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Coating Performance of SiO_2/ Epoxy Composites as a Corrosion Protector

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To solve the corrosion problem of industrial equipment and other constructions containing metals, corrosion protection can be performed by using coating which provides a barrier between the metal and its environment. Coatings play a significant role in protecting irons and steels in harsh marine and acid environments. This study was conducted to identify an anti-corrosive epoxy coating for carbon steel composite with 0.1, 0.3, and 0.5 wt% concentrations of nanoparticles of SiO_2 using the dip-coating method. The electrochemical behavior was analyzed with open circuit potential (OCP) technics and polarization curves (Tafle) in 3.5 wt% NaCl and 5 vol% H_2SO_4 media. The structure, composition, and morphology were characterized using different analytical techniques such as X-ray Diffraction (XRD), Fourier Transform Infrared spectrum (FT-IR), and Scanning Electron Microscopy (SEM). Results revealed that epoxy-nano SiO_2 coating demonstrated a lower corrosion rate of 2.51×10^{-4} mm/year and the efficiency of corrosion protection was as high as 99.77%. The electrochemical measurement showed that the nano-SiO_2 / epoxy coating enhanced the anti-corrosive performance in both NaCl and H_2SO_4 media.

Keywords: *Nano silica oxide, Surface modification, Corrosion protection, Metal coating, Anti-corrosion*

1. Introduction

Corrosion, in general, indicates the effect of a chemical or electrochemical reaction that takes place between metals and the surrounding medium. This action leads to the material's decomposition or destruction [1]. Corrosion is an irreversible process that occurs when metals are used. By producing high-performance metal materials and equipment, the corrosion problem of metal materials and equipment can be solved, pollution-free, and economic anti-corrosive coatings [2]. Because of their simplicity and effectiveness, corrosion resistance coatings have gotten a lot of attention [3].

Various corrosion coatings have been improved and tested to combat the damaging effects of corrosion on metal. Because different types of metals and alloys have different physical and chemical qualities, the protection afforded by a coating varies depending on the type of metal and the environment to which it is exposed [3,4]. The basic concept of coating is to keep oxygen and moisture from getting to the metal [5]. The organic coatings have excellent resistance to effectiveness, aesthetic properties, and substrate adhesion [6].

Organic coatings are widely employed to protect metallic structures against corrosion since they are simple to apply and inexpensive [7]. Material selection is an effective technique for dealing with severe corrosion, such as novel materials such as composites, nano-composites, and nano-particles. [2]. Since practically all engineering materials (composite, alloys, metals, polymers, and ceramics) can be employed as a reinforcement coating on material surfaces, anti-corrosive coatings are used in a variety of industries [8]. Organic polymeric coatings, which are commonly shown as paints, are one of the most widely used ways of protecting against metallic corrosion [9].

As protective coatings, organic compounds such as epoxy, polymethyl methacrylate (PMMA), polyurethane (PU), polyesters, fluoropolymers, and related paints, in combination with an anti-corrosive primer containing various types of pigments, are widely used [10]. Because of its outstanding toughness, adherence to metal substrates, and durability, epoxy coatings have been widely utilized to protect metal structures against environmental and corrosion attacks [11,12].

Yongxing Zhang *et al.* [13] investigated the corrosion

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resistance performance of epoxy composite coatings with silicon nitride to protect Q235 carbon steel in 3.5 wt% NaCl by electrochemical impedance spectroscopy (EIS), the result showed that modified silicon nitride coating exhibited good anti-corrosive performance. Hongli Cheng *et al.* [14] studied the synthesis and the characterization of poly (O-Ethoxyaniline)/ nano-silica (POEA/SiO_2) composite material by in-suite polymerization method which prepared on the surface of the treated carbon steel, also studied its anti-corrosive performance in 3.5 wt% NaCl solution via Tafel polarization curve and electrochemical impedance spectroscopy (EIS). The results revealed that coatings comprising POEA/SiO_2 have a decreased corrosion rate of 0.02 mm/year and a corrosion protection efficacy of 98 %. The addition of POEA/SiO_2 increases the epoxy coating's anti-corrosive properties.

Xianming Shi *et al.* [15] represented that the effect of anti-corrosive of epoxy coating containing nanoparticles of SiO_2, Zn, Fe_2O_3, and halloysite clay on steel in both 0.3 wt% and 3.5 wt% NaCl solutions over 28 days immersion. Potentiodynamic polarization and electrochemical impedance spectroscopy (EIS) were used to study the effect of combining different nanoparticles on the corrosion resistance of epoxy-coated steel. The result observed that the use of nanoparticles increased the corrosion resistance of coated steel, with the best nanoparticles being Fe_2O_3 and halloysite clay. The SiO_2 nanoparticles improved the anti-corrosive performance and microstructure of the coating matrix. M. Behzadnasab *et al.* [16] investigated the corrosion performance of mild steel coated with epoxy-containing nano-clay, and amino ProplyTrimethoxy silane (APS) treated zirconia nanoparticles using electrochemical impedance spectroscopy (EIS) technique. The results revealed that adding spherical ZrO_2 and layered clay nanoparticles at the same time causes clay nanoparticle exfoliation and improves the corrosion performance of nanocomposite coatings by improving barrier characteristics and ohmic resistance.

Wenhue *et al.* [17] and Bedaiwi *et al.* [18] reported that the modified coating of nano-ZrO_2 incorporated into phenolic-epoxy resin protects steel components in the acid industrial environment using electrochemical methods. The results revealed adding 1 wt% and 3 wt% nano-ZrO_2 to the coatings improved their corrosion resistance, however, adding 5 wt% nano-ZrO_2 decreased it.

In the present work, different concentrations of nanoparticles silica oxide SiO_2 composite with epoxy resin will be prepared and coated carbon steel using the dip-coating method. The anti-corrosive properties of the epoxy-nano silica oxide film will be investigated in two media (NaCl 3.5 wt% and 5 vol% of H_2SO_4 solution) for measuring the corrosion resistance and adhesion property on carbon steel. Different analytical techniques include Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectrum (FTIR), X-ray Diffraction (XRD) which are applied to identify the modified nanoparticles -silica oxide.

2. Materials and Methods

2.1 Materials

Epoxy resin purchased from (Sekadur 52, Egypt), SiO_2 nanoparticles (55-75 nm) amorphous particles purchased from (Nanografi, Germany) and ethanol alcohol 96 % supplied by (Scharlau, Spain), NaCl and H_2SO_4 supplied by (Merck, Germany).

2.2 Preparation of Epoxy/SiO_2 Nanocomposite

Epoxy resin-SiO_2 nanoparticles composite was prepared by mixing 50 mL of epoxy resin with 25 mL of hardener and 0.05 g SiO_2 nanoparticles (55-75 nm) and by manually mixing for 5 minutes to get different concentrations of SiO_2 (0.1 wt%, 0.3 wt%, and 0.5 wt%) respectively.

2.3 Characterization Techniques

Structure and phase analysis of the composite materials were performed using X-Ray Diffraction (XRD), (6000 Shimadzu, Japan). The spectrum of the Fourier-Transform Infrared (FTIR) was recorded with a Bruker and Scanning Electron Microscope (SEM) test (Tescan VEGA 3SB).

2.4 The Coating's Preparation

The carbon steel specimens with the size of (20 × 20 × 3 mm) are prepared in the following procedure smoothed by SiC abrasive paper from 180 to 500 grades, cleaned twice by ethanol alcohol solution, immersed in an ultrasonic bath for 20 minutes, then dried by air at room temperature before the coating process.

The dip-coating method was carried out by immersing

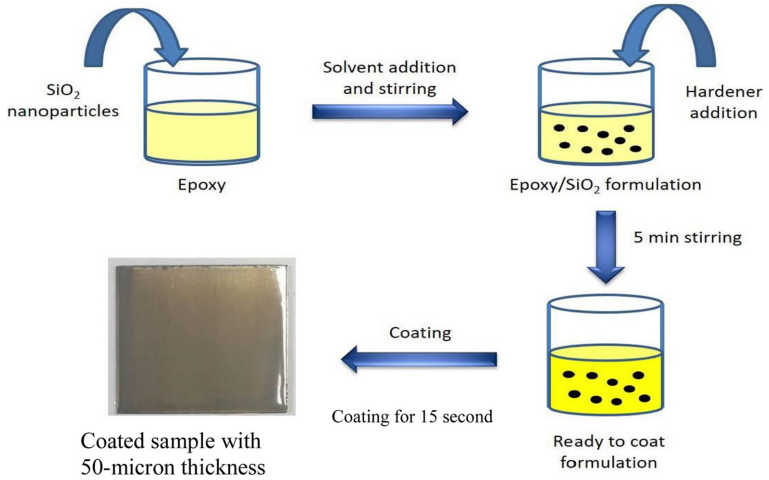


Fig. 1. The schematic preparation of an SiO_2/epoxy composite coating on a carbon steel substrate

carbon steel specimens vertically for 15 seconds in epoxy resin of 0.1wt% SiO_2 composite formulation to get a film layer of coating about 50 microns in thickness. Then the specimens were dried under atmospheric air for 24 hrs. The same procedure was repeated using 0.3 wt% and 0.5 wt% concentrations of SiO_2 nanoparticles. Fig. (1) depicts the preparation of an epoxy/SiO_2 composite coating on the surface of carbon steel in a schematic diagram.

2.5 Evaluation of Coating

The performance of anti-corrosive epoxy coating on the surface of the carbon steel specimens tested. Using the Tafel polarization curves and Open circuit potential (OCP) for the coated specimens after being immersed in 3.5 wt% NaCl, and 0.5 vol% H_2SO_4 solutions for a while in (25 ºC) laboratory ambient temperature using the potentiostat (Parstat 2273, USA). A flat cell with platinum was the counter electrode, and (Ag/AgCl/KCl) as a reference electrode with coated carbon steel specimen was the working electrode connected to the potentiostat.

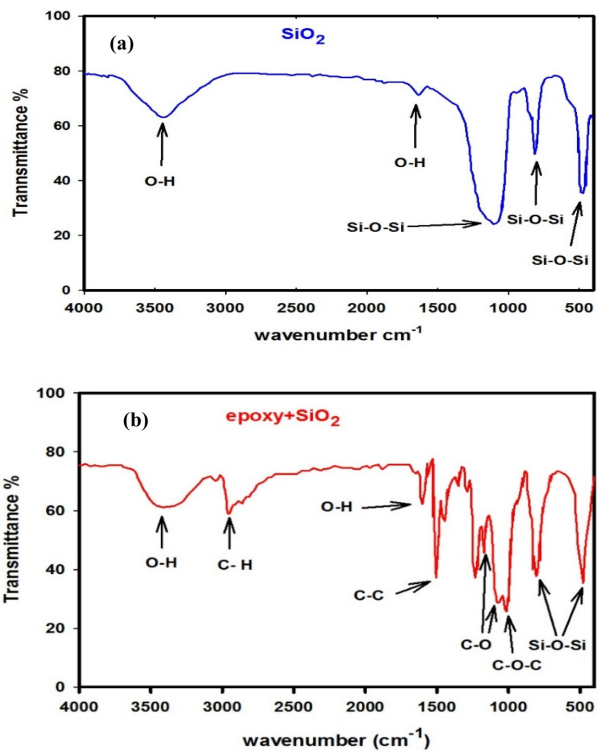


Fig. 2. FTIR spectrum for (a) SiO_2 nanoparticles epoxy (b) composite SiO_2

3. Results and Discussion

3.1 Structure and morphology

The FTIR analysis was used to identify the chemical bonds of organic, polymer, and inorganic material using infrared transmitted through the carbon steel surface. The FTIR spectrum for SiO_2 nanoparticles in Fig. 2a shows a peak of 3452 cm^{-1} is the structure of water O–H antisymmetric stretching vibration. Peak near 1638 cm^{-1} is the O–H bending vibration peak of water. The Si–O–Si antisymmetric stretching vibration has a strong and extensive absorption at 1081, 815, and 472 cm^{-1}.

Fig. 2b shows the FTIR spectrum for the epoxy-SiO_2 composite layer in a peak of O–H stretches at 3422 cm^{-1}. The symmetric stretching of C–H of the oxirane ring is shown at the peak at 2972 cm^{-1}. The peak at 1608 cm^{-1}

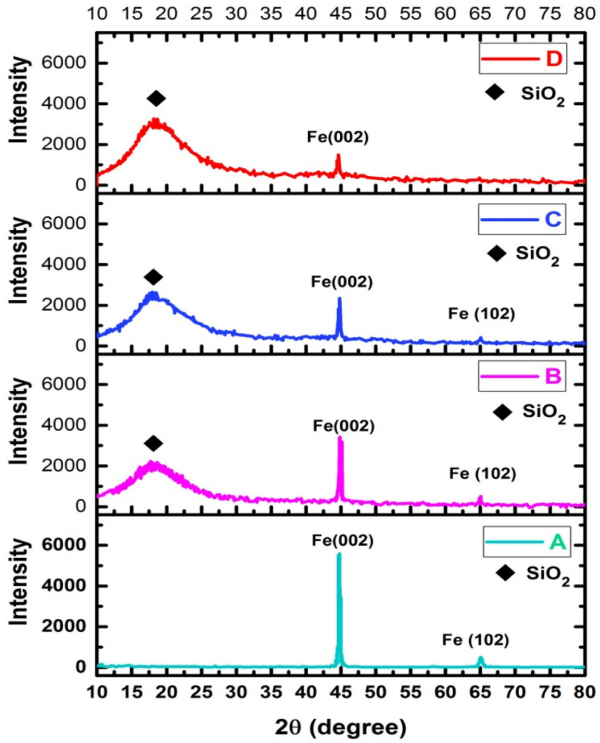


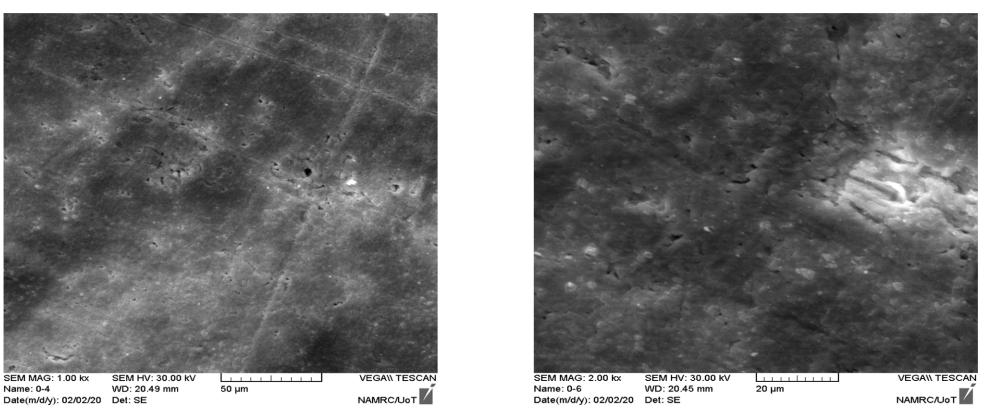
Fig. 3. XRD patterns for carbon steel coating with different concentrations of SiO_2/epoxy nanoparticles: (a) zero SiO_2, (b) 0.1 wt% SiO_2, (c) 0.3 wt% SiO_2 and (d) 0.5 wt% SiO_2

was due to O–H bending, then the peak of stretching aromatic ring C–C was observed at 1517 cm^{-1}, while the peaks at 1182 and 1097 cm^{-1} were due to stretching aromatic ring C-O. Finally, the peak at 1021 cm^{-1} was due to stretching ether group C–O–C. Also, Si–O–Si peaks were observed at 808 and 465 cm^{-1}.

Fig. 3. shows the X-ray patterns for different concentrations of amorphous SiO_2 nanoparticles. It can be seen from Fig. 3a Fe peaks appeared obviously at 2θ (49.5 and 64.7) while there is no obvious diffraction peak of SiO_2 nanoparticles, the observed 2θ value is consistent with the standard International Center for Diffraction Data (ICDD) in the values (ICDD No. 34-0529). The XRD patterns of epoxy/SiO_2 have a diffraction peak intensity that appears and is observed at $2\theta = 18.1^{o}$ at 0.1 wt% concentration in Fig. 3b while in Fig. 3a the high intensity of Fe is observed at $2\theta = 45^{o}$ and the other two diffraction peaks intensity appeared clearly with increasing amorphous SiO_2 nanoparticles concentration at 0.3 percent and 0.5 percent respectively, meanwhile the Fe peaks decreased as shown in Fig. 3c and d. This is due to the crystal behavior after modification coating is enhanced and the characteristic diffraction peak of SiO_2 centered on 18.1^{o} (2θ) confirmed its amorphous nature. The crystalline molecules of epoxy are covered on the surface of SiO_2 nanoparticles, which increases the mass-volume percentages of the nanoparticles to produce diffraction conditions, and thus increasing the concentration of amorphous SiO_2 nanoparticles gradually from 0.1, 0.3 and 0.5 wt% improves the surface coating on substrate carbon steel with disappearing of Fe peaks.

3.2 Surface Characterization:

Fig. 4. shows the surface topography of SiO_2/epoxy nanoparticles composite before and after coating. Fig. 4a-b illustrates the substrate surface after grinding also shows some cracks and pores on the surface due to the uncoating



(a) (b)

Fig. 4. SEM images with different magnification for Carbon steel coated with epoxy- SiO_2 at different concentration (a and b): substrate (c and d): 0.1 wt% SiO_2, (e and f): 0.3 wt% SiO_2 and (g and h): 0.5 wt% SiO_2

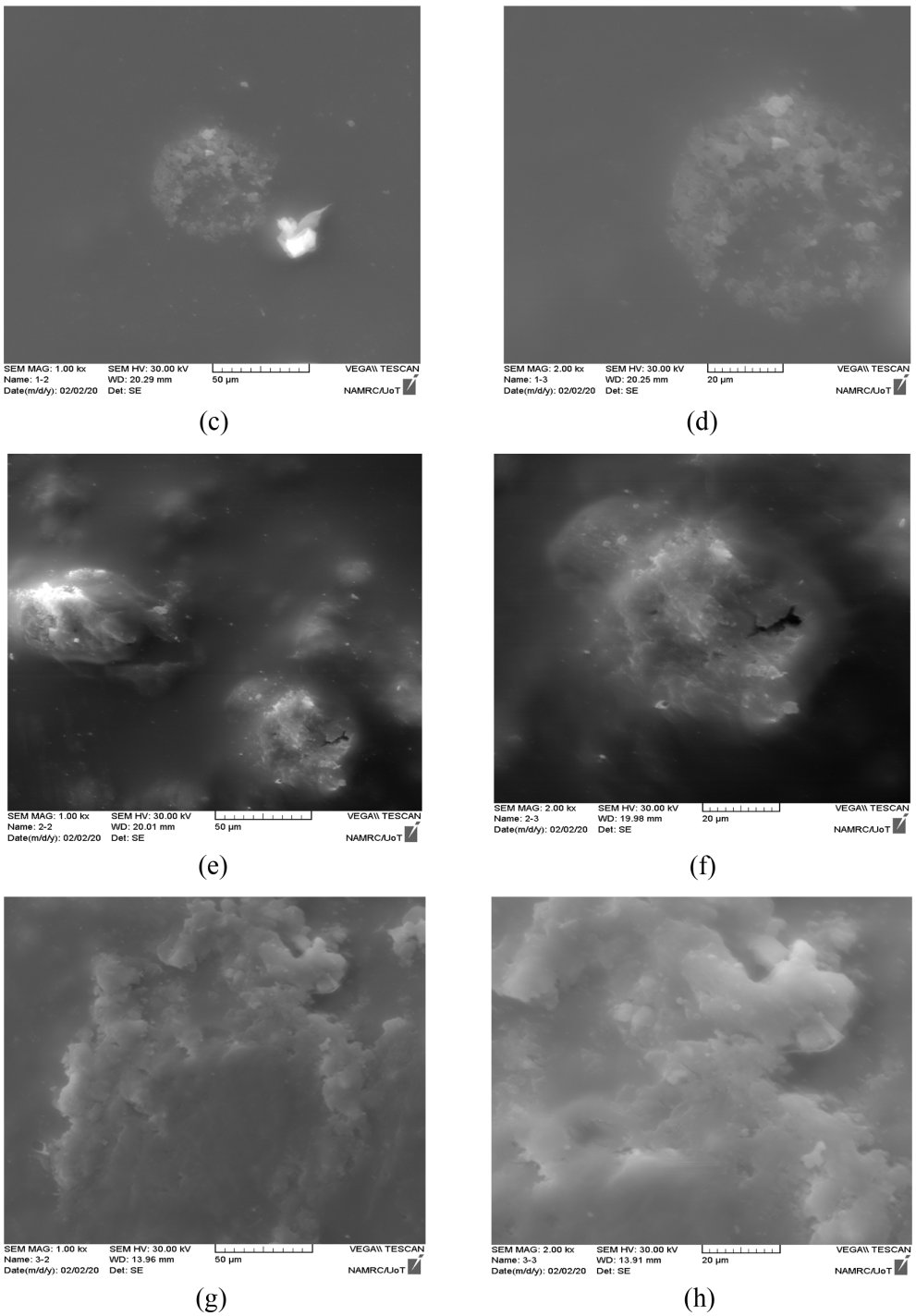


Fig. 4. Continued

substrate. While Fig. 4c-d shows the addition of SiO_2 particles in the concentration of 0.1 wt% began to appear and increased the coating's compactness, although micropores remained on the surface. As shown in Fig. 4e-f, the agglomerate of SiO_2/epoxy with 0.3 wt% began obviously on the surface substrate. While the SiO_2 with 0.5 wt% concentration was more agglomerate and covers the substrate surface as shown in Fig. 4g-h.

3.3 Electrochemical Behavior

Fig. 5 shows the open-circuit potential test for carbon steel specimen tested in NaCl media shows that the

increase in potential (the sample surfaces be more passive begin from -1.216 volt for uncoated one reached to -0.666 volt for the sample coated with 0.5 wt% SiO_2 composite). While Fig. 6. shows the specimen tested in sulfuric acid shows the same behavior begins with -0.962 volt for the uncoated sample and reaches -0.8325 for that coated with 0.5 wt% SiO_2. Both Table 1 and Table 2 show the corrosion data measured from the specimens experimentally immersed in media of 3.5 wt% NaCl and 5 vol% H_2SO_4.

The polarization curves (Tafel) for different specimen tested in NaCl 3.5 wt% media as shown in Fig. 7. The Polarization curve (Tafel) test for Carbon steel coated with epoxy- SiO_2 at different concentration of SiO_2, where curve (a) is a substrate, curve (b) 0.1 wt% SiO_2, curve (c) 0.3 wt% SiO_2 and curve (d) 0.5 wt% SiO_2. The results show a decrease in corrosion current as corrosion rate for all coated specimens compared to the uncoated ones. The corrosion rate is decreased from 1.590×10^{-2} mm/y uncoated to 2.519×10^{-4} mm/y for coated for specimens of 0.5 wt% SiO_2 composite as mentioned earlier in Table (1 and 2). That is represented the corrosion characteristics for specimens tested in 3.5 wt% NaCl media. So, the corrosion rate, reducing 63 times when dividing the corrosion rate for uncoated to the coated specimens at 0.5 wt% SiO_2.

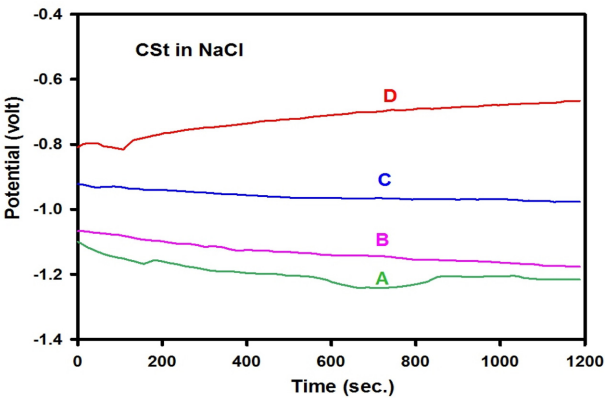


Fig. 5. Open circuit potential vs. immersion time curves for Carbon steel coated with epoxy- SiO_2 with at concentration (a): substrate, (b): 0.1 wt% SiO_2, (c): 0.3 wt% SiO_2 and (d): 0.5 wt% SiO_2 in 3.5 wt% NaCl media

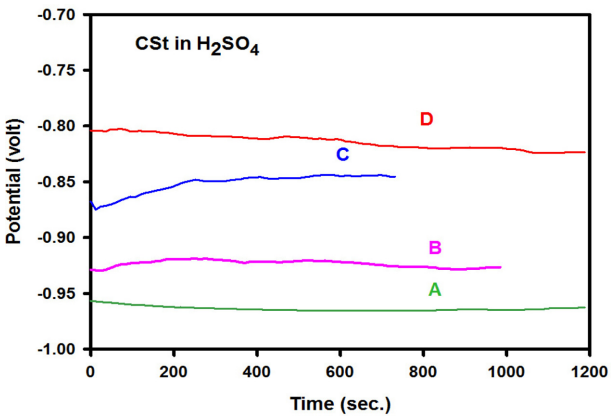


Fig. 6. Open circuit potential vs. immersion time curves for Carbon steel coated with epoxy- SiO_2 at different concentration (a): substrate, (b): 0.1 wt% SiO_2, (c): 0.3 wt% SiO_2 and (d): 0.5 wt% SiO_2 in 5 vol% H_2SO_4 media

Table 1. Corrosion characteristics for the specimens tested in 3.5 wt% NaCl media

b_c mVolt	b_a mVolt	Corr. Rate mm/y	I corr. Amp/cm^2	E corr. Volt	OCP Volt	Item
153	165	1.59×10^{-2}	1.410×10^{-6}	-0.616	-1.216	Base
69	74	8.45×10^{-3}	7.500×10^{-7}	-0.550	-1.176	0.1 wt% SiO_2
109	130	9.68×10^{-4}	8.620×10^{-8}	-0.490	-0.977	0.3 wt% SiO_2
118	121	2.51×10^{-4}	2.240×10^{-8}	-0.587	-0.666	0.5 wt% SiO_2

Table 2. Corrosion characteristics for the specimens tested in 5 vol% H_2SO_4 media

b_c mVolt	b_a mVolt	Corr. Rate mm/y	I corr. Amp/cm^2	E corr. Volt	OCP Volt	Item
135	38	4.169	3.712×10^{-4}	-0.418	-0.963	Base
172	173	1.504×10^{-1}	1.339×10^{-5}	-0.440	-0.926	0.1 wt% SiO_2
92	96	6.846×10^{-2}	6.846×10^{-6}	-0.443	-0.845	0.3 wt% SiO_2
80	86	9.550×10^{-3}	8.503×10^{-7}	-0.431	-0.823	0.5 wt% SiO_2

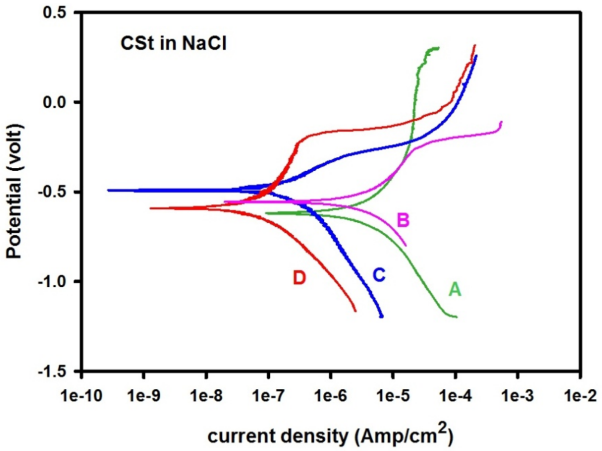


Fig. 7. Potentiodynamic Polarization curves were recorded for Carbon steel coated with epoxy- SiO_2 at different concentrations of SiO_2

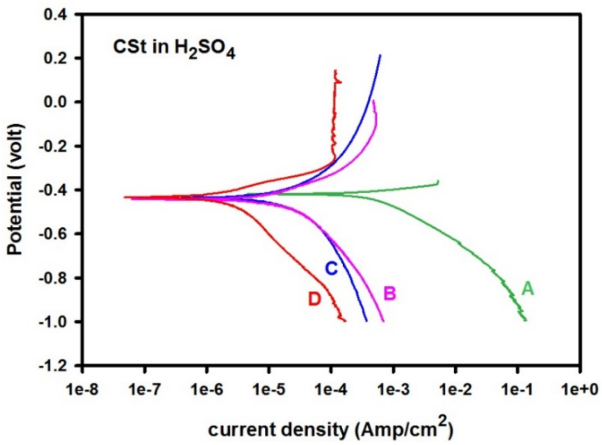


Fig. 8. Potentiodynamic Polarization curves recorded for carbon steel coated with epoxy- SiO_2 at different concentration (a): substrate, (b): 0.1 wt% SiO_2, (c): 03 wt% SiO_2 and (d): 0.5 wt% SiO_2 in 5 vol% H_2SO_4 media

Table 3. Calculated corrosion parameters for specimens tested in 3.5 wt% NaCl media

Porosity %	Rp $K\Omega.cm^2$	W.L. $mg.cm^{-2}.sec^{-1}$	PE%	Item
----------	24.44	1.167	----------	Base
33.83	28.76	0.620	46.8	0.1 wt% SiO_2
1.407	299.2	0.071	93.88	0.3 wt% SiO_2
1.408	1.158×10^3	0.018	98.4	0.5 wt% SiO_2

Table 4. Calculated corrosion parameters for specimens tested in 5 vol% H_2SO_4 media

Porosity %	Rp $K\Omega.cm^2$	W.L. $mg.cm^{-2}.sec^{-1}$	PE%	Item
----------	34.687	305.0	----------	Base
4.705	2796	11.04	96.63	0.1 wt% SiO_2
5.296	2979	5.025	98.15	0.3 wt% SiO_2
0.459	21164	0.701	99.77	0.5 wt% SiO_2

Fig. 8 shows the polarization curve (Tafel) test for specimens tested in 5 vol% H_2SO_4 media. The corrosion rate, reduced with the increase of SiO_2 quantity, is reduced from 4.169 mm/y for uncoated to 9.550×10^{-3} mm/y coated specimens with 0.5 wt% SiO_2 composite are present in Table 3 and 4. This result means reducing corrosion rate 436 times by dividing the corrosion rate for uncoated sample over that coated with 0.5 wt% SiO_2 composite, success for the coating to resistance towards corrosion.

3.4 Corrosion Protection Evaluation

The Protection Efficiencies (PE%) of the present work of coating specimens can be calculated by coating to uncoating corrosion current densities of the specimens equation 1 as follows [19],

$$PE\% = \left[1 - \frac{i_{corr_{coated\ specimen}}}{i_{corr_{uncoated\ specimen}}}\right] \times 100 \quad (1)$$

The Weight Loss (W.L.) can be calculated from the corrosion rate, also called penetration. The corrosion rate unit in the experimental test will be measured by (mm/year), then converted to (gm/cm^2.sec).

The Polarization Resistances R_p of the specimen calculated as [20];

$$R_p = \frac{b_a b_c}{2.303(b_a + b_c) i_{corr}} \quad (2)$$

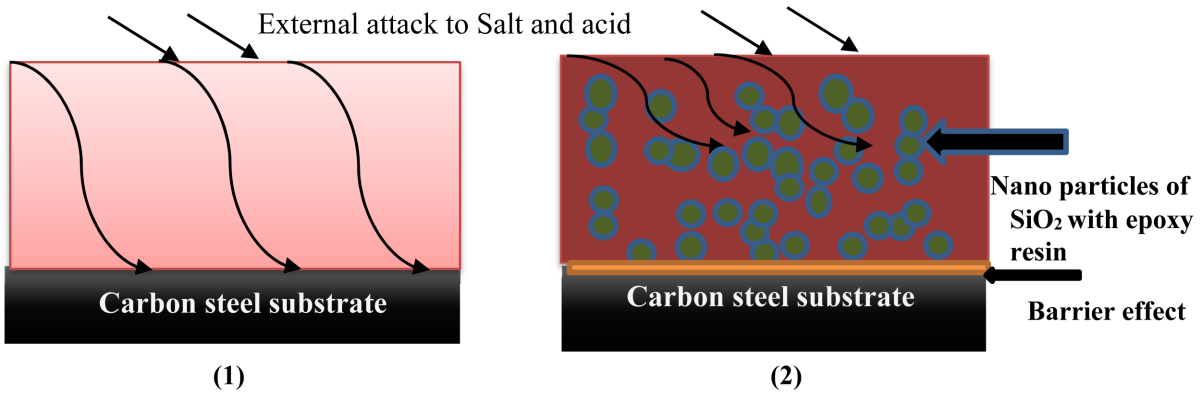


Fig. 9. The suggested mechanism for SiO_2 nanoparticles in reducing corrosion 1: without nanoparticles and 2: with nanoparticles

The Porosity Percentage (*PP%*) for both coating and uncoating will be calculated using the following equation [21]:

$$PP\% = \frac{R_{P_{uncoated}}}{R_{P_{coated}}} 10^{\frac{-\Delta E_{corr}}{b_a}} \times 10 \quad (3)$$

The Corrosion Potential (E) is calculated as;

$$\Delta E_{corr} = E_{uncoated} - E_{coated} \quad (4)$$

and b_a is the anodic Tafel slope of the uncoated and coated specimen.

The results of the protection efficiency (PE), the polarization resistance (Rp), and porosity percentage (PP%) are calculated from equations (1), (2), and (3) respectively, with the weight loss (W.L.) for NaCl and H_2SO_4 media are tabulated in Tables (3) and (4). The protection efficiency for specimens tested in H_2SO_4 media increased with increasing SiO_2 concentration to 99.77% while the protection efficiency for specimens tested in NaCl was 98.4%. This result indicates that corrosion resistance and high protection efficiency for coated specimens at a concentration of 0.5 wt% SiO_2 composite.

From Table 2 the W.L. was decreased from 1.167 mg $m^{-2} d^{-1}$ for uncoated substrate to 0.018 mg $m^{-2} d^{-1}$ for sample coated with 0.5 wt% SiO_2 in NaCl media. From Table 4 the W.L. was decreased also from 305 mg $m^{-2} d^{-1}$ for uncoated substrate to 0.701 mg.m^{-2} d^{-1} for sample coated with 0.5 wt% SiO_2 in H_2SO_4 media. The W.L. was very high in H_2SO_4 media for the sample uncoated, it was approximately 0.3 mg for each square meter every day, and that is not economically loss. The coating decreases the loss to economic range in both media especially in samples coated with 0.5 wt% SiO_2.

Because the nanoparticles filled the porosity in the epoxy resin layer and it took a long time for the corrosive media to reach the substrate's surface. Tables 3 and 4 show a decrease in porosity percentage with increasing SiO_2 concentration for both the NaCl and H_2SO_4 testing media, as shown in Fig. 9.

4. Conclusion

In this paper, an epoxy /nano SiO_2 composite has successfully coated on the surface of carbon steel by using the dip method. The investigation of structure and morphology reveals that epoxy and SiO_2 nanoparticles interact in some way. The corrosion evaluation of coated carbon steel in 3.5 wt% NaCl and 0.5 vol% H_2SO_4 by Open Circuit Potential (OCP) and Tafel polarization have demonstrated that epoxy/nano SiO_2 coating has the higher anti-corrosive performance which increased with the increase of nano-silica concentration from (0.1 wt%, 0.3 wt%, and 0.5 wt%). The corrosion rate reduces 63 times in the saline media meanwhile reduce to 436 times in acidic media. The corrosion protection efficiency increases with the increase of nano-silica concentration, which means that the epoxy/0.5 wt% nanoSiO_2 coating to carbon steel substrate can be up to 98.4% and 99.7% in saline and acidic solutions, respectively. Porosity percentage declined with the increase of nano-silica concentration was 1.4% and 0.45% respectively in NaCl and H_2SO_4. So, it can conclude that the Nanoparticles SiO_2

with epoxy improve the chemical properties and enhance the corrosion resistance in corrosive environments.

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