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RESEARCH ARTICLE

EFFECT OF APPLIED VOLTAGE AND EPOXY SUSPENSION'S AGEING TIME ON SHEET RESISTANCE OF ELECTRODEPOSITED EPOXY COATINGS

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Abstract. This study investigates the effect of applied voltages and epoxy suspension's ageing time on the sheet resistance of electrodeposited epoxy coatings. Electrophoretic deposition (EPD) was employed as a coating technique due to its precise control over thickness and uniformity, making it suitable for electrical applications. Different applied voltages (i.e. 30, 40, and 60 V) and epoxy ageing times (1, 72, and 144 hours) were examined to understand their impact on the resulting coating's sheet resistance. Characterisations of the electrodeposited epoxy coatings were conducted using the four-point probe method, weight gain measurement, field emission scanning electron microscopy and energy-dispersive X-ray spectroscopy. The results indicate that both the applied voltage and epoxy suspension's ageing time significantly affect coating thickness and sheet resistance, with highest sheet resistance value of 818.0 9 kΩ/sq. is achieved at an applied voltage of 60 V and an hour ageing time. Higher voltages initially increase sheet resistance, but this effect diminishes with longer ageing times. These findings are essential for industries utilising epoxy coatings, suggesting that adopting the optimal applied voltage and epoxy suspension's storing time can enhance coating performance and reliability. The study provides insight into tailoring electrodeposited epoxy coatings for customised sheet resistance properties, contributing to advancements in materials for electrical application.

Keywords: Electrophoretic deposition, epoxy resin, electrical conductivity, grain boundaries, sheet resistance

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1. INTRODUCTION

The field of materials science continually seeks to enhance coatings that protect and improve the properties of various substrates in industrial applications [1]. Among these, epoxy-based materials are notable for their exceptional mechanical strength, chemical resistance, and electrical properties [2]. They are widely used to mitigate corrosion, providing a protective barrier that prevents environmental interactions with metal surfaces [3,4].

The effectiveness of epoxy coatings relies on factors such as surface preparation, application technique, curing time, and environmental conditions. Proper surface preparation is crucial for optimal adhesion, requiring thorough cleaning to remove dirt and oil, and possibly roughening the surface through sanding or blasting [2]. Electrophoretic deposition (EPD) is a preferred technique for applying epoxy coatings, offering precise control over thickness and uniformity essential for consistent performance [5]. The process involves applying current or voltage to induce epoxy resin deposition on conductive substrates, resulting in solid coatings upon curing [6]. To achieve optimal coating performance, comprehensive studies on EPD suspension preparation are necessary, as stability and uniformity of the suspension directly affect deposition quality [7]. Key factors to optimize include particle size, concentration, surface charge, dispersant choice, and solvent selection. Understanding the interactions among these components is vital for controlling coating properties such as thickness, adhesion, and surface roughness [8].

Coating properties are also influenced by deposition conditions, especially the applied voltage during electrodeposition [9]. Applied voltage and the ionic state of EPD suspension define the resulted current and suspension stability, which are critical as they affect the microstructure, thickness, and cross-linking density of the epoxy coating, directly impacting its electrical property. The electrical characteristics of electrodeposited epoxy coatings play a crucial role in their effectiveness across various applications, including antistatic coating, and corrosion prevention [1,10]. Epoxy coatings are widely recognized for their superior mechanical strength and chemical resistance, making them suitable for demanding environments. However, their performance as conductor is especially vital in applications where electrical reliability is paramount [10]. Despite the importance of these characteristics, the relationships between the parameters involved in the electrodeposition process and the resulting sheet resistance remain inadequately defined, creating a gap in knowledge that this research aims to address.

The primary aim of this study will investigate how the EPD voltage and epoxy ageing time affect the electrodeposited (EPD) epoxy coating thickness and sheet resistance of cured epoxy coatings. By addressing these objectives, the research seeks to enhance understanding of the factors influencing the sheet resistance of epoxy coatings, ultimately contributing to improved coating performance in various applications. The research finding is to identify optimal conditions that can improve the electrical properties of the coatings, which contribute to the development of predictive models that facilitate better decision-making in coating processes, paving the way for advancements in material formulations and manufacturing techniques that enhance sustainability and product longevity.

2. MATERIALS AND METHODS

2.1 Modified Epoxy Suspension Preparation

A commercially available diglycidyl ether of bisphenol A (DGEBA), identified as Auto-Fix 8800-A from Chemibond Enterprise, was used as the starting material for the cationisation reaction process. According to Chemibond Enterprise [11], the as-received DGEBA liquid has a density of 1.13 g/ml, an epoxide equivalent weight (EEW) of 198–205 g/eq, and a dynamic viscosity ranging from 500 to 1,000 centipoise. The synthesis of cationic DGEBA epoxy resin suspensions involved a chemical reaction with six other reagent-grade chemicals, including N-Methylethanolamine (MEA), as detailed in Table 1. The formulations of the chemical reactants and the heating profile were adapted from the methodologies described by Bosso et al. [12] and Wismer et al. [13].

Table 1: Chemical Reactants for the Synthesis of Cationic Epoxy Resin Suspension [12,13]

Phase	Item No.	Chemical Reactant	Quantity (ml)
1. Modified Epoxy Resin Formation	1	DGEBA Epoxy Resin	420 ml
	2	Dimethylether of diethylene glycol (Solvent)	60 ml
	3	Monoalcohol A (1- Octanol)	123 ml
	4	Stannous Chloride	4.5 gram
2. Cationic Epoxy Resin Formation	5	N-Methylethanolamine (MEA)	1.5 ml
	6	Deionized water	180 ml
	7	Formic acid	0.3 ml

Initially, the as-received DGEBA liquid and the other components (Item No. 2-4) were mechanically stirred in a jacketed glass reactor, which was heated by a connected heating circulator. The mixture was maintained at a constant temperature of 130 °C for 2 hours and 50 minutes under an inert environment, created by flowing nitrogen gas (10 ml/min, 99.9% purity) into the reactor. This consistent flow of nitrogen removed air and minimized oxidation, thereby reducing unwanted reactions. Despite being initially immiscible, the mixture became homogeneous with continued stirring and heating. After this period, the mixture was allowed to cool to a temperature range of 70-92 °C before gradually adding MEA and the remaining chemicals (Items No. 5-7 in Table 1). The amount of MEA was fixed at 1.5 ml. MEA served as a cationisation agent to modify the as-received DGEBA polymer into cationic DGEBA polymer suspension. Once all components were added, the heating circulator was turned off to commence the natural cooling process, which took approximately 2-3 hours. Mechanical stirring and nitrogen flow continued until the cationic epoxy suspension reached room temperature. The suspension was divided equally into three suspensions (labelled as 1 H, 72 H, and 144 H) and then were stored in a dark place for 1 hour, 72 hours (3 days) and 144 hours (6 days) respectively. Each of the epoxy suspensions was then used separately in the electrophoretic deposition (EPD) process to deposit epoxy coating with the purpose to study the effect of the aging (storing) time on the epoxy coating's properties.

2.2 Electrophoretic Deposition Current and Electric Conductivity Characterisations

Electrophoretic deposition (EPD) employs two galvanised steel plates (length = 45 mm, width = 30 mm, thickness = 0.14 mm) as the anode and cathode, arranged in parallel with a 10 mm separation. Both electrodes, submerged 30 mm deep in the prepared cationic epoxy suspension, facilitate the deposition process. Deposition was performed at varying voltage levels (30, 40, and 60 V) for 15 minutes.

The EPD setup includes a DC power supply (model E3643A, with voltage ranges of 0–35 V or 0–60 V and current ranges of 1.4 A or 0.8 A), a digital multimeter (DMM, model 34465A), four probe wires, Keysight software for real-time monitoring of DMM readings, and a beaker containing 50 ml of the as-prepared water-based cationic epoxy resin. The Keysight software records the EPD current at deposition time-zero and time 15-minutes to monitor the EPD kinetic of the epoxy suspension.

The electrical conductivity of the EPD solutions was measured by a standard conductivity meter (model: 123-8777, brand: RS PRO) before and immediately after the EPD process to monitor the suspension stability condition.

2.3 Epoxy Coating Characterisations

Nine epoxy coating samples were obtained from the EPD process and subsequently cured in a dry oven at 100 °C for 24 hours. All characterisations of the cured epoxy coatings were carried out under ambient temperature and pressure conditions. The weight of epoxy deposited on the galvanised iron substrate was measured using a digital weighing balance (resolution: 0.001 grams, Explorer) and then the deposition weight of the epoxy coating was converted into the coating thickness through calculation [14]. The surface morphologies of the epoxy coating samples were analysed using field-emission scanning electron microscopy (FESEM) and Energy Dispersive X-ray Spectroscopy (EDS) with a Hitachi High-Tech SU5000 instrument. To improve the FESEM imaging, the coating samples were coated with a thin layer of gold-palladium (Au-Pd) using sputter coater (Qurom SC7620, applied current = 20 mA, deposition time = 180 s). JG M-3 Mini four-probe tester was used to measure sheet resistance (Rs) of the epoxy coatings for the electrical characterisation.

3. RESULTS AND DISCUSSION

3.1 Electrical Conductivity of Cationic Epoxy Suspensions and Related Electrophoretic Deposition Current

Table 2 presents the electrical conductivity of EPD suspensions and the EPD currents recorded at the beginning and end of the EPD process for various applied voltages and suspension ageing times. Figure 1 illustrates that the systematic decrease in both electrical conductivity and EPD current suggests a strong correlation between the reduction in EPD current and the depletion of ionic species in the epoxy suspension during the EPD process. This occurs because the generation of EPD current involves the movement of cations and anions within the suspension. As more of these ions are deposited and neutralised at the cathode and anode, fewer remain in the suspension, leading to a decline in electrical conductivity and, consequently, a reduction in current flow within the EPD cell. It is also evident that the effect of applied voltage is more significant than that of suspension ageing time on the electrical conductivity and EPD current, as greater decreases are observed with increasing applied voltage.

Table 2: Electrical conductivity of EPD suspensions and EPD currents recorded during EPD at different applied voltages and suspension ageing times. Data of the starting and the ending EPD conditions are labelled as t = 0 and t = 15 minutes

Ageing Time (hour)	Voltage (V)	Electrical Conductivity meter (mS.cm)			Deposition Current (mA)		
		t = 0 min	t = 15 min	Change	t = 0 min	t = 15 min	Change
1	30	11.50	8.60	-2.90	2.98	1.68	-1.30
	40	13.10	6.90	-6.20	3.96	2.29	-1.67
	60	13.10	6.80	-6.30	5.49	1.18	-4.31
72	30	12.20	9.20	-3.00	4.27	1.07	-3.20
	40	13.40	7.00	-6.40	5.14	2.71	-2.43
	60	14.10	7.80	-6.30	5.66	1.10	-4.56
144	30	13.60	10.00	-3.60	5.05	3.92	-1.13
	40	10.20	3.80	-6.40	6.04	3.77	-2.27
	60	14.40	7.90	-6.50	6.25	0.90	-5.35

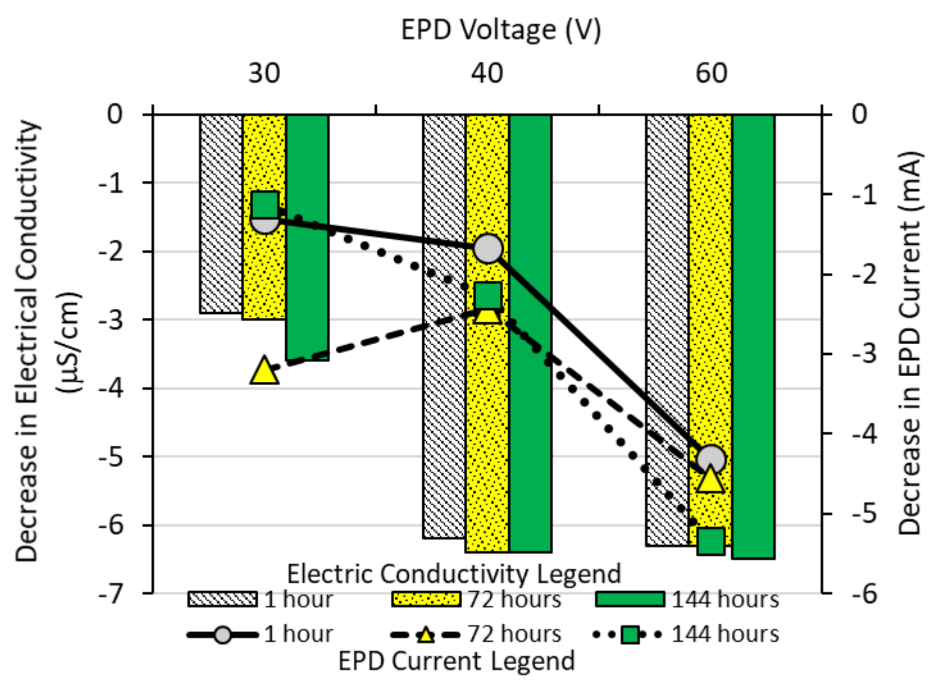

Figure 1: Decrease of electrical conductivity (indicated by bar chart) of EPD suspensions and EPD current (indicated by line chart) after 15 minutes deposition as function of EPD voltages and suspension's ageing times

3.2 Surface Microstructure of Epoxy Coating

Low and high magnification field emission scanning electron microscopy (FESEM) micrographs (Figures 2 and 3) provide a detailed view of the surface microstructure of epoxy coatings provided at different applied voltages using suspensions with different ageing times.

The surface microstructure of the coatings (Figure 2) are largely uniform and smooth, but rough microstructures are randomly appeared due to deposition of solid epoxy debris during the EPD process. It is suspected that the epoxy debris originate from the fracture of cured epoxy layers formed on the glass reactor wall during the cationic epoxy synthesis process. High magnification observations (Figure 3) at the uniform and smooth surface reveals a uniform distribution of cracks throughout the epoxy coatings irrespective of the EPD voltage and the ageing time. Thus, it is suggested the main factor causing the crack formation is thermal stress formation during the curing process in the drying oven. Detailed examinations on the high magnification micrographs (Figure 4) indicates a prominent difference in the microstructure of the cracks on the epoxy coating (shown as black coloured lines) and grain boundaries of the gold-palladium sputter coating (shown as grey coloured lines). The microstructure of the latter is similar to the previous reported study on the microstructure of gold-palladium sputter coating [15].

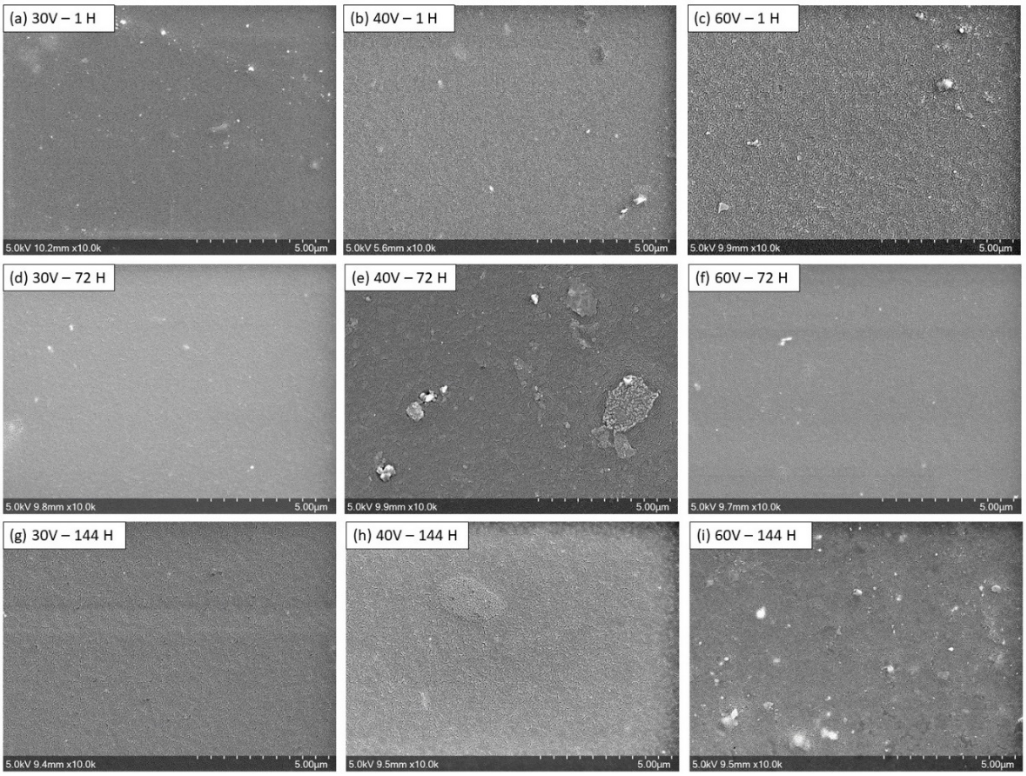

Figure 2: Low magnification (10,000×) surface micrographs of epoxy coatings deposited at different EPD voltages (i.e. 30, 40 and 60 V) using the cationic epoxy suspensions that underwent ageing time of 1 hour (a, b, c), 72 hours (d, e, f), and 144 hours (g, h, i)

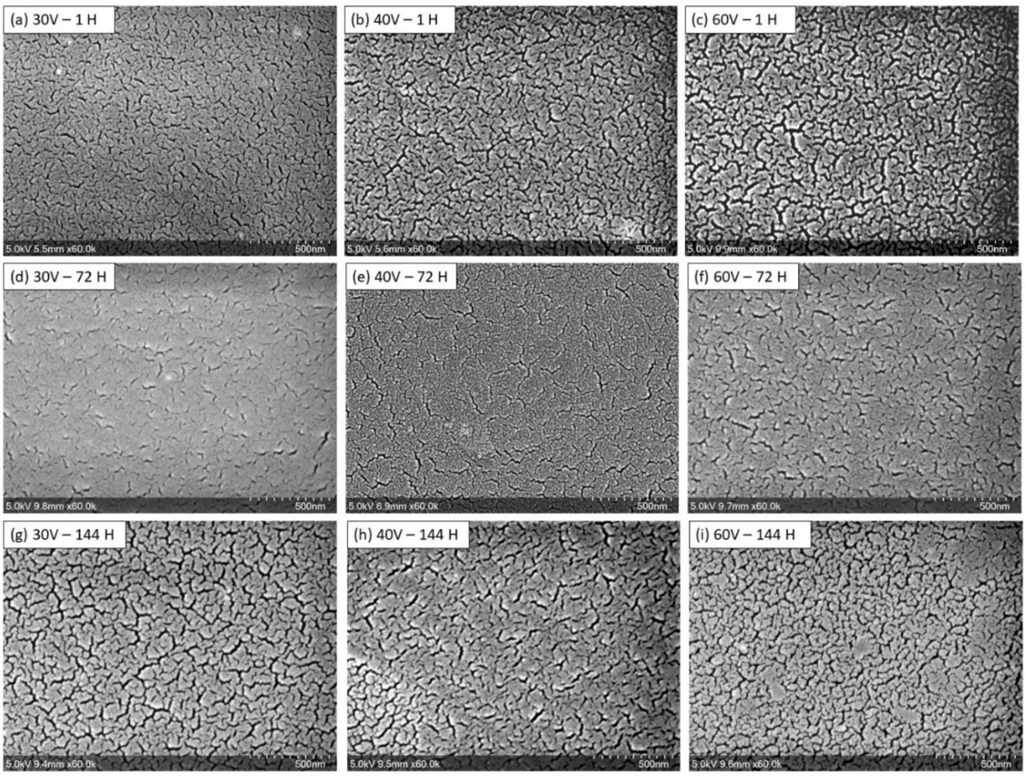

Figure 3: High magnification (60,000×) surface micrographs of epoxy coatings obtained at different EPD voltages (i.e. 30, 40 and 60 V) using the cationic epoxy suspensions that underwent ageing time of 1 hour (a, b, c), 72 hours (d, e, f), and 144 hours (g, h, i)

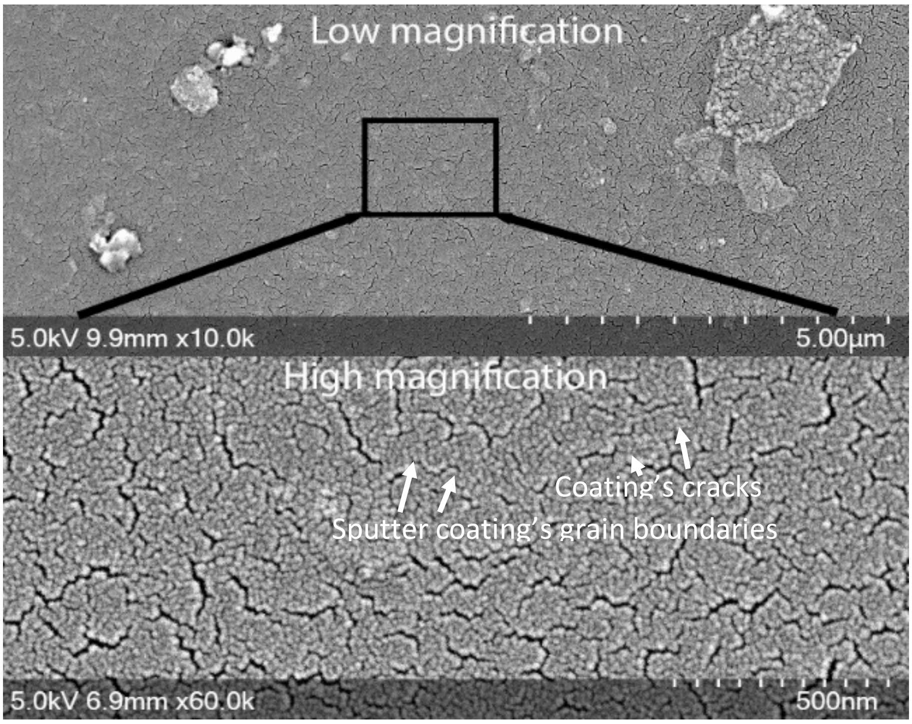

Figure 4: Cracks on the epoxy coating (black lines) and grain boundaries of the gold-palladium sputter coating (grey lines, as observed in the high magnification micrograph) for the coating prepared using EPD voltage of 40 V and suspension's ageing time of 72 hours

To analyse the elemental composition, Energy Dispersive X-ray Spectroscopy (EDS) can be employed. The EDS results show the presence of carbon, oxygen and nitrogen on the coating's surface, as illustrated in Table 3.

Table 3: EDS data of deposited epoxy coatings deposited at different applied voltages using suspensions with different ageing times

Ageing Time (hour)	**Voltage (V)**	**Element's Concentration (at%)**		
		Carbon	**Oxygen (%)**	**Nitrogen (%)**
1	30	88.6	10.9	0.9
	40	87.3	11.9	1.7
	60	92.7	7.1	1.1
72	30	87.6	11.4	1.5
	40	86.2	12.6	0.9
	60	88.3	11.3	1.3
144	30	87.6	11.5	1.2
	40	87.9	10.9	1.7
	60	84.6	14.8	1.5

3.3 Sheet Resistance of Epoxy Coating

Figures 5 and 6 show the thickness and sheet resistance of epoxy coatings deposited at different voltages using epoxy suspension from different ageing times. The sheet resistance measurements indicate a clear relationship between ageing time and sheet resistance. However, it is demonstrated that the decremental effect of suspension’s ageing time on the coating thickness is less significance than the incremental effect of the applied voltage. Higher EPD voltages initially lead to increased sheet resistance as result of increased coating thickness, this effect diminishes with prolonged suspension’s ageing which reduced the coating thickness. For an ageing time of 1 hour, sheet resistance and coating thickness increases with applied voltage, from 758.2 kΩ/sq. and 98.98 µm at 30 V to 818.0 kΩ/sq. and 157.51 µm at 60 V, suggesting that higher voltages enhance the epoxy's cross-linking density and EPD yield performance. With an ageing time of 72 hours, sheet resistance values are slightly higher than the 1-hour coating samples at lower voltages but increase less significantly at higher voltages, with 30 V at 776.6 kΩ/sq. and 60 V at 794.0 kΩ/sq. The coating thickness is relatively lower than the 1-hour coating samples. For an ageing time of 144 hours, sheet resistance values decrease, ranging from 746.9 kΩ/sq. at 30 V to 767.6 kΩ/sq. at 60 V. The respective coating thicknesses are the lowest in the similar voltages. The overall trends indicate that longer ageing time deteriorates the sheet resistance and EPD yield performance.

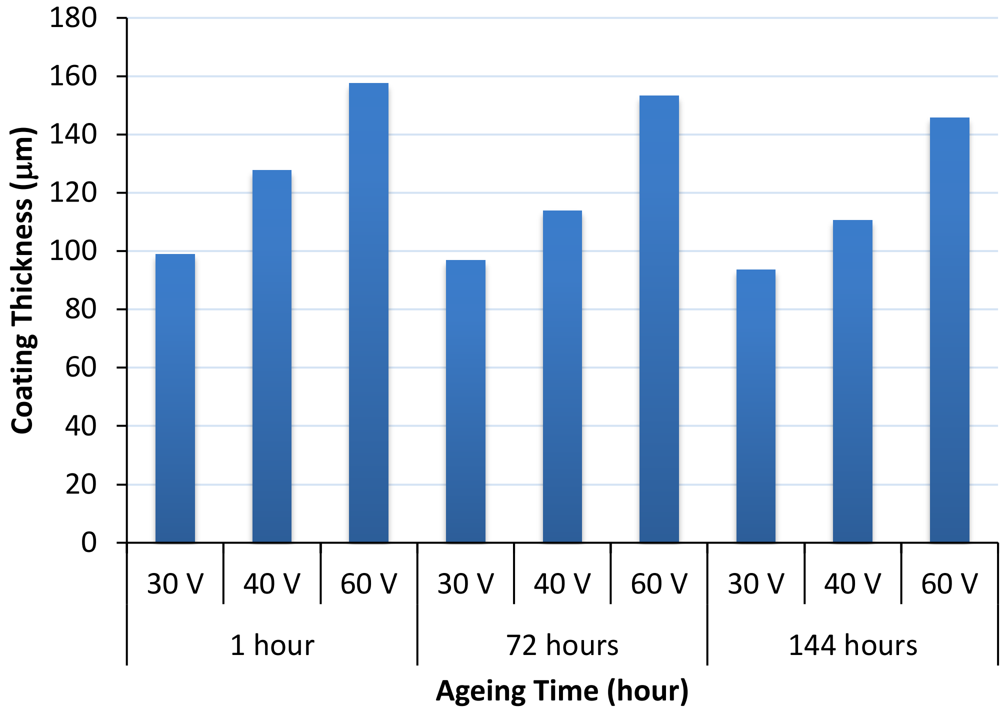

Figure 5: Calculated thickness of cured epoxy coating samples deposited at different applied voltages using suspensions with different ageing times

The decrease in the EPD yield performance when using suspension with longer aging time is likely due to the increased viscosity of the aged epoxy suspension as a result of cross-linking [16]. The increase in viscosity of the EPD suspension negatively affects electrophoretic mobility and, consequently, the electrophoretic yield [17]. Furthermore, longer ageing times reduce the sheet resistance of the epoxy coatings, suggesting that extended aging time of the EPD suspension also alter the cross-linking behaviour in the cured epoxy coating, resulted in reduced sheet resistance. Wang et al. reported that the formation of cross-linking points influences the dielectric constant of cured epoxy [18].

The sheet resistance of the coated samples is significantly higher than that of the uncoated substrate (Rs = 2.215 mΩ/sq.), demonstrating the effectiveness of the epoxy coating in enhancing surface resistivity. Therefore, the epoxy coatings exhibit much higher sheet resistance than the bare substrate. Both the EPD suspension's ageing time and the EPD voltage are critical for optimization. The best performance is achieved with an ageing time of 1 hour and an EPD voltage of 60 V, offering a balance between high sheet resistance, coating uniformity, and thickness stability. These findings underscore the importance of carefully controlling both the ageing time and the EPD voltage to achieve optimal electrical performance in epoxy coatings.

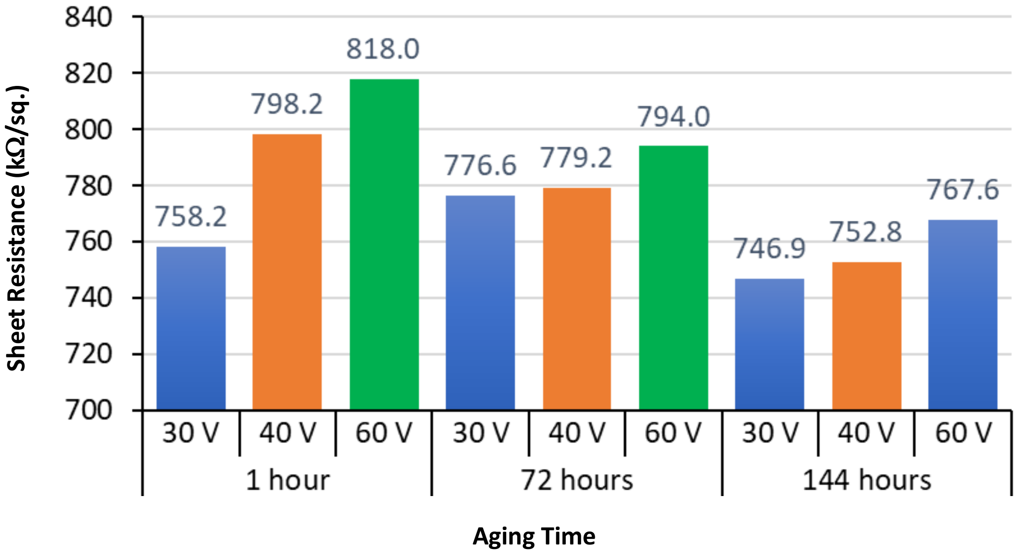

Figure 6: Sheet resistance (*R_s*) of cured epoxy coating samples deposited at different applied voltages using suspensions with different ageing times

4. CONCLUSIONS

The impact of EPD voltage and suspension's ageing time on the sheet resistance of electrodeposited epoxy coatings, revealing several key findings: optimal EPD voltage-suspension storing time settings for maximizing coating thickness were identified, with 60 V and an hour ageing time achieving the highest thickness of 153.26 µm. The characterization of sheet resistance indicated that optimized profiles enhance resistive properties, as demonstrated by sheet resistance values at an hour ageing time of 758.2 kΩ/sq. at 30 V, 798.2 9 kΩ/sq. at 40 V, and 818.0 kΩ/sq. at 60 V. Additionally, these findings have important implications for industrial applications such as electrical and corrosion prevention, allowing for improved resistive properties and reliability of epoxy coatings. Overall, the research provides valuable insights into the relationship between electrodeposition parameters and sheet resistance, contributing to advancements in coating technologies.

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Author Contributions

All authors contributed toward data analysis, drafting, and critically revising the paper and agree to be accountable for all aspects of the work. Nik Ahmad Luqmanul Hakim Nik Ab Rashid supporting facility and equipment, data collection and analyzed the data. Nurhaliana Shazwani Halim wrote the original manuscript and performed data analysis. Kok-Tee Lau, provided the main conceptual ideas, performed analyses and edited the manuscript. Kok-Tee Lau and Umar Al-Amani Azlan planned and supervised the work.

Disclosure of Conflict of Interest

The authors declare that they have no conflict of interest.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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