CAAP Quarterly Report

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Prepared for: *U.S. DOT Pipeline and Hazardous Materials Safety Administration*

Project Title: Development of New Multifunctional Composite Coatings for Preventing and Mitigating Internal Pipeline Corrosion

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Business and Activity Section

(a) Generated Commitments

No changes to the existing agreement.

Matthew Pearson, former undergraduate research assistant, decides to pursue his M.S. degree and continues the work in the fields of pipeline corrosion control.

PIs have submitted two abstracts to the coming pipeline conference: the *12th International Pipeline Conference (IPC 2018)* in Calgary, Canada.

(b) Status Update of Past Quarter Activities

The research activities in the 5th quarter include refinement and optimization of the nano-modified coatings developed in Task 2, and characterization of the properties of the new coating system and particularly the features of abrasion resistance, anti-fouling and surface roughness as planned in Task 4, as summarized below.

Task 4: Characterize the new coating systems

4.1 Background and Objectives in the 5th Quarter

As discussed in the previous report, nanoparticles reinforced polymer has been studied to develop multi-functional composite coatings. Despite many efforts, most studies have limitations of using one type of nanofillers in polymer matrices. Furthermore, a hybrid filler system, which synergized by the integration of different nanofillers, could be used to fabricate nanocomposites with better dispersion state and stability. The influence of incorporating different nanoparticles into polymer has been studied in our previous work. The overall results have indicated the potentials of fabricating high-performance coatings.

The overall results have proven the potentials that the nanofiller reinforced polymer could yield high-performance coatings. However, there is still a lack of knowledge about the microstructure of the nanocomposites. Scanning electron microscopy (SEM) and atomic force microscopy (AFM) is the commonly used techniques to analyze the material on micro or nanoscale. In this study, we also performed an evaluation of nanofiller reinforcement in the polymer matrix by using SEM and AFM.

In this report, the experimental study was focused on the following subjects:

- Characterization of the corrosive behavior of new coating;
- Characterization of the anti-fouling of new coating;
- Characterization of the abrasive resistance of new coating;
- Characterization of the mechanical properties and microstructures of the new coating; and
- Characterization of more durable superamphiphobic coating system.

4.2 Systematically Experimental Program

4.2.1 Test matrix design

This section describes the strategy and methods of the experimental study in this report. The study plan includes three nanoparticles and two prime bases, hybrid nanofiller reinforcement, and contact surface modification. The sample preparation procedures and test methods from the previous study were continually used in this study.

For developing a high-performance coating, both nanoparticles reinforcement and the quality of the polymer should be considered. The abrasion resistance, fouling resistance, and corrosion resistance of the organic coatings was characterized by Taber Abraser method (ASTM D4060), Contact Angle Measurement (ASTM D7334), and Electrochemical Impedance Spectroscopy (EIS), respectively. The dog bone tensile test was performed to characterize the Young's modulus and the maximum tensile strength. Meanwhile, scanning electron microscopy (SEM) and atomic force microscopy (AFM) were used to evaluate the nanofiller reinforcement in micro scales.

4.2.2 Atomic force microscopy (AFM)

Surface roughness analysis and nanoindentation were performed with an atomic force microscopy, carried out on a Nanoscope IIIa system. Both 2-D and 3-D images were collected in ‘‘tapping-mode’’. By scanning the surface of the coating with a sample-probe, the height changes were recorded, and three-dimensional images will be formed in the process. The obtained data can also be used to analyze the surface roughness of the tested samples.

4.2.3 Scanning electron microscopy (SEM)

The micrographs that obtained in this study were carried out by a field emission scanning electron microscope (FE-SEM), with a JSM-7600F Schottky at 2 KV. Visual assessment of the prepared samples was able to be performed with this advanced electron microscopy technique. In our study, the obtained images were used to study the dispersion and agglomeration of the different nanofillers at varied concentrations.

4.3 Results and Discussions

4.3.1 Characterization of the new coating

The test results of the new coating were summarized in the section. A comparative study of three coatings was performed. Furthermore, SEM and AFM images were taken to study their microstructures.

a) Corrosion barrier performance using the EIS

The results of the EIS test for the new coating were summarized in Figure *1*. The highest impedance values were observed in SE05 and SE1 groups (0.5 and 1 wt. %). In a comparison of 1 wt. % of graphene/epoxy (GE1), which exhibit the highest impedance value from the previous report, stronger corrosion barrier property was observed in SE05 and SE1 groups.

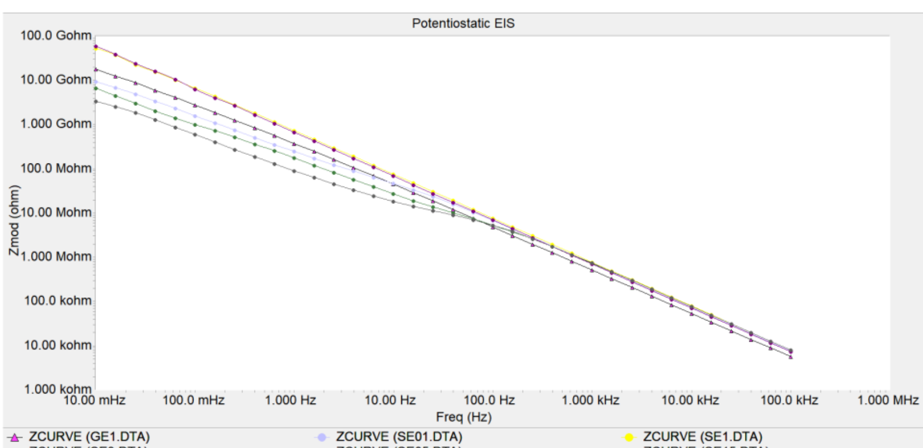

Figure 1 Impendence curve for nano modified composites

b) Abrasion resistance

The results of abrasion test for the new coatings were presented in Figure *2*. The abrasion resistance of the epoxy was significantly increased by the reinforcement of the nano particle. The lowest wear index value was observed in group SE3, which indicates maximum reinforcement was produced by the 3 wt. %. Also, the tendency of the curve shows that the wear index was continuously decreased by increasing the concentration and no degradation was observed in the test range.

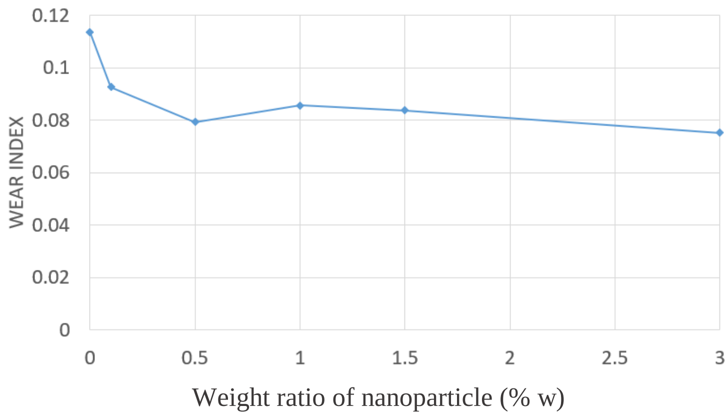

Figure 2 Wear index of nano modified composites

c) Contact angle test

From the previous work, the contact angle of water for pure epoxy is around 51 degrees. The contact angle of water was significantly decreased 16 degrees. This result indicates that incorporating such nano particles will not improve the hydrophobicity of the surface.

d) Dogbone tensile test

The tensile property of varied nanofiller reinforced epoxy was determined by the dogbone tensile test, following ASTM D638. The Nano-composites can be characterized by measuring tensile strength, strain at failure, and Young's modulus in this test.

e) Atomic force microscopy (AFM)

The surface roughness (Ra) of the nanocomposite coatings was measured by Atomic force microscopy on the nanoscale. The obtained height changes can be used to sketch a 3-D image of the tested samples. The surface roughness can also be calculated with these height change values.

The results of surface roughness of epoxy resin was dramatically decreased with the addition of the nanoparticles. The Ra value of pure epoxy is 29.4 nm, and it was decreased to less than 5 nm for most of the tested groups. These results indicate that a significant decrease in surface roughness can be obtained by adding a small amount of nanofillers.

f) Scanning electron microscopy (SEM)

Uniformly distributed, agglomeration size, higher concentration leads to larger agglomeration and more blank area, as typically shown in Fig. 4. These results can be used to explain the material degradation in the samples with high concentration of varying nanparticles.

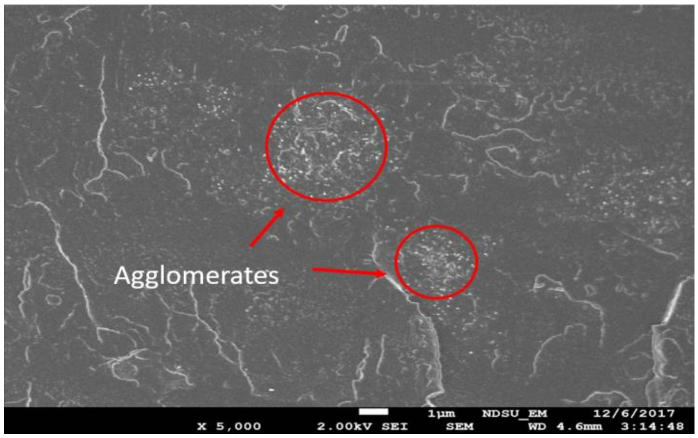

Figure 4 SEM micrograph of fracture surface of the new coating

4.3.2 Characterization of the new coating with hybrid nanofillers reinforcement

Like discussed in the previous section, three types of hybrid nanofillers were added into epoxy matrices to study their impacts to coating properties.

a) Corrosion barrier performance using the EIS

The results of corrosion resistance of the hybrid nanofiller/epoxy are shown in Figure . The obtained Zmod curves for each group were compared with the GE1 group as well. A significant improvement was observed in GSE1 group. The results indicate the combination has formed a nanoparticle network with extreme barrier protection properties.

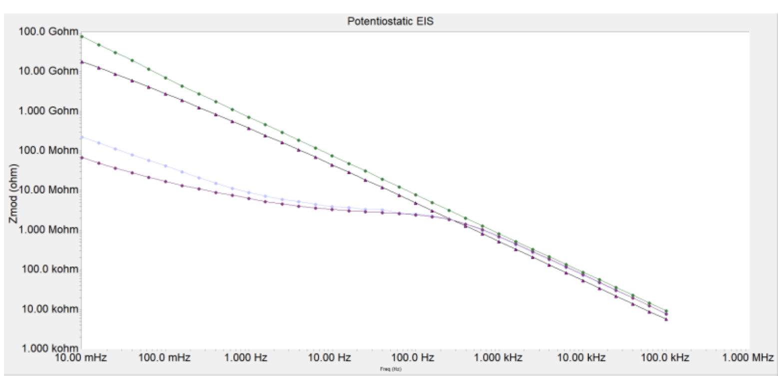

Figure 5 Impendence curve of hybrid nanofiller reinforced epoxy

b) Abrasion resistance

However, no significant improvement of the abrasion resistance was observed in the hybrid nanofillers groups. This result indicates the tested hybrid nanofillers will not significantly improve the abrasion resistance.

c) Contact angle test

The comparison of the contact angle with pure prime base revealed that the surface became extremely hydrophilic when the hybrid nanofiller were added to the epoxy resin.

d) Dogbone tensile test

The tensile properties of hybrid nanofiller reinforced epoxy were investigated. Compared with pure epoxy, there was no significant increases in max tensile stress and Young's modulus.

4.4 Future work

The following targets, as initially planned in Task 4, will be focused on the next step based on the current experimental results as follows:

- Characterization of the new coatings using different dosage for hybrid systems.
- Characterization of the new coatings using more detailed microstructures.
- Characterization of the new coatings particularly for anti-fouling properties.