Research Article

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Investigation of corrosion and thermal behavior of PU–PDMS-coated AISI 316L

Abstract: Polydimethylsiloxane (PDMS) is widely used from biomedical to industrial applications due to its nontoxic, hydrophobic, and transparent characteristics. PDMS has good thermal and adhesion properties; however, its mechanical properties are comparatively weak. Therefore, PDMS is blended with various polymers to effectively improve its mechanical properties. In this study, polyurethane (PU)–polydimethylsiloxane (PDMS) blended coatings of different concentrations were applied on the AISI 316L stainless steel surface. Their effects on corrosion and tribocorrosion properties were investigated in Ringer’s solutions. The blended polymer coatings were characterized using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS). The thermal properties of samples were examined by TGA and DSC. The surface images and cross-sectional were investigated using scanning electron microscopy (SEM). Tribocorrosion tests were carried out at open circuit potential (OCP). It was determined that hydrophobicity and thermal stability of polymer coating increased, while corrosion resistance slightly decreased with the increasing PDMS concentration in the polymer blended. The friction coefficient of blends decreased as the PU concentration increased. As a result, it was determined that the polymer-coated samples containing up to 50% PDMS prevented corrosive wear under the OCP wear test in Ringer’s solutions.

Keywords: polyurethane, polydimethylsiloxane, blends, contact angle, thermal behavior

1 Introduction

Polydimethylsiloxane (PDMS) is widely used from biomedical to industrial applications due to its advantages of being a transition polymer in between organic and inorganic polymers (1). PDMS is a non-toxic, highly hydrophobic, and transparent polymer (2). PDMS is used in biomedical industries due to its good blood compatibility, low toxicity, good thermal and oxidative stability, low modulus, and antiadhesive properties. PDMS exhibits mechanical properties similar to human tissue (elastic modulus of 2 MPa) (3). PDMS is used as a biomaterial in controlled drug release systems, cardiac medicine, drainage implants, artificial skin, contact lenses, oxygenators, medical adhesives, blood pumps, and catheters (4, 5). PDMS-coated materials are used in industrial applications because of their as anticorrosive, anti-icing, self-cleaning, antireflective properties (6, 7). The silicon–oxygen and silicon–hydroxide bonds of PDMS provide thermal stability, with strong adhesion to glass or metal surfaces (8).

PDMS has good thermal, adhesive, and corrosion-resistant properties, but it cannot precisely carry out its function due to its inadequate mechanical properties (9, 10). To improve the mechanic and wear properties of PDMS, various treatments such as blending, adding nanoparticles, bulk, and surface modification are applied (11–15). Among these, the blending method is the relatively simple, practical, and low-cost technique (16). PDMS is blended with various polymers to improve its poor mechanical properties. Polymer blending is the combination of at least two polymers to form a new material with different properties (17). Polymer blends exhibit various properties depending on the component’ amounts. These properties are a result of the mechanical, thermal, and chemical properties of each component (18, 19). Polyurethane (PU) is the most preferred material to improve the mechanical and corrosion-resistant properties of PDMS (20, 21). PU–PDMS blending is elastic, biocompatible, chemically inertness, and enhanced thermal and oxidative properties and can be used in surgical applications (22–24).
Studies investigating PU and PDMS blends as biomaterials can be found in the published literature (25). However, there is no study in the published literature investigating the corrosion and tribocorrosion of polymer-blended coatings consisting of PU and PDMS on AISI 316L stainless steel. Hence, this study fills an important deficiency of polymer-based biomaterials working under tribo-corrosion conditions. The working material exposed to various aqueous solutions or compounds is damaged over time. This situation based on solution–material interaction is expressed by tribocorrosion (26). Tribocorrosion is vital, especially in biomedical applications where both corrosion and mechanical wear occur. In this study, the effects of corrosion, tribocorrosion, and the thermal properties of different proportions of PU–PDMS blends on AISI 316L stainless steel were investigated.

2 Materials and method

In this study, AISI 316L stainless steel was used as the substrate material. PU Pelthane 2363-90A was obtained from Lubrizol. PDMS Sylgard 184 was supplied from Dow Corning in two components. Sylgard 184 PDMS has high crystallinity and viscosity, as well as nontoxic and inert properties. The molecular weight and density of PDMS are 27,000 and 0.982 g/mL, respectively. PU and PDMS were dissolved in a solution containing 20% (w/w) polymer in tetrahydrofuran (THF) + dimethylacetamide (DMAc). This solution was stirred for 1 day at 22°C. AISI 316L stainless steel samples were coated by dipping them into this solution. PU–PDMS blends of different ratios such as 75:25, 50:50, and 25:75 PU–PDMS were prepared to coat onto AISI 316L stainless steel. The prepared samples were left to dry at room temperature. The process was applied under vacuum conditions to avoid air bubbles in coatings. Figure 1 shows the schematic representation of the dip-coating process. FTIR measurements were carried out in the wavelength ranging from 400 to 4,000 cm⁻¹ MIR at a resolution of 2 cm⁻¹. The XRD patterns of polymer-coated samples were characterized by XRD diffraction (Panalytical Empyrean) with Cu-Kα. XPS measurements were conducted by Specs-Flex XPS equipment. The surface images and cross-sectional samples were analyzed with SEM equipment Zeiss Sigma 300. The potentiodynamic and electrochemical impedance spectroscopy (EIS) measurements of untreated and polymer-coated samples were investigated using a Gamry G750 potentiostat in Ringer’s solution. The chemical composition of Ringer’s solution is presented in Table 1.

A standard three-electrode cell was used for corrosion and tribocorrosion measurements, for which graphite was used as the counter electrode, Ag/AgCl was used as the reference electrode, and the sample was used as the working electrode. The potentiodynamic polarization curves were made at –1 to 2 V at a scanning rate of 1 mV/s over a surface area of 0.3848 cm². Tribocorrosion tests were performed in an electrochemical cell attached to the wear equipment. Tribocorrosion experiments were performed at a load of 3 N normal to the sample at a frequency of 1 Hz for 3,000 s under open circuit potential (OCP) conditions. Tribocorrosion tests were done with an 8 mm stroke length in an exposed area of 0.4 cm² in Ringer’s solution. The friction coefficient of samples was also recorded instantaneously in the tribocorrosion experiments. The static contact and the sliding angle of the polymer film were measured with an Attension Theta Flex contact angle device at the room temperature to evaluate hydrophobicity. The sample stage was rotated from 0° to 10° in the sliding angle test. Thermogravimetric (TGA) analysis was carried out using a Hitachi STA 7300...

Figure 1: The schematic representation of the dip-coating process. (a) Dipping, (b) formation of polymer layer, and (c) evaporation.
at a heating rate of 10°C/min from 25°C to 700°C under a nitrogen atmosphere. Differential scanning calorimetry (DSC) measurements were performed with DSCQ 2000 equipment at −90°C to 250°C with a heating and cooling rate of 10°C/min under a nitrogen atmosphere.

### 3 Results and discussion

#### 3.1 Characterization of polymer coatings

An FTIR graph for PU–PDMS coatings is shown in Figure 2. The adsorption peak at 3,321 cm⁻¹ represents the N–H urethane peak of PU. The intensity of the urethane peak decreased with the increasing PDMS content. The peaks between 2,950 and 2,970 cm⁻¹ correspond to the asymmetric Si–CH₃ bonds of PDMS. The peaks at 1,257 and 1,010 cm⁻¹ are attributed to CH₃ asymmetric deformation and Si–O–Si asymmetric deformation of PDMS. The spectrum of PU–PDMS blends shows 798 cm⁻¹ CH₃ release and Si–C cm⁻¹ vibrations (20). Polysiloxanes represent a big group of polymers with inorganic main chains. The surface properties of PDMS are a result of weak intermolecular forces between methyl groups as well as the strong Si–O bond and flexible Si–O–Si bond. PDMS shows nonpolar properties due to methyl groups outside the siloxane chain (21). Thus, PDMS shows excellent adhesion to metal surfaces due to these properties.

The XRD results of PU–PDMS polymer coatings are presented in Figure 3. It was observed that XRD peaks of PU and PDMS formed a wide range since polymeric structures were amorphous: 2θ = 12.5° PDMS peak and 2θ = 20.5° urethane peak of PU. XRD intensity peaks of PU–PDMS changed depending on the blending proportions. For the PU75–PDMS25 blend, the intensity urethane peak decreased and the PDMS peak began to appear. The PU50–PDMS50 blend formed equal urethane and PDMS peaks. As expected, the PU25–PDMS75 blend increased the intensity of the PDMS peak while decreasing the urethane peak significantly. The results indicated that as the PU content increased, the PDMS peak decreased. The XRD peaks of PDMS and PU were 2θ = 12.1° and 2θ = 20°, respectively, in the previous studies (27,28).

The crystallinity of PU, PU75–PDMS25, PU50–PDMS50, PU25–PDMS75, and PDMS are 62.8%, 64.9%, 69.7%, 71.1%, and 72.4%, respectively. It was revealed that the crystallization of the polymer increased with the increasing PDMS concentration in the coating due to the high crystallinity of PDMS. The XPS wide scan spectrum of PU–PDMS polymer blends is shown in Figure 4. Three main elements were detectable for neat PU and PDMS. The XPS spectra of PU are observed at 284, 399, and 532 eV, which are attributed to C 1s, N 1s, and O 1s, respectively. The peak positions of PDMS are O 1s at 532 eV, C 1s at 284 eV, Si 2s at 155 eV, and Si 2p at 102 eV. The presence of peak nitrogen and silicon indicates that PU and PDMS are effectively blended. As shown in Figure 4, a significant increase in silicon concentration and a decrease in carbon and nitrogen.

![Figure 2: FTIR graph of PU–PDMS coatings (a) from 3500 to 2600, and (b) from 1800–400.](image-url)
concentration occurred due to the increasing PDMS content in the blend (29–31).

3.2 Surface images

The cross-sectional views of AISI 316L coated with PU–PDMS polymers are shown in Figure 5. The thickness of the PU–PDMS polymer-coated sample layer was 45 µm according to the SEM images of cross-sectional view of samples. The surface view of AISI 316L coated with PU–PDMS polymers is shown in Figure 6. Homogeneous and uniform coatings were produced by the PU–PDMS blends. Figure 6 also shows irregular protrusions of PU, and it indicates that as the PDMS content increased, fracture structures were obtained. The SEM images showed that PU–PDMS blends had a porous structure on the surface, which might be due to the blend interface. It was also clear that there were irregular particles of various sizes on the surface of the blends. In Figure 6, these are particles containing silicon with a size of 1–2 µm. PU–PDMS-coated products attract a great deal of industry attention due to their versatile surface properties.

3.3 Contact angle measurements

The wettability and hydrophobicity of the polymer coatings were investigated using a water contact angle test. The contact angle of the PU–PDMS coatings on AISI 316L is shown in Figure 7. The contact angles of the PU and PDMS are 98° and 112°, respectively. The contact angles of PU75–PDMS25, PU50–PDMS50, and PU25–PDMS75 polymer blends are 101°, 107°, and 109°, respectively. The contact angle increased with the increasing PDMS content in the blend. These results showed that PDMS shows hydrophobic properties and is an effective barrier against water due to siloxane layers. Moreover, methyl groups on the outer surface of the PDMS support a hydrophobic structure and cause low surface energy at the polymer–air interface. The surface properties of PDMS have low surface energy due to the weak intermolecular forces between the methyl groups and the strong (Si–O) and flexible (Si–O–Si) siloxane chain (21). The water adsorption ability of PDMS is negligible due to its hydrophobic nature. Hydrophobic coated materials act as water repellents in aqueous environments. It prevents the adhesion of microorganisms by reducing the surface energy of polymeric coatings (32). However, hydrophobic coatings, especially from a biomedical point of view, cause surface contamination from organic solvents and proteins through the adsorption of small molecules due to its low wetting and heterogeneous surface charge (3). PDMS makes the surface hydrophobic by minimizing the surface energy between the substrate material and the coating. The Si–OH bond of PDMS provides strong bonding to glass or metal surfaces (8). It was determined that water contact angles of PDMS were 108° and 107°, respectively (33,34).
The static contact and the sliding angle of the PU–PDMS-coated AISI 316L steel are presented in Table 2. The sliding contact angles of PU and PDMS are 94° and 110°, respectively. The sliding angles of PU75–PDMS25, PU50–PDMS50, and PU25–PDMS75 polymer blends are 96°, 105°, and 107°, respectively. The sliding angle of polymer-coated samples decreased by 2°–5° compared to the static contact angle.

### 3.4 Potentiodynamic and EIS measurements

The potentiodynamic scans and Nyquist plots for AISI 316L coated with PU–PDMS polymers in Ringer’s solution are presented in Figure 8. Figure 8a shows that $E_{\text{corr}}$ values for all materials were very close to each other $-0.1 \, \text{V vs} \, V_{\text{ref}}$. The $I_{\text{corr}}$ untreated sample was $2.32 \times 10^{-4}$ (A/cm²). The $I_{\text{corr}}$ values of PDMS, PU25–PDMS75, PU50–PDMS50, PU75–PDMS25, and PU are $5.24 \times 10^{-5}$, $7.64 \times 10^{-5}$, $8.79 \times 10^{-5}$, $4.94 \times 10^{-5}$, and $1.20 \times 10^{-6}$ (A/cm²), respectively. All polymer-coated samples showed lower current density compared to the uncoated stainless steel. All polymer-coated samples improved the corrosion resistance properties of the substrate material and displayed a similar passivation behavior in anodic and cathodic regions. Figure 8b shows that the impedance spectra of all samples exhibited the semicircle which the diameter increased with the increasing PU concentration in the blend.
Bode plots for AISI 316 coated with PU–PDMS polymers is shown in Figure 9. Figure 9a shows that although the impedance value of all samples was high at low frequencies, the impedance value of samples decreased as the frequency increased. Polymer-coated samples had higher impedance untreated substrate material. While the phase angle had a high value at low frequencies, it decreased to 10^3 Hz and then increased again as the frequency increased as shown in Figure 9b. The polymer-coated materials acted as a barrier, which greatly improved the corrosion resistance of the substrate material. The PU-coated sample presented a better performance than the PDMS-coated sample, exhibiting lower corrosion density compared to that of the PDMS-coated sample. This indicated that the PU-coated sample increased the protection of the substrate material because of more oxidative resistance. The PDMS-coated sample exhibited lower corrosion resistance owing to its permeability. The corrosion resistance increased as the concentration of PU increased in the blend. This indicated that PU-coated materials improved corrosion resistance.

3.5 OCP wear

Tribocorrosion is very important in biomedical equipment, where there is mechanical loading and chemical interactions with the surrounding environment (36). OCP wear of AISI 316L coated with PU–PDMS polymers in Ringer’s solution is shown in Figure 10. The OCP wear test was conducted for 3,000 s. The samples were waited to stabilize for 1,000 s before and after the OCP wear test. The potential of all polymer-coated materials is approximately −0.15 V vs V_ref before and after the wear loading. When the wear started, the potential values of PU, PU75–PDMS25, and PU50–PDMS50 polymer-coated materials increased positively. This situation stably remained during the test. The results show that PU, PU75–PDMS25, and PU50–PDMS50 polymer coatings protected the surface of the substrate material. This indicated that the wear ball was not in contact with the substrate material. After unloading, the potential values of PU, PU75–PDMS25, and PU50–PDMS50 polymer-coated samples immediately decreased to their initial values. OCP results showed that PU, PU75–PDMS25, and PU50–PDMS50-coated materials can be used in biomaterial applications under wear-corrosion conditions. Potentiodynamic and OCP wear results clearly demonstrated that the PU-coated sample exhibited the best corrosion resistance and tribocorrosion properties.

The PU25–PDMS75 polymer coating exhibited different properties than others for OCP wear test. The PU25–PDMS75 coated sample increased positively first 300 s of the experiment, and then the potential value of this material suddenly dropped to −0.3 V vs V_ref. The decreased potential value of this coating gradually continued during wear. It showed that the polymer coating protected the surface for the first 300 s when the wear started. Then, the polymer layer was removed by the

<table>
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<th>Sample</th>
<th>Contact angle (°)</th>
<th>Sliding angle (°)</th>
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<tr>
<td>PU</td>
<td>98</td>
<td>94</td>
</tr>
<tr>
<td>PU75–PDMS25</td>
<td>101</td>
<td>96</td>
</tr>
<tr>
<td>PU50–PDMS50</td>
<td>107</td>
<td>105</td>
</tr>
<tr>
<td>PU25–PDMS75</td>
<td>112</td>
<td>107</td>
</tr>
<tr>
<td>PDMS</td>
<td></td>
<td>110</td>
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</table>

Table 2: The contact and sliding angles of PU–PDMS coated AISI 316L steel

Figure 8: Potentiodynamic scans and Nyquist plots for AISI 316L coated with PU–PDMS polymers in Ringer’s solution: (a) potentiodynamic and (b) Nyquist.
effect of the wear load, and the substrate material could not be protected. It gave a hint that mechanical wear has more effect on corrosion. This situation is clearly shown in Figure 11d, where the coating layer is broken due to the wear load. The potential value of the PU25–PDMS75 increases with the effect of repassivation when the wear ends. It is clear that friction coefficients of all polymer-coated samples showed oscillation due to stick-slip. The friction coefficients of PU, PU75–PDMS25, and PU50–PDMS50 polymer-coated samples are 0.2. The friction coefficient of the PU25–PDMS75-coated sample was slightly increased after 300 s when the coating layer was removed. The

Figure 9: Bode plots for AISI 316 coated with PU–PDMS polymers in Ringer's solution: (a) Bode magnitude and (b) Bode phase.

Figure 10: OCP wear of AISI 316L coated with PU–PDMS polymers in Ringer's solution.
PU25–PDMS75 polymer-coated material was 0.25. The friction coefficient of polymer-coated samples decreased with an increased concentration of PU in the blend.

OCP wear surface images of AISI 316L coated with PU–PDMS polymers in Ringer’s solution are shown in Figure 11. PU exhibited the lowest wear scar and the depth. The wear scar and the depth of the polymers significantly increased with incremental in the PDMS content. This can be attributed to the weakness of the PDMS mechanical properties (37). Furthermore, for all blends, it was observed that the wear volume increased with an increase in the PDMS concentration in the coating. The PU-coated sample showed better wear resistance compared to PU/PDMS blends. The wear mechanism of PU and PU75–PDMS25 polymer-coated material was abrasive. Figure 11d shows that the PU25–PDMS75 layer was broken due to the wear load. This can be attributed to the decrease in wear resistance in polymer wear resistance due to the weak mechanical properties of PDMS.

3.6 TGA, difference thermogravimetry, and DSC analysis

TGA, difference thermogravimetry (DTG), and DSC measurements were performed to determine the thermal properties of the polymers. TGA and DTG measurements of PU–PDMS polymers are shown in Figure 12. The TGA analysis of PU–PDMS polymers is shown in Figure 12a. PU showed almost no change in its weight up to 280°C and preserved its stability. PU weight decreased rapidly when the PU hard segment melted at 280°C, and this situation continued up to 390°C. The decrease in the amount of material up to 400°C in the PDMS is related to the loss of absorbed water (38). The weight of PDMS was sharply reduced in the temperature range from 400°C to 600°C. It shows depolymerization in the siloxane chains. The thermal degradation of PDMS is mainly related to Si–O’s heterolytic separation, where bonds interact significantly in the kinetic state and within the molecular structure (39).
The amount of thermal degradation of PDMS determines the intermolecular formation. PU–PDMS polymer blends are virtually nondegradable up to 280°C. The amount of the degraded substance increased as the amount of PU increased in the blend after 280°C. This situation is due to the low thermal stability of PU. PU showed two steps of degradation. The first weight loss at 330°C was due to the hard segment degradation, while the second weight loss at 390°C was due to the soft segment, as shown in Figure 12b. The hard segment's decomposition temperature is nearly the same in all blends, but the degradation temperature of the soft segment increased from 390°C to 420°C with the increasing PDMS content. This increase may be due to PDMS replacing the soft segment in the blend and the increased thermal stability. The thermal stability of PDMS provides silicon–oxygen bond strength.

The thermal stability of PU–PDMS polymer blends is presented in Table 3. The heat resistance index and residual weight were calculated from TGA measurements. The heat-resistant index temperatures of PU and PDMS are 153.97°C and 205.43°C, respectively. The heat resistance of the polymers increased with the increasing PDMS content. This increase is due to the silicon in the structure of PDMS. The residual weight of polymers increased with the increase in the PDMS content. This situation shows that PDMS considerably improved the thermal stability of the blends.

DSC measurement of PU–PDMS polymers and the glass transition temperature (T_g) and melting temperature (T_m) of PU–PDMS polymer from DSC analysis are presented in Figure 13 and Table 4, respectively. The glass transition temperature (T_g) of PU is −30°C. The glass transition temperature of PU50/PDMS50 decreased to −50°C with the effect of PDMS. An increasing concentration of PDMS moved the T_g of the polymers to lower values. The melting of the hard segment and the soft segment of PU are at 62.5°C and 150°C, respectively. The cold crystallization of PU occurred at 80°C. The melting of the soft segment of PU50–PDMS50 blending was not observed. These results showed that the PU and PDMS were chemically bonded. The melting of

Table 3: Thermal stability of PU–PDMS polymer blends

<table>
<thead>
<tr>
<th>Sample</th>
<th>T_d5 (°C)</th>
<th>T_d30 (°C)</th>
<th>T_d50 (°C)</th>
<th>Heat-resistant index (T_s) (°C)</th>
<th>Residual weight (%)</th>
</tr>
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<tr>
<td>PU</td>
<td>289.62</td>
<td>330.63</td>
<td>352.69</td>
<td>153.97</td>
<td>−</td>
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<tr>
<td>PU75–PDMS25</td>
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<td>347.66</td>
<td>404.51</td>
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<tr>
<td>PU50–PDMS50</td>
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<td>385.25</td>
<td>442.23</td>
<td>169.74</td>
<td>19</td>
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<tr>
<td>PU25–PDMS75</td>
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<td>398.01</td>
<td>472.81</td>
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<tr>
<td>PDMS</td>
<td>304.85</td>
<td>495.52</td>
<td>584.18</td>
<td>205.43</td>
<td>43</td>
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</table>

Table 4: The glass transition temperature (T_g) and melting temperature (T_m) of PU–PDMS polymer from DSC analysis

<table>
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<th>Sample</th>
<th>T_g (°C)</th>
<th>T_m (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>PU</td>
<td>−30</td>
<td>175</td>
</tr>
<tr>
<td>PU50/PDMS50</td>
<td>−50</td>
<td>165</td>
</tr>
<tr>
<td>PDMS</td>
<td>0</td>
<td></td>
</tr>
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</table>

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The properties of PU/PDMS polymer blends are presented in Table 5. The contact angle and the thermal stability of polymers increased with an increased concentration of PDMS in the blend. All the polymer coatings on stainless steel protected the substrate material very well in the corrosive environment. The PU coating showed the corrosion and tribocorrosion resistance. The polymer coatings containing up to 50% PDMS protected the substrate surface under tribocorrosion conditions.

4 Conclusion

The corrosion, tribocorrosion, and thermal properties of PU–PDMS blends on AISI 316L stainless steel were investigated in Ringer’s solution. The following results were obtained. The hydrophobicity and the thermal stability of coatings increased, whereas corrosion resistance of coating slightly decreased with the increased PDMS concentration in the blend. The wear volume and the friction coefficient of PU–PDMS coatings decreased with the increased PU concentration in the blend. EIS and potentiodynamic results showed that the PU-coated sample had better corrosion resistance than PU–PDMS blended coatings. PU, PU75–PDMS25, and PU50–PDMS50 polymer-coated samples protected the AISI 316L surface under the OCP wear test. However, PU25–PDMS75 coating could not protect the surface due to its weaker mechanical properties. As a result, it was determined that the polymer-coated samples containing up to 50% PDMS prevented corrosive wear under the OCP wear test in Ringer’s solutions.

Acknowledgments: The authors gratefully thank the Coordinatorship of Scientific Research Projects of Ataturk University for providing financial support (Project Code: FDK-2019-7078).

Funding information: This study was financially supported by Ataturk University, Coordinatorship of Scientific Research Projects (Project Code: FDK-2019-7078).

Author contributions: Muharrem Taşdemir: data analysis, paper writing, investigation, and methodology; Fatih Şenaslan: writing – review, editing; Ayhan Çelik: supervisor, paper writing.

Conflict of interest: The authors state no conflict of interest.

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