CAAP Annual Report

Date of Report: *January 7, 2021*

Contract Number: *693JK31850009CAAP*

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

Project Title: New Bio-Inspired 3D Printing Functionalized Lattice Composites for Actively Preventing and Mitigating Internal Corrosion

Prepared by: *North Dakota State University*

Contact Information: *Mr. Andrew Mahoney, M.S. student, Email: Andrew.mahoney@ndsu.edu; Mr. Matthew Pearson, M.S. student, Email: matthew.pearson@ndsu.edu, Phone: 701-231-7204; Mr. Xingyu Wang, PhD student, Email: xingyu.wang@ndsu.edu;Dr. Hong Pan, Postdoc, Email: hong.pan@ndsu.edu;Mr. Adeeb Ibne Alam, PhD student, Email: adeeb.alam@maine.edu; Dr. Zhibin Lin, Email:zhibin.lin@ndsu.edu, Phone: 717-231-7204; Dr. Bashir Khoda, Email: bashir.khoda @ndsu.edu, Phone: 701-231-7195*

For quarterly period ending: *January 7, 2021*

Business and Activity Section

(a) Generated Commitments

Purchase made for the nano-materials, and the 3-D printable polymers

(b) Status Update of Past Quarter Activities

The research activities in the 9th quarter report aimed at continuing efforts on Task 6-substask: Enhanced durability study of the new composite lattices.

(c) Cost share activity

Cost share was from the graduate students' tuition waiver.

(d) Summary of detailed work for Task 6

Task 6: Assess long-term durability tests of the new composite systems

By continuing the attempts to increase the applications of the designed lattice structures for accommodating pipe environment, this study will lead the effort to address the durability of the surface treatment and enhance their efficiency.

6.1 Step 1: Enhanced durability study of the new composite systems

6.1.1 Basic information of enhancement using Surface Modification Method II

Breakthrough height is a viable means of determining the filtration effectiveness of a mesh/coating setup. The maximum height of a column of hexadecane can be used to convert to a hydrostatic pressure, an indication of the effectiveness of the filtration system. Because the setup described previously involving a rubber stopper was unsuccessful, other setups were explored.

One such setup involved the use of two 3D printed flanges, into which an O-ring could be inserted. Between the flanges would lay the mesh. A glass cylinder could then be inserted into the flanges and a clamp placed around the flanges to create a seal, as shown in **Fig. 1**.

Fig. 1 Flange fixture for mesh Breakthrough height testing

After testing this concept, it was determined that making a seal was not possible with this setup. Liquid would penetrate past the O-ring as well as up the glass walls of the cylinder and over the top of the flanges. Therefore, another sealing setup was attempted in which a plug would be inserted into the cylinder, as shown in **Fig. 2**. Again, this setup was determined inadequate to direct liquid only through the mesh. Even when a nonporous material such as aluminum foil was used, liquid still was able to leak around the plug. Therefore, it was decided a disposable setup was needed, shown later in the document.

Fig. 2 Plug fixture for Breakthrough height testing

6.1.2 Enhancement using Surface Modification Method II

6.1.2.1 Improvement of Surface Modification Method II

To further enhance the performance of the surface modification method II, three improvements were attempts in this period.

It was thought that the PLA wafers could be degrading from a hydrolysis reaction with the dilute acid found in the coating. Therefore, it was surmised that vacuum drying could enable quick drying of the wafer to reduce exposure time of the PLA to the liquid coating. The wafers remained unchanged during this time with no evidence of degradation. Drops of hexadecane were placed on each wafer type, displaying high contact angle, shown by **Fig. 3**.

Fig. 3 LS and R PLA wafers coated with drops of hexadecane showing good contact angle and no visual degradation

6.1.2.2 Synthesis II Comparison Test

The setup was shown by **Fig. 4**, where a small setup was prepared using assessment of oil column height to evaluate the effectiveness of Synthesis II and their variants of Synthesis II, as well as number of layer of coats.

Fig. 4 Experimental setup for oil column height

Wafers were prepared with different combinations of coating chemistry, mesh type (LS or R), or numbers of coats. All wafers had the 0.6 mm mesh size. To test, dyed hexadecane was added to the sample in 4 mL increments. To start the experiment, 4 mL were added, and at every 3-minute interval thereafter, 4 mL more of hexadecane were added. When hexadecane broke through the mesh, the maximum height of the oil column was recorded. The data is shown in graph form by **Fig. 5**.

6.2 Step 2: Enhanced durability study of the new composite systems

6.2.1 Basic information of enhancement using Surface Modification Method III

The LS and R wafers in the centrifuged category were centrifuged for each of the four coatings except for the coating involving suspended SiO_2 nanoparticles (Coating 3) since this would have caused the nanoparticles to settle. The weights of the wafers were measured before and after application of each of the four coatings in order to determine if coating was being deposited. A plot of the average mass of the four wafers before and after each coating is shown by **Fig. 5.**

It can be seen from **Fig. 5** that the mass change in each step, with the exception of before and after Coating 3, was very similar between the centrifuged and dipped methods. This reveals that when coating the wafers with solutions (as opposed to suspensions) centrifuging can be used due to its higher throughput.

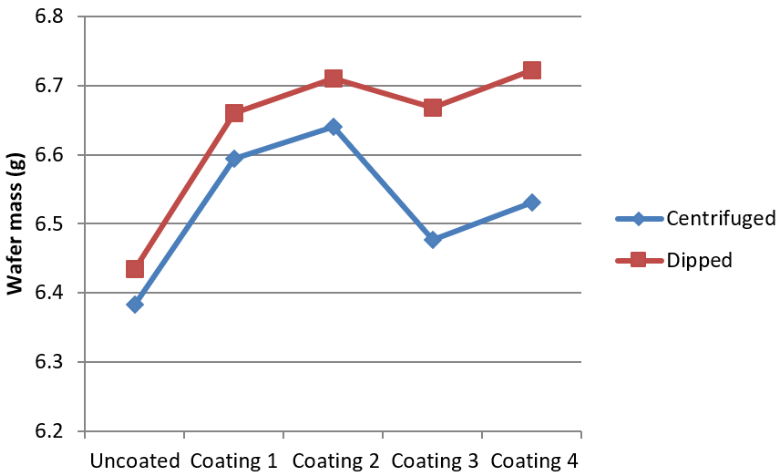

Fig. 5 Plot of average mass for each wafer over the course of each coating for both centrifuged and dipped coatings. Note that Coating 3 was applied by dipping for both cases.

6.2.2 Synthesis V Breakthrough Testing

A comparison reveals that results for LS mesh were generally consistent between Synthesis II and III chemistries. R type mesh showed greater breakthrough height with Synthesis II compared to Synthesis V and LS type consistently performed better than R type for each scenario. While the difference between chemistries for LS type is not large, Synthesis II has tentatively emerged as the better formulation, as it is a single-pot formulation (does not require multiple layers) and requires substantially less raw material.

6.3 Step 3: Enhanced durability study of the new composite systems with separation system concept and design

The 3D printing lattice with the proposed surface modification is able to separate water/oil mixture, as shown in Fig. 6. To improve the effectiveness of the lattice, the inclination will enhance the entire design and effectively implement the proposed concept in Task 8. As such, a new separation system is developed, as schematically shown in **Fig. 6**. As illustrated in **Fig. 6**, the system consists of: (i) a top tube with inlet (see Fig. 6c); (ii) 3D printing lattice with the proposed surface modification to separate water/oil mixture (see Fig. 6d) and allow them separately to flow to (iii) oil container (see Fig. 6b) and (iv) water container (see Fig. 6e).

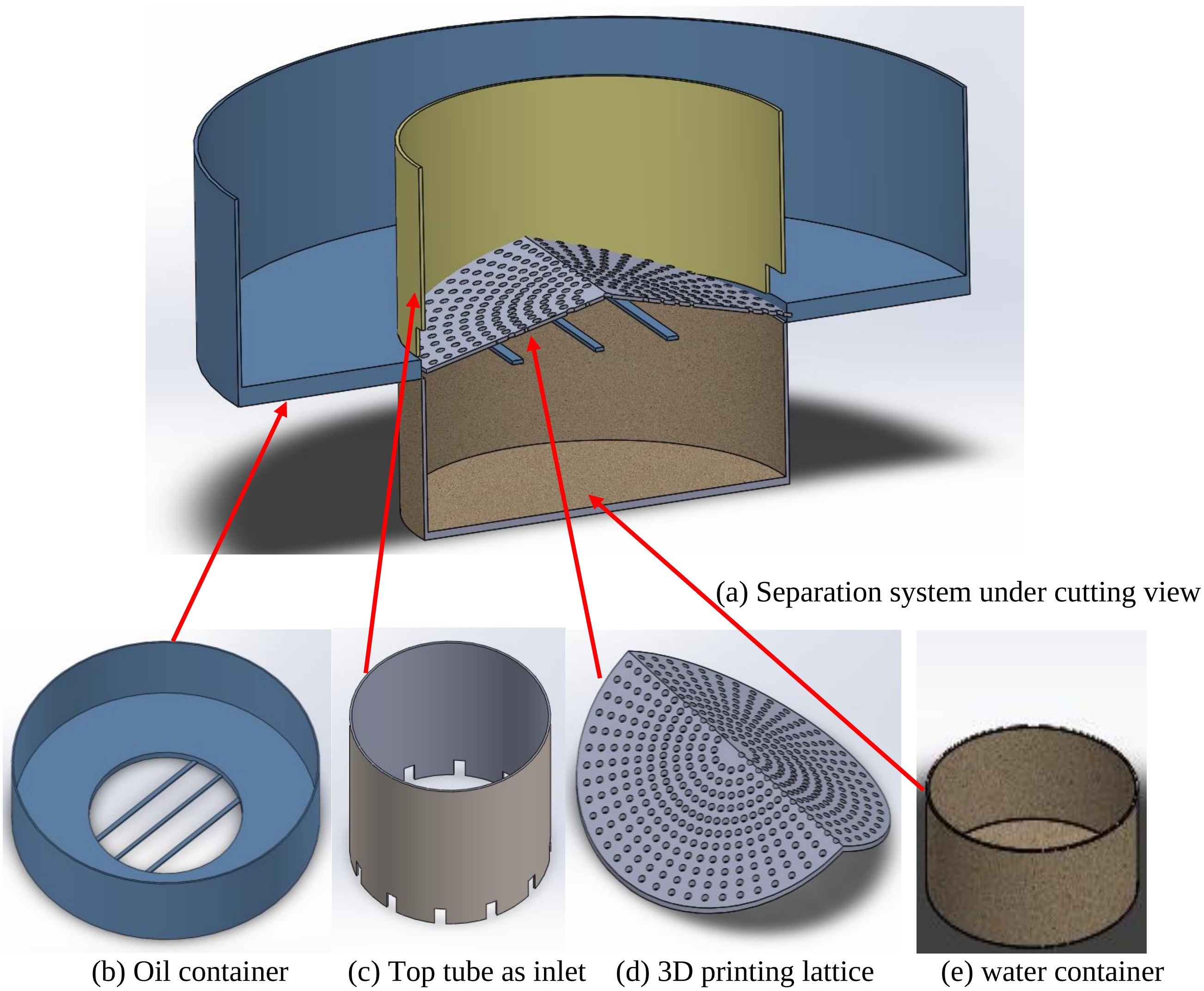

Fig. 6 Schematics of the test setup

-Summary of the work

This research activities in the 9th quarter included: (i) Continuing efforts on durability of the new 3D printing lattice composites in **Task 6**; and enhanced durability with separation system concept and design.

(e) Description of any Problems/Challenges

No problems are experienced during this report period

(f) Planned Activities for the Next Quarter

The planned activities for next quarter are listed below:

- More efforts on investigation of the sliding performance of the new composite with surface modification (Synthesis II).
- More efforts on investigation of the effectiveness of enhanced durability study of the new composite systems with separation system concept and design.