

SYNTHESIS OF PHOSPHAZENE IMINE/ EPOXY FOR HIGH PERFORMANCE APPLICATIONS

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Abstract:

In the present work, phosphazene modified imine was prepared and its molecular structure was confirmed using FTIR and ¹H NMR analysis. The modified phosphazene imine (PZ imine) of different weight percentages was incorporated into DGBEA epoxy resin to obtain composites and the formation of composites was confirmed by FTIR analysis. The composites were subjected to thermal, radiation resistant, electrical and antibacterial studies. Data resulted from thermal, UV absorption, antibacterial and surface behaviour studies indicate that PZ imine reinforced epoxy composites possess higher thermal stability, enhanced zone of inhibition, better UV shielding, improved hydrophobic behaviour and lower value of dielectric constant than those of neat epoxy matrix. The morphology of the samples was analysed using high resolution transmission electron microscope (HRTEM), which ascertained the homogeneous dispersion of PZ imine in epoxy matrix with fibrous structure.

Key Words: phosphazene imine, epoxy resin, antibacterial activity, dielectric constant, radiation resistance.

Introduction

Epoxy resins possess an excellent thermal stability, high modulus and enhanced electrical properties. The structure of epoxy resin influences high chemical resistance against wide range of corrosive conditions [1-5]. In order to make epoxy resins more suitable for high performance applications, its deficient behaviors like brittleness, moisture, absorption and flammability have to be improved [6-9]. In this direction continued research is on to improve the properties through

the modification of epoxy resins with flexible polymeric segments and chemical modifiers to an appreciable extent, but still some more effective enhancements are required with regard to thermo mechanical, dielectric and weather resistant behavior suitable for high performance applications [10-13]. The Schiff base is an efficient organic inhibitor preferred by coating industry, since it contains hetero atoms such as nitrogen, oxygen, phosphorus, conjugated double bonds in their skeleton through which they adsorbed on metal surface by the formation of an efficient adherent film [14]. Thus the role of oxygen, nitrogen, and phosphorus moiety present in epoxy containing Schiff base system is expected to exhibit synergism in establishing toughness, flame retardancy, thermal stability, flexibility and excellent corrosion resistance, good dielectric behaviour, good antifouling, radiation resistant and high affinity towards different substrates. These improved properties can be achieved because of their structurally modified chemical skeleton and the different hetero atoms and functional groups incorporated in the molecular structure of epoxy resin. The presence of $\text{CH}=\text{N}$ (imine) group in the modified epoxy resin improves an adhesion of the coating to a metal substrate with enhanced thermal stability and antimicrobial activity [15-21].

In this context, hexachlorocyclotriphosphazene (PZ), 4-hydroxy benzaldehyde and p-aminophenol were used for the synthesis of phosphazene based imine (PZ imine) with nanosized fibrous structure. Since PZ -imine is expected to impart good physiochemical, thermomechanical and surface properties suitable for modification of epoxy resin[22-26].

In the present work phosphazene based imine was synthesized and varying weight percentages were reinforced with DGEBA epoxy resin to obtain PZ-imine/epoxy composites with improved. Data obtained from different studies are discussed and reported.

Experimental

Materials

Epoxy resin (diglycidyl ether of bisphenol- A, DGEBA, with EEW 182-192, viscosity) and 4, 4-diaminodiphenylmethane (DDM) were obtained from Huntsman (India). Hexachlorocyclotriphosphazene ($\text{N}_3\text{P}_3\text{Cl}_6$), 4-hydroxy benzaldehyde and p-aminophenol were purchased from Alfa aesar (Germany). Chloroform, acetone, THF and toluene were purchased from Sisco Research Laboratories, India[27-31].

Synthesis of hexakis (4-aldehydephenoxy)-cyclotriphosphazene (PZ-CHO)

PZ-CHO was prepared by mixing 3.96 g of NaH (0.165mol) (70 wt % diluted in oil) and a solution of 14.04g (0.11 mol) of 4-hydroxybenzaldehyde in 50mL THF in a reactor with efficient stirring under nitrogen atmosphere. Subsequently, 5g (0.014 mol) of hexachloro cyclotriphosphazene ($N_3P_3Cl_6$) was dissolved in 50mL of THF and added in dropwise into the flask over a period of 60 min. Then the reaction system was maintained at 65 °C for 48 h. Light brown coloured product (PZ-CHO) obtained, was recrystallized with ethyl acetate and preserved for further use and it show in scheme 1 [32-39].

Synthesis of hexakis [(4-hydroxyphenylimino) methyl] phenoxy)-cyclotriphosphazene] (PZ-imine)

10g (0.0116mol) of PZ-CHO and 7.60g (0.069 mol) of p-aminophenol in dry toluene (50 mL) were refluxed in an oil bath using Dean-Stark water separator for 4-5 h (Scheme 4.2). The progress of the reaction was monitored on TLC plate. Excess of toluene was then distilled off and the product obtained was separated and recrystallized from alcohol [40-45].

Preparation of PZ-imine/ epoxy composites

The DGEBA epoxy resin and the varying weight percentages of PZ-imine (1, 3, 5 and 7 wt %) were separately stirred mechanically at 50°C for 24 h. A stoichiometric amount of DDM, corresponding to epoxy equivalents was also added. The product obtained was poured into a Petri dish. The samples were cured successively at 120 °C for 2 h, post cured at 180 °C for 3 h, and finally removed from the mould and characterized.

Characterization

Fourier transform infrared (FT-IR) spectra were recorded on a Perkin Elmer 6X FTIR Spectrometer at room temperature in the range of 4000–400 cm^{-1} . ^1H -NMR analyses carried out in $CDCl_3$ and Deuterated DMSO are recorded at 400 MHz on a Bruker NMR spectrometer (model Z123789). Thermogravimetric analysis of modified epoxy matrix was performed on a Netzsch STA 409 thermo gravimetric analyzer. The instrument was calibrated with calcium oxalate and aluminium supplied by Netzsch. The samples (about 50 mg in weight) were heated from ambient

temperature to 800 °C with a continuous flow of nitrogen (60 ml/min) and thermograms were recorded at a heating rate of at 10 °C/min.

A JEOL JSM-6360 field emission scanning electron microscope was used for morphological studies of different weight percentages of PZ-imine reinforced epoxy nanocomposites measured before and after UV exposure. The high resolution transmission electron microscopy (HRTEM) analysis was carried out using TECNAI-G2 (model T-30) operated at an accelerating voltage of 300 kV. A HRTEM samples were prepared by dispersed the composite samples in ethanol mounted on carbon-coated copper TEM grids and dried for 1 h at 70°C to form a film thickness of <100 nm. The dielectric studies of the neat epoxy and PZ-imine reinforced epoxy composites were determined with the help of an impedance analyzer (impedance/gain phase analyzer 1260, Solartron, UK) at room temperature using platinum (Pt) electrode at 30 °C at a frequency range of 1 MHz and this experiment was repeated four times at the same condition.

In order to ascertain the UV radiation resistant behavior of the composites, the epoxy matrix and varying weight percentages of PZ-imine reinforced epoxy composites were placed in the 365 nm UV photoreactor and subject to ultra-violet radiation for a period of one week at room temperature. The light intensity of UV lamp at 8W and 12 lamps are present in the UV photo reactor (Hebber Multilamp, HML Compact-Lp-Mp-812). The distance between the sample and the lamp is 10 cm and thickness of the samples is 3 mm. After UV exposure, the different weight percentages of PZ-imine reinforced epoxy composites surfaces were reanalyzed using SEM and the results are compared with that of UV unexposed samples of composites. The UV absorption and transmittance spectra were recorded in Jasco-(V-650) spectrophotometer.

Results and discussion

FTIR spectral analysis of phosphazene

The FTIR spectra of PZ-CHO and PZ-imine are presented in Figure 1(a-b). The disappearance of P-Cl bond and the formation of P-O linkage were observed 600 and 1019 cm^{-1} respectively. The strong absorption bands appeared at 1161 and 961 cm^{-1} indicate the presence of phosphazene linkage (P-O-Ph) and the band appeared at 2825 and 1706 cm^{-1} confirms the presence of an aldehyde group (-CHO) in the matrix it show in Figure 1(a) [8]. The FTIR spectrum of PZ imine is shown in Figure 1(b). From the spectrum, the disappearance of aldehyde

absorption band at 1706 cm^{-1} and the appearance of bands at 1599 cm^{-1} and 3273 cm^{-1} confirm the formation of PZ imine and terminal OH group.

The FTIR spectra of neat epoxy matrix and 1, 3, 5 and 7 wt% of PZ-imine/epoxy nanocomposites are shown in Figure 2. The high intensity bands obtained at 1182 , and 949 cm^{-1} are assigned to P-O-Ph linkage and 1609 cm^{-1} correspond to CH=N stretching vibration. The disappearance of band at 918 cm^{-1} confirms the curing reaction. The bands appeared at 2930 and 2850 cm^{-1} represent symmetric and asymmetric CH_2 groups respectively. The appearance of broad band at 3422 cm^{-1} corresponding to -OH, becomes weak and indicating that the DGEBA epoxy underwent a complete curing reaction with PZ-imine to form an inter cross linked network structure.

^1H NMR analysis

Figure.3 shows the ^1H NMR spectrum of PZ-CHO matrix and the single peak appeared at 9.8 ppm represent the aldehyde proton. The two doublet peaks appeared between 7.2 ppm and 7.8 ppm are represent the aromatic protons in the phenyl ring (Liu & Wang 2009). The ^1H NMR spectrum of PZ-imine is shown in Figure 4. The peak appeared at 10 ppm corresponds to the terminal hydroxyl group and the appearance of new peak at 8.5 ppm ascertains the presence of imine proton [15].

Thermal properties

Data obtained from the thermogravimetric analysis of neat epoxy matrix and varying weight percentages of PZ imine reinforced epoxy nanocomposites are presented in Figure 5 and Table 1. The incorporation of PZ imine (0, 1, 3, 5 and 7 wt %) into epoxy matrix increased the value of char yield. The value of char yield shows the increase in the thermal stability of PZ imine reinforced epoxy nanocomposites, which may be explained due to the presence of stable phosphorus and nitrogen present in the epoxy back bone. This may be attributed due to the increase in steric hindrance and high cross linking network formation between epoxy matrix and phosphazene group [8, 17]. The phosphorus rich char formation which delayed the degradation process of epoxy nanocomposites.

Morphology

Figure 6 illustrates the SEM analysis of fractured surfaces of UV unexposed and exposed PZ-imine reinforced epoxy nanocomposites. The SEM images observed for the neat epoxy matrix indicate the poor resistance to crack propagation. However the PZ-imine incorporation into epoxy protects them as self shielding against UV radiation [18-20]. The PZ-imine reinforced epoxy nanocomposites show smooth surface without any disturbance. This is due to absorption of energy by imine ($\text{CH}=\text{N}$) group and can prevent cracking and fading of polymer matrix due to prolonged exposure of high energy UV radiation.

Figure 7 shows the TEM images of 3 and 7 wt% PZ-imine/epoxy reinforced nanocomposites. From the Figure, it can be ascertained that the 3 wt% PZ-imine epoxy reinforced composites possesses lesser concentration of PZ-imine content, whereas 7 wt% PZ-imine/epoxy reinforced composites show higher concentration PZ-imine which can be seen in the Figure. The PZ-imine possesses fibrous morphology and uniformly dispersed within the epoxy matrix [20]. This is due to the strong interfacial interactions resulted between the PZ-imine and the epoxy matrix. The uniform dispersion of PZ-imine in the epoxy matrix also influences the thermal and dielectric properties of the resulting PZ-imine reinforced epoxy reinforced nanocomposites.

Dielectric constant

The values of dielectric constant and dielectric loss of varying weight percentages of PZ-imine reinforced epoxy nanocomposites are presented in Table.1 and Figures 8 & 9. The dielectric constant for neat epoxy, 1, 3, 5 and 7 wt % PZ-imine reinforced epoxy nanocomposites are 3.42, 3.10, 2.93, 2.75 and 2.41 respectively and dielectric loss of neat epoxy, 1, 3, 5 and 7 wt% PZ-imine reinforced epoxy nanocomposites are 0.25, 0.20, 0.18, 0.14 and 0.12 at 1 MHz respectively. When the frequency increases with a decrease in orientation of polarization and its dipole moments needs a longer time than the electronic and ionic polarization, this results in a decrease in the dielectric constant. In this connection, the introduction of PZ-imine into the epoxy contributes to the reduction in the values of dielectric constant and dielectric loss. The bulky group of PZ-imine was effectively restricts the orientation and relaxation of dipoles in the modified epoxy resins. The PZ groups tend to decrease the inter-phase interaction between the inorganic reinforcement and organic domain which strongly reduce the dielectric constant of the resulting nanocomposites [22, 23]. The reduction in the value of dielectric constant occurs due to

an enhanced free volume in certain extent by the influencing effect of PZ. The uniform dispersion of PZ-imine into the matrix was clearly observed from TEM images, in turn contributes to low k values of resulting PZ-imine reinforced epoxy nanocomposites.

From the Figure 9 the dielectric loss of the PZ-imine nanocomposites, this decreases with an increasing weight percentage of PZ-imine in epoxy matrix. The value of dielectric loss of materials is generally contributed from three distinct factors, such as direct current conduction, space charge migration and the movement of molecular dipoles. The lowered value of dielectric loss can be explained as polymer reinforced with PZ-imine, resulting in an insulating layer outside of the dielectric cores to control the migration and accumulation of the space charges within the nanocomposites.

Optical Properties

Among the various UV bands, the wave length range of 280-315 nm (UV-B) band and 315-400 nm (UV-A) are used to investigate the optical property of PZ-imine reinforced epoxy nanocomposites [24, 25]. Due to absorption of ozone layer in the upper atmosphere, (below 280nm) UV-C was filtered. From the Figure 10 it was inferred that the UV absorption behaviour of PZ-imine reinforced epoxy nanocomposites is increased with increasing weight percentages of PZ-imine. The UV absorption spectrum indicates the strong absorbance of the UV region, due to the presence of PZ-imine has a strong influencing effect on the absorption behaviour. Schiff base possesses the photochromic properties due to its cis-trans isomerization and intermolecular proton transfer which enhanced the absorption behaviour [26, 27]. The transmittance spectra of Figure 11. indicates the increasing shielding efficiency (82, 83, 84, 84 and 87 %) of PZ-imine reinforced epoxy nanocomposites in the mid of UVA region, whereas the neat epoxy matrix exhibits about 67% of shielding tendency.

CONCLUSION

PZ-imine reinforced epoxy nanocomposites were developed and their molecular structure was confirmed by FTIR and NMR spectroscopy. Thermal behaviour of the epoxy matrix was enhanced according to the incorporation of weight percentage of PZ-imine. The data

obtained from HRTEM images ascertain the uniform dispersion of PZ-imine in the epoxy nanocomposites. The SEM images of PZ-imine reinforced nano composites shows the efficient UV shielding behavior of PZ-imine reinforced nanocomposites. The 7 wt % PZ-imine reinforced epoxy nanocomposites found to possess the higher UV shielding effect and lower value of dielectric constant than those of other samples of nanocomposites developed in the present work.

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