ISSN: 2349 - 4891
International
Journal of Recent Research and Applied Studies
(Multidisciplinary Open Access Refereed e-Journal)

Modification of Epoxy coatings by conducting polymers for improved corrosion protection of Mild Steel

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Abstract
The use of conducting polymers is a very promising technique to improve the corrosion resistance of metals. In this study polyaniline (Pani) is used to improve the corrosion resistance of epoxy coatings Pani coated chitin and Pani coated silica were prepared by the insitu polymerisation of aniline in presence of silica and chitin respectively. Then they were used to prepare nanocomposites based on epoxy resin**.** The nanomaterials were incorporated in epoxy resin by ultrasonication process. Flexible epoxy coatings thus prepared were applied on mild steel. The coated samples were prepared by dipping process and the samples were used to study corrosion protection. The analysis was performed by electrochemical impedance spectroscopy in 3.5% NaCl solution. The structural characterisation of the resin, nanofillers and the nanocomposite coatings were done by IR spectroscopy, TEM and XRD. The mechanical properties were measured using UTM. The studies show that PANI coated chitin and PANI coated silica can significantly improve the corrosion resistance of epoxy coating.

KEYWORDS; Epoxy resin, Mild steel, Corrosion, PANI coated chitin, PANI coated silica.

Introduction

Corrosion is a natural process that has been a problem for human beings ever since the use of metals. Hence, efforts to develop more efficient and environment friendly methods to improve corrosion resistance have been an active area of research. Commonly used methods to reduce the rate of corrosion are cathodic protection, anodic protection and coatings[1]. Conducting polymers like polyaniline are new materials which have been studied extensively during the past two decades[2]. Conducting polymers used as modified electrodes find an application in biosensing, electroanalytical and drug delivery device. These materials possess electrical conductivity, electronic and optical properties such as metals while retaining processability and mechanical properties usually associated with conventional insulating polymers. Among conducting polymers, polyaniline is a potential material for various commercial applications and has been studied more than other conducting polymers because of its high electrical conductivity, environmental stability, ease of preparation in large quantities and low cost [3]. One of the most important applications of polyaniline is in the protection of metals against corrosion. The polymer works through the anodic protection route and significantly reduces the corrosion rate of metals. It is demonstrated that polyaniline is a good candidate as anticorrosive coating to replace chromium containing coating which have adverse health and environmental issues [4]. Polyaniline has been shown to protect metals against corrosion through its catalytic ability to form an oxide layer which can act as a passivation layer. Earlier study by DeBerry [5] confirmed that stainless steel, in presence of polyaniline, was passivated considerably under highly acidic conditions. Several strategies have been used to increase the effectiveness of polyaniline as anticorrosive coating on metals[6]. Layered materials eliminates the penetration of aggressive species and increase the barrier effect of polyaniline coating. In this study a novel method of coating polyaniline on two inert materials chitin and silica are proposed to be investigated.

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Experimental
Materials and methods

Epoxy resin used was ARALDITE AY 103 (Huntsman Advanced Materials, UK). The resin was cured using

Aradur® 76 Hardener. Chitin was supplied by India sea foods and silica was prepared from rice husk ash. Aniline and other chemicals used were AR grade

Preparation of Pani coated chitin and Pani coated silica

About 2 ml of aniline was mixed with 80 ml of 1N HCl and shaken vigorously using a magnetic stirrer for 30 minutes. 5 gm of chitin/silica powder was added to it and stirred again for 15 minutes. Then 120 ml of an acidic solution of ammonium persulphate solution (0.33 gm/mol) was added drop wise and stirred continuously for about 4 hrs. The precipitate obtained was filtered, washed with water and acetone and then dried in hot air oven at 50-60 ˚C and powdered.

The characterization of silica, chitin, Pani coated silica, Pani coated chitin and their composites with epoxy were done by TEM, FTIR and XRD.

Preparation of composites

Epoxy resin was taken in a beaker. 0.1% wt, 0.5% wt and 1% wt of the required nanofiller was taken in three separate beakers. Acetone was added to the beaker with the required amount of nanofiller to form a suspension. This solution was then poured into the beaker containing the required amount of epoxy resin. The mixture was mechanically stirred for a sufficient period of time. The stirred mixture was sonicated for 20 minutes. The hardener was mixed with the resin in the ratio 1:1.3. The mixture were cast in pre-shaped mould and cured for 48 hrs at ambient temperature. The samples for tensile testing were punched from the mould.

Corrosion study

Mild steel samples of the required size (2 cm × 1 cm) weighing approximately 2.5 gm were prepared. The samples were washed in dil. hydrochloric acid and dried to remove the acid. The sample size required for testing is a square size (1 cm × 1 cm). The metal pieces were then dipped in the mixture to get a uniform coating and then the coated samples were cured for 48 hrs at ambient temperature. The sample was then dipped in 3.5 wt% NaCl solution and kept it for further study.

The corrosion resistance of the coated samples was determined by an electrochemical technique (open circuit potential) in 3.5 wt% NaCl solutions at ambient temperature. The protective properties of such epoxy coatings on mild steel were evaluated using open circuit potential and electrochemical impedance spectroscopy (EIS). The open circuit potential (OCP) was measured over time using a saturated calomel electrode as the reference electrode, a platinum electrode as counter electrode using an electrochemical analyser

Results and Discussion

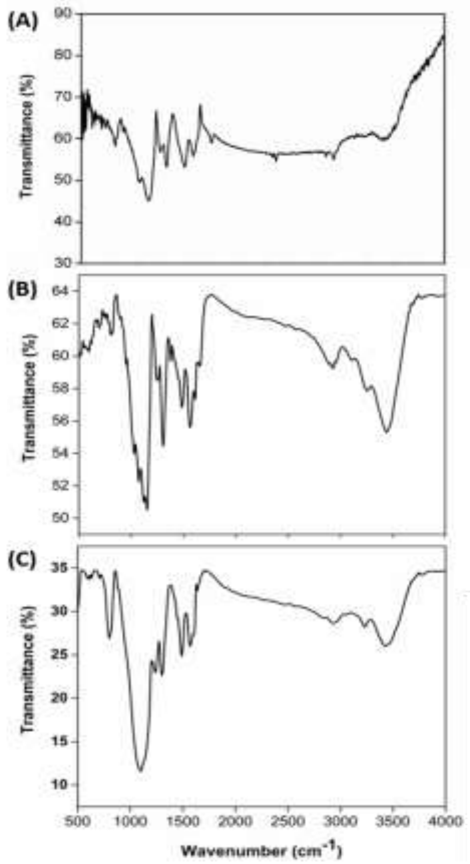

Figure 1. FTIR spectra of PANI (A), PANI coated Chitin (B), PANI coated Silica (C).

Pani coated chitin and Pani coated silica spectra show the characteristic peaks of silica and chitin (Figure 1).

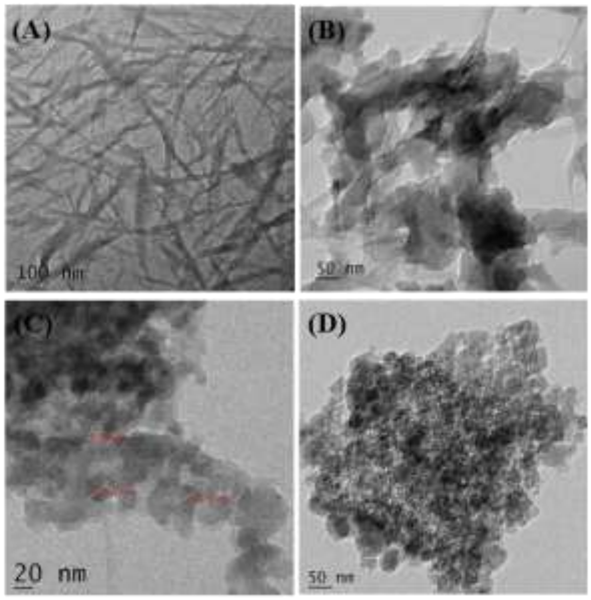

Figure 2. TEM image of chitin (A), PANI coated chitin (B), silica (C) and PANI coated silica(D).

From the TEM images (Figure 2) it is clear that there is no change in the structure of chitin and silica during in situ polymerisation of aniline. Polyaniline (PANI) gets coated over the surfaces of both silica and chitin without affecting its basic shape and structure.

Figs 3, 4 and 5 show the XRD of Epoxy resin, composites of epoxy resin with Pani coated chitin and composites of epoxy resin with Pani coated silica.

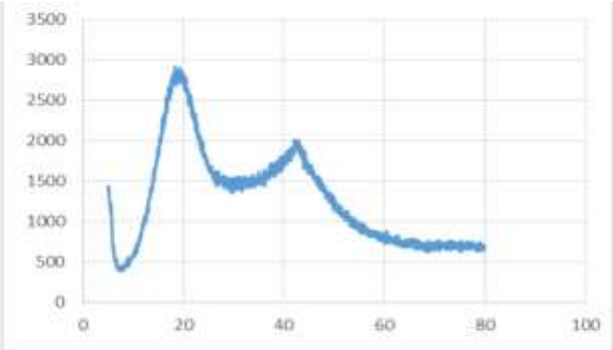

Figure 3. XRD pattern of epoxy resin

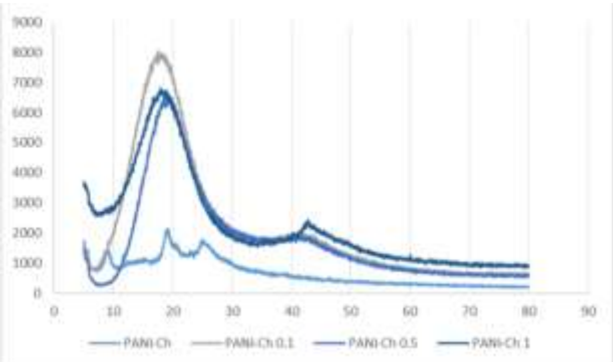

Figure 4. XRD pattern of PANI coated chitin and its composites with epoxy resin

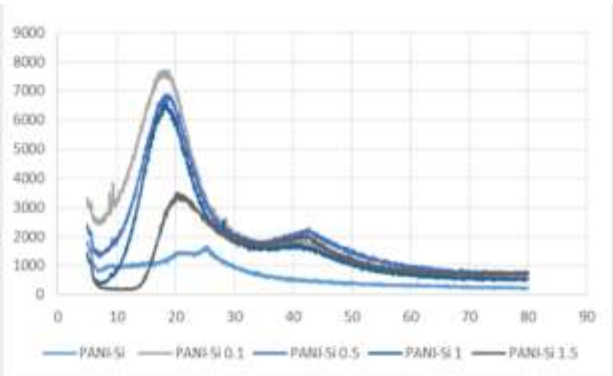

Figure 5. XRD pattern of PANI coated silica and its composites with epoxy resin

Fig 3 shows the XRD pattern of the cured epoxy resin. It shows two defined peaks at 2θ =18.405^{o} and the second peak at 42.35^{o} . Fig 4 shows the XRD pattern of Pani coated chitin and epoxy composites with Pani coated chitin. In the case of Pani coated chitin and epoxy composites similar peaks are observed. This suggests that coating of PANI does not cover the surface of the chitin fully.

Fig 5 shows XRD pattern of Pani- coated silica which reveals that the strong crystalline peak at 2θ =25.14^{o} corresponding to (200) plane of emeraldine salt form of polyaniline is merged with the amorphous broad peak of nano silica at around 2θ = 20.232^{o}. The neat epoxy resin registeres intense amorphous peaks at 2θ =18.405^{o} and 42.35^{o}, which is well pronounced in the XRD patterns of all Pani-Si/epoxy composites. The peaks corresponding to Pani-Si filler is merged with amorphous peaks of epoxy in the composites. It is well defined with increasing filler concentration, the peaks are well intensified in composites containing 1.5 wt% of silica.

The tensile properties of the epoxy composites are given in Table 1

Table 1. Tensile properties of the composites

Sample	Tensile strength N/mm2	Elongation at break %
Cured epoxy resin	1.10	98
Epoxy resin composite 0.1% Pani coated silica	1.20	85
Epoxy resin composite 0.5% Pani coated silica	1.41	106
Epoxy resin composite 1.0 % Pani coated silica	1.26	86
Epoxy resin composite 1.5 % Pani coated silica	1.13	95
Epoxy resin composite 0.1% Pani coated chitin	1.20	104
Epoxy resin composite 0.5% Pani coated chitin	1.5	98
Epoxy resin composite 1.0% Pani coated chitin	2.03	92
Epoxy resin composite 1.5% Pani coated chitin	1.72	95

Figs 6 and 7 show the thermo-gravimetric analysis of the composite containing 1% PANI coated chitin and silica respectively. As expected the composite containing silica shows higher thermal stability than that of the composite containing chitin.

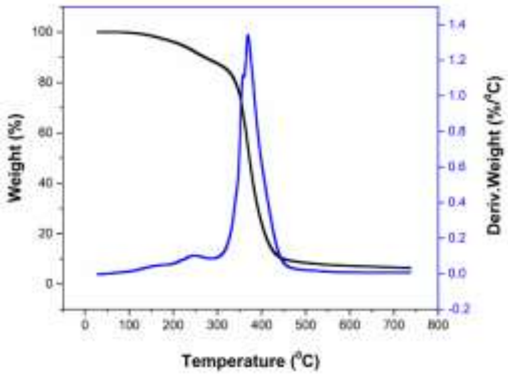

Figure 6. TGA of Epoxy resin with 1% PANI coated chitin

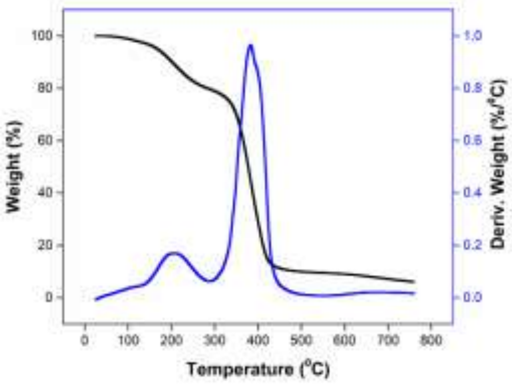

Figure 7. TGA of Epoxy resin with 1% PANI coated silica

Figs 8, 9 and 10 show the Tafel plots of the composite containing 0.1%, 0.5% and 1% chitin respectively and figs 11, 12 and 13 show the Tafel plots of the composite containing 0.1%, 0.5% and 1% silica respectively.

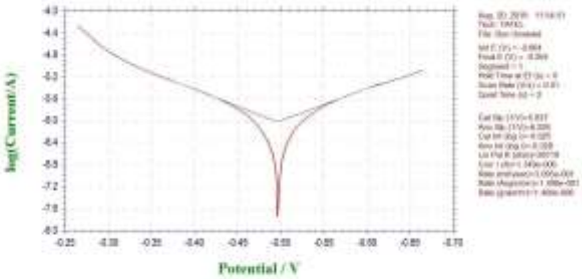

Figure 8. Tafel plot for epoxy composite with 0.1% PANI coated chitin

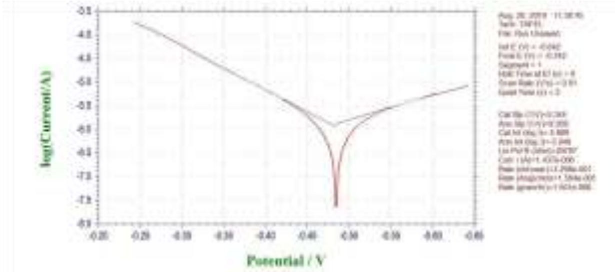

Figure 9. Tafel plot for epoxy composite with 0.5% PANI coated chitin

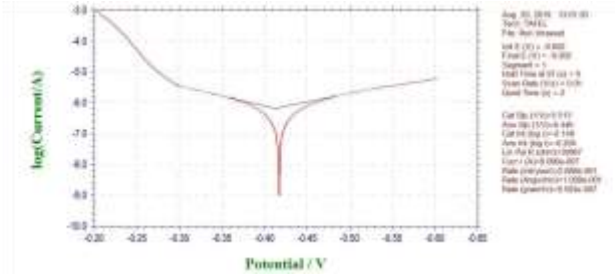

Figure 10. Tafel plot for epoxy composite with 1.0% PANI coated chitin.

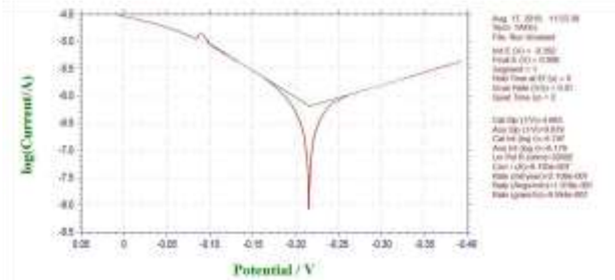

Figure 11. Tafel plot for epoxy composite with 0.1% PANI coated silica

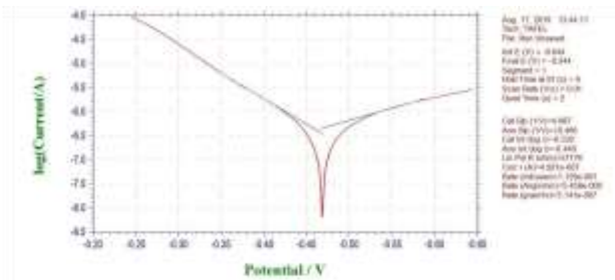

Figure 12. Tafel plot for epoxy composite with 0.5% PANI coated silica

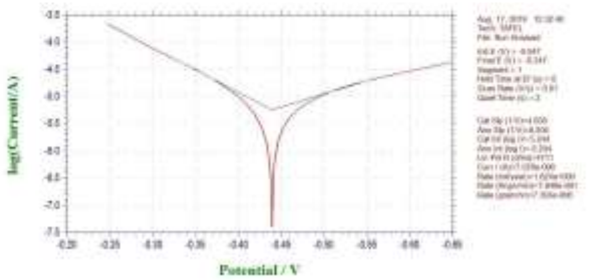

Figure 13. Tafel plot for epoxy composite with 1.0% PANI coated silica

It can be observed that the composite containing 1% chitin shows maximum corrosion protection while the composite containing 0.5% silica gives the maximum corrosion protection.

CONCLUSION

Pani coated chitin and Pani coated silica can significantly improve the corrosion resistance of epoxy coating over mild steel. There is an optimum concentration of the nanofiller which gives maximum corrosion protection. Coating Pani over chitin and silica permits the optimum concentration to be reached at very low levels. The tensile strength and thermal stability of the epoxy coating marginally improve with the addition of the nanofillers.

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Please cite this article as: DR.D.THIRUMALA RAO (2021). A Study on “Reverse Innovation” – A Rising Phenomenon from Indian Perspective . International Journal of Recent Research and Applied Studies, 8- 4(19), 122-126