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RECENT ADVANCES IN CATIONIC AQUEOUS EPOXY NANOCOMPOSITIONS CONTAINING ALUMINA AND TH EIR ELECTRODEPOSITION

Ravi Shankar*[1], K.G.Manjunath[2] A.K.Shuka[3] and K.S.Badari Narayan[4]
[*1]Associate Professor-Department of chemistry, M.S. Engineering College, Bangalore
[2]Professor-Department of Chemistry, Ghosia College of Engineering, Bangalore
[3]Professor, Department of Chemistry, East Point College of Engineering, Bangalore
[4]Principal, M.S. Engineering College, Bangalore

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ABSTRACT

Presently the researchers have focussed their attention on polymer nano-composites that show enhanced physico-mechanical and electrical properties compared to their conventional polymer composite counterparts. Electrophoretic deposition (EPD) of organic polymers and composite coatings on metal substrates is an important technique that finds wide application in industry. The process in essence involves application of DC voltage between the conducting anode and cathode suspended in the polyelectrolyte emulsion resulting in the electrodeposition of the polymer composite coating on theelectrode.This paper deals with the conversion of bifunctional epoxide resin into a water dispersible cationic emulsion, dispersion of nanoparticles, alumina powder and cathodic EPD of these nano composite coatings for application as dielectric and corrosion resistant coatings. Epoxy resin is chemically modified by reacting with a secondary amine to obtain an epoxy-amine adduct. The process was monitored by observing exotherm and the rise in epoxy equivalent weight using the standard volumetric method and supported by FTIR spectroscopy. IR spectrum of the adduct shows a very weak or nearly absence of typical absorption band of epoxy group at 914 cm^{-1} and appearance of sharp bands of tertiary amino group at 1361 and 1379 cm^{-1}. Neutralisation of adduct with an acid results in the formation of quaternary ammonium salt that disperses in water forming a stable cationic emulsion at a pH of 6.0 - 6.5. conventional Alumina and Natural montmorillonite (MMT) nanoclay was incorporated in aqueous epoxy emulsion using ultrasonication at 20 kHz to ensure intercalation of polymer in between the nanoclay platelets.
Adherent nanocoatings were electrodeposited on Copper metal cathode and were rinsed, dried and cured at 150°C. Effect of bath parameters viz., resin content, Alumina content, nanoclay content, pH, conductivity, and the deposition parameters viz., dc voltage, interelectrode spacing and voltage application time were studied. The neat epoxy coatingand Epoxy clay alumina coatingss SEM and XRD analysis confirmed the nanostructure of the clay particles in the composite. Salt fog Corrosion resistance of EPD epoxy Alumina-MMT nanocoatings is superior to neat epoxy coating.Epoxy- Resin content of the bath emulsion studied in the range 6 – 16% by weight, have direct impact on the mass of the deposited coating or thickness. MMT nanoclay content were varied from 3 to 7% by weight of resin, Alumina from 2 to 8%. beyond which it posed processing difficulty due to viscosity rise after ultrasonication. Thermogravimetric analysis (TGA) of the cured coating in air has shown thermal stability upto 175°C when the weight loss due to decomposition begins. There is remarkable increase in electrical strength of the cured nanocoatings i.e. 29 kV/mm at 5% MMT by wt., against 18 kV/mm for neat epoxy coating. There is a improvement in impact strength of the epoxy alumina -MMT coating than pure epoxy coating.

KEYWORDS: Epoxy resins, Electrodeposition, Alumina powder, Montmorillonite, Scanning Electron Microscopy(SE), Flexural strength, Thermo Gravimetric Analysis (TGA)

I. INTRODUCTION

Epoxy nanocomposites (ENCs) have been attracting the focus of researchers for over a decade. ENCs generally contain an inorganic nano-filler such as metal oxide, silicate or a nanoclay, and can be prepared by incorporating

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nano-sized inorganic filler particulate in the polymer matrix at as low loading volumes as <7 percent by weight. These have been reported [1-6] to possess enhanced physico mechanical and dielectric properties.

Natural montmorillonite nanoclays, have lamellar structure comprising of a layer of alumina octahedrons, surrounded by two layers of silica tetrahedrons [7,8]. The residual negative charge in the layer is compensated by the cation present in the interlayer spacing. The exchange of these cations by that of the intercalating agent or polymer results in the higher compatibility of the montmorillonite with the polymer. On treatment with liquid polymer or intercalating agent the layered nanoclay initially undergoes intercalation and finally exfoliation.

ENCs are prepared by dispersing the Alumina powder and nano filler in the liquid polymer using ultrasonication, followed by conventional processing, which are having enhanced physico mechanical and electrical properties[9,10].The problem of compatibility between the organic-inorganic interfaces, was addressed by using the organically modified nanoclay or organoclay[11,12].

Recently, a new class of polymer nanocomposites containing nano sized inorganic filler particles such as metal oxides, hydroxides, silicates, carbonates and nanoclays, in the polyelectrolyte matrix prepared by electrophoretic deposition (EPD) process, have been reported [13-20]. EPD Process is a novel method to deposit composite films containing inorganic nanoparticles and a polyelectrolyte under the influence of a DC voltage. The process involves dispersion of cationic or anionic polyelectrolyte in water and charged Nano clay or nanofiller particles and Alumina powder and their electrophoretic movement towards the respective electrodes and subsequent deposition.

Several papers and patents are available in literature[21-27] describing methods of preparation of electrodepositable epoxy based aqueous compositions containing conventional fillers, their electrophoretic deposition and study of electrodeposited coatings.

Recently a composite epoxy coating containing montmorillonite obtained by an electrophoretic deposition (EPD) process has been reported [28]. The microstructural analysis TEM and XRD confirmed the intercalation of montmorillonite layers with epoxy. Functional characterisation (EIS and TMA) demonstrated the improved properties of the reinforced epoxy coating with respect to the simple epoxy coating.

In the present work to the Epoxy montmorillonite, Alumina powder is added and a new composite is prepared. The addition of Alumina and MMT improves the thermal, mechanical and electrical properties

II. EXPERIMENTAL

Materials Used:

Commercial Epoxy resin based on diglycidyl ether of bisphenol-A (DGEBA) with epoxy equivalent weight of 833-895 from Atul India, laboratory reagent grade amines such as n-Butyl amine and Dialkyl amine from SD Fine Chemicals and Urea and formaldehyde of m/s Thomas baker make were used in the preparations. Ethylene glycol mono butyl ether, Methanol and Xylene of Laboratory reagent grade were used as solvent during the preparation. Alumina powder is purchased from Sigma Aldrich

Sodium Montmorillonite nanoclayCloisite Na+ from m/s Southern Clay products, USA, was procured for use as nanoclay particles. Aluminium and Copper metal sheets grade SI 304 of 1.75mm thickness cut to sizes 100 x 25 mm and 100 x 50 mm with edges rounded off, were used for cathode and anode during EPD process.

Preparation of Aqueous Cationic Epoxy Emulsion:

In order to make epoxy resin water dispersible it is necessary to introduce an ionisable group, a quaternary ammonium group in its molecular structure for making a cationic resin. A solution of DGEBA epoxy resin is treated with a secondary amine while stirring and allowed to react under controlled reaction conditions to form an Epoxy-amine adduct. The solutions of epoxy-amine adduct and a lab made amino aldehyde resin are mixed and acidified to form a quaternary ammonium salt which is dispersed in water by continuous stirring to form an emulsion, which is fairly stable at room temperature for long duration, without any separation or lump formation.

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ALUMINA POWDER: Al_2O_3

Aluminium oxide is a chemical compound of aluminium and oxygen with the chemical formula Al_2O_3.It is commonly called alumina, Alumina contributes 15% of the earth's crust and is amphoteric in nature. It has strong ionic inter-atomic bonding. It commonly occurs in its crystalline polymorphic phase α-Al_2O_3, in which it comprises the mineral corundum,. Al_2O_3 is significant in its use to produce aluminium metal, as an abrasive owing to its hardness, and as a refractory material owing to its high melting point. Al_2O_3 is an electrical insulator and has a relatively high thermal conductivity (35 W/m-K). Alumina powder with different weight percentage (2.%,4% ,6%,8%) is dissolved in ethanol and coupling agent and is added to epoxy emulsion followed by ultrasonication for 30 min, to get Epoxy - alumina emulsion. In this 6% weight of alumina gives optimum deposit.

Dispersion of Nano clay particles:

The montmorillonite nanoclayCloisite Na+ particles were dissolved in coupling agent and dispersed in epoxy-Alumina emulsion and subjected to ultrasonication at 20kHz intermittently in order to avoid heating. The ultrasonication process helps the intercalation of polyelectrolyte cation between the layers of the nanoclay platelets leading to exfoliation of the particles and thorough dispersion.

Study of EPD Process Parameters:

Montmorillonite nanoclayCloisite Na+ at various percentage is added to epoxy emulsion. Then it is dispersed in the cationic epoxy emulsion by subjecting to Ultrasonication at about 20 kHz frequency for 30-60 minutes The resulting dispersion of nanoclay and Alumina powder was very stable without any separation or agglomeration of nanoparticles for prolonged periods, storable for several weeks, indicating effective intercalation and exfoliation of the nanoclay composites. An electrophoretic cell comprising of a cathode and an anode connected to a DC source was fabricated. Electrodeposition of epoxy nanocomposite at different dc voltages was carried out on Copper metal plate specimens of size 100 x 25 or 50 mm, followed by drying and curing. For comparison, EPD of neat epoxy coating, without nanoclay particles, was also carried out and coatings were cured under the same condition. Quality of deposited coating, like uniformity, freedom from defects such assuch as blisters, cracks or peeling off, was critically observed. Coating build-up or deposition yield with respect to deposition time (Fig.1), was monitored by weight measurement of deposited coating after drying. To avoid gassing on the electrodes and to obtain the uniform and defect-free coating, the applied voltage was kept low, below 20V and the distance between the electrodes is varied while keeping all other parameters constant. Fig.2 shows the decrease in the deposition yield with respect to increase in the inter electrode spacing.

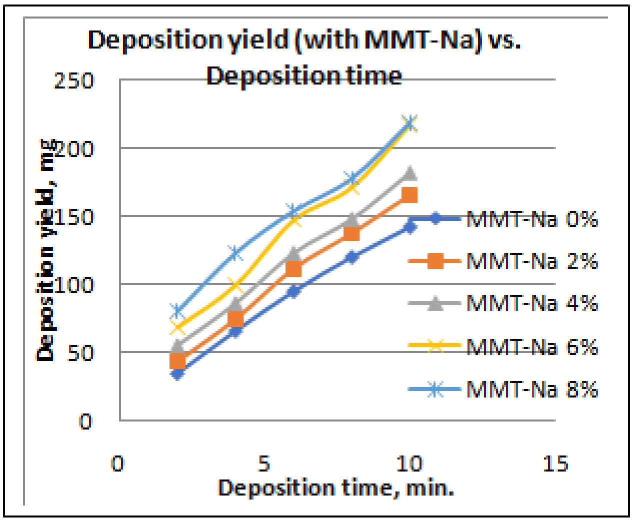

Fig1: ENC Deposition yield vs, deposition time at 6V

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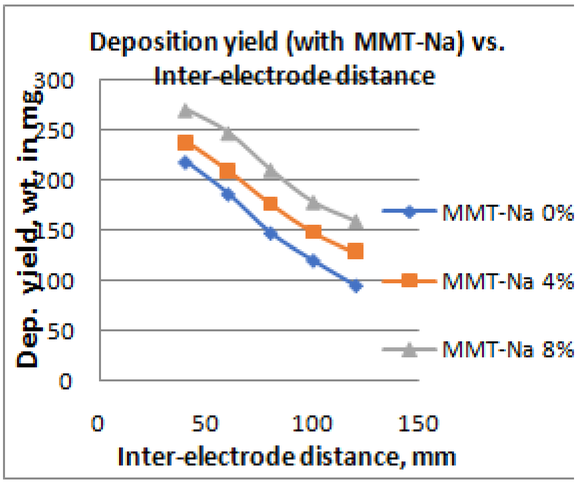

Fig.2: Deposition yield vs. inter-electrode pacing

Curing of Nanocomposite Coating
Extent of cure of coating was ascertained by determining the insoluble Gel Content using the Soxhlet Extraction method with acetone as solvent. The percentage of insoluble matter or the extent of cure at 150°C for different durations has been determined. Optimum cure schedule is established as the insoluble or cured polymer content approach almost constant (96-97%) after 3 hrs at 150°C.

III. CHARACTERISATION

FTIR Spectroscopy:
FTIR spectra of epoxy resin and epoxy amine adduct were taken on Perkin Elmer FTIR “Spectrum-BX” Spectrophotometer using KBr pellet medium at I.I.Sc., to confirm the epoxy-adduct formation by comparison [8]. IR spectrum of Epoxy resin (fig.3) shows the typical band at 914 cm^{-1} due to the terminal epoxy group confirmed by the presence of band at 1245 cm^{-1} besides the presence of other groups of a DGEBA epoxy resin.

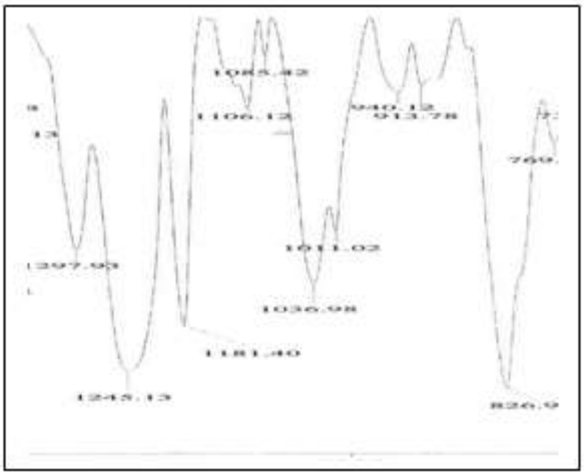

Fig.3. FTIR spectrum of Epoxy resin

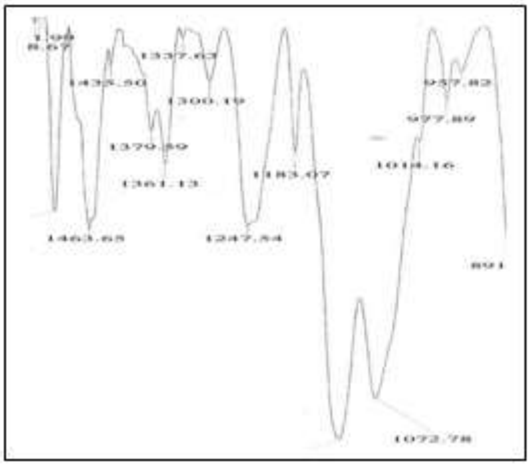

Fig 4. FTIR spectrum of adduct

IR spectrum of Epoxy adduct (fig.4) shows very weak or nearly absence of typical absorption band of epoxy group at 914 cm^{-1} indicating the consumption of epoxy groups in adduct formation. The sharp bands at 1361 and 1379 cm^{-1} indicate presence of tertiary amino group of the epoxy-amine adduct.

Epoxy Equivalent Determination:

Standard Volumetric Method as per ISO 3001:1997(E) for Determination of Epoxy Equivalent was used to monitor the epoxy-amine adduct formation. The method is fairly sensitive and the results are shown in the Table no. 1.

Table 1: Determination of Epoxy Equivalent of Epoxy resin and Adduct

Sl. no.	Resin	Ep. Equiv.(g/eq) (datasheet)	Ep. equiv.(g/eq) (measured)	Test method
1	Epoxy resin	833-895	886-928	ISO 3001:1997(E)
2	Epoxy-amine adduct	-	2745-2810	- do -

Dielectric properties measurements:

Dielectric strength and Volume resistivity of the nanocoatings obtained through electrophoretic deposition process from several nanocompositionscontaining different MMT contents by weight of resin in the epoxy alumina emulsion, was carried out. Fig.5 shows the effect of nanoclay content and alumina on the dielectric strength and Volume resistivity of the resulting nanocoatings.

Thermogravimetric Analysis (TGA):

TGA was conducted at Thermogravimetric Analyser Model V4.3A of TA Instruments at CPRI. The sample of EPD epoxy nanocomposite coating was subjected to TGA in air to check the thermal stability of the coating. The thermogram is shown in Fig.6. The coating is fairly stableupto 175°C when the weight loss or decomposition begins.

Scanning Electron Microscopy (SEM):

Microstructure and the morphology of EPD nanocomposite coatings were studied by using Scanning Electron Microscope Hitachi model SU-1500 and comparing the same with neat epoxy coating made through EPD process. The micrographs of the neat epoxy and epoxy with 4% MMT and epoxy containing Aluimina are shown in fig.7,8. and fig.9

Corrosion Resistance (Salt fog) Test:

Neat epoxy (without addition of nanoclay) emulsion based EPD coatings with and without addition of alumina and MMT nanoclay particles deposited over the flat Alplate specimens of size 100 x 50 mm, were placed in the test chamber and subjected to the salt fog with 5% sodium chloride solution at standard temperature 35°C for several hours/days of duration, followed by examining their Cross-Hatch Adhesion strength. As per ASTMD3359-87method. Observations given in Table 3 show higher corrosion resistance of the nanocomposite coating containing MMT and alumina than the neat epoxy EPD coating.

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Flexural Strength:
Flexural strength of pure Epoxy resin, epoxy with different concentration of Alumina and MMT were performed using Three-point transverse bending setup for a rectangular Cu metal coating.

Table.2 Flexural strength of neat Epoxy and Epoxy alumina nano composite coating

Sl. no.	Subs-trate	EPD Coating	Nanoclay content	Flexural Strength (Mpa)
1	Cu	Neat Epoxy	0	59.35
		Neat Epoxy +4% Al_2O_3	0	64.38
		Neat Epoxy+ 6% Al_2O_3	0	71.92
		Neat Epoxy+ 8% Al_2O_3	0	61.23
2	Cu	Ep + MMT+Al_2O_3 (6%)	2%	74.86
			4%	78.65
			6%	75.45

IV. RESULTS AND DISCUSSION:

It may be noted from section 2.3 that we have successfully developed an electro-active and stable aqueous emulsion based on cationic epoxy resin system which facilitates cathodic deposition of epoxy resin and the dispersed nanoclay particles. Formation of epoxy-amine adduct was monitored by determining the epoxy equivalent of the adduct and comparing with that of the epoxy resin as shown in the Table 1. This is confirmed by FTIR spectra of the two products respectively as given in Fig.3 and Fig.4 wherein the spectrum of the adduct shows the loss of terminal epoxy group and the appearance of tertiary nitrogen group in the molecule as reported in the section 3.2.

Curing schedule for the EPD-ENC has been optimised by Soxhlet extraction method and plotting the Insoluble content against the curing time at 150°C. The insoluble content in the cured coating, become almost constant at 96-97% after 3 hours curing at 150°C. Thermal Analysis by TGA of the cured EPD-ENC coating shows (Fig.6) thermal stability of the coating upto 175°C.

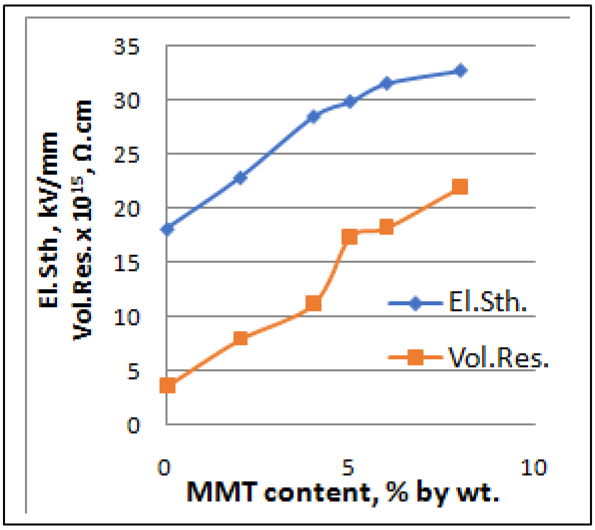

Fig.5: Electrical properties vs. MMT content

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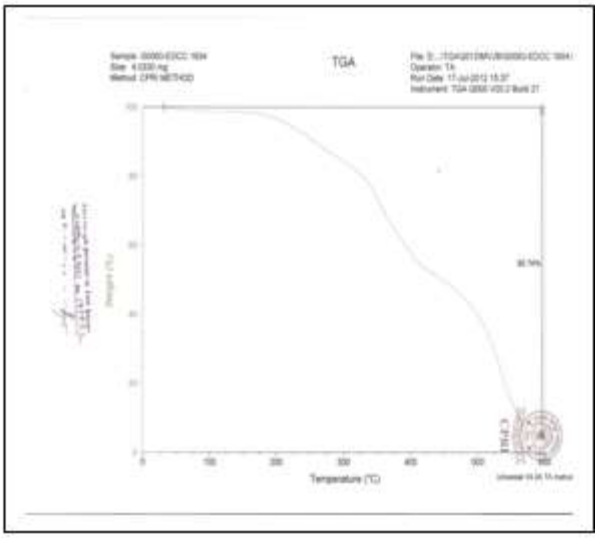

Fig.6: TGA Thermogram of EPD-ENC

Referring to the data plotted in Fig.1 pertaining to increase in deposition yield against increase in deposition time, it may be noticed that the thickness build-up of EPD coating is almost linear with respect to deposition time at a particular voltage at lower content of nanoclay MMT-Na. A defect free and uniform coating on cathode is obtained at a low voltage preferably at less than 20V dc. Higher voltages caused gassing on the electrodes leading to defects and unevenness. Fig.2 above shows that the thickness build-up decreases as the inter-electrode distance is increased.

The microstructures of the nanocomposites were characterised by means of Scanning Electron Microscopy (SEM) of the EPD neat epoxy coating and the EPD epoxy nanocomposite with 4% MMT, and with Alumina. The micrographs are shown in Fig.7,8 and fig 9 respectively

Fig7: SEM image of EPD-epoxy (neat) mag 2K

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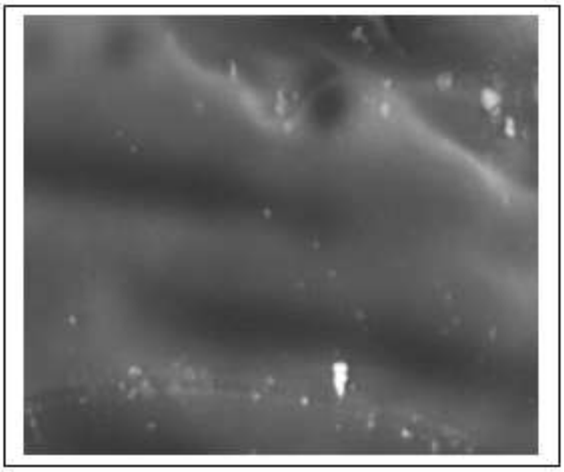

Fig.8 :SEM image of EPD-epoxy +4% MMT(mag. 3K)

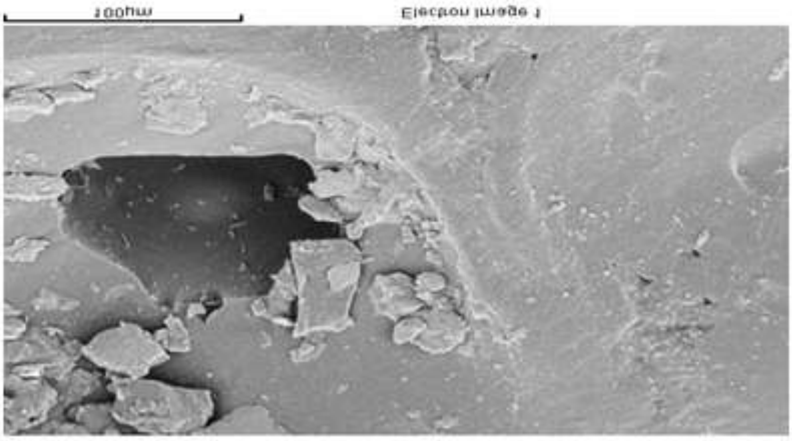

Fig.9: SEM image of EPD-epoxy +6%Alumina+4% MMT (mag. 3K)

Micrograph of the EPD nanocomposite with 4% MMT clearly shows the uniform Distribution of nano clay particles and Alumina in the resin matrix. However, presence of some white spots indicates the agglomeration of the nanoclay particles which were probably not disintegrated during ultrasonication.
Fig.5 shows the rise in Dielectric strength of EPD-ENC from about 18 to 37kV/mm due to addition of 6-7% MMT and 6%Alumina by weight of the resin. Similarly, the volume resistivity of EPD-ENC rises from 4 to 24 x $10^{15}\Omega$cm at the same 7% MMT and 6% Alumina content.

Table no. 3: Corrosion resistance of EPD-epoxy-MMT nanocoating against neat epoxy EPD coating

Sl. no.	**Subs-trate**	**EPD Coating**	**Nanoclay content**	**Salt fog exposure, hrs.**	**C.H.A. Rating**	**Observation/ Appearance**
1	Cu	Neat Epoxy	0	0	5B	No peeling
				400	4B	Upto 5% area peeling at scribes
				800	1B	35-65% peeling at scribes
2	Cu	Epoxy+MMT+Al_2O_3	4%	0	5B	No peeling
				400	5B	No peeling
				800	4B	Upto 5% area peeling at scribes

Corrosion resistance has been assessed by comparing the Cross-Hatch Adhesion strength rating of the EPD coating of neat epoxy and the nanocomposite containing 4% MMT and 6% alumina by weight of the resin given

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in Table-2. Epoxy+MMT+alumina coating shows better corrosion resistance (4B rating (5% peeling) after 800 hr exposure to salt fog spray) in Corrosion Test chamber, as compared to 1B rating (35-65% peeling) for neat epoxy EPD coating according to ASTM method.

V. CONCLUSION

i) Modification of DGEBA Epoxy resin has been successfully carried out into a water dispersible and electrodepositable cationic resin.

ii) A novel process for electrophoretic deposition (EPD) of epoxy nanocomposite (ENC) containing a montmorillonite nanoclay and Alumina is developed, the process parameters have been established.

iii) Thickness or deposition yield of EPD-ENC coating increases linearly with deposition time.

iv) Thickness/ deposition yield of EPD-ENC coating decreases linearly with inter-electrode spacing.

v) Optimum curing of EPD-ENC coatings is achieved at 150°C-3 hours.

vi) Thermal stability of the EPD-ENC coating is up to 175°C according to TGA.

vii) Dielectric strength (rises from 18 to 32 kV/mm) and Volume resistivity (rises from 4 to 22 x 10^{15}Ωcm)of EPD-nanocomposite, rise with increase in MMT content upto7wt% of the resin.

viii) SEM micrographs indicate nanostructure of the EPD coating surface.

ix) Corrosion resistance (with respect to Cross Hatch adhesion strength rating before and after the salt fog exposure for 800 hrs.) of EPD-Epoxy-MMT nanocomposite coating improves as compared to that of the neat epoxy-EPD coating.

x) There is a increase in the Flexural Strength for Epoxy resin with Alumina and nanoclay MMT compared to pure Epoxy Coating

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