



BIO-SILICA REINFORCED IMIDAZOLE CORE DIAMINE/EPOXY BASED COMPOSITES COATINGS FOR CORROSION RESISTANT APPLICATION

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ABSTRACT

Imidazole core diamine (IDA) was synthesized and used as a hardener for epoxy resin. The epoxy resin was reinforced with varying weight percentages of 3-glycidoxypropyltrimethoxysilane (GPTMS) functionalized rice husk ash bio-silica to obtain respective composites. Their composite morphology was studied using SEM analysis. The cure behaviour of imidazole core diamine hardener and DGEBA epoxy resin was studied. The thermal stability of the corresponding epoxy matrix and bio-silica reinforced epoxy composites were studied by thermogravimetric analysis (TGA). The values of limiting oxygen index calculated from TGA data infer that all the samples of epoxy matrix and composites possess good flame retardant behaviour. The glass transition temperature of composites was studied using DSC analysis. In order to ascertain their hydrophobic behaviour, the water-contact angle measurement studies were carried out. IDA/DGEBA matrix and functionalised bio-silica reinforced IDA/DGEBA composites were coated on the mild steel specimens and their corrosion resisting behaviour was studied by electrochemical impedance spectroscopy and potentiodynamic polarisation. Data resulted from corrosion studies (Nyquist plot and Tafel plot) inferred that among the coated specimens studied, the specimen coated with 7 wt% bio-silica reinforced IDA/DGEBA matrix exhibits the highest corrosion resisting efficiency due to its water repellent character and morphology.

Keywords: Imidazole core diamine, Epoxy resin, Bio-silica, Contact angle, Corrosion resistance.

1. INTRODUCTION

Mild steel (MS) is an important material for domestic and industrial applications and is widely used in construction, oil, gas, chemical processing, water and wastewater systems. However, mild steel has tendency to undergo structural deterioration due to corrosion. Hence, it needs surface protection to avoid loss of strength and to improve its lifespan and cost effective. In this context, the application of coatings on the MS surface is a cost effective way to inhibit the process of corrosion. Many approaches have been employed to reduce and prevent the corrosion of metals and their alloys, such as organic and inorganic coatings [1-5]. Although coatings are commonly used for corrosion protection, they may cause some environmental problems as they could contain some of the hazardous elements, e.g. cadmium, arsenic, and chromium. Generally, organic coatings have been employed to protect metal substrates from the adverse corrosion environments during their service life. The hydrophobicity, water uptake, and mechanical

properties of the organic coatings influence their surface protection ability by isolating the metal surface from the corrosive electrolyte [6-8]. Several organic polymers can be utilized for corrosion resistance application, such as epoxy, polyacrylates, polypyrrole, polybenzoxazine, epoxy matrix, etc [9-17]. Many researchers in the recent past are directed their attention towards epoxy based coating materials, because of their excellent properties suitable for different industrial applications. Epoxy resins are high-performance matrices widely used in the aerospace, automotive, corrosion resistance coating, electronic industries and other industries. The epoxy resins exist in the form of liquids or solids according to their starting materials. The ring-opening polymerization of epoxy resins takes place by amines, anhydrides, hydroxyl hardeners with release of no-by product without any shrinkage. The epoxy matrices possess an excellent mechanical and electrical, however their thermal behaviour and hydrophobic nature limit their applications to certain field. In this context, researchers

developing different epoxy resin and hardeners. Recently researchers reported the epoxy composites for superior corrosion resistant coating for steel plates. Many researchers are also working the utilization of epoxy as an effective coating material for mild steel specimen in order to prevent corrosion by introducing the different hardeners and fillers [18]. In this context, the heterocyclic compound based amine hardeners possess better film forming ability with enhanced hydrophobic nature and corrosion inhibition nature of the coating material. Many articles are reported with corrosion inhibitor obtained from different compounds. In this context, more attention is devoted to imidazole based inhibitors for anticorrosion behaviour [19]. Berdimurodov et al. [20] developed and reports new anti-corrosion inhibitor (3ar, 6ar)-3a,6a-di-ptolyltetrahydroimidazo[4,5-d]imidazole-2,5(1 h,3h)-dithione for carbon steel and Mendes et al. [21] reports on the nature of inhibition performance of imidazole on iron surface. Singh et al.(2019)[22] reports on corrosion inhibition performance of imidazolidine derivatives for J55 pipeline steel and novel imidazole derivatives as corrosion inhibitors for J55 steel in sweet corrosive environment. The prime objective of the present work is to develop bio-silica reinforced epoxy composite coatings to utilize them for protection of mild steel surfaces from corrosion. In this context, imidazole core diamine was synthesized and reinforced with varying weight percentages of functionalized bio-silica to obtain composite coatings. These coatings were coated on mild steel and their corrosion protection efficiency was studied by electrochemical method. Data obtained from different studies are discussed and reported.

2. MATERIAL AND METHODS

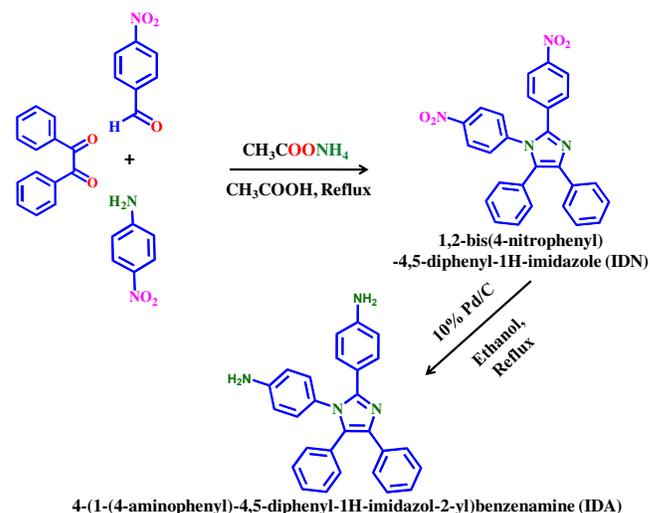
2.1. Material

Benzil, 4-nitrobenzaldehyde, 4-nitroaniline, ammonium acetate, hydrazine monohydrate, 10% palladium on activated carbon, were purchased from Sigma Aldrich, Bangalore, India. Hexane, ethyl acetate and acetic acid were received from qualigens Mumbai, India and DGEBA were purchased from Roto polymers chennai India.

2.2. Synthesis of 4, 4' (4, 5-diphenyl-1H-imidazol-1, 2-diyl) dianiline (IDA)

The synthesis of 4, 4' (4, 5-diphenyl-1H-imidazol-1, 2-diyl) dianiline (imidazole core diamine-IDA) involves two steps as shown in Scheme 1. Initially nitro substituted imidazole compound was synthesised in a

250 mL, two-necked, round-bottomed flask equipped with a reflux condenser, benzil (0.05 mol), 4-nitrobenzaldehyde (0.05 mol), 4-nitroaniline (0.05 mol) and ammonium acetate (0.01 mol) in 25mL of acetic acid were mixed and heated at 120°C. The progress of the reaction was monitored by thin-layer chromatography (TLC) in 7:3 ratio of hexane and ethyl acetate as eluting solvent. After completion of the reaction, the reaction mixture was cooled to room temperature and continued the stirring for overnight. The product was then filtered, washed with water and recrystallized from ethanol and collected the 1, 2-bis (4-nitrophenyl)-4, 5-diphenyl-1H-imidazole yellow solid product. Further, nitro compound reduced to amine compound in a 250 mL two-necked flask equipped with a dropping funnel and a reflux condenser, (0.05 mol) of nitro compound and 250 mg of 10% palladium on activated carbon (10% Pd/C,) were dispersed in 100 mL of ethanol. The suspension was heated to reflux, and 10 mL of hydrazine monohydrate was added slowly to the mixture. After 5 h of reflux, THF was added to the mixture and filtered hot to remove Pd/C, and the filtrate was cooled to obtain orange precipitate. The product was collected by filtration, recrystallized with ethanol and dried in vacuum at 80°C. The yielded amino compound was used as a hardener for epoxy resin.



Scheme 1: Synthesis of 4, 4' (4, 5-diphenyl-1H-imidazol-1, 2-diyl) dianiline (IDA)

2.3. Preparation of silane functionalization on bio-silica

The lignin from rice husk was first removed by the acid treatment, which is one of the most useful routes to

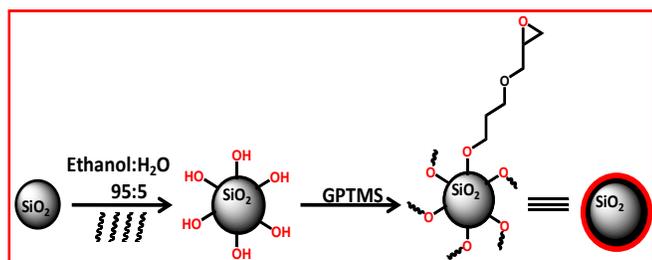
remove the wax and oils covering the wall of the surface of natural fibers (Scheme 2). Dried rice husk obtained was bleached with conc. HCl to remove the unwanted contaminants and subsequently washed with water continuously till the pH became neutral.

The product was then dried at 60°C for 4 h and calcinated at 600°C for 5 h in a muffle furnace, to obtain rice husk ash in which silica (SiO_2) is the major component (85 %) [23-25].



Scheme 2: Preparation of rice husk ash bio-silica

The prepared rice husk ash (biosilica) was then functionalized with (3-glycidyloxypropyl) trimethoxysilane (GPTMS) in order to reinforce with epoxy matrix. About 4 ml of GPTMS was stirred with 95% absolute ethanol and 5% deionized water and the resulting solution was sonicated for 15 min (Scheme 3). The pH of the solution was initially adjusted to 4.5 using acetic acid and subsequently sonicated for 1 h in order to ensure the complete hydrolysis of silane. Then 10 g of bio-silica was added, and the resulting mixture was sonicated for 2 h and then refluxed for 24 h at 80°C and centrifuged with the addition of water followed by ethanol and hexane and filtered. The functionalized bio-silica obtained was further dried in hot air oven at 100°C in order to remove the moisture.

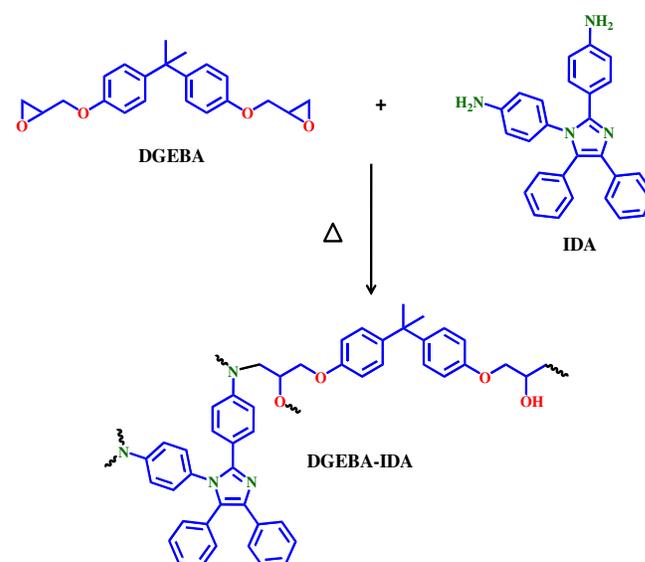


Scheme 3: Preparation of GPTMS functionalization on bio-silica

2.4. Preparation of neat DGEBA/IDA matrices

Different weight percentages of silane functionalized bio-silica was added into the DGEBA/IDA and subjected to agitation. The resulted homogenous mixture was then transferred into respective silane coated glass plates and the temperature was maintained

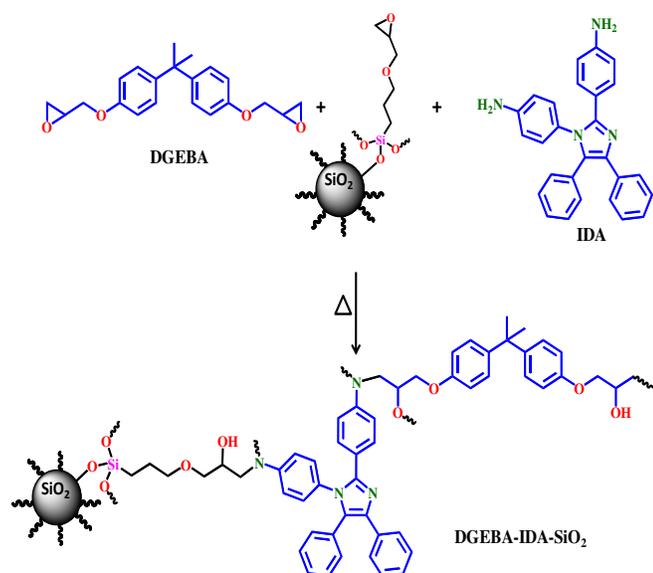
at 120°C for about 3 h. The obtained light brown coloured thin composite films were characterized.



Scheme 4: Synthesis of DGEBA/IDA polymer matrix

2.5. Preparation of bio-silica reinforced DGEBA /IDA composites coated MS plate

1, 3, 5, 7 and 10wt% of silane functionalized bio-silica incorporated DGEBA+IDA was coated on MS plate and subjected to curing process. Resulted homogenous blend was then transferred into respective silane coated glass plates and left to evaporate slowly at 100°C for 3h. Subsequently post cured at 120°C for 3h each until the formation of the light brown coloured thin composites film (Scheme 3).



Scheme 5: Preparation of bio-silica reinforced DGEBA/IDA composites

2.6. Characterization

FTIR spectra measurements were carried out with Agilent Cary 630 FTIR Spectrometer. NMR spectra were obtained with Bruker (400 MHz) using dimethylsulfoxide (d₆-DMSO) as a solvent and tetramethylsilane (TMS) as an internal standard. DSC measurements were recorded using NETZSCH STA 449F3 under N₂ purge (60 mL min⁻¹) at scanning rate of 10°C min⁻¹. Thermogravimetric analysis (TGA) was carried out from room temperature to 850°C using NETZSCH STA 449F3 with 5 mg of sample under N₂ flow (60 mL min⁻¹) at heating rate of 20°C min⁻¹. The morphology of the blended matrices and composites were analyzed from an FEI QUANTA 200F high-resolution scanning electron microscope (HRSEM). Contact angle measurements were obtained using a Kwoya goniometer with 5 μl of water as probe liquid. The epoxy matrices coated mild steel plates were tested for their corrosion protection behavior on mild steel in 3.5% sodium chloride solution. The corrosion experiments on mild steel specimens were carried out using open-circuit potential (OCP), electrochemical impedance spectroscopy (EIS) and potentiodynamic polarisation.

3. RESULTS AND DISCUSSION

3.1. IR spectral analysis

FTIR spectra of bio-SiO₂ reinforced epoxy composites are presented in fig. 1. The absence of peak at 965 cm⁻¹ in bio-silica reinforced DGEBA+IDA composites

confirms the formation of composites. The absorption peak appeared at 837 cm⁻¹ indicates the presence of aromatic substitutions rings. The appearance of peak at 1605 cm⁻¹ corresponds to the -N=C- group, which confirms that the imidazole core. The peak appeared at 3382 cm⁻¹ corresponds to the -OH stretching in the composites. Further, surface bio-silica possesses an active epoxy functional group that takes part in the ring-opening reaction with both DGEBA and IDA. This was confirmed from the existence of peak at 1115 cm⁻¹, that is, Si-O-Si linkage of silica. This confirms that the formation of silica network between epoxy resin and caprolactam is through the oxirane ring present on the surface of silica (Fig. 1).

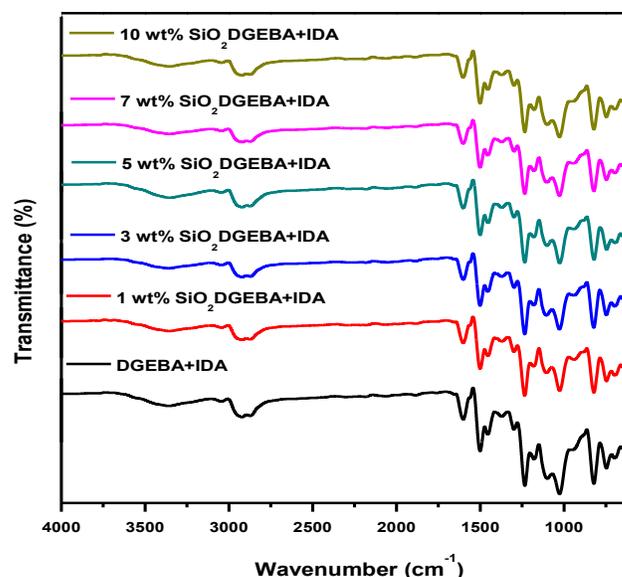


Fig. 1: IR spectra of neat and bio-silica reinforced DGEBA+IDA matrices

3.2. Thermal behavior

TGA thermograms for neat and bio-silica reinforced DGEBA+IDA matrices are shown in fig. 2. The thermal stability of neat and 1, 3, 5, 7 and 10 wt% bio-silica reinforced DGEBA+IDA were studied using the TGA at the heating rate of 20°C min⁻¹ under nitrogen atmosphere. The data obtained are presented in table 1 and fig. 2. From the TGA data, it is inferred that an increasing weight percentage of bio-silica reinforced DGEBA+IDA epoxy systems contributes higher thermal stability to the resulted composites via the strong covalent bonding that occurred between the epoxy and bio-silica reinforcements. The maximum degradation of neat and 1, 3, 5, 7 and 10 wt % bio-silica reinforced DGEBA+IDA epoxy are noticed at 405,

413, 415, 422, 426 and 428°C, respectively. The char yield of neat and 1, 3, 5, 7 and 10 wt% bio-silica

reinforced DGEBA+IDA epoxy matrices obtained at 15, 17, 19, 22 and 26% respectively.

Table 1: Thermal stability of neat and bio-silica reinforced DGEBA+IDA composites

| Sample name | 10 % weight loss (°C) | Maximum degradation temperature (°C) | Char yield percentage at 850°C | LOI |
|-------------------------------------|-----------------------|--------------------------------------|--------------------------------|------|
| Neat DGEBA+IDA | 325 | 405 | 11 | 21.9 |
| 1 Wt% SiO ₂ + DGEBA+IDA | 342 | 413 | 15 | 23.5 |
| 3 Wt% SiO ₂ + DGEBA+IDA | 349 | 415 | 17 | 24.3 |
| 5 Wt% SiO ₂ + DGEBA+IDA | 351 | 422 | 19 | 25.1 |
| 7 Wt% SiO ₂ + DGEBA+IDA | 352 | 426 | 22 | 26.3 |
| 10 Wt% SiO ₂ + DGEBA+IDA | 355 | 429 | 24 | 27.1 |

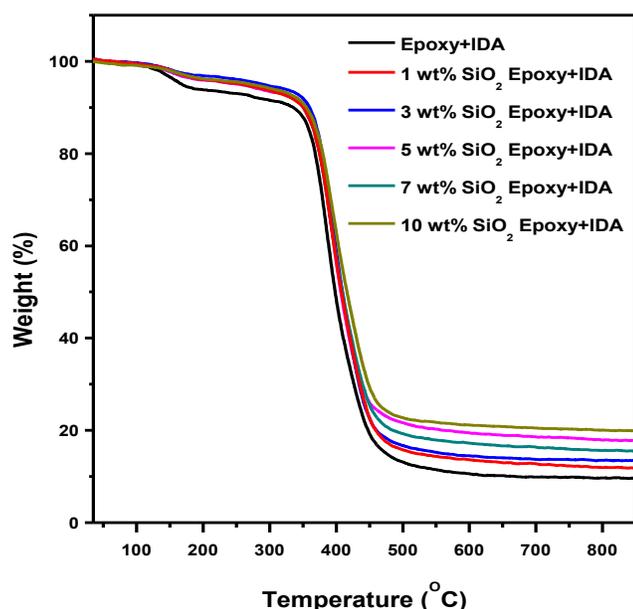


Fig. 2: TGA thermogram of neat and bio-silica reinforced DGEBA+IDA matrices

3.3. Flame Retardant behavior

The flame retardant behaviour of the polymer materials can be predicted from the value of Limiting Oxygen Index (LOI) calculated using char yield obtained from thermo-gravimetric analysis. The value of LOI is calculated using the Van Krevelen and Hoftzyer relation. $LOI = 17.5 + 0.4 CR$. The percentage char yield (CR) of the sample was obtained from TGA analysis at 850°C (Table 1). It is experimentally proven that the materials having higher LOI values are flame retardant in nature. Generally, polymers with LOI values greater than 26 are ranked as excellent flame retardant material.

The LOI values of neat and 1, 3, 5, 7 and 10 wt % bio-silica reinforced DGEBA+IDA composites are 21.9, 23.5, 24.3, 25.1, 26.3 and 27.1, respectively. It was

also noticed that the value of LOI increases with increase in silica content. The 10 wt% bio-silica reinforced composites shows the LOI value of 27.1, which is comparatively higher than that of neat matrix and other hybrid composites.

3.4. Microstructural behavior

In order to ascertain the microstructural behavior of the neat and bio-silica reinforced DGEBA-IDA composites, the scanning electron microscopic (SEM) as well as transmission electron microscopic (TEM) analysis were performed. Scanning electron microscopy (SEM) images of both neat and bio-silica incorporated DGEBA-IDA composites are presented in fig. 3. Neat matrix, 1, 3, 5, 7 and 10 wt% bio-silica reinforced DGEBA-IDA composite materials were taken for SEM analysis. It was observed from the images that the bio-silica reinforced DGEBA-IDA matrices exhibit a homogenous morphology without any voids in the matrix systems and thus possesses improved surface properties. The bio-silica reinforcement provides an effective formation of covalent bonding as well as inter cross linking network with epoxy matrix systems, which in turn develops the smooth surfaces and enhanced hydrophobic behaviour.

Furthermore, in order to ascertain the distribution of bio-silica in the DGEBA-IDA matrix, the HR TEM analysis was carried out and the images obtained are presented in Fig. 4. For TEM analysis 1 wt %, 5 wt % and 10 wt % of bio-silica reinforced DGEBA-IDA were taken as representative samples. The TEM images of 1 wt % (Fig. 4a), 5 wt % (Fig. 4b) and 10 wt % (Fig. 4c) bio-silica reinforced nanocomposites show uniform dispersion of silica in the DGEBA-IDA matrices. The distributions of bio-silica (F-SiO₂) are observed to be homogenous even at higher weight percentages. Further, it is interesting to note that the 10 wt % bio-

silica/DGEBA-IDA exhibit microspore morphology. This phenomenon influences and contributes to the elevation of high thermal stability and enhanced water contact angle properties achieved with homogeneity of

bio-silica. Among the hybrid composites studied, the 10 wt % DGEBA-IDA nanocomposites can be considered as better material for high performance micro-electronics applications.

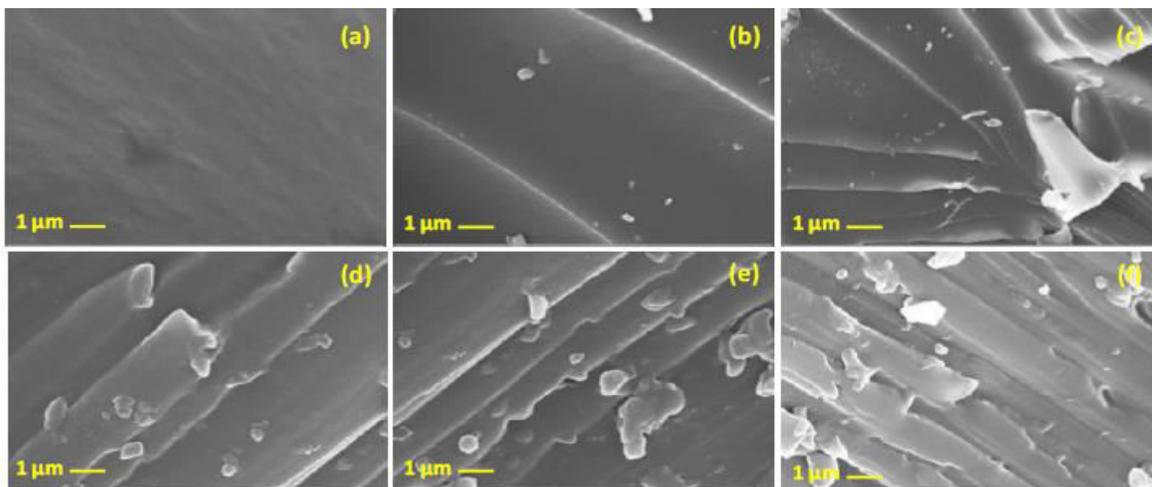


Fig. 3: SEM image of neat and bio-silica reinforced DGEBA-IDA matrices

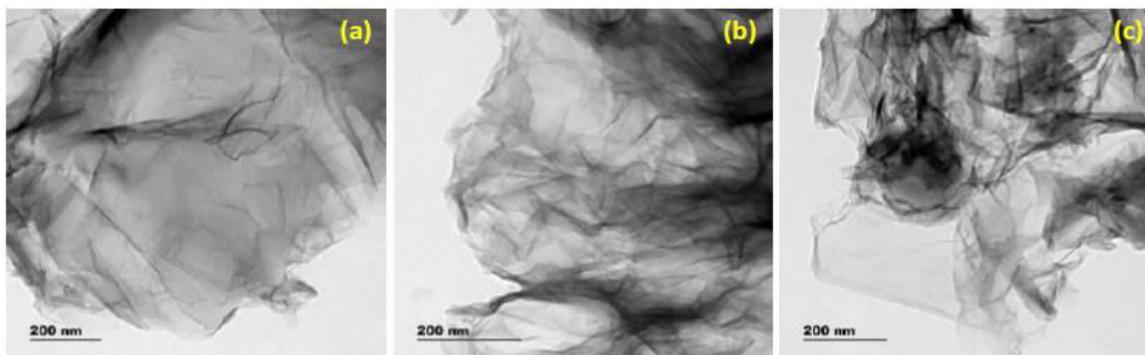


Fig. 4: TEM image of 1, 5 and 10 wt% bio-silica reinforced DGEBA-IDA matrices

3.5. Water contact angle studies

It is essential that the material should not be affected, when exposed to moisture environment. It is reported that when the MS are left unpowered under humid environments, the dust and moisture from the air deposited on the surfaces and this causes malfunctioning of the electronic devices by short-circuiting. Hence, the materials considered for electronic insulation applications should have inherent water repellency with lower surface free energy. In this context, the water contact angle properties of the developed materials were studied.

The contact angle images obtained for neat, 1, 3, 5, 7 and 10 wt % F-SiO₂ reinforced DGEBA-IDA composites are given in table 1. It can be observed that the reinforced composites possess the higher values of

contact angle when compared to that of neat DGEBA-IDA matrix. The value of contact angle of neat DGEBA-IDA matrix is 86°. Similarly, the values of water contact angle obtained for 1, 3, 5, 7 and 10 wt% of bio-silica reinforced DGEBA-IDA composites are 89°, 92°, 95°, 97° and 98° respectively. The values of contact angle tend to increase with increasing the concentration of F-SiO₂ in the composites. Thus, the 10 wt% F-SiO₂ reinforced bio-silica reinforced epoxy matrix show the highest values of contact angle of about 98°, respectively. In addition, the presence of intramolecular hydrogen bonding exist in the epoxy system also contributes to an enhanced values of water contact angle due to decrease in the surface free energy. It is further reiterated that the bio-silica reinforced epoxy composites with combination of higher values of water

contact angle can be considered for high performance corrosion resistant applications under humid environments.

3.6. Corrosion Studies

3.6.1. Preparation of the coatings

Sufficient numbers of uniform size mild steel plates having the size 2 cm x 1 cm were purchased from the local market. The mild steel plates were first thoroughly polished using emery papers. Then these plates were placed on the horizontal table. The required of coating was dropped on the mild steel plate and it was dried in the room temperature for 5h then it was cured in the air oven at 120°C for 3 h.

3.6.2. Corrosion studies using EIS measurements

Electrochemical impedance spectroscopy was used to find out the corrosion resistant property of the bio-silica reinforced DGEBA-IDA composites coating on the mild steel. An impedance analysis was performed using mild steel plate having 2 cm² area. The specimens after coating were immersed in 3.5% NaCl solution for different time periods.

EIS was used to evaluate the barrier properties of the bio-silica reinforced DGEBA-IDA composites films and the effect of silica reinforcement on the corrosion resistance. Nyquist plots derived from the EIS measurements for the coated and uncoated mild steel specimens in 3.5% NaCl solution are shown in fig. 5.

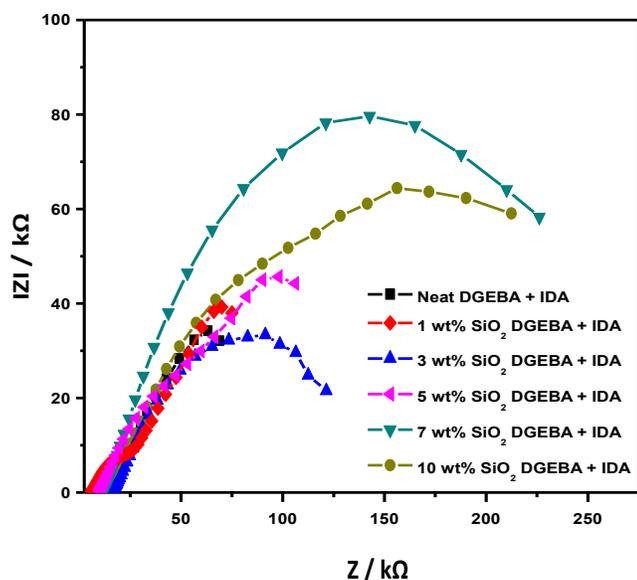


Fig. 5: EIS response of bare MS and bio-silica reinforced DGEBA-IDA composites coated specimen in 3.5% NaCl solution

Nyquist plots have only one capacitive semicircle for all the specimens represent the presence of only one time constant [26]. Hence, fitting of all EIS data is done using simple equivalent circuit model (Fig. 6).

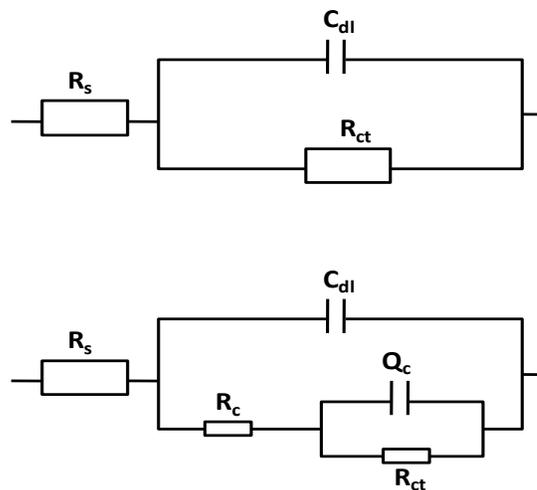


Fig. 6: The equivalent circuit used for impedance analysis

The equivalent circuit was used to evaluate the corrosion resistance data, where R_s is the solution resistance, R_{ct} is the charge transfer resistance and Q_c is the double layer capacitance.

Superimposition of the experimental data has been done using this equivalent circuit and corrosion parameters were obtained. R_s is the resistance of the solution between the working electrode and the counter electrode. R_s values are not only depending on the ionic conductivity of the solution but also depending on the geometrical area of the electrode. R_s values are insignificant data while studying the corrosion property of the film because it does not yield any information about the coating. R_{ct} values are used to measure the resistance of the electron transfer across the metal-solution interface, which is inversely proportional to the corrosion rate of the metal. The calculated values of the corrosion parameters from EIS measurements are given in table 2. R_{ct} values of the bio-silica reinforced DGEBA-IDA composites coated specimens are higher than that of the bare MS. In order to improve the corrosion resistance behavior of DGEBA-IDA matrix, the varying weight percentage of bio-silica were reinforced in the bio-silica reinforced DGEBA-IDA composites. When the content of bio-silica are increased from 1wt% to 10 wt%, into the bio-silica reinforced DGEBA-IDA composites, the increase in R_{ct}

and decrease in double layer capacitance values were observed. It infers an improvement of corrosion resistance behavior which arises from the reduction of pores/cavities present in the polymer coating which could be occupied by the bio-silica [15, 27-29]. The

roughness' factor values (n) are continuously decreasing while increasing weight percentage of bio-silica into the polymer matrix which also corroborates the reduction of pores/cavities on the mild steel surface.

Table 2: Corrosion parameters of the coated and bare mild steel specimens in 3.5 % NaCl solution calculated from potentiodynamic polarisation studies

| Samples | R _s (Ω cm ²) | CPE _c | | R _c (kΩ cm ²) | CPE _{dl} | | R _{ct} / kΩ cm ² |
|-----------------------------------|--|-------------------------|-------|---|--------------------------|-------|---|
| | | (F.s ⁿ⁻¹) | n | | (F.s ⁿ⁻¹) | n | |
| Bare MS | 7.20 | 09.11 x e ⁻⁶ | 0.878 | 2.29 | 2.23 x e ⁻² | 0.343 | 12 |
| Neat DGEBF/IDA | 5.93 | 5.64 x e ⁻⁶ | 0.723 | 2.03 | 4.906 x e ⁻⁴ | 0.504 | 40.05 |
| 1 wt% SiO ₂ DGEBF/IDA | 16.70 | 2.904 x e ⁻⁶ | 0.731 | 2.837 | 1.09 x e ⁻³ | 0.584 | 129 |
| 3 wt% SiO ₂ DGEBF/IDA | 6.45 | 9.83 x e ⁻⁶ | 0.680 | 03.84 | 02.884 x e ⁻³ | 0.496 | 168.5 |
| 5 wt% SiO ₂ DGEBF/IDA | 12.40 | 5.632 x e ⁻⁶ | 0.735 | 28.45 | 0.544x e ⁻⁴ | 0.659 | 266.1 |
| 7 wt% SiO ₂ DGEBF/IDA | 12.62 | 1.078x e ⁻⁶ | 0.799 | 42.23 | 1.335 x e ⁻³ | 0.505 | 292 |
| 10 wt% SiO ₂ DGEBF/IDA | 8.38 | 4.69 x e ⁻⁶ | 0.762 | 33.77 | 0.38 x e ⁻³ | 0.669 | 280 |

Generally, all the organic coatings are not completely impenetrable for long time, their barrier properties could decrease when immersion time increases because of the water/corrosion medium penetration into the coatings. For bare MS, the corrosion medium had a direct contact with the metal surface which led to the generation of many electroactive sites and corrosion will take place freely. Actually, corrosion reaction will takes place in the presence of moisture and oxygen. Silica containing polymer coatings prevent the diffusion of oxygen and aggressive medium into the polymer matrix due to the high cross linking of polymer network and -Si-O-Si- linking in the polymer network. From the contact angle measurement, it can also be understood that, all the silica containing polymers are hydrophobic, which could effectively reduce the wettability of polymer which ultimately reduces the sorption of water molecules in the coatings [30]. Among the different wt% of bio-silica reinforced DGEBA-IDA composites used in the present work, bio-silica reinforced DGEBA-IDA composites offers a better corrosion resistant property due to the presence of more nitrogen atoms in imidazole derivatives and alkyl side chain in the cardanol moiety. It helps to improve the water contact angle of DGEBA-IDA composites and in turn contributes to an enhanced corrosion resistant behavior.

Fig. 7 represents the Tafel plots and the values of the corrosion parameters for the coated MS specimens are given in Table 3. The curve a represents the Tafel plot of uncoated specimens while curves a-d represent the Tafel plot of the coatings loaded with the different amount of bio-silica reinforced DGEBA-IDA

composites. The corrosion rate (CR) was calculated using I_{corr} values in the equation (1),

$$CR = MI_{corr} / \rho nF \dots\dots\dots (1)$$

Where M is the molecular mass of copper (58.69 g mol⁻¹), I_{corr} is the corrosion current density(Acm⁻²), F is the Faradays constant (96500 A s mol⁻¹), ρ is the density of the mild steel specimen (7.85 g cm⁻³), and the number of electrons transferred during corrosion reaction is assumed to be 2 [15].

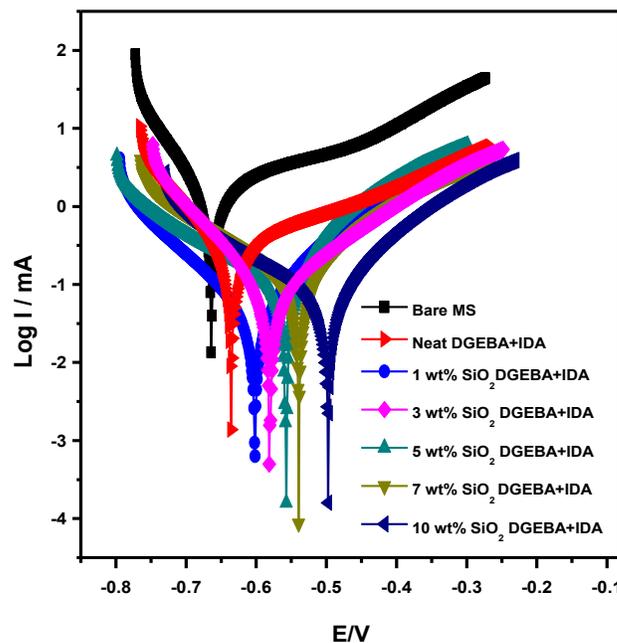


Fig. 7: Tafel plots of bare MS and bio-silica reinforced DGEBA-IDA composites coated specimens in 3.5% NaCl solution

Table 3: The values of the corrosion parameters of the coated and uncoated mild steel specimens in 3.5 % NaCl solution calculated from Tafel studies

| Specimens | E_{corr} (mV) | I_{corr} (μA) | CR mm year^{-1} | Efficiency η (%) |
|-----------------------------------|------------------------|-------------------------------------|--------------------------|-----------------------|
| Bare | -701 | 1350 | 0.0487 | 0 |
| Neat DGEBA/IDA | -636 | 187 | 0.0274 | 86.15 |
| 1 wt% SiO ₂ DGEBA/IDA | -560 | 108 | 0.0195 | 92.00 |
| 3 wt% SiO ₂ DGEBA/IDA | -501 | 62 | 0.0197 | 95.41 |
| 5 wt% SiO ₂ DGEBA/IDA | -583 | 58 | 0.0281 | 95.70 |
| 7 wt% SiO ₂ DGEBA/IDA | -510 | 40 | 0.0486 | 97.04 |
| 10 wt% SiO ₂ DGEBA/IDA | -543 | 46 | 0.0203 | 96.59 |

The E_{corr} values of the polymer coated samples are increased anodically and I_{corr} values the coated specimens are reduced by indicating that corrosion resistance of the coated specimens are improved after coating. This Effect is more pronounced when bio-silica are loaded in the coating. The improved corrosion resistance may be due to the suppression of the anodic corrosion reactions by the bio-silica reinforcement [31]. The 10 wt% bio-silica reinforced DGEBA-IDA composites coating shows the better positive shift in terms of E_{corr} value and lower corrosion current. Data obtained from corrosion studies, it is inferred that the presence of nitrogen rich imiazole core based IDA moiety influence the corrosion resisting behavior.

4. CONCLUSION

The present work is to develop bio-silica reinforced epoxy composite coatings to utilize them for protection of mild steel surfaces from corrosion. In this context, Imidazole core diamine (IDA) was synthesised and used as a hardener for epoxy resin. The epoxy resin was reinforced with varying weight percentages of 3-glycidoxypropyltrimethoxysilane (GPTMS) functionalized rice husk ash bio-silica to obtain respective composites. Their composite morphology and thermal stability was studied using SEM analysis, thermogravimetric analysis (TGA). The values of limiting oxygen index calculated from TGA data infer that all the samples of epoxy matrix and composites possess good flame retardant behaviour. In order to ascertain their hydrophobic behaviour, the water-contact angle measurement studies were carried out. The values of contact angle obtained for neat IDA/DGEBA matrix is 86°, where as that of 1, 3, 5, 7 and 10 wt% bio-silica reinforced IDA/DGEBA are 89°, 92°, 95°, 97° and 98° respectively. IDA/DGEBA matrix and functionalised bio-silica reinforced IDA/DGEBA composites were coated on the mild steel specimens and their corrosion resisting behaviour was studied by electrochemical impedance spectroscopy and

potentiodynamic polarisation. Data resulted from corrosion studies (Nyquist plot and Tafel plot), inferred that among the coated specimens studied, the specimen coated with 7 wt% bio-silica reinforced IDA/DGEBA matrix exhibits the highest corrosion resisting efficiency.

Conflict of interest

The authors confirm that this article content has no conflict of interest.

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