



Dielectric Property of Epoxy Coating Deposited using Electrophoretic Deposition Suspension Synthesised under Ambient and Inert Atmospheres

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ABSTRACT

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This study investigates the implications of N-methylethanolamine (MEA) concentrations and electrophoretic deposition (EPD) suspension synthesised under ambient air atmosphere and inert nitrogen gas environment on the chemical composition and dielectric properties of epoxy coatings. Cationised epoxy resin suspensions were synthesised from the modification of commercially available diglycidyl ether bisphenol A (DGEBA) epoxy using different MEA concentrations under ambient air and inert nitrogen gas environments. The cationic DGEBA suspension was then used in the EPD process to deposit an epoxy coating on a galvanised iron sheet. After elevated temperature curing, the epoxy coatings were characterised by Electrochemical Impedance Spectroscopy (EIS), Fourier-transform Infrared Spectroscopy (FTIR), Field-emission Scanning Electron Microscopy (FESEM), and Energy Dispersive Spectroscopy (EDS) analysis. The epoxy solution synthesised under the inert nitrogen gas environment produced an epoxy coating with a chemical composition identical to that of the as-received DGEBA functional groups. In contrast, the chemical composition of the epoxy coating synthesised under the ambient air environment displayed different chemical functional groups, suggesting a different chemical composition. For the ambient sample group, an increase in MEA concentration results in an increase in coating thickness and dielectric constant. In contrast, a similar change in the MEA level results in a decrease in epoxy thickness and dielectric constant.

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1. Introduction

Epoxy resins are the most commonly used thermosetting polymers in both industrial and domestic sectors. Their applications span various fields, including paints, coatings, adhesives, industrial tooling, aerospace, electronic materials, and biomedical systems [1-6]. It has been reported that surface coatings consume about 50% of all epoxy resins produced [7,8]. Epoxy resins are synthesised using different chemical reactants, resulting in various types of epoxy resins. The most common type is diglycidyl ether of bisphenol-A, commonly known as DGEBA epoxy resin. To convert the liquid epoxy resin into a solid form with a three-dimensional crosslinked structure, a curing agent must be added. There are various types of curing agents, such as aliphatic polyamines.

Cationic epoxy resins have garnered significant attention from researchers due to their unique response to applied electric fields, offering promising applications in coating processes such as electrophoretic deposition (EPD) [1,8]. The synthesis of these resins involves precise manipulation of molecular structures to achieve the desired chemical characteristics for EPD and curing processes. EPD is preferred over other coating methods because of its ability to deposit a uniform thickness of polymer coating on targeted metallic surfaces, adaptability to new coating formulations, and suitability for different surface materials and shapes [8].

Recent studies have emphasised the importance of understanding the synthesis and electrophoretic deposition of cationic epoxy for EPD [5,9]. The studies also emphasised that the synthesis of the cationic epoxy needs to be performed in an inert nitrogen environment. However, there is no explanation of the effect of ambient air environment on the EPD and the dielectric properties of the epoxy coating. This comparative analysis aims to elucidate the impact of environmental factors on the chemical reactions and physical properties of the epoxy coatings, enabling better control and optimisation of the Electrophoretic Deposition process for producing high-quality coatings.

In this study, we delve into the effect of N-methylethanolamine (MEA) concentration and the synthesis environment on the chemical composition and dielectric properties of epoxy coatings. By investigating the effect of these two factors during the synthesis process and correlating them with changes in chemical functional groups and dielectric properties of the deposited epoxy coating, we aim to elucidate the underlying mechanisms governing these phenomena. Such insights are vital for optimising epoxy coating formulations tailored for enhanced electrical insulation performance in demanding industrial environments.

2. Methodology

A commercially-available diglycidyl ether bisphenol A (DGEBA) (product number: Auto-Fix 8800-A, Chemibond Enterprise) was used as the starting material for the cationisation reaction process. Chemibond Enterprise [10] states in the product information that 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 of 500–1,000 centipoise. The synthesis of modified DGEBA epoxy resin suspensions involved the chemical reaction process with six other chemicals (reagent grade), including N-methylethanolamine (MEA) (see Table 1). The formulations of chemical reactants and heating profile were adapted from the studies by Bosso *et al.*, [11] and Wismer *et al.*, [12]. Initially, the as-received DGEBA liquid (item 1) and items 2-4 were mechanically stirred in a jacketed glass reactor chamber and heated by the connected heating circulator for a duration of 2 hours and 30 minutes at a constant temperature of 130°C under two distinct conditions: (i) Ambient environment: the mixture was heated in the normal air atmospheric environment, and (ii) Inert environment: the mixture was heated in the presence of

a flowing nitrogen gas atmosphere (flow rate into the reaction chamber: 10 ml/min, purity: 99.9%). The ambient condition allows for the observation of oxidation by oxygen gas on the chemical reactants.

Conversely, the consistent flow of pure nitrogen gas into the reactant chamber pushed out the air and minimised the oxidation risk, reducing oxidation and other unwanted reactions. Initially immiscible, the mixture became homogeneous after stirring and heating. Subsequently, the mixture was cooled down to 70-92°C before a selected amount of MEA and items 6-7 were added gradually. The amount of MEA was varied from 0.5 to 1.5 ml to create three different modified epoxy solutions. MEA was introduced as a cationisation agent of the DGEBA liquid to produce cationic DGEBA suspension. Immediately after all items were added, the heating circulator was switched off, which started the natural cooling process (estimated to take 2-3 hours). The mechanical stirring and nitrogen gas flow (for inert environment synthesis) processes continued to operate until the modified epoxy suspension reached room temperature.

Table 1
Chemical reactants and amount

No	Chemical reactant	Quantity
1	Epoxy resin	140 ml
2	Dimethylether of diethylene glycol	20 ml
3	1- Octanol	41 ml
4	Stannous chloride	1.5 grammes
5	N-methylethanolamine (MEA)	0.5, 1.0, and 1.5 ml
6	Formic acid	0.1 ml
7	Deionized water	60 ml

Electrophoretic deposition (EPD) employs two galvanised iron plates (dimensions of 45 mm × 25 mm × 0.14 mm) as the anode and cathode, arranged in parallel orientation with a separation distance of 10 mm. Submerged to a depth of 30 mm in epoxy solution, both electrodes facilitate the deposition process. Three types of modified epoxy solutions from each of the ambient and inert environment synthesis processes were used separately as suspension for EPD of epoxy coating. Deposition is conducted under a direct current (d.c.) voltage of 60 V for a duration of 15 minutes using a DC power supply (model E3643A, Keysight). Subsequently, curing of the samples is achieved by placing them in a dry oven at 100°C for a duration of 24 hours. The weight of epoxy deposited on the galvanised iron substrate was measured using a digital weighing balance (resolution: 0.001 grams, Explorer). The deposition weight of the epoxy coating was obtained by subtracting the weight of the blank galvanised iron from the weight of the galvanised iron with the deposited epoxy.

The characterisations of the coatings and the as-received DGEBA and MEA were performed at room temperature and pressure. The surface morphologies of the cured epoxy coating samples were investigated using field-emission scanning electron microscopy (FESEM) and Energy Dispersive X-ray Spectroscopy (EDS) with a Hitachi High-Tech SU5000 instrument. To facilitate sample conductivity during FESEM observation, the coating samples were subjected to gold sputtering, where a thin gold (Au) layer was deposited onto the epoxy coating. Planar surface images of the epoxy-coated galvanised iron substrate were taken using a mobile phone with normal settings. Fourier Transform Infrared (FTIR) spectroscopy, performed with a Jasco FTIR-6100 instrument, was conducted on the as-received DGEBA liquid, as-received MEA liquid and on the epoxy coating after curing to compare the change in the functional groups on the DGEBA epoxy molecules. Spectra were collected in Attenuated Total Reflectance (ATR) mode with a resolution of 2 cm⁻¹. Electrochemical Impedance Spectroscopy (EIS), utilising a Gamry Instrument model Reference 600 potentiostat, was employed to analyse the dielectric properties of the cured epoxy coating at AC current frequencies from 1 to

100,000 Hz. The real and imaginary impedance data obtained from EIS were converted into dielectric constants using the method proposed by Joshi *et al.*, [13]. Before characterisation, the cured epoxy coating with the galvanised iron sheet substrate was cut with scissors into a 24 mm diameter, and sandwiched between two spring-loaded stainless-steel electrodes inserted into a customised EIS sample holder with an effective diameter of 18 mm. EIS data analysis settings were based on a previous study [14].

3. Results

3.1 Physical Appearance of Epoxy Coating on Galvanised Steel Plates

Figure 1 illustrates the physical appearance of each sample (*i.e.* epoxy coating on galvanised iron substrate) deposited by EPD using modified epoxy suspension synthesised with different MEA concentrations under both inert nitrogen and ambient air environments. The distinctiveness of the samples' appearance is readily apparent between the inert and ambient sample groups, attributable to chemical composition variation in modified epoxy caused by oxidation of the chemical reactants (*i.e.* one or more reactants as mentioned in Table 1) during synthesis in the ambient air environment. A brownish colour was visible on the epoxy coating of the ambient sample group, particularly for samples synthesised using higher MEA concentrations. It appears that oxidation is more pronounced when higher N-methylethanolamine (MEA) concentrations are used during the synthesis stage, implying that MEA is strongly affected by the presence of oxygen during the high-temperature chemical reaction process.

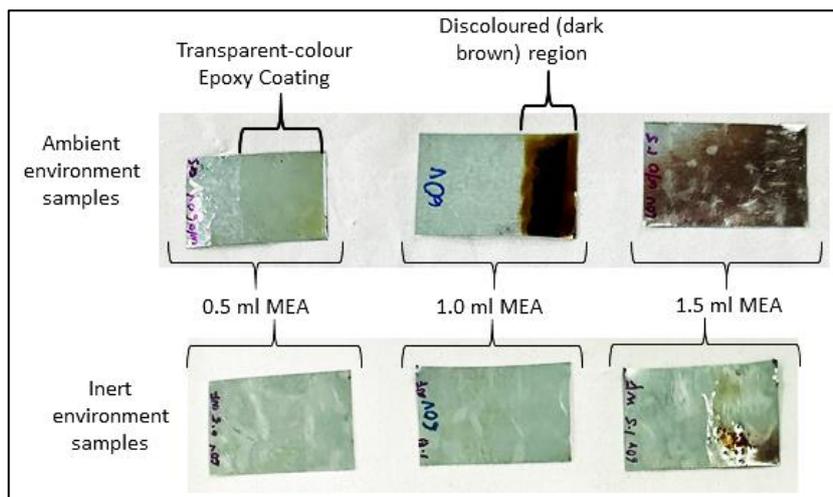


Fig. 1. Photograph image of planar surface of deposited epoxy coating on galvanised iron sheets obtained using EPD suspension synthesized in ambient air and inert nitrogen environments

Moreover, the discernible difference between the nitrogen and ambient environments serves as a poignant reminder of the significant role that environmental conditions play in shaping surface characteristics. Nitrogen environments act as a shield against chemical reactions, effectively minimising their occurrence and preserving the pristine state of the surfaces. In stark contrast, ambient conditions present a diverse array of gases and fluctuating moisture levels, creating a dynamic and often unpredictable setting. This environmental variability collectively influences the appearance and properties of the epoxy coating, contributing to the nuanced and multifaceted nature of epoxy coating in both ambient and inert synthesis environments.

3.2 Fourier-Transform Infrared Spectroscopy (FTIR) Spectra Data

The FTIR spectra depicted in Figure 2, encompassing spectra of the as-received DGEBA epoxy resin and as-received N-methylethanolamine (MEA) chemical reactants, serve as a pivotal tool for unravelling crucial insights into the chemical composition within the epoxy coatings. Peak labelled with '*' is the unique peaks of MEA where there is no overlapping with DGEBA peaks. Whereas, Table 2 lists the FTIR peaks of the two reactants and assigns most of them to the corresponding functional groups. Wavenumbers of the unique MEA peaks are highlighted in bold. This comprehensive FTIR analysis facilitates a comparative assessment of the functional groups inherent in each of these two main chemical reactants, thereby shedding light on their respective roles and contributions to the overall coating chemical composition [15]. By scrutinising the FTIR spectra's peaks of DGEBA and MEA from the epoxy coating's spectra, unique FTIR peak characteristics associated with specific functional groups from DGEBA and MEA reactants are discerned, enabling an understanding of the chemical composition of the epoxy coating materials.

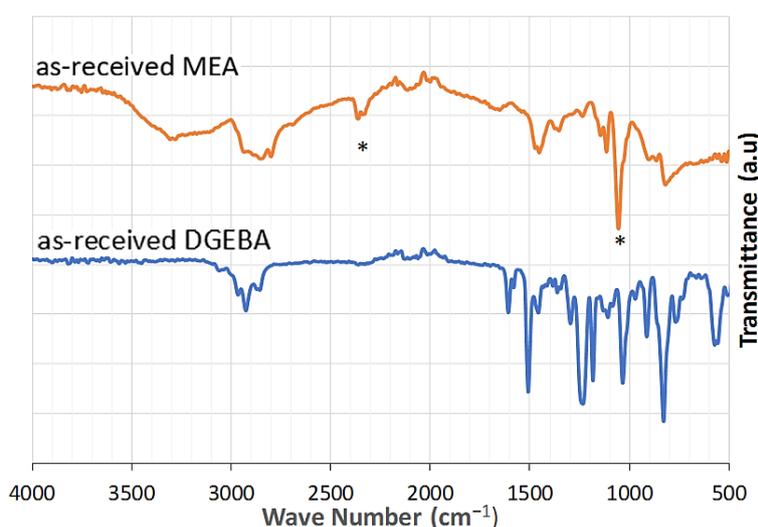


Fig. 2. FTIR spectra of as-received DGEBA epoxy resin and as-received MEA

Table 2

Assignments of diglycidyl ether of bisphenol A (DGEBA) and N-methylethanolamine (MEA) FTIR peaks as indicated by their FTIR spectra in Figure 2

Wavenumber ν (cm ⁻¹)		Assignment of functional group [15-18]
DGEBA	MEA	
3450		Hydroxyl group
2964		Methyl groups
2917	2942-2836	Methyl groups
2852		Methylene groups
	2796	unknown
	2358	unknown
	2327	unknown
1602		Phenyl groups
1581		Phenyl groups
1508		Phenyl groups
1455	1458-1450	Methylene groups
1367-1342	1360-1349	Methyl groups
1288		unknown
1230		Ether groups

	1225	unknown
1180		unknown
	1125	Secondary alcohol
	1113	Secondary amine
1107		Phenyl ether group
	1050	Primary alcohol
1030		Phenyl groups
910		Epoxide group
825		Phenyl groups
	815	unknown
766		unknown
555		unknown

The MEA spectrum exhibits a prominent peak corresponding to C-O stretching near the primary alcohol group (-OH) at 1050 cm^{-1} [16-18]. The secondary amine compound also shows a second unique but unidentified peak within the spectral range of 2327 cm^{-1} to 2796 cm^{-1} . Conversely, the DGEBA epoxy resin spectrum typically showcases peaks within the range of 825 cm^{-1} to 1602 cm^{-1} , indicative of aromatic (phenyl) and epoxy groups (C-O-C) functionalities. Additionally, the methyl and hydroxyl groups of the DGEBA compound occupy a significantly larger wavenumber region between 2852 cm^{-1} to 3450 cm^{-1} , as elucidated by prior research [15].

The FTIR analysis presented in Figure 3 shows the effects of varying MEA concentrations from 0.5 to 1.5 ml under ambient atmospheric conditions on the chemical composition of the synthesised epoxy coating. FTIR Spectra of as-received DGEBA is provided as comparison. Dashed line indicates the peak position of epoxide group of DGEBA epoxy. There are distinct differences in the spectrum properties in terms of the transmittance peaks and patterns compared to the as-received epoxy from the manufacturer. The transmittance peak intensity of the epoxy compound diminishes after the synthesis process, with the peak intensity decreasing as the MEA concentration decreases. Furthermore, the epoxy coating produced using lower MEA concentrations (i.e., 0.5- and 1.0-ml MEA) shows distortion in the transmittance patterns, implying the possible formation of new chemical compounds. This is accompanied by the disappearance of the important epoxide group peak at 910 cm^{-1} .

Analysing Figure 4, which illustrates less distinct transmittance spectra obtained from epoxy coating synthesised using different MEA concentrations under a nitrogen atmosphere. FTIR Spectra of as-received DGEBA is provided as comparison. Dashed line indicates the peak position of epoxide group of DGEBA epoxy, whereas shadow region indicates the peak position of one of the MEA unique peaks. All the transmittance peaks observed in as-received DGEBA liquid were also observed in the deposited epoxy coating samples. However, the peak intensity diminishes, particularly the epoxide group peak at 910 cm^{-1} for the MEA concentration of 0.5 ml. Interestingly, new transmittance peaks appear in the spectral range of 2327 cm^{-1} to 2796 cm^{-1} , suggesting possible chemical bonding of new compounds onto the DGEBA polymer, which may be related to MEA addition. These peaks are located exactly at the peak position of the unique MEA peak. However, further investigation is required to verify and determine the nature of the chemical bonding. We argue that the MEA peak observed in the coating samples is unlikely to originate from unreacted MEA molecules because of the following reasons:

- i. The unique MEA peak at 1050 cm^{-1} does not appear in the samples' spectrum.
- ii. The MEA-to-DGEBA epoxy concentration added during the synthesis process is significantly small, which is 0.5-1.5 ml to 140 ml.

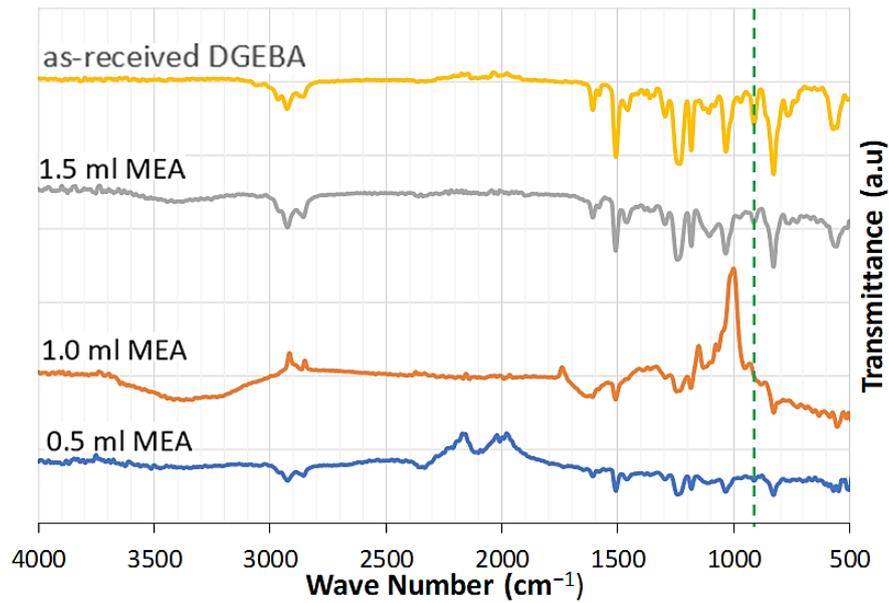


Fig. 3. FTIR spectra of epoxy coating deposited using modified DGEBA prepared in ambient air environment at different MEA concentrations

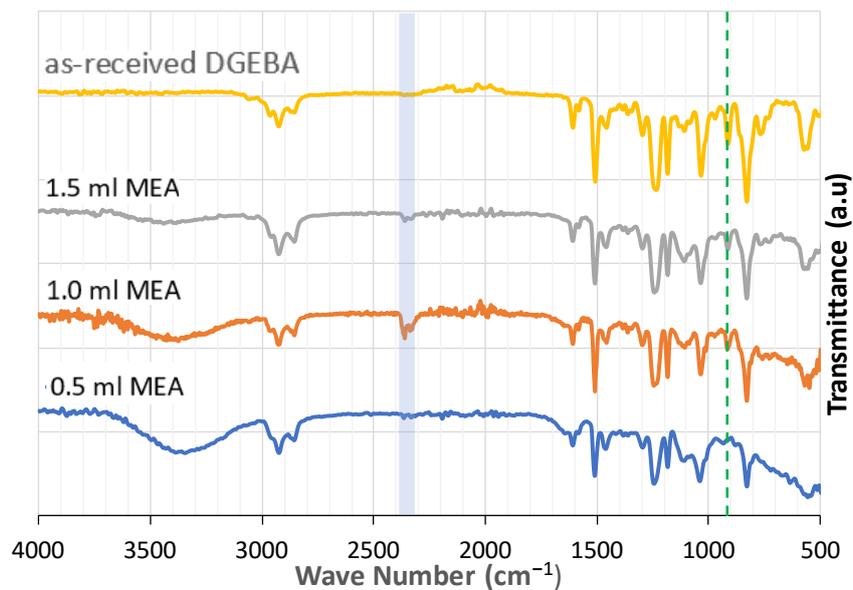


Fig. 4. FTIR spectra of epoxy coating using solution produced in inert nitrogen gas environment

3.3 Electrochemical Impedance Spectroscopy (EIS)

Figure 5 illustrates the epoxy coating's dielectric constant across different synthesis settings, encompassing both ambient and inert synthesis environments, as well as different MEA concentrations. While, the insert of Figure 5(a) shows the enlarged view of the respective data. All samples exhibit an exponential decrease in their dielectric constant as the logarithmic angular frequency of the applied voltage increases, consistent with the study by Joshi *et al.*, [13]. This decline can be attributed to the limited ability of the electric dipoles to respond to changes in the applied alternating current (AC) electric field. It becomes evident that the dielectric constant drops drastically in the low-frequency region between 1 and 50 Hz for both ambient and inert environment samples. However, as the MEA concentration increases, there is a noticeable decline in the dielectric constant

for inert environment samples throughout the entire frequency range. At 50 Hz, the dielectric constant decreases from 11.39 to 2.74 and 2.65 as MEA concentration increases from 0.5 to 1.5 ml. In contrast, for the ambient environment samples, there is a slight increase in the dielectric constant across the entire frequency range as MEA concentration increases from 0.5 to 1.5 ml. For example, the dielectric constant at 50 Hz increases from 3.6 to 3.9 and 4.0 for the respective MEA concentrations. Although the dielectric constant of ambient environment samples records higher values, it remains lower than the previously reported dielectric constant values for DGEBA-type epoxy, which typically range from 4.0 to 4.8 [19,20].

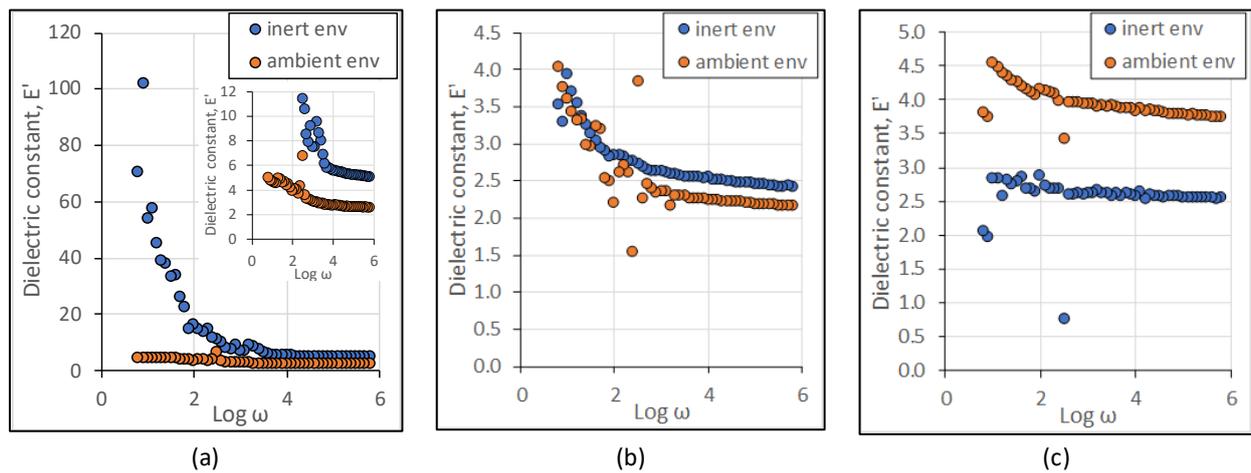


Fig. 5. Graph dielectric constant, E' vs. $\log \omega$ of epoxy coating deposited using MEA concentration (a) 0.5 mL (b) 1.0 mL (c) 1.5 mL in the ambient and inert synthesis environments

3.4 Field Emission Scanning Electron Microscopy (FE-SEM)

Figures 6 and 7 portrays the planar surface microstructure of the cured epoxy coating deposited using modified epoxy synthesised in both ambient air and nitrogen environments at different MEA concentrations. The FE-SEM examination reveals agglomerates deposited as part of the epoxy coating. Apart from the agglomerates, the coating surface is smooth.

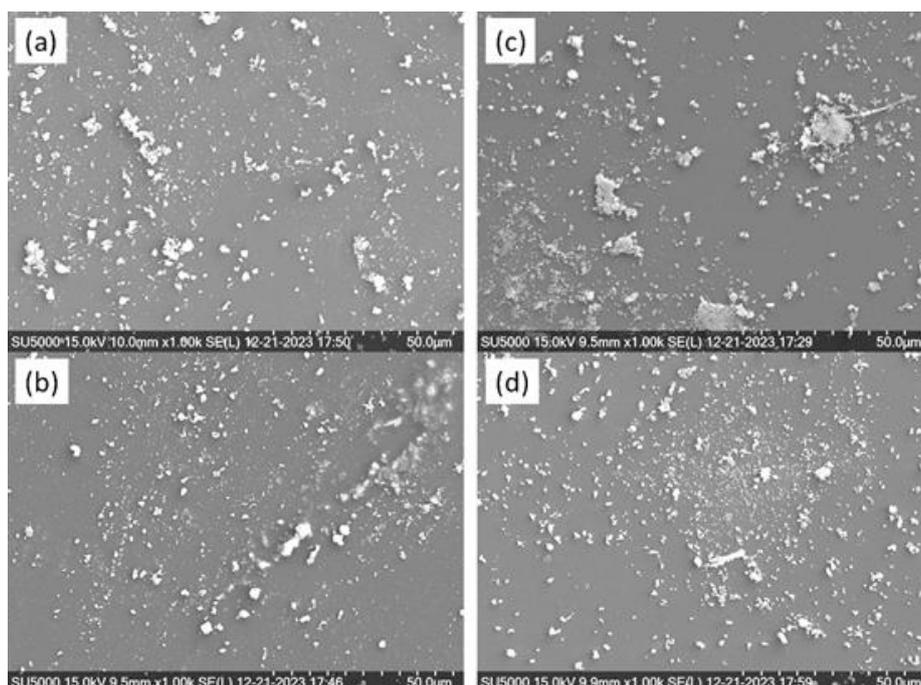


Fig. 8. FESEM micrographs of planar surface microstructure of cured epoxy coating deposited using EPD suspension synthesised with different environment and MEA concentrations (a) Ambient air (0.5 mL) (b) Ambient air (1.0 mL) (c) Inert nitrogen (1.0 mL) (d) Inert nitrogen (1.5 mL)

3.5 Calculated Thickness of Deposited Epoxy Coating

Tables 3 and 4 present the calculated thickness and EDS data of the planar surface, respectively, of the epoxy coating deposited using epoxy suspension synthesised with different MEA concentrations in ambient and nitrogen environments. The thickness of the epoxy coating deposited on the galvanised iron substrate was calculated using the method published in our previous studies [21]. While the ambient sample group displays an increase in coating thickness with increasing MEA concentrations, the inert sample group shows a decrease in coating thickness with the rise in MEA concentrations.

Table 3

Calculated thickness from the weight of epoxy coating deposited using epoxy suspension synthesised with different MEA concentrations in ambient and nitrogen environments

Environment	MEA concentration (mL)	Weight (g)	Thickness (μm)
Ambient	0.5	0.094	0.635
	1.0	0.095	0.641
	1.5	0.122	0.844
Inert	0.5	0.069	0.466
	1.0	0.077	0.520
	1.5	0.034	0.230

Table 4

EDS data of the planar surface of epoxy coating deposited using epoxy suspension synthesised by different MEA concentration and using EPD coating obtained in ambient and nitrogen environment

No.	Element (at%)			
	Carbon	Nitrogen	Oxygen	Silicon
a	69.7	2.1	27.6	0.4
b	78.0	2.8	17.1	1.5
c	67.2	4.0	26.3	2.2
d	90.7	0.0	8.6	0.0

4. Conclusions

The study demonstrates that varying concentrations of N-methylethanolamine (MEA) and different synthesis environments of cationic epoxy solutions significantly affect the chemical composition and dielectric properties of deposited epoxy coatings. Epoxy solutions synthesised under an inert nitrogen gas environment produced epoxy coatings with a chemical composition identical to that of the as-received DGEBA functional groups. In contrast, the chemical composition of epoxy coatings synthesised under ambient air environments displayed different chemical functional groups, suggesting a varied chemical composition.

Electrophoretic deposition (EPD) of epoxy coatings on galvanised iron substrates was successfully performed, and weight gain of the respective epoxy coatings was measured. The effect of MEA concentration increases on epoxy thickness and dielectric constant differed depending on whether the epoxy solutions for EPD were synthesised in the two environments. In the ambient sample group, an increase in MEA concentration resulted in increased coating thickness and dielectric constant. In contrast, similar changes in MEA concentration led to a decrease in epoxy thickness and dielectric constant in the inert sample group. The distinct behaviours of the two sample groups may be attributed to changes in the chemical composition of the epoxy solutions synthesised for use in the EPD process to produce epoxy coatings with specific dielectric properties and coating thicknesses.

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