energy storage at present. According to Anna V. et al. (2022), the combination of polyisobutylene (PIB) and paraffin wax comes with two benefits and functions which can act as pressure-sensitive adhesives (PSA) to lower the viscosity of the flow and accumulate the latent thermal energy. This makes adhesive processing easier and enables the use of adhesives as liquids that flow smoothly. When polyisobutylene is heated to high temperatures, paraffin wax melts and entirely dissolves, reducing the adhesive melt's effective viscosity by several decimal orders. As a result, it is feasible to produce adhesives using low-power mixing equipment while spending less time and energy heating and stirring the mixture.

2.3.1.2 Beeswax

Beeswax is the most common natural wax that is produced from honeycomb by honeybees. Figure 2.8 shows the appearance of the beeswax. The eight unique wax glands on the bottom of the worker bees' abdomens subsequently exudes the wax. The wax is released as a cool liquid and becomes a solid state. In terms of composition, the qualitative average for beeswax is composed of hydrocarbons (14%), monoesters (35%), diesters (14%), triesters (3%), hydroxy monoesters (4%), hydroxy polyesters (8%), monoacid esters (1%), acid polyesters (2%), free acids (12%), free alcohols (1%), and unidentified compounds (6%) (I. Bonaduce & Colombini M.P., 2004). Usually, this wax comes in yellow or white colour with melting points ranging from 61°C to 66°C. The colour of beeswax actually depending on the nation, forage, and types of bees. In industry, beeswax is the favourite material used by manufacturers since it releases a sweet smell and is edible. The application of beeswax is very prominent in cosmetics including lip balm, moisturizer skin and stretch mark removal. Food and Drug Administration (FDA) has approved the substance in beeswax composition that there is no cytotoxic behavior present, demonstrating their potential use for biomedical applications (Brito-Pereira R. et al., 2022).

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Figure 2.8 Beeswax

2.3.1.3 Carnauba wax

Carnauba wax is extracted from the leaves of carnauba palm, *Copernicia cerifera*, which only grows in northeastern Brazil. It is also known as Brazil wax or palm wax that widely used for various applications, especially in coatings and the food industry. This is because carnauba wax has a glossy finish, low solubility and the highest melting point compared to other natural waxes which is about 85°C. As shown in Table 2.2, the composition of carnauba wax has been done by Ullman's, 1997, and Vandenburg and Wilder, 1967. Esters, free alcohols, aliphatic acids, aromatic acids, ω-hydroxycarboxylic free acids, hydrocarbons, and diols triterpenes make up the complex combination that makes up carnauba wax (De Freitas, et al., 2019). The result may vary due to atmospheric factors such as the amount of rain or sun, the local moisture content, and the properties of the soil. In the food industry, carnauba wax has multiple functions, including as a glazing agent, anticaking agent, acidity regulator, and bulking agent for the surface treatment of the foods. The effectiveness of the wax-based coating in carrying out these tasks mostly depends on the emulsion's quality traits, such as viscosity, dispersibility, stability, wettability, spread-ability, and particle size (Susmita Devi L. et al., 2022).

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Table 2.2 The composition of carnauba wax (De Freitas, et al., 2019)

Constituent	Amount (%)	
	Ullmann's (1997)	Vandenburg and Wilder (1967)
Aliphatic acids	40	38-40
Diesters of 4-hydroxcinnamic acid	21	20-23
Ester of ω- hydroxycinnamic acid	13	12-14
Free alcohols	12	10-12
Diesters of 4-methoxycinnamic acid	7	5-7
Free aliphatic acids	4	-
Free aromatic acids	1	-
Hydrocarbons (paraffins)	1	0.3-1
Free ω-hydroxycarboxylic acids	0.5	-
Triterpene diols	0.5	0.4
Aromatic compounds and/or resins	4.4	-
Free acids and other unknown constituents	-	5-7

2.3.1.4 Soy wax

Soy wax is produced from extracted oil from soybeans. The beans are washed, split, de-hulled, and then rolled into flakes after harvesting. The oil is then separated from the flakes and hydrogenated. Significant amounts of polyunsaturated fatty acids are present in the soybean oil after it has been synthesized, hydrogenated, and altered by the addition of polar groups. Normally, this natural wax is used to produce candles. It is a fantastic option for any candle maker due to its reliable performance, simplicity of use, and flexibility to various mixes and formulas. Another explanation for using soy wax in candles is that it makes a great scent carrier and does not need chemical amplifiers. Compared to paraffin wax, soy wax candles emit less soot, proven that is much cleaner and friendly to the environment. Figure 2.9 shows the making of candles by using soy wax.

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Figure 2.9 Soy wax production

2.3.1.5 Coconut wax

Coconut wax as shown in Figure 2.10 is another popular option in candle making. It was extracted from coconut oil to be refined and hydrogenated to be an additive. The hydrogenation process significantly raises the melting point from 24°C to 38°C while the refining step eliminates the coconut scent. The melting point of the coconut oil is then raised by blending it with additional natural waxes, usually soy wax, rapeseed, and beeswax, to ensure it solidifies at ambient temperature since it is in a liquid state at room temperature. Besides, coconut wax is odourless, colourless, and bright that easier to blend with colour pigments for producing interesting colours of candles.

Figure 2.10 Coconut wax

According to Fernandez P. A. (2021), coconut wax is a sustainable additive that brings many benefits compared to other natural waxes in candle making. It became favourable due to its slower burning rate that makes it last longer and has a greater aromatic scent compared to other waxes. In addition, coconut wax has a better burning quality which does not emit smoke and free of soot. Thus, the consumers can enjoy the scents without worrying about their health issue since it is non-toxic.

2.3.2 Colour pigment

Pigment is a substance that alters the colour of transmitted or reflected light due to wavelength-selective absorption. Pigments are mostly used for adding colour to materials, whether it is from textiles or paints. There are two types of pigments: organic pigments and inorganic pigments. The chemical composition of organic pigments is pretty simple, and the mineral and metals consist of named its colour. Washing, drying, powdering, and mixing into a formulation is the common process in organic pigments. Meanwhile, inorganic pigments are completely opposite to organic pigments. It is formulated in laboratories and provides inorganic pigment with a wide range of control over the oxidation process. This pigment is also known as synthetic pigment. Actually, pigments and dyes function differently; whereas dye can dissolve on its own, pigments require a binder to help them dissolve. This is due to the fact that the solubility of these two substances varies.

Derivation of Lithol Rubine (LR) to integrate a new pigment has been done by Abdelmaksoud W. M. A. et.al. (2022). Lithol Rubine (LR) was successfully utilised to react with salts of Cu (II) and Zn (II) ions in ethanolic solutions to generate a new colour pigment that is crucial to the inks industry and is used as special inks for printing. Naturally, Lithol Rubine comes with red colour. The combination of LR with Cu (II), LR-Cu, will form worm red colour while Zn (II), LR-Zn formed magenta red as in Figure 2.11.

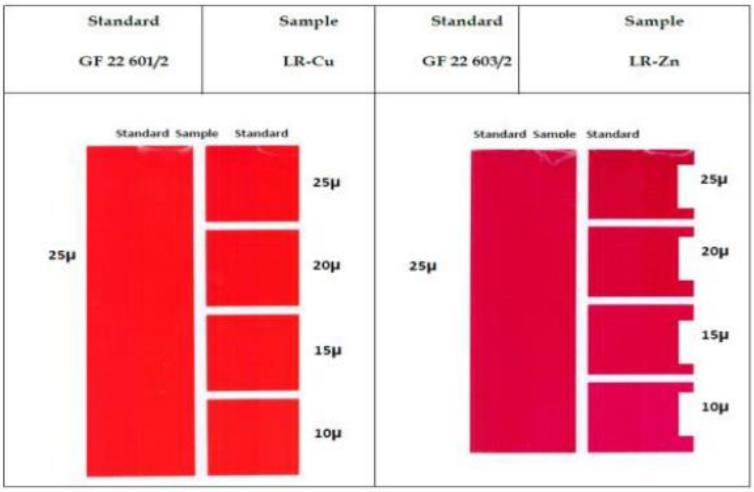

Figure 2.11 The inspection visual of LR-Cu and LR-Zn (Abdelmaksoud et.al., 2022).

2.3.3 Plastic

Apart from being used for drawing and colouring, crayons are also can be used as marking instruments since a long time ago. Based on prior findings, there is no clear statement on how to deliver good erasability along with intriguing characteristics, while also preventing the blooming of the crayons. Bloom is a phenomenon where the white-grey surface deposits hide the actual colour of crayons. This phenomenon makes the crayon seem unappealing aesthetically. In 1990, Brachman and Armand E. patented their findings about plastic crayons. Their findings are focused on several physical properties which are erasability, breaking strength, ease of application, sharpenability and the absence of bloom through 15 samples with different ratios of high-density polyethylene and stearic acid. Table 2.3 shows the result for the sample number 8 to number 15 with the scale from 1 to 10 where 10 is the best result. As a consequence of this test, it was discovered that a matrix mixture of 25% index intermediate melt (M.I. = 12) high-density polyethylene with 38% stearic acid produces the best qualities in high-density polyethylene and stearic acid. The increasing degree high-density polyethylene produces better erasability, increased tip-breaking strength, and increased barrel-breaking strength arise from. However, these benefits come at the cost of a minor reduction in ease the usability.

Table 2.3 The physical properties with combination of high-density polyethylene and stearic acid (Brachman and Armand E., 1990)

Sample	8	9	10	11	12	13	14	15
12 melt index high density polyethylene	20%	20%	25%	25%	25%	30%	30%	30%
Stearic acid	28%	38%	28%	38%	48%	28%	38%	48%
Erasability	8.3	8.4	9.2	8.8	8.8	9.0	9.8	9.8
Ease of application	8.0	8.4	7.2	8.5	7.1	6.7	7.2	5.6
Tip breaking strength	6.4	5.1	6.4	6.1	6.8	7.8	8.7	8.2
Barrel breaking strength	-	10.6	10.9	12.9	12.2	13.7	14.3	14.3
Sharpenability	10.0	10.0	10.0	10.0	10.0	10.0	10.0	10.0
Bloom	none	none	none	none	none	none	none	none

2.4 Waste cooking oil (WCO)

Waste cooking oil (WCO) is oil that has been used in cooking or frying processes and is no longer suitable for further use in food preparation. It is a byproduct of various cooking activities in households, restaurants, and food processing industries. The main content in vegetable oil is triacylglycerols (88–98%), are a crucial part of a balanced diet and make up around 15–20% of total calories consumed in the majority of developed countries (Awogbemi O. et al., 2021). Unfortunately, improper disposal of WCO negatively affects human and animal health, worsens environmental degradation, clogs drain, and contaminates land and marine environments. It has a high possibility to obstruct the oxygen for animals and plants when it covers the oxygen's path. In addition, cooking oil goes through a lot of physical and chemical changes when being fried. After extended heating, these

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modifications render the oil unsafe for human consumption (Panadare D.C. & Rathod V.K., 2015).

Rather than being disposed, WCO also can be reused to produce a beneficial product such as biodiesel, candles, soap, and crayons. However, the WCO needs to be treated before safely to be used. In this project, WCO is used as an additive to produce organic crayons. Transesterification processes are used to treat the WCO. However, the catalyst is not added in this process, thus, it cannot be claimed as a transesterification process.

2.4.1 Biodiesel

Biodiesel is a renewable fuel and is created from organic sources, typically from recycled cooking oil, animal fats, or vegetable oil. Biodiesel has many enticing traits such as high calorific value, low sulfur content, lubricating ability, and a high cetane number. In addition, it is sustainable and biodegradable (Rocha-Meneses L. et al., 2023). Normally, the process to purify the WCO is called transesterification process. The process of transesterification as in Figure 2.12, involves the reaction of a fat or oil that contains triglyceride with an alcohol to produce esters and glycerol. WCO is a feasible option to produce biodiesel (Awogbemi O. et al., 2021). At present, numerous studies investigated innovative catalysts for the transesterification of WCO into biodiesel and found that under moderate production circumstances, WCO may be transesterified into high-quality biodiesel.

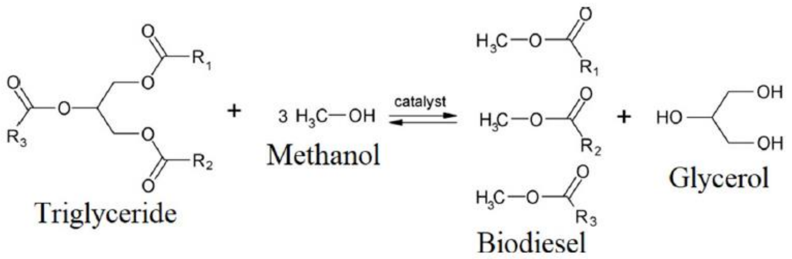

Figure 2.12 Transesterification reaction to produce biodiesel (Ahmed et al., 2023).

As time passed, biodiesel has been produced in supercritical conditions. This method is still using the transesterification process but did not use catalyst to speed up the reaction time. It substitutes the usage of alcohol to supercritical alcohol such as supercritical methanol. Even though it has a great advantage which can reduce capital costs, it also may bring a disadvantage which needs high energy consumption since it needs a very high temperature to let the reactions occur.

2.4.2 Cosmetics

Oil is a key component in the creation of cosmetics since it is mainly composed of triglycerides. Reusing of WCO can be repurposed and used in the production of cosmetics, particularly in skincare and haircare products. Naturally, oil has a silky texture and an easy-to-spread quality depending on its oil content, which ranges from 1% to 99%. In this era, many manufacturers of cosmetics add yeast to WCO to enable fermentation and the production of biodegradable surfactants. It is a natural by-product that manufacturers find to be quite appealing due to its being environmentally friendly.

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2.4.3 Candle

WCO also can be used to produce scented candles. The oil must be treated or filtered repeatedly to ensure that all the impurities have been removed before it is utilised to make the scented candles. The filtered oil is then mixed with the scented powder to eliminate the oil smell like fried chicken or fried fish. The scent and smell vary based on the natural fragrance used. For instance, combining blueberry-scented powder with WCO, any oily smell will be replaced by the blueberry aroma (Jocelyn Tan, 2020). Furthermore, the condition of the oil needs to be addressed because it is thick, has an unpleasant smell, and is dark in colour and makes the process more difficult (Azni, 2022). The condition of oil depends on the source and purpose of the oil before it becomes waste.

2.4.4 Soap

Animal fats and vegetable oils mostly consist of esters that contain glycerol and fatty acids (Azme et al., 2023). Generally, the reaction between vegetable or animal fat with lye in saponification process will create a solid cleaning agent known as soap. Figure 2.13 shows the chemical reaction in the saponification process. Other than candles, WCO also can be used as a raw material to create soaps that have been filtered, degummed, and decoloured (Li et al., 2020). However, the WCO treatment process for making soap requires the stages of filtering, washing, bleaching, and deodorization. Hydrogen peroxide, H_2O_2, was used throughout the bleaching process to make the oil's colour brighter.

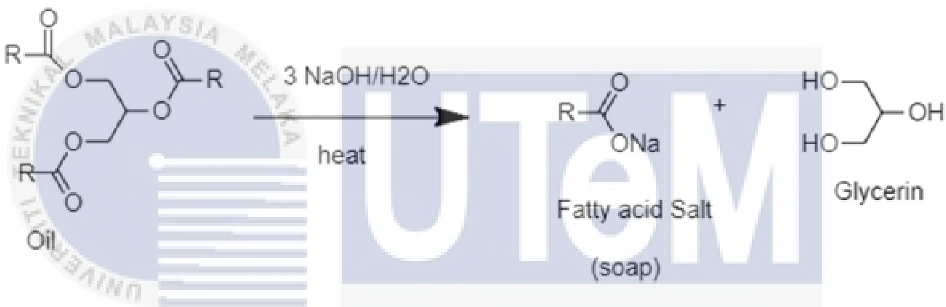

Figure 2.13 Saponification process in soap production (Azme et al., 2023).

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In the saponification process, a beaker containing around 40 g of treated WCO was weighed and heated on the heating plate. At the same time, 4 g of NaOH were added to the hot deionized water beaker. The NaOH solution and the treated oil were both heated up to 37°C. The WCO was then poured with a NaOH solution. Then, the mixture in which the fatty acid was converted into an alkali salt was constantly agitated until it reached a desirable thickness.

2.5 Toxic elements that may contain in crayons

Crayons are also considered children’s products often used by children below 12 years old because it is one of the easiest medium to use to showcase their creativity and imagination. However, the chemicals contained in the crayons might be exposed to the children in a number of ways because they spend a lot of time playing with it. The exposure scenario determines the exposure route and exposure intensity. Children under the age of three have a natural hand-to-mouth (HTM) behavior, which leads to oral exposure. The children's products that made of powder or pliable material such as crayons can be ingested by children HTM behavior (Kang et al., 2022). Children's product safety standards are a collection of federal safety regulations that apply to children's products, which also applies in manufacture of crayon.

A study done by Cui et al (2015) found that toxic metals like Pb, As, Cd, Cr, Ni and Sb may be present in high concentrations in crayon, which can cause major health issues. European Union introduced the EU Toy Safety Directive 2009/48/EC that sets a regulation for toys intended for children under 14 years of age to ensure they are safe to play with. According to the recently revised European Union Directive, the following heavy metal concentrations are acceptable in children's toys: Pb 2 mg/kg, As 3.8 mg/kg, Sb 45 mg/kg, Cd 1.3 mg/kg, Ni 75 mg/kg, and Cr 37.5 mg/kg. Meanwhile, Pb that contained in children's crayon has a range of concentration of 29-91mg/kg of sample. According to section 101 of the Consumer Product Safety Improvement Act of 2008 (CPSIA), all children's products made in the United State of imported there must adhere to CPSC regulations and have a total lead concentration in accessible portions of no more than 100 parts per million (ppm) and no more than 90 parts per million (ppm) in paint or any similar surface coating. On the other hand, As that was marked as human carcinogen by World Health Organization (WHO), the International Agency for Research on Cancer (IARC), and the Environmental Protection Agency (EPA) also been used in the crayon making. High concentrations of Cd are used to give pigment by creating brilliant yellow, orange, crimson and maroon colour in paints and

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coatings. Besides the water and food intake in daily lives, chewing and ingestion of non-edible objects are also the primary source of exposure to Cd.

2.5.1 Arsenic, As

Arsenic is a chemical element that is located in group 15 and period 4 in the periodic table. It is a naturally occurring metalloid that is prevalent in the environment, with an average worldwide concentration of around 10 mg/kg in natural soil. Arsenic is a deadly metalloid with mutagenic and carcinogenic characteristics. It exists in 2 forms, organic and inorganic. According to WHO, inorganic arsenic is found in soils, sediments and groundwater considered toxic and usually is associated with environmental contamination whereas organic arsenic is less toxic and can be found in marine organisms. Since children consume more food, air and water than adults do, they are more likely to be exposed to arsenic. They are also more likely to put their hands in their mouth. According to American Academy of Pediatrics, long term exposure of arsenic in early childhood gives negative impacts on cognitive development and caused fatality. Depending on the level of exposure, arsenic causes both acute and chronic diseases to humans. It is also known to cause a variety of illnesses, including melanosis, liver cirrhosis, muscular weakness, nausea, and vomiting (Sevak & Pushkar, 2023). According to a study by Mračević et al. (2022), the toxic elements of arsenic must not exceed 5.78 mg kg^{-1}.

2.5.2 Lead, Pb

Lead is a chemical element that is located in group 14 and period 6 in the periodic table. It is a hefty, solid metal that can be bent and stretched without breaking. Lead is a crucial toxic of heavy metal. It can be found in all parts of environment, even in daily anthropologic activities can cause to lead level increase such as emission from vehicle, smelting, electrical waste dismantling, etc (Zwolak, 2019). The digestive system is the major way that lead is exposed to people. Children are more vulnerable because they absorb more lead, up to 50% of ingested quantity compared to adults which is 10% (Rebelo A. et al., 2015). High lead concentrations can seriously harm the brain and kidneys and result in death depending on their exposure levels (Norkhadijah et al., 2017). Continuous exposure causes an amount of the lead that enters the body to not be completely removed, which causes it to attach to body tissues, particularly in the bone. Children frequently experience low amounts of lead exposure, which is especially pernicious due to the absence of physically observable symptoms that might make it difficult to detect its effects. Lead can have neurotoxic effects and long-lasting consequences on children's neurobehavioral functioning even at extremely low blood levels.

2.5.3 Chromium, Cr

Chromium is a chemical element that is located in group 6 and period 4 in the periodic table. Chromium is a transition metal that has a hard, brittle, and shiny property. It has a silvery grey tint and is extremely polishable. Chromium forms a thin oxide layer on its surface to prevent further oxidation when exposed to oxygen. It may exist in oxidation states ranging from 2 to +6, with +3 being the most stable (Hasan et al., 2023). Besides, chromium is antiferromagnetic at normal temperature in its elemental or uncombined state. Some researchers suggested that consumer goods must include fewer than 5 µg g^{-1} of nickel, chromium, and cobalt, or preferably less than 1 µg g^{-1} to reduce the risk for those who are extremely sensitive (Rebelo A. et al., 2015).

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2.5.4 Cadmium, Cd

Cadmium is a chemical element that is located in group 12 and period 5 in the periodic table. It usually can be found as bluish-white metal in zinc ores with soft and ductile properties. Nowadays, a significant amount of cadmium is manufactured from recovered nickel-cadmium batteries and zinc byproducts. In light of the fact that cadmium pollution is getting worse globally, cadmium has always been a significant contaminant in agricultural soil. Cadmium may rapidly develop in soil due to its difficulty in being degraded. Particularly in some confined, rain-washed crop planting settings, the soil's cadmium level frequently exceeds 0.3 mg kg^{-1} cap authorized by the agricultural industry standard (Cheng et al., 2023). However, it is considered acceptable since the maximum content is 0.615 mg kg^{-1} (Mračević et al., 2022). Besides, cadmium can be built up in the human body. The renal toxicity of cadmium in children, however, is less extensive. Children who are exposed to this cumulative element may experience long-term negative effects, especially in the neurological system, such as developmental delays and hyperactivity, even at extremely low levels (Rebelo A. et al., 2015).

2.5.5 Nickel, Ni

Nickel is a chemical element that is located in group 10 and period 4 in the periodic table. It is malleable, ductile and has great strength and corrosion resistance. Nickel has been used extensively in the manufacture of stainless steel, including cooking utensils, building materials and cutlery appliances, due to its low cost, high temperature resistance, corrosion and oxidation resistance. Due to its abundance in the Earth crust, humans are constantly exposed to Nickel. According to A. Rebelo et al. (2015), dermal exposure to even modest levels of nickel after sensitization might result in dermatitis outbreaks. Agency for Toxic Substances and Disease Registry (ATSDR) stated that about 10–20% of people, either by ingesting or skin contact, are sensitive to nickel and can experience cutaneous issues even at low concentrations. Nickel exposure can have a variety of negative health impacts on people, including the emergence of immunologic, neurologic, and reproductive disorders as well as

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carcinogenic effects (Mračević et al., 2022). Furthermore, nickel's maximum concentration is 25 mg kg^{-1} in playing materials (Kindi & Ali, 2020).

2.5.6 Antimony, Sb

Antimony is a chemical element that is located in group 15 and period 5 in the periodic table. Antimony naturally occurs in ores with arsenic and is linked to sulfur as stibnite. It is a silvery-white metal that is too fragile to be used by itself. Instead, it is frequently alloyed with lead and copper to make batteries, sheet and pipe metal. Besides, antimony sulfide and lead antimonite are the two primary forms of this element that have been used as pigments for ages. Occupational exposure to high levels of antimony can cause health issues, including respiratory problems, skin irritation and digestive disorder. But in long-term and chronic exposure may produce antimoniosis, cardiac issue, gastrointestinal, and lung disorders such pneumoconiosis and lung cancer (Mračević et al., 2022). This metal is considered hazardous since it can bring harm to human health. The maximum concentration of antimony must not exceed 45 mg kg^{-1}.

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CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter discusses the appropriate planning and methods utilised in this research to guarantee that all objectives listed are achievable. This section examined and described the method to purify the WCO using denatured alcohol from the best ratio of prior studies. Next, it will be utilised in organic crayons production before running the sample using XRF analysis. The procedure to run XRF analysis on the organic crayon has been discussed in this chapter.

3.2 Experimental method

The workflow or procedure of the project is shown in chronological sequence on the flow chart in Figure 3.1. The project started with a study on chemical substances that are probably contained in crayons to ensure they do not exceed the limitation. Next, WCO must be collected before it comes to purification process. The purification process used in this project is by adding denatured ethanol in order to obtain the ideal acid value. For this project, denatured ethanol was used to compare the acid value from prior study (Iqmal, 2023) that utilised food-grade ethanol for WCO treatment. This is because denatured ethanol is much cheaper compared to food-grade ethanol. If the acid value is significantly different from previous study, the process of purification is compulsory to be repeated. Then, the treated WCO will be used by mixing it with the hardening agent (beeswax and carnauba wax) and colour pigments. Finally, the sample of organic crayons will be analysed using XRF analysis.

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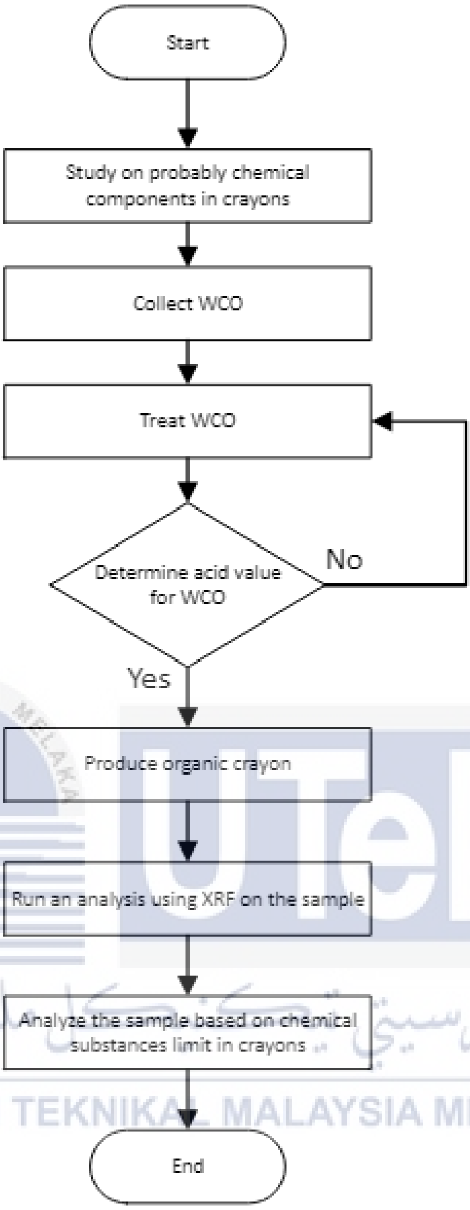

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Figure 3.1Flow chart

3.3 Waste cooking oil treatment

This procedure is necessary to guarantee that the WCO collected is safe to use. WCO and ethanol were used to perform the purification process in this project. The materials and apparatus for this process are listed in APPENDIX C. Initially, the excessive water in WCO must be removed. Fill the 200 g of WCO into the beaker glass and weigh it weight. Then, heat up the beaker at temperature between 110°C and 120°C on the hotplate stirrer, while stirring the WCO using magnetic stirring capsule. In a mean time, cover the top of beaker by using aluminium foil to prevent evaporation of WCO. Acid value is a crucial parameter to determine the best ratio of WCO and ethanol. Figure 3.2 shows the flow on determination of acid value.

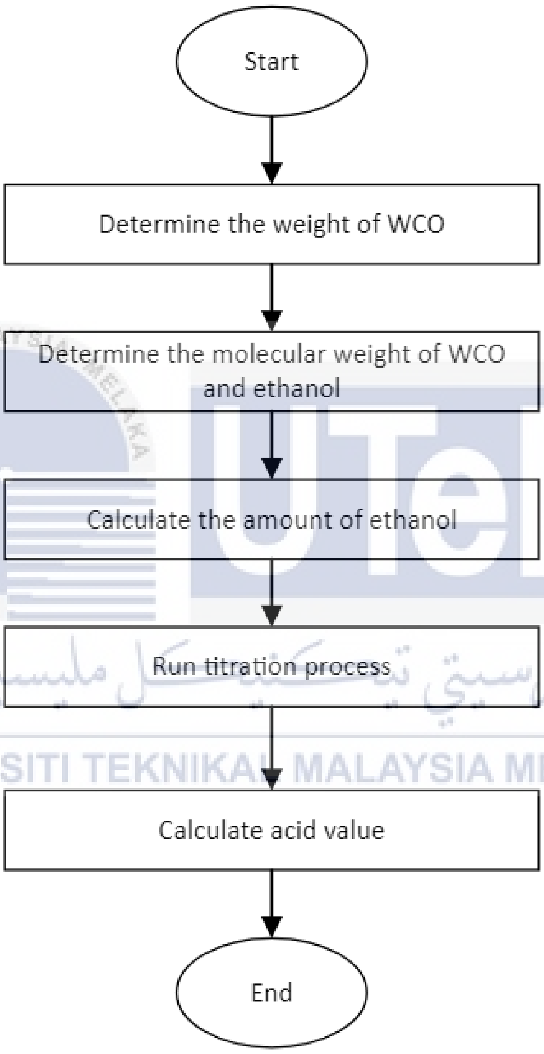

Figure 3.2 Flow chart on determining acid value

After removing the excess water in WCO, add an amount of ethanol into the beaker that contains WCO before heating it up at temperature between 60°C and 70°C and continue stir for 120 minutes. The required amount of ethanol is shown in Equation (3.1). Therefore, the molecular weight of ethanol and WCO must be determined first. Based on previous findings by Jeffri (2023), the 15:1 ratio is an ideal ratio where 15 represents the amount of ethanol and 1 represents the amount of WCO compared to other ratio based on stoichiometric ratio which consists 9:1, 12:1, 15:1, and 18:1. This is because this ratio (15:1) has the lowest acid value which is 2.08, and produced a brighter colour compared to others ratio. Table 3.1 shows the comparison of acid value for a various ratio from prior study. After 2 hours, the mixture is transferred in beaker into separating funnel and let it rest for 24 hours. After 24 hours, three layers of oil will form on the mixture which is fat, treated WCO and ethanol. The treated WCO was taken from the middle layer of oil and filtered using filter paper.

$$\text{Weight of ethanol} = \text{Ratio} \times \frac{\text{Molecular Weight of Ethanol}}{\text{Molecular Weight of WCO}} \tag{3.1}$$

Table 3.1 Result of acid value (Jeffri, 2023)

No.	**Ratio**	**Weight of WCO (g)**	**Weight of Ethanol (g)**	**Average Fatty Acid Value**
1.	9:1	200	101.2	2.50
2.	12:1	200	135.0	2.30
3.	15:1	200	168.8	2.08
4.	18:1	200	202.0	2.19

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3.4 Measuring acid value

In this process, acid value can be determined using treated WCO and KOH solution through the titration process. 25 ml of propanol and 4 drops of phenolphthalein added into the treated WCO that act as an indicator. Then, titrate KOH until the mixture changes to red colour. Equation 3.2 shows the calculation to determine acid value after titration process.

$$\text{Acid Value} = \frac{\text{Molar mass KOH} \times \text{Titration value} \times \text{Molarity KOH}}{\text{Weight of WCO}} \tag{3.2}$$

In this study, denatured alcohol is employed to compare the acid value results with those from previous studies (Iqmal, 2023) that utilised food-grade ethanol, specifically focusing on the optimal ratio of 15:1. The titration process is repeated 2 times to get a precise value. Figure 3.3 shows the process on the titration process to determine acid value.

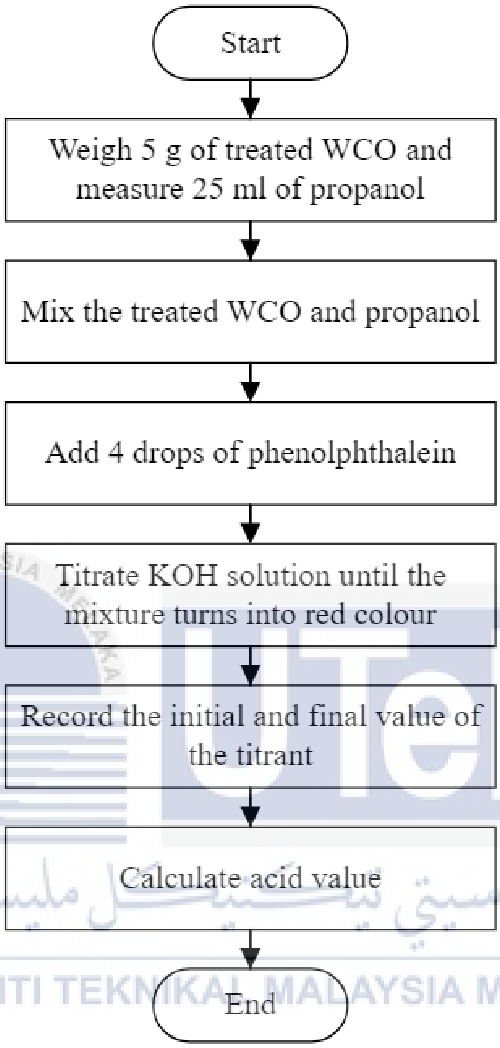

Figure 3.3 Titration process for determining acid value

3.5 Production of organic crayon

The organic crayons are produced using materials and apparatus listed in APPENDIX D. The main component for this process is treated WCO as an additive, carnauba wax and beeswax as hardening agent, and colour pigments. Firstly, measure 10 ml of treated WCO into the beaker. Then, weigh the carnauba wax and beeswax to ratio (7:3) in gram, respectively. The combination ratio of natural waxes also found by Jeffri (2023). Based on his study, this is a solid ratio since it has a faster time taken to harden the crayon which is

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657 second compared to others ratio. In addition, in his study, it was proved that this ratio (7:3) also has the longest time for the crayon to start melting which is 2 minutes 40 second. The melting test has been conducted by using water bath machine at constant temperature which is 65°C for black colour sample. After weighing, natural waxes are mixed into the treated WCO beaker. Heat up the mixture on the stirring hotplate while stirring it by using stirring rod until the wax is totally melted. After that, add the colour pigment powder in the beaker while continue stirring it. Finally, pour the mixture of treated WCO, natural waxes, and colour pigment powder into the mould and rest the mixture until it becomes hard. The analysis of XRF spectrometer was run using the sample from previous study conducted by Jeffri, (2023).

3.6 XRF analysis

The elemental composition of materials can be ascertained using the non-destructive analytical method known as XRF. Usually, this method is used to identify the chemical composition in the sample. In this project, XRF analysis were perform directly on two difference colour of sample which is dark (black) and bright (orange) by a high-performance Wavelength Dispersive XRF spectrometer from S8 Tiger Series 2 (Bruker) as shown in Figure 3.4 at Faculty of Science & Technology laboratory, Universiti Sains Islam Malaysia (USIM). In addition, the XRF spectrometer equipped with Lithium Fluoride (LiF) 200 crystals which have a high reflectivity and high sensitivity followed with Rhodium tube operated at 75 μA and 50 kV.

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Figure 3.4 XRF spectrometer

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3.6.1 Procedure on XRF analysis

Before starting the analysis, sample preparation is an important step to run this analysis because the quality of sample preparation determines the accuracy of the results, and improper sample preparation can result in completely incorrect results. The sample of organic crayons should be in a powder or solid form before being placed into the sample cup. However, the properties of samples cannot be crushed into powder form and need to be used in solid. Figure 3.5 shows the state of sample when being crushed.

Figure 3.5 The state of samples when being crushed

Next, the outer surface of the samples needs to be removed to eliminate any surface contaminants from the sample. Then, the samples are placed into the sample cup followed by thin film. The use of thin film is to increase the elemental analysis's accuracy by ensuring that x-rays enter the sample uniformly. Figure 3.6 shows the sample preparation ready to be analysed.

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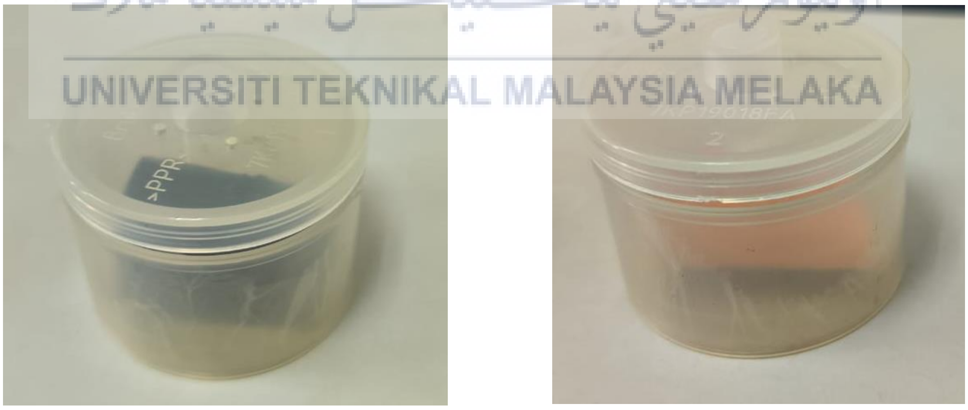

(a) (b)

Figure 3.6 The complete preparation of the samples (a) dark sample; (b) bright sample

After that, the samples are placed in the XRF spectrometer as shown in Figure 3.7 according to the coordinate which are dark sample in A1 and bright sample in A2 where the alphabet and number represent the row and column, respectively. To start the analysis, the start button was pressed which was located on the operator's control panel screen. The analysis run separately started with dark sample. A controlled x-ray tube emits high intensity x-rays at the sample. A dislodged electron from one of the atom's inner orbital shells occurs when an atom in the sample is impacted by an x-ray with sufficient energy. When an electron from one of the atom's higher energy orbital shells fills the hole left in the inner orbital shell, the atom regains its stability. Each sample takes about 20 minutes to completely analyse. After 20 minutes, the result of XRF appeared on operator's control panel screen. The expected element that may be present in the samples should be safe for human and environmental since the full production of crayon is using natural sources except WCO. If there is any heavy metal detected, it should be in a small quantity.

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Figure 3.7 The samples in XRF spectrometer

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

The results in this study are discussed in this chapter. There are two main studies which are the titration of WCO with KOH solution and the analysis of the organic crayon using XRF spectrometer to determine the elemental composition with the use of carnauba wax and beeswax and treated WCO as an additive.

4.2 Acid value

In this analysis, the treated WCO is combined with KOH through titration process to measure the acid value. The ratio used in this analysis is 15:1, where 15 represents the amount of ethanol and 1 represents the amount of WCO which is the best ratio based on previous study (Iqmal, 2023). The weight of ethanol can be determined using Equation 3.1, where molecular weight of ethanol and WCO is 46.07 g/mol and 872.36 g/mol, respectively. From the calculation, the weight of ethanol obtained is 158.43 g.

$$\text{Weight of ethanol} = \frac{15}{1} \times \frac{46.07\ \text{g/mol}}{872.36\ \text{g/mol}} = 0.79$$

$$\text{Weight of ethanol} = 0.79\ \times 200\ \text{g} = 158.43\ \text{g}$$

From the data acquired, the acid value can be determined through titration process using KOH until the mixture turned into red colour permanently. Table 4.1 shows the titration value of mixture treated WCO and denatured ethanol.

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Table 4.1 Titration value of treated WCO

No.	**Ratio**	**KOH volume (ml)**		**Final - initial**
		Initial	**Final**	
1.	15:1	26.3	32.6	6.3
2.		32.8	39.5	6.7

The result of the titrant can be used to determine acid value in the treated WCO. Using Equation 3.2, the acid value can be ascertained for both attempts to get the average. Acid $Value_1$ represents the first attempt of titration while Acid $Value_2$ represents the second attempt. The normality of KOH solution is 0.1. The average acid value of this ratio is 7.30, considerably high since the value from prior study is 2.08. This result shows the use of food-grade ethanol is better compared to denatured ethanol from acidity aspect.

$$\text{Acid Value}_1 = \frac{56.1 \times 6.3 \times 0.1}{5} = 7.07$$

$$\text{Acid Value}_2 = \frac{56.1 \times 6.7 \times 0.1}{5} = 7.52$$

$$\text{Average Acid Value} = \frac{7.07 + 7.52}{2} = 7.30$$

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The acid value obtained can be compared from prior study which use food-grade ethanol that guaranteed much safer than denatured ethanol. Table 4.2 shows the comparison of acid value between food-grade and denatured ethanol. The weight of denatured ethanol used is higher than food-grade ethanol due to different calculation ratios. This is because, molar mass of WCO for this project is higher that may be caused by the usage of WCO. However, the range between these two acid values is quite big. This proved that the use of denatured ethanol is unfit to be used in WCO treatment. The denatured ethanol itself contains toxic and bad odour that is unsuitable for human consumption. Moreover, consumption of denatured ethanol can lead to serious health issue such as blindness and death.

Table 4.2 Comparison of Acid Value between food-grade and denatured ethanol

Type of ethanol	**Esterification Ratio**	**Weight of WCO (g)**	**Weight of ethanol (g)**	**Average Acid Value**
Food-grade ethanol (Iqmal, 2023)	15:1	200	168.8	2.08
Denatured ethanol			158.43	7.30

4.3 XRF result

In this analysis, there are two different colour of sample which is dark (black) and bright (orange). Fortunately, there are no heavy metals such as Pb, Cr, As, Sb, Cd and Ni detected that have potential to harm the users or toddlers for both samples. Each sample has a different elemental composition presented. This event occurs due to different sources of natural colour pigment. The unit used to indicate the element for this analysis is kilocounts per second, %. The result of each sample can be referred to Appendix E and F.

4.3.1 Dark sample

The natural pigment used to provide a dark colour (black) for this sample is extracted from bamboo charcoal. For dark sample, the element composition present are potassium (K), iron (Fe), silicon (S), zinc (Zn), chlorine (Cl), rubidium (Rb), copper (Cu), aluminium (Al), sulfur (S), manganese (Mn), niobium (Nb) and phosphorus (P) with the intensity of 31.2%, 9.54%, 7.11 %, 5.37%, 5.29%, 3.68%, 2.30%, 2.18%, 1.97%, 1.66%, 1.1 % and 0.986%, respectively. The impurity content is about 27.604%, which mainly includes oxygen that has been oxidized. Table 4.3 shows the elemental composition of dark sample using XRF spectrometer.

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Table 4.3 Elemental composition of dark sample

Dark sample, elemental composition, %												
K	Fe	S	Zn	Cl	Rb	Cu	Al	S	Mn	Nb	P	Other
31.2	9.54	7.11	5.37	5.29	3.68	2.30	2.18	1.97	1.66	1.1	0.986	27.604

Potassium is detected as the highest composition percentage because the source of this pigment enriched with potassium content (Samadhi et al., 2018). It is commonly found in food especially, fruits and plants. Even though the composition of potassium is at peak, there is no significant evidence to prove that consuming potassium in low amounts can bring a negative impact to human health. Moreover, potassium is essential for human since it comes with minerals to appropriate bodily cell function, including cardiac muscle cells. However, excessive intake of potassium can lead to hyperkalemia (levels above 5.5 mmol/L), which can potentially result in kidney failure. This is because the kidneys remove excess potassium. Consequently, an accumulation of this mineral in the blood may be caused by impaired kidney function.

Iron is detected as the second highest composition percentage in dark sample. The correlation between iron and bamboo charcoal is prominent in the chemical industry. The combination of these two or known as Ferum-bamboo charcoal based (Fe-BC), act as a great adsorbent to remove pollutant, pigments, and flocculants (Wang et al., 2011). For human health side, the body requires iron as a mineral for growth and development. The body uses iron to produce myoglobin, a protein that gives muscles oxygen, and hemoglobin, a protein found in red blood cells that transports oxygen from the lungs to every part of the body. The maximum daily intake that is unlikely to have a negative impact on health is known as the tolerable upper intake level (UL). The recommended daily allowance for iron is 45 mg for all males and females aged 14 years and older and 40 mg applies to younger ages. Therefore, iron is not harmful for human health since it provides minerals for human growth but

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consuming too much of iron can lead to severe health risk such as inflammation of stomach lining and ulcers.

Silicon is a metalloid metal that is located at Group 14 in periodic table. Silicon is the third most abundant trace element in the body. It is essential for the health of the connective tissues, bones, cartilage, tendons, and joints as well as for the synthesis of collagen and elastin. However, due to its strong affinity for oxygen, this crystalline metalloid is nearly always found as silicon dioxide/silica. Up to this point, consumption of silicon supplements has no known adverse effects. The UL intake for silicon is 50 mg daily. Kidney damage can arise from prolonged inhalation of silica, particularly when it is in the crystalline form, which is known to induce silicosis, a lung disease.

For Zn, Cl, Rb, Cu, Al, S, Mn, Nb and P, there is a small amount of these elements detected. These elements did not bring a significant impact to harm humans and the environment. Thus, these elements can be neglected.

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4.3.2 Bright sample

The natural pigment used to provide a bright colour for this sample is extracted from carrot. For bright sample, the element composition present are titanium (Ti), tin (Sn), iron (Fe), aluminium (Al), potassium (K), zinc (Zn), chlorine (Cl), ruthenium (Ru), rubidium (Rb), sulfur(S) with the intensity of 35.1 %, 7.76 %, 6.30 %, 4.55 %, 4.54 %, 2.65 %, 2.14 %, 1.16 %, 0.609 % and 0.484 %, respectively. The impurity content is about 34.707%. Table 4.4 shows the elemental composition of bright sample using XRF spectrometer.

Table 4.4 Elemental compostion of bright sample

Bright sample, elemental composition, %										
Ti	Sn	Fe	Al	K	Zn	Cl	Ru	Rb	S	Other
35.1	7.76	6.30	4.55	4.54	2.65	2.14	1.16	0.609	0.484	34.707

Titanium is detected as the highest composition percentage in bright sample. This is because the main ingredient of this pigment is titanium dioxide (TiO_2) which are widely used in cosmetics and pigment because of its more desirable characteristics, which include a higher refractive index, greater optical qualities, a higher covering power, a higher dispersion, a higher brightness, and greater chemical inertness (Tian, 2020). Naturally, TiO_2 comes in pure white colour but the combination of TiO_2 and other pigments can produce a variety of colours. In this case, TiO_2 is combined with extracted carrot which has an abundance of β-carotene that gives orange to yellow colour to impart natural orange pigment (Nabi et al., 2023). According to FDA, TiO_2 is safe for use in cosmetics and foods. The FDA outlines a guideline on the usage of TiO_2 in food which is no more than 1%.

Tin is detected as the second highest composition percentage in bright sample. Tin is a metal that widely used for catalyst and coating due to its properties which is non-corrosive and non-toxic. It can exist as an isolated pure compound or as a constituent of other organic or inorganic compounds. Tin can combine with other elements like oxygen, sulfur, fluoride, or chloride to form inorganic tin compounds. Tin binds to carbon to form organic tin compounds. In the faeces, inorganic tin salts are quickly eliminated and poorly absorbed. It indicates that tin has low toxicity. At this point, no evidence of mutagenicity, teratogenicity, or carcinogenicity has been found in experimental studies. However, according to WHO 1973 permissible limit for tin should be below 250 mg kg^{-1} for any tinned food.

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Aluminium is a metal that is commonly used in food additives for raising agent, stabiliser, firming agent and anticaking agent to enhance their flavour and texture. Basically, vegetation can absorb aluminium that is naturally present in the soil and water since it is the 3rd most abundant element in earth’s crust. For human health, aluminum's relatively low bioavailability led to its not being regarded as harmful to human health (Stahl et al., 2011). According to latest studies, the WHO suggests that Provisional Tolerable Weekly Intake

(PTWI) for aluminium is 2 mg/kg bw in foods. At this point, there are no significant evidence of extreme aluminium exposure can lead to any serious health issue since this element is an essential part of a balanced diet to human.

For K, Zn, Cl, Ru, Rb, and S, there is a small amount of these elements detected. These elements did not bring a significant impact to harm humans and the environment. Thus, these elements can be neglected.

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CHAPTER 5

CONCLUSION

5.1 Conclusion

This chapter serves as a wrap-up for the project or overall research and offers suggestions regarding ways to improve product development. The purpose of this project needs to be examined in the final research after all the steps have been completed to ensure that all the objectives have been achieved.

The first objective is to compare the acid value for treated WCO using denatured ethanol instead of food-grade ethanol from prior study. From this study, even though denatured ethanol served low cost compared to food-grade ethanol, it is shown that denatured ethanol is not worthy to be use for WCO treatment. The acid value of this alcohol is too high compared to food-grade ethanol with the value 7.30 and 2.08, respectively.

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The second objective is to conduct chemical testing using XRF spectrometer to identify any toxic element and compare to ASTM D4236. From the data obtained, there is no heavy metal detected in the crayons. The dark sample exhibits the highest concentration of potassium, derived from the charcoal bamboo pigment, which is enriched with potassium content. Meanwhile, titanium is the highest concentration from bright sample that comes from TiO_2 that widely used in pigmentation for white colour. Moreover, the elemental composition present in the samples is commonly found in dietary and did not impair human health. ASTM D4236 outlines standard practice for labeling art materials for chronic health hazards. In this study, the XRF analysis revealed the absence of any concerning heavy metals, such as As, Pb, Cr, Cd, Ni, and Sb, in the crayons. Moreover, the elements present in the crayon are not harmful and do not pose health risks. Hence, this study adheres to the regulatory standards outlined in ASTM D4236.

5.2 Recommendation

In the future, the characterisation of organic crayons could be improved by including the following concern:

i. Conduct the XRF analysis on the other colour of samples to elucidate a comprehensive composition profile for crayons.

ii. Conduct another chemical test such as Scanning Electron Microscopy (SEM) or X-Ray Diffraction (XRD) analysis to compare the compositional elements in the crayons.

iii. Identify another parameter and method for crayon testing such as pH levels.

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APPENDICES

APPENDIX A Gantt Chart PSM 1

Gantt Chart PSM 1																	
No.	Activity	Plan/ Actual	Week														
			1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Registration of PSM title	Plan														
		Actual														
2	Briefing on project title by supervisor	Plan														
		Actual														
3	Briefing on Chapter 1	Plan														
		Actual														
4	Writing Chapter 1	Plan														
		Actual														
5	Sunbission of Chapter 1	Plan														
		Actual														
6	Drafting and writing on Chapter 2	Plan														
		Actual														
7	Present the draft on Chapter 2	Plan														
		Actual														
8	Submission on Chapter 2	Plan														
		Actual														
9	Drafting and writing on Chapter 3	Plan														
		Actual														
10	Submission on Chapter 3	Plan														
		Actual														
11	Writing Chapter 4	Plan														
		Actual														
12	Submission of draft report	Plan														
		Actual														
13	Last correction for the report	Plan														
		Actual														
14	Submission of final report of PSM 1	Plan														
		Actual														
15	Presentation and slides preparation	Plan														
		Actual														

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APPENDIX B Gantt Chart PSM 2

Gantt Chart PSM 2																	
No.	Activity	Plan/ Actual	Week														
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Correction for PSM 1	Plan															
		Actual															
2	Treat WCO	Plan															
		Actual															
3	Find acid value	Plan															
		Actual															
4	Produce organic crayon	Plan															
		Actual															
5	Run analysis on sample	Plan															
		Actual															
6	Completing Chapter 3	Plan															
		Actual															
7	Research on element present	Plan															
		Actual															
8	Completing Chapter 4	Plan															
		Actual															
9	Completing Chapter 5	Plan															
		Actual															
10	Submission of draft report	Plan															
		Actual															
11	Submission of final report of PSM 2	Plan															
		Actual															
12	Submission for corrected report	Plan															
		Actual															
13	Presentation and poster preparation	Plan															
		Actual															

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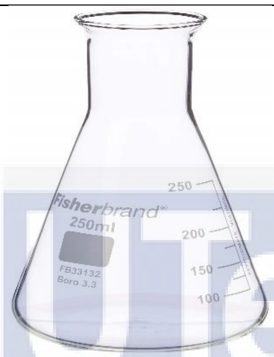
APPENDIX C Material and Apparatus For WCO treatment

No.	Apparatus's name	Apparatus	Description
1.	Beaker Glass 250 ml, 500 ml, 1000 ml.		To fill the mixture of WCO and ethanol
2.	Conical Flask 250 ml		To store the treated WCO while filtered
3.	Stirring Hotplate		Use to heat up and stir the mixture
4.	Digital Scale Weight		To measure the weight of WCO and ethanol

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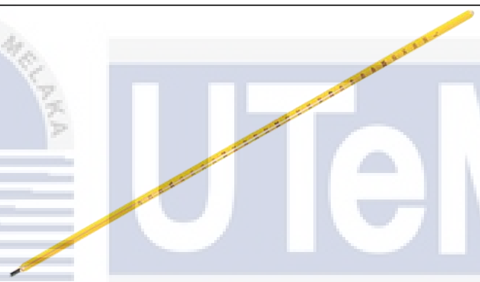
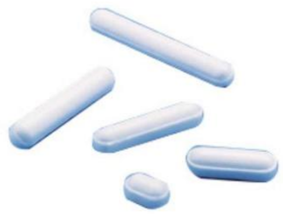
5.	Separating Funnel		To store the mixture of WCO after purification process
6.	Filter Funnel		Used to filter the treated WCO into conical flask
7.	Thermometer		To measure the temperature when heat the mixture
8.	Filter Paper		To filter impurities of WCO
9.	Magnetic Stirring Capsule		To stir the mixture in the beaker on the stirring hotplate

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10.	Aluminium Foil		To cover the upside of beaker when heating process

No.	Material's name	Material	Description
1.	Waste cooking oil		The treated of WCO can be an additive in organic crayon production
2.	Ethanol		Use to treat the WCO in purifiying process

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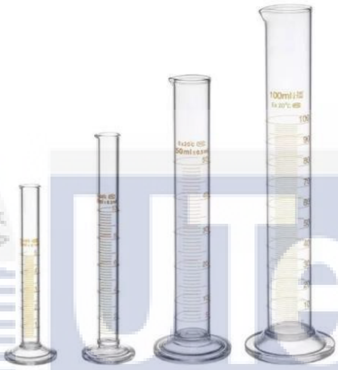
APPENDIX D Material and Apparatus Used in Crayon Production

No.	Apparatus's name	Apparatus	Description
1.	Beaker Glass 250 ml		To fill the mixture of treated WCO, and waxes for heating process
2.	Measuring Cylinder		To measure the volume of treated WCO before mix with waxes
3.	Digital Scale Weight		To measure the weight of beeswax and carnauba wax
4.	Stirring Hotplate		To heat the mixture of treated WCO and waxes

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5.	Stirring Rod Glass		To stir the mixture
6.	Crayon Mould		To let the mixture harden for produce crayons

No.	Material's name	Material	Description
1.	Treated WCO		Used as an additive to produce crayons
2.	Beeswax		Act as hardening agent in production of crayons

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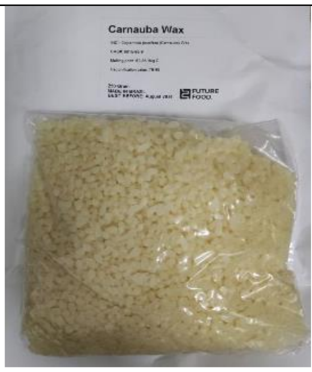
3.	Carnauba wax	 	Act as hardening agent in production of crayons
4.	Colour pigment powder		To add the colour on the crayons

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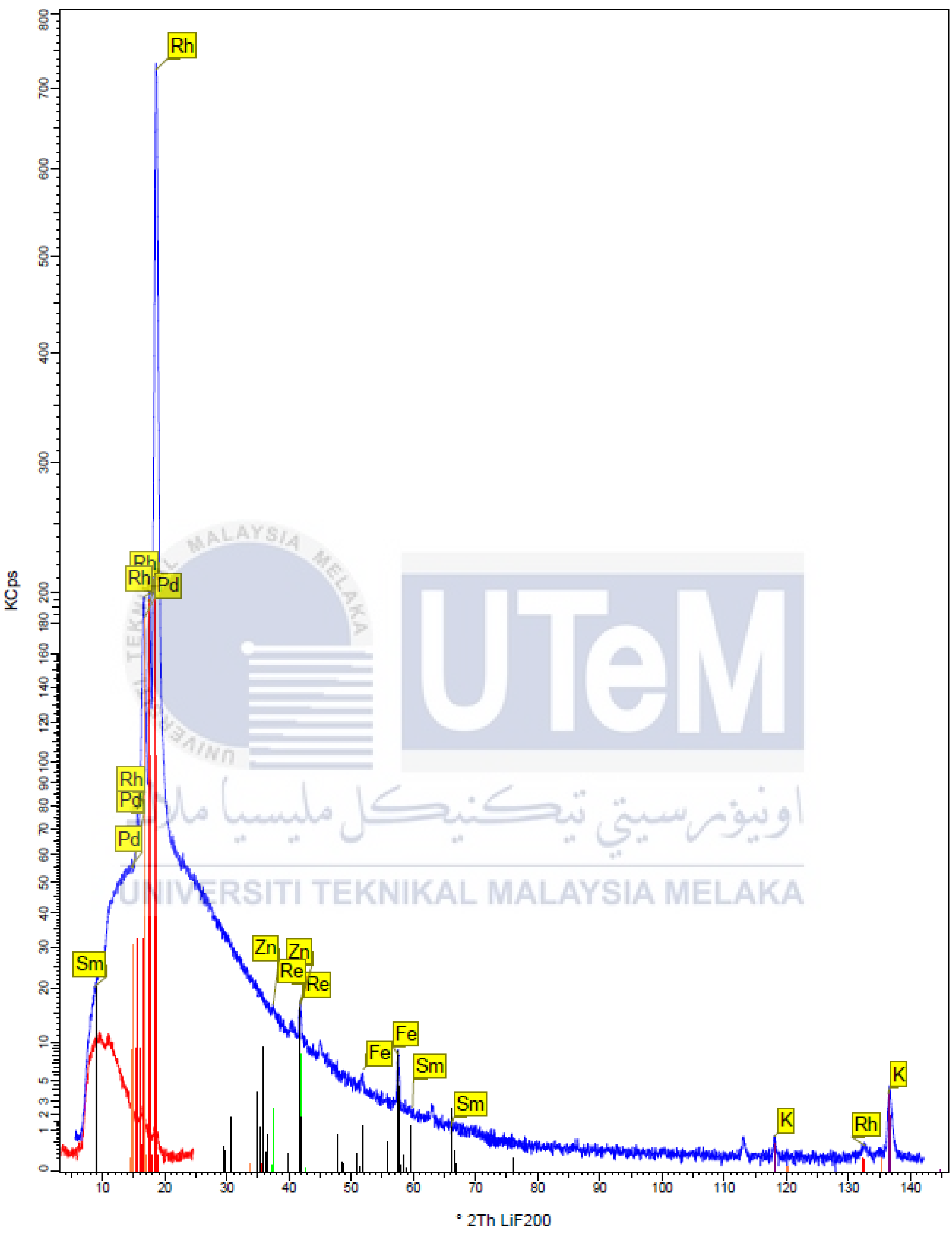
APPENDIX E Graph of dark sample



APPENDIX F Graph of bright sample