

Corrosion Resistance of PANI/Polymorphic-ZrO₂ Modified Epoxy Paint in High Salinity Environment

Munaji^{1*}, Rizki Dwi Ardika^{1*}, Nanang Suffiadi Akhmad¹, Albet Eka Pratama¹, Ahmad Rifai¹, Alfandy Kurnia Azam¹, Triwikantoro²

¹ Department of Mechanical Engineering, Universitas Muhammadiyah Ponorogo, Indonesia

² Department of Physics, Faculty of Science and Data Analytics, Institut Teknologi Sepuluh Nopember, Surabaya Indonesia.

Corresponding Authors E-mail: munaji@umpo.ac.id, rizkidwiardika@umpo.ac.id

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Abstract

Corrosion in high-salinity environments remains a major concern in maintaining the durability and performance of metallic infrastructure. The use of conductive polymers and ceramic nanoparticles, such as polyaniline (PANI) and zirconia (ZrO₂), has emerged as a promising strategy to improve the corrosion resistance of protective coatings. This study aims to evaluate the effect of ZrO₂ polymorphic phases – tetragonal (t-ZrO₂), monoclinic (m-ZrO₂), and a mixture of tetragonal-monoclinic (tm-ZrO₂) – on the corrosion protection performance of PANI/ZrO₂-modified epoxy coatings applied on ST42 steel. The coatings were applied using spray coating. Compositions of ZrO₂ were varied at 2.5%, 5%, 7.5%, and 10% by weight. To simulate a marine environment, corrosion resistance was assessed using Tafel plot measurements in a 3.5% NaCl solution at room temperature. The results revealed that coatings containing t-ZrO₂ and tm-ZrO₂ phases exhibited significantly lower corrosion rates than those with m-ZrO₂. At 7.5% composition, the addition of ZrO₂ reduced the corrosion rate from 0.6710 mpy (without PANI/ZrO₂) to 0.3988 mpy (with PANI/m-ZrO₂), 0.0364 mpy (with PANI/t-ZrO₂) and 0.0212 mpy (with PANI/tm-ZrO₂). These findings highlight the critical role of ZrO₂ phase composition in improving coating performance. Incorporating t-ZrO₂ and tm-ZrO₂ into epoxy coatings presents a promising pathway to enhance corrosion resistance, offering valuable potential for applications in aggressive saline environments.



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Introduction

Construction materials applied in high-salinity environments are susceptible to corrosion [1] [2] [3]. Corrosion can cause damage to ship structures, reducing the strength and toughness of the structure and causing economic losses [4]. To address this problem, ship structures are often manufactured using medium carbon steel AISI 1042 or ST42. The advantages of ST 42

steel are its high tensile strength and excellent welding, making it suitable for building ship hulls, decks, and other structural components [5]. Although this steel has fairly good corrosion resistance, it also requires additional protection with paint to extend its service life and maintain its structural integrity.

The use of paint as an anti-corrosion coating has long been carried out [6]. Although the industry has provided many choices of anti-corrosion coating paints, current anti-corrosion coating paints still have several weaknesses, including not being resistant to high temperatures, being less resistant to humid areas, and being less effective in the long term [7]. Therefore, research on increasing corrosion resistance continues to be carried out. These efforts include adding additives from plant extracts [8] [9] natural fibers [10], metal oxide ceramic materials [11] [12] [13] And polymer/oxide-based materials. Polymers/metal oxides that have been used for anti-corrosion materials include PANI/SiO₂ [14], PANI/Fe₂O₃ [15], and PANI/ZrO₂ [12] [16] [17].

Polyaniline/ZrO₂ (PANI/ZrO₂) composite is a widely studied polymer/metal oxide composite material due to its unique properties and vast application potential. Compared to other polymer/metal oxide composites, PANI/ZrO₂ has superior corrosion resistance due to its enhanced adhesion and high thermal stability. This material combines the properties of polyaniline with ZrO₂. The reported research starts with synthesis, characterization, and application. PANI/ZrO₂ composite is generally synthesized using the in-situ polymerization method. Exploration of PANI/ZrO₂ composite properties includes characterization of thermal properties, electrical properties, and electrochemical properties. PANI/ZrO₂ composite is more thermally stable than pure PANI. This composite thermal stability increase is considered to originate from the interaction between PANI and nano-ZrO₂ [18] [19]. Other research results show that based on TGA data analysis, the thermal stability of PANI/ZrO₂ is higher than PANI. The conductivity of PANI/ZrO₂ composite is around 11.27 S/cm, which is typical of semiconductors [20].

The corrosion resistance test shows that PANI/ZrO₂ shows better corrosion inhibition efficiency [16]. The application of a steel coating coated with PANI/ZrO₂ composite has a corrosion rate value in between, which is lower than paint-coated steel but higher than ZrO₂-coated steel [21] [22]. In this study, the utilization of PANI/ZrO₂ composite material as an additive for anti-corrosion coating paint will be developed.

As is known, ZrO₂ from previous studies has different phases, namely amorphous (a), tetragonal (t), monoclinic (m), and a mixture of tetragonal-monoclinic (tm). Then, the electrical properties of PANI/a-ZrO₂, PANI/m-ZrO₂, PANI/t-ZrO₂, and PANI/tm-ZrO₂ composites have also been studied [16]. While previous studies have focused on the electrical properties of PANI/ZrO₂ composites, this study specifically aims to determine the effect of ZrO₂ phase variations (tetragonal, monoclinic, and tetragonal-monoclinic) on the corrosion inhibition performance of PANI/ZrO₂ composites when used as anticorrosive coating additives. Previous studies that discuss the effect of various ZrO₂ phases on the corrosion inhibition efficiency of PANI/ZrO₂ when applied as paint additives are still very limited. Therefore, this study needs to be conducted.

Experimental Method

PANI/ZrO₂ composites were prepared using the in-situ polymerization method, where the polymerization of aniline monomers occurs together with the presence of ZrO₂. The ZrO₂ used includes three phases, namely tetragonal (t), monoclinic (m), and tetragonal-monoclinic (tm). The detailed synthesis and characterization of PANI/polymorphic-ZrO₂ has been reported in previous research [23]. This PANI/ZrO₂ composite is in the form of dry sediment, then made into powder and sieved using a 400 mesh sieve.

Furthermore, the substrate specimen made of ST42 steel was formed into a cylinder with a diameter of 20 mm and a thickness of 5 mm as seen in Figure 1. The surface of the steel to be painted was then smoothed with sandpaper with grades 300, 600, and 1000. While the other side was connected with a cable using solder, this was intended as a connection to the corrosion test tool. Furthermore, all sides other than the surface coated with paint were wrapped with resin.

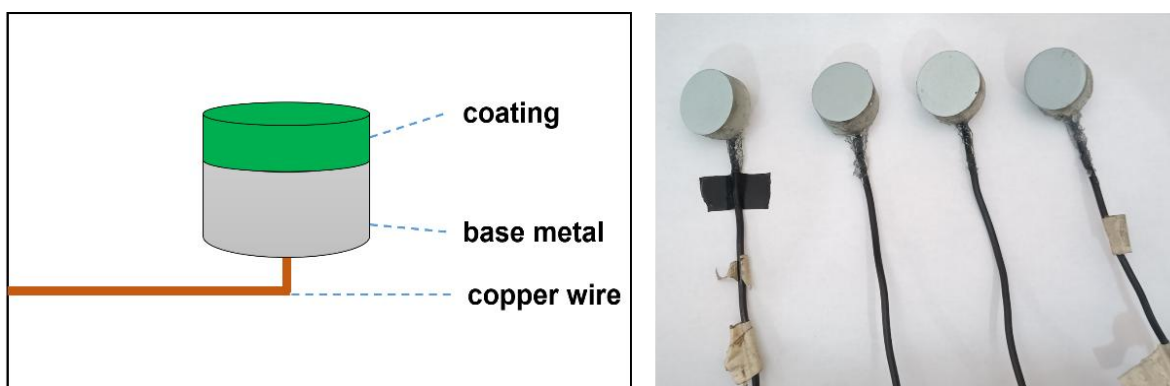


Figure 1. Test specimen

The next step is mixing the base coat (epoxy) with PANI/m-ZrO₂, PANI/t-ZrO₂, and PANI/tm-ZrO₂ materials with a specific ratio. Painting is done using the spray coating method. Painting is done in an optimal environment with a room temperature of 30°C, relative humidity of 40%, and good air circulation. The paint drying time is 24 hours. The sample list is shown in Table 1. After the paint is dry, the sample is tested for corrosion in a 3.5% NaCl solution according to ASTM G 44. Testing is carried out on days 1, 7, 14, and 21 of immersion. This corrosion test uses a three-electrode cell connected to a potentiostat/Galvanostat brand Gamry Reference 600 and a set of computers as data processors, as shown in Figure 2. The data obtained are then analyzed using Gamry Echem Analyst software, a Tafel plot.

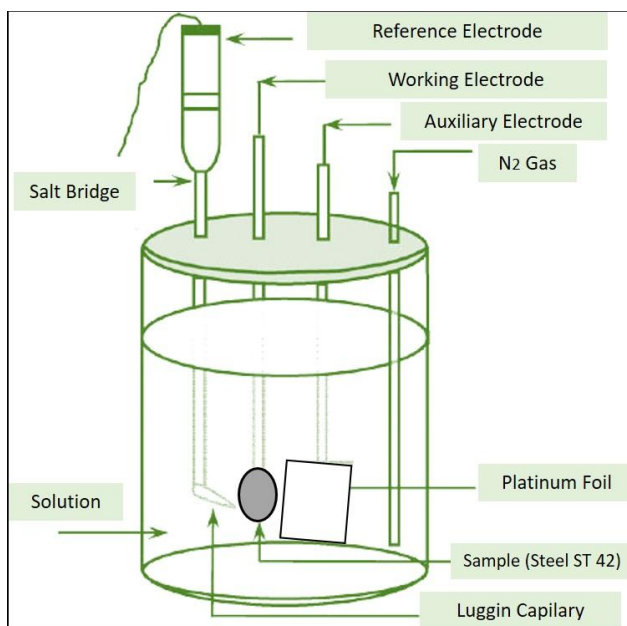


Figure 2. Test instrument for three-electrode potential cell polarization

Table 1. Coating composition on the sample

No.	Sample code	Annotation
1.	ST0	ST 42 without paint
2.	STC	ST 42 + paint
3.	STCM2	ST 42 + paint + PANI/2,5% <i>m</i> -ZrO ₂
4.	STCM5	ST 42 + paint + PANI/5% <i>m</i> -ZrO ₂
5.	STCM7	ST 42 + paint + PANI/7,5% <i>m</i> -ZrO ₂
6.	STCM10	ST 42 + paint + PANI/10% <i>m</i> -ZrO ₂
7.	STCT2	ST 42 + paint + PANI/2,5% <i>t</i> -ZrO ₂
8.	STCT5	ST 42 + paint + PANI/5% <i>t</i> -ZrO ₂
9.	STCT7	ST 42 + paint + PANI/7,5% <i>t</i> -ZrO ₂
10.	STCT10	ST 42 + paint + PANI/10% <i>t</i> -ZrO ₂
11.	STCTM2	ST 42 + paint + PANI/2,5% <i>tm</i> -ZrO ₂
12.	STCTM5	ST 42 + paint + PANI/5% <i>tm</i> -ZrO ₂
13.	STCTM7	ST 42 + paint + PANI/7,5% <i>tm</i> -ZrO ₂
14.	STCTM10	ST 42 + paint + PANI/10% <i>tm</i> -ZrO ₂

Result and Discussion

Corrosion resistance of paint coating-PANI/*m*-ZrO₂

The first additive used in metal primers is PANI/*m*-ZrO₂, a composite material with PANI matrix and monoclinic ZrO₂ filler. The mixture of paint-PANI/*m*-ZrO₂ shows varying corrosion resistance depending on the composition of *m*-ZrO₂, as shown in Figure 3.

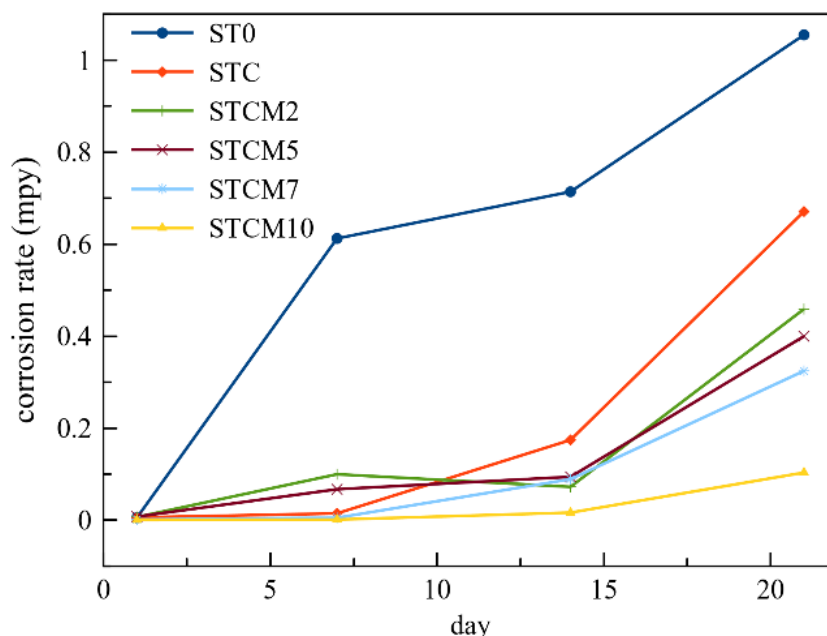


Figure 3. Corrosion rate of PANI/m-ZrO₂ paint coating

Based on Figure 3, it can be seen that the corrosion rate values in all samples increased, but the increase values varied. A high increase occurred in the ST0 sample, a metal substrate without anti-corrosion coating protection. The STC sample experienced a high increase in corrosion rate on days 14 to 21, caused by damage to the paint layer exposed to NaCl liquid, so the paint layer was not able to withstand the corrosion reaction that occurred. The STCM2 sample had the highest corrosion rate among the STCM5, STCM7, and STCM10 samples. This indicates that the percentage of m-ZrO₂ composition in the paint-PANI/m-ZrO₂ composite affects corrosion resistance. The higher the percentage of m-ZrO₂ composition, the better it inhibits the corrosion rate. However, further observations are still needed for m-ZrO₂ compositions above 10%.

The corrosion resistance of paint coating-PANI/t-ZrO₂

The next additive is PANI/t-ZrO₂, a composite material with PANI matrix and tetragonal ZrO₂ filler. The corrosion resistance of the PANI/t-ZrO₂ paint layer with various t-ZrO₂ compositions is shown in Figure 4. From the figure, it can be seen that there is a trend of increasing corrosion rate every day for each sample tested. Samples with the addition of PANI/t-ZrO₂ have a lower corrosion rate value than uncoated steel (STO) and coated with paint only (STC); this shows that the addition of PANI/t-ZrO₂ can increase the corrosion resistance of the paint layer.

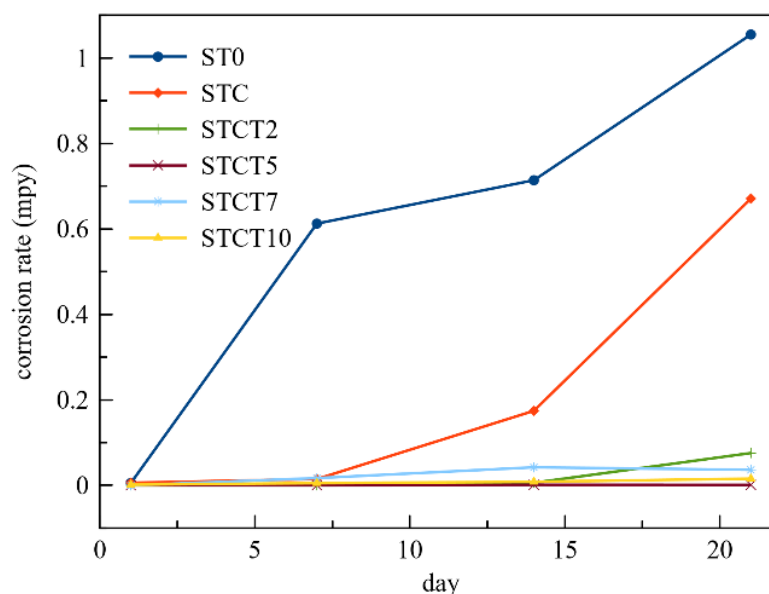


Figure 4. Corrosion rate of the PANI-ZrO₂ paint coating

The effect of composition variation also affects the effectiveness of corrosion protection provided by PANI/t-ZrO₂. STCT5 shows the best performance in protecting metals from corrosion. The relatively low corrosion rate indicates this compared to other compositions. STCT5 has an optimal balance between the protective effect of PANI and the barrier properties of ZrO₂, which provides effective protection against corrosive ion attacks. This composition can stabilize the passive layer and reduce the penetration rate of corrosive ions such as Cl⁻, which is the main cause of corrosion in NaCl environments. On the other hand, the results for the compositions of PANI/t-ZrO₂ 7% and 10% show higher corrosion rates, which are caused by the instability of the paint layer or the decrease in the effectiveness of the interaction of PANI and t-ZrO₂ at higher concentrations. With the addition of PANI/t-ZrO₂ 7% and 10%. At these concentrations, although the corrosion protection increases compared to the paint sample without any addition, the effectiveness is not as high as at the concentration of STCT 5. This is likely due to the more significant agglomeration of ZrO₂ at high concentrations, thereby reducing the effectiveness of protection. At higher concentrations (7.5% and 10%), PANI/t-ZrO₂ particles are likely to tend to agglomerate [24], which reduces the effective surface area of the particles interacting with the polymer matrix and creates a preferential pathway for penetration of corrosive ions [25]. As a result, the protection provided is reduced when compared to the 5% composition.

Corrosion resistance of paint coating-PANI/tm-ZrO₂

Another additive used as a paint mixer is PANI/tm-ZrO₂, a composite material with a PANI matrix and ZrO₂ filler with a tetragonal-monoclinic mixed phase. The results of the corrosion resistance test of the PANI/tm-ZrO₂ paint coating are shown in Figure 5. The STCTM2 sample has the highest corrosion rate among the STCTM5, STCTM7, and STCTM10 samples. However, the corrosion rate of the PANI/tm-ZrO₂ coating remains lower than that of the paint coating alone.

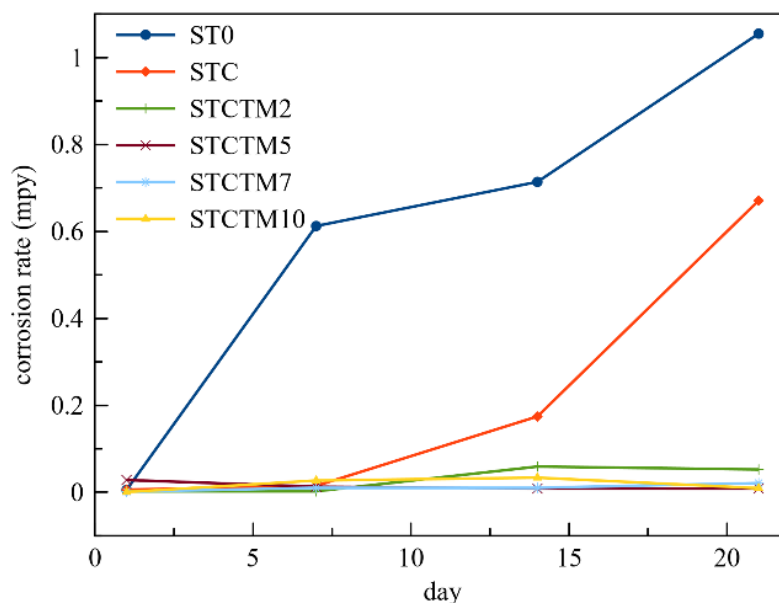


Figure 5. Corrosion rate of the PANI/tm-ZrO₂ paint coating

In contrast to the trend of corrosion rate on the PANI/m-ZrO₂ paint layer, where the higher the composition percentage, the better the ability to inhibit corrosion rate [26], the PANI/tm-ZrO₂ paint layer does not show a regular pattern. Samples STCTM5 and STCTM7 showed stable corrosion resistance, indicated by a relatively small increase in corrosion rate.

Effect of ZrO₂ polymorphism on corrosion resistance

The effect of ZrO₂ polymorphism on the corrosion resistance of the PANI/ZrO₂ paint layer can be seen from the comparison of the corrosion rate of the layer over a certain period, as shown in Figure 6. Based on the figure, it can be seen that the PANI/ZrO₂ paint layer with the monoclinic ZrO₂ phase tends to experience a faster increase in corrosion rate than other phases. The layer containing 5% and 7.5% t-ZrO₂ and tm-ZrO₂ has a relatively stable corrosion rate. The corrosion rate of PANI/ZrO₂ modified paint coating at 7.5% ZrO₂ is shown in Table 2. Overall, the layer containing tm-ZrO₂ has good corrosion resistance (low corrosion rate). This shows that ZrO₂ polymorphism affects corrosion resistance. Monoclinic crystals tend to have a cube shape with the same length and sides [25] [27]. This shape causes the tendency of PANI/m-ZrO₂ particles to be round. Tetragonal crystals tend to have a block shape with one side longer [17] [28]. This shape causes the tendency of PANI/t-ZrO₂ particles to be slightly oval [2]. This plays an important role in inhibiting the corrosion rate. An illustration of the role of ZrO₂ in inhibiting the corrosion rate is shown in Figure 7.

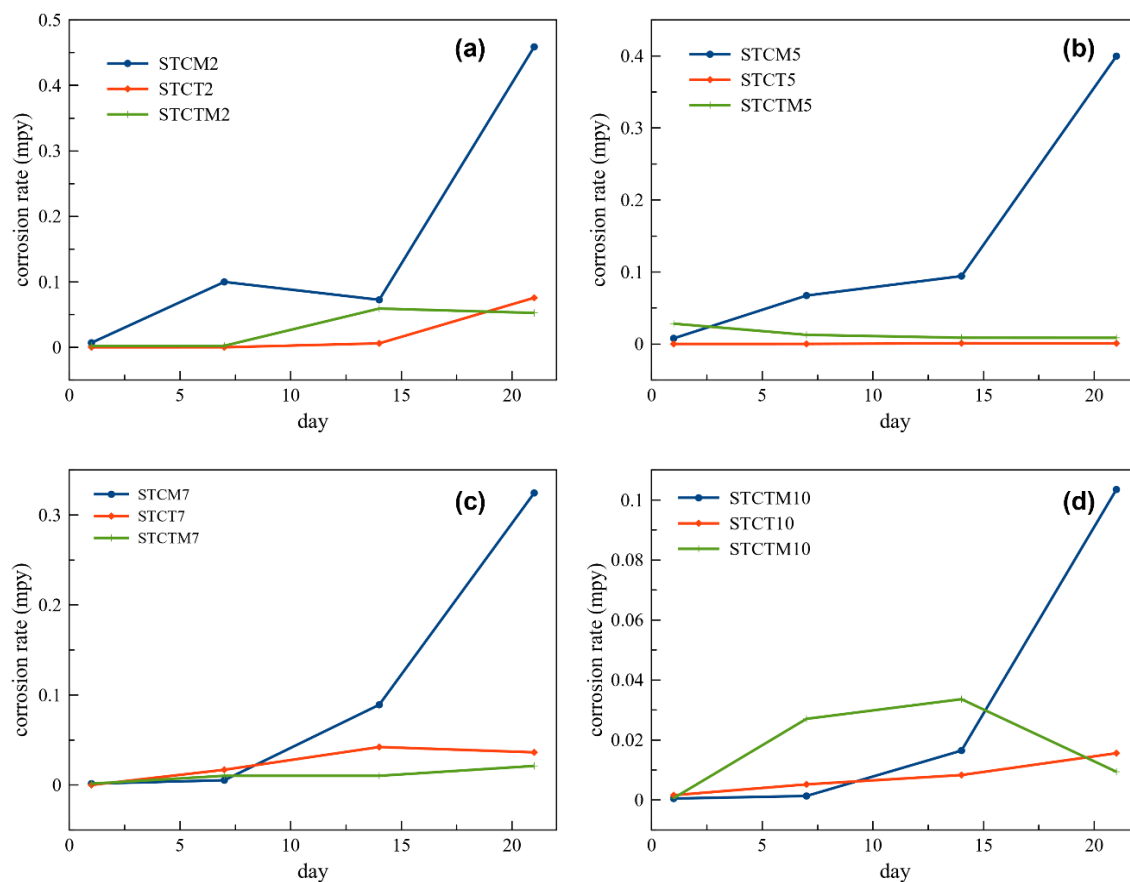


Figure 6. The corrosion rate of the PANI/ZrO₂ paint coating with ZrO₂ content: (a) 2.5%, (b) 5%, (c) 7.5%, and (d) 10%

Table 2. Corrosion Rate of PANI/ZrO₂ Coating (with 7.5% ZrO₂)

Day	Corrosion rate (mpy)			
	STC	STCM	STCT	STCTM
7	0.0148	0.0053	0.0169	0.0102
14	0.1744	0.0892	0.0422	0.0102
21	0.6710	0.3988	0.0364	0.0212

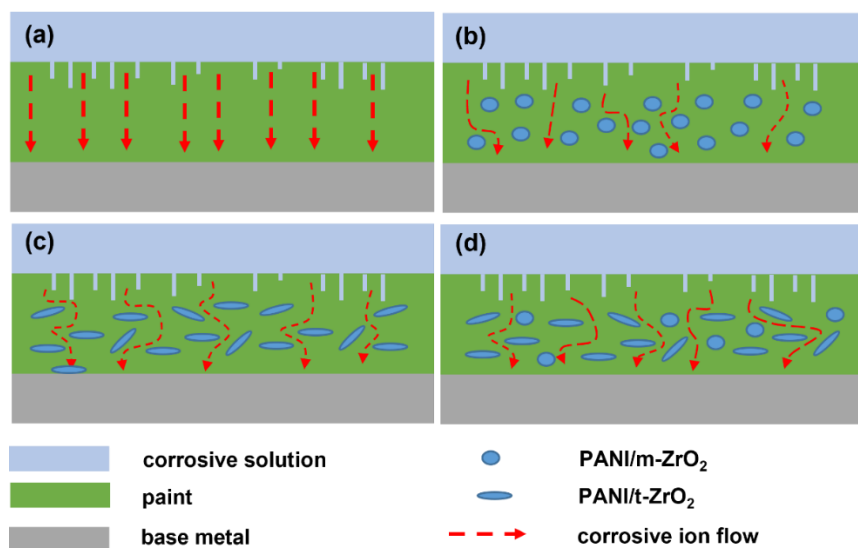


Figure 7. The corrosion protection mechanism of coatings: (a) pure paint, (b) paint-PANI/m-ZrO₂, (c) paint-PANI/t-ZrO₂, and (d) paint-PANI/tm-ZrO₂

Figure 7 (a) shows the relatively easier path of corrosive ions penetrating the paint layer. This is because no barrier particles are mixed with the paint, so the corrosive ion liquid penetrates straight through the paint layer. Figure 7 (b) explains the mechanism of corrosive ions penetrating the paint layer-PANI/m-ZrO₂. It appears that PANI/m-ZrO₂ functions as an inhibitor of the movement of corrosive ions penetrating the layer. Because PANI/m-ZrO₂ is spread across the layer, the path of the corrosive ion liquid has a winding path. Figure 7 (c) explains the mechanism of corrosive ions penetrating the paint layer with the addition of PANI/t-ZrO₂. It can be seen that PANI/t-ZrO₂ forms an inhibitor of the movement of corrosive ions penetrating the paint layer by forming a longer path than PANI/m-ZrO₂. Because there is PANI/t-ZrO₂ spread across the paint layer creates a winding and narrow path for corrosive ions to pass through, making the path of the corrosive ion liquid more difficult and hampered in penetrating the paint layer. Figure 7 (d) shows how corrosive ions penetrate the PANI/tm-ZrO₂ layer. This layer's tetragonal and monoclinic crystal structures create a more difficult path for corrosive ions to pass through. This mixed crystal form results in very few gaps, thus slowing the passage of corrosive ions to the metal surface. This mechanism illustrates how the PANI/tm-ZrO₂ layer effectively reduces the corrosion rate by blocking the penetration of ions that can cause damage to ST42 metal.

Conclusion

Adding PANI/polymorphic-ZrO₂ additives has been proven to increase the corrosion resistance of paint layers. The corrosion rate of the PANI/polymorphic-ZrO₂ paint layer is smaller than that of the pure paint layer. In sequence, corrosion resistance increases in paint layers that are given additional PANI/m-ZrO₂, PANI/t-ZrO₂, and PANI/tm-ZrO₂. The composition of polymorphic ZrO₂ affects corrosion resistance, where the compositions of 5% and 7.5% show relatively more stable performance. The mechanism of increasing corrosion resistance by PANI/polymorphic-ZrO₂ is related to its morphology, namely, by the formation of an inhibition of corrosive ion movement in the paint layer.

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