



A study on the anticorrosion performance of epoxy nanocomposite coatings containing epoxy-silane treated nano-silica on mild steel substrate



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ABSTRACT

Anticorrosion performance of a clear epoxy coating was enhanced by the incorporation of modified nano-silica into the polymer matrix. The surface of nano-silica was treated with 3-Glycidioxypropyl-trimethoxysilane (GPTMS) in order to achieve proper dispersion of nanoparticles. The surface grafting of GPTMS on the nanoparticles was detected using fourier transform infrared spectroscopy (FTIR) and dispersion stability test. Corrosion performance of the coated mild steel specimens was investigated employing electrochemical impedance spectroscopy (EIS) and salt spray test. Incorporation of 4–6 wt% SiO₂ nanoparticles possessed the best corrosion performance. The electrochemical results are completely in agreement with the morphological results of the surface obtained from scanning electron microscopy (SEM).

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Introduction

Among the metallic structural materials, steel is by far the most common and most valuable for industries. Mild steel is frequently used in different industries because of its mechanical properties and machine-ability at a low price. The mild steel will corrode in the atmospheric condition causing the consuming of material resources [1–3]. Organic and metallic coatings, using inhibitors and cathodic protection are usually employed to protect steel constructions against corrosion damages [3]. Isolating of the metal surface by organic coatings from corrosive media is still the most conventional method to prevent corrosion [4]. For this purpose, polymeric coatings can provide protection either by a barrier action of the coating or by the aid of active corrosion inhibition supplied by pigments in the coatings [5]. However, in practice, all organic coatings are permeable to corrosive species such as oxygen, water and ions to some extent [6–8].

In the last few years, study of the performance of nanocomposite coatings has been in the center of attention of coating scientists due to their beneficial properties [9–11]. Various nanoparticles have, therefore, been employed as reinforcements

in order to improve coatings' performance in the corrosive environments including, TiO₂ [12], ZnO [13], ZrO₂ [2,14], CeO₂ [15], CaCO₃ [16], Fe₂O₃ [17] and SiO₂ [18,19].

Nano-silica is a multi-purpose nanoparticle, utilized to produce multifunctional nanocomposite coatings. It possesses high hardness (7 Mohs), low refractive index (1.46), and reasonable price among other nanoparticles that make it ideal to produce scratch-resistant, transparent and cost-effective coatings [19–21]. Furthermore, silica is an inorganic UV-absorber, exhibiting more stability than the usual organic UV-absorbers or light stabilizers. Additionally, nano-silica particles do not cause/accelerate polymer degradation compared to UV-absorbent TiO₂ nanoparticles [22,23]. It is worth to note that the nano-silica particles have OH functionalities, making a hydrophilic nature to their surface [20].

Surface treatment of silica nanoparticles with organosilanes is commonly recommended in order to make a compatible nature of the nano-silica particles with non-polar or weakly polar matrices. Organic chains of the organosilanes increase the miscibility of silica nanoparticles with the organic matrices [24–26]. The influence of surface treatment on the electrochemical behaviors of the nanocomposite coatings has been studied using electrochemical impedance spectroscopy (EIS) [27,28]. EIS has been one of the most successful technique in the field of anticorrosion characterization of organic coatings [29,30]. This technique with

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providing quantitative data was extremely used to rank the efficiency of different nano anticorrosive pigments in organic coatings [31]. Dolatzadeh et al. [19] reported the modification of the corrosion properties of a two-pack polyurethane coating using various organosilane-treated SiO₂ nanoparticles. Furthermore, Behzadnasab et al. [2] demonstrated that the silane-modified zircon nanoparticles could significantly improve the anticorrosion properties of the epoxy coatings.

Nano-silica-based nanocomposites were extensively utilized to improve scratch and abrasion resistance of organic coatings [20]. Investigation of the quantitative (EIS) as well as qualitative (salt spray) anticorrosion performance of the epoxy resin, as an organic coating primer, containing treated nano-silica is the main target of this research.

In this work, a new anticorrosion organic coating formulation was investigated based on silane-treated nano-silica/epoxy resin. In this way, an epoxy-based organosilane namely 3-Glycidoxypropyltrimethoxysilane (GPTMS) was used to modify the surface properties of nano-silica. In the next step, the corrosion performance of treated silica-epoxy nanocomposite on mild steel was studied in 3.5 wt% NaCl solution. Moreover, a high-resolution SEM technique was employed to study the dispersion properties of the nanoparticles on the coating surface.

Experimental

Materials

St-37 type mild steel sheets (with dimensions of 10 × 8 × 0.2 cm³) were prepared from Mobarake Steel Co. with the following composition: C ≤ 0.1%, Mn ≤ 0.4%, P ≤ 0.04%, Fe remainder. The mill scale layer on the surface of mild steel sheets was carefully removed using a magnetic polisher (at 2000 rpm) and followed by an acetone degreasing to remove organic contaminants.

A conventional bisphenol-A type epoxy resin (Epikote 828) and polyamide hardener (Epikure 3175) from shell chemicals were used as an organic matrix for dispersion of nano-silica. The dry film thickness (DFT) of the coated samples were 60 ± 3 μm and the coatings were applied on the metal surface using a wet film applicator and cured for 30 min at 120 °C. Hydrophilic fumed Nano-silica (AEROSIL® 200) with an average primary particle size of 12 nm and with a specific area of 200 m²/g were purchased from EVONIK industries. 3-Glycidoxypropyltrimethoxysilane (GPTMS) (Fig. 1) was purchased from Merck Chemicals and used without further purification.

Surface modification of silica nanoparticles:

Firstly, 10 g of the silica nanoparticles dispersed in 50 ml ethanol (Merck Chemicals) via stirring at 800 rpm for 1 h at ambient temperature. The mixture was homogenized in an Ultraturrax homogenizer (IKA Labortechnik) for 30 min. In the next step, 12 ml of GPTMS and 3.8 ml of deionized water was

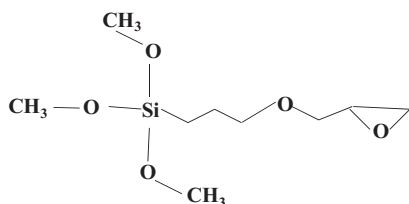


Fig. 1. The chemical structure of 3-Glycidoxypropyltrimethoxysilane (GPTMS) used to modify the nano-silica surface.

gradually added to the dispersion and stirred for further 2 h in ambient temperature. In order to have a maximum efficiency of the silane hydrolysis, pH value of this step was adjusted to 2 using hydrochloric acid [32]. The amounts of silica and silane were calculated according to a stoichiometric value (unity) as follows [32]:

$$M = \frac{3 \times S_{\text{Si}} \times m_{\text{Si}} \times M_{\text{Silane}} \times \text{OH.No} \times 10^{19}}{N_A} \quad (1)$$

where M is the amount of silane (in grams), S_{Si} is the surface area of silica, m_{Si} is the weight of silica, and M_{Silane} is the molecular weight of silane. OH.No is the number of hydroxyl groups per nm² on silica surface, N_A is the Avogadro number and 10^{19} is a conversion factor. It should be noted that the reaction was performed at hydrolysis ratio of 3 (the ratio of water to silane).

The mixture was then stirred and heated in reflux condition for 48 h at pH 2 [32,33]. Finally, it was centrifuged (4000 rpm) and the residue washed with ethanol and deionized water. The washing procedure was repeated for two times and the remained precipitate was dried in an oven at 60 °C for 4 h. The obtained dry powder was mixed in xylene solvent for 1 h and then was homogenized for 30 min to make a pre-dispersed nano-silica. Fig. 2 shows the schematic reaction of the surface nano-silica particles and GPTMS.

Preparation of epoxy nanocomposite coatings

The pre-dispersed nano-silica was added slowly and gradually to the epoxy resin and stirred with a mechanical stirrer (1200 rpm) for 2 h at ambient temperature and degassed for further 3 h to remove remained trapped air during the mixing steps. 2, 4, 6 and 8 wt% nano-silica in the epoxy-hardener film was prepared using the mentioned method. In order to make a suitable coating properties, an appropriate concentration of leveling agent (BYK 306) and defoamer (EFKA 2022) were used in the coatings' formulation. The stoichiometric ratio of the hardener to the resin was adjusted in 1.1 in order to obtain an appropriate efficiency of the curing reaction. Finally, the prepared nanocomposite coatings were applied on the mild steel surface and were cured in a conventional oven.

Dispersion stability test

Dispersion stability of nanoparticles (untreated and treated), was evaluated in water and xylene as an inorganic (polar) and organic (non-polar) solvents, respectively. Dispersions of 1 wt% nanoparticles in water and xylene were stirred for 1 h and then homogenized for further 30 min followed by sonication for 30 min. The dispersion was then allowed to stand for 1 day and sedimentation behavior of nanoparticles was visually evaluated.

Characterization

The grafting of GPTMS on the silica nanoparticles was evaluated by FTIR. FTIR spectra of the nanoparticles and GPTMS in KBr pellets were recorded in transmission mode on a Bruker IFS 88 FTIR spectrometer, collecting 35 scans in the 400–4000 cm⁻¹ range with 4 cm⁻¹ resolution.

The corrosion properties of the organic coated samples were studied by electrochemical impedance spectroscopy (EIS) in 3.5 wt% NaCl solution up to 60 days (AUTOLAB PGSTAT12). The test was carried out at open circuit potential (OCP) within frequency range of 10 mHz to 10 kHz (with the perturbation of ±10 mV). Frequency response analysis (FRA) software was used to analyze the information obtained from EIS measurements. EIS tests were carried out in a three-electrode electrochemical cell including reference

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