EFFECT OF SECONDARY AMINE OXIDATION ON DIELECTRIC PROPERTY OF EPOXY COATING DEPOSITED BY ELECTROPHORETIC DEPOSITION

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DEDICATION

I dedicate this project to God Almighty my creator, my strong pillar, my source of inspiration, wisdom, knowledge and understanding. He has been my source of strength throughout this programme, and I have only ever flown on His wings.

Not forget to mention my beloved parents and siblings who encouraged me every step of the way and who guided me and ensured that I gave it everything I had to accomplish what I had started.

ABSTRACT

With the goal to ensure corrosion resistance and longevity, this study explores the effects of different secondary amine concentrations on the thickness of epoxy coatings applied using electrophoretic deposition (EPD). The goal of the study is to understand the connection between coating qualities and secondary amine concentration, filling in a major knowledge gap. It involves regulating various parameters such as suspension concentration, applied voltage, deposition time, electrophoretic mobility, and others to better understand the behavior of these components during the coating process. Importantly, the change in secondary amine concentration aims to explore how different materials interact and can be applied at varying thicknesses on desired conductors. The mixing of amine and epoxy resin, followed by controlled heating and pH adjustment using formic acid, is a crucial part of the process. However, it's also noteworthy to consider the potential impact of secondary amine oxidation during EPD. Secondary amines can undergo oxidation, which might affect epoxy behavior during the deposition process, potentially influencing the properties of the resulting coatings. The main objective is to use electrochemical impedance spectroscopy (EIS) to assess dielectric characteristics and optimise EPD settings to produce a better coating microstructure. Preparing the substrate, creating the epoxy solution, processing the EPD with care, curing, and using sophisticated characterization methods such as FTIR, SEM, and EIS are all part of the extensive process. Moreover, the optimal dielectric constant for a 1.5 ml secondary amine concentration and a 40-volt EPD voltage is determined via EIS analysis. Interestingly, these results highlight the usefulness of the reported characteristics in many contexts, such as ambient and nitrogen conditions, providing important information for developing epoxy coatings customised for certain uses.

ABSTRAK

Dengan matlamat untuk memastikan rintangan kakisan dan umur panjang, kajian ini meneroka kesan kepekatan amina sekunder yang berbeza pada ketebalan salutan epoksi yang digunakan menggunakan pemendapan elektroforesis (EPD). Matlamat kajian adalah untuk memahami hubungan antara kualiti salutan dan kepekatan amina sekunder, mengisi jurang pengetahuan utama. Ia melibatkan mengawal selia pelbagai parameter seperti kepekatan ampaian, voltan terpakai, masa pemendapan, mobiliti elektroforetik, dan lain-lain untuk lebih memahami kelakuan komponen ini semasa proses salutan. Yang penting, perubahan dalam kepekatan amina sekunder bertujuan untuk meneroka bagaimana bahan yang berbeza berinteraksi dan boleh digunakan pada ketebalan yang berbeza-beza pada konduktor yang dikehendaki. Pencampuran resin amina dan epoksi, diikuti dengan pemanasan terkawal dan pelarasan pH menggunakan asid formik, adalah bahagian penting dalam proses tersebut. Walau bagaimanapun, adalah wajar untuk mempertimbangkan kesan potensi pengoksidaan amina sekunder semasa EPD. Amina sekunder boleh mengalami pengoksidaan, yang mungkin menjejaskan tingkah laku epoksi semasa proses pemendapan, yang berpotensi mempengaruhi sifat salutan yang terhasil. Objektif utama adalah untuk menggunakan spektroskopi impedans elektrokimia (EIS) untuk menilai ciri dielektrik dan mengoptimumkan tetapan EPD untuk menghasilkan struktur mikro salutan yang lebih baik. Menyediakan substrat, mencipta penyelesaian epoksi, memproses EPD dengan berhati-hati, pengawetan dan menggunakan kaedah pencirian yang canggih seperti FTIR, SEM dan EIS adalah sebahagian daripada proses yang meluas. Selain itu, pemalar dielektrik optimum untuk kepekatan amina sekunder 1.5 ml dan voltan EPD 40 volt ditentukan melalui analisis EIS. Menariknya, keputusan ini menyerlahkan kegunaan ciri yang dilaporkan dalam banyak konteks, seperti keadaan ambien dan nitrogen, memberikan maklumat penting untuk membangunkan salutan epoksi yang disesuaikan untuk kegunaan tertentu.

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LIST OF SYMBOLS AND ABBREVIATIONS

EPD	Electrophoretic Deposition
FTIR	Fourier-Transform Infrared Spectroscopy
EIS	Electrochemical Impedance Spectroscopy
SEM	Scanning Electron Microscope
h	Hours
V	Voltage

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CHAPTER 1

INTRODUCTION

1.1 Background

Corrosion is a major problem for the many metal materials that are utilized in daily life and industrial production (Jamwal et al. 2020). Every year, about one-third of the world's metal materials are predicted to corrode beyond repair. The causes of metal corrosion are various. Among these, the corrosion caused by microorganisms may be found in various industrial production domains (Lang et al. 2021). This problem could be solved by using epoxy coating that widely industries used it as a troubleshooter.

Epoxy coatings are widely used in many industries as a solution to prevent corrosion. Epoxy coatings provide a protective layer over the metal surface, preventing contact with the surrounding environment and thus preventing corrosion (Song et al. 2021). However, the success of the epoxy coating process depends on several factors, such as surface preparation, application technique, curing time, and environmental conditions.

Proper surface preparation is critical to the success of the epoxy coating process. The metal surface must be cleaned thoroughly to remove any dirt, grease, or other contaminants that may interfere with the adhesion of the epoxy coating (Tian et al. 2022). The surface may need to be sanded or blasted to create a roughened surface that allows for better adhesion.

The capacity to accomplish precise and controlled deposition on intricate forms and surfaces is a benefit of electrophoretic deposition (EPD) as a targeted coating method. EPD makes it possible to cover substrates uniformly and conformally, even irregular and three-dimensional objects, which is difficult to do with conventional coating techniques. EPD is a very effective and specialised coating method that works by creating an electric field to allow charged particles or colloids to migrate and deposit onto the target surface. The EPD's targeted coating capabilities minimises material waste, speeds up the manufacturing process, and improves the overall quality and functionality of the coated goods. The application technique also plays a significant role in the success of the epoxy coating process. The epoxy coating must be applied uniformly, and the thickness of the coating must be consistent. The coating must be applied in a dry environment, and the temperature and humidity must be within the recommended range (Thakur et al. 2019).

To get the best coating performance and quality, EPD suspension preparation for epoxy coating has to be thoroughly studied. The stability, homogeneity, and dispersion of the particles or colloids in the suspension are guaranteed by proper suspension preparation, and this directly affects the deposition and film formation during EPD. To create a homogeneous and defect-free coating, variables such particle size, concentration, surface charge, dispersant selection, and solvent choice need to be carefully studied and optimised. Controlling layer thickness, adhesion, porosity, and surface roughness requires an understanding of the interactions between suspension components and how these interactions affect coating characteristics. By investigating and optimizing the EPD suspension preparation, researchers and engineers can enhance the coating process efficiency, coating adhesion, corrosion resistance, and overall performance of epoxy coatings, leading to improved durability and functionality in various industrial applications.

Curing time is also essential to the success of the epoxy coating process. The epoxy coating must be allowed to cure completely before the metal is put into service. The curing time depends on the type of epoxy coating used, the temperature, and the humidity (Michel and Ferrier 2020).

Humidity and temperature are important factors that influence the curing process. Higher temperatures often shorten the curing period whereas lower temperatures lengthen it. Similar to low humidity, high humidity has the potential to speed up the curing process. It's crucial to adhere to the manufacturer's instructions on the ideal curing circumstances for the particular epoxy coating being applied (Ndukwu et al. 2020).

For numerous reasons, it is crucial to properly enable the epoxy coating to dry completely before using the metal. First off, if the coating is used too soon, it may not adhere well, be as durable, or have impaired protective qualities (Fattah-alhosseini, Chaharmahali, and Babaei 2022). When the coating is not completely dry, it could not create a solid connection with the substrate, reducing durability and raising the possibility of coating breakdown over time. In turn, the coating's ability to protect the metal against corrosion, abrasion, and other types of damage may be compromised.

Additionally, if the epoxy coating doesn't dry completely, it may be more vulnerable to physical harm. It's possible that the coating hasn't fully developed its hardness and toughness, rendering it vulnerable to chipping, cracking, or delamination. The integrity of the coating may be jeopardised by these flaws, exposing the underlying metal to hazardous environmental elements and decreasing the coating's overall efficiency as a barrier of defence (Farh, Ben Seghier, and Zayed 2023).

In conclusion, epoxy coatings are an effective solution to prevent corrosion caused by microorganisms. However, to ensure the success of the epoxy coating process, proper surface preparation, application technique, curing time, and environmental conditions must be considered.

1.2 Problem Statement

Electrophoretic deposition (EPD) is a process that involves depositing a charged material onto a conductive surface using an electric field. In the case of epoxy coatings, the EPD process involves preparing an epoxy suspension that contains both the epoxy resin and secondary amine as curing agent. The epoxy suspension is prepared, it is then placed into a container that serves as the anode or cathode in the EPD process (Aghili et al. 2021a). The container is filled with an electrolyte solution, which is typically a salt solution that allows the electric field to be established.

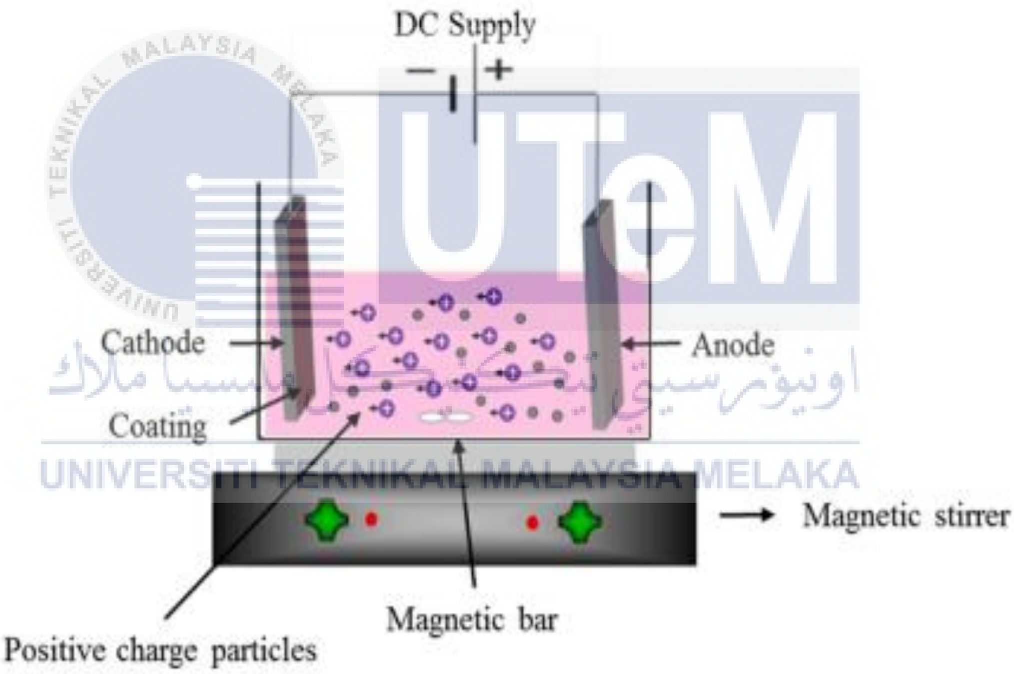

Figure 1.2 Schematic representation EPD process(Aghili et al. 2021b)

The thickness of the epoxy coating required for an electrical isolation application depends on various factors, including the type of metal, the corrosive environment, and the expected service life of the coated material (Ahmad et al. 2023). The concentration of secondary amine as curing agent in the epoxy suspension can affect the properties of the coating, including its thickness. Optimization of amine concentration is needed. When amine concentration is too low, degree of curing may be reduced(Nadagouda et al. 2022). Whereas, too high amine concentration may result in thinner coating due poor surface tension which in low wettability (Su et al. 2019).

The effect of varying concentrations of secondary amines in epoxy suspensions on the resulting epoxy thickness is the primary focus of this study. The investigation aims to discern how different concentrations of secondary amines influence the final thickness of cured coatings. It's imperative to acknowledge that various factors, including the type of epoxy resin utilized, curing temperature, and duration, could impact how alterations in secondary amine concentration affect epoxy thickness. Therefore, to ensure the reliability and accuracy of outcomes, comprehensive research is essential, meticulously monitoring these variables.

The relationship between secondary amine concentration and epoxy thickness can be better understood through a detailed examination involving controlled experiments. By systematically adjusting the concentration of secondary amines while considering other influential factors like epoxy type, curing conditions, and timeframes, a clearer correlation between secondary amine content and epoxy thickness can be established (Wand 2019). These controlled tests are vital for advancing the development of optimized epoxy coating systems. This investigation provides insights into how variations in secondary amine concentrations influence the dielectric characteristics and oxidation of the chemical reaction of epoxy and secondary amine. The ideal balance between amine concentration management and coating thickness. Additionally, amine level optimisation strengthens the coating's dielectric strength, which is important for applications needing dependable electrical insulation (Shuqi Wang et al. 2021).

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1.3 Research Objective

The objective consists of the following :

1. To identify the effect of the secondary amine concentration on the epoxy coating's thickness and microstructure produced by electrophoretic deposition (EPD).
2. To determine the influence of secondary amine oxidation on coating' chemical property throught Fourier Transform Infra Red (FTIR) spectroscopy.
3. To assess dielectric property of the epoxy coating by Electrochemical Impedance Spectroscopy (EIS)

1.4 Scope of Research

This research initiative primarily focuses on scrutinizing the influence of varying concentrations of secondary amines on the thickness of epoxy coatings obtained through electrophoretic deposition. The scope involves a meticulous exploration through controlled experiments, systematically altering the concentrations of secondary amines in epoxy solutions. The objective is to establish a direct correlation between the concentration levels of secondary amines and the resulting thickness of the coatings. With electrophoretic deposition (EPD), coatings are created when charged particles suspended in a liquid migrate in the direction of a substrate while it is subjected to an electric field. This adaptable technique is useful in a variety of sectors because it provides accurate control over coating thickness and uniformity. The versatility of EPD allows for the uniform coating of complicated structures and the modification of coating characteristics through the adjustment of parameters such as deposition duration or electric field strength. The method is used to create protective layers, functional films, and innovative materials in the automotive, electronics, and biomedical industries. Analysing coating attributes like thickness, shape, chemical composition, and mechanical and electrical properties is part of the process of characterising EPD. Comprehending these characteristics facilitates the customisation of the EPD procedure to fulfil particular coating demands for a range of industrial uses.

For the second objective, Another significant facet of this research endeavor is the examination of secondary amine oxidation's impact during the electrophoretic deposition process and its subsequent effects on coating characteristics. The scope encompasses a detailed exploration into the mechanisms and implications of secondary amine oxidation. Based on how molecules interact with infrared light, Fourier-transform infrared spectroscopy (FTIR) is an analytical method used to identify chemical compounds and comprehend molecular structures. It works by detecting how much infrared light a sample absorbs, which results in molecular vibrations that are unique to the functional groups in the molecules. In FTIR, a material is exposed to a wide range of infrared light, and the resulting absorption patterns are examined to pinpoint distinctive peaks connected to various chemical interactions. The composition, bonding, and functional groups of the molecule are revealed by the correlations between each peak and certain vibrational modes inside the molecule.

Expanding the scope further, this research involves an evaluation of the optimized dielectric properties of the epoxy coatings. It integrates the use of Electrochemical Impedance Spectroscopy (EIS) as a crucial analytical tool. EIS enables a detailed assessment of the coatings' dielectric characteristics, such as impedance and capacitance, under various conditions. Through the application of a tiny AC signal across the system and the observation of the voltage response that results, EIS offers a thorough understanding of impedance changes over a variety of frequencies. The resistance, capacitance, and charge transfer mechanisms of the system may be better understood by analysing the impedance data using plots such as Nyquist or Bode plots. This will help to clarify the electrochemical behaviour of the system.

Electrical Impedance Sampling (EIS) is a vital technique in the characterization and electrical performance optimisation of a wide range of materials and systems. It is extensively used in corrosion research, battery analysis, and the creation of electrochemical devices.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter describes the data that was gathered for the final year project research from the internet, newspapers, publications, and books. This chapter also provides a summary of epoxy resins. The data was collected to provide a comprehensive understanding of the topic being studied and to support the research objectives.

2.2 Epoxy resin

Epoxy resins are a type of the thermosetting polymer that are formd by the reaction between hardening agent. Epoxy resins are also known as high-performance materials, corrosion resistance, high mechanism strenght and high flexibility (X. F. Liu et al. 2022). To produce the succeeded epoxy resin must react with curing agent. It is to make sure the strenght and durability develope into the material. Epoxy resins are converted into solid, infusible, and insoluble three-dimensional thermoset networks by curing with cross-linkers (Qian et al. 2022). In real reactions, the density of cross linking is typically less than 100%, and this affects the chemical resistance, electrical properties, mechanical properties, and heat resistance of the cured thermosets.

Figure 2.1 illustrates how these favorable properties can be directly related to the resin chemical structure, which is available most commonly in the form of diglycidyl ether of bisphenol A (DGEBA) (Zhu et al. 2022). DGEBA is formed from the reaction of bisphenol A and epichlorohydrin, which when further reacted with an appropriate curing agent will eventually form a thermoset polymer, with aromatic groups from the bisphenol A component evenly distributed throughout the entire structure (X. Wang et al. 2019). They are one of the best materials available when comparing performance against relative cost, and this has in turn led to epoxies being the substance of choice for use in many high-performance polymeric applications (Paolillo et al. 2021).

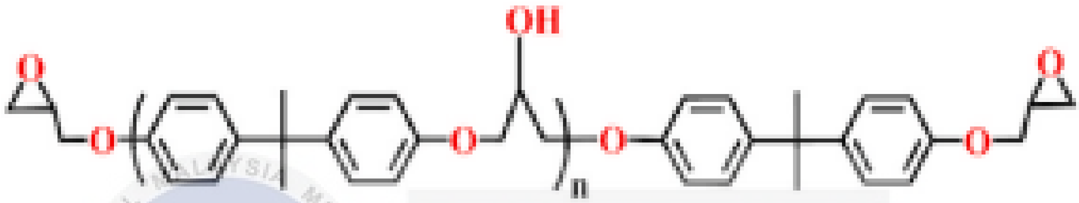

Figure 2.1 the chemical structure of a diglycidyl ether of bisphenol A (DGEBA) (X. Wang et al. 2019)

2.3 The system of epoxies

Epoxy resins are commonly formulated as either a one-pack system or a two-pack system, depending on the specific application requirements. In a one-pack system, all the necessary components, including the epoxy resin, curing agent, and any additives or fillers, are pre-mixed and supplied as a single package. This formulation offers convenience and ease of use, as it eliminates the need for precise measuring and mixing of multiple components. One-pack epoxy systems are often used in smaller-scale applications or situations where simplicity and time efficiency are important.

2.3.1 One-pack system

For a one-pack system, also known as a single-component system or one component epoxies. One-component epoxies can be used straight from the tube. No mixing, metering or degassing is required. However, these epoxies require heat for curing. One-component versions have an initiator, which is the heating element, contained along with the resin. The epoxy then requires an oven or other heating mechanism to reach full cure (Yang et al. 2021). As the system will either not react at all or react very slowly at ambient temperature. They were frequently identified and created as epoxy curing agents because of their high-temperature capabilities and greater selection for curing temperature. Typically having strong activity, imidazole may cure epoxy even at ambient temperature. The curing reaction occurring before actual application might shorten the storage period when imidazole is utilised as a curing agent or catalyst in one-component epoxy resin systems (Shi et al. 2021).

2.3.2 Two-pack system

The most common epoxies are two-part ones. The user of these epoxies must combine a resin and a hardener (Cort, n.d.). The polymerization necessary for curing is started by hardeners. An exothermic reaction takes place when the two are combined physically or manually when the molecules start to cross-link. In this case, need to apply it to the material faster before mixing between resin and curing agent growing the molecular which cannot be used anymore (Xiang and Xiao 2020). The low viscosity is related to filming the epoxy onto the substrate. To achieve this must dissolve the resin and the curing agent into solvent. Then, it will evaporate and allow the liquid to be converted into a solid form. Starting with the mixing of the two components, the most crucial need for retaining the varied characteristics of cured epoxy is established. Depending on the breadth of the application and the necessary features, a wide range of component formulas and different coating techniques are used (Choi and An 2022).

2.4 Type of the epoxy coating

The two most common kinds of epoxy coatings used in a variety of industries and applications are water-based coatings and liquid-applied coatings.

Water is used as a solvent or carrier in water-based coatings rather than from more conventional solvents like turpentine or mineral spirits. Water-based coatings' reduced emission of volatile organic compounds (VOCs) is one of its main advantage (Cunningham et al. 2019). Chemicals called VOCs, which may evaporate at room temperature and cause air pollution, are to blame. When compared to solvent-based coatings, they are frequently thought of as being more environmentally friendly and emitting fewer VOCs (David and Niculescu 2021). The example of water-based coatings are paint, varnish, and sealants.

There are further benefits to using water as a solvent in these coatings. Water is a safer alternative to conventional solvents since it is widely accessible, affordable, and non-toxic (Li et al. 2019). It does away with the need to handle and dispose of dangerous chemicals, lowering possible dangers to worker health and safety and minimising environmental effect.

Liquid-applied coatings, on the other hand, are coatings that are applied in liquid form and then cure to form a solid, protective layer. These coatings can be applied to various surfaces such as roofs, walls, and floors to provide waterproofing, weatherproofing, and protection against corrosion and other damage (Kavitha et al. 2023). Liquid-applied coatings can be made from a variety of materials such as acrylics, polyurethanes, and epoxies.

The choice between water-based and liquid-based epoxy depends on a number of parameters related to the particular application. Epoxy systems that are liquid-based often perform exceptionally well in terms of chemical resistance, making them the best choice for situations where strong chemicals or solvents will be present (Ghosal and Nayak 2022). Additionally, they often provide stronger adhesion and bonding, which is useful in load-bearing or structural applications that need for solid connections between substrates. On the other hand, water-based epoxies have the advantage of faster curing times, enabling quicker project turnaround (Lakhdar et al. 2021). Additionally, they often contain less volatile organic compounds (VOCs), which makes them safer to handle and more ecologically friendly. When emissions must be kept to a minimum in confined or delicate environments, this lower VOC content can be especially significant.

2.4.1 Behavior of water-based coating

When used as a protective or ornamental layer over a variety of substrates, water-based coatings exhibit certain behaviours based on how they function, behave, and are made up of certain qualities (Fang, Huang, and Fu 2022). Water is used as the principal solvent in coatings that use water rather than more conventional organic solvents. They have a reduced environmental impact and safer handling than solvent-based coatings, which is why they are utilised extensively across a variety of sectors (Liu et al. 2022).

In several applications, water-based coatings are favourable due to a number of fundamental behaviours they display. They are firstly capable of generating excellent films, which enables them to cover the substrate with a coating that is both homogeneous and continuous (Zhang et al. 2020). As a result, surfaces like metals, plastic, or wood adhere to them with exceptional strength. When it comes to application, water-based coatings often have low viscosities and are simple to use with a variety of techniques, such as brushing, spraying, or dipping. Smooth and even covering is made possible by their strong flow and levelling qualities (Kong and Meng 2022). The rapid drying period of these coatings also enables quicker manufacturing and less downtime.

The durability and resistance to corrosion of water-based coatings are also quite good. Depending on the formulation, they can offer protection against chemicals, moisture, UV rays, corrosion, and more (Peltier and Thierry 2022). In addition, they frequently have exceptional flexibility and hardness, enabling the coated surface to endure mechanical forces like impact or abrasion.

2.4.2 Behavior of liquid-based coating

Coatings that are applied in liquid form and subsequently cured to produce a solid, protective layer are referred to as liquid-based coating (Marlinda et al. 2023). Several variables, such as the kind of coating, the substrate being coated, and the application technique, can affect how liquid-based coatings behave. The following are some typical features of liquid-based coatings. Liquid coatings tend to flow and level off after application, creating a smooth, uniform surface (Fu and Dudley 2021). However, if the coating is put too thickly or the substrate is not level, this might potentially result in problems like sagging or running.

Liquid coatings normally go through a drying and curing process in order to solidify and provide a protective layer (Croll 2020). While the curing process requires a chemical interaction between the coating components, the drying step involves the evaporation of solvents or water

(Dehm et al. 2023). Temperature, humidity, and airflow are a few examples of the variables that may affect how quickly and well this process occurs.

Liquid coatings can be applied at various film thicknesses, ranging from extremely thin to relatively thick (Le et al. 2019). Although thicker coatings may offer more protection against harm, they may also be more prone to problems like peeling or cracking.

2.5 Epoxy coating characterization

Epoxy coatings are well regarded for their exceptional protective, visually pleasing, and practical qualities, making them an ideal choice in a variety of industries. Epoxy coatings go through a rigorous and thorough characterization process to assure their quality, performance, and compatibility for certain applications (Mirabedini et al. 2020). To assure the performance and quality of an epoxy coating, a thorough evaluation and study of its numerous characteristics is required. The assessment of anti-corrosion, microstructure analysis, and thickness measurement are the three main components of epoxy coating characterization (Sowa et al. 2022).

2.5.1 Thickness

Epoxy coating The process of characterization of thickness entails measuring and assessing the thickness of the epoxy coating that has been applied to a substrate (Guo et al. 2021). The coating's thickness is an important factor since it directly affects the coating's barrier and protective qualities. To offer sufficient defence against corrosion, abrasion, and other environmental variables, it is imperative to make sure the coating complies with the necessary thickness criteria (Arnal et al. 2022).

Epoxy coating thickness is often described by measuring both the average thickness and the uniformity of the coating throughout the whole surface (Center, Resource 2021). An incorrect application or possible regions of weakness may be indicated by variations or irregularities in the coating thickness. For the purposes of quality control and assurance, an accurate assessment of epoxy coating thickness is crucial. It aids in ensuring that the coating offers the desired level of protection and satisfies the application's criteria (Benrezgua et al. 2022). Furthermore, accurate thickness characterization enables efficient problem-solving and identifies any difficulties that could have an impact on the performance of the coating (Verma and Khanna 2023).

In overall, epoxy coating thickness characterization is a critical component of assessing the effectiveness and quality of the coating. Accurate thickness measurement and assessment guarantee that the coating offers sufficient protection and adheres to the required standards (Sousa et al. 2021). Manufacturers are able to offer high-quality epoxy coatings for a variety of industrial and commercial applications by following standardised techniques and rules that assure consistent and reliable coating thickness measurements (Lucherini and Maluk 2019).

2.5.2 Microstructure

Analysing the microstructure of an epoxy coating is an essential part of describing it. It requires looking at the coating's internal structure and structure in order to comprehend its composition, component distribution, and general level of quality (Shuaixing Wang et al. 2020). The physical, chemical, and performance features of the coating may all be learned by microstructure analysis.

Analysing the microstructure with the aim of determining the integrity and uniformity of the epoxy coating is one of the main objectives. The efficiency of the coating may be hampered by imperfections like voids, porosity, or inclusions, which can be found by analysing the microstructure (Serrano et al. 2022). These imperfections may result in diminished mechanical capabilities, poor adhesion, or decreased corrosion resistance.

Microstructure analysis usually involves the use of methods including optical microscopy, scanning electron microscopy (SEM), and atomic force microscopy (AFM) (Falsafi et al. 2020). Visual examination of the coating's surface and cross-section using optical microscopy can provide details about its general structure and the distribution of fillers, colours, and additives (Calovi and Rossi 2023). The topography, particle morphology, and interfacial properties of the coating may be seen using SEM's increased magnification and detailed imaging (Jaques et al. 2022). AFM offers high-resolution imaging and accurate surface roughness measurements, enabling nanoscale investigation of the topographical characteristics of the coating (Ruggeri et al. 2019).

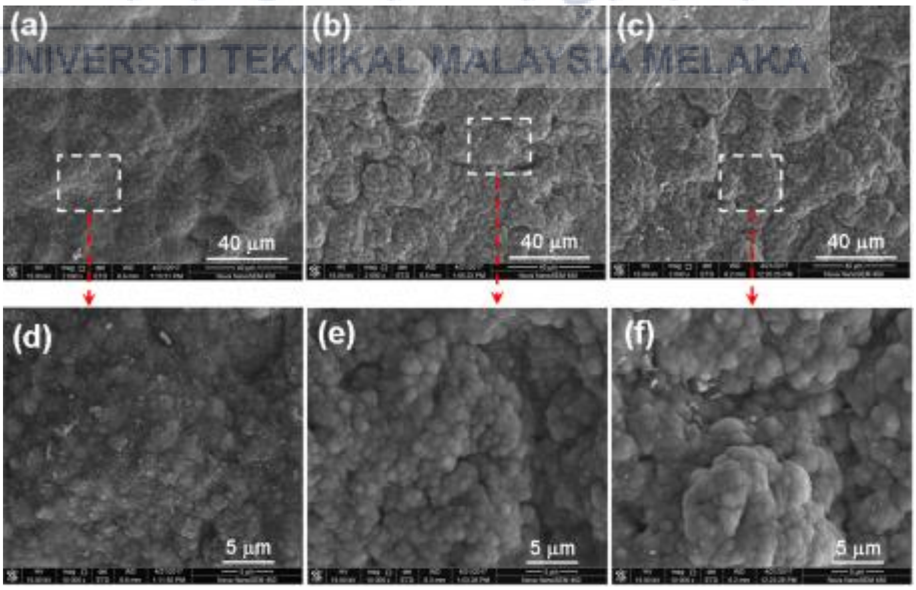

Figure 2.2 SEM morphology and microstructure of aluminum coating
(Shuaixing Wang et al. 2020)

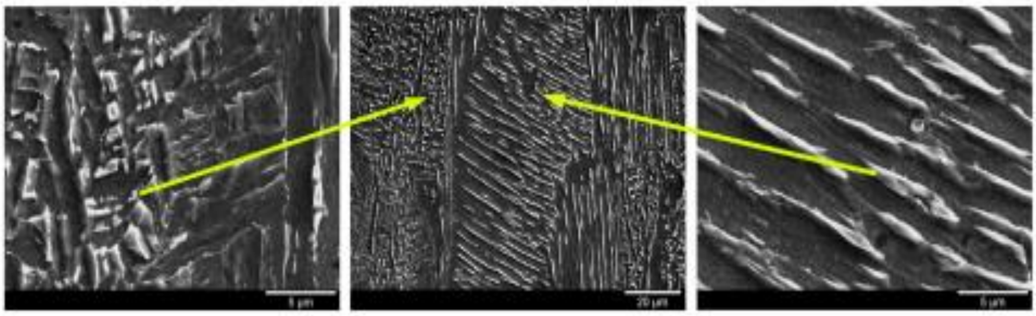

Figure 2.3 SEM image of the microstructure (Jorge et al. 2021)

In broadly, microstructure analysis is crucial for comprehending the quality, durability, and effectiveness of an epoxy coating. It aids in spotting any flaws, improving the formulation and application of the coating, and making that the coating complies with the required standards and requirements. Manufacturers may create epoxy coatings with better characteristics and increased resistance to corrosion, abrasion, and other environmental variables by characterising the microstructure (Fathi et al. 2022).

2.5.3 Electrophoretic Deposition of Dielectric Epoxy

Electrophoretic Deposition (EPD) is a versatile technique used to apply a dielectric layer onto an existing epoxy coating. This method begins with the preparation of a suspension containing dielectric particles dispersed within a liquid medium. These particles are carefully selected based on their dielectric properties and size to achieve the desired electrical insulation or other functionalities (Atiq Ur Rehman et al. 2021). The suspension is prepared to ensure uniform dispersion and stability, crucial for an effective EPD process.

Prior to the EPD application, the surface of the epoxy coating undergoes meticulous cleaning and preparation. This step is essential to remove any contaminants or impurities that might hinder the adhesion of the dielectric layer. Cleaning might involve degreasing, etching, or other surface treatments to create an ideal substrate for the subsequent deposition.

During the EPD process, an electric field is applied, causing the dielectric particles within the suspension to migrate and deposit onto the prepared epoxy-coated surface. The electric field facilitates the movement of charged particles, guiding their precise placement onto the substrate. This method allows for controlled and uniform deposition, enabling the creation of a tailored dielectric layer with specific thickness and properties, augmenting the functionality and performance of the epoxy coating for various applications, such as improving insulation or modifying surface characteristics (Shen, Zheng, and Kim 2021).

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2.6 Secondary amine as curing agent

Secondary amines are utilized as curing agent in various materials, particularly in the production of epoxy resins. These compounds possess a nitrogen atom bonded to two carbon atoms and one hydrogen atom, making them pivotal in the curing process of epoxy resins. When combined with epoxy resins, secondary amines act as curing agents, initiating the cross-linking reaction necessary for the resin to solidify and gain its durable properties.

The hardening process involves a chemical reaction between the epoxy resin and the secondary amine hardener. This reaction, commonly known as the amine curing reaction, occurs through the nucleophilic attack of the amine nitrogen on the epoxy groups present in the resin (Emad Aziz 2010). This triggers the opening of the epoxy ring, leading to the formation of a three-dimensional network, thereby solidifying the material. The choice of secondary amine hardener greatly influences the curing process and the final properties of the hardened material. Different secondary amines exhibit varying reactivity levels and pot life (the duration during which the mixture remains workable), allowing for control over the curing speed and flexibility in application.

Moreover, secondary amines contribute to the modification of epoxy resin properties. The nature of the amine structure can impart specific characteristics to the hardened material, such as improved flexibility, thermal resistance, or impact strength, depending on the application requirements (Saeedi, Andritsch, and Vaughan 2019).

2.6.1 N-Methylethanolamine

The methyl group that has been added to the nitrogen atom of N-Methylethanolamine (NMEA) makes it stand out among other members of the ethanolamine family. The chemical compound in question is significant since it has a wide variety of uses in many industries. NMEA is used in the pharmaceutical industry as a precursor in the synthesis of different compounds, which helps to create necessary treatments. Its function also includes organic synthesis, where its chemical characteristics are used to help create a variety of compounds and create novel materials and substances (Boekell et al. 2023).

N-Methylethanolamine is also useful in agriculture, where it is frequently used in the creation of agricultural compounds. Its characteristics influence crop protection and yield by augmenting the effectiveness of fertilisers or insecticides. Beyond this, its use as a corrosion inhibitor or catalyst highlights how crucial it is to a number of production processes, helping to protect against corrosive effects or speed up reactions, thus enhancing the lifetime and efficiency of industrial systems.

2.6.2 Properties of N-Methylethanolamine

N-Methylethanolamine demonstrates hygroscopic properties, which means it has the capacity to absorb moisture from its surroundings. When exposed to high levels of humidity, this trait may cause clumping or stickiness. According to the Table 2.2 show the general properties.

Table 2.1 general properties of the N-Methylethanolamine(Benabid et al. 2019)

Chemical Formula	CH3NHCH2CH2OH
Density	0.935 g mL−1
Molecular Weight/ Molar Mass	75.111 g·mol−1
Boiling Point	158.1°C
Melting Point	-4.50 °C

2.7 Electrophoretic deposition (EPD)

A method called electrophoretic deposition takes use of the motion of charged particles in suspension when an adequate electric field is present. The consolidation of such particles into films, cast onto any form of substrate, or thick, bulk components is made possible by this electric field. It is essential to have an overview of the deposit's limited drying and sintering problems, colloidal stability, and deposition kinetics in order to properly use electrophoretic deposition (Mostafapour et al. 2021).

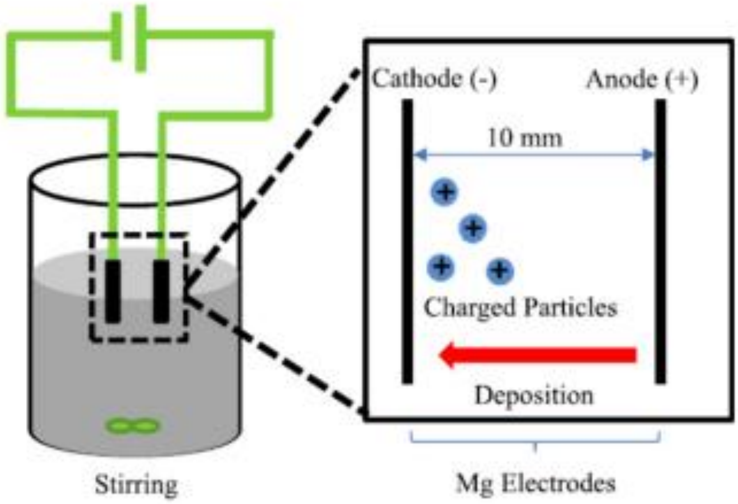


Figure 2.4 A scheme showing EPD (Saji 2021)

A charged substance is deposited onto a conductive substrate using the electrophoretic deposition (EPD) method, which is influenced by an electric field (C. Wang et al. 2022). Several EPD process factors, such as the following, have an impact on the thickness of the deposition of material. One of the most crucial factors impacting the thickness of the deposited material is the applied voltage. Increasing the voltage can speed up the deposition process but can also result in imperfections and uneven layer deposition.

The quantity of material that is deposited into the substrate is determined by the deposition time, which also impacts the final coating's thickness. Though thicker coatings are often the consequence of longer deposition durations, this may also increase the chance of imperfections and non-uniformity.

2.7.1 Parameter applied in EPD

The suspension's concentration, or the number of particles in the solution, can have an impact on the thickness of the substance that is being deposited. Higher concentrations can result in formation and non-uniformity in the presence of thicker coatings. The distance between the electrodes has an impact on both the deposition rate and the intensity of the electric field. A shorter distance may provide thicker coatings and a greater deposition rate, but it might also make imperfections and non-uniformity more likely (Schäfer et al. 2020).

In simple terms, several process variables, such as voltage, time, suspension concentration, and distance between the electrodes, affect the thickness of the deposited material in electrophoretic deposition (EPD). To create a final coating that is uniform and the proper thickness, these factors must be carefully regulated (Varlık, Göncü, and AY 2022).

Table 2.2 Example variable suspensions (Varlık, Göncü, and AY 2022).

Variable Parameters	level			
Application Voltage (V)	25	50	75	100
Deposition Time (s)	30	60	120	-
Chitosan Concentration(%)	0.05	0.1	-	-

2.8 Performance Tests

For epoxy coatings, performance tests like Fourier-Transform Infrared Spectroscopy (FTIR), Application of Electrochemical Impedance Spectroscopy (EIS), and Application of Scanning Electron Microscope (SEM) are essential for assessing and comprehending their properties, characteristics, and performance. These tests offer important details regarding the coating's composition, structure, and morphology, allowing for a thorough evaluation of its performance and applicability.

2.8.1 Application of Fourier-Transform Infrared Spectroscopy (FTIR)

For locating and describing functional groups in epoxy coatings, FTIR analysis is an effective technique. Researchers may learn more about the chemical components of the coating and its stage of curing by using this technique, which works on the idea of detecting the infrared light absorption by various chemical bonds.

Epoxy resin interacts chemically with a curing agent or hardener during the curing of epoxy coatings, causing crosslinking and the development of a three-dimensional network. The epoxy system's functional groups may be identified by distinct absorption bands that can be found via FTIR analysis (ElFaham, Mostafa, and Nasr 2020). The degree of conversion and whether the epoxy has fully cured may be assessed by contrasting the FTIR spectra of the uncured epoxy resin with those of the cured coating.

Monitoring the development of the curing process over time can also be aided by FTIR analysis. One may notice changes in the absorption peaks, which signify the transformation of functional groups and the production of new chemical bonds, by capturing spectra at various phases of the curing process (Silva et al. 2022). This information is useful for checking that the intended chemical reactions have occurred and that the curing process is complete. As the figure 2.5 shown the example of the graph that obtain from the FTIR spectra.

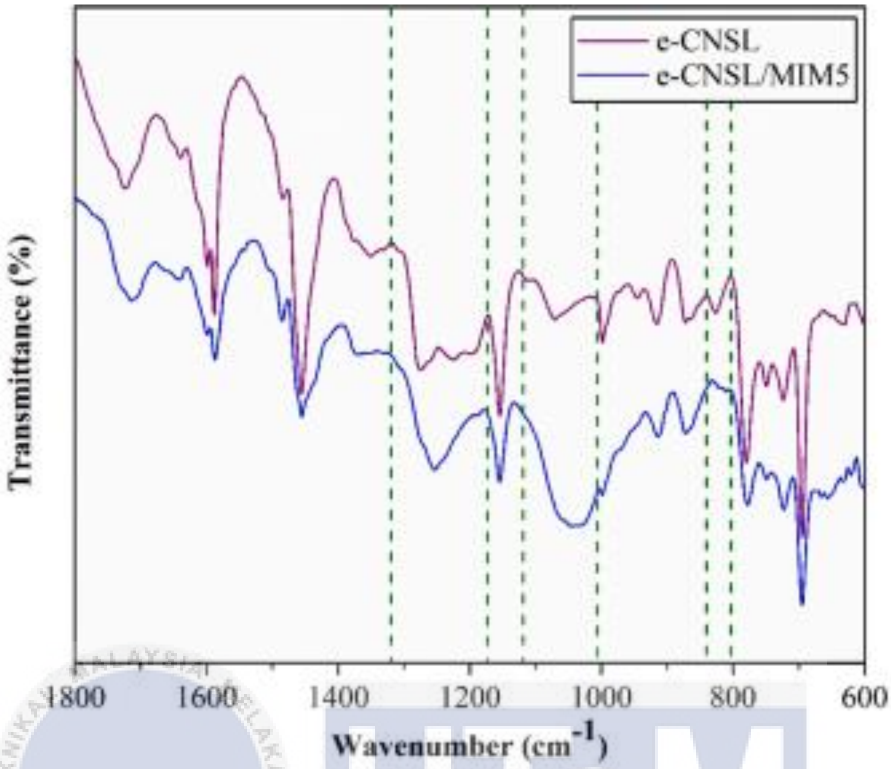


Figure 2.5 Example of FTIR spectra of the epoxy resin and the cured coating (Silva et al. 2022).

Additionally, the epoxy coating's unreacted components can be found via FTIR analysis. Any lingering unreacted components can be located by comparing the FTIR spectrum of the dried coating with the reference spectra of the epoxy resin and curing agent. This will reveal the presence of their distinctive absorption bands. According due figure 2.6 shown the result of ammonium phosphate by using FTIR. (Jaramillo et al. 2021)

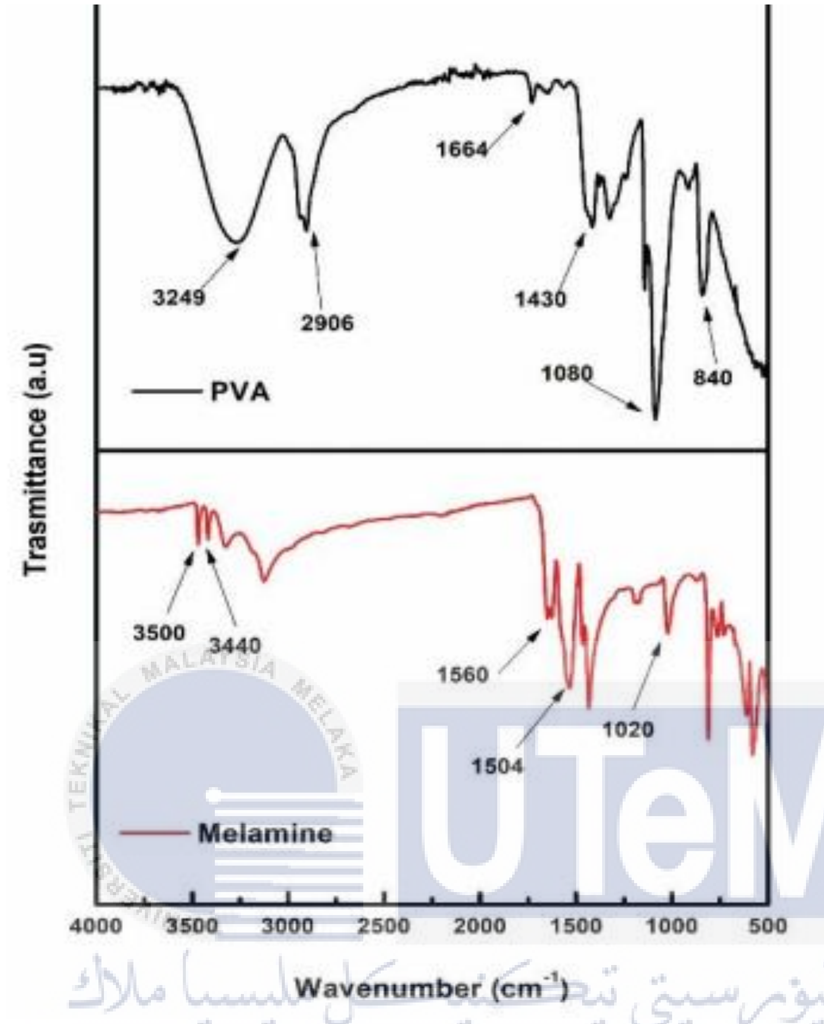


Figure 2.6 (FTIR) spectra of the microencapsulation of dihydrogen ammonium phosphate (Jaramillo et al. 2021).

In summary, FTIR analysis enables the identification and characterisation of functional groups, confirmation of the chemical composition, assessment of the degree of crosslinking in epoxy coatings, and determination of the curing state. It is an effective method for tracking the progression of the curing procedure, locating unreacted materials, and confirming the necessary chemical reactions have taken place, eventually assisting in the assessment and improvement of epoxy coating performance.

2.8.2 Application of Electrochemical Impedance Spectroscopy (EIS)

Electrochemical impedance spectroscopy (EIS) can extract comprehensive electrochemical information from systems, it is a flexible analytical tool used in many scientific areas. Corrosion investigations are among its main applications. EIS is essential for assessing changes in impedance across a wide frequency range in order to assess the corrosion resistance of metals, coatings, and alloys (Joshi et al. 2017). By assisting in the identification of corrosion products, the comprehension of corrosion mechanisms, and the evaluation of protective coating efficacy, this approach considerably enhances the durability and integrity of materials in sectors such as infrastructure, transportation, and construction .

Moreover, EIS is essential to the creation and improvement of innovative coating materials. EIS is a tool used by researchers to analyse the electrochemical behaviour of coatings and modify their formulations for better results. Scientists may improve the composition of coatings, comprehend their protective processes, and strengthen their resistance to a variety of environmental variables including moisture, chemicals, and temperature variations by examining impedance spectra. As show Figure 2.7 is the schematic EIS setup (Bakalli et al. 2023).

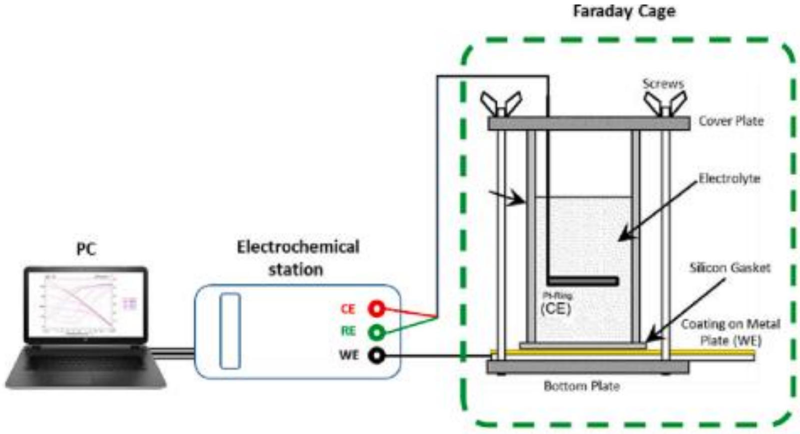


Figure 2.7 Schematic of the Electrochemical impedance spectroscopy (EIS) setup (Bakalli et al. 2023).

Additionally, EIS helps track how coatings degrade over time. By means of accelerated ageing experiments or exposure to severe environments, EIS enables researchers to monitor changes in impedance, offering valuable insights into the durability and long-term performance of coatings (Mcintyre and Pham 1996). With the use of this data, coating lifespan predictions may be made, allowing for the creation of stronger materials or preventative maintenance.

Furthermore, EIS is a non-destructive and effective method for large-scale coating integrity assessment in quality control procedures. It enables speedy decision-making in production or maintenance settings by enabling the fast evaluation of coatings' performance without causing damage to the underlying substrate. All things considered, EIS makes a substantial contribution to the creation, evaluation, and upkeep of coatings, guaranteeing improved lifetime and protection for a range of industrial applications.

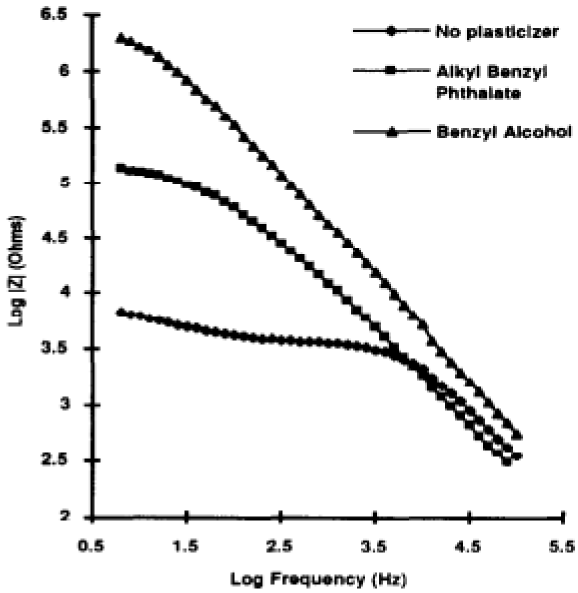


Figure 2.8 Example of Electrochemical impedance spectra (EIS) result (Mcintyre and Pham 1996).

As the Figure 2.8 show the example of EIS result on the coating plate. The graph definitely shows impedance readings over a variety of frequencies (Mcintyre and Pham 1996). This research presents unique characteristics and metrics that offer important insights into the performance of the coating.

2.8.3 Application of Scanning Electron Microscope (SEM)

For surface imaging and material characterisation, scanning electron microscopy (SEM) is an essential instrument. Surface morphology, microstructures, and nanostructures may all be seen and examined with SEM due to its high-resolution imaging capabilities. The electron beam moves across the sample's surface, interacting with the atoms to produce secondary electrons, backscattered electrons, and distinctive X-rays as signals.

Surface imaging and morphology analysis are both important SEM applications. It offers finely detailed pictures that show the texture, surface roughness, and distribution of characteristics like pores, fissures, and particles on the sample's surface. This knowledge is essential for comprehending the surface characteristics of materials and assessing their calibre, effectiveness, and usability. Due to the Figure 2.9 illustrate the compact microstructure with the skinkage of some crack of the of di-ammonium phosphate (Pesce et al. 2019).

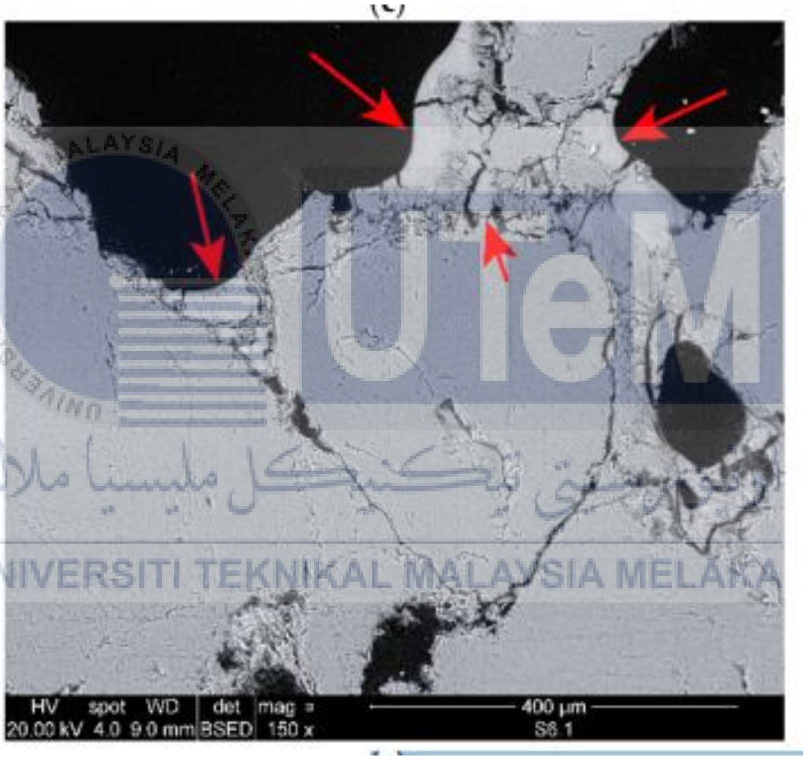


Figure 2.9 microstructure of di-ammonium phosphate (Pesce et al. 2019).

As the figure that shows above SEM is useful for analyzing the structure and composition of materials. It enables the investigation of interfaces, grain boundaries, phase distribution, and microstructures. This makes it especially helpful in failure analysis, process optimization, and materials research in fields including metallurgy, semiconductor production, and nanotechnology.

2.9 Conclusion

As a result, it is essential to characterise epoxy coatings to ensure that all of the parameters are uniform. The purpose of the performance test is then to confirm that and These tests provide crucial information on the composition, structure, and morphology of the coating, enabling a thorough evaluation of its performance and applicability.

CHAPTER 3

METHODOLOGY

In this chapter, an overview of the study design, which includes the project's overarching strategy and organisation, opens the chapter. The justification for the selected design and its applicability for the study aims are examined, emphasising both its benefits and drawbacks. The techniques used to gather the data, such as observations, or experiments, are then described together with the standards used to choose the samples.

3.1 Experimental Flow Chart

The investigation's technique is shown in Figure 3.1 which flow chart. The complete process and all of the elements that make up the study's scope in order to achieve the research objectives are referred to as methodology. The method's objective was to determine the coating process's thickness by analysing the microstructure of the sample. From planning to characterisation, Figure 3.1 shows the entire process of finishing this study.

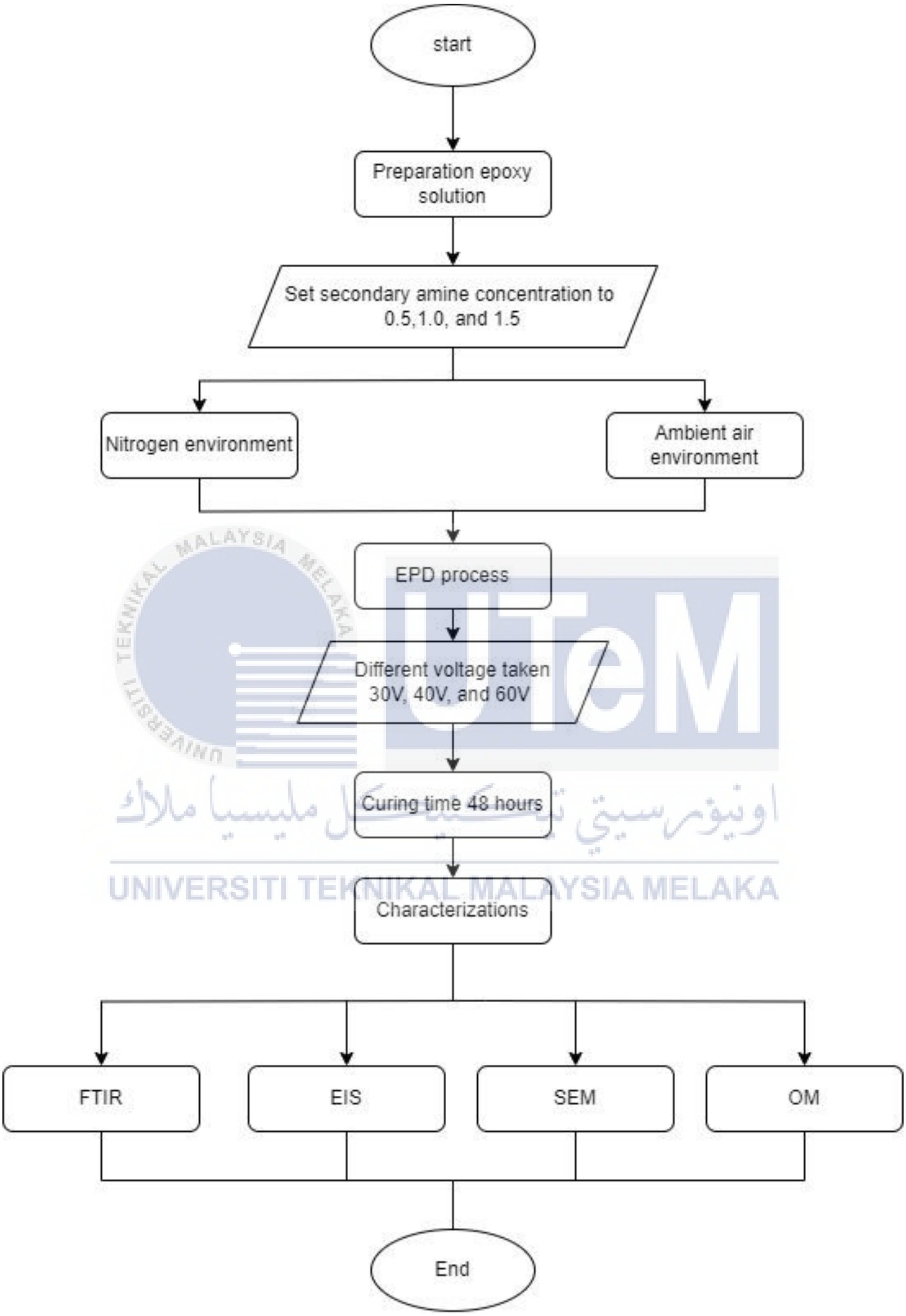


Figure 3.1 The flow chart

3.2 Raw Materials

Raw materials that have used for this study epoxy resin, ammonium salt, and stainless steel. For a precise ratio, utilise epoxy resin and ammonium salt as the raw material. This is to ensure that the mixed solution turns out perfectly. As a result, it will be possible to make sheet metal plates with the ideal thickness.

3.2.1 Epoxy Resin

For the epoxy resin we are using DGEBA (Diglycidyl ether of bisphenol A) as figure 3.2. it is also come with the physical properties of the DEGBA in table 3.1.

Figure 3.2 epoxy resin of DGEBA

Table 3.1 Physical properties of DEGBA

Diglycidyl ether of bisphenol A	DGEBA
FW	340.4
Density (g/mL)	1.160
T_m(°C)	40
Equiv (g^{-1})	172-176

3.2.2 Stainless Steel

29mm x 44mm x 0.5mm stainless steel has been cut to use as a base to lay up the mixture of the epoxy resin, that illustrated in figure 3.3. For this stainless steel plate are also used as electrode while conducting the EPD process. It is shown as figure 3.4.

Figure 3.3 stainless steel plate

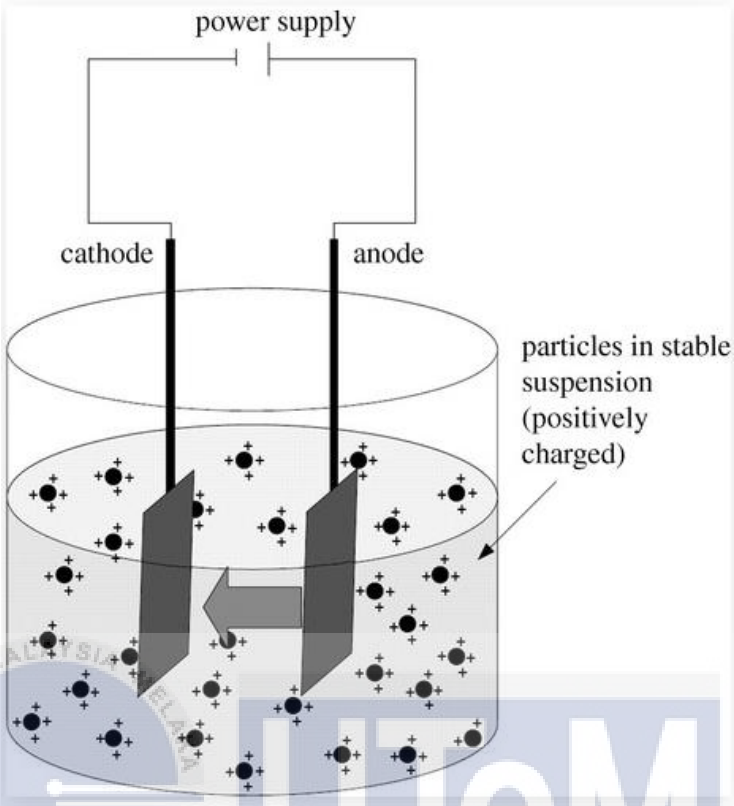


Figure 3.4 illustration of EPD

3.3 Sample Preparation

Epoxy resin (DGEBA) and N-methylethanolamine are the only solutions that are utilised during the sample preparation process. The epoxy must be preheated to 70°C and 21 minutes stirred before mixing the two solutions. The hardener should be added once the temperature reaches that level.Synthesis of Epoxy Resin. For the formation of the finished product, the temperature must reach 92°C while stirring after the epoxy and hardener have been combined.

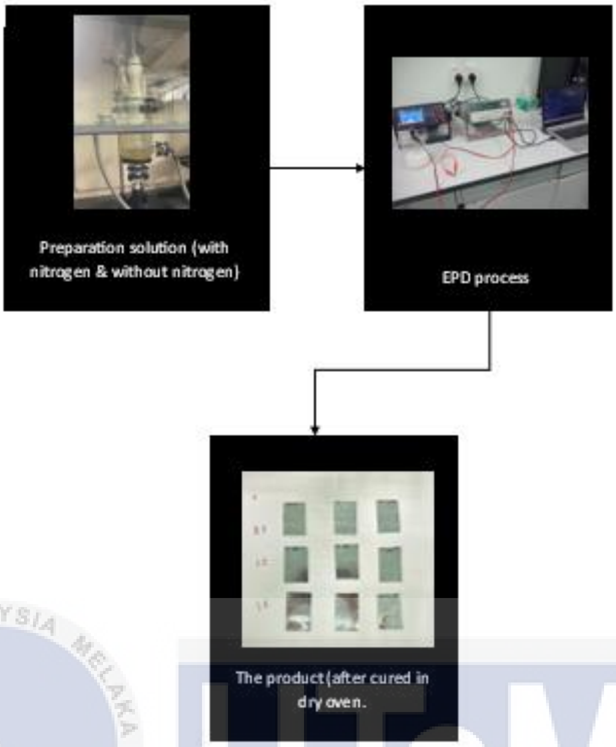


Figure 3.5 Preparation of sample

3.3.1 Labelling Sample

Several factors are taken into consideration while labelling samples. Labelling the sample is crucial to ensuring the organised structure of the outcome, as seen in Table 3.2.

Table 3.2 Labelling table

Lable	Concentration	Voltage (V)	With N2	Without N2
A	0.5	30		
B		40		
C		60		
D	1.0	30		
E		40		
F		60		
G	1.5	30		
H		40		
I		60		

3.4 Coating Techniques

A variety of procedures are utilised for applying protective or ornamental coatings onto substrates using coating techniques. These methods are useful for several applications and have various advantages. The application of coating material with brushes is a simple and economical process known as brushing.

Electrophoretic Deposition is referred to as EPD in the context of coating processes. An electric field is used to induce the deposition of particles or colloidal suspensions onto a conductive substrate during the electrophoretic deposition coating process.

Depending on the substrate type, application size, desired finish, and functional needs, these coating processes offer many alternatives to accomplish desired coating results.

3.4.1 EPD Set-up

The figure 3.5 illustrated the setup of the EPD. The digital multimeter and power supply with the same brand which Keysight Technology and both model is 34465A and E3643A has two sections, the first of which has a red box that displays a monitor for controlling voltage. The area where the electrodes are placed because of the anode and catode is next for the second red box.

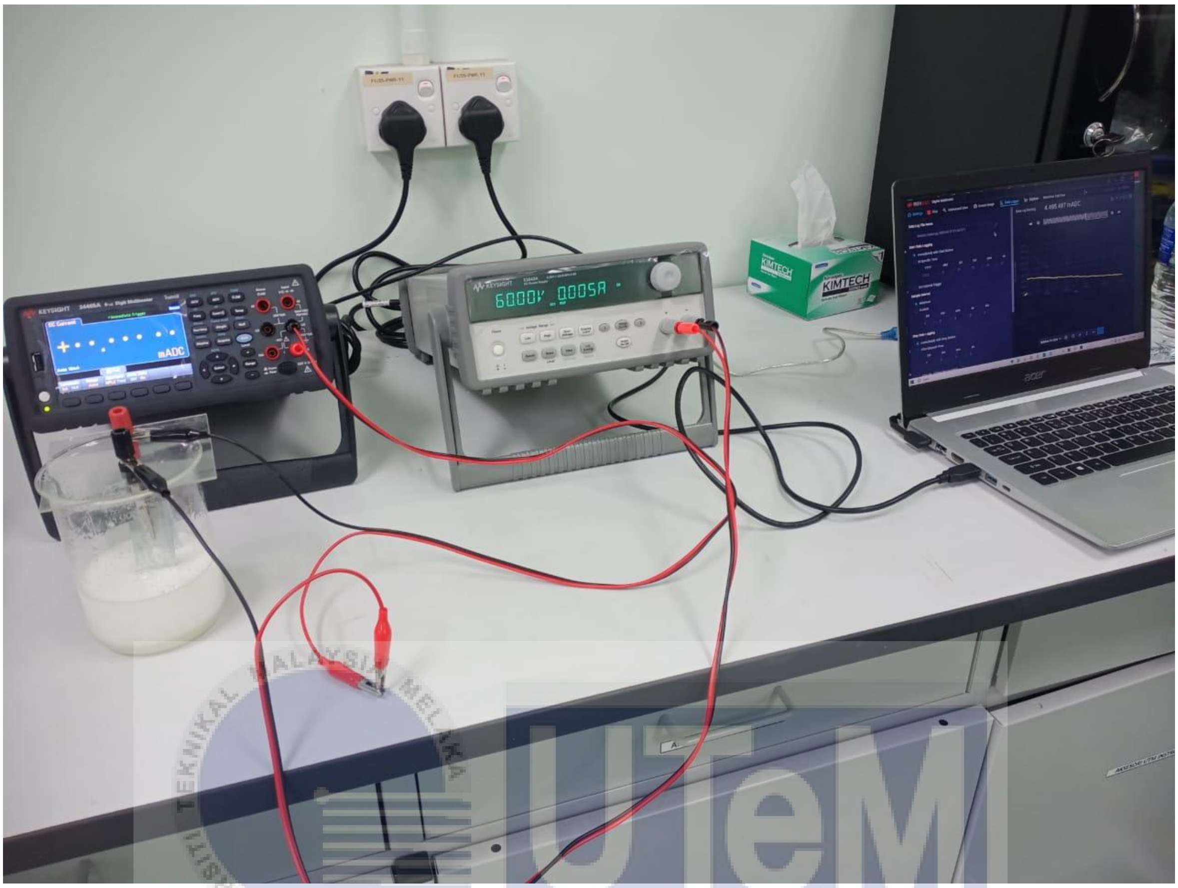


Figure 3.6 EPD setup

According due to figure 3.6 demonstrating the movement of positively charged particles in suspension towards the negative electrode.

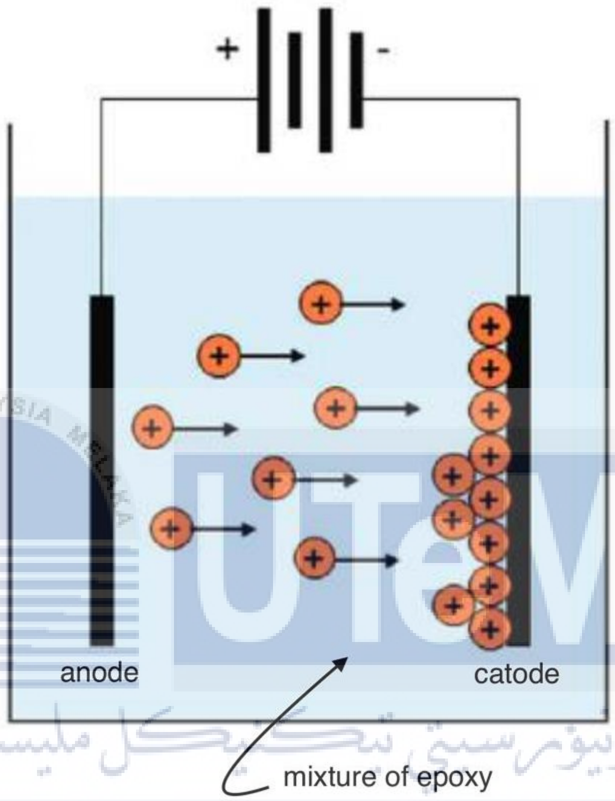


Figure 3.7 the movement positive charged to negetive charged

3.5 Characterization Test

Few methods have been utilised to describe the formation of nanoparticles, including Electrochemical impedance spectroscopy (EIS), scanning electron microscopy (SEM), and Fourier-Transform Infrared Spectroscopy (FTIR). Characterization methods were quickly and thoroughly discussed. These methods have the capability of displaying the outcomes of the produced samples.

3.5.1 The Mass Difference Before and After EPD

The term "mass difference before coated and after curing" describes the difference in mass that a substrate or material experiences before to and following the application of a coating through the Electrophoretic Deposition (EPD) process.

Firstly, before any coating is applied using EPD, the mass of the substrate is measured and recorded as the baseline value. Once the coating is applied using the EPD technique, the mass of the substrate is measured once more. The quantity of material that has been deposited into the substrate during the EPD process is indicated by the difference between these two measured masses.

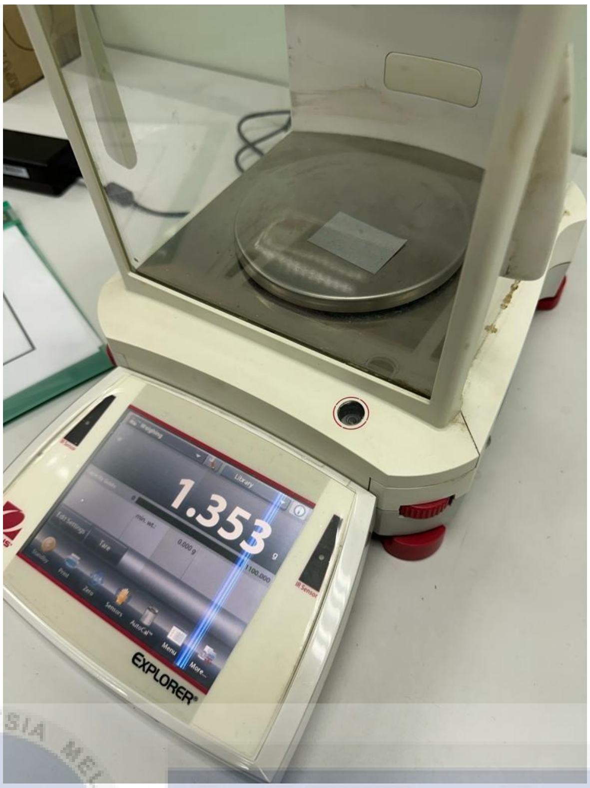


Figure 3.8 Weighing scale for mass.

When assessing the efficacy and efficiency of the EPD technology, this mass difference is quite significant. When the EPD method is applied successfully, a larger mass differential usually indicates a more significant coating material deposition onto the substrate. It functions as a measurable indicator of how much coating material is bonded to the substrate's surface.

Understanding this mass differential helps explain the thickness and homogeneity of the coating produced by EPD. A more dependable and repeatable EPD method is indicated by a constant and considerable mass difference across several samples; variations or discrepancies may suggest irregularities in the coating application, necessitating more research into process parameters for optimization.

In overall, the mass difference measurement is an important indicator for evaluating the effectiveness and dependability of the EPD method for coating deposit, impacting.

choices regarding process optimization and the caliber of the final coatings applied using this approach.

3.5.2 Fourier-Transform Infrared Spectroscopy (FTIR)

The skilled method of Fourier-Transform Infrared Spectroscopy (FTIR) which brand Jas.co with model Jacco FTIR-6100 allows for the analysis of the chemical composition of epoxy coatings. FTIR measures the different wavelengths of infrared light absorption by the coating material. The epoxy coating's functional groups absorb infrared light at certain frequencies, creating individual fingerprint spectra that may be utilised for characterisation and identification. It is possible to ascertain the existence of several functional groups, including hydroxyl groups, epoxy groups, carbonyl groups, and aromatic groups, which are crucial for epoxy chemistry, by analysing the FTIR spectrum. The degree of curing or crosslinking of the epoxy resin, as well as the presence of impurities or contaminants, may all be determined using FTIR.

The epoxy coating might undergo changes over time, such as deterioration or chemical interactions, which can be seen by FTIR analysis. FTIR is a rapid, non-destructive method that may be used for both research and quality control in the field of epoxy coatings. For the purpose of obtaining precise and representative FTIR spectra of epoxy coatings, the sample must be properly prepared, which may entail developing a thin layer or employing attenuated total reflectance (ATR) methods. As figure 3.8 shown the machine of FTIR.

Figure 3.9 Fourier-Transform Infrared Spectroscopy (FTIR) machine

3.5.3 Electrochemical impedance spectroscopy (EIS)

An alternating current (AC) signal is applied to an electrochemical system over a range of frequencies, usually from millihertz to megahertz, in order to perform Electrochemical Impedance Spectroscopy (EIS) which brand Gamry Instrument with model 600. It monitors the system's impedance response, or the resistance the system puts up to the AC current flow. This answer provides important details on the electrochemical behaviour of the system.

Fundamentally, EIS operates on the idea that various electrochemical processes inside a system behave differently in terms of impedance at various frequencies. Processes like as capacitance, diffusion, and charge transfer, for example, appear differently in the impedance spectrum at different frequencies. Plotting the resultant impedance data on Nyquist or Bode plots provides a visual depiction of the behaviour of the system, highlighting distinct shapes and patterns that are indicative of certain electrochemical processes.

Analysing these plots to extract factors that clarify the characteristics of the system is part of the process of interpreting EIS data. For example, the slope and peaks in a Bode plot provide information on capacitive and resistive elements, whereas the semicircular arc shown in a Nyquist plot usually shows charge transfer resistance. EIS makes it possible to derive quantitative information on reaction kinetics, diffusion coefficients, and the integrity of protective coatings by fitting experimental data to analogous electrical circuits.

The method's adaptability may be used to a wide range of tasks, such as materials research, corrosion investigations, sensor development, and battery characterisation. It is a very instructive and non-destructive method for studying the electrochemical behaviour of systems, which helps engineers and researchers create more efficient designs, improve performance, and create more effective materials.

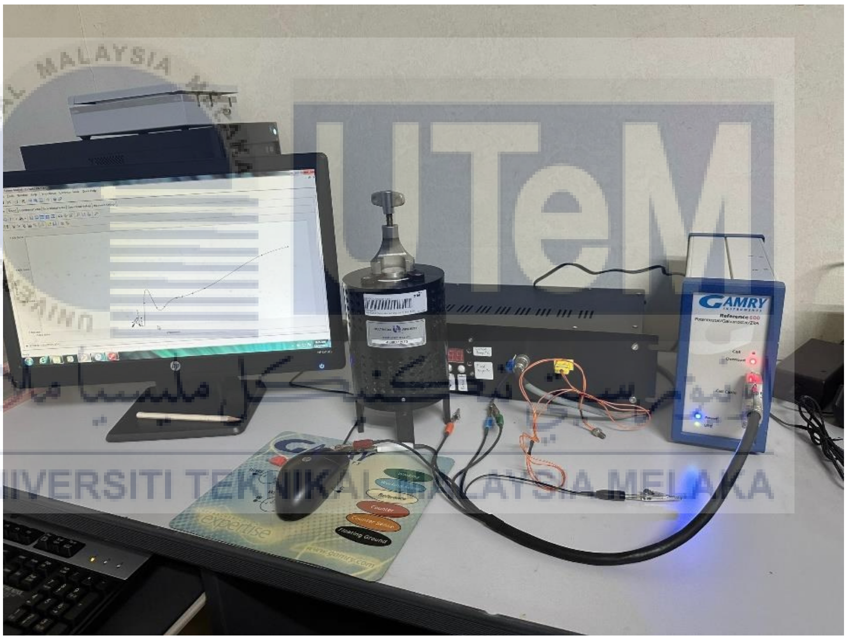


Figure 3.10 Electrochemical Impedance Spectroscopy (EIS) setup machine

3.5.4 Scanning Electron Microscope (SEM) and Optical Microscopy

SEM model Carl zeiss evo 50 under an accelerating voltage of 15 kV at a magnification of 100x, 300x, 500x, and 1000x were used to observe and analyse the accumulation and dispersion image of produced 40 samples. The machine as shown in Figure 3.8 is located at UteM laboratory. Prior to being scanned under a microscope, the sample was coated with gold-palladium to prevent electrostatic charge and inadequate picture resolution. The machine illustrated as figure 3.10.

Optical Microscopy as brand Leica with model DM 2500M is the process of seeing and magnifying tiny objects or samples using visible light. Scientists can see details as fine as a few hundred nanometers with the use of lenses, which reveals the composition and characteristics of materials, cells, and living things.

An effective tool for assessing the surface characteristics of epoxy coatings is a scanning electron microscope (SEM). SEM scans the coating's surface with a concentrated electron beam to provide high-resolution pictures and comprehensive data on its microstructure. The surface morphology of the coating, including elements like fractures, voids, particles, and changes in coating thickness, may be seen using SEM. The approach can also show if pollutants or other impurities are present, as well as how well the coating adheres to the substrate. Energy-dispersive X-ray spectroscopy (EDS) or wavelength-dispersive X-ray spectroscopy (WDS) systems with SEM provide elemental analysis in addition to imaging.

Understanding structure-property correlations, evaluating the coating's quality, looking into failure processes, and optimising the coating process for desired performance are all made possible with the help of SEM analysis of epoxy coatings. It should be noted that accurate and trustworthy findings from SEM examination depend on good sample preparation, including meticulous cleaning, drying, and coating stabilisation.

Figure 3.11 scanning electron microscope (SEM) and Optical Microscopy

3.6 Conclusion

The study's desired objectives were achieved through a staged approach and methodical structuring of the research technique. The upcoming review will focus on examining and rating the results obtained from the epoxy coating technique used in the investigation. The purpose of this evaluation is to determine the performance, consistency, and effectiveness of the epoxy coating technique in achieving the specified objectives and specifications of the research.

CHAPTER 4

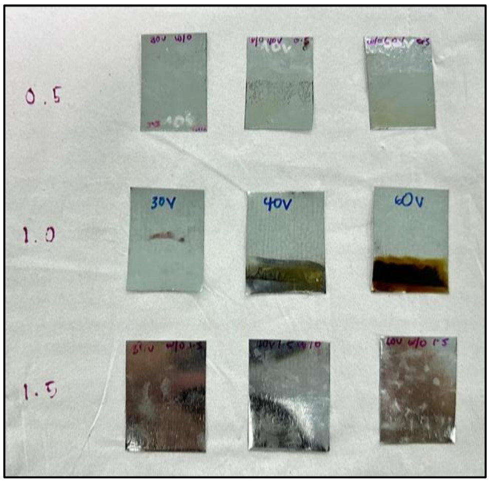
RESULT AND DISCUSSION

4.1 Introduction

The information acquired following the completion of the sample, chemical preparation, and testing tests is mostly explained in this chapter. Every theory and discussion based on the structural and physical properties of epoxy coating deposited will be supported by earlier research findings. The visual inspection of the artificial epoxy coating deposited was done after processing. Furthermore, A scanning electron microscope (SEM) was used to examine the surface properties. Fourier-transform infrared spectroscopy (FTIR) was utilized to gain insights into the molecular structure and chemical composition, while Electrochemical Impedance Spectroscopy (EIS) was utilized to investigate the electrical behavior of the particles across frequencies. The combination of these methods provided a thorough understanding of the electrical, structural, and chemical characteristics of the nanoparticles.

4.2 Physical Appearance of Substrates

The physical appearance of each sample with nitrogen environment and ambient air environment was depicted in Figure 4.1. It is obvious that every sample was present with different appearance due to the concentration of amine and voltage applied in EPD.

A) 

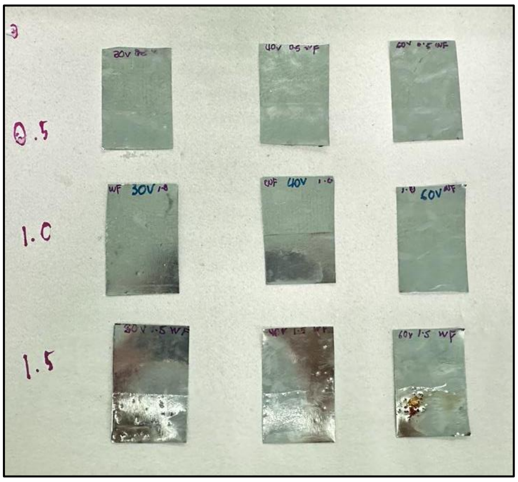
B) 

Figure 4.1 (A) sample in ambient air enviroment (B) sample in nitrogen enviroment

Additionally, the contrast between the nitrogen and ambient environments underscores the impact of environmental conditions on surface characteristics. Nitrogen environments minimize reactions, while ambient conditions encompass varying gases and moisture levels, which collectively influence the appearance of the deposited materials on the samples.

4.2.1 Thickness Analysis of Substrates

Assessing the efficacy and efficiency of the deposition process is significantly impacted by Table 4.1 and Table 4.2, which shows the mass of the plate both before and after Electrophoretic Deposition (EPD) for nitrogent environment and ambient environment. Some may determine how much material was deposited onto the plate during EPD by comparing the original and final masses. Removing or adding material is indicated as a drop or increase in mass, accordingly.

Table 4.1 weight before and after EPD in nitrogen environment

CONCENTRATION OF SECONDARY AMINE (mol/L)	APPLIED VOLTAGE ON EPD (V)	WEIGHT, (g)		THICKNESS (mm)
		BEFORE	AFTER	
0.5	30	1.353	1.455	6.89×10^{-5}
	40	1.351	1.426	5.07×10^{-5}
	60	1.349	1.418	4.66×10^{-5}
1.0	30	1.346	1.436	6.08×10^{-5}
	40	1.354	1.421	4.53×10^{-5}
	60	1.348	1.425	5.202×10^{-5}
1.5	30	1.352	1.416	4.32×10^{-5}
	40	1.354	1.414	4.05×10^{-5}
	60	1.347	1.381	2.30×10^{-5}

Table 4.2 weight before and after EPD in ambient air environment

CONCENTRATION OF SECONDARY AMINE (mol/L)	APPLIED VOLTAGE ON EPD (V)	WEIGHT, (g)		THICKNESS (mm)
		BEFORE	AFTER	
0.5	30	1.352	1.449	6.55×10^{-5}
	40	1.334	1.423	6.01×10^{-5}
	60	1.324	1.418	6.35×10^{-5}
1.0	30	1.366	1.512	9.86×10^{-5}
	40	1.356	1.466	7.43×10^{-5}
	60	1.341	1.436	6.41×10^{-5}
1.5	30	1.341	1.466	8.44×10^{-5}
	40	1.359	1.483	8.38×10^{-5}
	60	1.356	1.478	8.24×10^{-5}

The thickness formula that has been applied ;

$$\rho = \frac{m}{Vo}$$

$$\rho = \frac{m}{A \cdot t}$$

$$t = \frac{m}{A \cdot \rho}$$

This quantitative analysis is an essential indicator for assessing the amount of material transferred from the suspension to the plate, the process efficiency, and the deposition yield.

Tables 4.1 and 4.2 both indicate that the thickness value at lower voltage is greater. Lower voltages in electrophoretic deposition (EPD) may appear like a more economical choice because of their alleged energy efficiency. Nevertheless, this decision poses difficulties with the particle movement speed in the suspension. Extended processing periods resulting from slower deposition rates tie up important industrial resources and raise labour costs (Y. Wang et al. 2022). Furthermore, coatings become harder to achieve precisely and consistently at lower voltages, requiring more work and money for quality control. This might require specific methods or modifications, adding to the overall cost increase.

4.3 Material Properties

In material analysis, structural characteristics can be investigated using electrochemical impedance spectroscopy (EIS), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR). FTIR facilitates accurate identification and characterisation by revealing the complex composition and functional groups present in a sample through the probing of molecular vibrations. Simultaneously, SEM provides high-resolution imaging, which is essential for comprehending material topography and composition because it sheds light on particle sizes, surface morphology, and intricate structural details at microscopic scales. In addition to these, electrochemical behaviour is assessed by EIS, which offers information on conductivity, surface properties, and structural stability. When combined, these methods offer an extensive comprehension of the structural composition of a material, enabling careful examination and well-informed choices in a wide range of research.

4.3.1 Fourier-transform infrared spectroscopy (FTIR)

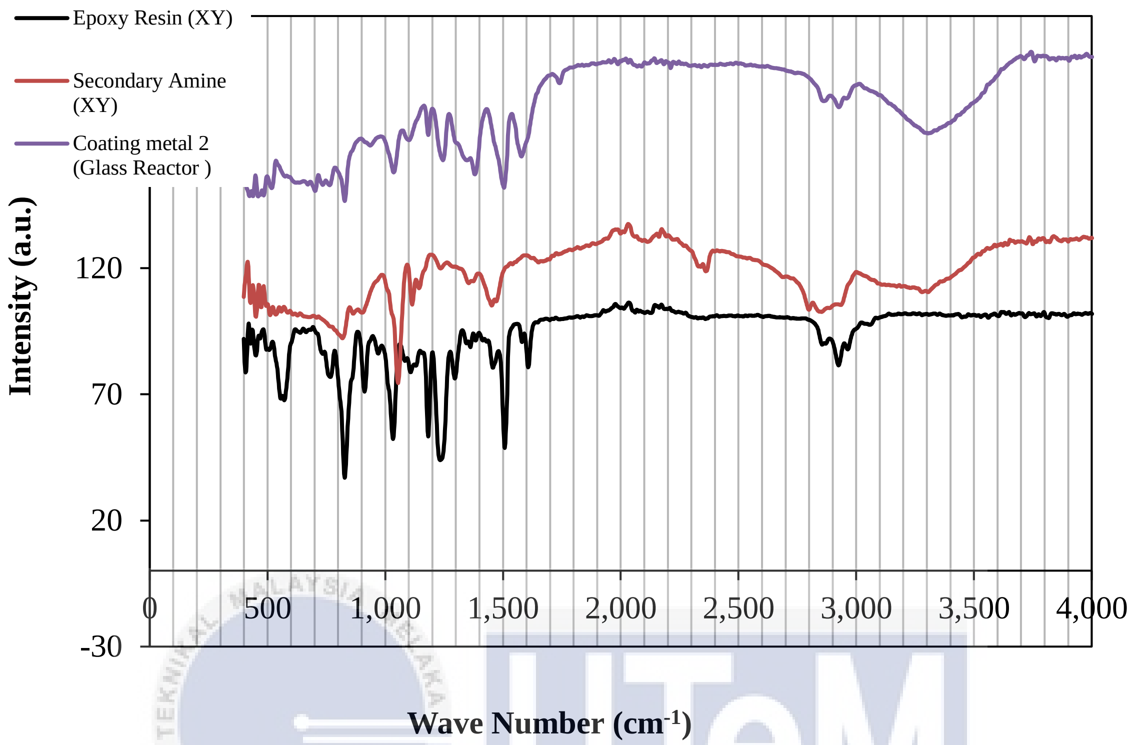


Figure 4.2 Combination FTIR graph of epoxy resin, secondary amine, and coating produce in ambient environment.

The FTIR graph shown in Figure 4.2, which includes the coating metals, secondary amine, and epoxy resin spectra from the glass reactor, offers important information on the chemical makeup and interactions of these constituents in the coatings.

Comparative evaluation of the functional groups and chemical bonds found in each component is made possible by the spectra's analysis. The spectra's peaks and distinctive bands show unique characteristics connected to certain chemical group. As the shown as, the secondary amine spectrum display peaks N-H stretching or amine groups (such as -NH or -NH2) on peaks 3290cm^{-1} - 3217cm^{-1}, whereas the epoxy resin spectrum would normally show peaks lies on 1660cm^{-1} - 1726cm^{-1} relating to epoxy groups (C-O-C) or aromatic. As for secondary amine is lies on a significant region on 1200cm^{-1} - 600 cm^{-1} functionalities as stated on (Morsch et al. 2020).

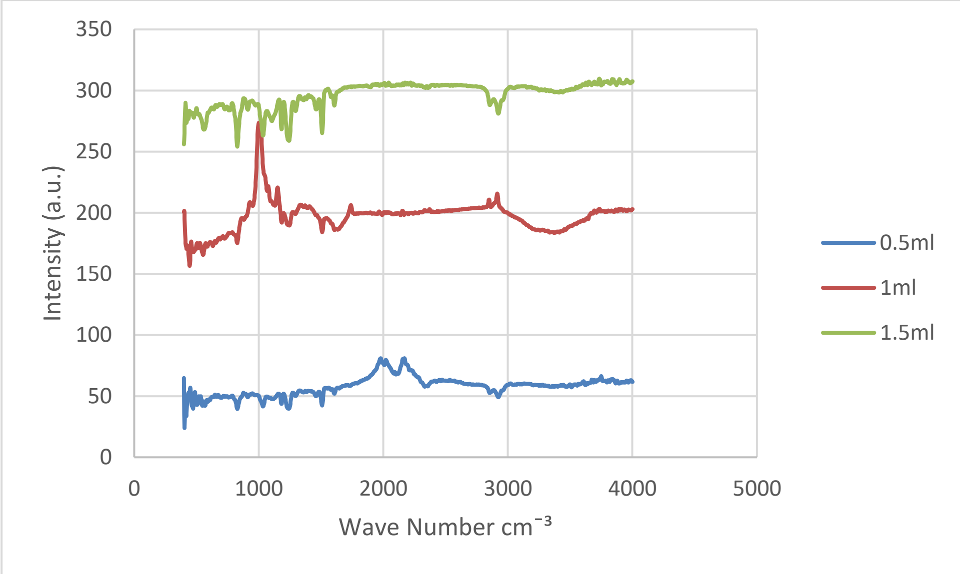


Figure 4.3 FTIR spectra of epoxy coating using solution produced in ambient air environment.

An FTIR analysis of the effects on different secondary amine concentrations (0.5, 1.0, and 1.5ml) in an ambient atmosphere is shown in Figure 4.3. Unique spectrum properties, such as absorption peaks and patterns matching to the different concentrations, are probably revealed by this spectroscopic data. Concentration-dependent alterations in molecular interactions and chemical compositions in the inert environment can be identified by analysing peak intensities, shifts, or the appearance of new peaks across various concentrations (da Silva et al. 2022). These spectra may be compared to provide insight into the effects of changes in secondary amine concentration on molecule bonding, structural configurations, and intermolecular interactions.

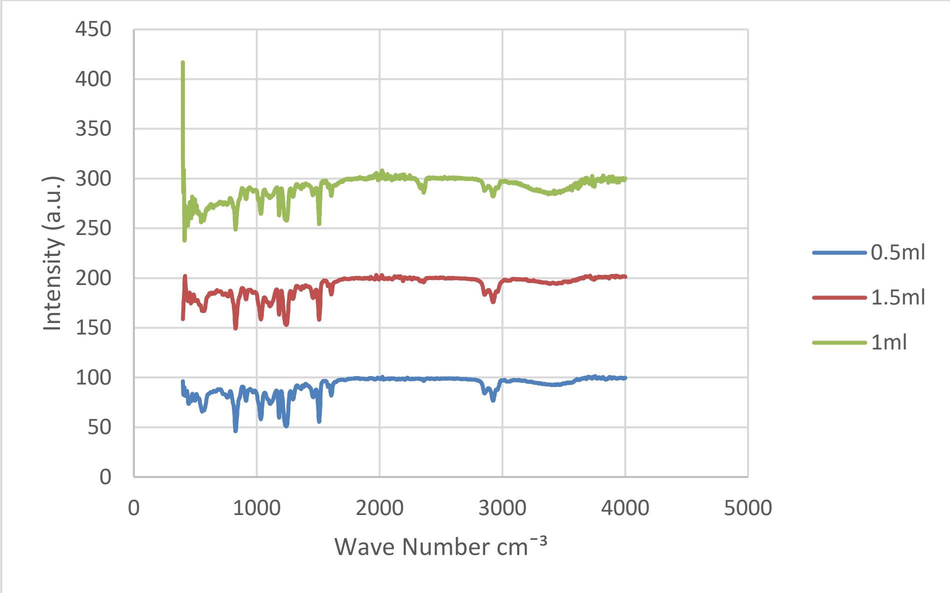


Figure 4.4 FTIR spectra of epoxy coating using solution produced in nitrogen gas environment.

Observing Figure 4.4, which shows three different secondary amine concentrations (0.5, 1.0, and 1.5mL) in a nitrogen atmosphere, provides an insight into the molecular interactions and chemical changes typical of this particular configuration. The varied quantities of secondary amine present are likely represented by distinct absorption peaks or patterns in the FTIR spectrum. Examining peak intensities, changes, or the appearance of new peaks connected with varying concentration levels are some of the aspects of delving into this spectrum data (C. Wang et al. 2022). These subtle variations are frequently indicative of modifications in molecular structures, chemical bonding, or unique interactions that are unique to the nitrogen environment when different secondary amine concentrations are present.

In overview, the FTIR evaluation is a useful instrument for comprehending the chemical properties, interactions, and possible bonding mechanisms among the constituents involved in the coating process. This knowledge can be applied to improve the properties of the final coated materials for a range of applications and to optimise the deposition parameters.

4.3.2 Electrochemical impedance spectroscopy (EIS)

Different behaviours may be seen in nitrogen and ambient air environments using Electrochemical Impedance Spectroscopy (EIS). Ambient settings emphasise the dynamics of charge transfer while highlighting intrinsic system features. On the other hand, environments rich in nitrogen add more complexity by changing impedance profiles and affecting surface responses. An important way to understand how different surroundings affect electrochemical behaviours is to compare the EIS results between them.

Using the formula below, the dielectric constant ($\mathcal{E}'$) for making a

$$\mathcal{E}' = \frac{t}{\omega A \mathcal{E}_{\circ}} \cdot \frac{Z''}{Z'^2 + Z''^2},$$

Where A represents the substrate's cross-section area, t = thickness, $\mathcal{E}_{\circ}$ its permittivity of free space, Z' = real part of impedance, and Z" its imaginary part of impedance.

The rate at which the complex impedance of a material or system oscillates as a function of time is referred to as the angular frequency in the context of dielectric constant analysis and Electrochemical Impedance Spectroscopy (EIS). It is represented by the symbol ω (omega) and has an equation that relates it to the regular frequency (f):

$$\omega=2\pi f$$

where,

- ω is the angular frequency,
- π is a mathematical constant (approximately 3.14159),
- f is the frequency in hertz.

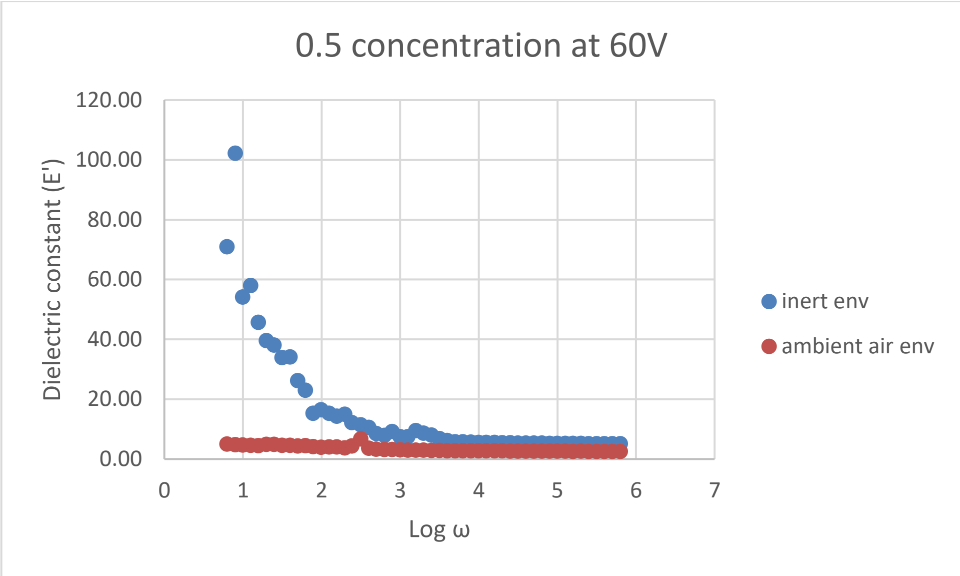
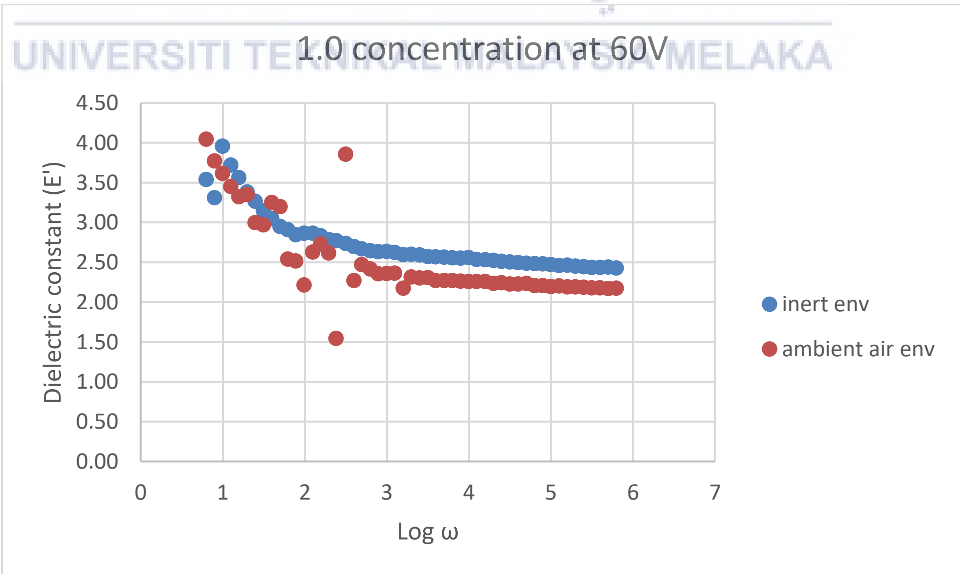


Figure 4.5 Graph dielectric constant (E’) vs. Log ω 0.5mL concentration at 60V

Figure 4.5 illustrates how the dielectric constant changes with frequency in a variety of settings, including ambient and inert, along with a value at 60 volts. As can be observed from this figure, the dielectric constant is high for both environments in the low frequency area, but it falls with increasing frequency due to the electric dipole's inability to adapt to changes in the applied a.c. electric field (Joshi et al. 2017).



4.6 Graph dielectric constant (E’) vs. Log ω 1.0mL concentration at 60V

Analysing the dielectric constant (ε) vs Log ω for a secondary amine solution at 1.0 concentration and 60V in Electrochemical Impedance Spectroscopy (EIS) in Figure 4.6 yields interesting results. Changes in Log ω in an inert environment can reveal a trend in the dielectric constant that corresponds to the solution's reaction to the applied electrical field. This connection provides insight into the material's electrical characteristics in this controlled environment by explaining its polarisation behaviour or the way its molecular structure interacts with the electric field (Joshi et al. 2017). Conversely, investigating the relationship between the dielectric constant and Log ω in an ambient setting may reveal different patterns as a result of various environmental factors. These changes may result from external influences that affect the conductivity of the solution and the consequent dielectric response, providing unique insights into the behaviour of the material under real-world settings.

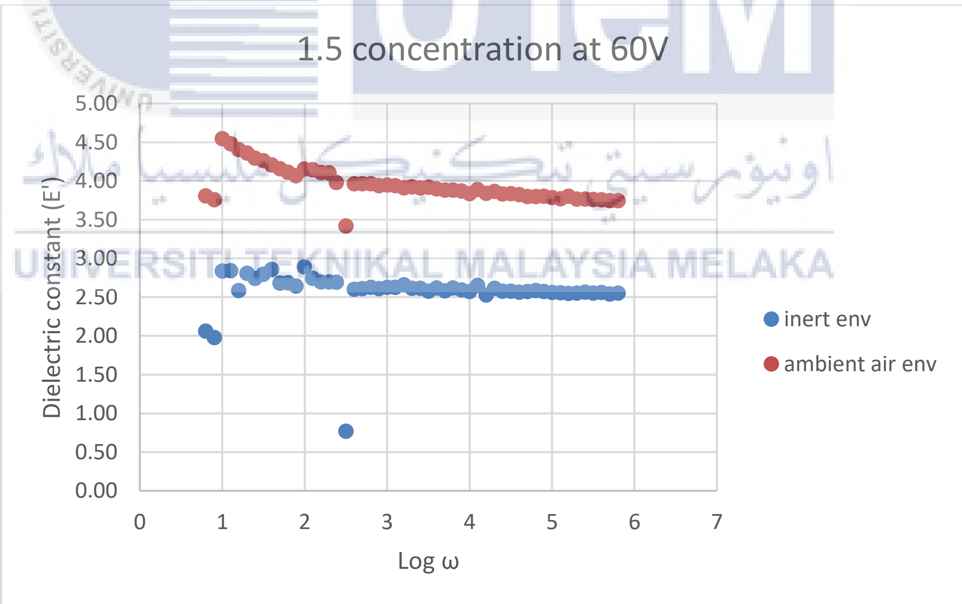


Figure 4.7 Graph dielectric constant (E’) vs. Log ω 1.5mL concentration at 60V

Based on Figure 4.7 referring to Dielectric constants can change depending on frequency, and concentration. The electrical energy-storing capacity or degree of polarisation is shown by the dielectric constant (E').

This graph most likely illustrates how the dielectric constant varies with a substance's logarithmic angular frequency at 60V. It may show how, under a constant voltage of 60V, the material's capacity to the electric components varies with varying concentrations. Depending on the characteristics of the material and how it behaves in an electric field, the connection between these variables may be linear, exponential, or exhibit another type of relationship.

4.3.3 Scanning electron microscopy (SEM) and Optical Microscopy

The morphological features were examined using the scanning electron microscope Model SU5000. The Figure 4.8 depict the morphology of the best microstructure of coating both in ambient air environment and nitrogen environment. This result is related to the EIS result as the best graph's curve. Refer the Figure 4.8 pictures a until d is in ambient air environment while e and d is in nitrogen environment. The high intensity surface of the epoxy coating, as revealed by SEM inspection, causes the as-synthesised BP to appear as micron and nanoscale agglomerates.

However, the BP is seen in a distinct environment in the photos e and f in the nitrogen environment. It is essential to comprehend the coating's microstructure in a nitrogen environment in order to evaluate its performance and stability in different settings (Hwang et al. 2019). This setting might mimic actual situations where the coating might be exposed to various gas compositions, which would affect how it behaves and how long it lasts.

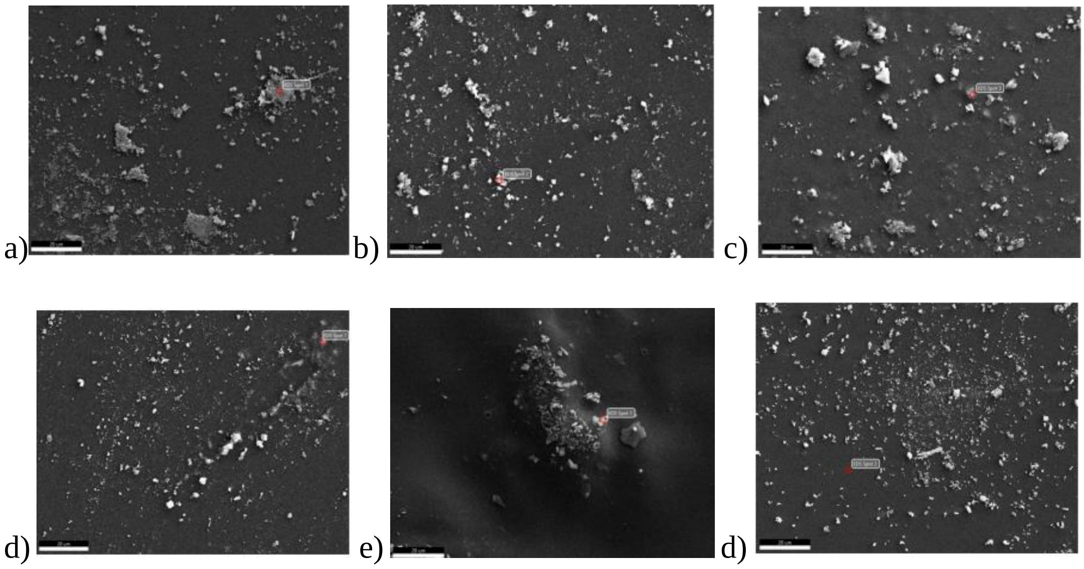


Figure 4.8 SEM images (Figure a until d are in ambient air environment while e and d are in nitrogen environment)

The material's natural condition is captured in a microscopic image of the surface that shows it before any coating has been placed as in the Figure 4.9, especially before using electrophoretic deposition (EPD) to apply an epoxy coating. All the important information regarding the surface texture is revealed in this photograph, including whether it is smooth, uneven, or has any flaws like scratches. This image provides as a baseline by showing the bare surface, which enables us to evaluate how effectively the upcoming coating could adhere and cover the substance. Moreover, it aids in the detection of any impurities or residues that can obstruct the epoxy coating's ability to adhere during EPD. To achieve consistent adherence and a strong protective layer, it is important to ensure a clean surface before coating.

Furthermore, the material's intrinsic faults or imperfections, such as fractures, voids, or structural abnormalities, may be found using this microscopic picture. These flaws might weaken the coating's integrity after it has been applied, which could lead to problems with adherence or lessen the protective layer's efficacy. Assessing the substrate's state in advance helps determine if it is appropriate for the epoxy coating to be applied using EPD, guaranteeing the best possible adherence and overall effectiveness of the protective layer.

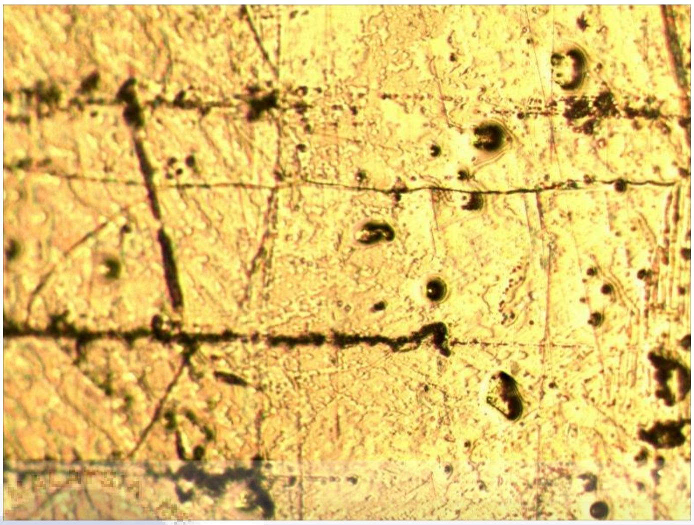
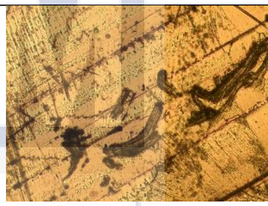
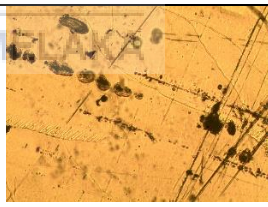
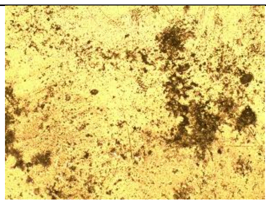
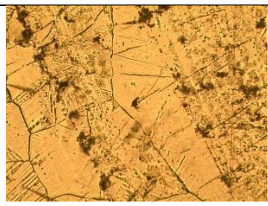


Figure 4.9 Microscopy structure for plain metal plate

As illustrated in Table 4.3 with subtle observations, one may discern the existence and properties of epoxy coatings deposited by electrophoretic deposition (EPD) at different voltages (30V, 40V, and 60V) in microscope pictures. The first clues to the existence of a coating are differences in texture and contrast with respect to the bare substrate. A discernible alteration in hue or feel on top of the substrate indicates the coating layer's presence. Variations in thickness and coverage are also visible characteristics in these pictures. Greater adhesion and better coverage are suggested by thicker, denser areas in microscope pictures taken at higher voltages, such as 60V. These regions also frequently indicate more considerable coating deposition. Lower voltage settings, on the other hand, may show thinner or less densely packed coatings, which would show up as lighter or more transparent patches on the substrate.

Analysing the dispersion of particles in the microscope pictures is essential for determining the quality of the coating. A homogeneous and equally distributed dispersion of particles throughout the substrate suggests consistent coverage and is indicative of a well-applied coating. Surface texture analysis is also very important. In microscope photographs, a smoother surface often indicates a more uniform and securely adherent coating. On the other hand, areas with more texture might be a sign of imperfections or insufficient coating, which could reduce the coating's ability to protect (Coto et al. 2021). By allowing for the evaluation of the effects of varying EPD voltages on the thickness, homogeneity, and general quality of epoxy coatings, these observations across voltage-varied microscopy pictures facilitate the optimisation of deposition parameters for the required coating qualities in real-world applications.

Table 4.3 Images of microscopy for ambient air environment

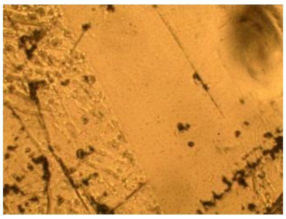
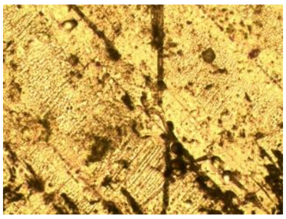
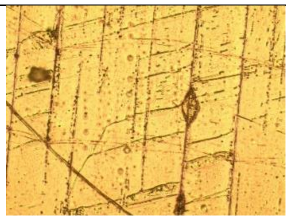
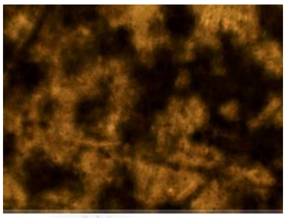
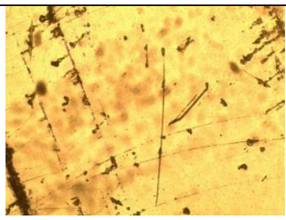
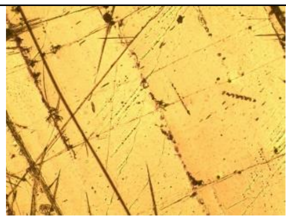
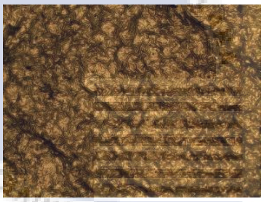
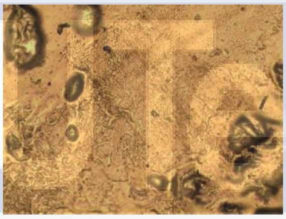
Ambient air environment	0.5 mol/ml	1.0 mol/ml	1.5 mol/ml
30V			
40V			
60V			

Electrophoretic deposition (EPD) of epoxy coatings at different voltages (30V, 40V, and 60V) in a nitrogen atmosphere is shown in Table 4.4 of microscopy pictures for nitrogen environment, which show different surface properties. Images taken at 60V in particular show far smoother surfaces than coatings placed at lower voltages (30V and 40V). This smoother look may indicate improved adherence and coverage over the substrate in the nitrogen environment, as it shows a more uniform and uniformly dispersed coating layer at the maximum voltage setting (Ghosh et al. 2023).

The microscope pictures taken at various voltages show how changes in the EPD voltage impact the epoxy coatings' shape and quality, particularly in the nitrogen environment (Aghili et al. 2021a). The more uniform and well-adhered coating suggested by the smoother surface seen at 60V suggests ideal deposition conditions. This smoothness might indicate fewer wrinkles or inconsistencies in the coating, which could lead to better protective qualities in an environment high in nitrogen.

These findings highlight how EPD voltage affects epoxy coating surface properties in a nitrogen atmosphere. The noticeable smoothness at 60V indicates that the coating quality is optimised. This shows how important voltage management may be in customising coatings for particular environmental circumstances and improving performance and protection in nitrogen-rich environments.

Table 4.4 images of microscopy for nitrogen environment

Nitrogen environment	0.5 mol/ml	1.0 mol/ml	1.5 mol/ml
30V			
40V			
60V			

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

The study's results were examined to give crucial information and analysis of Effect of Secondary Amine Oxidation on Dielectric Property of Epoxy Coating Deposition by Electrophoretic Deposition. The findings and discussion of the goal outlined in Chapters 1 through 4 led to the conclusions that were drawn.

5.1 Conclusion

The evaluation of secondary amine concentrations' effects on epoxy coating thickness using electrophoretic deposition (EPD) highlights the complex correlation between different amine concentrations and the final coating thickness. This study shows a direct association using controlled studies with amine concentrations that are consistently changed. Because EPD can be used to adjust coating properties and cover complicated structures consistently, it is an invaluable tool for managing coating thickness and uniformity. Its application to a wide range of industries is demonstrated. Comprehending these attributes facilitates the customization of EPD protocols to satisfy industrial coating specifications.

Examining secondary amine oxidation and its effects during electrophoretic deposition is another essential aspect of this study that will help clarify the workings and consequences of this process. Using Fourier-transform infrared spectroscopy (FTIR), the investigation of molecular interactions reveals characteristic peaks associated with different chemical interactions, revealing the molecular makeup and bonding. This in-depth knowledge of secondary amine oxidation enhances our understanding of molecular structures and how they affect coating properties by shedding light on the complex changes that take place during the coating process.

On top of that, an essential component of this research endeavor is the assessment of the optimized dielectric characteristics of epoxy coatings in conjunction with the utilization of Electrochemical Impedance Spectroscopy (EIS). EIS enables a thorough evaluation of dielectric properties and provides information on charge transport, capacitance, and impedance processes at different frequencies. This analysis makes the electrochemical behavior of the system more understandable by using analytical tools like Nyquist or Bode plots. This establishes EIS as an essential technique for characterizing and optimizing the electrical performance of materials and systems in a variety of applications, including corrosion research and the development of electrochemical devices. When taken as a whole, these diverse studies greatly advance our knowledge of and ability to use EPD in customized coating development and industrial processes.

5.2 Recommendations

It is advised to carry out in-depth performance testing of the epoxy coating under various environmental circumstances in light of the noted microstructural differences between inert and nitrogen environments. To assess the flexibility and robustness of the coating, this testing should include exposure to conditions that resemble real-world operational scenarios, such as nitrogen-rich and inert environments. Further investigation into the long-term stability and protective effectiveness of the coating by accelerated ageing or durability testing in these conditions would be highly beneficial. These evaluations would help determine how resistant and suitable the coating is for a range of operating situations, which would direct its optimal deployment in various industrial settings.

For the purpose of to enhance the comprehension of the ideal circumstances for obtaining the greatest epoxy coating by electrophoretic deposition (EPD), more research into microscope analysis is advised. Including more precise factors in the microscopy studies, such the coatings' surface roughness, interfacial properties, and particle size distribution, may provide important discoveries. A more thorough understanding of the morphology and structure of the coating may be obtained by investigating these tiny features at a more detailed level. This can help identify the exact circumstances that lead to the best coating characteristics. Furthermore, combining microscopy with other analytical methods like scanning probe microscopy or atomic force microscopy (AFM) may provide a more comprehensive knowledge of the surface characteristics and interfacial interactions of the coating. This thorough microscopic examination may be used as a guide to improve the parameters of the EPD process and create epoxy coatings that are more durable, consistent, and adhere well to a variety of industrial applications.

5.3 Sustainable Development

An area for sustainable material development is the investigation of the effects of secondary amine concentration on epoxy coatings using electrophoretic deposition. Through a knowledge of the complex interaction between coating thickness and amine levels, companies may be able to optimise their coating processes. Based on this knowledge, coating processes might be customised to minimise environmental impact, maximise resource efficiency, and cut down on material waste. This information promotes more economical material usage, which helps industrial applications adopt sustainable practices.

In addition, the examination of secondary amine oxidation during electrophoretic deposition in conjunction with Fourier-transform infrared spectroscopy (FTIR) study advances both sustainable material design and our understanding of coating properties. By comprehending molecular interactions and how they affect coating qualities, coatings with enhanced performance and durability may be developed, resulting in an extended lifespan. Longevity and improved performance in coating design minimises material consumption and waste by reducing the need for reapplications or replacements. This strategy supports the development of more robust, long-lasting materials that lessen resource depletion and environmental effect across a range of sectors, in line with the ideals of sustainable development.

5.4 Project Potential

The analysis of the influence of secondary amine oxidation on the dielectric characteristics of epoxy coatings produced by electrophoretic deposition offers a promising direction for future research and material science developments. Important information on the stability, electrical behaviour, and longevity of these coatings may be uncovered by this research, which is important for a variety of industrial applications.

Firstly, knowing how secondary amine oxidation affects epoxy coatings may improve their overall lifetime and function. Changes in the coating's chemical composition or cross-linking density might result from modifications to the amine structure, which could have an impact on the coating's dielectric characteristics. This might pave the way for the customisation of coatings with particular dielectric properties, which are essential in industries like electronics where accurate insulation qualities are crucial.

Second, understanding this link might help create coatings that are more durable and dependable. Understanding how oxidation affects the dielectric characteristics might help researchers develop ways to counteract or take advantage of these modifications. This might result in the development of epoxy coatings with enhanced resistance to electrical breakdown, decreased susceptibility to environmental deterioration, and greater dielectric strength—all of which are critical in high-voltage applications or challenging operating environments.

Furthermore, this research may open the door to improving the electrophoretic deposition procedure itself. Comprehending the impact of secondary amine oxidation on the dielectric behaviour of the coating may provide valuable information for altering the deposition settings or including additives that improve or stabilise particular dielectric characteristics. Such developments might simplify the coating procedure, increasing its effectiveness and adaptability to a range of industrial needs.

Finally, this study's findings may inspire new developments in multifunctional coatings. It could be possible to create coatings with several functions if secondary amine oxidation can be carefully controlled to customise dielectric qualities without sacrificing other desired qualities. These coatings might be highly dielectric and also have improved mechanical strength, resistance to corrosion, or thermal stability. As a result, they could be used in a variety of industries, including electronics, automotive, and aerospace.

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APPENDICES

APPENDIX A

Result of Turnitin


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APPENDIX B

Gantt Chart for the whole Project