Measuring Performance of Paint Coating via Laboratory and Field Testing

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By
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Approval Page

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CAL POLY STATE UNIVERSITY
Materials Engineering Department

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Abstract

There is significant interest in the corrosive behavior of superhydrophobic surfaces because of their unique water-repelling and self-cleaning properties. Specifically, the petroleum industry is interested in their use for off-shore oil rigs. The purpose of this research was to investigate how the pigment concentration of coating affects the superhydrophobic property in marine environments. The tested urethane and epoxy coatings were multi-layered systems with epoxy primer. The tested single layer system was polyurethane coatings. The superhydrophobic samples were prepared by coating diatomaceous earth particles with hydrophobic coating on carbon steel panels. Samples were scribed and tested in QUV weathering tester for 1000 hours outlined by ASTM Standard D4587-11 and in salt fog chamber outlined by ASTM Standard B117-11. They were also placed at the Cal Poly pier for atmospheric exposure. In the accelerated weathering test, single layered coating systems outperformed the multi-layered systems in general. Epoxy coating systems also showed a significant amount of shrinkage and sigmoid flaking. In Q-fog test, PU SSC7 and PU SSC10 outperformed the rest of 2-layer PU-EP coatings.
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I. Introduction

Corrosion is a major problem throughout the world that causes serious structural damage in buildings, bridges, offshore structures, pipelines, etc. The damage due to corrosion in the United States alone has an annual cost of about $100 million. As a result corrosion protection is becoming a major industry in which there is a significant amount of research opportunities to develop new methods for reducing the amount of damage due to corrosion. There are many different methods that are used to hinder the rate of corrosion of different materials in certain corrosive environments. The main method that will be utilized for our senior project on corrosion protection is the use of protective coatings. In particular the protective coatings that we will be testing and analyzing are protective coatings like epoxies or multi-layer coatings that have a special additive giving it superhydrophobic properties. Superhydrophobic coatings are a relatively new type of protective coating that is starting to be utilized for corrosion resistant applications.

Applications

Corrosion rate of offshore platforms is mainly dependent on the concentration of water salinity, oxygen content, temperature, and flow rate of seawater. Corrosion rates can range from 0.05 to 0.64 mm/yr\(^3\). As the temperature of the seawater decreases the solubility of oxygen in the seawater rises, and therefore, the corrosion rate will increase. Corrosion at the offshore facilities is severe especially at crevices and sharp-edged regions, such as skip-welded plates and steel structural shapes. An offshore structure has a significant exposure to various salty environments. As shown in Figure 1, the splash zone, which is above the mean high tide level, is the region which suffers the most severe corrosive attack due to the erosive effect by the actions of tidal waves and continuous contact with salt air. The interval of splash zone of the structure can range from 1.5 m to more than 12 m, depending on the location\(^3\). It might be expected that most of the corrosion control design of an offshore structure would be similar to that of ships, as they are operated in the same environment. High performance epoxies which are commonly used on ships are the main coating systems for the offshore platforms as well. Cathodic protection is often used below the water level, however, it is inefficient in the areas, such as splash zones, which do not have continuous contact with seawater. Unlike ships, offshore platforms rarely or are unable to return to ports for repair and maintenance. For this reason a long-lasting coating system with good corrosion resistance is desired for offshore structures. The corrosion resistance of a coating is its ability to prevent the formation of a corrosion cell on the surface of the substrate.
General methods of corrosion prevention

Passivating barrier
Some active metals and alloys, under certain environmental conditions, naturally become less chemically reactive and form a hard and inert surface. Passivation layer on the metallic surface is a thin layer of oxide that produces to varying degree, depending on the availability of oxygen. This oxide layer can serve as a protective barrier to greatly reduce the transport of corrosive substances to the underlying surface and the formation of rust. However, a change in the character of the environment, for example, an alteration in the concentration of the active corrosive species, might result a passivated material to revert to an active state. In the splash zone of the offshore structure, the continuous wetting and drying helps destroy the passivating film on the metal surface.

Cathodic protection
Cathodic protection can control the corrosion on the metallic surface by supplying electrons to the metal that needs to be protected and making it a cathode of an electrochemical cell. The metal to be protected is electrically connected to another metal that is more reactive in the certain environment. The sacrificial anode is oxidized and giving up electrons to protect the cathodic metal from corrosion. Zinc and aluminum are the common sacrificial metals for the offshore structures as they are at the anodic end of the galvanic series and are more reactive compared to most metals. Nonetheless, the choice of the sacrificial anode material depends on the applications.
and the environment. The number as well as the distribution of anodes in the system will affect the performance life. A design life of 20 years is common. Poor distribution and the use of too few anodes will result in under protection, especially at the welded joints. However, the sacrificial anode cannot function properly when it is intermittently in and out of the seawater and it suffers possible abrasion and impact from floating debris.

**Coatings**
One of the most widely utilized methods for corrosion protection is with a protective coating or film. This method involves the application of a specific coating or film onto a substrate, which is generally a material that is susceptible to corrosion. Metal corrosion is a particularly significant type of corrosion that affects the functionality of many engineering systems including aircrafts, automobiles, offshore structures, pipelines, etc. As a result protective coatings are often used on metal substrates because they are susceptible to metal corrosion. There are many different types of protective coatings that are used for corrosion protection in industry applications including epoxies, polyurethanes, superhydrophobic surfaces, etc.

**Galvanization**
The galvanization protection method is the application of an anodic material, often zinc, as a protective coating providing an extra layer of protection for the metal substrate. It is often used for preventing corrosion of pipelines in corrosive conditions such as underground or offshore environments. There are a number of different types of galvanization methods to apply the layer of zinc to the surface of the substrate including electroplating, metal spraying, and hot dip galvanizing. The different methods can produce zinc layers of various thickness and durability on a metal surface.

**Protective coatings**
There are numerous coatings used protect the metal substrates from corrosion by preventing the metal from coming into contact with an environment that would promote the metal corrosion of the substrate material. Many of the protective coatings today are polymer based coatings such as epoxies or polyurethanes. The coating performance is dependent on a number of factors including chemical composition, coating application method, and surface preparation. However the main determining factor that has the greatest effect on the performance of the coating or surface that is produced and applied to a metal substrate is the surface preparation. Surface preparation of the substrate has a significant impact on several important coating properties that are key to the coatings’ performance. These coating properties that are integral to its performance are the degree of adhesion between the coating or surface and the metal substrate, the surface profile (roughness), and the degree of hydrophobicity exhibited by the coating or surface.
Superhydrophobic Coatings

Superhydrophobic coatings are surfaces that display an extremely high water repellency, which make them highly effective for corrosion resistant applications. The effectiveness of the superhydrophobic surfaces’ water repellency is dependent on two critical factors, which are the surface energy and the surface morphology of the material. Substrate materials with a lower surface energy and a roughened surface morphology exhibit the most superhydrophobic behavior. The roughened surface improves the hydrophobic nature of the surface the most by increasing the solid-liquid interface and trapping air in between the surface and the liquid. However for a material to display superhydrophobic behavior it requires a combination of lowered surface energy and increased surface roughness. The hydrophobic behavior of the surface is directly related to the water contact angle it forms with liquid droplet. For a surface to exhibit hydrophobic behavior the water contact angle of the surface needs to be in the range of $90^\circ < h < 150^\circ$. The two major liquid-solid surface interactions that occur on surfaces with different water contact angles are shown in Figure 2.

![Figure 2](image)

Figure 2- The hydrophilic behavior (Top) occurs at small water contact angles of $h<90^\circ$ while hydrophobic behavior (bottom) occurs at larger water contact angles of $h>90^\circ$.5

The properties exhibited by these superhydrophobic coatings make them an incredibly versatile coating that can be utilized for many practical applications. Their main applications include self-cleaning, anti-biofouling, and corrosion resistant applications. The self-cleaning applications seem to possess a lot of promise in their ability to help keep surface free of contaminate making the surface easier to clean.5 Anti-biofouling applications also act as a repellent for biological organisms on structures or substrates that are submerged. However its high level of corrosion resistance for preventing or slowing the breakdown of the oxide layer on metal substrates in corrosive media is potentially its most significant application and is the subject of this project. The superhydrophobic polymer coatings are supposed to be applied to offshore structures existing in a coastal (saltwater and air) environment. These coatings are supposed to be applied specifically in the splash zones of these offshore structures since they are the most corrosive area of an offshore structure and will result in the largest cost benefit.
Properties
Protective coatings with the combination of lowered surface energy and increased surface roughness are known as superhydrophobic. Their properties include water repellency, self-cleaning, transparency, and flexibility. Transparency and hydrophobicity however are a particularly difficult combination to find since they both tend to compete with each other because the surface features like the surface roughness for hydrophobic surfaces are generally light scattering.

Performance
The level of performance exhibited by superhydrophobic coatings is dependent upon two factors, surface preparation and pigment volume concentration (PVC). In order to ensure adhesion of the coating to the base metal and prolong the service life of the coating system, a proper surface preparation is essential before applying the coating. Up to 80% of all coating failures result from inadequate surface preparation, which affects the adhesion of coating significantly. When all contamination on the surface is removed, the surface profile would allow a strong bonding between coating and substrate and reduce the probability of corrosion initiating from the presence of any surface contaminants. The process of surface preparation consists of removal of visible and invisible contaminants and roughening the surface. A clean surface that is free of visible contaminants such as rust, dust, salts and so on is required. In general, higher performance coating systems require a higher degree of cleanliness of the surface. Chemical contamination before coating, which is more difficult to detect, can result in poor adhesion, blistering or other defects in the applied coating system. Besides cleanliness, the surface of the substrate is required to be roughened to provide for a mechanical bond of the coating to the base metal.

The PVC of a coating is another significant factor that affects the coating performance. The coating PVC can be defined as the volume of pigment/filler contained in the dry coating film. A superhydrophobic coatings PVC affects its hydrophobic behavior and properties. As the PVC of the coating is altered the coatings surface morphology and film properties also change. By increasing the PVC the coatings surface roughness and the film density can be improved until the critical pigment concentration (CPVC) is reached. The CPVC is the volume of pigment/filler that is required to use all the binder to cover the particles. Once the PVC of the coating is above the CPVC the coatings surface roughness increases significantly while the film density decreases.

Research Question
In this project we are testing and analyzing a kind of protective coating that uses a superhydrophobic additive with a polymer binder. The additive is a powder that is composed of diatomaceous earth particles. Diatomaceous earth is a chalk-like porous sedimentary rock that has a low density and is composed of diatoms, which are any
microscopic organisms with cell walls made of silica. The silicate surface it produces is amorphous in nature and contains a number of silanol, Si-OH, groups. Using a QUV, a Q-FOG, and the pier at Avila we will test over 50 samples and observe their progress over 5-6 weeks. Our final analysis will include the optimum pigment volume concentration (PVC) of the hydrophobic additive used to produce the best corrosion resistance in the coatings being tested.

II. Tested Coating
The coating systems that were tested are shown below in Table I. Each Epoxy coating system was a two-layered system, which consisted of a epoxy primer and a epoxy top coat. Each PU coating system consisted of a epoxy primer and a high solids polyurethane layer as top coat. MP2 coatings were a single layered polyurethane system. In addition to the difference in material used as top coat, the amount of diatomaceous earth particles contained in coatings varied from 10 to 50% by dry coating weight. The coatings provided by Chevron ETC was prepared by different solvents. The coated samples with 20% and 30% diatomaceous earth (DE) additives were prepared using Oxsol 100/MAK solvent while the samples with 10% diatomaceous earth additives were prepared with Novec 7500 solvent.

Table I - Various Layers and PVC of DE in Each Coating System

<table>
<thead>
<tr>
<th>Panel ID</th>
<th>First coat</th>
<th>Second coat</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy SSC1</td>
<td>Macropoxy 646</td>
<td>Macropoxy 646 w/ 20% DE</td>
</tr>
<tr>
<td>Epoxy SSC2</td>
<td>Macropoxy 646</td>
<td>Macropoxy 646 w/ 30% DE</td>
</tr>
<tr>
<td>PU SSC6</td>
<td>Macropoxy 646 w/ 20% DE</td>
<td>HS Polyurethane w/ 20% DE</td>
</tr>
<tr>
<td>PU SSC7</td>
<td>Macropoxy 646</td>
<td>HS Polyurethane w/ 10% DE</td>
</tr>
<tr>
<td>PU SSC8</td>
<td>Macropoxy 646</td>
<td>HS Polyurethane w/ 20% DE</td>
</tr>
<tr>
<td>PU SSC9</td>
<td>Macropoxy 646</td>
<td>HS Polyurethane w/ 30% DE</td>
</tr>
<tr>
<td>PU SSC10</td>
<td>Macropoxy 646 w/ 20% DE</td>
<td>HS Polyurethane w/ 10% DE</td>
</tr>
<tr>
<td>MP2 -40</td>
<td>Polyurethane w/ 40% DE</td>
<td>N/A</td>
</tr>
<tr>
<td>MP2 -50</td>
<td>Polyurethane w/ 50% DE</td>
<td>N/A</td>
</tr>
</tbody>
</table>
Scribing
In order to artificially inflict damages on the coatings, all samples were scribed before testings according to ASTM Standard D1654 -08 Evaluation of Painted or Coated Specimens Subjected to Corrosive Environments. A “X” shaped scribe was used as a well-defined defect for the testings. It was to conduct reproducible tests regarding the protective as well as water-repelling properties of the superhydrophobic coatings in combination with the metal substrate. Corrosion resistance of each coating can be determined by the corrosive damage in and around the scribe. Razor blade was used as a scribing tool. The scribe width of each coating was consistent, as shown in Table II, so the influence of scribe layout on the testing results was insignificant.

Table II - Comparison of Average Scribe Width in Each Coating System

<table>
<thead>
<tr>
<th>Coating System</th>
<th>Average Scribe Width, mm</th>
<th>Standard Deviation</th>
<th>Number of Cuts</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy</td>
<td>0.49</td>
<td>1.0</td>
<td>5</td>
</tr>
<tr>
<td>PU</td>
<td>0.46</td>
<td>1.3</td>
<td>5</td>
</tr>
<tr>
<td>MP2</td>
<td>0.43</td>
<td>0.6</td>
<td>10</td>
</tr>
</tbody>
</table>

III. Testing Procedures

In order to compare the performances of different coating systems, 3 different tests were performed - Natural weathering test, QUV accelerated weathering test and salt spray test. Natural weathering testing was outlined by ASTM Standard G7/G7M-11 Practice for Atmospheric Environmental Exposure Testing of Nonmetallic Materials. For accelerated weathering test, the samples were tested according to ASTM Standard D4587-11 Fluorescent UV-Condensation Exposures of Paint and Related Coatings. Q-fog salt spray test was performed using ASTM Standard B0117-11 Practice for Operating Salt Spray (Fog) Apparatus to test the corrosion resistance of the coatings.

Natural Weathering Testing
Coated samples were placed at Cal Poly Marine Sciences Pier, which is located at Avila Beach, to analyze the performance of the superhydrophobic coatings under coastal environment. The samples were checked and taken pictures every 1-2 weeks. The exposure rack, shown in Figure 3, was constructed according to ASTM Standard G7/G7M to hold the samples at a 45° angle facing towards the ocean. Moreover, the testing holder was placed at the end of the Cal Poly Pier where no shadows were cast on the samples during daytime.
QUV Accelerated Weathering Testing
Samples were put in a Q-lab fluorescent UV/condensation cabinet, of which cross section is shown in Figure 4, in order to evaluate the damage caused by outdoor weathering such as sunlight, dew and rain. Outlined by ASTM Standard D4587, the coatings were exposed to alternating periods of 700 hours in the tester. The fluorescent UV/condensation cycle was 4 hours of UV at 0.89 W/(m²-nm) at 340 nm at at 60°C and 4 hours of condensation without UV exposure at 50°C. Brand new UV lamps, which were placed at the cabinet as shown in Figure 5, were used at the beginning of the test and they were replaced every 400 hours. Due to non-uniform irradiance within the chamber, the samples were rotated horizontally every 100 hours so that each sample was able to receive same amount of UV exposure.
Figure 4 - During condensation period, the water molecules that are evaporated in UV cycles condensates on the samples.\textsuperscript{11}

Figure 5 - There were 4 UV-340 lamps on each side of the tester and the coated side of samples were facing toward the lamps during testing.
Salt Spray Testing

Q-fog cyclic corrosion tester was used to find out the coating’s resistance of corrosion in the exposure of humid and salty environment. Coated samples were exposed to 5% salt spray at a rate of 0.4 to 0.8 mL/second at operating temperatures between 23 °C and 30 °C according to ASTM Standard B117-11. The samples were held at an angle of 30° from the vertical and were parallel to the principal direction of flow of spray through the cabinet, as shown in Figure 6. The salt solution was prepared by dissolving 5 portions by mass of sodium chloride in 95 portions of distilled water. Although the testing cabinet was shared with a class from Cal Poly Coatings Department, spacing between 2 different sets of panels was adequate so that there was no contamination caused by foreign sources.

![Figure 6- Schematic of Salt Spray Tester.](image)

IV. Results and Discussion

From all three testings, the results showed that the single-layered polyurethane MP2 coating systems outperformed the two layered PU coating systems in terms of corrosion resistance and durability. Using ASTM Standard D610-08R2012 Evaluating Degree of Rusting on Painted Steel Surfaces, the amount and distribution of visible surface rust on each coated panel was evaluated.\(^\text{12}\) Coated samples were assigned a rust rating followed by the type of rust distribution to quantify the corrosion resistance. The degree of rusting was determined using a 0 to 10 scale based on the percentage of surface area rusted. Coated samples rated as 0 generally have more than 50% of surface area rusted while samples having the least or no rusting with less than 0.01% of visible rust are rated as 10. The type of distribution of rusting was classified as general rust, pinpoint rust, hybrid rust or spot rust, which are identified by G, P, H and S respectively.
Natural Weathering Testing
In the third week of atmospheric exposure at Cal Poly Pier, pinpoint rusting and light corrosion in the scribe was visible on samples PU SSC9 and PU SSC6. After 3 months of testing, however, there was no observable growth of the surface rust on these 2 PU samples. PU SSC9 was rated as 8-P while a rate of 9-P was given to PU SSC6. There were also no samples showing signs of blistering or chalking through visual inspection. Except samples PU SSC9 and PU SSC6, all of the coatings had performed well in this test. The PU SSC9 sample is shown below in Figure 7.

![Figure 7- Panel PU SSC9 at 0 hours (left) and at 3 months (right)](image)

QUV Accelerated Weathering Testing
After 700 hours of cyclic UV/condensation testing, PU SSC7 and PU SSC10 performed better than the rest of the PU coating systems. Both samples showed only slight corrosion in the scribe. The epoxy coatings did not perform as well, a picture of the degradation of epoxy SSC2 is shown below in Figure 8.

![Figure 8 - Panel Epoxy SSC2 at 0 hours (left) and at 1000 hours (right)](image)
The results also showed that all Epoxy coatings were vulnerable to UV light as a significant amount of checking and discoloration was noticed on the surfaces. Using ASTM Standard D660-94 Evaluating Degree of Checking of Exterior Paints, the degree of chalking on Epoxy samples was evaluated. Epoxy SSC1 sample had a large amount of shrinkage, in which the breaks formed individual short breaks in the surface, giving it a rating of 5. Epoxy SSC 2 sample, on the other hand, had numerous sigmoid checking, in which the breaks in the surface form oval patterns, providing a rating of 4.

**Salt Spray Testing**
The test samples were run for 120 hours in the Q-FOG salt spray machine. The results of the 120 hour salt spray test are shown below in Table III.

### Table III - Rust Rating Assigned to Each Coated Sample

<table>
<thead>
<tr>
<th>Panel ID</th>
<th>Rust Grade</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy SSC1</td>
<td>7-G</td>
<td></td>
</tr>
<tr>
<td>Epoxy SSC2</td>
<td>7-G</td>
<td></td>
</tr>
<tr>
<td>PU SSC6</td>
<td>7-G</td>
<td></td>
</tr>
<tr>
<td>PU SSC7</td>
<td>9-P</td>
<td></td>
</tr>
<tr>
<td>PU SSC8</td>
<td>7-G</td>
<td></td>
</tr>
<tr>
<td>PU SSC9</td>
<td>5-G</td>
<td></td>
</tr>
<tr>
<td>PU SSC10</td>
<td>9-G</td>
<td></td>
</tr>
<tr>
<td>MP2 -40</td>
<td>10</td>
<td></td>
</tr>
<tr>
<td>MP2 -50</td>
<td>10</td>
<td></td>
</tr>
</tbody>
</table>

As the results show the single layer polyurethane coatings performed the best overall. However among the best two layered coating, PU SSC7 and PU SSC10, also performed well. However sample PU SSC9 performed the worst overall getting a rating of 5. The results of the PU SSC9 samples salt spray test are shown below in Figure 9.
Figure 9- Panel PU SSC9 at 0 hours (left) and at 120 hours (right)

V. Conclusion
From our test results we determined that the single-layered polyurethane coating system outperformed all other coating systems in terms of corrosion resistance. However the film formed by single-layered polyurethane coating was malleable and weak. As a result of this coating’s low durability we determined that it would not function well in the ‘splash zone’ on offshore structures. After the single-layered polyurethane system the best results were seen in the two layer polyurethane system. In the QUV test and Q-FOG test sample, PU SSC7 and PU SSC10 outperformed the rest of 2-layer PU-EP coatings. Among PU-EP samples, the hydrophobicity and corrosion resistance of the coating appeared to decrease with increasing amounts of DE additive. The results of the analysis indicate that the CPVC of the PU-EP coatings was around 10% DE additive, since the coatings with that PVC exhibited good performance.
References

4. J. Peter Ault, P.E. The Use of Coatings for Corrosion Control on Offshore Oil Structures. Ocean City, NJ
Appendix A- Surface preparation techniques

Surface preparation is one of the most important parts in a coating system. The performance of the coating is affected by the techniques of surface preparation, as indicated in Table IV, more than any other variable. Poor surface preparation would lead to poor performance of the coatings.

Table IV- Various Techniques of Surface Preparation for Coating System

<table>
<thead>
<tr>
<th>Technique</th>
<th>Uses</th>
</tr>
</thead>
<tbody>
<tr>
<td>Solvent cleaning</td>
<td>Used to remove oil, grease, soil, and various other contaminants, except rust or mill scale.</td>
</tr>
<tr>
<td>Hand/power tool cleaning</td>
<td>Used to remove loose rust, mill scale and any other loose contaminants.</td>
</tr>
<tr>
<td>Waterjetting</td>
<td>Using high pressure waterjetting to clean prior to coating.</td>
</tr>
<tr>
<td>Commercial blast cleaning</td>
<td>Used to remove all contaminants from surface, except discolorations caused by rust stain or slight residues of rust or old coatings.</td>
</tr>
<tr>
<td>Pickling</td>
<td>Used for complete removal of all mill scale, rust, and rust scale by chemical reaction</td>
</tr>
</tbody>
</table>
Appendix B: QUV Result Pictures

Pictures below are the coated samples before and after QUV accelerated weathering test.

Figure 10 - Epoxy control samples without superhydrophobic Diatomaceous Earth additive

Figure 11 - PU control samples without superhydrophobic Diatomaceous Earth additive
Appendix C: Coastal Environment Weather Data
Factors such as temperature, dew point, wind speed, humidity and precipitation would have influence on the results of natural weathering testing.

Table V - Average Weather Data in The Months Coated Samples Were Tested 14

<table>
<thead>
<tr>
<th>Month</th>
<th>Average Mean Temp, °C</th>
<th>Average Dew Point, °C</th>
<th>Average Windspeed, km/h</th>
<th>Average Morning Relative Humidity, %</th>
<th>Average Afternoon Relative Humidity, %</th>
<th>Average Precipitation, mm</th>
<th>Days With Precipitation</th>
</tr>
</thead>
<tbody>
<tr>
<td>March</td>
<td>12</td>
<td>7</td>
<td>20</td>
<td>86</td>
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<td>14</td>
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