

Evaluation of Mechanical Properties and Corrosion Protection Performance of Surface Modified Nano-alumina Encapsulated Epoxy Coated Mild Steel

G. Boomadevi Janaki¹ · Joseph Raj Xavier¹

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Abstract

The incorporation of imidazole-modified nano-alumina on the corrosion protection properties of epoxy coating on mild steel was studied by electrochemical impedance spectroscopy (EIS) and scanning electrochemical microscopy (SECM) in 3.5% NaCl solution. The dispersability of the alumina nanoparticles was greatly improved by the surface modification using imidazole in the epoxy matrix. The imidazole-modified nanoparticle was analyzed by Fourier transform infrared spectroscopy. Both EIS and SECM studies confirmed that the corrosion resistance is higher for the alumina–epoxy nanocomposite coated steel than that of the pure epoxy-coated steel. It is evidenced that alumina–epoxy nanocomposite coated mild steel has higher charge transfer resistance values, 156,344 Ω cm², compared to pure epoxy coated mild steel, 79,546 Ω cm², at 40 days of immersion. The modified nanoparticles enhanced the adhesive properties of the coatings. Possible chemical interactions between epoxy matrix and surface-modified alumina nanoparticles in nanocomposites cause high protection properties and ionic resistances. FE-SEM/EDX analysis showed the presence of Fe, Al, and O in the corrosion products. Hardness and tensile strength measurements showed that the improved mechanical properties were noticed for alumina–epoxy nanocomposite coated mild steel.

Keywords Nano-Al₂O₃ · Surface modification · Imidazole · Corrosion · EIS · Mild steel

1 Introduction

The metal corrosion is one of the vital issues in the protection of steel structures from the corrosive environments. The protection of metal structures from environmental and corrosion attacks was carried out using epoxy coatings because of its very good toughness, adhesion to metal substrates, and durability [1–4]. However, the epoxy coatings are susceptible to perforation by water and corrosive ions and might experience degradation during long-term usage [5, 6]. Extensive research is being conducted on polymer composites reinforced with rigid nanoparticles. Epoxy resins have excellent material properties, but they are, however, generally brittle materials. Therefore, to fully exploit the

Joseph Raj Xavier drjosephrajxavier@gmail.com properties, epoxy polymers often need to be reinforced by the incorporation of nanoparticles. Generally, inorganic particles may improve the stiffness and other properties of the materials [7]. A range of inorganic nanoparticles may be used as reinforcements, including carbon nanotubes [8, 9], nanofibers [10], clay [11], and silica [12, 13]. The inorganic fillers can modify the stiffness, strength, and toughness of polymers. In general, the mechanical properties of polymer composites depend primarily on the particle size, the particle/matrix interface adhesion, and the particle loading. The stiffness depends significantly only on particle loading while the particle/matrix adhesion particularly affects the strength and toughness. The main difference between using nanosized fillers, compared to microsized fillers, is the much higher specific surface area of the nanomaterials. The evaluation of alumina-reinforced composite coated mild steel has also been reported [14, 15].

Corrosion inhibition efficiency increases with compounds possessing hetero atoms like N, O, and S due to the characteristics of adsorbed film on a metal surface. The inhibitory activity of imidazole is significant due to its well-known

¹ Department of Chemistry, Vel Tech Rangarajan Dr. Sagunthala R&D Institute of Science and Technology, Avadi, Chennai, Tamil Nadu 600 062, India

planar, heterocyclic, aromatic, and 5-membered organic compounds with two nitrogen atoms in their structure. The nitrogen atom plays predominant role as inhibitor by forming coordinate bond with empty orbital of the metal [16]. When adsorbed on metal surfaces, molecules act as a protective barrier film against corrosive agents. Therefore, the anticorrosion performance of metals is greatly enhanced by the incorporation of nanoparticles with epoxy matrix [17–20]. The anticorrosion performance and mechanical properties of the epoxy coatings were also improved by the incorporation of metal oxide nanoparticles [21–25].

Our aim is to investigate the anticorrosion behavior of the surface-modified nano-alumina (nano-Al₂O₃)-incorporated epoxy nanocomposite-coated surface in 3.5 wt% NaCl solution. Surface-modified alumina with imidazole increases the dispersion of alumina in epoxy matrix. Fourier transform infrared spectroscopy (FT-IR) was used to analyze the modified alumina. The corrosion resistance of the coated surface was investigated by electrochemical impedance spectroscopy (EIS) and scanning electrochemical microscopy (SECM). The adhesive property of the coated steel was studied by adhesion strength test. The surface morphology and elemental composition of epoxy–alumina nanocomposite coated substrates were studied by FE-SEM with EDX techniques. Mechanical properties of nanocomposite coated surface are analyzed by hardness and tensile strength tests.

2 Experimental

2.1 Substrate Preparation

Mild steel was used as substrate for coating alumina along with organic moieties of imidazole. Mild steel of $10 \times 10 \times 3$ mm size was abraded using silicon carbide papers of different grit size (120 to 1000). Soap solution, water, and acetone were used to wash the mild steel after polishing. Then, degreasing was done by ultrasonication for 20 min using acetone. After rinsing them with distilled water, the sample was dried in the oven and kept in the vacuum desiccator.

2.2 Preparation of Epoxy Composite Coatings

The equivalent molar concentrations of imidazole and alumina were dissolved in 25 ml of anhydrous ethanol to form a homogeneous solution and kept in the ultrasonicator for 30 min. Then, the mixture was stirred at 78 °C for 1 h. The resultant product was washed with a minimum quantity of water, followed by ethanol. The resulting sample was dried in a vacuum oven at 60 °C for 12 h to obtain the product. The epoxy resin was mixed with imidazole-modified alumina (2 wt%) and hardening agent with a blending ratio of

3:1, followed by stirring for 2 h at 3000 rpm to get homogeneous and stable nanocomposite. The coating has been done by drawdown process.

2.3 Characterization Techniques

The surface modification was analyzed by FT-IR Perkin Elmer spectrum II series instrument using KBr pellet in the range of 4000–400 cm⁻¹ wavenumber with a resolution of 4 cm⁻¹. Electrochemical measurements were done by EIS and polarization studies (Biologic SP-240 instrument; ECLab V10.37 software) for the coated samples immersed in 1, 5, 10, and 40 days. Three electrode systems were used at 35 ± 1 °C. A saturated calomel electrode (SCE), platinum, and coated samples acted as reference electrode, counter electrode, and working electrode, respectively. The frequency was varied from 100 kHz to 0.01 Hz at 10 points per decade.

SECM measurements were carried out at the tip potential of +0.6 V vs. Ag/AgCl/KCl to analyze imidazole-modified alumina–epoxy nanocomposite- and pure epoxy-coated samples in 3.5% NaCl solution. Platinum microelectrode (diameter 20 µm) acted as the ultramicroelectrode (UME) tip. Ag/AgCl and Pt strips were employed as reference electrode and counter electrode, respectively. A micro-flat cell was used to mount the coated sample horizontally, thus baring the coated side upwards to test solution. SECM scans were done by rastering UME over the coated surface by applying the tip potential of +0.60 V. At this potential, the presence of ferrous ion can be detected through their oxidation to ferric ions.

The surface morphological studies of scratched area of imidazole-modified alumina–epoxy nanocomposite coatings were analyzed by FE-SEM/EDX technique (Model CARL ZEISS SUPRA 55, Germany) for 1 day and 40 days. The crystalline nature of imidazole-modified alumina–epoxy nanocomposite-coated scratched surface in 3.5% NaCl for 1 day and 40 days was analyzed with X-ray diffractometer (XRD; Bruker model D8, Germany) using Cu K α radiation λ -1.5406 Å. The 2 θ angles were swept from 10° to 70° in steps of one degree.

The adhesion strength of the coatings to the substrate was calculated before and after immersion in 3.5% NaCl solution by pull-off measurements on Electronic Universal Material Testing Machine (Instron Corporation, USA). The testing was done according to ASTM D3359 standard. The tensile test was performed in the universal testing machine at a crosshead speed of 10 mm/min. The measurement of microhardness was carried out using HM113 Vickers hardness tester. The right-angle pyramid with a square-base diamond indentor angle of 136° between opposites faces compressed the composite specimen under a load of F = 20 N for a loading time of 15 s. Six number of indentation was made on

each sample to get the mean value of the hardness. Figure 1 displays the flow chart of the evaluation of mechanical properties and corrosion protection performance of surface-modified nano-alumina-encapsulated epoxy coated mild steel.

3 Result and Discussion

3.1 FT-IR Spectroscopy

The FT-IR spectra of imidazole, pure Al₂O₃ and modified Al₂O₃ are shown in Fig. 2 The characteristic absorption peaks of pure imidazole are as follows: N-H stretching frequency at 3413 cm⁻¹, C–N stretching at 1463 cm⁻¹, C=N at 1615 cm⁻¹, and C-H stretching vibrations in the region of 3040–3101 cm^{-1} . The bands between 1400 cm^{-1} and 1650 cm⁻¹ were assigned to C-C stretching vibrations. The broad absorption band in the region of 523 to 764 cm^{-1} was attributed to the Al-O-Al group of nano-Al₂O₃ and the peak at 3400 cm^{-1} corresponded to O–H groups. It can be clearly seen that there was a significant increase of N-H, C-N, and C=N band intensities for the surfacemodified coatings. The bands at 3451 cm^{-1} , 2927 cm^{-1} , and 2843 cm⁻¹ corresponded to N-H stretching frequency. The bands at 1475 cm⁻¹ and 1641 cm⁻¹ were attributed to C–N and C=N, respectively. The polymer backbone (epoxy C–O–C) showed bond at 1023 cm⁻¹. The Al–O–Al group is due to the broad band in between 555 cm^{-1} and 734 cm^{-1} [26]. These changes indicated that the nano-Al₂O₃ had been successfully modified by the addition of imidazole. Those absorption peaks did not appear on the corresponding region in the pure Al_2O_3 .

3.2 Polarization Studies

Figure 3 depicts the potentiodynamic polarization curves for both epoxy- and alumina–epoxy nanocomposite-coated



Fig. 2 FTIR spectra obtained for imidazole, alumina nanoparticle and Al_2O_3 nanoparticle-imidazole

samples in 3.5% NaCl solution for various days of immersion. The electrochemical parameters (corrosion potential $E_{\rm corr}$ and corrosion current density $I_{\rm corr}$) derived from polarization curves after stabilization are presented in Table 1. The shifting of $E_{\rm cor}$ in the noble positive direction observed with lower $I_{\rm corr}$ suggests that the remarkable increase in the corrosion resistance of the epoxy coated mild steel by the incorporation of imidazole-modified alumina nanoparticle.

It is shown that higher $E_{\rm corr}$ and lower $I_{\rm corr}$ values were obtained in 1 day of immersion and lower $E_{\rm corr}$ and higher $I_{\rm corr}$ values were obtained in 40 days of immersion for both imidazole-modified alumina–epoxy nanocomposite and pure epoxy coated samples. The $E_{\rm corr}$ and $I_{\rm corr}$ values of imidazole-modified alumina–epoxy nanocomposite-coated sample immersed for 1 day are – 185 V and 0.17 μ A/cm², whereas the $E_{\rm corr}$ and $I_{\rm corr}$ values of pure epoxy-coated sample immersed for 1 day are – 345 V and 1.24 μ A/cm². On the other hand, the $E_{\rm corr}$ and $I_{\rm corr}$ values of epoxy–alumina

Fig. 1 Flow chart of the evaluation of mechanical properties and corrosion protection performance of surface modified nano-alumina encapsulated epoxy coated mild steel





Fig. 3 Potentiodynamic curves of **a** alumina–epoxy nanocomposite and **b** pure epoxy coated mild steel immersed in 3.5% NaCl solution for 1, 10, 20, and 40 days

Table 1	Polarization	data for	coated	steel

Coating	Immersion time (day)	<i>E</i> _{Corr} (mv) vs. SCE	$I_{\rm Corr}$ (µA cm ²)	$R_{\rm P} ({\rm k}\Omega~{\rm cm}^2)$
	1	- 345	1.24	6.89
Epoxy	10	-424	1.95	5.92
	20	-492	2.16	4.63
	40	-743	2.86	2.79
	1	-185	0.17	10.25
Epoxy-	10	-246	0.69	8.95
nano- alumina	20	-354	1.25	7.32
	40	-490	1.83	5.24

nanocomposite coated sample immersed for 40 days are -490 V and 1.83 μ A/cm². The E_{corr} and I_{corr} values of pure epoxy-coated sample immersed for 40 days are -743 V and 2.86 μ A/cm². The higher values of corrosion potentials and lower values of corrosion current for imidazole-modified

alumina–epoxy nanocomposite coatings than pure epoxy coatings indicate the better bonding due to the incorporation of imidazole formation along with epoxy on the metal surface. Thus, imidazole-modified alumina–epoxy nanocomposite-coated sample showed maximum corrosion resistant compared to pure epoxy-coated sample.

3.3 Electrochemical Impedance Spectroscopy Test

EIS measurements were carried out to evaluate the corrosion protection performance of the coated samples. Figure 4 displays the Nyquist plots of imidazole-modified alumina–epoxy nanocomposite- and pure epoxy-coated steel in 3.5% NaCl solution for various immersion days. Figures 5 and 6 show Bode plots for imidazole modified alumina–epoxy nanocomposite and pure epoxy coated mild steel, respectively. It is observed that imidazole-modified alumina–epoxy nanocomposite coated steel shows maximum



Fig. 4 Nyquist plots obtained for **a** alumina–epoxy nanocomposite and **b** pure epoxy coated mild steel immersed in 3.5% NaCl solution for 1, 10, 20, and 40 days



Fig. 5 Bode plots obtained for modified alumina–epoxy nanocomposite coated mild steel immersed in 3.5% NaCl solution for 1, 10, 20, and 40 days

impedance and maximum phase angle shift from low-frequency to high-frequency region compared to pure epoxycoated sample. The imidazole-modified alumina–epoxy nanocomposite coated mild steel has higher charge transfer resistance compared to pure epoxy coated mild steel [27–29].

Moreover, the electrical equivalent circuit was used to fit and simulate two times constant impedance data as shown in Fig. 7. In the circuit model, R_s is the solution resistance, R_{coat} indicates coating resistance at a constant phase element (C_{coat}), and R_{ct} indicates charge transfer resistance in the coating/substrate interface paralleled with C_{dl} for the coating/substrate interface and polarization resistance. Table 2 displays the data of capacitance (C_d) and resistance (R_c) for the epoxy and epoxy nanocomposite coating as a function of immersion time. It is shown that imidazole modified alumina–epoxy nanocomposite- and pure epoxy-coated samples possess n values from 0.79 to 0.85 and from 0.62 to 0.75, respectively.



Fig. 6 Bode plots obtained for pure epoxy coated mild steel immersed in 3.5% NaCl solution for 1, 10, 20, and 40 days



Fig. 7 Equivalent electrochemical circuit for epoxy and imidazolemodified alumina-epoxy coated mild steel

Table 2EIS data of the coatedsample

Coating	Immer-	$R_{\rm S} \Omega {\rm cm}^2$	$R_{\rm f}\Omega{\rm cm}^2$	$Q_{\rm f} ({\rm Fcm^{-2}s^n})$	$R_{\rm ct} \Omega{\rm cm}^2$	$C_{\rm dl} ({\rm F}{\rm cm}^{-2}{\rm s}^{\rm n})$	$ Z \Omega \ cm^2$	n _{coat}
	sion (day)							
Ероху	1	28.1	75,280	3.581×10^{-5}	111,802	8.112×10^{-5}	103,318	0.79
	10	30.6	70,356	5.893×10^{-4}	96,587	9.311×10^{-4}	64,782	0.80
	20	31.8	55,822	8.298×10^{-3}	84,116	11.24×10^{-3}	36,836	0.82
	40	34.2	38,240	4.276×10^{-2}	79,546	6.895×10^{-2}	30,319	0.85
	1	11.2	195,112	7.241×10^{-10}	208,564	8.564×10^{-11}	67,694	0.62
Epoxy– nano- alumina	10	15.6	187,210	4.110×10^{-10}	195,620	3.465×10^{-11}	57,401	0.69
	20	17.8	154,234	9.253×10^{-8}	170,281	6.289×10^{-9}	46,322	0.73
	40	19.3	138,451	6.132×10^{-7}	156,344	4.321×10^{-8}	34,865	0.75

With increasing test time, the film resistance is decreased from 195,112 Ω cm² at 1 day to 138,451 Ω cm² at 40 days for imidazole modified alumina-epoxy nanocomposite coating and from 75,280 Ω cm² at 1 day to 38,240 Ω cm² at 40 days for pure epoxy coating. Charge transfer resistance (R_{ct}) is the resistance against the process of electron transfer from electrode to the electrolyte. The higher charge transfer resistance was obtained at 208,564 Ω cm² for 1 day and decreased to 156,344 Ω cm² at 40 days for alumina-epoxy nanocomposite-coated sample. However, $R_{\rm ct}$ value was found to be 111,802 Ω cm² at 1 day and lowered to 79,546 Ω cm² at 40 days for pure epoxy. The high porous nature of pure epoxy system leads to increased corrosion rate. The dissolution of corrosion products at higher test time and subsequently higher diffusion of the corrosive ions in the coating cause the reduction of corrosion resistance.

3.4 SECM

Figures 8 and 9 show the SECM measurement for imidazole-modified alumina-epoxy nanocomposite and pure epoxy-coated samples in 3.5% NaCl solution for immersion in different days at +0.60 V. The scratched metal surface in all samples of pure epoxy coatings showed increased current at +0.60 V. It was due to the possible oxidation of Fe^{2+} to Fe^{3+} . The current distribution on the coated surface (scratched area) increases with increasing immersion time. Figure 10 depicts SECM line analysis for scratched imidazole modified alumina-epoxy nanocomposite and pure epoxy coated mild steel immersed in 3.5% NaCl solution for 1, 10, 20, and 40 days at + 0.60 V. In pure epoxy system, there is a drastic increase in the current from 2.0 Å at 1 day to 10.0 Å at 40 days. This is because the epoxy coatings are porous in nature. In case of modified system, the current is increased from 0.5 Å at 1 day to 2.5 Å at 40 days. It is indicated that the anodic dissolution of Fe²⁺ is much lower

Fig. 8 SECM 2D images obtained for scratched alumina– epoxy nanocomposite coated mild steel immersed in 3.5% NaCl solution for 1, 10, 20, and 40 days at tip potential + 0.60 V vs. Ag/AgCl/KCl reference electrode



Fig. 9 SECM 2D images obtained scratched pure epoxy coated mild steel immersed in 3.5% NaCl solution for 1, 10, 20, and 40 days at tip potential +0.60 V vs. Ag/AgCl/KCl reference electrode



for epoxy–alumina coating when compared with pure epoxy coating. This could be because of the active participation of nitrogen donor atoms of imidazole moiety that inhibits the formation of Fe^{3+} [30].

3.5 SEM/EDX Analysis

Figure 11 displays the SEM images of nano-alumina grafted epoxy nanocomposite coated substrates in 3.5% of NaCl for 1 day and 40 days. It is shown for a 1-day immersion that the surface modified alumina-epoxy nanocomposite possesses uniform coatings due to the homogeneous distribution on its surface. The better compatibility between its components leads to crack free coating with the substrates. It was observed at 40 days of immersion that no severe cracking was noticed on the substrate because of surface modification using imidazole moiety. The presence of pore structures leads to greater exposure of the substrate metal to oxidation and results in the formation of corrosion products. The incorporation of imidazole-modified alumina moiety hinders the possible interaction of chloride ions with metal substrate and results in a low rate of corrosion. It is concluded that no agglomeration takes place because of the homogeneous distribution of 2 wt% Al₂O₃ nanoparticles. Figure 12 displays elemental analysis using EDX of epoxy-alumina nanocomposite coated substrate. It is seen that there is no change in weight percentage of C, O, and Al but significant changes in the concentration of Fe. This is due to the degradation of mild steel substrate.

3.6 X-Ray Diffraction (XRD) Analysis

Figure 13 shows the XRD pattern obtained for the epoxy/ alumina/imidazole nanocomposites immersed in 3.5% NaCl for 1 and 40 days, respectively. XRD analysis for 1 day and 40 days immersion of epoxy/alumina/imidazole nanocomposites showed formation of corrosion products on the metal surface. The corrosion product of the upper surface was α -FeOOH and that of the lower surface was γ -FeOOH. However, on prolonged immersion in NaCl, y-FeOOH could be converted to Fe₃O₄. This indicates that complete corrosion takes place on the metal surface. It is also observed that the intensity of these iron peaks (γ -FeOOH) is raised in 40 days of immersion. These findings confirm that there is a corrosion formation both in 1 day and in 40 days of immersion, but higher corrosion product formation in 40 days compared to 1 day of immersion leads to the slowing down of the corrosion process. In 40 days, we could see the enhanced peaks obtained for γ -FeOOH and small peak obtained for Fe_3O_4 , which reveals that the corrosion process occurs with controlled process along with the epoxy/alumina/imidazole nanocomposite coatings on mild steel.

3.7 Mechanical Properties

Figure 14 displays the bonding strength of pure epoxy coating and epoxy-alumina nanocomposite coating with mild steel system. The bonding strength of pure epoxy and epoxy-alumina nanocomposite coating at 1 day of



Fig. 10 SECM line analysis obtained **a** alumina–epoxy nanocomposite and **b** pure epoxy coated mild steel immersed in 3.5% NaCl solution for 1, 10, 20, and 40 days at tip potential +0.60 V vs. Ag/AgCl/ KCl reference electrode

immersion was found to be around 5 MPa and 8 MPa, respectively. In the same way, at 40 days of immersion, it was observed nearly 1.5 MPa for pure epoxy coated sample and 4.5 MPa for epoxy–alumina nanocomposite coated sample. From these results, it was concluded that better adhesive strength was found even after 40 days of immersion for the epoxy–alumina nanocomposite coating. It is due to the presence of nitrogen donor atoms present in epoxy–alumina nanocomposite coating for that leads to strong protection from degradation [31].

Figure 15 shows the results of hardness test as well as tensile strength test for the pure epoxy- and epoxy-alumina nanocomposite coated samples for different days of



Fig. 11 SEM images of alumina-epoxy nanocomposite coated sample for 1 day and 40 days

immersion in 3.5% NaCl solution. It can be seen from the figure that the hardness value of 80 MPa at 40 days is calculated for pure epoxy-coated samples. However, the hardness value of 620 MPa at 40 days is found for epoxy–alumina nanocomposite coated samples. The higher coating hardness for epoxy–alumina nanocomposite coated samples. The higher coated sample shows lower porosity. Similarly, the tensile strength of epoxy–alumina nanocomposite coated sample was found to be 70 MPa at 40 days but pure epoxy-coated sample showed 30 MPa at 40 days. Therefore, the results of mechanical testing confirm that the addition of imidazole-modified alumina to the epoxy matrix improves the adhesion, hardness, tensile strength, and anticorrosion properties of the coated sample.

4 Conclusion

The dispersability of nano-alumina in the epoxy matrix has been enhanced by the surface modification of nanoalumina with imidazole. The resultant alumina–epoxy



Fig. 12 EDX analysis of alumina-epoxy nanocomposite-coated sample for a 1 day and b 40 days





Fig. 13 XRD analysis of alumina–epoxy nanocomposite coated sample for $a\ 1$ day and $b\ 40$ days in 3.5% NaCl solution

Fig. 14 Bar graph of adhesion strength of alumina–epoxy nanocomposite- and pure epoxy coated mild steel before immersion and after immersion in 3.5% NaCl solution for 1, 10, 20, and 40 days



Fig. 15 Bar graph of hardness and tensile strength of alumina–epoxy nanocomposite and pure epoxy coated mild steel before immersion and after immersion in 3.5% NaCl solution for 1, 10, 20, and 40 days

nanocomposite coating on mild steel has been investigated by SECM and EIS techniques in 3.5% NaCl.

- The enhanced anticorrosion performance was displayed by the modified nano-alumina–epoxy coating compared to pure epoxy coating. This could be attributed to the surface modification of nano-alumina by imidazole to speed up the possible chemical interactions between nano-alumina and epoxy matrix.
- EIS studies showed higher charge and film resistance for the epoxy-alumina nanocomposite coated steel.
- SECM studies showed lower current at the scratched surface of the epoxy-alumina nanocomposite-coated steel in comparison with pure epoxy-coated steel sample at the scratched surface.
- FE-SEM/EDX analysis confirmed the presence of Fe, Al, and O in the corrosion products.
- The modified nano-alumina–epoxy coated sample shows good adhesive strength, improved hardness, and tensile strength and better corrosion resistance ability than a pure epoxy coating.

4.1 Future Work

Future work concerns the coating of different nanocomposites on mild steel in the industrial applications and deeper analysis of corrosion mechanisms using X-ray Photoelectron Studies (XPS).

Compliance with Ethical Standards

Conflict of interest The authors declare that there is no conflict of interest.

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