

Corrosion and Adhesion Behavior of Recycled High Density Polyethylene (HDPE) Coated Mild Steel

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Abstract— This research utilized 100% recycled HDPE as a powder coat on mild steel (used as a simulated automotive chassis). The recycled HDPE as a powder form was applied to mild steel using electrostatic powder coating equipment. The coated steel was characterized, and the corrosion property was determined by immersion in NaCl, and H₂SO₄ solutions. Scanning Electron Microscope (SEM) analysis revealed that the coating was non-porous and on average, 80 μm thick. Corrosion test in 5% H₂SO₄ acid solution was performed in accordance with ASTM standards ASTM C694-90a and ASTM G31. All specimens showed corrosion, specifically on the edges and sharp corners, with a maximum corrosion rate of 12.54 mm/year. Corrosion test was also performed in a 5% NaCl salt solution in compliance with ASTM B895-16 and ASTM D610-08 standards. Visual inspection suggested that the samples were corroded mildly as some corrosion products can be seen at the bottom of the beaker. However, no measurable weight loss was found. After corrosion tests, the coated samples were subjected to adhesion tape test according to the ASTM standard D3359 adhesion by tape test. The tape tests provided a 5B, which is the maximum for a tape test, for as coated samples, and a minimum 4B for samples performed immersion tests in salt solution. These results suggested that a well adhesion was achieved between the steel substrate and HDPE coating. This study indicated that the recycled content HDPE would be a viable candidate to protect chassis from road salt corrosion.

Index Terms— Recycling, HDPE coating, Adhesion Tape Test, Corrosion, Salt Immersion Test, Acid Immersion Test, ASTM Tape Test.

1 INTRODUCTION

ACCORDING to the United States Environmental Protection Agency, 2016, the amount of municipal solid waste produced by United States was 258 million tons in 2014 [1]. Less than half of them (34.6%) was recycled while a bigger portion was landfilled. Recycling of any portion of this otherwise landfilled waste will add to the global efforts to minimize the landfilled waste. Consequently, it reduces the amount of land used for dumps, which pollute the property and environment. Also, the waste dumps make the land unusable for years afterward. The recycling also reduces humanity's carbon footprint. The recycling completed in 2014 saved the annual carbon dioxide emissions by over 181 million metric tons, which is comparable to the emissions produced by approximately 38 million passenger cars [1]. Additionally, by improving the sustainable management of resources, the strain to excavate and process the Earth's finite natural resources is relaxed. Therefore, to better manage sustainability, recycling rates must be increased. Plastics are major contributor to landfilled wastes. In fact, 18.5% of the 136 million tons of landfilled materials were classified as plastics [1]. Due to the recyclability of most plastics, this number could easily be decreased. One solution to reducing plastic waste, in addition to further en-

would be to create a corrosion inhibiting coating with recycled HDPE.

HDPE is considered as one of the most popular and versatile plastics in industry [2]. HDPE constitutes a sizable portion of recycled plastics because of its wide use in consumer products. According to 2014 statistics collected by the Environmental Protection Agency, over 29% of HDPE products were recycled. The repeated processing due to recycling, may eventually compromise some of the properties exhibited by first-made HDPE. As plastic is recycled repeatedly, it ages and eventually becomes unsuitable for its traditional uses [3]. Contaminants leftover from the recycling process may also affect the material properties of the plastic [3]. Since most recycling centers only clean the post-consumer plastics with water and mechanical work, impurities such as detergent may remain in the processed materials as determined through simulated testing, the detergent can slightly decrease the mechanical strength of HDPE as the number of extrusion cycles increase. Detergent was also proven to degrade the stability of the polymer by decreasing its oxidation onset temperature. However, the contaminants appeared to have trivial effect on most other properties. Therefore, although the recycling of HDPE may affect some mechanical properties, it generally remains durable after several cycles [3].

The injection molded HDPE products, such as milk jugs or other consumer containers, can withstand tensile stresses ranging from 1450 to 6530 psi [4]. In addition to its high strength, HDPE is also resistant to impact load, weather, and insect [2]. These characteristics make it possible to use in a range of products i.e. from car fuel tanks to plastic decking [2, 5]. Samimi and Zarinabadi [6] investigated a coating of polyethylene applied to steel oil and gas pipelines as protector from corrosion. This coating is common in the petroleum in-

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couraging consumers to recycle, is to develop new products which utilize recycled materials. For instance, one possibility

dustry and will often utilize HDPE. Also, the coating is applied in three layers as opposed to a direct application. Generally, this coating was deemed effective except for in circumstances where careless or improper installment of the pipes compromised the coating [6]. Although these studies focused solely on piping, they are an example of HDPE's durability, even under the harshest conditions. However, given that these properties establish it as a considerable corrosion inhibitor for automotive components, like the chassis, the three-layer method of application is unrealistic for this case. In the case of a pipe, after the base primer is applied, the top two layers of the coating are wrapped around the pipe like a tape. However, due to the complicated geometry of a chassis, wrapping the HDPE around it would be very difficult to producing a seal against its surroundings.

Corrosion is always a costly issue for many common applications, including steel components for automobiles [7, 8]. On the other hand, the United States landfills over 100 million tons of waste annually [1]. Understandably, a corrosion protection coating, such as that created with recycled HDPE, has the potential to address both these issues by reducing costly corrosion damages to automobiles as well as increasing sustainability by lessening plastic waste. However, given that metal and plastic do not generally bond well, it was initially speculated that adhesion issues could inhibit the coating's functionality. In this project, mild steel samples were coated with recycled content, post-consumer HDPE using powder coating method. These samples were then subjected to corrosion immersion and adhesion testing to determine recycled HDPE's viability as a powder coat for the application of automotive chassis. Thickness of the HDPE coating was determined using SEM analysis.

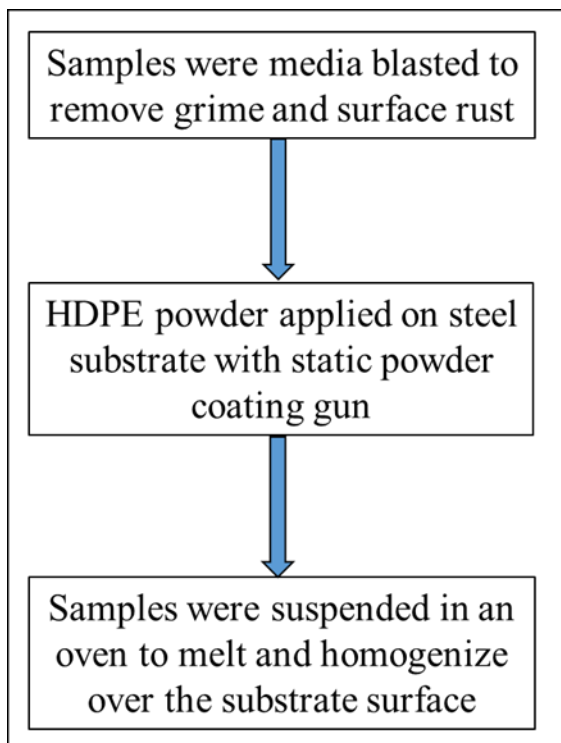


Figure 1: Summary of powder coating process.

2 EXPERIMENTAL PROCEDURE

2.1 Application of HDPE Coating

Figure 1 shows the summary of the HDPE powder coating process. Mild steel samples with dimensions of 75 mm x 25 mm x 6.25 mm were cut from flat bar stock with a bandsaw. After cutting, all burrs and rough edges were removed with a bench grinder. Following deburring, 6.25 mm holes were drilled and deburred 9.5 mm from the top of each sample. The starting materials for coating (recycled HDPE) were in the powder form. The substrates were first media blasted and then coated. The coating was performed with an unmodified static powder coating gun. This device gave the HDPE particles a static charge which allowed them to cling to the steel. The coated samples were then suspended in an oven. This process melted the plastic particles transforming into a singular, uniform coating around the entire surface of the mild steel substrate. The melting point of HDPE is between 121°C and 137°C, which is lower than most traditional powder coats.

2.2 Microscopic Analysis/Characterization of Coating

The coated samples were cut in the transverse direction with a wet/dry horizontal bandsaw. The cut faces were then polished with emery paper and the finest paper used was 1200 grit size. The samples were coated with carbon in a carbon coater before checking them under SEM. Scanning Electron Microscopy images were taken using Hitachi SU6600.

2.3 Corrosion Testing

To investigate the corrosion behavior of coated mild steel, two different corrosion tests were performed. The first was immersion in an acid bath in accordance with ASTM standard C694-90a, while the other was immersion in a salt bath in accordance with ASTM B8951-16. Before testing, each sample was weighed and tagged with a letter-number combination. The letter "A" represented those used for acid immersion test while S was given to those chosen for immersion in NaCl solution. Also, weight of each sample was recorded before being subjected to any immersion tests.

2.3.1 Immersion Test in Diluted Sulfuric Acid

The acid immersion testing was inspired by ASTM C694-90a, which evaluates the weight loss of sheet steel when immersed in a sulfuric acid solution [9]. A sulfuric acid solution, 5% by volume, was prepared using deionized water. Each solution was mixed with a magnetic stirrer in order to ensure that the solution was homogenous. This test was conducted at room temperature and no heat was added from outside sources. In one 500 ml beaker, one sample was tested at one time to avoid any cross contaminations. Also, it allowed to observe each sample individually. During the immersion test, the samples were suspended from a steel bar with wires. The samples were attached in a way that it would submerge approximately one-half of the length in the acid solution. During acid immersion tests, the samples were immersed for 5.5 hr. to 70 hrs. The samples were then air dried in a chemical fume

hood for several days. After drying, they were weighed, and mass losses were calculated. The corrosion rates were calculated in accordance with ASTM standard G31 [10]. Also, any physical changes to the samples were noted and photographed.

2.3.2 Immersion Test in Sodium Chloride Solution

Since the application for this coating was intended to prevent corrosion on automotive chassis, it was decided to perform an immersion test in a sodium chloride solution in order to simulate exposure to road salt. This test was conducted in compliance with ASTM B895-16. The samples were individually immersed in the 5% by mass NaCl solution for 0.5 hr to 70.5 hr. The corrosion rating of each sample at specific time intervals was recorded. The corrosion ratings used were A, B, C, and D; where A being no corrosion and D being the highest corrosion based on a visual examination. Once the samples were removed from solution, they were washed with a multipurpose cleaner and then air dried. After drying, the corrosion of the samples was given a second rating in accordance with ASTM D610-08 [11]. The rating used were General (G), Pinpoint (P) or Hybrid (H) based on a visual examination after salt immersion test. These ratings may be more accurate because it addresses the types of corrosion that occur with painted surfaces as opposed to the rating B895 which was intended for uncoated stainless steel. Additionally, the samples were weighed to determine if there were changes in mass.

2.4 Adhesion Testing by Tape Test

The coating's adhesive strength was measured with the tape test method B described in ASTM D3359-17 [12]. On each sample, the coating was cut with a specialized cross-hatch cutter to form a grid pattern on the surface. This was then analyzed with a lighted magnifying glass to determine if the cuts were deep enough to reach the steel substrate. After this, any loose particles were brushed away with a small brush. Pressure sensitive tape was then applied over the cuts and then smoothed out with a rubber eraser. This eliminated air bubbles and ensured that the tape adhered evenly across the cut pattern. The tape was then steadily pulled back on itself at an angle close to 180°. The surface was then analyzed for missing or loose pieces of coating. Based on the amount of flaking a rating from 0B to 5B was given where 5B is being the best and 0B is being the worst according to the explanation given in standard.

Each side of a sample was tested twice, with one cut near the top and the other near the bottom. For all non-immersed samples, this rating was averaged to give the sample an overall score. For immersed samples, the surface was first cleaned with a multipurpose cleaner and lint-free towel. Each side was tested twice, when possible. However, the top and bottom test ratings were kept separate to distinguish from the immersed and non-immersed sections of the specimen. Also, the time between immersion and adhesion testing was recorded for all relevant specimens.

3 RESULTS AND DISCUSSION

3.1 HDPE Coating

Figure 2 shows a HDPE powder coated mild steel sample. Based on the visual inspection, it was found that some areas of steel substrate were insufficiently coated for few samples out of 30 coated samples. Additionally, the coating was not uniform or sufficient around the sharp corners or edges in the samples. The thin coatings that were found on few samples were a result of powder falling off before curing. This phenomenon occurred when turning the sample to coat the other side and can occur even with normal powder coats. These results suggest that the HDPE may not be able to hold a static charge if the powder particles were too large. If the powder particles were finer, the static charges used could have been enough to hold them onto the steel substrate surface. The static charges may also have been easily conducted away from the HDPE powder after coating because the mild steel is a good conductor of electricity.



Figure 2: HDPE powder coated mild steel.

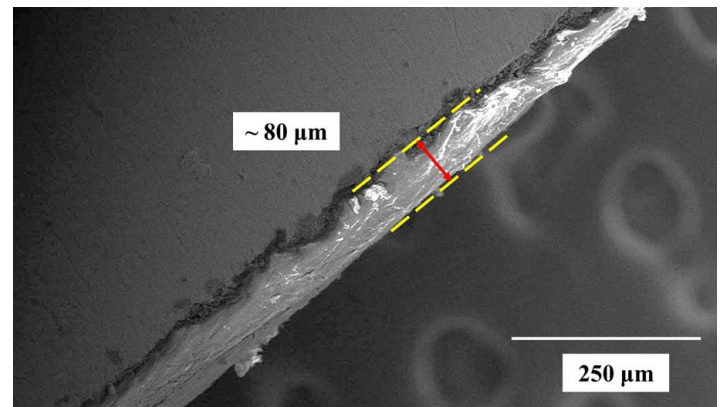


Figure 3: SEM image of substrate/coating interface showing the thickness of the coating.

Figure 3 shows SEM image of substrate/coating interface showing the thickness of the coating. The average thickness of the coating obtained was $\sim 80 \mu\text{m}$. On nearly all samples, there was a dark line that followed the substrate/coating interface. This line indicates that there was some separation between the polymer and the steel. This was likely a result of the polishing process. As the sample was moved back and forth on the abrasive surface, it could have pulled the coating away from the

substrate. The substrate surface on which the polymer is attached appears rough and uneven due to the media blasting process. However, the coating blended with this complex geometry. This is promising for the viability of the coating because the geometry helps to increase the coating's adhesive strength.

3.2 Corrosion Test

3.2.1 Immersion test in 5% H₂SO₄

The visual observation found that the bubbles were formed as soon as the samples were immersed in the acid solution. The bubbles were concentrated around the corners and edges of the samples. This suggested that the coating may have had poor coverage at the sharp fillets. However, the bubbles appeared to be stagnant with no signs of vigorous reactions. The acid solution also discolored because of the formation of corrosion products. However, the corrosion reactions were not same as for all samples. Some specimens were coated very well and showed less corrosion compared to other samples. Figure 4 shows photographic images of samples; (a) before immersion, (b) after corrosion in acid solution - edge view, and (c) flat surface near the bottom end. It was evident that the steel had been corroded. This again confirmed that the coating was not sufficient at the sharp fillets of the steel and allowed the acid to penetrate behind the coating. This damage affected approximately up to 2 mm from the bottom of the sample.

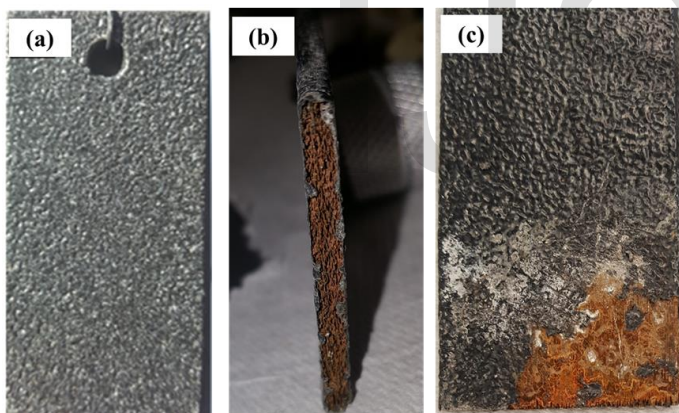


Figure 4: Photographic images of samples (a) before immersion, (b) after corrosion in acid solution - edge view, and (c) flat surface near the bottom end.

Table 1: Weight Loss, Immersion Times, and Corrosion Rates of Samples in 5% H₂SO₄.

Sample #	Weight Loss (grams)	Time (hours)	Area (cm ²)	Corrosion Rate (mm/y)
A1	2.7	70.45	34.03	12.54
A2	1.7	70.45	34.15	7.87
A3	1.4	70.45	33.55	6.59
A4	2.3	70.45	33.72	10.78
A5	2.1	70.45	33.71	9.85
A6	0.6	23.25	33.89	8.48
A7	0.0	5.58	34.19	0.00
A8	0.1	23.25	33.71	1.42
A9	0.2	23.25	33.96	2.82

Table 1 summarizes the weight loss and corrosion rates for

all samples subjected to the acid immersion test. The corrosion rates were calculated using the theoretical density of mild steel 7.87 g/cm³. The corrosion rate varied from 7.87 to 12.54 mm/y for 70.45 hr of immersion. The corrosion rate became as small as zero for 5.58 hr of immersion. This indicated that the proper controlling of the coating method can lead to a remarkably effective corrosion resistant coating, even in the acidic environment.

3.2.2 Immersion Test in 5% NaCl Solution

The samples immersed in the salt solution did not immediately exhibit any signs of reaction. However, after 5.5 hours, there were small spots of corrosion found on all samples. This spot corrosion continued to progress over several days. The ratings given to each sample related to time are summarized in Table 2. The ratings are given in letter grades where an A represents 0% coverage, a B is less than 1%, a C is between 1 and 25%, and a D is greater than 25% coverage of corrosion. However, since the samples quickly developed rust, it was clear that the coating did not completely seal the substrate from the environment. Since ASTM B895-16 is typically used for examining uncoated samples, determining the ratings based on the percentage of the surface covered with corrosion was difficult. Therefore, after the samples were removed from the salt solution and cleaned, the corrosion was reevaluated with the system described in ASTM D610-08 [11].

Table 2: Corrosion ratings of HDPE coated samples after immersion in salt solution according to ASTM B895-16 [26].

Specimen number	Submersed time (hour) in 5% NaCl solution					
	0	0.5	5.5	24	48	70.5
S1	A	A	B	C	C	D
S2	A	A	B	B	C	C
S3	A	A	B	B	C	C
S4	A	A	B	C	C	D
S5	A	A	B	B	C	C

Table 3: Corrosion ratings of HDPE coated samples after immersion in salt solution according to ASTM D610-08 [27].

Sample #	Sample Side	Evaluation Number
S1	a	5-P
	b	4-P
S2	a	6-H
	b	3-H
S3	a	2-H
	b	6-P
S4	a	1-H
	b	4-P
S5	a	5-P
	b	4-P

*General (G); Pinpoint (P); Hybrid (H)

As summarized in Table 3, each side of each sample was given a separate rating. The higher the number, the smaller the percentage of rust. For example, a 10 would correspond to 0.01 percent rust, a 6 would be greater than 0.3 and up to 1.0 percent, and a 1 would be between 33.0 and 50.0 percent cover-

age [11]. It was believed that this happened due to the non-uniformity of the coating. Most of the rust was very localized and therefore labeled as pinpoint corrosion. However, some samples did exhibit spots that resembled general corrosion, thereby yielding a hybrid rating. The lower the number, the higher the percentage of corrosion with a 9 being 0.01% and 0 being greater than 50%. As shown in the table, none of the 5 samples received the same rating for both sides. Some samples, such as S3, have significant difference in ratings thereby demonstrating the varying nature of the coating's protective abilities. Additionally, despite having a faster visible corrosion rate than several samples as revealed in Table 3, sample S1 did not have the most widespread corrosion. This could possibly be attributed to the types of corrosion occurring on each sample. S1, with only pinpoint corrosion, may appear to have less corrosion than a different sample which has relatively large spots of general corrosion. Also, unlike the corrosion on the acid samples, the corrosion on this group appeared to have stayed localized. Therefore, the polymer coating, despite not fully sealing the steel, may have the correct properties to hinder the spread of rust and any subsequent damage. Although the visual inspection showed corrosion occurred on the samples, no samples showed measurable weight loss after immersion in salt solution even after 241 hours of immersion. This may indicate that the coating on automotive chassis may perform very well in response to corrosion from road salt.

3.3 Adhesion Tape Test of Coating

Table 4: Summary of adhesion tape test of as coated (T), after immersion test is salt solution (S), and acid solution (A).

Sample #	Rating		Time between immersion and testing
	Not-Immersed	Immersed	
T1*	5B	N/A	N/A
T2	4.75B	N/A	N/A
T3**	5B	N/A	N/A
T4	5B	N/A	N/A
T5	5B	N/A	N/A
S1	4B	5B	7 Days
S2	4.5B	4.5B	7 Days
S3**	4B	4B	7 Days
S4	5B	5B	7 Days
S5	5B	5B	7 Days
A1***	N/A	N/A	23 Days
A2	4.5B	5B	23 Days
A3	5B	4.5B	23 Days
A4	4.5B	1B	23 Days
A5	5B	4B	23 Days
A6****	4B	3B	8 Days
A7	5B	4.5B	9 Days
A8	5B	1B	8 Days
A9	4.5B	5B	8 Days

*1 mm cut; **Coating too thin; ***Damage from immersion prevented testing; ****Peeled when cutting.

In addition to determining the adhesion property of five as coated samples, all samples from the immersion tests were also used. This was done to determine if the corrosive agents affected the adhesion between the coating and substrate. The results of the testing are summarized in Table 4. The letter designations for sample number used in Table 4 were T for as coated samples, S for samples tested in salt solution, and A for samples tested in acid solution. T2 was the only sample that resulted with a rating of 4B in one location, thereby making the average 4.75B. This may be due to inconsistencies in the coating and numerous other factors that could affect the adhesion at that location. While conducting testing, the coating overall exhibited high adhesive strength both to the substrate and itself. Cutting would often leave loose burrs of polymer. Most of these burrs could not be removed by brushing and they would often still be attached after conducting the tape test. Although slightly lower than the ratings given to T1-T5, the samples subjected to salt testing also demonstrated high adhesive strength with no ratings below 4B. Therefore, despite the presence of corrosion, it appears that the corrosion remained localized. As a result, the adhesion between the coating and substrate was barely affected.

According to the visual inspection, the acid immersion test samples demonstrated clear signs of adhesive degradation. The tape test rating were lower for acid immersion test samples and few samples showed a rating as low as B. It proved that the acid compromised adhesion in the majority of samples. With sample A6, the coating was damaged to the extent that it peeled when using the cross-hatch cutter. This severe degradation may have resulted from the formation of corrosion products on exposed surface and advancement of it underneath the HDPE coating. Despite, adhesive degradation in most samples, there were also several specimens which demonstrated a resilience to the acid's effect on adhesion. This, in combination with the reality that this coating would not be intended for use in highly acidic environment, suggests that the coating has some viability as an anti-corrosive coating. However, it appears that this effectiveness depends on the uniform coating and sealing of the substrate. Therefore, if a seal cannot be achieved, then acid corrosion will occur and spread thereby compromising the coating.

4 CONCLUSION

The viability of using recycled HDPE coating on mild steel (simulation of automotive chassis) has been investigated. According to the SEM analysis, the coating was non-porous and filled the uneven surface of the mild steel substrate very well. The acid immersion tests indicated the corrosion occurred only at corners and other sharp fillets. This happened because the coating was not uniform and/or sufficient enough to prevent attack from acid solution. Similarly, the corrosion test in salt solution demonstrated that the corrosion started on the corners and edges and advanced other areas. However, no weight loss was observed for salt immersion test. Finally, the tape tests yielded high adhesive strengths in all categories of samples such as after salt immersion test, and as coated sam-

ples except acid immersion test. Notably, as coated samples and salt immersion samples exhibited the more or less similar ratings. This suggest that the salt solution had only minor effects on the bond between the coating and substrate. Considering the overall performance of the coating, specifically in the salt immersion test and adhesion tape test, the powder coat recycled HDPE can be used on automotive chassis to prevent corrosion. The coating performance can be improved further by improving the coating parameters so that the uniform coating can be achieved on sharp corners and edges.

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