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# Effect of Incorporating MoS<sub>2</sub> in Organic Coatings on the Corrosion Resistance of 316L Stainless Steel in a 3.5% NaCl Solution

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**Abstract:** This study discusses a new coating method to protect 316L stainless steel (SS) from pitting corrosion in high chloride environments. The SS surface was coated using a simple, eco-friendly method, and sunflower oil (SunFO) was used as a base coating and binder for molybdenum disulfide (MoS<sub>2</sub>). The coated surface was observed using scanning electron microscopy (SEM) with an energy dispersive spectrometer (EDS) and X-ray diffraction (XRD). Corrosion behavior was examined by open-circuit potential (OCP) measurement and electrochemical impedance spectroscopy (EIS) in an 3.5% NaCl solution. The SunFO coating with MoS<sub>2</sub> showed the highest corrosion resistance and coating durability during the immersion time relative to the SunFO coating and bare 316L SS. The increased corrosion resistance is thought to be because of the interactions with the aggregations of the SunFO lamellar structure and MoS<sub>2</sub> in the coating film, which acted as a high order layer barrier providing protection from the metals to electrolytes.

**Keywords:** stainless steel; sunflower oil; molybdenum disulfide (MoS<sub>2</sub>); organic coating; corrosion resistance; electrochemical impedance spectroscopy (EIS)

## 1. Introduction

Austenitic stainless steel (SS) has excellent corrosion resistance in various environments, so they are used as materials in multiple applications such as pipelines, pumps, and structural steels in many industries [1]. Among them, 316L SS has good mechanical and welding properties with high corrosion resistance; therefore, it is regarded as one of the most effective materials in the field of corrosion [2,3]. The high corrosion resistance of 316L SS comes from a thin chromium oxide film which has high stability against corrosion [4]. In marine atmospheric environments, however, it often suffers from pitting-type corrosion due to exposure to stagnant seawater and through the deposition of airborne sea salts, which have high humidity and high chloride concentration [5]. When pitting corrosion is initiated, the pit propagates aggressively because the pH within the pit inside turns acidic due to the generation of CrCl<sub>3</sub>, which is called the autocatalytic mechanism [6]. Moreover, the pitting corrosion of SS could cause stress corrosion cracking (SCC) which results in unpredictable, brittle fracture [7]. For these reasons, corrosion protection of stainless steel in high chloride environments is a major concern in many industries. As such, several corrosion mitigation methods are being studied and developed in various fields.

Alkyd coatings are extensively used for the surface coatings as binders and adhesives. This class of coating is generated from polyols, dibasic and fatty acids or oils by condensation polymerization [8]. Alkyds have attracted significant attention among coating materials because they are lower cost and incur fewer film defects during applications. Recently, vegetable oils have been highlighted as a new effective organic coating that are nontoxic, nondepletable, domestically abundant, nonvolatile and a biodegradable resource [9]. The vegetable oils are triacylglycerols of fatty acids with high degrees of unsaturated sites which can have the ability to polymerize via cross-linking under certain conditions [10]. Polar molecules present in the oils can be absorbed on metal surfaces and form the corresponding metal oxides, which will enhance the stability of passivation and promote adhesion [9]. Sunflower oil (SunFO) coating is reported to be an effective inhibitor of corrosion for carbon steel, which is likely due to the lamellar-like layered structures of the organic film [9,11]. However, there are few studies surrounding the corrosion inhibition mechanism of the SunFO coating for SS and methods for synthesizing the SunFO coating using other effective materials. Therefore, in this study, the sunflower oil is selected as a base coating and binder for 316L SS.

The two-dimensional (2D) materials have been extensively researched within the context of several applications as coating materials because of their interesting, atomically thin, physical, chemical and electrical properties [12,13]. Among 2D materials, molybdenum disulfide (MoS<sub>2</sub>) has been widely applied as a lubrication and thin film protection coating materials because of its tribological and corrosion resistance properties [14,15]. Moreover, MoS<sub>2</sub> remains stable in various solvents and oxygenated environments, and it can also withstand high temperatures and pressures [16,17]. Currently, however, many surface modifications, such as chemical vapor deposition (CVD), water transfer are being phased out due to their high cost, lengthy processing time, low output and harmful effects on environments. This has motivated current research, which is being conducted to improve the stability and corrosion resistance with a mixture consisting of MoS<sub>2</sub> particles and several organic coating materials without imparting the toxic effects [18].

This study discusses a new coating method to protect 316L SS from the pitting corrosion when exposed to a 3.5% NaCl solution. Scanning electron microscopy (SEM) with energy dispersive spectrometry (EDS) was used to observe the dispersion of MoS<sub>2</sub> on the SS surface and cross section. In addition, X-ray diffraction (XRD) measurements were performed to evaluate the coating materials on SS. After that, the electrochemical properties of organic coatings according to MoS<sub>2</sub> were evaluated by using open-circuit potential (OCP) measurement and electrochemical impedance spectroscopy (EIS) tests.

#### 2. Materials and Methods

#### 2.1. Specimens and Solution Preparation

The chemical composition of the 316L SS is given in Table 1. For the electrochemical tests and the coating procedure, the surface of the specimen was polished with 2000-grit silicon carbide (SiC) paper, rinsed with ethanol and then dried with  $N_2$  gas.

Elements	Composition					
Fe	Balance					
С	0.03 Max.					
Cr	16-18					
Ni	10-14					
Mo	2–3					
S	0.03 Max.					

 Table 1. Chemical Composition of 316L stainless steel (wt.%).

Figure 1 shows a schematic diagram of the experimental procedures and expected coating structure. A 1 mg amount of  $MoS_2$  particles (commercially purchased, Sigma-Aldrich, St. Louis, MI, USA) was mixed with 10 mL of sunflower oil (commercially purchased) in 20 mL beakers. The mixture was sonicated and then stirred at 1000 rpm at room temperature for 3 h. Then, 100  $\mu$ L of the mixture was dropped on the SS substrate and heated at 275 °C for 10 min on a hot plate and slowly cooled. At this temperature, the triglycerides which is a main composition of sunflower oil undergo polymerization through oxidation [10]. The SunFO with MoS<sub>2</sub> coatings were presumed to have a layered oil structure which contained evenly distributed MoS<sub>2</sub> particles.



Figure 1. Schematic diagram of the experimental procedures and expected coating structure.

To observe the cross section of the coated specimens, the cross section was polished with 2000-grit silicon carbide (SiC) paper. All electrochemical experiments were conducted in a 3.5% NaCl solution at ambient temperature.

## 2.2. Surface Analysis

The surface morphology and the cross-sectional image of the specimens were observed using SEM/EDS (JSM-7600F, Jeol Ltd., Tokyo, Japan) to verify the dispersion of MoS<sub>2</sub> in organic coating. X-ray diffraction (XRD, D8 Advance, Bruker Co., Karlsruhe, Germany) measurements were performed on the specimens to identify the effective bonding within the organic coating, MoS<sub>2</sub> and 316L SS. The XRD analysis of the coated specimens was conducted to confirm the crystalline properties of the coating film using Cu K $\alpha$  radiation ( $\lambda = 1.54178$  Å) in the 2 $\theta$  range 0–60° at a scan rate of 0.017° 2 $\theta$ .

#### 2.3. Electrochemical Investigation Method

All electrochemical experiments were performed using a three-electrode system in a 1000 mL Pyrex glass corrosion cells connected to an electrochemical apparatus (VSP 300, Bio-Logic SAS, Seyssinet-Pariset, France). The test specimens were connected to a working electrode, graphite rods were used as the counter electrode and a saturated calomel electrode (SCE) was used as the reference

electrode. The exposed area of the test specimen to the electrolyte was  $0.25 \text{ cm}^2$  (0.5 cm × 0.5 cm). An initial open-circuit potential (OCP) was established within 3 h to carry out the entire electrochemical test. The electrochemical impedance spectroscopy (EIS, VSP 300, Bio-Logic SAS, Seyssinet-Pariset, France) was carried out with an amplitude of 20 mV in a frequency range from 100 kHz to 10 mHz. The EIS tests were performed at 3, 12, 36 and 63 h to investigate variations of the coating durability in corrosive media. The impedance plots were interpreted on the basis of equivalent circuits using a suitable fitting procedure through the ZSimpWin software (ZsimpWin 3.20).

## 3. Results

## 3.1. Surface Analysis

The SEM/EDS analysis is used to probe the morphologies of the SunFO coated SS. The addition of the pristine  $MoS_2$  in the SunFO significantly impacted the surface morphology. As shown in Figure 2, the SunFO with the  $MoS_2$  coating film shows the presence of a large number of  $MoS_2$  flakes which averaged an even layer 1–3 µm thick on the entire surface (Figure 2a–d). The EDS data indicated that the surface consisted of Mo, S, Cr, Ni, and Fe corresponding to particles of  $MoS_2$  and the surface of the SS (Figure 2e).



**Figure 2.** Surface morphology of sunflower oil (SunFO) with molybdenum disulfide (MoS<sub>2</sub>) coating on the stainless steel (SS) observed by SEM and EDS: (**a**)  $1000\times$ ; (**b**)  $3000\times$ ; (**c**)  $5000\times$ ; (**d**)  $10000\times$ ; (**e**) EDS results on the red spot.

Figure 3 shows cross-sectional images of SunFO with the  $MoS_2$  coating specimen. The thickness of the coating was verified at about 3–4 µm and shows uniform and dense surface features. According to the EDS mapping results (Figure 3b,c), it was revealed that the  $MoS_2$  particle existed in the SunFO coating layer. However, it was difficult to observe the layered structure of the oil, so the SEM analysis was re-conducted on an unpolished condition. In Figure 4, a layered structure was observed, and it was demonstrated that the coating structure of the specimen corresponded to the expectation outlined in Figure 1.

The XRD patterns in Figure 5 show the crystalline nature of the  $MoS_2$  and substrate. In previous reports, the diffraction pattern of bulk  $MoS_2$  has strong and sharp peaks which correspond to (100), (002), (100), (103), (105), and (110) planes [19]. After SunFO with  $MoS_2$  coating, the  $MoS_2$  peaks were only detected via the (002) peak corresponding to the angle of 15° because the  $MoS_2$  was oriented to a layered structure on the SS. The additional diffraction peaks at 44.5° and 19.6° indicate crystalline iron in the SS substrate and SunFO.



**Figure 3.** Cross sectional images of SunFO with MoS<sub>2</sub> coating on the SS observed by SEM and EDS: (a) 10000× (Cross-section of the specimen); (b) Fe observed by EDS mapping; (c) Mo observed by EDS mapping; (d) S observed by EDS mapping.



**Figure 4.** SEM images of unpolished SunFO with MoS<sub>2</sub> coating on the SS observed by SEM: (**a**) 1000×; (**b**) 3000×; (**c**) 5000×.



Figure 5. XRD results of (a) Bare SS; (b) SunFO coated SS; (c) SunFO with MoS<sub>2</sub> coated SS.

## 3.2. Electrochemical Analysis (EIS Tests)

The impedance spectra were obtained in the form of Nyquist plots of the data from the three-electrode system as shown in Figure 6. The tests were conducted with the following specimens, bare, SunFO coating and SunFO with a MoS<sub>2</sub> coating at room temperature. The specimens were tested in a 3.5% NaCl solution for a total of 63 h. The Nyquist plot consisted of a depressed capacitive loop at high frequency values [20,21]. As shown in Figure 6a, the capacitive loop of bare SS increased as time progresses, because the passive film on the surface goes to stable in the solution [22–24]. While, in case of the SunFO coated SS, the loops decreased with immersion time, which was caused by a deterioration of oil coating (Figure 6b) [25,26]. The capacitive loop of SunFO coating with MoS<sub>2</sub> on SS had little change during the test period, so it was regarded as a non-time-dependent effect in the solution as shown in Figure 6c.



**Figure 6.** Electrochemical impedance spectroscopy (EIS) results in the form of Nyquist plot in 3.5% NaCl solution; (**a**) Bare SS; (**b**) SunFO coated SS; (**c**) SunFO + MoS<sub>2</sub> coated SS.

In Figure 7a, the capacitive loop of bare SS was the smallest initially, however it displayed a reversed trend compared with the plot of the SunFO coated SS at the final time (Figure 7b). The SunFO coating with MoS<sub>2</sub> on the surface maintained the loop tendency during the entire immersion time. It can be also verified in the OCP graph seen in Figure 8. The SunFO with MoS<sub>2</sub> coating SS revealed very stable potential, which obviously differed from the bare and SunFO coated SS. Moreover, the potential of SunFO coated SS continuously decreased and reversed compared with the potential of bare specimen after about 28 h, which was the same tendency observed in the time-dependent variation of the Nyquist plot.



**Figure 7.** Time-dependent EIS results in the form of Nyquist plot in 3.5% NaCl solution: (**a**) initial stage (3 h); (**b**) final stage (63 h).



Figure 8. Open-circuit potential of specimens during the immersion time.

Figure 9 presents the EIS results in the form of Bode phase plots during immersion for 63 h. The Bode phase plot provided more clear description of the electrochemical frequency-dependent behavior than did the Nyquist plot, where the frequency values are implicit [27]. The high frequency spectra detects local surface defects, whereas the low frequency spectra detects the processes within the film and at the metal/film interface, respectively [28,29]. As shown in Figure 9a, in case of the bare SS, the phase angle maximum is slightly increased, and the width of the graph is wider in the low frequency region according to the test time. It means the passive film on the surface goes to more uniform and thicker [30–33]. In the case of SunFO coated SS, there was evidence of variations in surface conditions (Figure 9b). The initial stage of the specimen had two-time constants, but it changed to one-time constant at the final stage [34]. In addition, the Bode plot of SunFO coated SS has a low phase angle maximum and narrow shoulder width. This indicated that the SunFO and MoS<sub>2</sub> did not exhibit strong time-dependent behavior in the graphs, however it had a very wide frequency area as shown in Figure 9c. This is thought to be due the maintenance of a thick and uniform film throughout the entire test period.

Figure 10a shows the equivalent electrical circuits for bare SS, which had a passive film, and was used to analyze the results of the EIS tests (one-time constant circuit) [27]. In this figure,  $R_s$  is the solution resistance, *CPE*1 is the dielectric strength of the film and water absorbed by the film, and  $R_{film}$  is the electrical resistance resulting from the formation of an ionic conduction path through the pores in the film. The capacitance generated by the metal dissolution reaction and by the electric double layer at the electrolyte/substrate interface is designated by *CPE*2, and  $R_{ct}$  is the resistance caused by the metal dissolution reaction. In the case of SunFO coated specimens, however, more circuit parameters should be added because the oil coating generates a new layer which forms a two-layered film (two-time constant circuit) [39]. As shown in Figure 10b,  $R_{coating}$  is the SunFO coating resistance and *CPE*3 is the dielectric strength of the SunFO coating. The ZSimpWin program of the defined equivalent circuits was used to fit the EIS data to determine the optimized values for the resistance parameters, which are presented in Table 2. The  $R_{film}$  and  $R_{ct}$  of bare SS increased according to the immersion time. These values indicated an increase of the passive film's stability, which was the same tendency identified in the Nyquist and Bode plot. The film and coating capacitance ( $C_{film}$ ,  $C_{coating}$ ) is described by the expression [20,40]:

$$C = \frac{\varepsilon A'}{d} \tag{1}$$

where A<sup> $\prime$ </sup> is the surface area of specimen,  $\varepsilon$  is the dielectric constant, and *d* is thickness of the passive film in solution. This equation suggests that a decrease of  $C_{dl}$  is related to an increase of passive film's thickness. The  $C_{film}$  of bare SS decreased with time, therefore this indicated that the passive film on the surface becomes thicker at final stage. In the case of the SunFO coated specimen, there were too many error values when it was applied to a two-time constant circuit after the initial stage. As shown in the Bode plot, two-time constants were displayed only at the initial stage and one-time constant

was shown after that. In other words, the SunFO coating on the SS surface became degraded prior to 12 h. For this reason, the simulation was conducted to the point of one-time constant after the initial stage. According to the data shown in Table 2,  $R_{\text{film}}$  and  $R_{\text{ct}}$  decreased according to immersion time, which meant the film had deteriorated. Especially, the  $R_{\text{film}}$  and  $C_{\text{film}}$  rapidly decreased after 36 h which indicates that a thick and porous non-protective layer was generated on the surface [41–43]. The SunFO with MoS<sub>2</sub> coated SS showed an obvious two-time constant, which fit well with the two-time circuit. Both values,  $R_{\text{coating}}$  and  $R_{\text{ct}}$ , increased according to the test period, and the  $R_{\text{coating}}$  value at 63 h was markedly greater than the  $R_{\text{film}}$  of bare SS at the same time, which indicated excellent protective film properties. Moreover, the  $R_{\text{film}}$  value remained very small relative to that of bare SS. It is presumed that the SunFO coating with the MoS<sub>2</sub> blocks the electrolyte and oxygen so that the passive film could not form.



**Figure 9.** EIS results in the form of Bode plot in 3.5% NaCl solution: (**a**) Bare SS; (**b**) SunFO coated SS; (**c**) SunFO + MoS<sub>2</sub> coated SS.



Figure 10. An equivalent circuit used to fit the results of the EIS tests: (a) one-time constant circuit; (b) two-time constant circuit.

Specimen	Time	$R_{\rm s}~(\Omega~{ m cm^2})$	CPE1 (C <sub>film</sub> ) (F/cm <sup>2</sup> )	$n_1$	$R_{ m film}$ ( $\Omega$ cm <sup>2</sup> )	CPE2 (C <sub>dl</sub> ) (F/cm <sup>2</sup> )	<i>n</i> <sub>2</sub>	$R_{\rm ct}$ ( $\Omega \ {\rm cm^2}$ )	<i>n</i> <sub>3</sub>	CPE3 (C <sub>coating</sub> ) (F/cm <sup>2</sup> )	$R_{\text{coating}} (\Omega \text{ cm}^2)$
Bare	3	118.9	$1.37 \times 10^{-5}$	0.92	$9.85 \times 10^4$	$2.06 \times 10^{-6}$	0.37	$1.01 \times 10^{6}$	-	-	-
	12	118.2	$1.37 \times 10^{-5}$	0.91	$5.65 \times 10^{5}$	$1.88 \times 10^{-6}$	0.61	$1.83 \times 10^{6}$	-	-	-
	36	119.5	$1.34 \times 10^{-7}$	0.91	$3.91 \times 10^{6}$	$3.74 \times 10^{-6}$	0.80	$2.14 \times 10^6$	-	-	-
	63	120.1	$4.73\times10^{-6}$	0.91	$2.12 \times 10^7$	$7.61\times10^{-6}$	0.83	$2.12 \times 10^7$	-	-	-
SunFO	3	120.4	$7.72 \times 10^{-7}$	0.93	$1.22 \times 10^4$	$1.88\times10^{-6}$	0.83	$6.33 \times 10^{7}$	0.80	$2.31  imes 10^{-4}$	$4.38 \times 10^{-6}$
	12	127.6	$2.39 \times 10^{-6}$	0.88	$3.19 \times 10^{4}$	$3.61 \times 10^{-6}$	0.77	$4.88 \times 10^{5}$	_	-	-
	36	134.1	$4.55 \times 10^{-7}$	0.86	181.5	$7.39 \times 10^{-6}$	0.91	$1.58 \times 10^{6}$	-	-	-
	63	126.9	$7.66\times10^{-8}$	0.81	427.7	$4.80\times10^{-6}$	0.94	$9.05 \times 10^5$	-	-	-
SunFO + MoS <sub>2</sub>	3	135.7	$4.80  imes 10^{-5}$	0.56	223.5	$6.20 \times 10^{-7}$	0.97	$1.01 \times 10^4$	0.90	$5.12 \times 10^{-6}$	$2.836 \times 10^{9}$
	12	135.1	$5.05 \times 10^{-5}$	0.56	238.5	$4.65 \times 10^{-7}$	1	$1.48 \times 10^{4}$	0.90	$5.29 \times 10^{-6}$	$3.05 \times 10^{10}$
	36	141.4	$3.76 \times 10^{-5}$	0.63	6742	$4.50 \times 10^{-5}$	0.77	$3.48 \times 10^{7}$	0.89	$4.96 \times 10^{-6}$	$1.34 \times 10^{14}$
	63	140.1	$4.52 \times 10^{-5}$	0.60	6855	$2.69 \times 10^{-5}$	0.98	$1.24 \times 10^{9}$	0.89	$5.07 \times 10^{-6}$	$1.19 \times 10^{16}$

Table 2. EIS results of specimens according to the coating materials.

The SunFO is a complex mixture of triacylglycerol consisting of tri-esters of glycerol and fatty acid [44,45]. At high temperature, the unsaturated fatty acid of SunFO undergoes oxidation reactions and cross links with the SS. According to the XRD results (Figure 5), the SunFO layer has crystalline peaks at 19.6° which means the liquid states of the fat were crystallized on the SS. According to the expected coating structure (Figure 1), the SunFO coating, which has fatty acid hydrocarbon chains, makes lamellar structures as identified by the subcell concept [9,11]. However, the lamellar structures of the SunFO are aggregated structures that have a number of defects. In the SunFO with  $MoS_2$  coating film, the MoS<sub>2</sub> has layered structures that, during the coating process on SS, are well layered [8,9,11,46]. The interactions with aggregations of the SunFO lamellar structure and layered MoS<sub>2</sub> in coating the films acts as a high ordered layer barrier for the protection of the metals from electrolytes. The defects of both materials act as active centers for molecular adsorption and functionalization, therefore the combination of both layered materials could reduce the defects and build denser film [47]. Additionally, previous studies have reported that MoS<sub>2</sub> particles impart a negative effect on the corrosion because of their cathodic partial reactions during the corrosion process which leads to the destruction of the surface film [48]. Moreover, the electrochemical potential of  $MoS_2$  particle is higher than the 316L stainless steel, so it could generate galvanic cell between steel and MoS<sub>2</sub> which accelerates corrosion reaction on the steel surface [49]. However, in case of the MoS<sub>2</sub> with the SunFO film, the lamellar structure of the oil acted as an electrical insulating barrier between  $MoS_2$  and steel surface, which prevents the galvanic corrosion and improves the corrosion resistance.

## 4. Conclusions

This study evaluated the reinforcement effect of  $MoS_2$  in organic coatings on 316L SS in 3.5% NaCl solution. Firstly, SEM with EDS and XRD methods were performed to verify the  $MoS_2$  distribution on the SS surface. Through the experiments, the following results were drawn:

- The SEM images of SunFO with MoS<sub>2</sub> coating film showed the presence of a large number of MoS<sub>2</sub> flakes which averaged 1–3 μm evenly on the entire surface. The XRD results showed that the crystalline nature and orientation of the MoS<sub>2</sub> have strong and sharp peaks, which are the (002) peak corresponding to the angle of 15°.
- In the EIS results, the bare SS showed stable passive film generation during the test time. The SunFO with the MoS<sub>2</sub> coating on the SS surface showed the largest coating resistance and durability throughout the immersion time compared to SunFO coating and bare specimen. This is because the interaction with the aggregations of the SunFO lamellar structure and MoS<sub>2</sub> in coating film acted as a high order layer barrier to protect the metals against electrolytes.

Consequently, the incorporating  $MoS_2$  in organic coatings could considerably improve the localized corrosion resistance of stainless steel in high chloride environment.

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