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RESEARCH ARTICLE

Preparation and corrosion protection of polypyrrole/SiO₂ nanocomposite doped with 3-nitrosalicylic acid on aluminum

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Funding information

Vietnam National Foundation for Science and Technology Development (NAFOSTED), Grant/Award Number: 104.02-2019.327

Abstract

Polypyrrole/SiO₂ nanocomposite doped with 3-nitrosalicylic acid was synthesized in three concentrations of 3-nitrosalicylic acid: 0.01 M, 0.005 M, and 0.0025 M. FTIR spectroscopy, thermogravimetric analysis (TGA), Raman scattering spectroscopy, and FESEM imaging further assisted in determining the shape and structure of the polypyrrole/nanocomposite product. The aluminum protection arrangement of the paint film comprising polypyrrole/SiO₂ nanocomposite doped with 3-nitrosalicylic acid was proven by electrochemical methods. The salt spray test results also demonstrated the protective efficacy of the paint film comprising polypyrrole/SiO₂ nanocomposite doped with 3-nitrosalicylic acid to suppress corrosion. The best corrosion prevention results were obtained with a polypyrrole/SiO₂ nanocomposite dispersion paint film doped with 0.01 M 3-nitrosalicylic acid at a concentration of 2%.

KEYWORDS

3-nitrosalicylic acid, aluminum, corrosion protection, nanocomposite, polypyrrole, self-healing

1 | INTRODUCTION

Conducting polymers (CPs) have attracted a lot of interest in recent years due to their environmentally friendly nature and wide range of applications such as anti-corrosion and metal protection, sensor materials, fuel cells, and so on.^{1–3} Polypyrrole (PPy) is one of the most promising CPs because of its stability and environmental friendliness, remarkable durability, ease of synthesis, and high electrical conductivity.^{4,5} The most important aspect is that when PPy

is combined with additional elements such as silica, a material with specific corrosion resistance and metal protection qualities is formed.

Silica (SiO₂) features great hardness, low refractive index, thermal and chemical durability, large surface area, and reasonable price.^{6–8} As a result, SiO₂ nanoparticles are employed to improve the mechanical characteristics and minimize the thermal breakdown of polymer films at high temperatures. Furthermore, the surface energy of nano SiO₂ is so high that the development of a polymer/SiO₂

core-shell structure not only prevents agglomeration of nano SiO₂ but also improves polymer sphere dispersion. As a result, the material's mechanical and chemical characteristics can be improved.^{9–12}

When electropolymerizing polypyrrole on aluminum, the creation of Al₂O₃, which acts as a barrier impeding electron transmission and the polymerization process, makes the process more complex.^{13,14} Despite this, many published researches have revealed the potential of PPy-epoxy coatings in inhibiting the corrosion of aluminum alloys, including AA1200,¹⁵ AA2024,^{16–19} AA6082,^{18,19} AA7075,^{20,21} and AA2019-T6.²² Several of these articles have explored the influence of the substrate's microstructural and compositional aspects on the electrodeposition and electrochemical properties of PPy coating.

In this article, we focused on the synthesis of polypyrrole-based silica nanocomposite coatings with the corrosion inhibitor anion 3-nitrosalicylic acid and used them in paint as a corrosion inhibitor for aluminum. Electrochemical techniques and salt spray tests were used to evaluate the corrosion protection efficacy of paint films comprising polymer/SiO₂ nanocomposites doped with corrosion inhibitors.

2 | MATERIALS AND METHODS

2.1 | Chemicals and materials

Pure pyrrole (Sigma-Aldrich, USA), pure 3-nitrosalicylic acid (Leyan, China), pure ammonium persulfate (APS) (Xilong, China), high purity industrial nano silica powder (Kangyexin China), pure isopropyl alcohol (Xilong, China), pure xylene (Xilong, China), epoxy D.E.R 671-X75 (epoxy resin in the original form of diglycidyl ether bisphenol-A dissolves with the concentration of 75% in xylene), polyamine-polyamide curing agent have been used in this research.

A 6061-T6 aluminum panel with a size of 150 × 150 × 2 mm was used as substrate.

2.2 | Synthesis of polypyrrole/SiO₂ nanocomposite doped with organic dopant

Firstly, the solution (S1) consisted of 3-nitrosalicylic acid (30% vol/vol) in isopropyls alcohol was prepared by uniformly dispersed. Next, S2 solution was obtained by dissolving 3 g of SiO₂ in 250 mL of S1 ultrasonically. 3.5 mL of pyrrole monomer were added in S2 solution and kept stirring in 30 min using a magnetic stirrer. Dissolving 18 g of ammonium sulfate in 50 mL of S1 and gently adding it to the reaction mixture (S2) for 15 min.

After 3 h of reaction, the products was filtered, washed with distilled water and dried in vacuum for 24 h at 500 °C. Polypyrrole/SiO₂ nanocomposite doped with 3-nitrosalicylic acid (0.01 M, 0.005 M, 0.0025 M) was obtained.

TABLE 1 Component for making anion-doped polypyrrole/SiO₂ nanocomposite to inhibit corrosion.

No.	Products' code samples	Components			
		SiO ₂	Pyrrole	APS	3-NiSA
1	T ₁	3 g	3.5 mL	18 g	–
2	T ₂	3 g	3.5 mL	18 g	–
3	T ₃	3 g	3.5 mL	18 g	0.01 M
4	T ₄	3 g	3.5 mL	18 g	0.005 M
5	T ₅	3 g	3.5 mL	18 g	0.0025 M

Dissolving 3 g of SiO₂ in 250 mL of solution (isopropyl alcohol/3-nitrosalicylic acid = 30/70). Add 3.5 mL of pyrrole to the mixture and stir for 30 min using a magnetic stirrer. In 50 mL of solution (isopropyl alcohol/3-nitrosalicylic acid = 30/70), dissolve 18 g of ammonium persulfate (APS) and gently add it to the reaction mixture for 15 min. Allow for 3 h of reaction time, then switch off the stirrer, filter the product, wash with distilled water, and vacuum dry for 24 h at 500 °C to get a consistent weight.

Polypyrrole/SiO₂ nanocomposite doped with 3-nitrosalicylic acid (0.01 M, 0.005 M, 0.0025 M) is the final product (Table 1,2).

2.3 | Preparation of paint coatings on aluminum

Amount of 2% of the produced nanocomposite particles was dispersed in 15% xylene solvent and mixed for 1.5 h using ultrasound. Then, 5 g of X75 epoxy resin was added, followed by 2 h of ultrasonic vibration and 1 h of magnetic stirring. Finally, 4 g of polyamine-polyamide curing agent was added and then pattern it on the metal substrate.

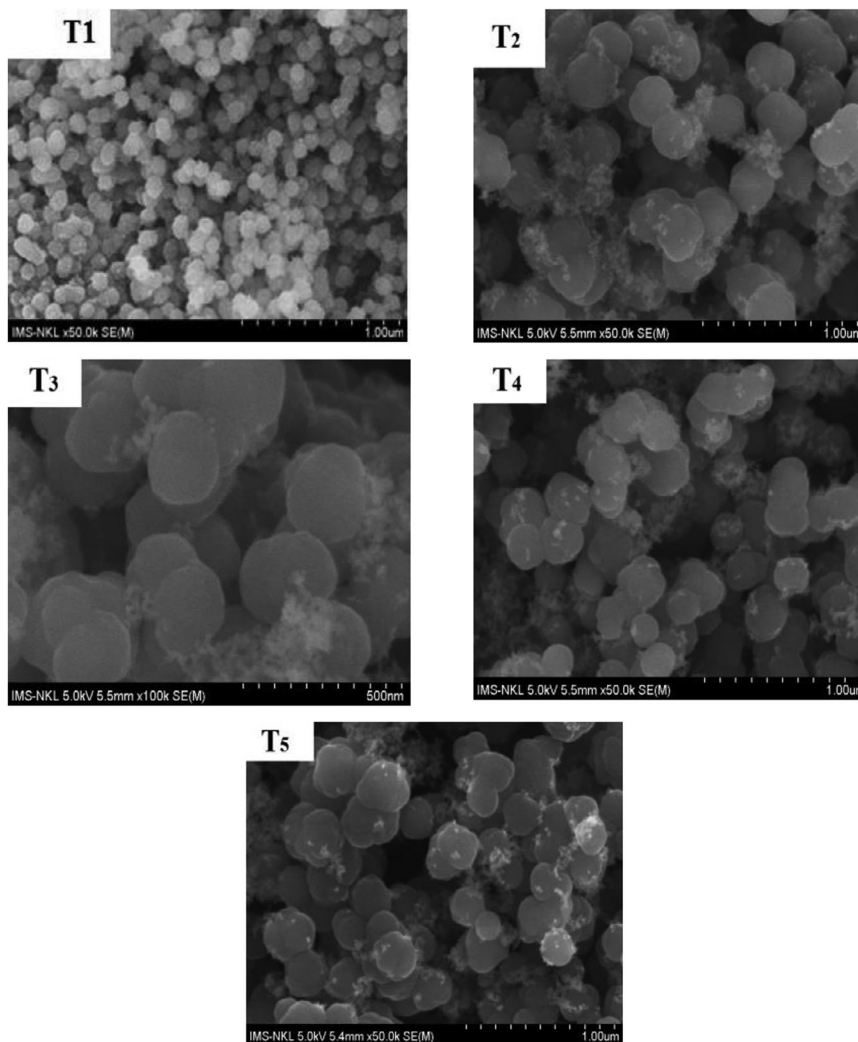
2.4 | Methods and devices

Field emission scanning electron microscopy (HITACHI-4800 FESEM, Japan), thermogravimetric analysis (TGA-50H thermogravimetric analyzer, Shimadzu), Fourier-transform

TABLE 2 Aluminum samples coated with paint film tested in salt spray.

No.	Code	Components
1	I	SiO ₂ 1%, epoxy X75, curing agent polyamine-polyamide
2	II	SiO ₂ 2%, epoxy X75, curing agent polyamine-polyamide
3	III	SiO ₂ 4%, epoxy X75, curing agent polyamine-polyamide
4	IV	T ₂ 1%, epoxy X75, curing agent polyamine-polyamide
5	V	T ₂ 2%, epoxy X75, curing agent polyamine-polyamide
6	VI	T ₂ 4%, epoxy X75, curing agent polyamine-polyamide
7	VII	T ₃ 1%, epoxy X75, curing agent polyamine-polyamide
8	VIII	T ₃ 2%, epoxy X75, curing agent polyamine-polyamide
9	IX	T ₃ 4%, epoxy X75, curing agent polyamine-polyamide

FIGURE 1 FESEM images of powder samples T₁, T₂, T₃, T₄, and T₅.



infrared spectroscopy (Prestige-21, Shimadzu), Raman spectroscopy (Lazar Raman Spectrophotometer, Ramalog 9I, USA), open circuit potential (OCP) method in NaCl 3%

solution (Zenium Potentiostat EIS, Zaehner, Germany), and salt spray test (Q-FOG CCT 600 salt spray test instrument) were used in this research.

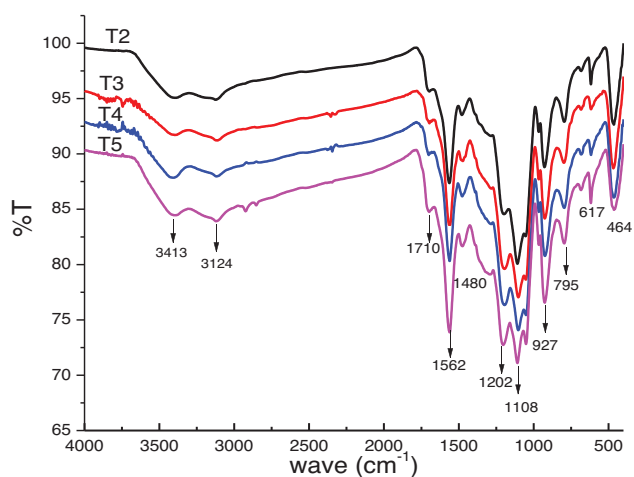


FIGURE 2 FT-IR spectra of samples T₂, T₃, T₄, T₅.

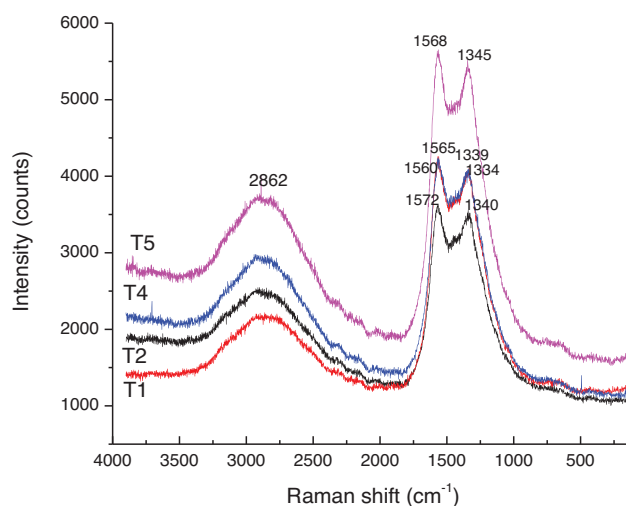


FIGURE 3 Raman spectra of samples T₂, T₃, T₄, T₅.

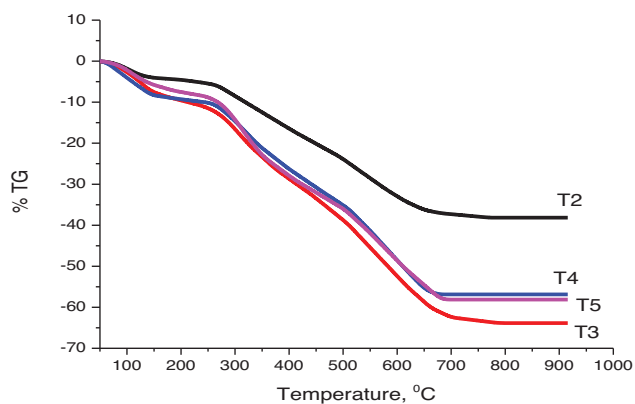


FIGURE 4 TGA curves of samples T₂, T₃, T₄, and T₅.

3 | RESULTS AND DISCUSSION

3.1 | FESEM images of polypyrrole/SiO₂ nanocomposites

Field emission scanning electron microscopy was used to study the morphology of nano-silica (T₁), polypyrrole/SiO₂ nanocomposite (T₂), polypyrrole/SiO₂ nanocomposite doped with 0.01 M 3-nitrosalicylic acid (T₃), polypyrrole/SiO₂ nanocomposite doped with 0.005 M 3-nitrosalicylic acid (T₄), polypyrrole/SiO₂ nanocomposite doped with 0.0025 M 3-nitrosalicylic acid (T₅) (Figure 1).

All FESEM images of the samples demonstrate that the nanocomposite has a spherical form, comparable to that of nano-silica particles and a smooth texture. The polymer mixture's microstructure revealed a cluster of spherical polypyrrole matrix mixed with identical SiO₂ particles.²³ This is because the pyrrole monomer molecule adsorbed on the silica surface is polymerized in the presence of the oxidizing agent APS and forms a core-shell structure.²⁴

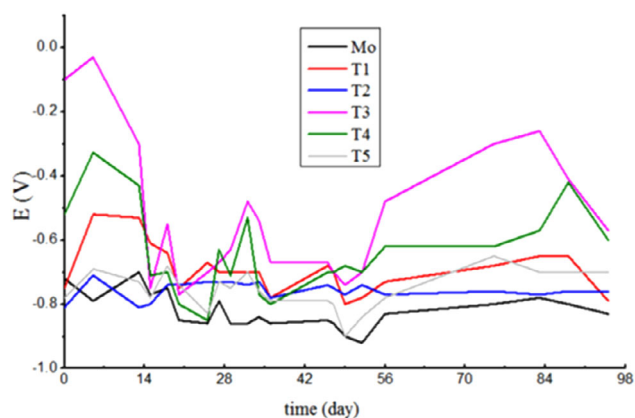


FIGURE 5 Open circuit potential curves of aluminum coated with original paint film (Mo), paint film containing SiO₂ (T₁), and paint film containing T₂, T₃, T₄, and T₅ according to immersing time in 3% NaCl solution.

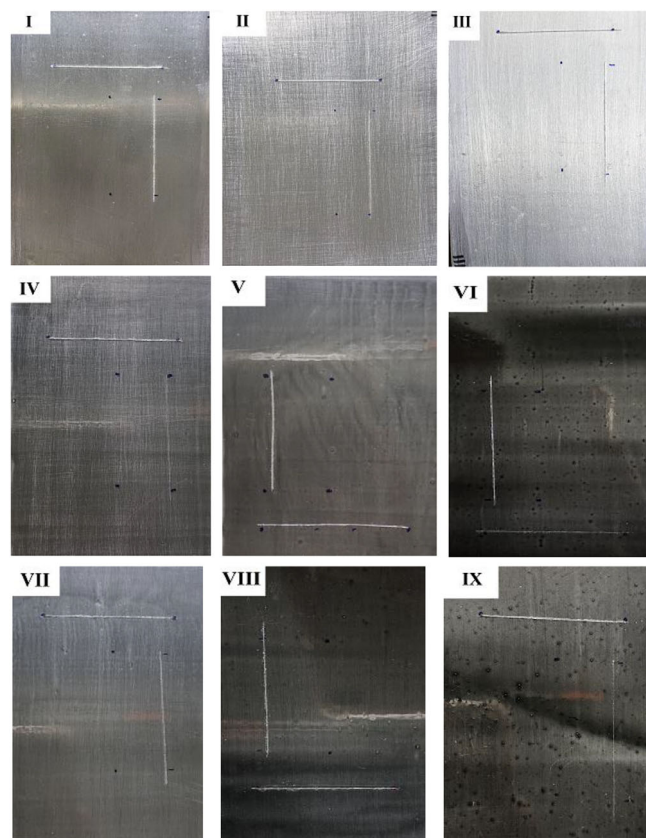


FIGURE 6 Aluminum panels covered with paint film before salt spray test.

3.2 | FTIR spectra

At wavelength 1108 cm⁻¹ and 464 cm⁻¹, the FTIR spectra of samples T₂, T₃, T₄, and T₅ have typical stretching and bending vibration ranges of asymmetric vibrations and deformation vibrations of the Si—O—Si bond (Figure 2).²⁵ The peaks at 964 cm⁻¹ and 795 cm⁻¹ characterize the Si—OH symmetric valence vibration of nanosilica. The peak at 1562 cm⁻¹ corresponds to typical pyrrole ring vibrations, the valence vibrations of the aromatic C=C double bond.²⁶ The low-intensity peak at 1480 cm⁻¹ is attributed to the valence vibration of the C—N group.²⁷ The vibration of the =C—H group is at approximately 1054 cm⁻¹. The vibration region below 1000 cm⁻¹ may be due to the deformation vibrations of C—H in the pyrrole ring, in addition to the vibrations of aromatic C—H with adjacent 2H. The characteristic peaks of the samples all have slight shifts. This can be explained by the fact that NH₂⁺ cation was generated in the polypyrrole chain by protonation and the formation of a bond between polypyrrole and silica through the —OH bond. The above results show the strong interaction between polypyrrole and SiO₂ particles of the nanocomposite material and also show that polypyrrole has adsorbed onto the silica surface. In addition, the slight shift of the characteristic peaks may also be due to the association between PPy and the doping anion. Thus, the doped

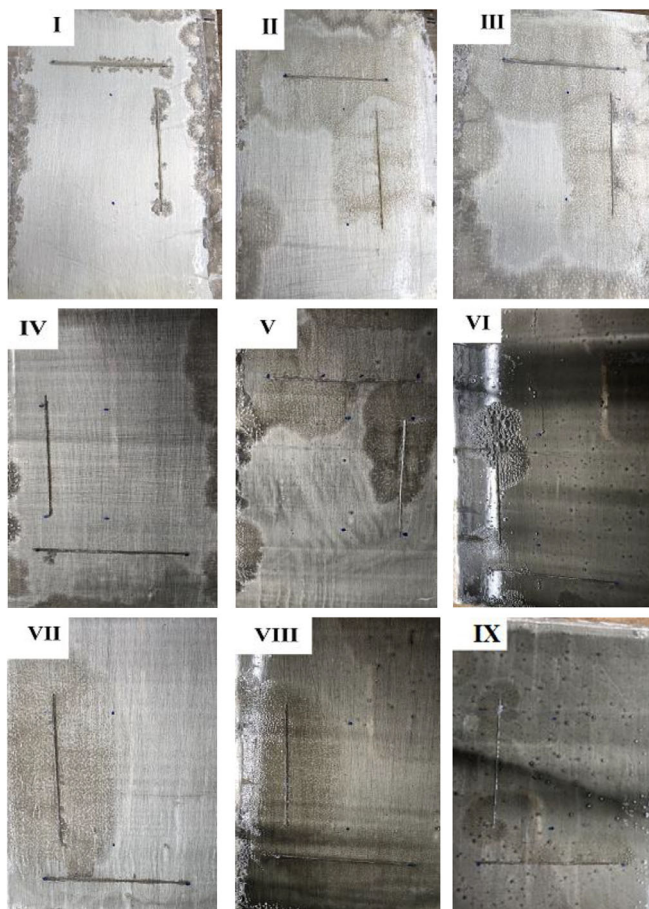


FIGURE 7 Aluminum panels covered with paint film after 480-h salt spray test.

polypyrrole/SiO₂ nanocomposite samples all have characteristic peaks of polypyrrole/SiO₂ nanocomposite, but the presence of doping substances is not really clear.

3.3 | Raman spectra

The Raman spectrum (Figure 3) shows that the forms and locations of the spectrum's peaks are comparable among the samples. The figures of all the samples register their highest peaks at around 1560 cm⁻¹ and 1350 cm⁻¹. These peaks are consecutively assigned to the stretching of conjugative backbone C=C and N-C ring.²⁸ The broad bands in the 2800–3000 cm⁻¹ region are the C-H stretching of CH₂ and CH₃.²⁹ It can be concluded from the similarity between the samples' Raman spectra that the composition and state of the produced polypyrrole are very stable.

3.4 | Thermogravimetric analysis (TGA)

The evaporation of water and certain oligomers in the sample causes the sample weight to drop to around 100 °C. The samples' mass then remained almost unchanged before plummeting at around 250 °C. This is possible because

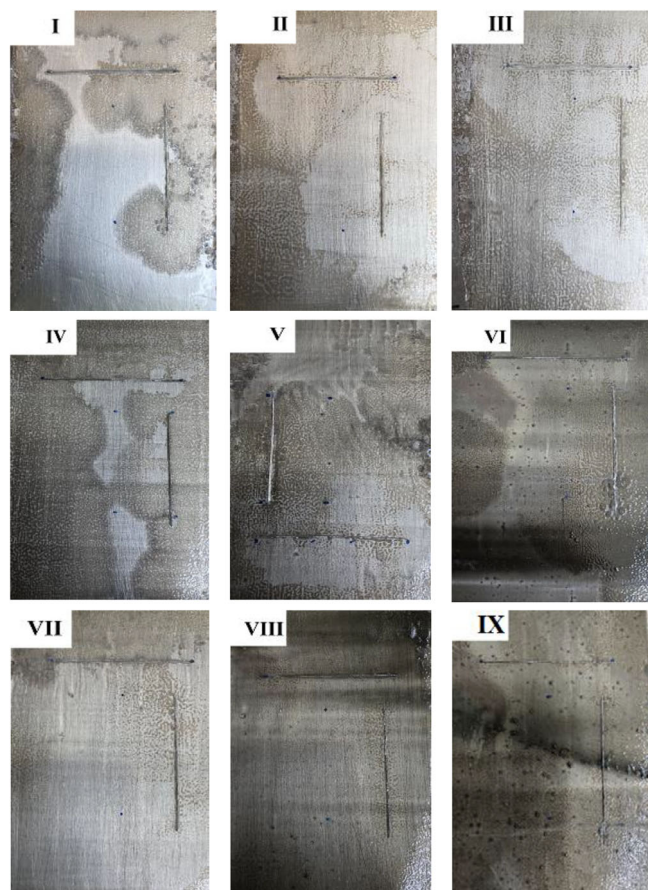


FIGURE 8 Aluminum panels covered with paint film after 960-h salt spray test.

of the decompositions of dopants and residue monomers. When the temperature reaches 450 °C, the polymer chain decomposes next and further decreases the weight of all the samples. After reaching 700 °C, almost all samples hit their all-time low and remained static (Figure 4). Overall, T₃ has the highest thermal stability among the samples due to the lowest mass loss (38.06%).

3.5 | Open circuit potential measurement

Compared to the original Mo sample, T₁, T₂, T₃, T₄, and T₅ exhibit higher open circuit potential values. After five days, the open circuit potential of the Mo sample decreased to -0.79 V. However, the open circuit potential of the remaining paint films increased, specifically -0.52 V, -0.71 V, -0.03 V, -0.33 V, and -0.69 V for the paint film containing T₁, T₂, T₃, T₄, and T₅, respectively (Figure 5).

During the 14 weeks, open circuit potential levels underwent fewer fluctuations after week 5. During this period, aluminum samples covered with paint film exhibited no symptoms of corrosion with T₃ recorded the most positive OCP.

The corrosion prevention ability of the paint film comprising polypyrrole/SiO₂ nanocomposite doped with

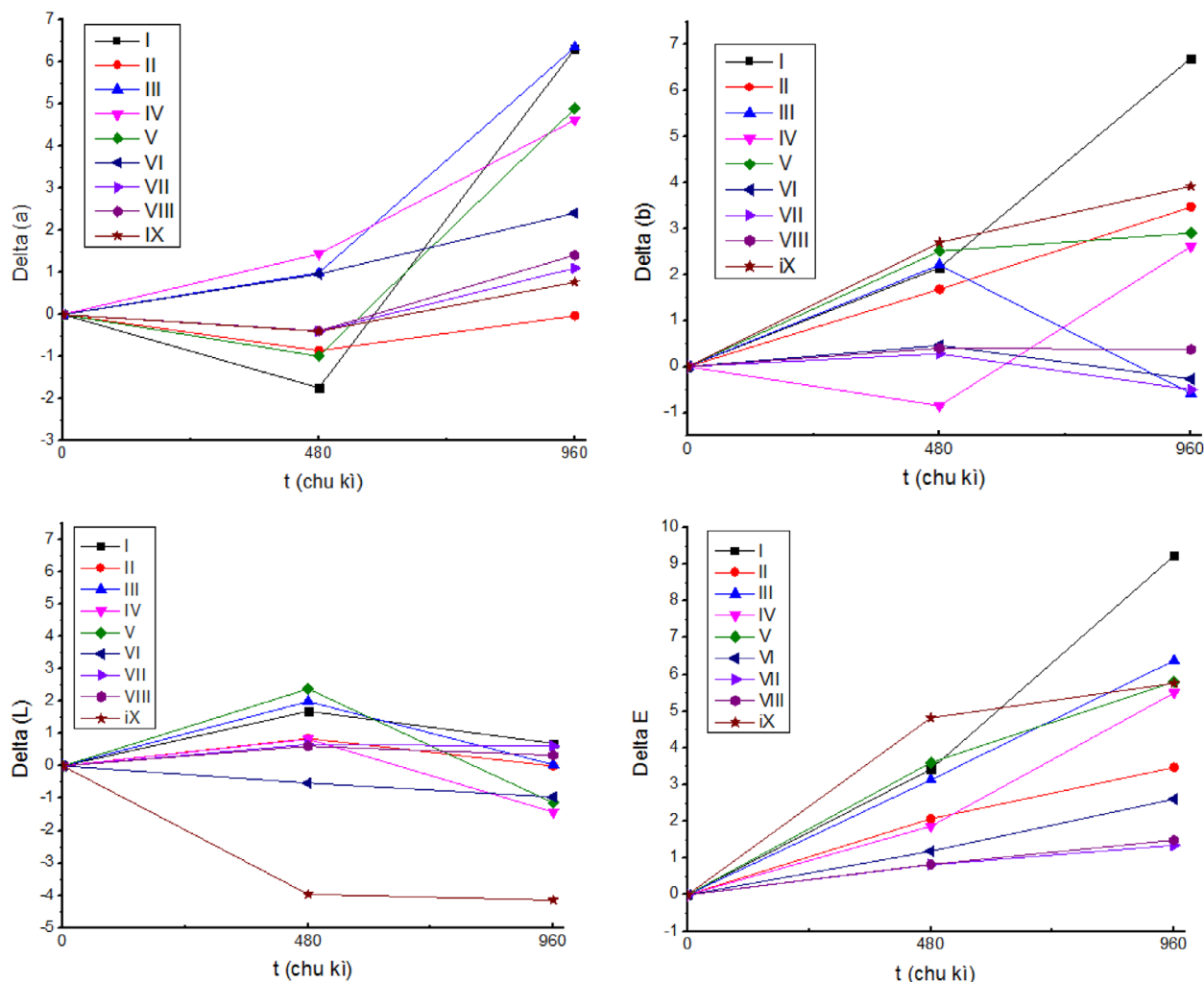


FIGURE 9 Color indexes of samples over time of testing.

3-nitrosalicylic acid was demonstrated by measuring the open circuit potential of the samples. It is clear that, the paint film comprising polypyrrole/SiO₂ nanocomposite doped with 0.01 M 3-nitrosalicylic acid provided the best corrosion prevention results. Although the open circuit potential value of the paint films containing T₂, T₄, and T₅ is lower than that of the paint film containing T₃, it is still bigger than that of the original paint film Mo.

3.6 | Salt spray test

Some corrosion pits began to appear on the sample surface in the early stages of salt mist corrosion (Figure 6). As the corrosion time increases, the number of pits gradually increases, the depth and diameter of the pits gradually expand, and different corrosion streaks begin to form and connect. After 480 h, each small corrosion streak develops into large ones, increases the corrosion rate. Finally, the sample surfaces were covered by corrosion points as shown in Figures 7 and 8. Some corrosion pits could be seen on the sample surface in the early stages of test. After 480 h, panel IV had little corrosion, and panels I, VI, and

IX all had a few small corrosion streaks. Panel II, III, and V displayed a clear appearance of severe corrosion along the incision. This indicates loss of adhesion of the epoxy coating during prolonged exposure to salt spray. The presence of a polypyrrole/SiO₂ nanocomposite mixture doped with 3-nitrosalicylic acid in the epoxy coating has helped improve the anti-corrosion properties of the paint film. This can be seen from the Figure 8 after the salt mist test for 960 h, membrane VIII is the least corroded.

Figure 9 shows that over the time of the experiment, the samples had changes in color indices (Δa , Δb , ΔL and ΔE). The ΔE index of samples VIII is the smallest, which coincides with the comment on surface corrosion above.

4 | CONCLUSIONS

Polypyrrole/SiO₂ nanocomposite materials doped with 3-nitrosalicylic acid at three different 3-nitrosalicylic acid concentrations: 0.01 M, 0.005 M, and 0.0025 M were synthesized to repress corrosion. Electrochemical measurement results of the samples demonstrated the metal protection ability of the paint film containing polypyrrole/

SiO₂ nanocomposite doped with 3-nitrosalicylic acid. The paint film containing polypyrrole/SiO₂ nanocomposite doped with 0.01 M 3-nitrosalicylic acid gave the best corrosion inhibition results. The salt spray test results also showed the corrosion inhibition ability of the paint film containing polypyrrole/SiO₂ nanocomposite doped with 3-nitrosalicylic acid, in which polypyrrole/SiO₂ nanocomposite dispersion paint film doped with 0.01 M 3-nitrosalicylic acid with 2% gave the best corrosion inhibition results.

ACKNOWLEDGMENTS

This research was funded by the Vietnam National Foundation for Science and Technology Development (NAFOSTED) under grant number 104.02-2019.327.

FUNDING INFORMATION

Vietnam National Foundation for Science and Technology Development (NAFOSTED) under grant number 104.02-2019.327

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How to cite this article: L. Van Khoe, H. M. Hung, H. T. Thuy, P. H. Binh, B. T. Anh, L. M. Duc, N. T. Huong, D. T. Y. Oanh, N. X. Thai, V. Q. Trung Preparation and corrosion protection of polypyrrole/SiO₂ nanocomposite doped with 3-nitrosalicylic acid on aluminum, *Vietnam J. Chem.* **2024**, *1*.
<https://doi.org/10.1002/vjch.202400042>