Influence of Silane-Post Treatment on Sodium Dodecylbenzene Sulfonate:Zinc Sulfate Pre-Treated Mild Steel and Its Effect on Adhesion Properties of Organic Coating

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Abstract: This paper describes the effect of post-treated mild steel substrates containing a sodium dodecyl benzenesulfonate:zinc sulfate (SDBS:ZnSO$_4$) inhibitor mixture as the first layer pre-treatment with 3-glycidoxypropyltrimethoxysilane (3- GPS) on the overall corrosion protection and adhesion of an epoxy protective coating. The protective layer was characterized by linear polarisation resistance measurement (LPR), electrochemical impedance spectroscopy (EIS), Fourier transform infrared spectroscopy (FTIR), and X-ray photoelectron spectroscopy (XPS) tests, while the adhesion of the epoxy coating to this pre-treated mild steel was evaluated by a pull-off test. The silane-post treated SDBS:ZnSO$_4$ mild steel showed excellent corrosion resistance in sodium chloride solution with good adhesion to the epoxy coating. However, the adhesion was highly dependent on the silane post-treatment methods. The increase in corrosion resistance and adhesion is due to increased surface passivation and the formation of strong interfacial interactions.

Keywords: *Silane, corrosion inhibitor, corrosion resistance, adhesion, mild steel*

1. Introduction

Organic coatings are frequently used to protect metal surfaces from corrosion by preventing aggressive ions from reaching the metal surface. However, due to imperfections such as micro pores, a low cross-link density area, or a high pigment volume concentration (PVC), organic coatings are often unable to offer long-term corrosion protection on their own [1]. These imperfections allow ions such as water, oxygen, and chloride to permeate the coating, creating a pathway that allows further electrolyte diffusion towards the coating/metal interface. As a result, corrosion cells will form underneath the coating, resulting in a premature adhesion failure [2].

Pre-treating the metal substrate with suitable chemicals could enhance the corrosion resistance and adhesion properties of the coatings. In the early stages, inorganic compounds like chromates were mainly employed. Unfortunately, because of the hazardous and carcinogenic properties that chromates impart, their use is currently prohibited [3].

In recent years, government and environmental bodies have pushed for the removal of those chemicals from the

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manufacturing process. As a result, the use of environmentally friendly chemical treatments such as organosilanes can be an alternative.

Organosilanes are the most commonly used precursors in sol-gel technology. There are various advantages to using sol-gel technology, such as low processing temperatures, the ability to cast a complex coating with a liquid precursor, and the ability to produce a thin film without machining or melting. Another benefit is that it does not introduce pollutants into the finished product, making it a waste-free approach.

Organosilanes have been widely used as a surface pre-treatment on metal [4, 5] or directly mixed into adhesive or coating [6]. These compounds have been shown to promote adhesion between organic coatings and metal substrates by providing high surface energy as well as chemical compatibility with the organic coatings [7, 8]. In principle, the silane molecules were adsorbed on the metal surface via hydrogen bonds between the silanols (-SiOH) and the hydroxyl groups (Me-OH), resulting in a strong chemical interaction [9–10].

Nowadays, pre-treatment with a silane solution is gaining popularity as an alternative to chromate conversion coating against metal corrosion. Commonly, corrosion inhibitors used in conjunction with silane coatings are able to effectively inhibit corrosion reactions on metal substrates [11–13]. Corrosion inhibitors form a protective layer on metal surfaces, creating physical barriers to the metallic interface, consequently reducing the corrosion reactions [14–16].

The latest research trend is towards promoting the use of non-toxic, economical, and more environmentally friendly corrosion inhibitors. Sodium dodecyl benzene sulfonate (SDBS) is an anionic surfactant that has been discovered to be effective as a non-toxic corrosion inhibitor [17–19]. This surfactant molecule contains both polar and non-polar functional groups that enhance its ability to be adsorbed on the metal surface. The molecules can be positioned at the interface so that the hydrophobic component of the molecule escapes the aqueous environment while the hydrophilic head group remains submerged. The adsorption of this hydrophobic compound on metal can prevent water from reaching the metal surface, reducing the corrosion of the metal.

Incorporating metal cations such as Zn^{2+} in surfactant inhibitors has significantly enhanced their inhibition properties [19]. Our latest studies suggest that combining SDBS with $ZnSO_4$ in a sodium chloride solution can minimise the rate of mild steel corrosion when compared to SDBS alone [20, 21].

In this work, a two-layer model was employed to pre-treat mild steel in order to increase corrosion protection and the adhesion properties of the organic coating. The mild steel was first pretreated with SDBS $ZnSO_4$ inhibitors and then post-treated with 3-GPS silane. The post-treatment with 3 GPS silane was performed by two methods: washing and immersion. The protective properties of the pretreated mild

steel substrate were investigated by electrochemical impedance spectroscopy (EIS) and polarisation measurements. The adhesion strength of the epoxy coating to pretreated mild steel was measured using a pull-off test. In addition, the surface chemistry of the pretreated mild steel was investigated by Fourier transform infrared spectroscopy (FTIR) and X-ray photoelectron spectroscopy (XPS).

2. Materials and Methods

2.1. Materials

The mild steel substrate used in this work has the following chemical composition (wt %): 0.60% Mn, 0.15% C, 0.03% P, 0.04% S, and 99.18% Fe. All mild steel substrates were ground with emery paper (grits 500, 800, 1200, and 2400), followed by cleaning twice with deionized water and acetone, and air dried before pre-treatment.

The pre-treatment formulation consisted of 200 ppm sodium dodecyl benzene sulfonate (SDBS) and 200 ppm zinc sulphate ($ZnSO_4$) [22] and 1.5% v/v 3 GPS, hydrolysed for 24 hours in a deionized water/ethanol 1/1 (v/v) mixture, and adjusted to pH 5.5 with acetic acid. All chemicals were purchased from Aldrich. The epoxy coating used for this study was a two-component epoxy resin with 50% solids content and polyamide hardener (PARALUX 4HG) provided by Kansai Paint Sdn. Bhd., Malaysia.

2.2. Surface Pretreatment

The mild steel substrate was immersed in a 200:200 ppm SDBS: $ZnSO_4$ bath solution for 24 hours [23]. Subsequently, the mild steel was dried with nitrogen gas and allowed to dry in a desiccator for an additional 24 hours before post-treatment with 3 GPS silane. The post-treatment was performed using two methods. In the first method, the mild steel was rinsed with a 3-GPS silane solution for 5 seconds before being air dried for 2 hours. In the second method, the mild steel was immersed in a 3-GPS silane solution for 20 minutes and then air dried for 2 hours. Subsequently, an epoxy coating was applied to the pretreated mild steel. Otherwise, some of the pretreated mild steel was stored in a desiccator for an additional 24 hours prior to surface characterization.

2.3. Epoxy Coating Application

A K Control Coater, KCC 101 coating applicator was used to apply a 3:1 v/v epoxy resin and polyamide hardener composition ratio to the mild steel substrates. They were allowed to cure for 24 hours at room temperature before being placed in a desiccator for at least three days before testing. The epoxy coating had a dry film thickness of 25 ± 5 µm.

2.4. Characterization

2.4.1. Electrochemical Test

All electrochemical measurements were performed with a three-electrode system that included a platinum counter electrode and an Ag/AgCl coupled to a luggin capillary as a reference electrode. A mild steel working electrode was prepared by embedding a mild steel substrate in epoxy resin and leaving 0.054 cm^2 exposed. Electrochemical impedance spectroscopy (EIS) was performed at the open circuit potential (OCP) with AC signals of amplitude 10 mV peak to peak over a frequency range of 10 kHz-20 mHz. All measurements were taken after 0.25, 1, 2, and 3 hours of immersion in a 0.05 M NaCl solution. Polarization measurements were taken with a scan rate of 1 mV/s from -100mV to +100mV vs. OCP. All electrochemical measurements were made with an Autolab PGstat302N potentiostat/galvanostat. The Nova software version 1.11 was used to analyse the data.

An electrical equivalent circuit (EEC) was used to analyse the experimental data of the EIS measurement [24]. Figure 1 presents the EEC used to fit the EIS data for this study. In this circuit, the solution resistance, charge transfer resistance, and double layer capacitance were denoted as R_s, Rct, and Cdl, respectively. A constant phase element (CPE) was introduced instead of a pure capacitor due to its non-ideal capacitive behaviour [25, 26].

The inhibition efficiency (η) was calculated from impedance parameters using (1):

$$\eta = (R_{cti} - R_{cto}) / R_{cto} \times 100 \qquad (1)$$

Where, R_{cti} and R_{cto} are the charge transfer resistance in the presence and absence of inhibitor.
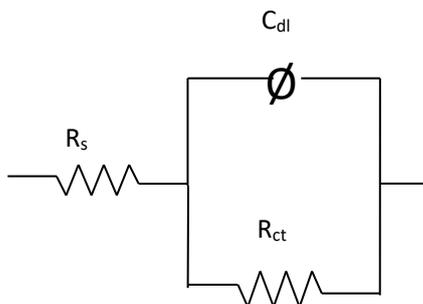
Figure 1. Equivalent Electrical Circuit (EEC) used to fit experiment impedance data

2.4.2. Surface Characterization

Thermo Nicolet 6700 Fourier Transform Infrared Spectroscopy (FTIR) was used to study the protective coating formed on the mild steel. All spectra were recorded in simple reflectance mode (ATR) using 36 scans with a resolution of 4 cm-1 in the 400–4000 cm-1 range. Otherwise, the surface binding energy was evaluated by X-ray photoelectron spectroscopy (XPS) model Omicron Nanotechnology with a Magnesium 1200 eV X-ray gun at a pressure of 10-10 torr.

2.4.3. Adhesion Test

The adhesion strength of epoxy coating on untreated and pre-treated mild steel was determined by a pull-off adhesion test as described in ASTM D4541 [27].

3. Results and Discussion

3.1. Electrochemical Measurements

Figure 2 and Figure 3 show the Nyquist and Bode diagrams for SDBS: ZnSO$_4$ pretreated mild steel that has been post-treated with 3-GPS either by washing or immersion methods. There was no significant difference in the EIS results, indicating that the silane post-treatment method had no effect on the overall protective properties of the mild steel.

The larger semicircle diameter in the Nyquist plots for both pretreated samples, either by washing or immersion, compared to untreated samples, demonstrated the effect of significant inhibition on mild steel corrosion processes. Furthermore, the high total impedance value at a low frequency confirmed the significant increase in corrosion resistance. The phase angle plots shifted to a higher frequency, also indicating increased overall corrosion resistance of the mild steel [22]. This is associated with the deposition of an insoluble layer of zinc hydroxide/oxide [21, 22]. It could also be related to zinc chelation with SDBS compounds. The hydrophobic properties of the SDBS compound could also prevent the penetration of electrolytes. This argument is confirmed when the results are compared with SDBS: ZnSO$_4$ pretreated mild steel without silane post-treatment (see Figure 4). The results indicated that the 3-GPS post-treatment has no significant effect on the overall corrosion protection of mild steel.
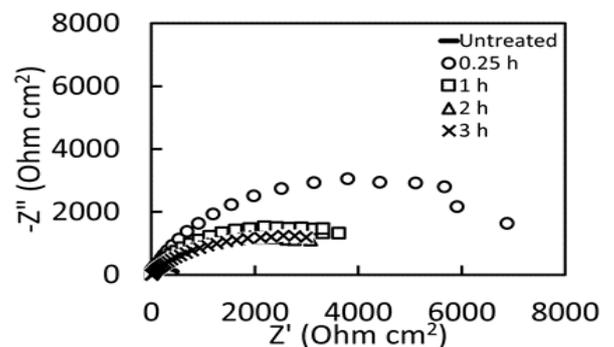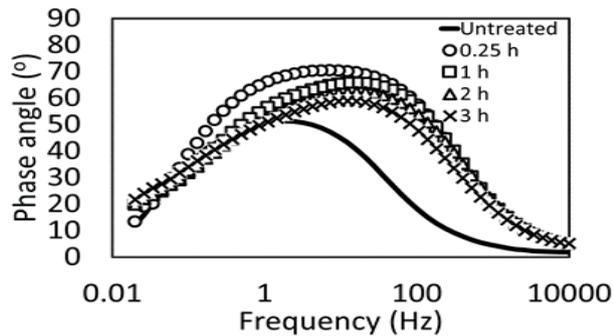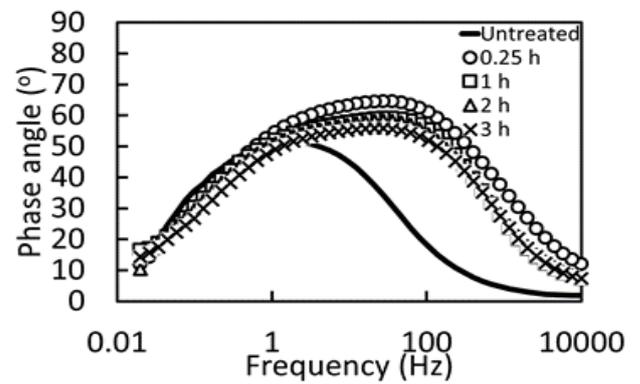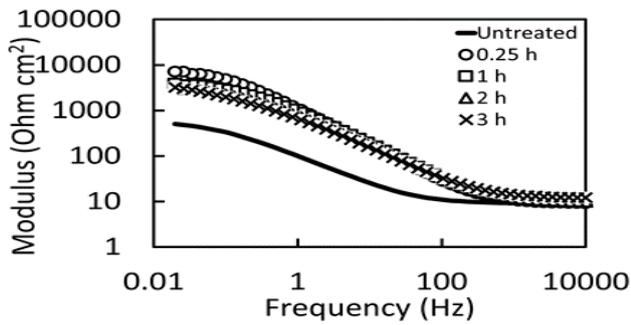
Figure 3. Nyquist and Bode diagrams for SDBS:ZnSO₄ mild steel pre-treated followed by 3-GPS post-treated (immersion) exposed in 0.05 M NaCl for 0.25, 1, 2 , and 3 hours

Figure 2. Nyquist and Bode diagrams for SDBS:ZnSO₄ mild steel pre-treated followed by 3-GPS post-treated (washing) exposed in 0.05 M NaCl for 0.25, 1 , 2 , and 3 hours
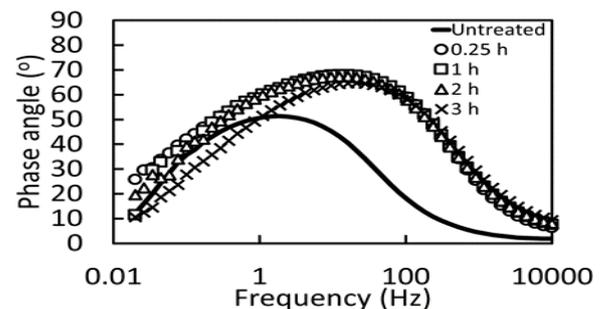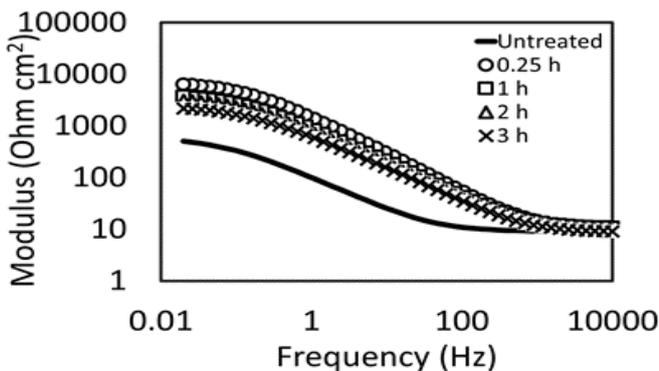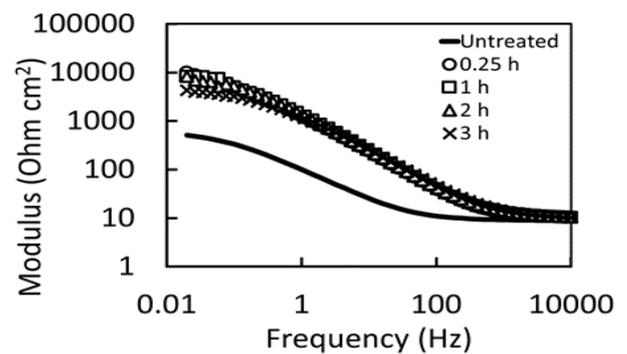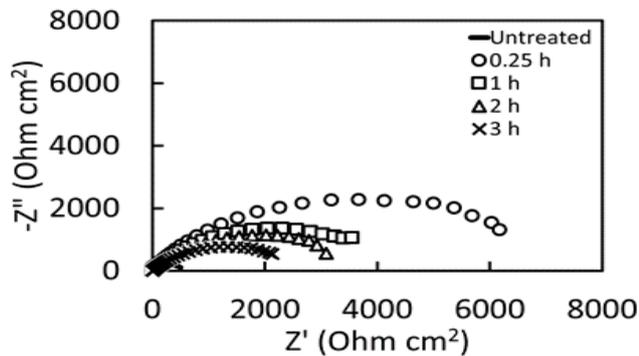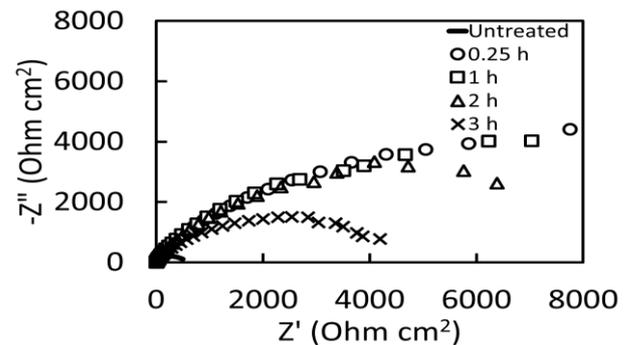
Figure 4. Nyquist and Bode diagrams for SDBS:ZnSO₄ mild steel pre-treated exposed in 0.05 M NaCl for 0.25, 1, 2 , and 3 hours

The EEC was used to fit the impedance experimental data, and the parameters extracted from the fitting are summarized in Table 1. Figure 5 plots the corrosion inhibition efficiency (η) over the time of exposure. The

results demonstrated that η does not increase significantly either by washing or immersion methods compared to the sample without post-treatment. This is thought to be due to the poor inhibition properties of 3-GPS silane as reported in the earlier works [13, 28].The η for both of the samples remained the same over the exposure time.

Table 1. Time dependence electrochemical parameters extracted from EIS analysis for untreated and pretreated mild steel in 0.5 M NaCl solution

Samples	Time (hours)	R_{ct} (Ohm cm^2)	C_{dl} (µF)	η (%)
Untreated	0.25	603	175	-
	3	603	176	-
SDBS:ZnSO$_4$ 3-GPS post treated by washing	0.25	7871	13	92
	1	4151	20	84
	2	3443	31	81
	3	3658	52	82
SDBS:ZnSO$_4$ 3-GPS post-treated by immersion	0.25	7603	9	91
	1	4580	17	86
	2	3937	19	83
	3	2767	24	76
SDBS:ZnSO$_4$	0.25	10692	14	94
	1	9742	12	93
	2	8316	11	92
	3	4299	10	85
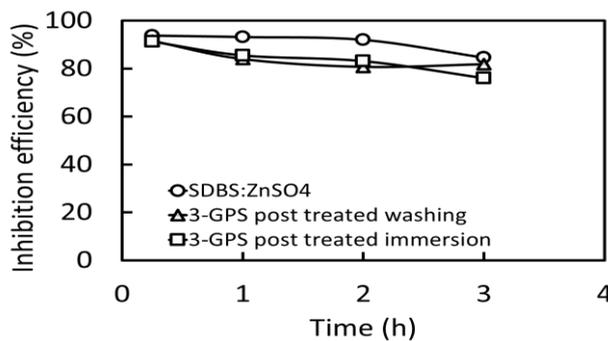
Figure 5. Inhibition efficiency of pretreated mild steel by SDBS:ZnSO$_4$ (a) no post treatement (b) 3- GPS post-treated-washing (c) 3-GPS post-treated -immersion

Figure 6 depicts the polarization curves of the pretreated mild steel by SDBS: ZnSO$_4$ followed by 3-GPS post-treatment, washing, and immersion methods. Consistent with the EIS results, higher corrosion resistance for the pretreated mild steel compared to the untreated mild steel was obtained. However, no differences were found in the polarization plots for the sample with post-treatment either by washing or immersion method. These samples retarded the mild steel's corrosion reactions equally. The protective coating of mild steel promoted a significant decrease in i$_{corr}$ compared to the untreated sample. As discussed earlier, zinc oxide/hydroxide precipitated on the

mild steel surface, thereby reducing the active metallic surface area. SDBS further hinders the penetration of aggressive electrolytes from reaching the mild steel surface. The reduction of both anodic and cathodic current density might be a reflection of blocking the anodic and cathodic reactions. Therefore, it retards both anodic dissolution and oxygen reduction reactions on mild steel [29, 30]. The E$_{corr}$ for the pretreated mild steel was shifted towards a more positive value compared to the untreated steel indicated that the retardation of the anodic reaction was more dominant.
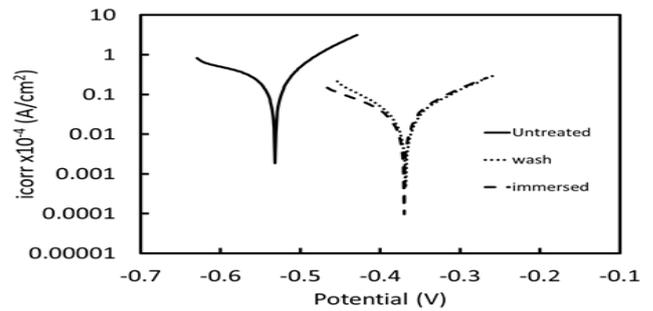
Figure 6. Polarization curves of mild steel samples (a) untreated and SDBS:ZnSO$_4$ pretreated (b) 3-GPS post treated-washing (c) 3-GPS post treated-immersion after three hours exposed in 0.05 M NaCl.

3.2. Surface Characterization

Figure 7 depicts the FTIR spectra of pretreated mild steel after 3-GPS post-treatment. The spectrum demonstrates that 3-GPS silane is not detectable on mild steel after post-treatment by washing. The spectrum obtained represents the SDBS: ZnSO$_4$ protective layer [22]. In contrast, by the immersion method, the spectrum revealed the presence of 3-GPS silane [13]. The broadband near 3400 cm^{-1} was related to the O-H stretching vibration of the adsorbed water and Si-OH bonds [5]. The characteristic bands of the epoxy ring were located at 750–900 cm^{-1} [31]. The region at 1000–1200 cm^{-1} can be assigned to the absorption of the siloxane group Si-O-Si [32].
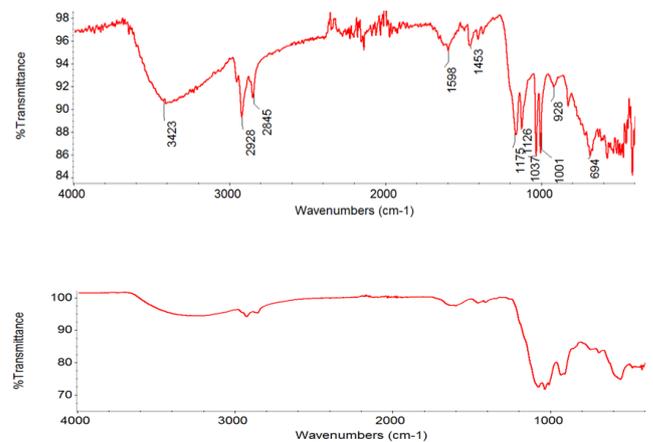
Figure 7. FTIR spectra on SDBS:ZnSO$_4$ pretreated mild steel post-treated by (a) 3-GPS silane washing (b) 3- GPS silane immersion method

Figure 8, Figure 9, and Table 2 depict the results for XPS analysis of pretreated mild steel. In the presence of C, O, and Zn binding energies, this confirms the deposition of inhibitors on mild steel. The appearance of a Zn 2p peak at a binding energy of 1022 eV and an O 1s peak at a binding energy of 531 eV are associated with zinc oxide and zinc hydroxide, respectively [5, 29, 33–34]. However, after being post-treated with 3-GPS silane by washing method, no Si binding energy was detected on mild steel. This result indicated that no 3-GPS silane was deposited on the surface, which was consistent with FTIR analysis. Otherwise, the Si binding energy was detected on pretreated mild steel after it was post-treated with 3-GPS via immersion. According to the findings, the post-treatment method is critical for obtaining the presence of a 3-GPS silane. 3-GPS silane can be absorbed through SDBS: $ZnSO_4$ pores during immersion to form bonds. On the other hand, when the washing method is used, the silane does not have the opportunity to interact and form bonds with the mild steel.

Table 2. The data extracted from deconvolution of the XPS spectra of the pretreated mild steel post-treated by 3-GPS silane

Spectrum	Compound	Washing Binding energy (eV)	Immersion Binding energy (eV)	References
C 1s	C-C	284.61	284.55	[35]
	C-O-C	-	286.11	[35]
O 1s	Zn-OH	531.21	531.72	[29]
Zn 2p	Zn-O	1022.21	1022.22	[29]
Si 2p	Si-O	-	102.02	[36]
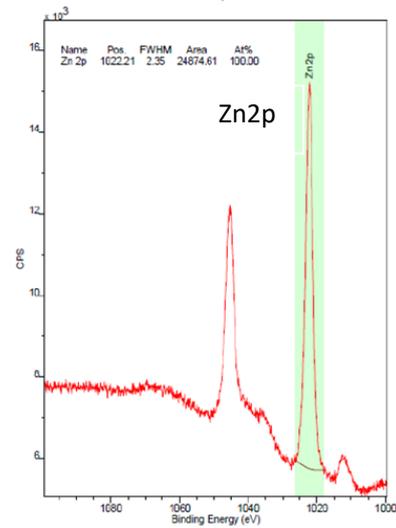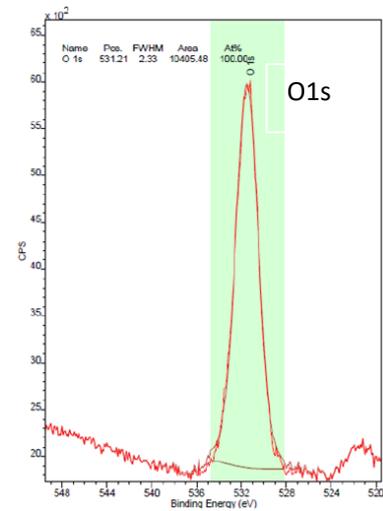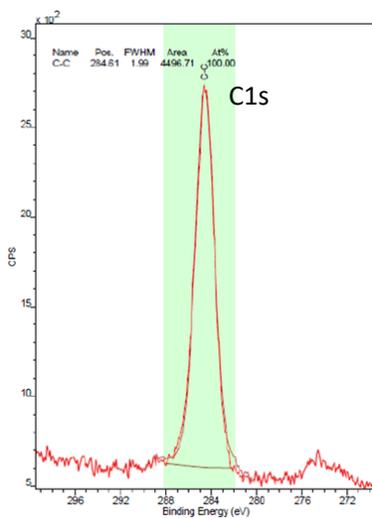
Figure 8. High-resolution spectra of C 1s, O 1s, Zn 2p, and their deconvolutions for sample 200:200 ppm SDBS:$ZnSO_4$ with 3-GPS post-treated (washing)
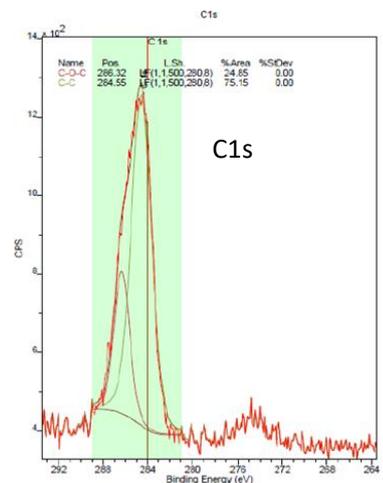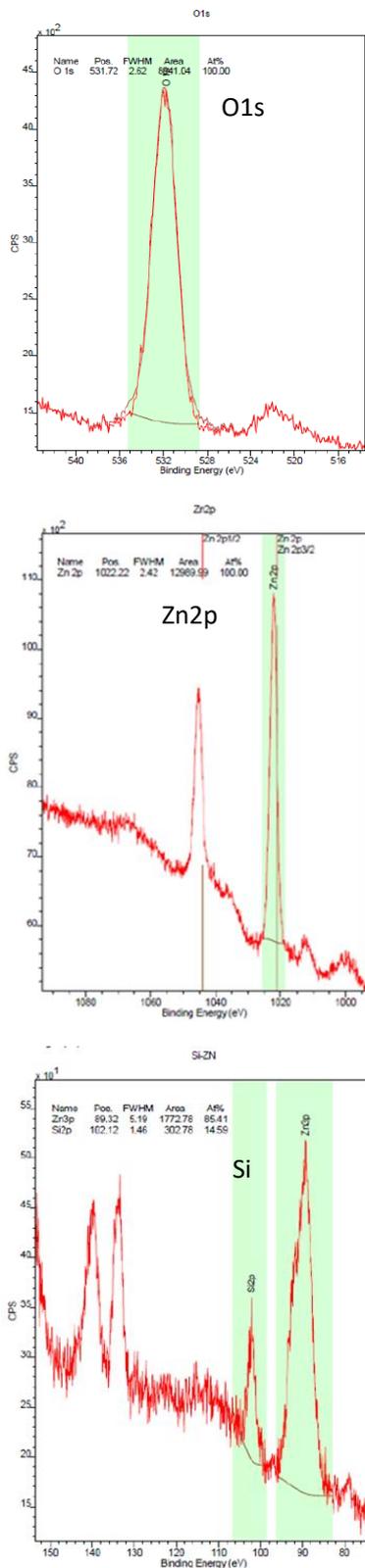
3.3. Adhesion Strength

Pre-treatment of the mild steel with SDBS: $ZnSO_4$ significantly reduces the bond strength of the epoxy coating, as shown in Figure 10. There may be no interaction between the epoxy coating and the protective coating to form a bond. This is because the protective coating is composed of zinc hydroxide/oxide and SDBS compounds, which are not able to react with the functional groups of the epoxy coating. On the other hand, post-treatment with 3-GPS silane by immersion method increased the adhesion strength of the epoxy coating. Silane may have penetrated the coating and formed a covalent bond with a mild steel substrate. Due to its functional groups, this 3-GPS silane also forms strong covalent bonds with the epoxy coating, resulting in improved adhesion strength. The deposition of 3-GPS silane was confirmed by XPS and FTIR analysis. However, when silane was applied via the washing method, the adhesion strength of the epoxy coating was not significantly increased. This was believed to be due to no 3-GPS silane depositing on the protective coating, as confirmed through XPS and FTIR analysis.

It has been shown that the post-treatment method is important for depositing a 3-GPS silane on the SDBS: $ZnSO_4$ coating. In contrast, when the silane is applied to bare mild steel, both washing and immersion methods yield no significant differences, as shown in Figure 11. On bare metal, 3-GPS silane applied by both methods increased the adhesion strength of the epoxy coating.
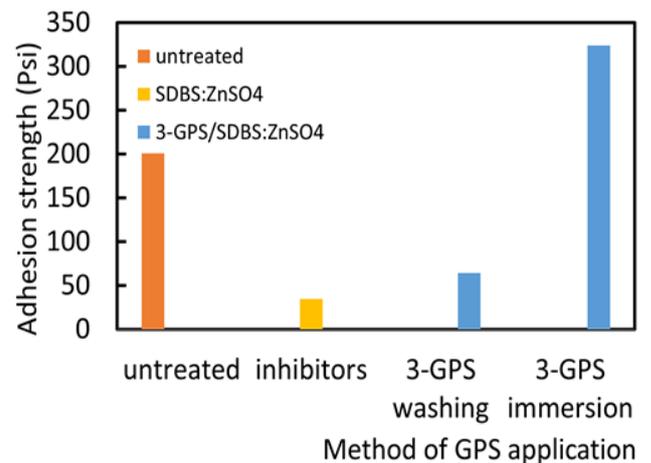
Figure 10. Dry adhesion strength of epoxy coating on (a) untreated (b) SDBS:$ZnSO_4$ inhibitors treated (c) post-treatment with 3-GPS silane by washing and immersion methods

Figure 9. High-resolution spectra of C 1s, O 1s, Zn 2p,Si and their deconvolutions for sample 200:200 ppm SDBS:$ZnSO_4$ with 3-GPS post-treated mild steel (immersion)
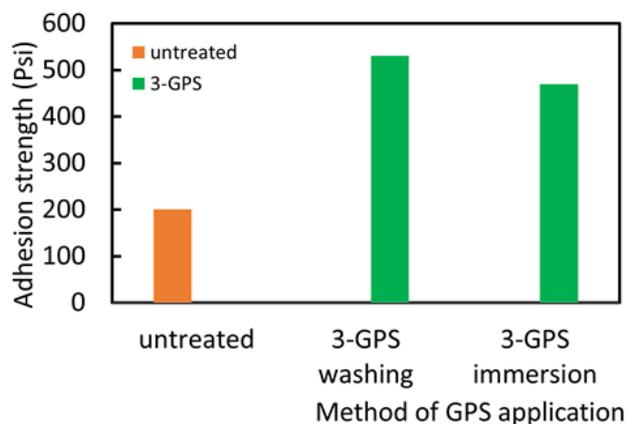
Figure 11. Dry adhesion strength of epoxy coating on mild steel (a) untreated (b) 3-GPS treated by washing method (c) 3-GPS treated by immersion method

4. Conclusion

The results demonstrated that the protective properties of SDBS: ZnSO$_4$ treated mild steel following post-treatment with 3-GPS silane were higher than those of untreated mild steel in sodium chloride solution. The formation of a protective layer composed of zinc hydroxide/oxide and SDBS compounds was responsible for the increase in corrosion resistance. The penetration of aggressive electrolytes was prevented by this protective layer from reaching the mild steel surface. As a result, it reduces the corrosion reaction of mild steel. The electrochemical barrier properties and corrosion resistance of mild steel were not affected by 3-GPS post-treatment methods.

On the contrary, the post-treatment method has a substantial impact on the adhesion strength of epoxy coating on mild steel substrates. The findings revealed that the presence of 3-GPS silane is highly dependent on the post-treatment method, which affects the adhesion strength of the epoxy coating. In this case, the immersion method enhanced the adhesion of the epoxy coating on the mild steel substrate. The presence of both of silane and inhibitors compounds is critical to enhancing the corrosion resistance and adhesion properties of organic coatings.

Acknowledgements

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