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

NORSHAZLINA BINTI ROMZEI
B092010086

BACHELOR OF MANUFACTURING ENGINEERING TECHNOLOGY (PROCESS AND TECHNOLOGY) WITH HONOURS

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SESI PENGAJIAN: **2023-2024 Semester 1**

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Disahkan oleh:

Alamat Tetap:

NO. 155, JALAN PERIKANAN, TG. SEDILI,
81910 KOTA TINGGI,
JOHOR.

Cop Rasmi:

DR. LAU KOK TEE
Profesor Madya
Fakulti Teknologi & Kejuruteraan Industri & Pembuatan
Universiti Teknikal Malaysia Melaka

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Date : 25 January 2024

DEDICATION

I would want to dedicate my thesis to my incredible family, who have always been there for me, offering everlasting love, strength, and encouragement. Your unfailing support, unconditional love, and consistent encouragement throughout my academic career. Thank you for sticking by me through the long hours of research, late nights, and several sacrifices. Your faith in me has been my driving force, and I am grateful for the sacrifices you have made to make my aspirations a reality. This work is dedicated to you as a sign of my heartfelt thanks and appreciation for everything you have done for me.

This report is also dedicated to my wonderful supervisor, Associate Professor Ts Dr. Lau Kok Tee, whose advice and knowledge have impacted my intellectual evolution. Your devotion to study and thirst for knowledge have encouraged me to stretch the limits of my ability. Their mentoring has influenced my thinking and pushed me to achieve new heights. I appreciate their patience, support, and dedication to my academic achievement. I'm also appreciative for the opportunity you've given me and the vital skills I've learned under your tutelage.

To Miss Nurhaliana Shazwani Binti Mohd Halim and the rest of the study team, thank you for your assistance and support throughout this difficult undertaking. Whose willingness to donate their time, expertise, and experiences made this research feasible. Your encouragement, insightful talks, and shared experiences have made this journey more meaningful and enjoyable. Your insights have expanded my comprehension and been critical to the accomplishment of my thesis. I am glad for the friendship we have formed and the memories we have made together.

Finally, this thesis is dedicated to those of you who have helped shape my academic and personal development. Your support, contributions, love, and trust in me have all been driving factors in my accomplishments. I am thrilled to dedicate my work to each of you individually. Thank you for being a part of my journey and leaving such a lasting impression on my life.

ABSTRACT

This study looks at the effect of secondary amine content on the thickness of epoxy coatings applied using electrophoretic deposition (EPD). Epoxy coatings play an important role in corrosion resistance and durability; thus, appropriate coating thickness is critical for efficacy. This work fills a major knowledge gap in understanding how different secondary amine concentrations affect coating characteristics. The study goal is to investigate the effect of secondary amine concentration on epoxy coating thickness, optimise EPD parameters for improved coating microstructure, and evaluate dielectric characteristics using electrochemical impedance spectroscopy (EIS). The technology includes substrate preparation, epoxy solution formulation, precision EPD processing, curing, and sophisticated characterization methods including EIS, Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). The results show that an ideal thickness for 0.5 ml and an EPD voltage of 30 volts improve layer homogeneity, adhesion, and deposition efficiency. The EIS study also determines the optimal dielectric constant for a secondary amine concentration of 1.5 ml and an EPD voltage of 40 volts. These findings offer important insights for designing epoxy coatings for specific applications, emphasising the practical importance of the observed characteristics.

ABSTRAK

Kajian ini melihat kesan kandungan amina sekunder terhadap ketebalan salutan epoksi yang digunakan menggunakan pemendapan elektroforetik (EPD). Salutan epoksi memainkan peranan penting dalam rintangan kakisan dan ketahanan; Oleh itu, ketebalan salutan yang sesuai adalah penting untuk keberkesanan. Kerja ini mengisi jurang pengetahuan utama dalam memahami bagaimana kepekatan amina sekunder yang berbeza mempengaruhi ciri salutan. Matlamat kajian adalah untuk menyiasat kesan kepekatan amina sekunder pada ketebalan salutan epoksi, mengoptimumkan parameter EPD untuk struktur mikro salutan yang lebih baik, dan menilai ciri-ciri dielektrik menggunakan spektroskopi impedans elektrokimia (EIS). Teknologi ini termasuk penyediaan substrat, formulasi penyelesaian epoksi, pemprosesan EPD ketepatan, pengawetan, dan kaedah pencirian yang canggih termasuk EIS, spektroskopi inframerah transform Fourier (FTIR), dan mikroskop elektron pengimbasan (SEM). Hasilnya menunjukkan bahawa ketebalan ideal untuk 0.5 ml dan voltan EPD sebanyak 30 volt meningkatkan homogeniti lapisan, lekatan, dan kecekapan pemendapan. Kajian EIS juga menentukan pemalar dielektrik optimum untuk kepekatan amina sekunder 1.5 ml dan voltan EPD sebanyak 40 volt. Penemuan ini menawarkan pandangan penting untuk mereka bentuk salutan epoksi untuk aplikasi tertentu, menekankan kepentingan praktikal ciri-ciri yang diperhatikan.

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LIST OF SYMBOLS AND ABBREVIATIONS

T_g	-	glass transition temperature
ºC	-	degrees Celcius
DGEBA	-	diglycidyl ether of bisphenol A
VOCs	-	volatile organic compounds
EPD	-	electrophoretic deposition
DFT	-	density functional theory
FTIR	-	Fourier transform infrared
XDR	-	x-ray diffractometer
SEM	-	scanning electron microscope
EEW	-	epoxy equivalent weight
DMA	-	dynamical mechanical analysis
DDS	-	diaminodiphenyl sulfone
DoB	-	degree of branching
HAp	-	hydroxyapatite
ATR	-	attenuated total reflection
UF	-	urea-formaldehyde
WEP	-	water-borne epoxy
GO	-	graphine oxide
PA	-	phytic acid
kV	-	kilovolt
EDAX	-	energy dispersive absorption X-ray
NaCl	-	sodium chloride
K	-	Kelvin
QC	-	quality control
ml	-	milliliter
mm/s	-	millimeters per second
µm	-	mikrometer
ω	-	omega
ε	-	dielectric constant
ρ	-	density
m	-	mass of coating substrate

A	-	Area of the coating region
t	-	thickness of the coating substrate
ε_o	-	permittivity of free space
Z’	-	real part of impedance
Z’’	-	imaginary part of impedance
f	-	frequency

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CHAPTER 1

INTRODUCTION

This chapter contains a detailed overview of the project's background, the problem statement, the research objective, and the scope of the research. The study explores the effect of secondary amine concentration on epoxy coating thickness using electrophoretic deposition (EPD). Understanding this relationship is crucial for optimising protective performance in industries. The research aims to determine optimal thickness conditions while maintaining coating integrity, providing practical insights for coating technology, and enhancing substrate preservation and corrosion prevention in various industries.

1.1 Background

This study investigates the impact of secondary amine concentration on the thickness of epoxy coatings through electrophoretic deposition (EPD). Epoxy coatings are crucial for their corrosion resistance and durability, and achieving optimal coating thickness is essential for their effectiveness. Secondary amines, as additives in epoxy formulations, influence coating properties. EPD is chosen for its efficient production of uniform and adherent coatings. The research aims to address a research gap and contribute to the advancement of coating technology and tailored solutions for enhanced industrial protection.

Organic coatings are thought to be one of the most promising ways to protect metals against corrosion. Epoxy resins are frequently used as coatings due to their stable chemical characteristics, superior corrosion resistance, remarkable adhesion, minimal curing shrinkage, high tensile strength, and flexural strength (Pourhashem et al., 2017). As explained by Xiang and Xiao (2020), the worldwide market for epoxy resin is being pushed by increased demand in industries such as civil engineering, chemical, aerospace, etc. Figure 1.1 shows the global demand for epoxy coating.

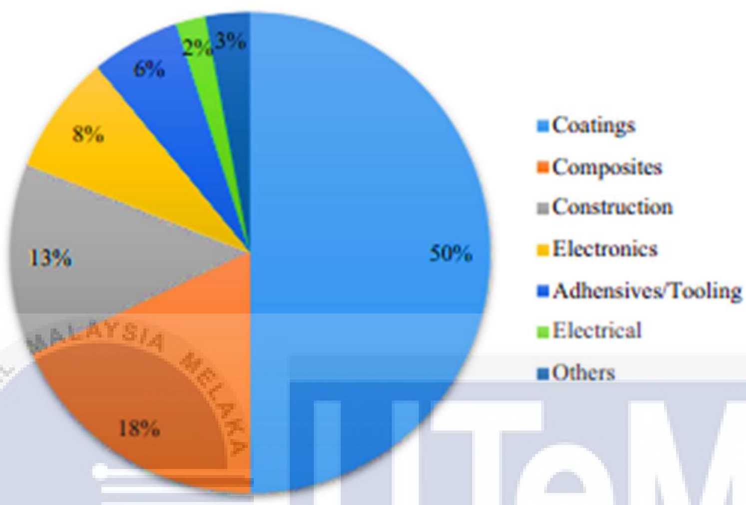


Figure 1.1 Global epoxy coating demand (Xiang and Xiao et al., 2020)

Most epoxy resins on the market are polymers of diglycidyl ether of bisphenol A (DGEBA). Based on Saba et al (2016), when these polymers combine with the hardener, the epoxy resin cures and transforms into a thermosetting polymer. The fundamental property of the epoxy monomer is the oxirane functional group of DGEBA (a three-membered ring composed by two carbon atoms and one oxygen atom. Carbon atoms in the ring are electrophilic because carbon and oxygen have a different electronegativity. Because of its high strain, this atomic arrangement is more reactive than typical ethers (Saba et al. 2016).

Aside from that, according to Jin et al. (2022), epoxy resin is classified into two types which are two-package binder and three-package binder. The main distinction is that the curing temperature regulates the setting of the epoxy resin, whereas the promotor regulates the setting of the unsaturated polyester resin. Aside from the kinds, there are two sorts of epoxies curing agents: apparent curing agents and latent initiators. Curing epoxy resins using apparent curing agents is achieved by addition polymerization or ion polymerization (Chen et al. 2020).

In addition, according to D'Almeida and Monteiro (1996), a higher processing temperature can yield a higher degree of cure in epoxy resins. As a result, the polymeric structure has a larger crosslink density, is likely stiffer, and has a higher ultimate strength. Apart from temperature, there are many other factors that affect the results of the coating process, such as the hardener types and the curing time. All the elements that contribute to coating processing must be managed and continuously monitored in order for the coating produced to be durable and of good quality.

When compared to other typical thermoplastic or thermoset polymers, epoxy resins have significant benefits. There are various advantages to using this coating process. Among these are corrosion resistance (anticorrosive), long shelf life, impact resistance, improved mechanical and fatigue strength, outstanding moisture and chemical resistance, minimal shrinkage during curing, and improved electrical properties. One of the most important benefits of employing epoxy resins is that no volatile organic compounds (VOCs) are produced (Saba et al. 2016). It is critical because it has the potential to harm our ecosystem if it occurs on a regular basis.

In other ways, it is the best suited for the coating process due to its benefits and the properties of epoxy resins. Consequently, because this procedure is one of the most promising ways to shield metals from corrosion, it has piqued the interest of our industries. However, the influencing aspects must also be considered in order to get a superior coating process finish. The ability of epoxy resins in coating processes cannot be disputed because there have been several successful cases.

This research explores the electrophoretic deposition (EPD) process, a popular method for producing coatings, films, and composite materials. EPD involves charged particles moving in an electric field and deposition on a solid substrate. Factors such as particle selection, size, distribution, suspension medium, dispersants, pH control, suspension concentration, stability, ageing, and cleaning are crucial. EPD offers selective, controlled deposition, uniform thickness, and versatile applications, ensuring consistent thickness for functional or aesthetic reasons on complex substrates.

1.2 Problem Statement

The charge on the particle, as well as the electrophoretic mobility of the particles in the solvent under the effect of an applied electric field, are the primary driving forces for electrophoretic deposition (EPD) (Besra and Liu 2007). According to Besra and Liu (2007), charged particles in a suspension are deposited onto an electrode under the influence of an applied electric field in the mechanism of EPD. In the case of epoxy coatings, the charged particles are typically epoxy resin particles dispersed in a liquid medium containing a solvent and other additive. Meanwhile, amine concentration study refers to the amount or concentration of secondary amines in a given substance or solution. They react with the epoxy functional groups to initiate crosslinking and curing, resulting in a solid, durable coating.

The effect of using different amine concentrations in coatings can vary depending on the specific type of amines (primary, secondary, or tertiary) and the coating system being employed. The differences of amines classes will produce different coating thickness. Figure

1.2 gives an overview of the amine reactivity that occurred from relevant data sources. This is due to the differences in each class of amines. Some general effects that can be observed are related to the curing and crosslinking. The concentration of amines can affect the curing process and the degree of crosslinking in the coating. As stated in Mora et al. (2020), for example, used density functional theory (DFT) approaches to investigate the mechanism of the epoxy-amine curing process. They emphasised that secondary amines would respond faster than primary amines (Mora et al. 2020).

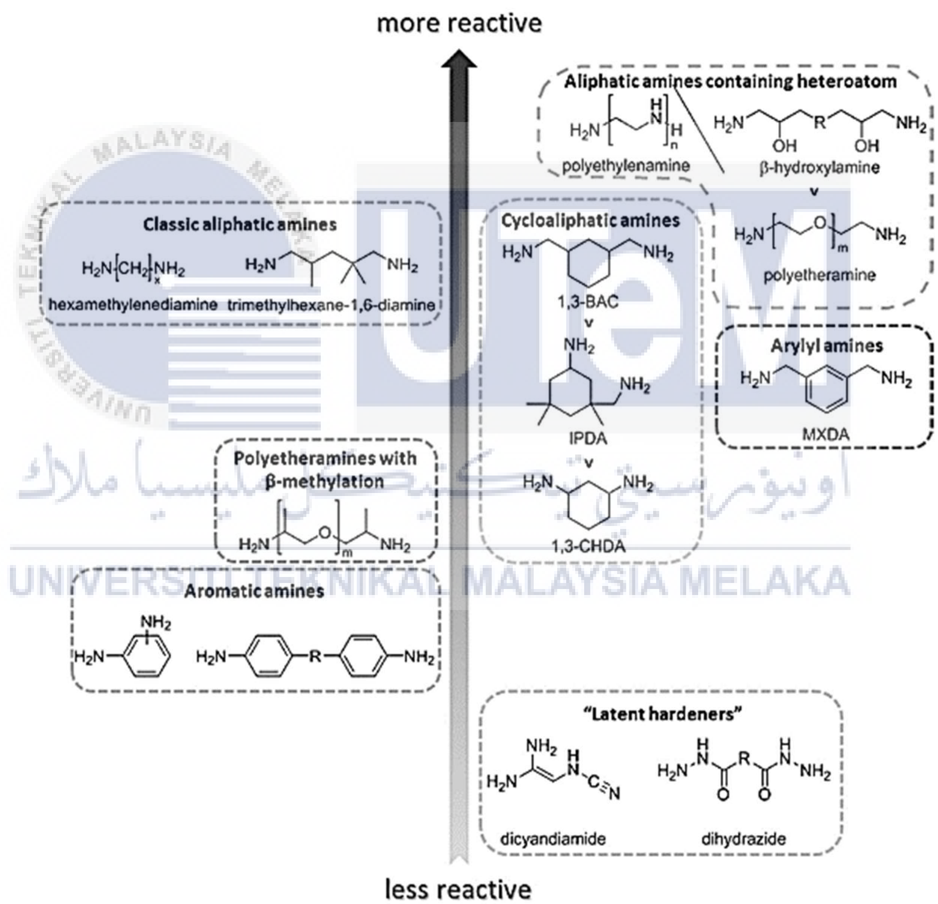


Figure 1.2 Summary of the amine reactivity scale (Mora et al. 2020)

Adjusting the amine concentration with various values in the epoxy coating process is very essential to identify how far the effect of the secondary amine concentration influences the coating thickness. Based on the results obtained, we can determine the differences in thickness if they mix with different values of concentrations. This is most important because our research is about the effect of secondary amine concentrations on the thickness of epoxy coatings by electrophoretic deposition (EPD). Based on the data received, we can conclude if the concentration value used increases, what will happen to the thickness and the dielectric property of epoxy coating, and the time taken for the curing process.

1.3 Research Objective

a) To investigate the influence of secondary amine concentration on epoxy coating's thickness deposited by electrophoretic deposition (EPD).

b) To determine the effect of parameters uses in electrophoretic deposition (EPD) in order to get a better coating microstructure.

c) To assess the dielectric property of the epoxy coating by using electrochemical impedance spectroscopy (EIS).

1.4 Scope of Research

The scope of this research are as follows:

This research focuses on investigating how varying concentrations of secondary amines in epoxy formulations influence the thickness of coatings deposited through electrophoretic deposition (EPD). The study involves systematically adjusting secondary amine levels to identify optimal conditions for achieving the desired coating thickness. According to the Gibson (2017), the primary and secondary amines, in general, operate as reactive hardeners, whereas the tertiary amines are catalytic. This is most likely due to the fact that secondary amine hydrogens are two to three times less reactive than primary amine hydrogens in aromatic amines (Carrasco et al. 2005). It encompasses a detailed examination of the EPD process, employing precise measurement techniques to quantify coating thickness and assess coating integrity and adherence. Other than that, when switching from a primary amine to the matching N-methyl secondary amine, aliphatic amine reactivity rose consistently (Marsella and Starner, 2000). As a result, it is obvious that secondary amines can influence the thickness of the epoxy coating. This study aims to optimise coating parameters in industrial contexts, focusing on precise control over epoxy coating thickness for enhanced performance and durability. Multiple procedures will be employed to determine the final coating thickness, with efficacy tested through changing secondary amine ratios.

Other than that, this research seeks to determine the impact of key parameters in electrophoretic deposition (EPD) on the coating microstructure. The study analyses the microstructural characteristics of suspensions through systematic variations in parameters like voltage, deposition time, and composition using techniques like scanning electron microscopy and optical microscopy. The study aims to identify optimal conditions for superior coating microstructure, balancing mechanical, electrical, and thermal properties. It will deepen understanding of EPD parameters and microstructure relationships, guide process optimisation, and enhance coating quality and durability across various industries. In this research, the parameter that will be manipulated is the voltage used in the EPD process. According to Aghili et al. (2021), when the EPD process is conducted at various voltages, uniform coatings of varying thicknesses will be produced.

This research aims to comprehensively assess the dielectric properties of epoxy coatings through the application of electrochemical impedance spectroscopy (EIS). The scope involves using EIS techniques to measure dielectric behaviour across different frequencies, considering factors like epoxy formulation, coating thickness, and curing conditions. According to Hernández et al. (2020), EIS is a successful novel electrochemical method that has undergone tremendous evolution, becoming a vital analytical tool in materials science research. Additionally, the study will explore the correlation between EIS data and the structural integrity of the coatings. By examining the impact of dielectric properties on overall performance, the research aims to provide insights crucial for optimising epoxy formulations and coating processes. The study highlights the importance of understanding and improving dielectric performance in industrial and technological settings to ensure the effectiveness of epoxy coatings in electrical insulation applications.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction of Epoxy Resin

Epoxy resins are a kind of polymer that is used practically every day. According to Rehim and Turky (2022), they were first synthesised in 1891 but were not commercially available until the 1940s. Epoxy refers to pre-polymers and monomers that include an epoxy group, either within the molecule or as a terminal group. Epoxy resins are used as intermediates in UV coatings and as adhesion enhancers in certain coatings. Other than that, according to Ahmadi (2019), epoxy resin is the most potential as an adhesive and the choice of most engineers in the coating industry due to its long-life span. Figure 2.1 depicts the synthesis process and how epoxy resin is mixed to produce the emulsion. Epoxy resin, also known as polymer resin, is defined by Chen et al. (2020) as a material having numerous epoxy groups that can change its physical state from liquid to solid.

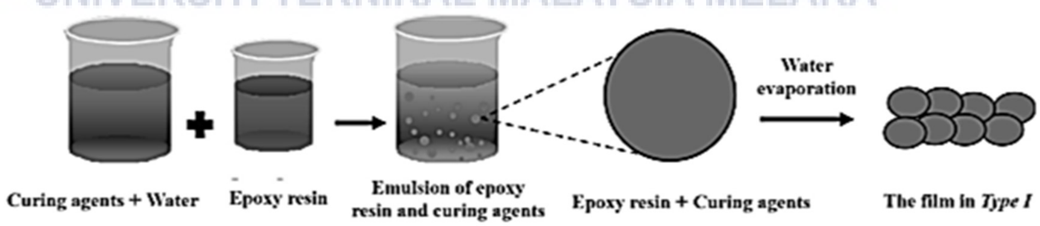
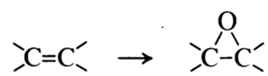
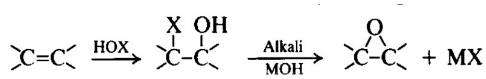
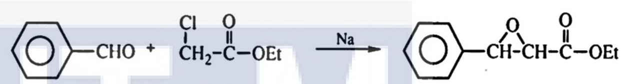
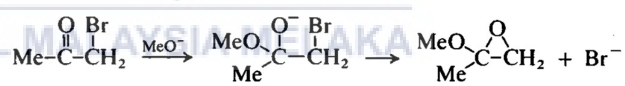
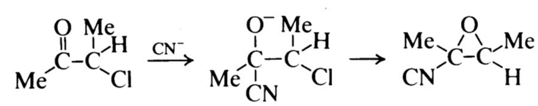
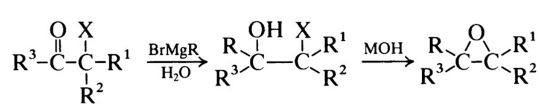


Figure 2.1 The synthesis of a typical Type I aqueous epoxy resin (Xiang and Xiao et al., 2020)

2.1.1 The Chemistry of Epoxy Group

According to Ellis (1993), Wurtz discovered oxirane, the parent molecule, in 1859 by synthesising it from ethylene chlorohydrin via an aqueous alkali process. Epoxy resins, widely used in adhesives and coatings, are created using a chemical that allows for customisation of their characteristics through epoxide precursors and curing agents. Understanding epoxy group chemistry is crucial for optimising formulations and applications across various industries. Based on the review article, Table 2.1 summarises the various methods for synthesising epoxy rings.

Table 2.1 An overview of epoxide synthesis techniques (Ellis et al., 1993)

Method	Types	Diagram
Oxidations of alkenes	Direct Oxidation	
	Inorganic Oxidants	
	Organic Peroxides	
	Hydrogen Peroxide	
From Halohydrin	Hypohalous addition to alkene → cyclodehydrohalogenation	
From α-Halocarbonyl Compounds	Darzen's Condensation	
	Reduction	
	Addition of Alkoxide Ion, Followed by Ring Closure	
	Addition of Cyanide Ion	
	Grignard Reagent	

Oxidations of alkenes, halohydrin production, and halocarbonyl compounds are the synthetic ways for producing epoxides. The technique of choosing is determined by specific aims, substrate reactivity, and stereochemistry needs. Epoxides are produced by oxidizing alkenes with peracids or organic hydroperoxides, used in epoxy resins manufacturing, with oxidising agent and reaction conditions determined by substrate. According to Morsch et al. (2020), a high link between aliphatic amine oxidation products and those detected in resins during the early phases of thermal ageing.

Based on the table, the halohydrin synthetic method is a technique for synthesising epoxides from halohydrin precursors. A base deprotonates a hydroxyl group, producing an alkoxide ion and attacking the carbon atom, resulting in the formation of a three-membered oxirane ring. This approach is frequently utilised in organic synthesis and the manufacturing of epoxy resins. The enantioselectivity of these enzymes has sparked a lot of interest in the generation of optically pure epoxides and halohydrins (Gul et al. 2020).

The synthesis of epoxides from halocarbonyl compounds includes a nucleophilic assault that results in the creation of an enolate ion and subsequent reaction with an electrophile, adding to the variety of techniques accessible in organic synthesis. According Gontala et al. (2021), the formation of radical intermediates from halocarbonyl compounds using a visible light photocatalyst, as well as the addition reactions of the halocarbonyl radicals with diverse alkenes, have been extensively explored.

2.1.2 The Synthesis and Manufacture of Epoxy Resins

Epoxy resins are used in the construction, electronics, and automotive industries due to their adhesion strength and chemical resistance. They are synthesised by reacting epichlorohydrin with bisphenol, which is controlled by adjusting reactant stoichiometry and conditions. Continuous developments in synthetic methods enhance their versatility and performance. The state-of-the-art involves the synthesis of various polymers, including epoxy resins, polyurethanes, polyacrylics, and cyanate resins, through reactions that open the way to a wide variety of polymers (Eid, Ameduri, and Boutevin 2021). According to

Ellis (1993), the most crucial processes to produce epoxy resins are halohydrin dehydrohalogenation and alkene epoxidation using peracids or their esters.

In addition, the absence of particular knowledge on bio-laminate composite creation compels the search for appropriate manufacturing alternatives, since the manufacturing method has a substantial influence on the mechanical performance of these materials (Torres-Arellano, Renteria-Rodríguez, and Franco-Urquiza 2020). Then, according to Kim et al., (2021), to lower costs, epoxy resin is made using polymerization with bisphenol A and epichlorohydrin condensation, which necessitates good ECH recovery or separation. Processes that combine distillation and pervaporation can improve energy efficiency and product competitiveness.

2.1.3 Pack System of Epoxy Resin

A system is an element that interacts with each other to produce a whole that is united in the end according to the rules that have been set. A system is one of the most important things to make sure the process can be done systematically and smoothly. In epoxy resin, there are two pack systems: the one-pack system and the two-pack system.

For one-pack systems, due to their very slow reaction at ambient temperature or no reaction at all, the resin and curing agent can be mixed earlier than the actual time (Daniel Suckley, 2021). If heat is introduced to this one-pack system, the process of curing will be accelerated. When this curing reaction is completed, the finished polymer will be perfectly formed. According to Xu et al. (2020), epoxy resin with a one-pack system will remain reactively inert throughout storage, but it is effective when in use.

It is different for the two-pack system, where the resin and curing agent cannot be mixed earlier than the actual time. When these resins and curing agents are mixed, the beginnings for the formation of the final polymer are formed As a result, the applicator has a limited amount of time after combining the resin and curing agent to utilise and apply the material before the system's rising molecular weight gets too high and it can no longer be employed (Daniel Suckley, 2021).

2.1.4 Characterization of Uncured Epoxy Resins

The study evaluates the chemical composition, viscosity, reactivity, pot life, solubility, compatibility, molecular weight, thermal characteristics, and shelf-life stability of uncured epoxy resins using spectroscopy, chromatography, and thermal analysis. The exothermic reaction peaks differ owing to the protection of epoxy resin shell materials, which prohibits contact between microcapsules containing ethylenediamine (EDA) and uncured epoxy resin. As the temperature rises, EDA infiltrates and volatilizes from the interior microcapsules (Yuan et al. 2019). As mentioned by Ellis (1193), different species with varying n values are present in resin, with the concentration of epoxy groups being a crucial approximation for characterizing the resin, as it is used to calculate the required hardener amount.

2.1.4.1 Chemical Analysis

The chemical analysis of epoxy resin employs a variety of methods to determine its composition, structure, and thermal characteristics. Scanning electron microscopy (SEM) analysis of raw surface RS indicated that increasing the glass powder amount increased the silicon mass content in the coating and substrate top zone, indicating silicon penetration into substrate pores. SEM maps were generated to visualise individual components (Chowaniec, Sadowski, and Żak 2020).

2.1.4.2 Quantitative Analysis

Some techniques used to quantify epoxy resin are titration, gas chromatography, UV-visible spectrophotometry, and nuclear magnetic resonance. Each technique is calibrated to meet specified criteria for exact analysis. According to Bell et al., (2021), to identify and quantify chemicals from untreated and UV-A-aged coating systems, the researchers used an HPLC system and a high-resolution mass spectrometer. FTIR is a viable method for quantitatively analysing the curing process of DGEBA, with the near-IR region offering accurate findings for determining oxirane and amine group concentrations (Cholake et al. 2014). Based on the finding of Min et al., (1993), the combined epoxy and primary amine group band was identified at 4535 cm^{-1} using near infrared spectroscopy for quantitative investigation of cure processes in diglycidylether of bisphenol A (DGEBA).

2.1.5 Types of Epoxy Resin

Epoxy resin has a lot of type where it will be blended with curing agent to produce an emulsion. Table 2.2 displays the examples of epoxy resin, its curing agent, and the application. According to Ozeren Ozgul and Ozkul (2018), to create an epoxy combination, three types of epoxy resins were used are diglycidyl ethers of bisphenol A and F, and a third generated by combining the two resins, as well as three types of reactive diluents based on glycidylether.

Many varieties of novel epoxy resins have been produced from epoxides since the 1930s, when the synthesis of epoxy resins was patented (Agarwal and Agarwal, 2019). Behalf of that, as identified by Sukanto et al. (2021), epoxy resins are classified to two families known as aromatic epoxy saturated ring epoxy (aliphatic) and nonaromatic saturated ring epoxy (cycloaliphatic). The monomer diglycidylether of bisphenol A (DGEBA) is an example of aliphatic epoxy family. For cycloaliphatic epoxy resin, the example is (3,4-

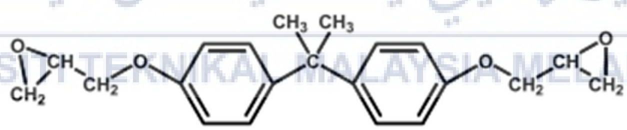
epoxycyclohexylmethyl-3',4'-epoxycyclohexane carboxylate) where its employed as the epoxy matrix (Tomasi et al. 2018).

Table 2.2 Epoxy resin and curing agents commonly used in productions and researches (Sukanto et al. 2021)

Resin Epoxy	Curing Agent	Application
DGEBA	Cycloaliphatic diamine, bis *ρ*-amino cyclo-hexyl methane	CF Sizing
DGEBA	Aliphatic polyether triamine	CFRP Sheet
DGEBA	Anhydride	CF Sizing
DGEBA	Combination of diethylene-triamine and triethylenetetramine	Civil Construction

2.1.6 Structure of DGEBA

Consequently, diepoxides are molecules that include two oxirane groups in a molecule, such as DGEBA (Capricho, Fox, and Hameed 2020). As a result, DGEBA exhibited long-term stability in liquid form at room temperature while curing fast under heating, exhibiting temperature-dependent latent curing behaviour (Xu et al. 2020). Figure 2.2 is depicted the chemical structure of DGEBA.



DGEBA

Figure 2.2 Chemical structure of DGEBA (Liu et al. 2023)

As explained by Sukanto et al. (2021) the rise in crosslinking density has led to the ideal structure of the epoxy crosslinking network. In addition, the molecular structure of organic and inorganic components can be determined using fourier transform infrared spectroscopy (FTIR). It is one of the most adaptable analytical methods for non-destructive chemical evaluation of geological materials (Jilani et al. 2020).

2.1.7 Thermal Properties of DGEBA

DGEBA is one of the epoxy resins with high mechanical strength, great adhesion, strong chemical and corrosion resistance, high heat resistance, dimensional stability qualities, and endurance in hostile situations (Talukdar et al. 2022). Properties like this aid today's engineers in determining the proper option to guarantee that the epoxy coating manufactured may persist for a long time and generate an effective coating. According to Khan and Chavan (2019), the glass transition temperature, T_g of DGEBA is 56.23 ºC. T_g also known as the point of "melting of amorphous material".

Furthermore, Hsissou et al. (2021) expressed the opinion that Tg is an essential indication to determine the thermal characteristics of epoxy resin, which directly impacts the performance and process performance of the material. To evaluate how the polymer product characteristics develop as the reaction continues, systematic investigations under varied electric field circumstances have been done by combining the dielectric and exclusion chromatography approaches (Tu et al. 2022).

2.1.8 Mechanical Properties of DGEBA

There are a lots of information that we can gains from the mechanical properties such as the behaviour of the material. In this research, the mechanical properties such as tensile and adhesive strength, flexural, and impact of DGEBA composites increase initially and subsequently drop when the epoxy equivalent weight (EEW) decreases (Mi et al., 2022).

The majority of experimental methodologies, such as dynamical mechanical analysis (DMA), have been used to investigate the molecular dynamics of epoxy resins exposed to high energy irradiation (K. Chen et al. 2019). Dynamic mechanical analysis (DMA) was used to assess the mechanical properties of cured epoxy resin (W. Zhou et al. 2022). Table

2.3 is the summarized of dynamic mechanical analysis of DGEBA. According to Liu et al. (2023), the results demonstrated that the epoxy-rich mixes produced tight and brittle macromolecular structures and exhibited brittle behaviour.

Table 2.3 DMA analysis of a DGEBA epoxy matrix (Dhanapal, Kumar, and Ranjitha et al., 2022)

DGEBA	Storage Modulus (GPa)	Glass transition temperature, T_g (ºC)
100	2.5	153
80	3.8	160
80	4.3	165
80	5.6	172
80	4.4	161

2.1.9 Synthesis of DGEBA

DGEBA type resins, the most common of which are synthesized from polyhydric compounds like bisphenol-A and epichlorohydrin (Capricho, Fox, and Hameed 2020). Figure 2.3 shown the synthesis process of DGEBA from bisphenol-A and epichlorohydrin. According to Xu et al. (2022), a 100 mL three-necked flask with a pressure-equalizing addition funnel, thermometer, cooling condenser, magnetic stirrer, and oil bath was used for the resin synthesis experiment. In a nitrogen atmosphere, phenol precursors were melted, hydrochloric acid was added, and aldehyde was added drop by drop. The resin was rinsed three times with distilled water before being dried at 125 degrees Celsius for 24 hours.

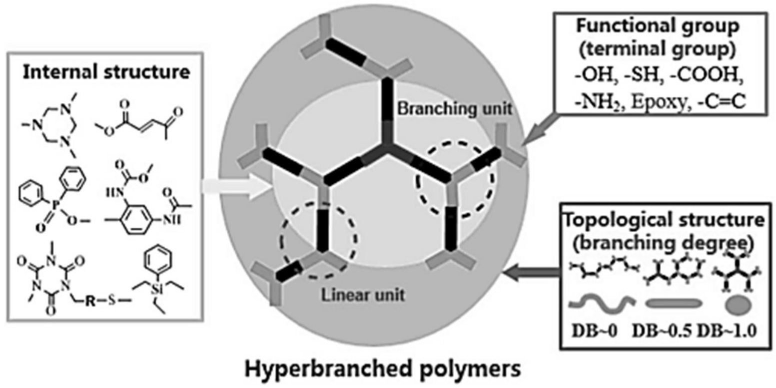


Figure 2.3 Hyperbranched epoxy resin synthesis techniques and structural regulation (Mi et al., 2022)

2.2 Advantages of Epoxy Coating

Nowadays, epoxy resin is a new technology that is commonly applied in engineering compared to traditional methods of coating. There are many advantages to the coating process with epoxy resin. One of the advantages of epoxy coatings is that they have dual functions: bactericidal and antiadhesive. Other than that, epoxy coating also has chemical inertness, a low friction coefficient, and high corrosion resistance (Jin et al., 2022). Moreover, Robert et al. (2020) stated that technology's capacity to store powder-epoxy preforms for subsequent usage is a significant competitive advantage.

2.2.1 Volatile Organic Compounds (VOCs) Contents

The epoxy/amine coating is a very challenging task. It is because the process carried out must meet the regulatory standards of Volatile Organic Compounds (VOCs) and improve the cost or performance ratio of the coating. VOCs have a deleterious influence on the human body, particularly in children due to insufficient physical and cognitive development, and can target multiple systems depending on whether exposure is acute or chronic (Ozge et al., 2021). Liang et al. (2021) found that samples with VOC concentrations less than 10 g L^{-1} contributed the most to water-based waterproof coatings, accounting for 96% of the total. From this article, we can determine that solvent-based adhesives have greater VOC levels than water-based and bulk adhesives. Table 2.4 depicted the contents of VOCs in architectural adhesives.

Table 2.4 Architectural adhesive VOC content (Liang et al. 2021)

Adhesive Type	Subordinate	Number of Samples	VOC Content Range	VOC Comprehensive Content	Standard Limit	
					2014	2008
Solvent-based adhesives	Neoprene	41	483-840	621	680	700
	Acrylic	8	412-775	539	600	700
Water-based adhesives	Polyvinyl Acetate	40	9-84	43	100	110
	Formal	18	58-179	107	150	350
Bulk-based adhesives	Epoxy	-	75	75	50	100
	Silicon	18	11-195	74	100	100

2.3 Amines as a Curing Agent

According to Babahan-Bircan et al. (2022), amines are often utilised as hardeners in epoxy coating systems due to their excellent mechanical durability, chemical resistance, and good adhesive qualities. Epoxy curing agents are classified as either additive or catalytic. Formers, especially amines, have a direct role in polymerization and cross-linking structures. Most amines are toxic before curing, necessitating modification. Apparent activation energy between 50 and 70 kJ/mol is the typical range of epoxy-amine curing processes (Y. Huang et al. 2019). Murmu et al. (2019) identifies that aliphatic and aromatic amines were used to cure double Schiff base epoxy (DSBE) to create a strong crosslinked structure. As we know, curing is the process of hardening a polymer material by cross-linking it using heat or chemical additions. Apart from that, amine is reacted as a hardener in epoxy-amine coating. Amine has three categories: primary, secondary, and tertiary amines.

2.3.1 Structure of Secondary Amine

Secondary amines are one of the category of amines' classes. In low-stoichiometric ratio samples, the epoxy-secondary amine reaction and etherification occurred, resulting in molecular structural development beginning at diaminodiphenyl sulfone (DDS), acting more like a tetrafunctional component, resulting in a branching dominating structure with a greater degree of branching (DoB) (Odagiri et al. 2021). The chemical structure of secondary amines is depicted in Figure 2.4. As identified by Grunenberg et al. (2021), postsynthetic functionalization is enabled by secondary amines on pore walls, resulting in customised covalent organic frameworks with improved hydrolytic stability. In addition, because of the properties of secondary amine, it was used to prevent the development of imine bonds (Jiang et al. 2019).

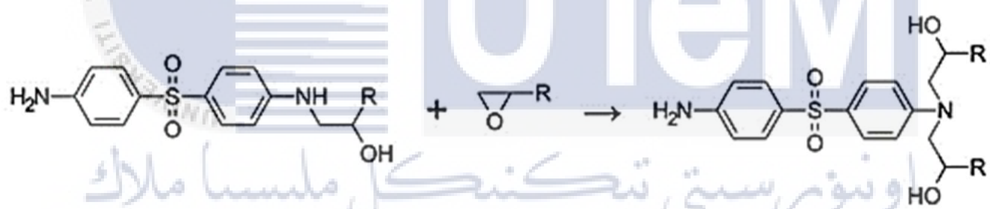


Figure 2.4 Chemical Structure of Secondary Amines (Odagiri et al. 2021)

2.3.2 Characterisation of Secondary Amine

The electronegativity of secondary amines decreases due to inductive effects, while primary amines are more reactive to electrophilic chemicals. So, it is logical that primary amines will react faster than secondary amines. However, Mora et al. (2020) suggest that secondary amines react faster compared to primary amines. C. Huang et al. (2020) pointed out that, as curing progresses, the amount of epoxy groups and primary amines decreases, but the rate of secondary amine production surpasses consumption, resulting in an increase in secondary amine quantity.

2.3.3 Composition of Secondary Amine as a Curing Agent

According to Ekbrant et al. (2021), temperature, solvent, electrical, amine type, steric hindrance, and resin mixture homogeneity all have an effect on the kinetics of epoxy curing. Epoxy equivalent weights (EEW) are used to calculate epoxy quantities in commercially relevant systems. Based on this research, we know that secondary amines are monofunctional.

Curing agents have a big influence on epoxy resin curing because they change the kinetics, reaction rate, gel time, degree of cure, viscosity, and cycle. Their stoichiometric connection has a considerable impact on physical and mechanical properties. Estimating and optimising curing agent and resin volumes is critical for attribute evaluation. In secondary amine-cured systems, hardeners are often used at a near-stoichiometric ratio (Agarwal and Agarwal 2019).

Furthermore, according to Delannoy et al. (2022), DGEBA and amine hardeners were mixed to create epoxy-amine systems, which thickened in closed vials for up to 3 hours. Thermoset films with 20 to 60 m thickness were produced using a heated hydro press and cured for 1 hour at 50ºC, 90ºC, and 30 minutes of post curing at 170ºC under vacuum.

2.3.4 Curing Mechanism of Secondary Amines

During the coating process, the secondary amines was synthesised and worked as a reactive epoxy curing agent. The resultant material's compatibility with epoxy resins was greatly enhanced (B. Chen et al. 2020). Because of secondary amines is a reactive, it is the reason why secondary amines was chosen as an epoxy curing agent. The polymerization mechanism of epoxy and amine is depicted in Figure 2.5. According to the nanofiller fills the pores of epoxy resin, increasing strength and corrosion resistance while solving an important need (Duan et al. 2021). It is vital to note that the epoxide ring is very reactive and easily experiences ring opening reaction with active hydrogen of curatives or hardeners. Stated that cycloaliphatic amine is reactive compared to aromatic amine (Verma et al. 2020).

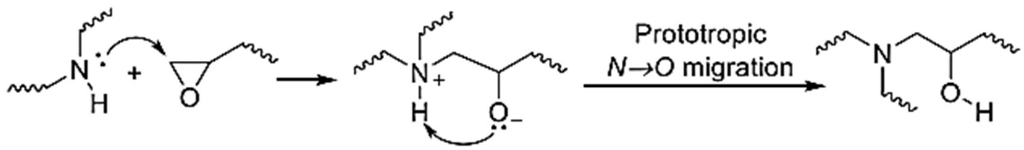


Figure 2.5 Mechanism of polymerization epoxy with amines (Duan et al. 2021)

2.4 Nitrogen Gas

Nitrogen oxides are secondary species produced by nonthermal plasma in air or nitrogen-containing carrier gases, likely involving atomic oxygen and N_2 and N (Qiongyu Li 2018). Nitrogen gas is necessary in epoxy coating solutions because it creates inert atmospheres that prohibit interactions with oxygen and moisture, maintaining resin stability. It aids in solvent evaporation, viscosity adjustment, and curing. Nitrogen decreases the concentration of oxygen in combustible materials, improving safety and reducing the risk of fire or explosion. Other than that, according to Luo et al., (2020), the synthesis of flame retardants, including phosphorus, nitrogen, and silicon, improves the self-extinguishing performance of epoxy thermosets, consequently improving the flame-retardant qualities of epoxy resin.

Furthermore, at high temperatures, nitrogen dopants have a considerable influence on film surface morphologies, generating needle-like sp^2-carbon structures. Nitrogen incorporation at grain borders is thermodynamically preferable to grain inclusion because grain boundaries have less energy (Wanninayake et al. 2020). Plasma-Enhanced Atom Layer Deposition (PEALD) is a method of depositing an oxide coating on a template using nitrogen gas as a carrier and dilution gas, covering the exposed top surface and patterned structures

(Fukazawa 2020). According to the findings, coatings with a greater nitrogen concentration are more resistant to oxidation between a high-entropy alloy (HEA)-based coating and ambient air (Sha et al. 2020).

2.5 Process of EPD

Electrophoretic deposition (EPD) is a process to fabricate epoxy coatings. EPD is a cost-effective and adaptable coating method that deposits thin, homogeneous coatings on conductive surfaces. It generates an electric field between parallel electrodes, forcing charged particles to collide with the substrate, resulting in thin films of different quality (Aghili et al. 2021). As identified by Wang et al. (2022a), an electric field and water-cationic epoxy resin molecules are used to investigate the EPD repair technique for efficiently fixing rust-cracked reinforced concrete. EPD repair involves wrapping corroded reinforcement with cationic epoxy resin and curing agent to prevent hazardous interaction (Wang et al. 2022b).

2.5.1 Parameters of EPD

Electrophoretic deposition (EPD) is a low-cost and easy approach. The microstructure layer may be adjusted by adjusting processing parameters such as deposition duration, voltage, and suspension concentration (Rahmadani et al., 2022). According to Figure 2.6, the higher the conductivity, the larger the current density. Higher current density accelerates cationic epoxy resin and curing agent electrophoresis, reducing deposition in cracks and surfaces, according to Wang et al. (2022a). In addition, the dimensions of two parallel electrodes are 100mm x 3mm x 0.6mm (Aghili et al., 2021). The parameters utilised in EPD that were gathered from various resources are summarised in Table 2.5.

Table 2.5 Parameter of EPD process (Rahmadani et al. 2022) (Aghili et al., 2021)

Types of electrodes	**Voltage (V)**	**Distance between electrodes (mm)**	**Deposition Time (min)**	**Thickness (µm)**	**Temperature (ºC)**
CP-Ti (cathode) a platinum foil (anode)	20	20	30	32.2	25
	30			50.5	
	40			60.6	
Steel strips (cathode) Stainless steel strip (anode)	30	20	20	5±0.5	160±5
	50			7±0.5	
	70			10±0.5	

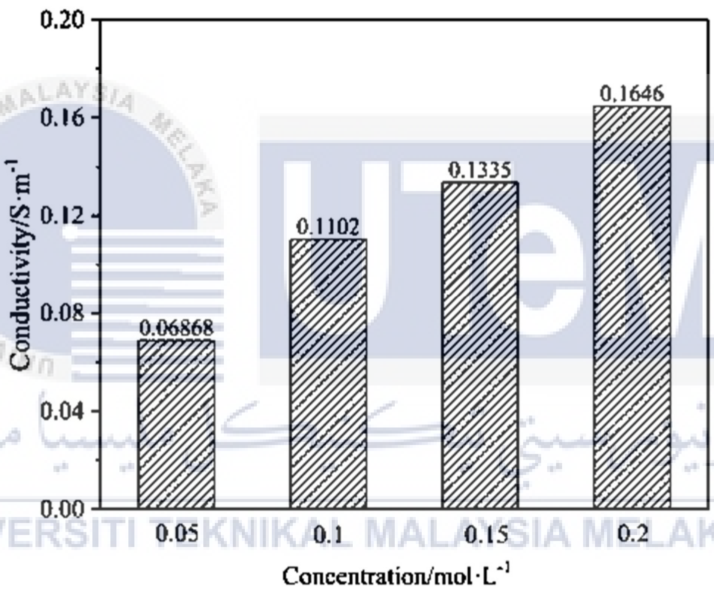


Figure 2.6 Conductivity of high-performance epoxy resin solutions at various concentrations (Wang et al., 2022a)

In summary, based on the articles, EPD factors such as voltage, deposition duration, particle size, and suspension have a major impact on coating qualities. Optimisation of these parameters is critical for attaining desired film properties, making EPD a strong tool for thin-film production in a variety of industries. Continuous research and control help to enhance and innovate EPD procedures.

2.6 Curing Process

The curing process, which converts a liquid to a solid, improves qualities such as strength and stiffness, with curing factors such as temperature and time influencing the process. Post-curing exposes cured resin objects to temperatures above curing temperature, resulting in enhanced strength, a higher glass transition temperature, lower stress, and reduced outgas propensity (Moller, Berry, and Foster 2020).

Other than that, according to Muc, Romanowicz, and Chwal, (2019), polymerization is a chemical process that connects monomers or prepolymers to generate polymer networks. Catalysts, or curing agents, are used in this procedure. Thermosetting polymers go through irreversible curing and hardening, which may be done in phases, with the interfacial bonding regulated in a simple way (Muc, Romanowicz, and Chwał, 2019). Figure 2.7 shows phenomena in resin curing technology, including exceeding the gelling threshold, curing near the infusion channel, and curing the entire volume.

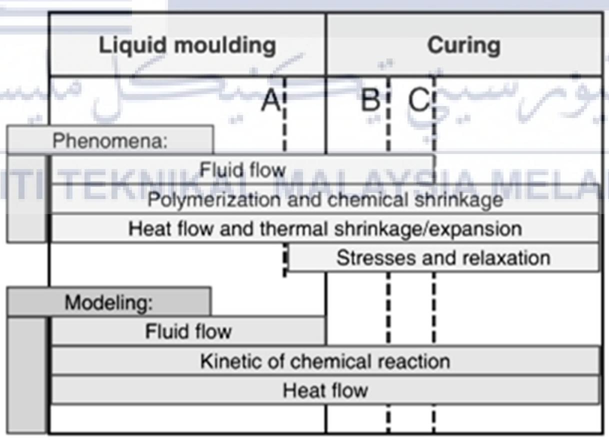


Figure 2.7 Phenomena in resin curing technology (Muc, Romanowicz, and Chwał 2019)

In addition, based on the finding of Zhang et al., (2010), the exothermic process accelerates cure, resulting in a non-uniform cure field, potentially entrapping voids, and volatile byproducts, and promoting warpage and residual stress buildup in epoxy plates. Figure 2.8 depicted the graph of time versus degree of cure for the study investigates the impact of the temperature ramp on non-uniform curing in an epoxy part with a convective boundary condition.

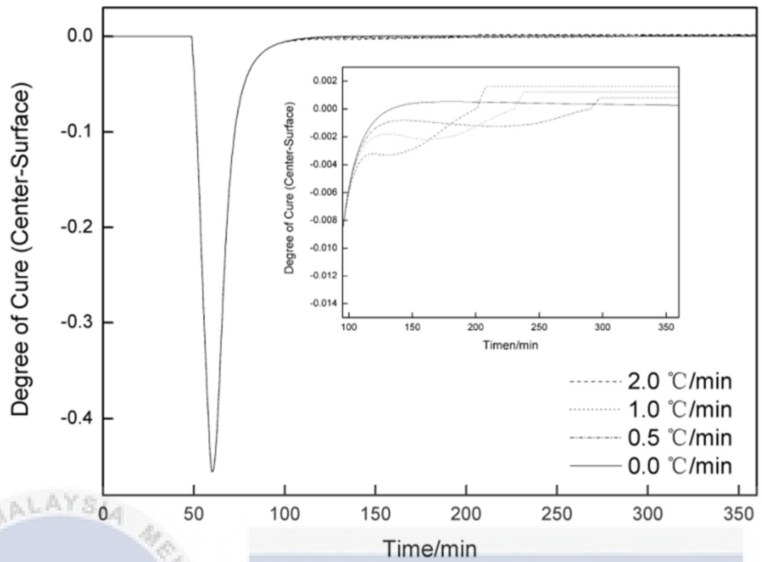


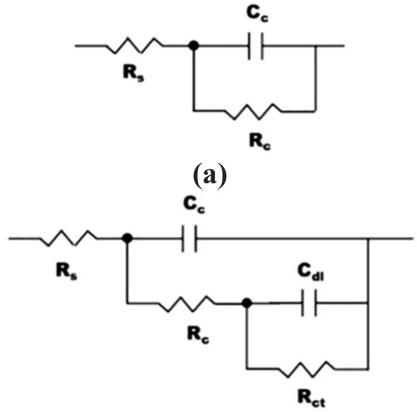
Figure 2.8 The effect of a temperature ramp on non-uniform curing in a convective boundary condition epoxy component (Zhang, Xu, and Huang, 2010)

2.7 EIS Analysis

The method of electrochemical impedance spectroscopy (EIS) is used to assess the electrochemical behaviour of epoxy coatings on metal substrates. It detects possible faults or deterioration by measuring resistance, capacitance, and total impedance. EIS is used in

industry to guarantee the quality of protective coatings. According to Oliveira et al., (2021), the study uses electrochemical impedance method EIS to explore the effect of flash rust inhibitors on hydro blasted surface corrosion protection and substrate or coating interfaces during ageing testing. Its goal is to evaluate the reliability of epoxy coating methods on offshore platforms.

In electrochemical impedance spectroscopy (EIS), the equivalent electric circuit (EEC) is a theoretical model. It consists of electrical components such as resistors, capacitors, and inductors that reflect certain electrochemical events. Figure 2.9 depicts the equivalent electric circuits used to gather the EIS data.



(a)

(b)

Figure 2.9 Equivalent electric circuits (EEC) used for the EIS data (Oliveira et al. 2021)

According to Koochaki et al., (2021), the EIS findings on a scratched insulating polymer coating over a steel conductive surface were analysed using a two-time constant electrical equivalent circuit (EEC). In addition, equivalent circuit models were utilised to analyse electrochemical impedance data for three coating systems and evaluate their protective characteristics depending on coating and breakpoint frequency parameters (X. Liu et al. 2009).

In summary, electrochemical Impedance Spectroscopy (EIS) is a critical technique for evaluating epoxy coating effectiveness by analysing electrical parameters such as resistance and capacitance. It facilitates the detection of defects, the monitoring of changes, and the evaluation of coating protective properties in industrial applications.

2.8 FTIR Analysis

Fourier transform infrared (FTIR) spectroscopy was used to characterize the prepared samples. Spectrum Two FTIR spectrophotometer was used to measure FTIR spectra (Cheng et al. 2022). Meanwhile, according to (RuRudawska et al. 2019) curing resin entails two processes: the formation of linear chains and the assembly of linear pieces into a three-dimensional network structure. The percentage conversion of reactive resin groups is determined by the degree of conversion. FTIR spectroscopy is a tool for assessing the degree of conversion during curing and monitoring changes in distinctive groups or bonds. Attenuated total reflection (ATR) is an FTIR spectrophotometer accessory used to assess the surface characteristics of solid or thin film materials rather than their bulk properties. FTIR-ATR spectroscopy with a diamond crystal with a 4 cm^{-1} wavelength resolution was utilized to detect functional groups in adhesive formulations. The functional groups found in the evaluated adhesive formulations were identified using the FTIR-ATR spectrum as in Figure 2.10.

Based on findings of (Cordeiro Neto et al. 2020), it can be argued that to validate the encapsulation of Tung oil in urea-formaldehyde (UF) microcapsules, FTIR investigations were performed. The FTIR measurements were made with a spectrometer (Bruker, vertex 70) through ATR in transmission mode in the 400-4000 cm^{-1} range. Based on the various findings, we can identify that FTIR is most useful in determining the chemical characterization of the specimen.

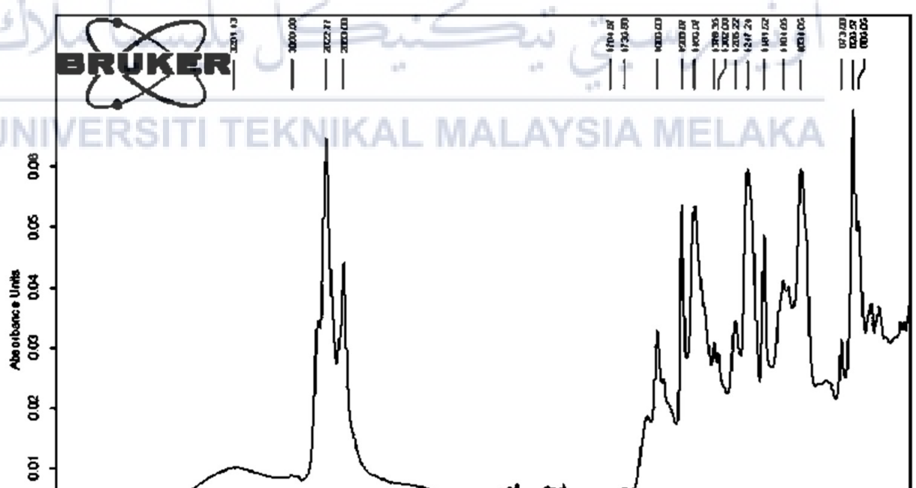


Figure 2.10 FTIR spectrum of Specimen 1 (combination of epoxy resin called as Epidian 6 and the curing agents is called as PF with the values of ratio is 100:50) (RuRudawska et al. 2019)

In summary, based on the articles, EPD factors such as voltage, deposition duration, particle size, and suspension have a major impact on coating qualities. Optimisation of these parameters is critical for attaining desired film properties, making EPD a strong tool for thin-film production in a variety of industries. Continuous research and control help to enhance and innovate EPD procedures.

2.9 Optical Microscopy Analysis

Optical microscopy study of epoxy coatings allows thorough microstructure investigation, revealing faults and fractures, and helps in quality control and performance assessment in industries such as aerospace and automotive. Poole and Mostaço-guidolin (2021) state that, due to its great sensitivity and specificity for key extracellular molecules, optical microscopy is a viable alternative to traditional histology for researching extracellular matrix (ECM) remodelling.

Other than that, limited image data might stymie deep neural network applications in optical microscopy, particularly in rare or difficult samples or pictures. Deep learning is successful on small data sets, but generalisation increases with bigger training data sets, which can range from a few thousand to hundreds of thousands of patches (De Haan et al. 2020). Furthermore, Balasubramanian et al. (2023) state that optical microscopy is a fast-evolving method of scientific study that allows for the visualisation of biology in its physiological context, outperforming other life science approaches.

In conclusion, optical microscopy is an important technique for analysing epoxy coatings since it provides extensive information on microstructure, surface morphology, and fault identification. Its high-resolution photographs assist in the optimisation of coating processes for increased durability and usefulness in industries such as aerospace and automotive.

2.10 SEM Analysis

Scanning electron microscopy (SEM) was used to analyse the surface morphologies of various metal substrates acquired by peeling off coated water-borne epoxy (WEP) coatings exposed in salt spray for 450 hours to further evaluate the anti-corrosion capacity of all WEP coatings. By monitoring the fracture surface of all WEP coating samples, SEM (Hitachi SU-8010 SEM) was utilised to analyse the compatibility of graphine oxide (GO) and nontoxic phytic acid-graphine oxide (PA-GO) with WEP coating (X. Zhou et al. 2020). Moreover, as explained by (Ibrahim et al. 2020), SEM with a 5 kV accelerating voltage and a 6mm working distance was used for morphological investigations. The contrast and brightness were altered to make the particles more visible. Energy Dispersive Absorption X-ray (EDAX) analysis is an elemental technique using electron microscopy to detect elements in specimens through distinctive X-rays. SEM scans confirmed agglomeration and detected composite particles with sizes smaller than 100 nm. Figure 2.11 depicts a SEM investigation of steel substrates untreated and coated with two cured epoxy resins before and after 24 hours of immersion in 3.5% NaCl solution at 298 K (Hsissou et al. 2021).

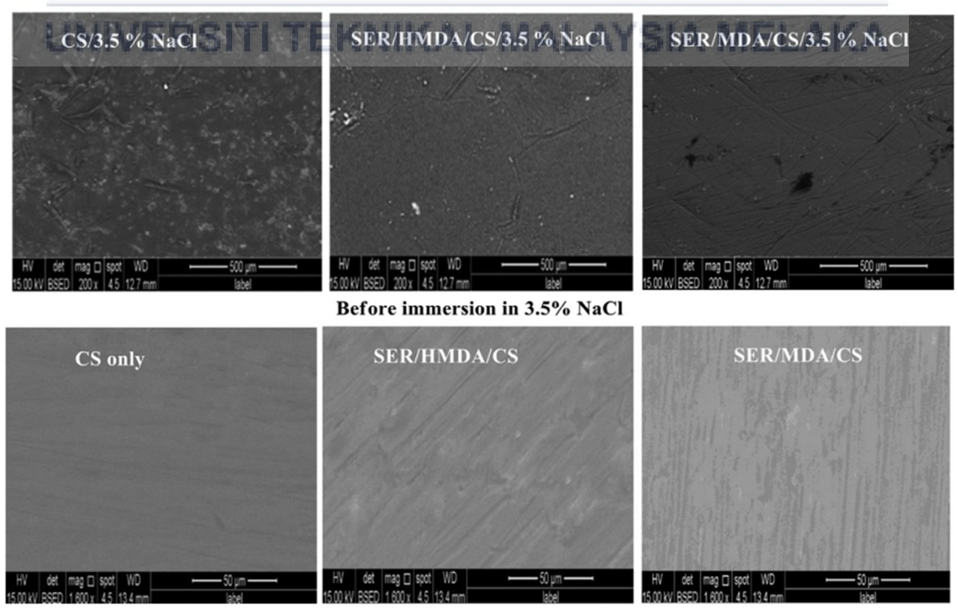


Figure 2.11 SEM photos of carbon steel before (Hsissou et al. 2021)

2.11 Conclusion

In conclusion, based on the various findings, the properties and characterization of epoxy resin coating can be illustrated. There are some ways to determine the characterization methods that were by using electrochemical impedance spectroscopy (EIS), fourier transform infrared (FTIR) spectroscopy analysis, scanning electron microscopy (SEM) analysis, and optical microcopy. Based on the finding, it will helps us in future to handle the epoxy coating with a proper ways and to get a better results.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter discusses the study of the impact of secondary amines as hardeners on epoxy coating properties. It provides a systematic approach and experimental methodologies for assessing the performance of epoxy coatings using secondary amines. The introduction provides context, insights into the selection of secondary amines, expected outcomes, and study objectives. The methodology section lays the groundwork for further investigation into the interactions between epoxy resins and secondary amine hardeners in coating applications.

3.2 Experimental Flow Chart

The flow chart in Figure 3.1 depicts the methodology of this investigation.

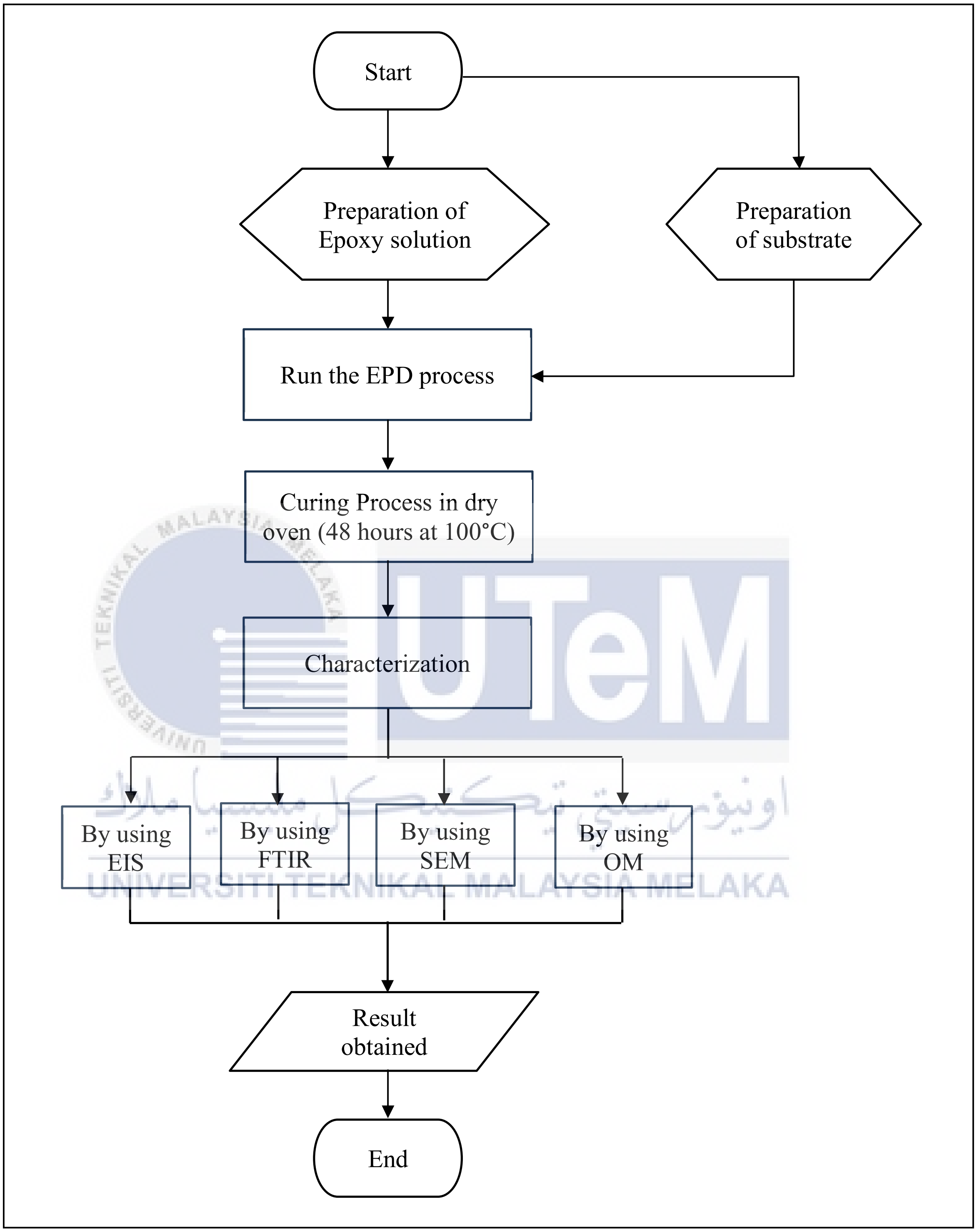


Figure 3.1 Process Flow Chart

3.3 Preparation of Substrate

In this research, we used a plate of Zinc-plated Steel Sheet as a coating plate. Sheet metal is a common material for epoxy coating research due to its practicality, uniform surface, ease of handling, cost-effectiveness, and variety in testing coating performance. The plate as shown in Figure 3.2 (a) was cut into small strips as in Figure 3.2 (b). The description of the strip's sample is depicted as in Table 3.1. Before we coated, the strips were cleaned and rinsed with ethanol to make sure there was no foreign matter or dust on the strip's surface.

(a) (b)

Figure 3.2 Zinc-plate Steel Sheet (a) Plate of sheet metal, (b) substrate of zinc

As shown in Figure 3.2, the plate was cut into a small piece, and the description of the strips is mentioned in Table 3.1. The strip will then act as an electrode in the electrophoretic deposition (EPD) process. An electrode is composed of two pairs of strips that will be the anode and cathode. In this research, there are nine sets of electrodes. It is because there are three differences in concentration and voltage applied during the EPD process.

Table 3.1 Properties of the Zinc-plated Steel Sheet

Properties	**Description**
Dimension	29 mm x 44 mm
Thickness	0.140 mm
Area	1276 mm^2
Volume	178.64 mm^3
Weight	1.35 g
Colour	silver
Surface Condition	smooth surface

3.3.1 Labelling Sample

The labelling of samples is based on several factors, such as the differences in voltage usage during the EPD process and the value of the secondary amine concentration in the solution. Because of that, the set of electrodes must be done in nine sets. Labelling the sample is crucial to ensuring the results obtained are not changed with other samples and are easy to analyse. Table 3.2 depicts the labelling the coating substrate used in this research.

Table 3.2 Labelling the coating substrate

Number of Sample	Secondary Amine's Concentration (ml)	EPD Voltage (V)
1		30
2	0.5	40
3		60
4		30
5	1.0	40
6		60
7		30
8	1.5	40
9		60

3.4 Epoxy Resin

In this research, the epoxy type that we used is Auto Fix 8800-A as shown in Figure 3.3. It was purchased from the Chemibond Enterprise Sdn Bhd. The epoxy resin was choosen because it passed the quality control (QC) inspection. It is important to make sure the material that we used is safe and under control. The properties of the epoxy resins are in Table 3.3.

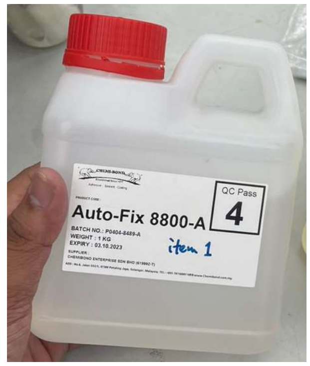


Figure 3.3 Type of epoxy used for coating process

Table 3.3 Properties of epoxy resin

Properties	**Evaluation**
Product Code	Auto-Fix 8800-A
Type	Adhesive
Colour	white
Weight	1 kg
State of Aggregation	liquid
Density	1.160
T_m(°C)	40

3.5 Preparation of the Epoxy Solution

The preparation of the coating solution completely occurs in a double-jacketed glass reactor. The model of the glass reactor is the R10 10-Litre, with power up to 90 Watts. Glass reactors provide transparency, chemical inertness, real-time reaction observation, accurate temperature control, ease of cleaning, corrosion resistance, scalability, and safety through immediate visual inspection. Figure 3.4 shows the setup of a jacketed glass reactor integrated with a heat circulator.

Figure 3.4 The Double Jacketed glass reactor integrated with heat circulator

There are three differences in the solution used to differentiate the concentration of secondary amines. Hence, the overall method used for each solution is similar. To produce the coating solution, there are three parts that need to be done (part 1, part 2, and part 3) to make sure the solution is fully synthesised.

The first part is the process of heating the epoxy resin. The temperature of the epoxy solution must rise to 145 ºC for three hours. After 3 hours, the secondary amines are added to the glass reactor so that they can be mixed with the epoxy solution. The second part took 2 hours, and the temperature was controlled between 70 ºC and 90 ºC.Then, the last part, which is the third part, involves the addition of formic acid, deionized water, and nitrogen gas into the solution of epoxy resin and secondary amines. In the last part, the pH scale value should be controlled at about 4.4 (acidic). Figure 3.5 is a summary of the three parts needed to produce a coating solution.

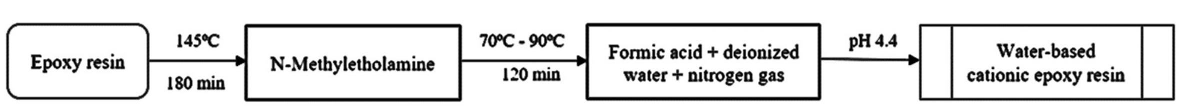


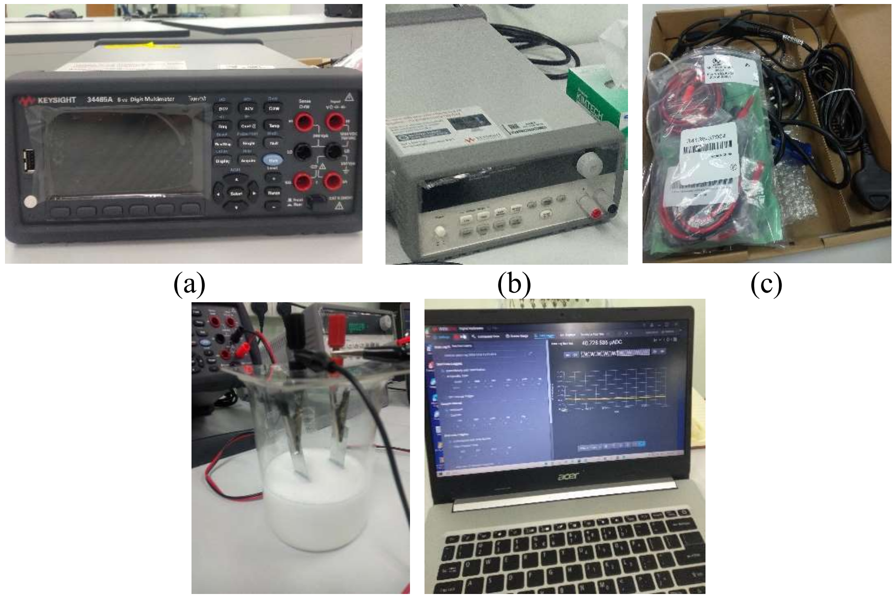
Figure 3.5 Synthesis process of water-based cationic epoxy resin

3.6 Work Process of EPD

Electrophoretic deposition (EPD) is a process that consists of two electrodes that are used as anodes and cathodes, as depicted in Figure 3.6. The anode electrode has a positive charge, while the cathode electrode has a negative charge. Epoxy liquid was used as a suspension. The coating is formed when charged particles move to the cathode electrode. So, the cathode will be coated, and the result of that coating will be analysed in this research. In this EPD process, various voltage values are applied, which are 30V, 40V, and 60V. This is to determine the differences in the coating areas when the applied voltages are different.

Figure 3.6 Electrophoretic Deposition (EPD) setup

The important things in the setup of the EPD process are the DC power supply with model E3643A (0 to 35 V, 1.4 A, or 0 to 60 V, 0.8 A), the digital multimeter (DMM) with model 34465A, four wires (two red and two black), Keysight Software, and also a beaker containing water-based cationic epoxy resin, as shown in Figure 3.7. To run and observe the data for the current values of DMM, the Keysight software is used. The EPD process was held for 15 minutes, and then the completed graph of the EPD process will appear in the Keysight software.



(a) (b) (c)

(d) (e)

Figure 3.7 Parts of EPD setup (a) DMM, (b) DC Power Supply, (c) Wires, (d) Beaker containing solution, (e) Keysight Software

3.7 Curing Process of Coated Sample

The curing process involves placing the material in a dry oven for a duration of two days at a temperature of 100ºC. During this thermal treatment, the material undergoes a curing reaction, resulting in enhanced properties such as increased hardness, strength, and durability. The prolonged exposure to the specified temperature ensures thorough curing and cross-linking of the material's molecular structure. This controlled heating process is crucial for achieving the desired performance characteristics in the final product, making it suitable for various industrial applications. Figure 3.8 shows the dry oven that was used in the curing process.

Figure 3.8 Dry oven for curing process

3.8 Characterization Technique

The characterization technique is the last process that occurs in this research. It is very important to determine the surface morphologies, the performance of the coating, and the properties of the coating sample. In this research, we used several methods such as thickness measurement, electrochemical impedance spectroscopy (EIS), Fourier transform infrared (FTIR), and scanning electron microscopy (SEM).

3.8.1 Thickness Measurement

The concept of "mass variance pre-coating and post-coating" refers to the difference in mass experienced by a substrate or material before and after the application of a coating through the electrophoretic deposition (EPD) procedure. Initially, before initiating any coating using EPD, the substrate's mass is gauged and documented as the baseline measurement. Following the application of the coating through the EPD technique, the substrate's mass is measured again. The disparity between these two measured masses indicates the amount of material deposited into the substrate during the EPD process

The significance of this mass difference becomes evident when evaluating the effectiveness and efficiency of EPD technology. A larger mass differential, typically observed with successful EPD applications, signifies a more substantial deposition of coating material onto the substrate. It serves as a quantifiable metric, indicating the extent to which the coating material bonds to the surface of the substrate.

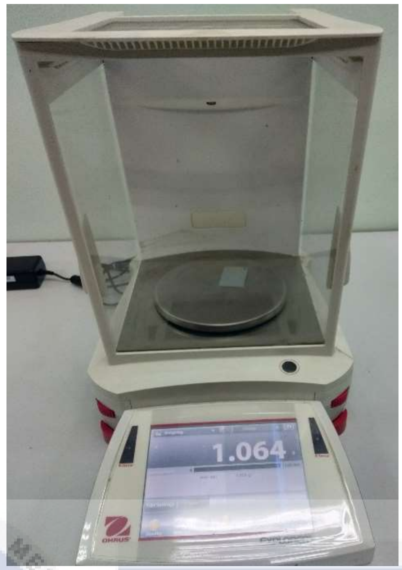


Figure 3.9 A mass-weighing scale

Furthermore, by understanding this mass discrepancy helps to clarify the thickness and homogeneity of the EPD-generated coating. A constant and significant mass difference across numerous samples points to a more reliable and reproducible EPD approach. Variations or inconsistencies, on the other hand, may indicate abnormalities in the coating application and necessitate additional investigation of process parameters for optimisation.

To summarise, mass difference measurement is critical in determining the efficacy and reliability of the EPD technique for coating deposition. It has a considerable impact on process optimisation decisions as well as the quality of the final coatings produced using this method..

3.8.2 EIS

The characterization using electrochemical impedance spectroscopy (EIS) is held at Multimedia University. There are several important parts for the EIS process, which are potentiostats or galvanostats with a Reference 600 model, two electrode measuring cells for

EIS, and EIS software. First of all, the sample should be cut into a small circle. The diameter of the sample should be ± 18 mm. It is because the diameter of the EIS tool is 22 mm. So, the sample size should be less than the size of the tool. Figure 3.10 depicts the parts of EIS.

(a) (b) (c)

(d)

Figure 3.10 Parts of Electrochemical Impedance Spectroscopy at Mutimedia University (a) sample holder, (b) two electrode measuring cell, (c) potentiostat/ galvanostat, (d) fully setup of EIS

In this research, there are nine coating substrates to be tested, and the differences between each sample are the secondary amine concentration and the voltage values of the EPD process. Then, each sample displayed the differences readings as the parameters used were differences. Figure 3.11 depicts the sample used to obtain the data by EIS characterization.

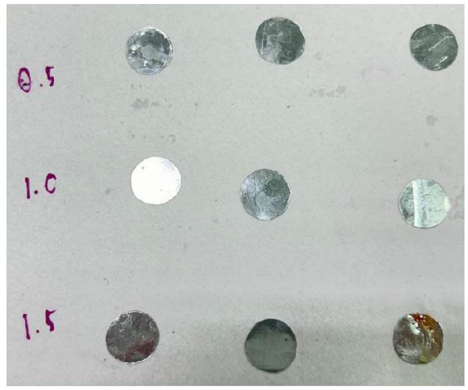


Figure 3.11 Coating substrate to characterise by using EIS

EIS characterises coating samples by applying an alternating current signal over a wide frequency range. The dielectric constant value of each different amine concentration and voltage has been summarised into a dielectric constant graph against log ω. The result is EIS is valuable for monitoring coating integrity, assessing durability, and correlating results with other techniques like microscopy for a comprehensive understanding of coating performance.

3.8.3 FTIR

Fourier transform infrared spectroscopy (FTIR), with the name Dee Multi Technology and the model Jasco FT/IR-6100, as shown in Figure 3.12, is capable of analysing material molecular composition, including coatings, providing information on functional groups and chemical bonds, and aiding in inferring coating processes. As usual, first of all, we need to prepare the coated sample for FTIR analysis by grinding, powdering, or coating it on a suitable substrate. Ensure the sample is clean and free of contaminants. Load the sample into the FTIR instrument and set up measurement parameters, including sampling technique, beam path, and instrument settings.

After that, before analysing the coated sample, measure the background spectrum of the reference material to account for interference or absorption. On FTIR equipment, measure the infrared spectrum of the coated sample using transmission, reflection, or attenuated total reflection modes. Then, the infrared spectrum of the coated sample was measured using an instrument scanning mid-infrared wavelengths and measuring absorbance or transmittance at each wavelength.

The FTIR spectrum is important to identify functional groups and chemical bonds in the coating. Compare the peaks and patterns with reference spectra from libraries like NIST or commercial databases to match bands with known compounds. Lastly, we should interpret the FTIR results, considering identified functional groups, chemical bonds, and other relevant information, and compare the findings with known coating processes, reference samples, or literature to infer the most likely coating process used.

Figure 3.12 Fourier Transform Infrared Spectrometer

3.8.4 SEM

The HITACHI SU5000 Scanning Electron Microscope (SEM) is a sophisticated tool for high-resolution imaging of material surfaces. The SEM, which is equipped with an electron gun and lenses, focuses an electron beam on a specimen in a vacuum chamber. SEM is increasingly being used in a variety of applications, including medical, biological, metals, semiconductors, and ceramics. Its capability is growing as more attachments and gadgets are added. There are many features of SEM machines. Among them are superior operability, navigation assist, LV included as standard, fully integrated EDS, auto functions, and magnification. The adjustable pressure in the specimen chamber is 10 Pa to 100 Pa. Moreover, the resolution for high-vacuum mode is 4.0 nm, 3.7 nm, 8.0 nm, and 15.0 nm. Meanwhile, the low-vacuum mode is 5.0 nm.

SEM is a powerful imaging technique that uses a focused beam of electrons to examine the surface of a sample. While SEM itself cannot directly identify the coating process, it can provide detailed information about the coating's morphology, composition, and thickness, which can be used to infer the coating process.

The procedure for using SEM to analyse coatings starts with preparing the coated sample by ensuring it is clean and dry. Then, we need to mount the sample securely using a conductive adhesive. Next, if the coating is non-conductive, a small layer of conductive material can be sputtered onto the sample using a specialty coating apparatus. After that, we should load the sample holder into the SEM chamber and configure the accelerating voltage, working distance, and other imaging settings.

Thenceforth, to get the clearest and best image, we need to adjust the focus, astigmatism, brightness, or contrast settings. We need to start with low magnification, then raise it to analyse coating morphology, and take many images to catch the complete covering. Furthermore, we need to analyse the elemental composition of the coating using thickness measurement methods like cross-sectional SEM. The next step is to assess the thickness of the coating using techniques such as cross-sectional SEM. To assess the deposition process, the last phase comprises interpretation and analysis, which includes analysing SEM images and elemental composition data and comparing coating morphology with known techniques and reference samples.

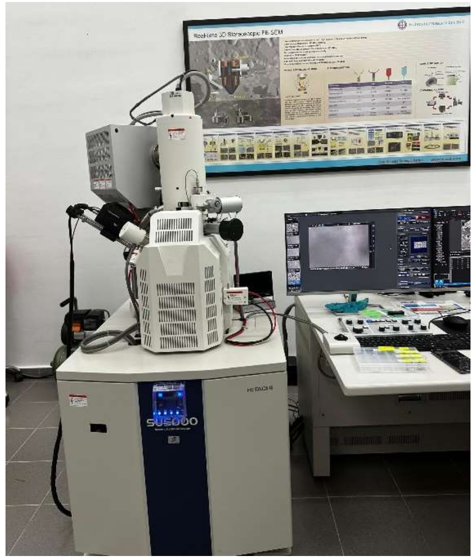


Figure 3.13 Scanning Electron Microscopy (SEM)

3.8.5 Optical Microscopy

Optical microscopy (OM), made by Leica Microsystems with the model DM 2500 M, as shown in Figure 3.14, is a technique that uses visible light and lenses to magnify and observe small objects or specimens that are not visible to the naked eye. It involves a light source, condenser, objective lens, specimen, stage, and eyepiece. The objective lens magnifies the specimen's details, and the eyepiece further magnifies the image for observation. Optical microscopes are essential tools in biology, medicine, materials science, and other fields, enabling scientists to study the microscopic structures and properties of various samples.

Figure 3.14 Optical Microscopy

The key parts of an optical microscope include the light source for illumination, the condenser to focus light on the specimen, the objective lens for primary magnification, a stage for holding the specimen, and coarse and fine focus knobs for adjusting clarity. The eyepiece further magnifies the image for observation. Additional components like the diaphragm control light intensity, mechanical stage controls enable precise movement, and a camera may be present for image capture. These parts work collaboratively to facilitate the observation and magnification of specimens in optical microscopy.

In summary, the methodology for optical microscopy involves meticulous sample preparation, effective utilisation of key components like the light source and objective lens, and systematic observation techniques. This approach ensures clear and precise imaging of microscopic structures. Regular maintenance is emphasised for sustained, optimal performance. Optical microscopy remains an indispensable tool, facilitating advancements in various scientific fields through detailed microscopic exploration.

3.9 Summary

Overall, in this research, towards achieving the stated objectives in Chapter 1, the research methodology has been performed in phases. Some of the standard performance testing of epoxy coatings has been carried out to measure the thickness and characteristics. As a conclusion, to obtain accurate results during the process, every single thing should be taken seriously. Coating techniques also play an important role in ensuring that the results obtained are accurate and reliable.

3.10 Gantt Chart

As shown in Appendix B

CHAPTER 4

RESULT AND DISCUSSION

4.1 Introduction

The previous chapters have gone over the research strategy, methodology, and systematic data gathering. This chapter focuses on the presentation and interpretation of the outcomes of these exhaustive research endeavors. The primary goal is to examine the data in order to extract useful insights, answer research questions, and eventually contribute to the field's greater body of knowledge. After the curing process, the visual observation of the coating substrate was addressed. The thickness of the coating substrate was examined using the specific formula. The coating substrate's properties were assessed using a specific characterization method, specifically electrochemical impedance spectroscopy (EIS), to determine its dielectric property data. In addition, several methods are used to characterize coating substrates, including Fourier transform infrared spectroscopy (FTIR) for chemical composition insights. Other than that, to examine the structure properties, scanning electron microscopy (SEM) and optical microscopy are used to analyze surface properties and assess coating quality.

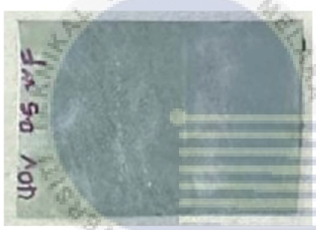
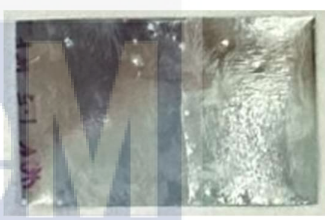
4.2 Coating Substrate Appearance

The visual characteristics of an epoxy-coated substrate contribute to both its aesthetic appeal and functional performance. Epoxy coatings are noted for their smooth and homogeneous surface. The look of epoxy-coated substrates is critical in a variety of sectors, where both performance and visual aesthetics are significant concerns.

4.2.1 Electrophoretic Deposition (EPD)

In the EPD process, the distance between two electrode and time are fixed. Figure 4.1 shown the coating substrate applied by various value of secondary amine concentrations and the EPD voltages.

Table 4.1 Coating substrate with different secondary amine concentration and applied EPD voltage

	0.5 ml	1.0 ml	1.5 ml
30 V			
40 V			
60 V			

Based on the observations, all of the substrates have been successfully coated through the EPD process using different parameters. After the coating process, by visual inspection, the surface of the coating area turns to glossy and looks exhibits a reflective surface compared to an uncoated one.

4.2.2 Thickness Analysis

Understanding the thickness of a coating substrate is crucial for its performance and quality, as it influences factors like corrosion protection, adhesion, and durability. Consistency is essential for meeting performance standards, quality control, and cost efficiency by minimising material use while maximising coating properties.

The thickness of the coating substrate was measured by the derivation formula for density, as in Formula (4.1). Formula (4.3) is the calculation formula for thickness of the coating substrate.

$$\rho = \frac{m}{V_O} \quad (4.1)$$

$$\rho = \frac{m}{A.t} \quad (4.2)$$

$$t = \frac{m}{A.\rho} \quad (4.3)$$

Where t is the thickness of the coating substrate (mm); ρ is the density of the epoxy resin (g/mL); m is the difference mass between after and before coating (g); and A is the area of the coating zinc plate (mm^2). Based on epoxy obtained from Chemibond Enterprise Sdn Bhd., the epoxy density (ρ value) is 1.160 g/mL. Meanwhile, the value of the coating area, A, is 1276 mm^2. The values of ρ and A are fixed. When the values are filled, as in formula (4.3), the thickness as in Table 4.2 is obtained.

Table 4.2 The data of the substrate before and after coating

Concentration of Secondary Amines (ml)	Applied EPD Voltage (V)	Weight,w (g)			Thickness, t (µm)
		Before	After	Difference	
0.5	30	1.353	1.455	0.102	69.0
	40	1.351	1.426	0.075	51.0
	60	1.349	1.418	0.069	47.0
1.0	30	1.346	1.436	0.090	61.0
	40	1.354	1.425	0.071	48.0
	60	1.348	1.421	0.073	49.0
1.5	30	1.352	1.416	0.064	43.0
	40	1.354	1.414	0.060	41.0
	60	1.347	1.381	0.034	23.0

Based on the calculations, when the voltage of the EPD process increases, the thickness of the coating substrate decreases. It may be because the bubbles occur more when the voltage applied is higher. As stated in the research of Wang et al. (2022b), the anode electrolysis of water results in oxygen bubbles during electrophoretic deposition repair. These bubbles provide a gradient that influences current conduction and discharge uniformity. The oxygen bubbles float because they are encased in epoxy resin, which makes them tough to break. As a result, the surface of the epoxy resin coating is thin. Figure 4.1 represents the thickness of the coating substrate with a different voltage during EPD process.

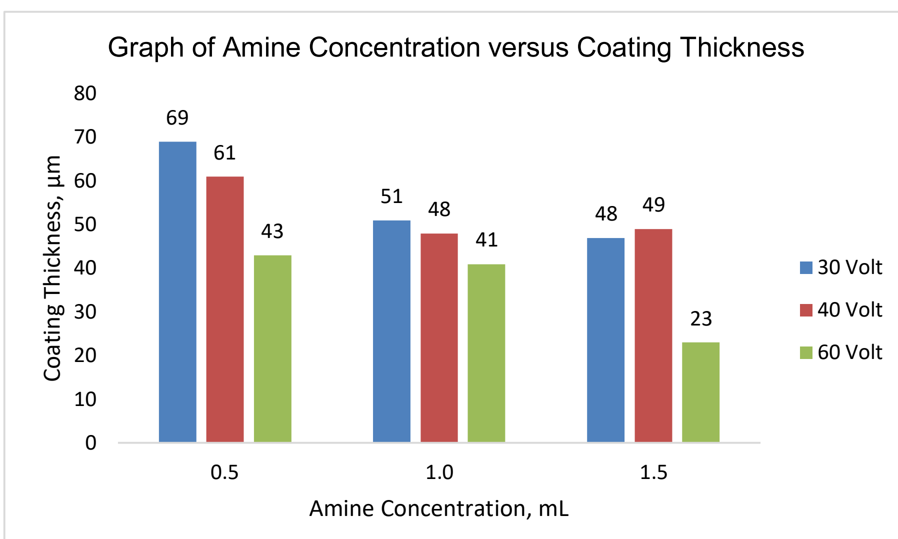


Figure 4.1 Thickness of the coating substrate after cure in a dry oven

In conclusion, both hypotheses are the same based on the information collected from the reading material and the findings of the investigation. If the voltage value supplied during the EPD process is high, the resulting thickness will decrease. As clearly can be seen as in Figure 4.1, the best parameters in order to produce the best coating thickness is 0.5 ml amine concentration and 30 volts of EPD voltage.

4.3 Dielectric Properties

The dielectric properties of a substrate coated with an epoxy coating refer to the electrical characteristics of the material when subjected to an electric field. Epoxy coatings are commonly used for their insulating properties, making them suitable for applications where electrical insulation is essential. Understanding the dielectric properties of a coating substrate with an epoxy coating is crucial in applications where electrical insulation is required, such as in electronic components, electrical devices, or cable insulation. These properties ensure the reliable performance and safety of the coated materials in various electrical environments.

Furthermore, the dielectric constant, ε, of the coating substrate was measured using the formula dielectric constant as stated by Joshi et al. (2017) in 4.4.

$$\varepsilon' = \frac{t}{(2\pi f)(A\varepsilon_0)} \cdot \frac{Z''}{Z'^2 + Z''^2} \tag{4.4}$$

Where, A = cross-section area of the pallet in m^2, t = thickness of the pallet in m, ε_o= permittivity of free space, f = frequency in kHz, Z' = real part of impedance and Z” = imaginary part of impedance. From the calculation, a graph of dielectric constant against log ω will be plotted (where ω = 2πf). Figure 4.2, 4.3, and 4.4 shows the variation of dielectric constant with frequency at different voltages.

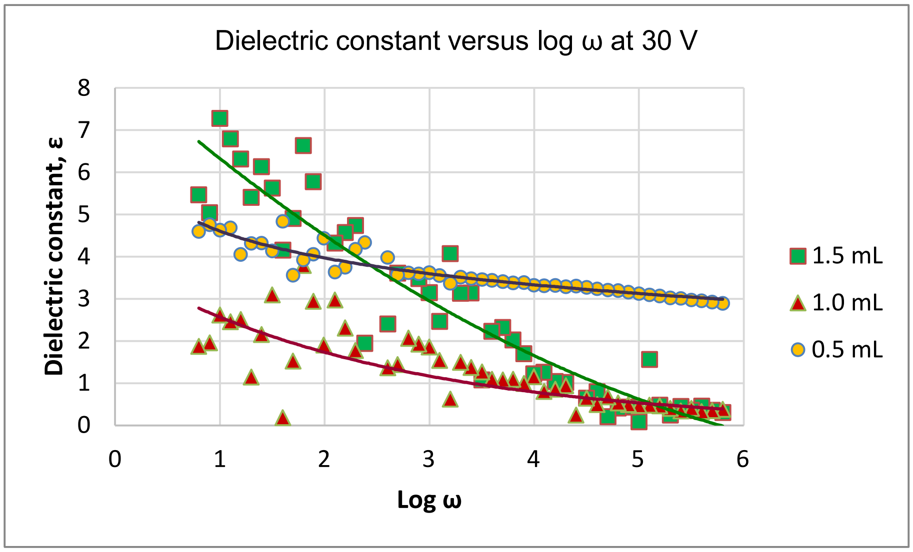


Figure 4.2 Dielectric Constant versus log ω at 30 volts

Figure 4.2 displays the graph of the dielectric constant versus log ω for the difference in amine concentration at 30 volts. According to Joshi et al. (2017), the dielectric constant is high in the low-frequency zone, but as the frequency increases, the dielectric constant drops due to the difficulty of the electric dipole conforming. Based on Figure 4.2, it can be seen that the dielectric constants are different for all concentrations. For a concentration of 0.5 ml, the dielectric constant is lower compared to a concentration of 1.5 ml. At 30 volts, the dielectric constant is in the range of 0 to 8. Meanwhile, for log ω, the range is between 0 and 6. Significantly, the results shows that when log ω increases, the value of the dielectric constant decreases.

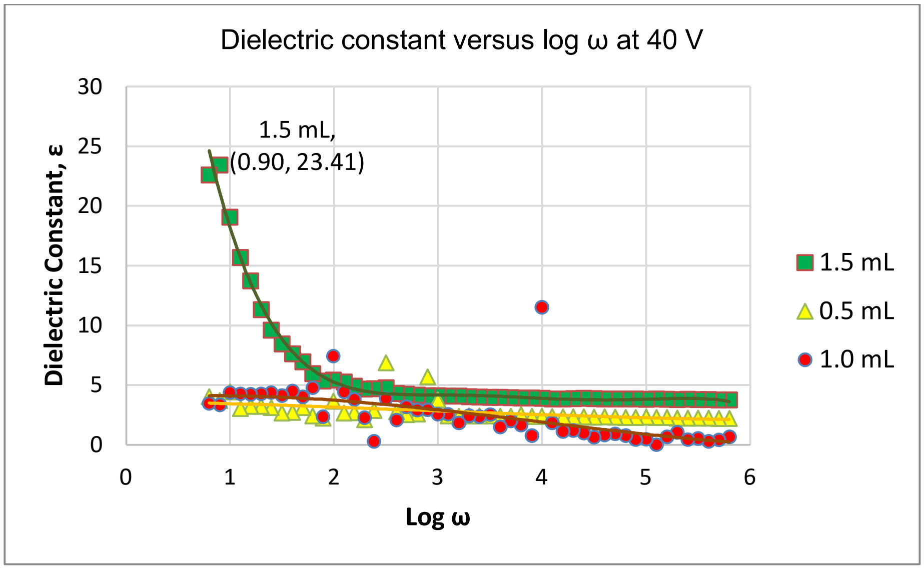


Figure 4.3 Dielectric Constant versus log ω at 40 volts

Whereas, Figure 4.3 demonstrates the dielectric constant versus log ω at 40 volts. In this figure, the amine concentration of 1.5 ml is clearly shown, and the dielectric constant is inversely proportional with a smooth curve. Otherwise, for amine concentrations of 0.5 ml and 1.0 ml, the dielectric constant is in the range of 0 to 12. Indeed, at 40 volts, the best curve of dielectric constant against log ω is 1.5 ml.

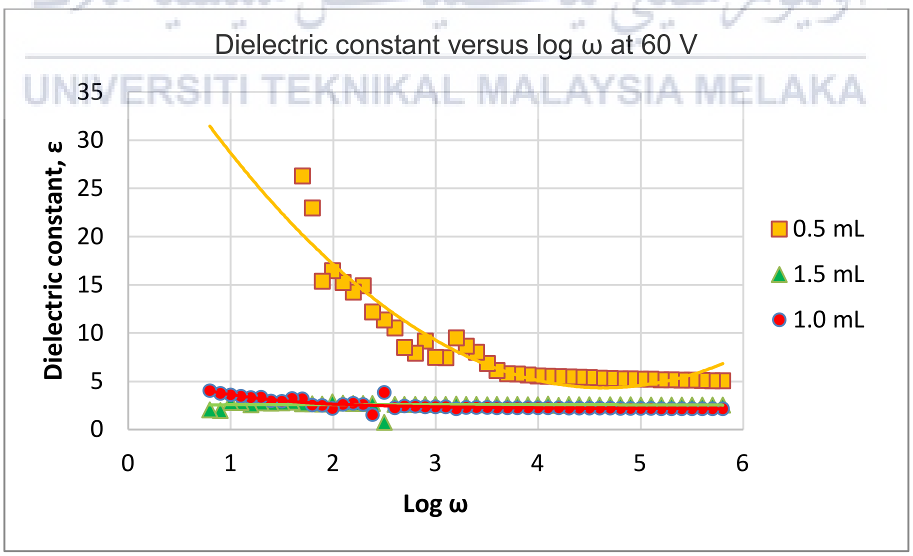


Figure 4.4 Dielectric Constant versus log ω at 60 volts

Moreover, Figure 4.4 depicts the dielectric constant against log ω at 60 volts. The graph in Figure 4.4 is almost similar to the graph's pattern in Figure 4.3. When the concentration is 0.5 ml, the curve is clearly inversely proportional, which means if the dielectric constant is decreases, the log ω is increases. Meanwhile, for 1.0 ml and 1.5 ml, the dielectric constant is lower compared to 0.5 ml which is the range of dielectric constant is between 0 to 5. Based on the Figure 4.4, as conclude, the amine concentration of 0.5 ml is the best curve compared to others.

To sum it up, the best concentration, according to Joshi et al. (2017), at 30 volts is 1.5 ml, at 40 volts is 1.5 ml, and at 60 volts is 0.5 ml. It is because Joshi et al. (2017) said that the greater the change in dielectric constant and log ω, the better the dielectric property of the coating substrate.

4.4 Coating's Molecular Structure and Microstructure

The structural characteristics of an epoxy coating are investigated using FTIR for chemical composition analysis, SEM for high-resolution imaging of surface morphology, and optical microscopy for defect detection. These methodologies give a full understanding of the epoxy coating, assisting in quality evaluation and optimisation for a variety of applications.

4.4.1 Fourier Transform Infrared Spectroscopy (FTIR)

Figure 4.5 shows the FTIR spectra of different types of material which are epoxy resin, secondary amine, and coating substrate by using reactor. The result of Figure 4.5 is taken before do the coating process is to examine the chemical composition of the material itself before adding with other solution. So, it will make it easier for the comparison to be done based on the chemical composition involved before and after the coating process.

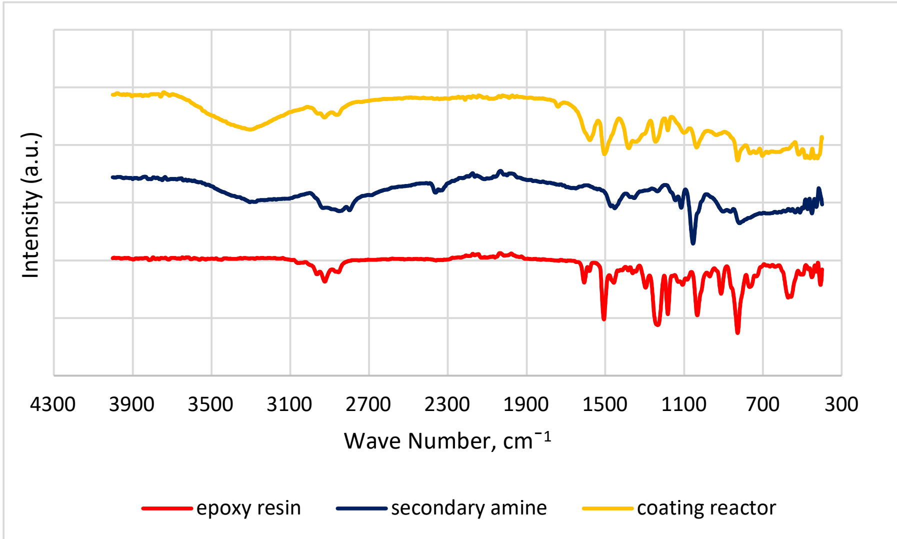


Figure 4.5 FTIR spectra of epoxy resin, secondary amine, and coating metal produced in ambient air environment.

Figure 4.5 demonstrates the difference in chemical bonding between the three compounds. According to Morsch et al. (2020), the region between 950 cm^{-1} - 400 cm^{-1} is responsible for the significant attenuations of numerous bands, including the symmetric stretching vibrations of C-N-C in secondary amine at 1004 cm^{-1}. Furthermore, at 3000 cm^{-1}–2800 cm^{-1}, peaks in the secondary amine spectrum of N-H stretching or amine groups (such as -NH or $-NH_2$) are occurring. There are several regions that are notably different when examined through the differences in the FTIR graph. Regions of 1084 cm^{-1}–958 cm^{-1} are among them. Based on the region, the chemical bonds of the peak contain an alkyl amine group, as mentioned by Morsch et al. (2020).

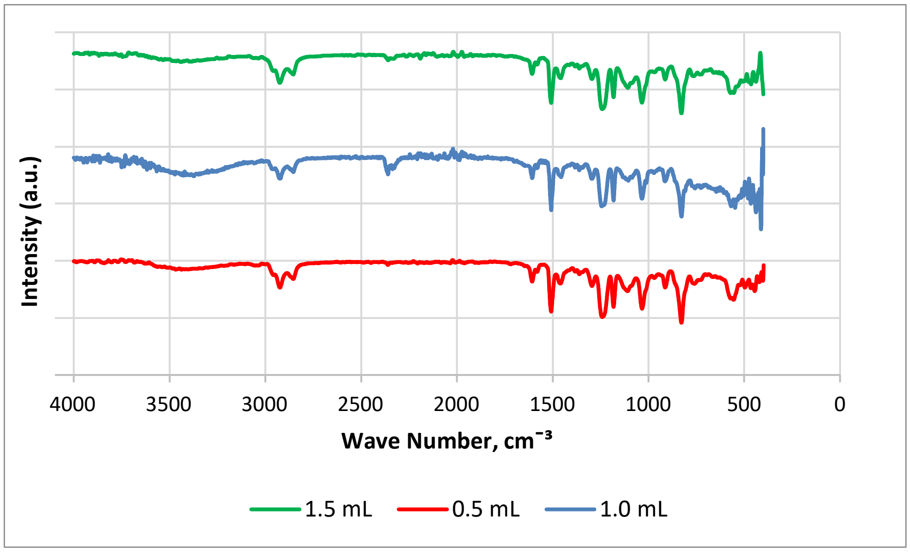


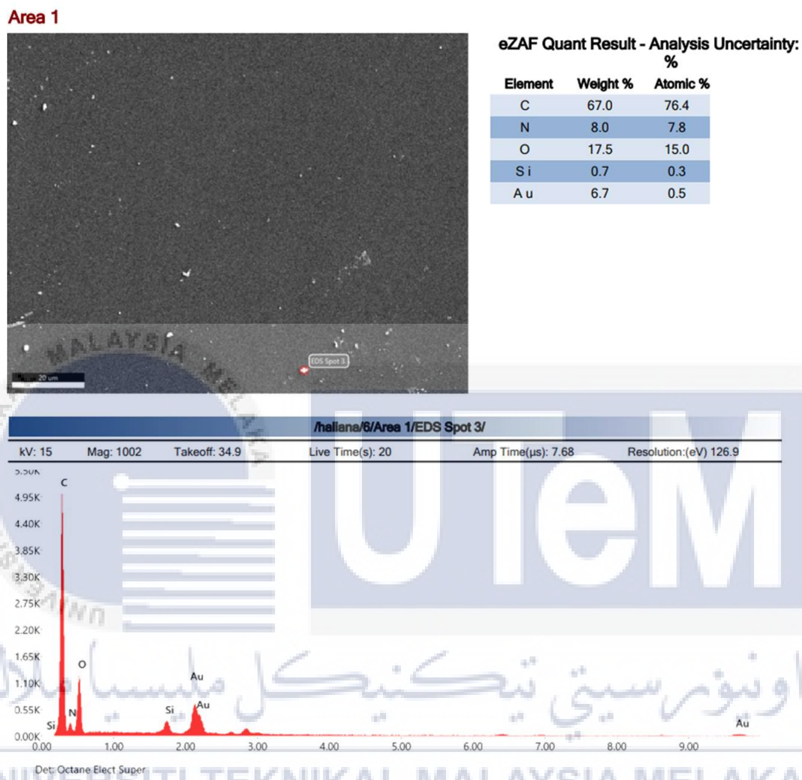
Figure 4.6 FTIR spectra of epoxy coating produced in N_2 gas environment with different amine concentration

The FTIR spectra in Figure 4.6 shown the three graph of coating chemical's bond with differences amine concentration which are 0.5 ml, 1.0 ml, and 1.5 ml. In addition, according to Deus et al. (2021), those connected to amine vibrations stood out among the wavenumbers that contributed the most to amine estimate, as expected of N-H bending vibration of basic amines can be ascribed to wavelengths between 1618 cm^{-1} and 1569 cm^{-1}. Furthermore, the N-H bending band is rarely found in the spectra of aliphatic secondary amines, but it is identified at 1515 cm^{-1} in the spectra of secondary aromatic amines.

To summarise, FTIR is extremely useful in determining the chemical composition or chemical bonds present in a sample. This substantially simplifies the scientific search, particularly the search for structural features.

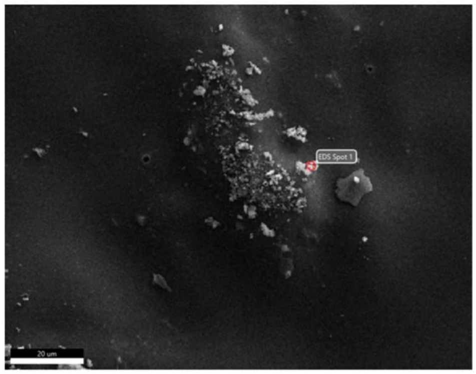
4.4.2 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) analysis revealed that the coating prevents any corrosion by forming a protective layer on the zinc surface. This coating was considered waterproof and useful as a protective film against the formation of any corrosion products. In order to zoom in on the coating surface, the magnification and resolution of the SEM are 20 µm and 126.9 eV. For SEM testing, the best-of nine solution is selected to view the coating surface in detail.



(a)

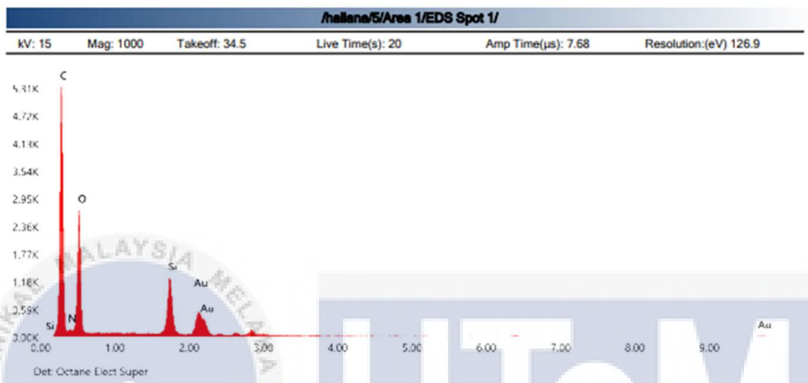
Area 1



eZAF Quant Result - Analysis Uncertainty: 13.14 %

Element	Weight %	Atomic %
C	64.5	73.4
N	2.9	2.8
O	26.0	22.2
S i	2.6	1.3
A u	4.1	0.3

(b)



Area 1

eZAF Quant Result - Analysis Uncertainty: 26.93 %

Element	Weight %	Atomic %
C	79.4	90.7
N	0.0	0.0
O	10.0	8.6
S i	0.0	0.0
A u	10.6	0.7

(c)

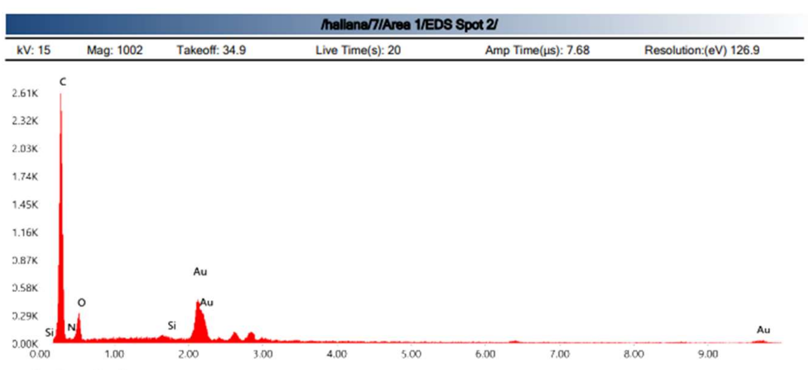


Figure 4.7 SEM images of differences amine concentration (a)0.5 ml, (b)1.0 ml, (c) 1.5 ml, at 60 volts during EPD process

Figure 4.7 depicts the coating surface of various amine concentration at 60 volts during EPD process. As mentioned in the result, there are several compositions found on the surface of the coating which are carbon, nitrogen, oxygen, silica, and aurum. The dominant composition is carbon for all amine concentration because has the highest percentage among all of the four compositions. The value of atomic's percentage for 0.5 and 1.0 ml is over 70%, but reaches 90% for 1.5 ml The lower composition onto the coating surface is silica and aurum. The percentage of silica is rises only 1.3% for 1.0 ml amine concentration. Then, based on the surface's images, we can see that surface of 0.5 ml is smoother compared to 1.0 and 1.5 ml.

To summarise, the image of a material can reveals its inherent flaws, such as fractures or structural irregularities, which can degrade the coating's integrity and reduce its efficacy. Assessing the substrate's status beforehand ensures the optimal application of the epoxy coating using EPD.

4.4.3 Optical Microscopy

The other way to determine the structural properties is by using optical microscopy. The magnification is change to 200μm to make sure that the surface images is clear. Figure 4.8 is represent the uncoated zinc substrate.

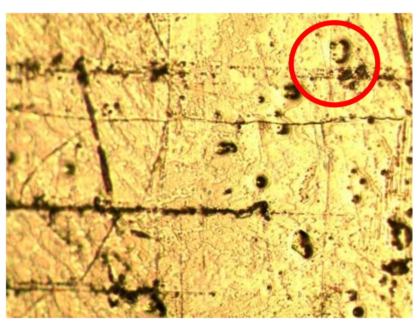
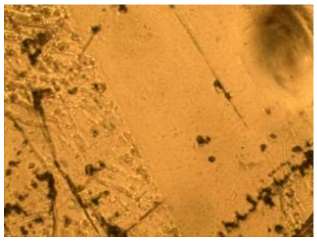
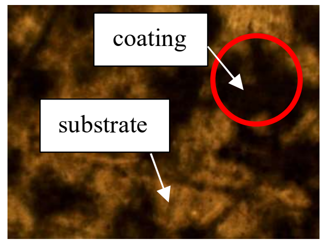
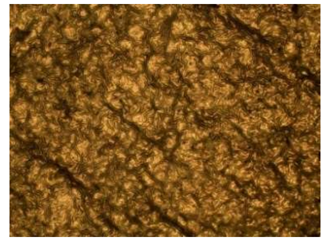
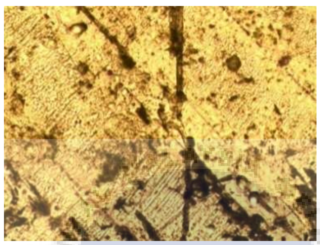
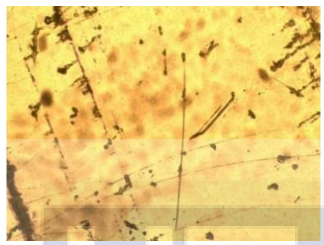
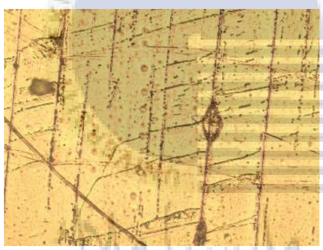
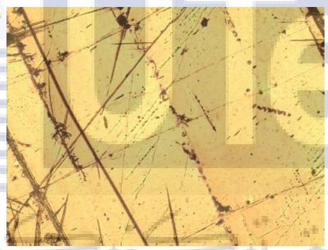


Figure 4.8 The image of an uncoated zinc surface

As depicted in Figure 4.8, the zinc surface is clearly unsmooth, rough, has a lot of scretch mark, and also has a porosity. Apart of that, epoxy coating is the bet method to make the zinc surface covered by another layer in order to prevent the material before rusting.

Table 4.3 Morphology surface of the coating substrate by using optical microscopy

	30 V	40 V	60 V
0.5			
1.0			
1.5			

There is a variation in the image for 60 volts EPD voltages while seeing the photographs of 0.5 ml. It might be because the bubble shows significantly more at higher volts than at lower levels. During EPD in aqueous solutions, higher voltage increased the number of bubbles in the solution and the oxidation of the substrate. When water decomposes into oxygen and hydrogen, bubbles emerge on the electrode surface, resulting in water electrolysis. Electrolysis was boosted by increasing the water volume percentage (Maciag et al., 2021).

Furthermore, as stated by Maciag et al., (2021), the darker region onto the substrate coating is the appearance of epoxy coating. Based on the surface picture obtained using optical microscopy, the optimal epoxy coating parameter is a 0.5 ml solution with an EPD voltage reading of 40 volts.

However, all of the parameters also produce a layer of coating on the surface of the substrate. As a result, the porosity seen in the Figure 4.8 has been covered by another layer, as shown in Table 4.3. The two porosity images are clearly different before and after the coating process.

4.5 Discussion

The optimal thickness of 0.5 ml and an EPD voltage of 30 volts are crucial for achieving optimal film uniformity, adhesion, and deposition efficiency. Understanding the mechanisms behind electrophoretic deposition and the applied voltage is essential for achieving a uniform and well-adhered layer, emphasizing the importance of precise control in the deposition process.

Electrochemical Impedance Spectroscopy (EIS) is a crucial technique for determine the value of dielectric constant. Based on the calculations of dielectric constant, a graph of dielectric constant against log ω is illustrated. From the result, the ideal curve of the dielectric constant versus log ω is 1.5 ml at 40 volts.

Comparative analysis with other thicknesses or EPD voltages can provide a deeper understanding of the trade-offs and advantages of the chosen parameters. Exploring potential applications where these dielectric properties play a crucial role emphasises the practical significance of these findings. The discussion can be expanded to highlight the material's potential and advantages in various fields, based on the observed optimal thickness and EPD voltage.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

The findings of the investigation were scrutinised in order to provide critical information and an analysis of the influence of secondary amine concentration on the dielectric property of epoxy coatings applied by electrophoretic deposition. The conclusions were formed based on the facts and discussion of the purpose given in Chapters 1 to 4.

5.1 Conclusion

Finally, this research has shed light on the effects of critical factors, notably amine concentration and EPD voltage, on the EPD process of epoxy coatings. The rigorous investigation of these factors has shown a complicated connection, demonstrating the deposition process's sensitivity to tiny variations in both amine concentration and EPD voltage. The findings highlight the significance of accuracy and optimisation in attaining the desired properties of epoxy coatings.

In addition, the analysis of amine concentration revealed that the thickness of the epoxy coating is intricately linked to the concentration of the amine solution used in the EPD process. The study demonstrated that a concentration of 0.5 ml yielded the most favourable coating thickness. This understanding is crucial for applications where precise coating thickness is a critical factor, such as in electronic components or protective layers. The interplay between amine concentration and coating thickness offers a blueprint for tailoring coatings with specific thicknesses, catering to the diverse requirements of various industries.

Furthermore, the investigation into the effects of EPD voltage on dielectric properties post-coating has unveiled noteworthy outcomes. The study found that an EPD voltage of 40 volts contributes to enhanced dielectric properties in the epoxy coating. This observation is paramount for applications demanding superior electrical insulation or specific dielectric characteristics. The correlation between EPD voltage, coating thickness, and dielectric properties provides a comprehensive understanding of the interrelated factors governing the material's electrical performance, paving the way for informed choices in material design and applications. In essence, this study contributes essential knowledge that can guide the optimisation of EPD parameters for tailored epoxy coatings with the desired thickness and superior dielectric properties.

5.2 Recommendations

For future improvements, accuracy of the epoxy coating estimation results could be enhanced. First of all, future research should explore the impact of electrophoretic deposition (EPD) on epoxy coating thickness and the dielectric properties. Investigating variations in amine concentration and EPD voltage levels beyond the optimum, as well as deposition time and temperature, can provide a comprehensive understanding of the process's sensitivity to different conditions. This will help tailor epoxy coatings for diverse applications.

The curing process of epoxy coatings requires a holistic approach that considers the curing environment, curing time, temperature parameters, and the interaction between curing conditions and the final mechanical and thermal properties of the coating. This holistic approach ensures that the coatings achieve full cure and exhibit the desired properties for their intended applications, thereby optimising the curing process for enhanced performance.

5.3 Project Potential

Through electrophoretic deposition (EPD), this study aims to unleash the full potential of epoxy coatings by exploring the effect of regulated secondary amine concentrations on the coating's thickness, microstructure, and dielectric characteristics. Because epoxy coatings are used in a variety of sectors to provide insulation and protection, it is critical to understand and optimise their properties. The purpose of this research is to contribute to the field by investigating the complex link between secondary amine concentration, coating microstructure, and dielectric performance.

This goal attempts to unveil the subtle impact on the thickness of coatings deposited using electrophoretic deposition by systematically altering the concentration of secondary amines in the epoxy formulation. Controlling coating thickness precisely is critical for modifying the protective capabilities of epoxy coatings in a wide range of applications, from electronics to industrial components.

This goal is to improve the microstructure of epoxy coatings by investigating and optimising the factors involved in electrophoretic deposition. Variations in voltage, deposition duration, and particle concentration will be examined in depth to gain a better understanding of their individual and combined impacts on the microstructure of the coating. This technique is expected to result in coatings with increased adhesion, homogeneity, and overall structural integrity.

The third goal is to use electrochemical impedance spectroscopy (EIS) to completely examine the dielectric properties of epoxy coatings. This study intends to give significant insights into the coating's impedance, capacitance, and overall dielectric behaviour by exposing coated surfaces to EIS analysis. The results of this examination will lead the formulation process towards coatings with optimised dielectric characteristics for use in electronic and electrical systems.

To sum it up, the outcomes of this research study have the potential to significantly advance the state-of-the-art in epoxy coating technology. Controlled secondary amine concentrations and modified EPD parameters are projected to provide optimised coatings with increased thickness, better microstructure, and superior dielectric characteristics. These breakthroughs have far-reaching ramifications for sectors that rely on epoxy-coated materials, such as electronics, energy systems, and aircraft, where reliable insulation and protection are critical. Finally, this project has the potential to not only improve our fundamental understanding of coating processes but also to provide practical solutions for optimising epoxy coatings, fostering innovation, and improving the performance and reliability of coated materials in a variety of industrial applications.

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APPENDICES

APPENDIX A Result of Turnitin

NORSHAZLINA FULL REPORT

ORIGINALITY REPORT

10%	4%	7%	4%
SIMILARITY INDEX	INTERNET SOURCES	PUBLICATIONS	STUDENT PAPERS

PRIMARY SOURCES

1	Xiaoming Liang, Xibo Sun, Qing Lu, Lu Ren et al. "VOC emission inventory of architectural coatings and adhesives for new buildings in China based on investigated and measured data", Atmospheric Environment, 2021 Publication	<1%
2	www.researchgate.net Internet Source	<1%
3	Submitted to Universiti Teknikal Malaysia Melaka Student Paper	<1%
4	W Suchanski. Journal of Physics Condensed Matter, 11/20/2000 Publication	<1%
5	Manju V., Rohith R, Anandhu T P, Bhavija B.V., Sreekanth J Varma. "Dielectric and Electrochemical Performance of Rhombohedral Lanthanum Manganite Perovskite Nanostructures", New Journal of Chemistry, 2022 Publication	<1%

APPENDIX B Gantt Chart for thesis project

No.	Task	Status	Week																							
			1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24
1	Discussion about research's flow with supervisor	Plan																								
		Actual																								
2	Purchased order for research's items	Plan																								
		Actual																								
3	Analyse the parameters uses in EPD process	Plan																								
		Actual																								
4	Finalize the parameters uses in EPD process	Plan																								
		Actual																								
5	Setup the place for EPD process	Plan																								
		Actual																								
6	Run EPD process : with 0.5 mL amine concentration	Plan																								
		Actual																								
7	Curing process : in dry oven for 2 days (0.5 mL)	Plan																								
		Actual																								
8	Booking the FTIR machine for characterization	Plan																								
		Actual																								
9	Run EPD process : with 1.0 mL amine concentration	Plan																								
		Actual																								
10	Curing process : in dry oven for 2 days (1.0 mL)	Plan																								
		Actual																								
11	Characterization by using FTIR analysis (0.5 & 1.0 mL)	Plan																								
		Actual																								
12	Run EPD process : with 1.5 mL amine concentration	Plan																								
		Actual																								
13	Curing process : in dry oven for 2 days (1.5 mL)	Plan																								
		Actual																								

14	Book the slot at MMU for EIS characterization	Plan
		Actual
15	Characterization by using FTIR analysis (1.5 mL)	Plan
		Actual
16	Characterization by using EIS at MMU (all sample)	Plan
		Actual
17	Characterization by using SEM at MMU (all sample)	Plan
		Actual
18	Analyse the results from EIS, FTIR, & SEM characterization	Plan
		Actual
19	Present the results to the supervisor	Plan
		Actual
20	Preparation for the final report and presentation session	Plan
		Actual

APPENDIX C Excel Formula for dielectric constant calculation

	A	B	C	E	F	G	H	I	J	K	L	M	N	O	P	Q
1	Hz			m	m2		permittivity									
2	**Freq**	**Z' (a)**	**Z'' (b)**	**Radius**	**Area**	**logf**	**e0**	**Z'2**	**Z''2**	**Z'2 + Z''2**	**Z''/Z'2+Z'**	**Z'/Z'2+Z''2**	**Omega**	**Ae0**	**wAe0**	**t/wAe0**
3	100078	748.1436	19560.39	0.009	0.00025	5.00034	8.85E-12	559719	382608857	383168576	5.1E-05	1.9525E-06	6.28E+05	2.25E-15	1.42E-09	9.93E+04
4	79453.1	769.8564	24590.39	0.009	0.00025	4.90011	8.85E-12	592679	604687280	605279959	4.1E-05	1.2719E-06	4.99E+05	2.25E-15	1.12E-09	1.25E+05
5	63140.6	1075.285	30795.15	0.009	0.00025	4.80031	8.85E-12	1156238	948341264	949497501	3.2E-05	1.1325E-06	3.97E+05	2.25E-15	8.93E-10	1.57E+05
6	50203.1	1395.395	38521.54	0.009	0.00025	4.70073	8.85E-12	1947127	1483909044	1485856171	2.6E-05	9.3912E-07	3.15E+05	2.25E-15	7.10E-10	1.98E+05
7	39890.6	1656.04	48349.64	0.009	0.00025	4.60087	8.85E-12	2742468	2337687688	2340430157	2.1E-05	7.0758E-07	2.51E+05	2.25E-15	5.64E-10	2.49E+05
8	31640.6	2037.88	60629.87	0.009	0.00025	4.50025	8.85E-12	4152955	3675981136	3680134091	1.6E-05	5.5375E-07	1.99E+05	2.25E-15	4.47E-10	3.14E+05
9	25171.9	3177.716	75939.41	0.009	0.00025	4.40092	8.85E-12	1E+07	5766793991	5776891870	1.3E-05	5.5007E-07	1.58E+05	2.25E-15	3.56E-10	3.95E+05
10	20015.6	3666.342	94846.26	0.009	0.00025	4.30137	8.85E-12	1.3E+07	8995813036	9009255100	1.1E-05	4.0695E-07	1.26E+05	2.25E-15	2.83E-10	4.96E+05
11	15890.6	4556.284	118977.3	0.009	0.00025	4.20114	8.85E-12	2.1E+07	1.4156E+10	1.4176E+10	8.4E-06	3.214E-07	9.98E+04	2.25E-15	2.25E-10	6.25E+05
12	12609.4	6196.476	149631.4	0.009	0.00025	4.10069	8.85E-12	3.8E+07	2.239E+10	2.2428E+10	6.7E-06	2.7628E-07	7.92E+04	2.25E-15	1.78E-10	7.88E+05
13	10078.1	8576.835	186198	0.009	0.00025	4.00338	8.85E-12	7.4E+07	3.467E+10	3.4743E+10	5.4E-06	2.4686E-07	6.33E+04	2.25E-15	1.43E-10	9.86E+05
14	8015.63	11377.78	233305.3	0.009	0.00025	3.90394	8.85E-12	1.3E+08	5.4431E+10	5.4561E+10	4.3E-06	2.0853E-07	5.03E+04	2.25E-15	1.13E-10	1.24E+06
15	6328.13	18314.84	292456.6	0.009	0.00025	3.80128	8.85E-12	3.4E+08	8.5531E+10	8.5866E+10	3.4E-06	2.1329E-07	3.97E+04	2.25E-15	8.95E-11	1.57E+06
16	5015.63	26111.03	365890.1	0.009	0.00025	3.70033	8.85E-12	6.8E+08	1.3388E+11	1.3456E+11	2.7E-06	1.9405E-07	3.15E+04	2.25E-15	7.09E-11	1.98E+06
17	3984.38	37261.79	455442	0.009	0.00025	3.60036	8.85E-12	1.4E+09	2.0743E+11	2.0882E+11	2.2E-06	1.7844E-07	2.50E+04	2.25E-15	5.63E-11	2.49E+06
18	3170.96	46999.4	568235.4	0.009	0.00025	3.50119	8.85E-12	2.2E+09	3.2289E+11	3.251E+11	1.7E-06	1.4457E-07	1.99E+04	2.25E-15	4.48E-11	3.13E+06
19	2527.57	68446.56	707449.9	0.009	0.00025	3.4027	8.85E-12	4.7E+09	5.0049E+11	5.0517E+11	1.4E-06	1.3549E-07	1.59E+04	2.25E-15	3.57E-11	3.93E+06
20	1976.1	93975.33	893646.9	0.009	0.00025	3.29581	8.85E-12	8.8E+09	7.986E+11	8.0744E+11	1.1E-06	1.1639E-07	1.24E+04	2.25E-15	2.79E-11	5.03E+06
21	1577.52	156017.3	1105239	0.009	0.00025	3.19798	8.85E-12	2.4E+10	1.2216E+12	1.2459E+12	8.9E-07	1.2523E-07	9.91E+03	2.25E-15	2.23E-11	6.30E+06
22	1265.63	268111.7	1330135	0.009	0.00025	3.10231	8.85E-12	7.2E+10	1.7693E+12	1.8411E+12	7.2E-07	1.4562E-07	7.95E+03	2.25E-15	1.79E-11	7.85E+06
23	998.264	476123.4	1576776	0.009	0.00025	2.99925	8.85E-12	2.3E+11	2.4862E+12	2.7129E+12	5.8E-07	1.755E-07	6.27E+03	2.25E-15	1.41E-11	9.95E+06
24	796.875	469513.4	2043812	0.009	0.00025	2.90139	8.85E-12	2.2E+11	4.1772E+12	4.3976E+12	4.6E-07	1.0677E-07	5.00E+03	2.25E-15	1.13E-11	1.25E+07
25	627.79	1006808	2088895	0.009	0.00025	2.79781	8.85E-12	1E+12	4.3635E+12	5.3771E+12	3.9E-07	1.8724E-07	3.94E+03	2.25E-15	8.88E-12	1.58E+07
26	505.515	1326742	1961178	0.009	0.00025	2.70373	8.85E-12	1.8E+12	3.8462E+12	5.6065E+12	3.5E-07	2.3665E-07	3.17E+03	2.25E-15	7.15E-12	1.96E+07