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Sol-gel Spin-coated SiO_2 Multilayers for Anticorrosion Applications

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ABSTRACT

A silicon dioxide (SiO_2) film protects steel or other metals by acting as a highly dense, impermeable barrier against corrosive agents. SiO_2 thin-film coatings are widely used for their superior performance and economic benefits. In this work, a sol-gel-synthesized and spin-coated SiO_2 thin film was deposited on mild steel, which was calcined at 300°C. The prepared thin films were examined for their optical, structural, functional, morphological, elemental, and corrosion properties using XRD, FTIR, SEM, EDX, and EIS. The XRD spectrum revealed sharp, narrow diffraction peaks, confirming the quality of the crystallite-nature enhancement induced on the mild steel surface. In addition, sharp diffraction peaks confirmed the presence of SiO_2 and the formation of an ultrafine crystal structure on the mild steel. FTIR transmittance spectrum shows the stretching vibration modes of the primary material, Si-O-Si, between 927 and 439 cm^{-1}. The surface morphology of the prepared multilayer thin films was examined using SEM, whereas the EDX spectrum indicated the presence of Si and O. Finally, electrochemical impedance spectroscopy (EIS) is useful for evaluating strong anticorrosion properties. In future, the anticorrosion property can be studied by using TiO_2, ZrO, and SiO_2/TiO_2 etc.

Keywords: SiO_2; Thin film; Corrosion; XRD; FTIR.

1. INTRODUCTION

Metal oxides (SiO_2, ZnO, TiO_2) have been used in important corrosion-resistance applications. Among these metal oxide materials, SiO2 is one of the most environmentally friendly and silicon-abundant materials in nature (Jeong et al., 2024). Controlling corrosion in metals is a significant concern for the scientific community. It should be economical, technically sound, and environmentally important. For example, marine industries nowadays play a significant role in economic growth not only for the country but also for fishermen, including the production and development of marine resources that are directly or indirectly related to industrial growth. In general, corrosion is a major and complex problem that affects the economic progress of countries as well as marine fleets. We know that seawater is a strong corrosive medium for metal surfaces, including mild steel. For many decades, marine personnel have used common methods, such as painting or coating metal or steel surfaces, to isolate the corrosive environment and the metallic matrix (Sanjeev et al., 2024). Corrosion is an unambiguous problem that has prevailed worldwide, so for many decades, corrosion protection studies have gained momentum and have become an extensive research area for academics. Nowadays, nanothin-film technology has been used for corrosion protection of metal or steel surfaces by separating them from corrosive environments (Reyes-Tesillo et al. 2024; Zhang et al. 2019), and as a result, this technology is a significant tool in high-temperature applications. Generally, when metals are exposed to an oxidizing gaseous environment at high temperatures, corrosion occurs and is referred to as high-temperature oxidation or gaseous corrosion (Rajendran et al., 2022). Therefore, it is necessary to protect the corroding material from the highly corrosive atmosphere by applying protective methods such as functioning coatings, injection of corrosion inhibitors, cathodic protection, etc. Among metallic oxides, silicon dioxide (SiO2) is a significant and emerging nanomaterial with a wide range of applications in thin-film technology, particularly its ability to enhance the corrosion resistance of steels at different temperatures due to its high thermal and chemical resistance. We have various techniques, for synthesis of multilayer thin films which will be used as a coating material on a steel substrate, such as chemical vapor deposition technique, plasma spraying, laser cladding, and chemical plating or sol- gel methods, among which sol- gel technique stand out first because it is an ideal and eco-friendly as well as economically viable method for its efficient production of coatings while spin coating method is also important method for synthesis of nano scale thin films. Maia *et al.* (2012) presented SiO_2 mesoporous nanoparticles loaded with 2-MBT and studied their corrosion-protection properties. Balaskas *et al.*(2017) reported that a novel two-shell structured inhibitor-loaded methacrylic acid with CeO_2 nanocontainers was synthesized and studied for its corrosion protection of damaged epoxy-coated AA 2024-T_3. Ribeiro *et al.* (2023) experimentally examined the reduction in mild steel degradation caused by green garlic peel at various pH values, which acted as an inhibitor. Stambolova et al. (2018) prepared SiO2 coatings on steel to enhance corrosion resistance using the sol-gel dip-coating method. They examined (250 hrs) the weight loss of uncoated steel and SiO_2 thin film as 1.81 and 1.06 mg/cm^2. After 346 hours, no weight changes were observed. Wei *et al.* (2022) reported improved anticorrosion performance using silicon- and nitrogen-incorporated hydrogenated diamond. It has shown better corrosion resistance in seawater and is environmentally friendly, making it a promising protective material. Annie et al. (2026) gave experimental and theoretical insights into TiO_2-SiO_2-ZnO hybrid nanocoating. The sol-gel synthesis process and COSMOL simulations were used throughout this study.

The corrosion resistance is a major drawback of mild steel, as discussed in various scientific works in the literature survey. The aim of this work is to synthesize and fabricate SiO_2 multilayers on mild steel for anti-corrosion applications. The SiO_2 solution was synthesized by the sol-gel process and coated on mild steel using the spin-coater technique. The SiO_2 thin film was deposited in four layers on mild steel to enhance its anti-corrosion properties. After the calcination process, SiO2 thin films were characterized for their optical, morphological, structural, elemental, and anti-corrosion properties using XRD, SEM, EDX, and electrochemical impedance (EI) spectroscopy.

2. MATERIALS AND METHODS

2.1 Materials

Tetraethyl orthosilicate (TEOS, $Si(OC_2H_5)_4$), acetic acid (CH_3COOH), and ethanol (C_2H_6O), acetone (C3H6O) are procured from Sigma–Aldrich, along with distilled water and mild steel as a substrate, for the synthesis of the multilayer nano thin films. TEOS is used as the primary source, ethanol as the solvent, and acetic acid as the catalyst for preparing the SiO_2 solution. Further, acetone, ethanol, and distilled water have also been used to clean the mild steel substrate prior to the deposition of the SiO_2 thin film.

2.2 Methods

The sol-gel synthesis is a simple method for preparing a SiO_2 solution. Among numerous synthesis methods, the sol-gel method is a well-established method that was developed by J.J. Ebel in the year 1842 (Gasiorek et al.2018) for obtaining homogeneous coatings free of microcracks on steel substrates. This method is a significant one for preparing organic or inorganic solutions by adding two or more chemicals at the atomic level, and it could greatly support the use of silicon (Si) and titanium (Ti) as primary elements for depositing thin films, which generate the highest aspect ratio.

Figure 1 shows the sol-gel synthesized SiO_2 solution deposition process on a mild steel substrate. The preparation of SiO_2 solution by the sol–gel method is as follows: Three chemicals, namely TEOS (a source element), ethanol, and acetic acid, are used in either liquid or powder form. This process involves converting monomers into a colloidal solution (sol) that serves as the precursor to an integrated network (gel) of either discrete particles or network polymers (Geetha et al., 2024; Saravanan and Dubey, 2020).

The sol-gel synthesis was used to prepare a SiO2 solution by adding TEOS (1.5 ml) to 20 ml of ethanol, the solvent. Next, the acetic acid (2.3 ml) was added drop by drop, acting as a catalyst and chelating agent. This solution was stirred for 90 minutes using a magnetic stirrer to achieve homogeneity. Finally, the prepared transparent solution is left to age for 24 hours. Figure 2 shows the schematic diagram of a SiO_2 film on mild steel substrates. The chemical reaction of the sol-gel synthesis process is as follows,

$$\mathrm{Si(OC_2H_5)_4 + 2H_2O + 2CH_3COOH \rightarrow SiO_2 + 4C_2H_5OH + 2CH_3COOCOOC_2H_5}$$

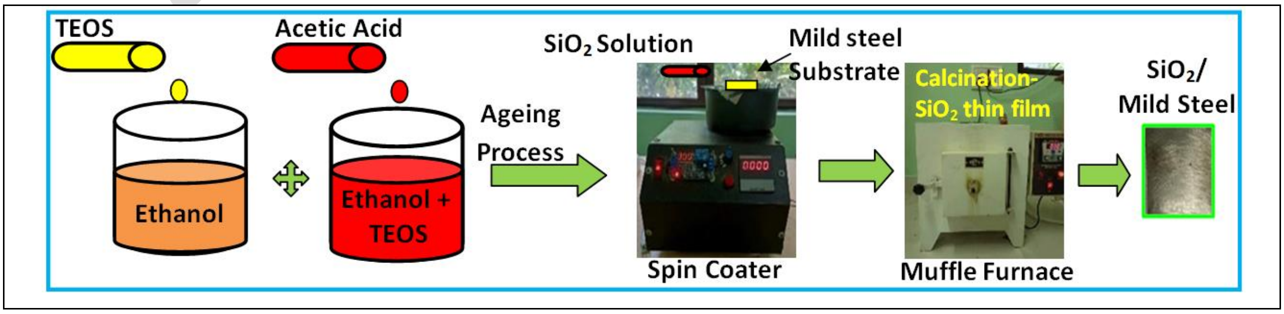


Fig. 1: The procedural steps of preparation of SiO_2 thin film by Sol-gel spin-coating technique

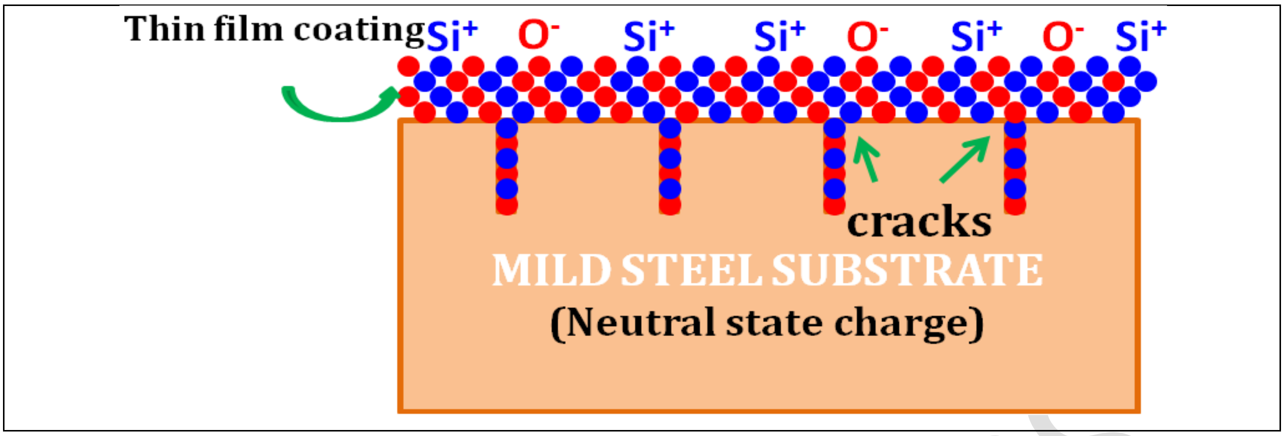


Fig. 2: The illustration of corrosion resistance on mild steel with SiO_2 multilayers

The process of depositing the coatings was carried out through the spin-coater technique and is demonstrated in Figure 1. In this process, the prepared solution is dripped onto the substrate using a spinning mechanism to achieve homogeneous spreading. The thickness of the mild steel substrate is 0.3 cm. The length and breadth of this substrate are considered as 2 × 2 cm. Initially, the substrate was cleaned with acetone and ethanol to remove undesirable elements such as grease, oil, and dust, using an ultrasonic cleaner at 40°C for 30 minutes. The ultrasonically cleaned substrate was coated with a thin SiO_2 film at room temperature (28 °C). Magnetic stirring was maintained at 2000 rpm for 30 seconds. Consequently, four layers of thin films were coated on mild steel to enhance anticorrosion properties. Each thin film was calcined at 300 °C for 1 hour, and this temperature was maintained for each coating to enhance the adhesion of the SiO_2 thin layer on the substrate.

2.3 Characterization

X – ray diffraction (RigakuMiniflex, UK), FTIR (JASCOFT/IR6600, Japan), Scanning electron Microscopy (TESCOW VEGA 3, Czech Republic), Energy dispersive X –ray spectroscopy, EDS (Oxford Instrument, UK), Electrochemical Impedance spectroscopy (CH1100) techniques have been used to know the crystalline as well structural properties of prepared SiO_2 multilayer thin films on mild steel substrate including the functional and elemental groups present in the sample and corrosion resistance studies have been done.

3. RESULTS AND DISCUSSION

We prepared four thin films coated on mild steel using simple sol-gel and spin-coating deposition techniques. Further, we investigated their optical, functional, structural, and corrosion properties by using different characterization techniques as follows,

3.1 Structural Analysis: X-ray Diffraction (XRD)

Figure 3shows the X-ray diffraction pattern of SiO_2 multilayer thin films as a function of scattering angle 2θ (degree). The intensity peaks (sharp and narrow) are observed with increasing diffraction angle, which are due to constructive interference of scattered X-rays for the periodic structure of the prepared sample (Dwivedi *et al.*2017).

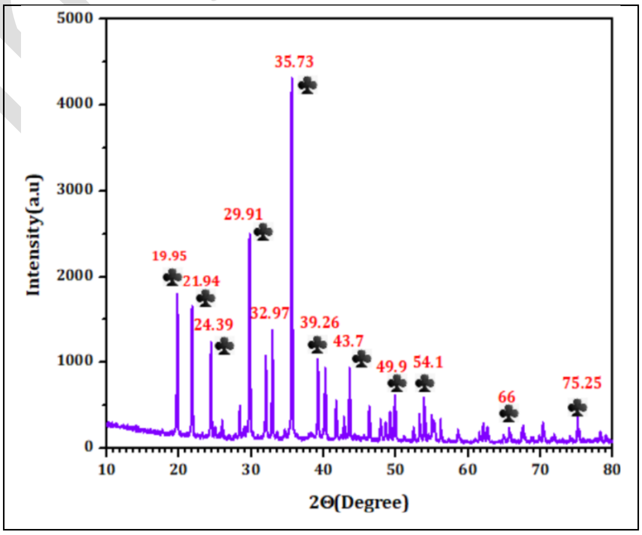


Fig. 3: The XRD pattern of SiO_2 thin film on mild steel

The sharp peaks indicate the quality of the crystalline enhancement induced in the steel substrate at a specific annealing temperature (Bajorek et al. 2022; Yadav et al. 2016), confirming the purity of the SiO_2 sample. The low-intensity peaks observed as the diffraction angle increases indicate the presence of the prepared samples. First, the diffraction peak at 66° (2θ) appeared due to the presence of the SiO_2 phase, as reported by Halin et al. (2017). As observed from the XRD spectrum, the sharp peaks indicate the ultrafine crystal structure of the prepared SiO2 thin film, while the remaining diffraction peaks confirm the presence of a steel (Fe2O3) substrate, as reported by Ghosh *et al.* (2015). The diffraction peaks are 21.94, 35.73, 39.26, 46.7, 48.7, 54.1, 56.9, 62, 66, and 79° associated with the planes (110), (111), (200), (201), (112), (202), (210), (211), (300), and (213). All the XRD peaks are well matched with JCPDS Card: 00-046-1045(Ali *et al.*2023). We have calculated the average crystallite size from the half-width of the full maxima (HWFM) of specific XRD peaks by using Debye Scherrer's formula (Bajorek *et al.*2022).

$$D = \frac{K\lambda}{\beta \cos\theta} \quad ...(1)$$

Where K is the Scherrer constant, which for a spherical particle equals 0.89; β is the full width at half maximum (FWHM); θ is the diffraction angle; and λ is the wavelength of the incident X-ray (Cu K_α line, 1.54059). The wavelength ranges are considered from 10 to 80°C with a step of 0.02 and a speed of 0.04. Silicon bonded with oxygen molecules and the formation of SiO_2, confirming on mild steel.

3.2 Functional Group Analysis: Fourier Transform Infrared Spectroscopy

Figure 4 shows the FTIR transmittance spectrum of the SiO_2 solution, a representative spectrum of the synthesized sample, confirming the formation of SiO_2. This spectrum is taken over the wavelength range of 400 to 4000 cm^{-1} for SiO_2 multilayer thin films on a mild steel substrate. The broader absorption bands are noticed from 3740 to 2763 cm-1, corresponding to the presence of O-H or hydroxyl groups, as a result of which the O-H stretching mode is obtained at 1621 cm^{-1} (Saravanan and Dubey, 2020). It is noteworthy that the primary material of the Si-O-Si stretching vibration bond is observed between 927-439 cm-1 (Table 1). The bands observed at 1353 cm^{-1} and 1219 cm^{-1} confirm the presence of the O-H group of bending vibration (Zaved et *al.*2016).

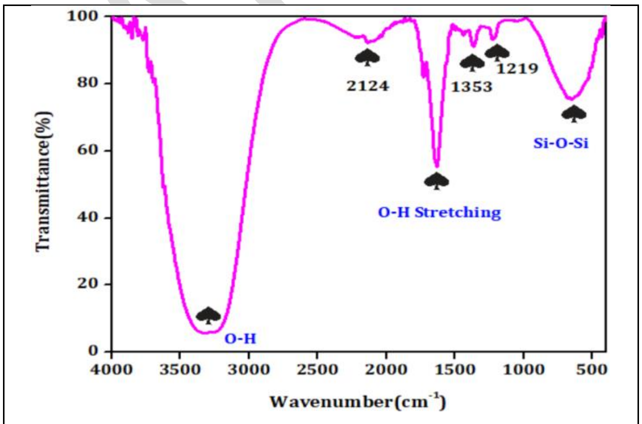
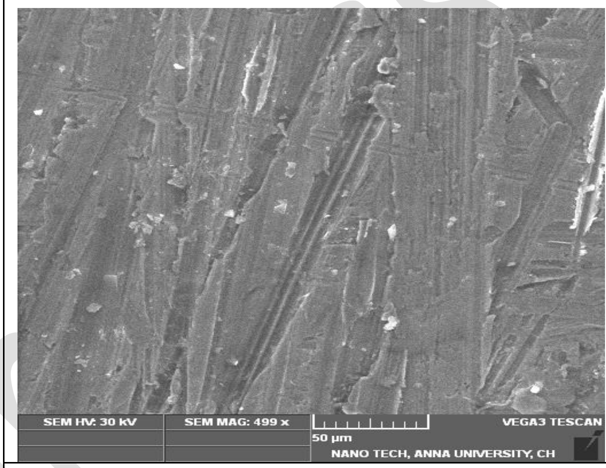


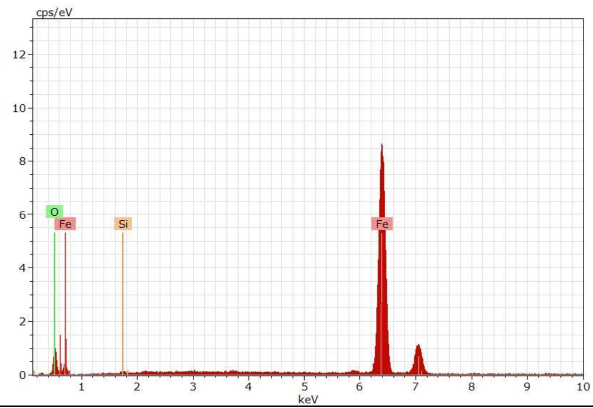
Fig. 4: The FTIR spectrum of SiO_2 multilayer thin film

Table 1. The functional groups of the prepared SiO_2 thin film

S.No.	Absorption Peaks	Vibration Modes	Ref.
1	439 - 927cm^{-1}	Si-O-Si stretching vibration	(Nariyal *et al.*2014)
2	1219 cm^{-1} and 1353 cm^{-1}	O-H group of bending vibration	(Zaved *et al.*2016)
3	1621 cm^{-1}	O-H stretching	(Saravanan and Dubey, 2020)
4	2124 cm^{-1}	Si-OH	(Ali *et al.*2023)
5	2763 to 3740 cm^{-1}	O-H or hydroxyl group	(Saravanan and Dubey, 2020)



(a)



(b)

Fig.5: The SEM morphology and EDX spectrum of SiO_2 thin film on mild steel

3.3Study of Morphology: Scanning Electron Microscope (SEM)

Figure 5 (a)revealed the surface morphology of SiO_2 multilayered structure, which strongly depended upon the annealing temperature. During annealing, the nanoparticles are deposited onto the mild steel substrate, forming numerous nucleation centers. For SiO_2 thin films, we observed significant growth of grains or crystallites on the substrate, which has an almost flat surface. Each coated thin film is maintained in a calcination atmosphere at 300°C for one hour. We observed that during corrosion testing, these thin films undergo significant changes at the microscale due to the steel substrate. For this reason, microscale cracks are observed in the micrograph images. It is also observed that, at the same time, the SiO_2 thin films are coated homogeneously on the mild steel substrate. This homogeneous coating is deposited on mild steel under environmental conditions such as room temperature and humidity.

3.4 Elemental Analysis: Energy Dispersive X-ray (EDX) Spectroscopy

Energy-Dispersive X-ray (EDX) spectroscopy is a useful tool for evaluating the chemical composition of prepared, sol-gel-synthesized, and spin-coated SiO_2 thin films. Figure 5(b)revealed the purity of the prepared sample as well as the primary elements such as Fe, Si, and O in the presence of SiO_2 thin films (multilayers). Consequently, no other impurities were present.

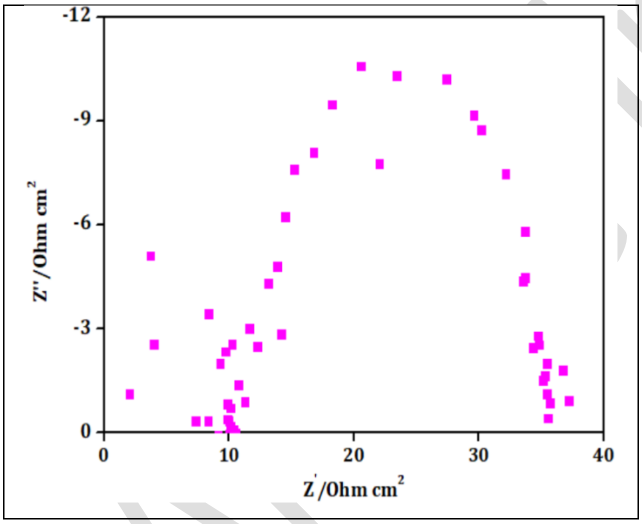


Fig.6: Electrical impedance spectrum of SiO_2 thin film on mild steel

3.5 Electrochemical Analysis: Corrosion Resistance

The corrosion resistance of mild steel could be improved by pre-treating with cleaning processing before the coating process. Electrochemical impedance is an ideal tool for studying the kinetics of corrosion processes. It provides dynamic information on the atomic-level chemical reaction between the mild steel and the coated thin films on the substrate. Corrosion resistance is achieved by depositing a SiO_2 multilayer coating on mild steel and integrating suitable inhibitors (Emmanuel, 2024; Thamaraiselvi and Nalini, 2022). Generally, mild steel does not have any neutral charges. However, when exposed to the prepared solution on mild steel, the steel acquires a positive charge that depends on the solution's pH and other physical and chemical parameters. We know that mild steel is highly reactive with air and water due to oxygen, ions, and molecules; therefore, it quickly rusts, so a protective coating or painting will be necessary. Figure 6 shows the electrical impedance spectrum of SiO_2 thin films on mild steel. The spectrum revealed that the corrosion potential varies greatly with optimized time. These measurements indicated that the superhydrophobic composite surface improved corrosion resistance when coated with SiO_2.

In these studies, a very thin SiO_2 layer was deposited on mild steel, thereby enhancing corrosion resistance and providing significant protection against corrosive agents such as oxygen and moisture (Mohapatra et al., 2023). At the same time, the corrosion protection efficiency of thin-film SiO_2 coating decreases with increasing precursor concentration, as reported by Irina Stambolova et al. (2018); Sanjeev et al. (2024) reported that the corrosion rate gradually decreases with increasing thin-film layer thickness on the substrate. In the present work, the number of SiO2 thin-film coatings on the substrate was increased using the spin-coating process. This process is repeated four times, ultimately producing thin films on mild steel, ensuring full coverage and enhancing corrosion resistance, as reported by researchers (Bajorek et al., 2022).

4. CONCLUSIONS

In this summary, multilayer silicon dioxide (SiO_2) thin films are deposited on mild steel using sol-gel and spin-coating techniques. The structural and crystalline properties of the coated SiO2 multilayers on the mild steel surface were confirmed by XRD. These diffraction peaks indicate the presence of SiO2 and are well matched with the JCPDS card. Improved adherence to mild steel is evident in the SEM micrograph, showing that SiO_2 thin films are homogeneously coated on the mild steel substrate. This work shows that multilayer thin films and annealing temperature are significant factors that strongly affect the surface structure, morphology, and optical properties of the materials. In addition, the electrochemical impedance analysis indicated good corrosion resistance against mild steel, which depends on the concentration of the SiO2 solution and the duration of treatment. In the future, these works could be extended through long-term corrosion testing, salt spray testing, and marine-environment evaluation.

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CONFLICTS OF INTEREST

The authors declare no conflict of interest.

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