Polymeric Nanocomposite Coatings for Corrosion Protection of Steel

### Research Problem

Corrosion is a major problem in the oil and gas industry, and it is one of the main concerns in the durability of the steel pipelines. As pipelines are mostly buried in seabed, the steel is subjected to various corrosion factors (water, oxygen, PH changes) causing corrosion to occur easily.

![Figure 1: picture showing corrosion in pipelines](image)

Corrosion is an electrochemical process where chemical reaction occurs between the steel pipelines and the surrounding environment producing oxides, which occupies more space which causing the pressure and cracks that weakens the pipelines gradually [5]. This problem negatively affects the economy, the safety, and the environment.

- The global cost of corrosion is about $2.5 trillion, which is equivalent to 3.4 % of GDP. Most of the cost is due to inspection, repairs, maintenance and safety. Studies showed that corrosion prevention results in 15-35% of the global savings of the cost [6].

### Research Hypothesis

A new nanocomposite smart coating that can be synthesized and evaluated for its ability to protect steel from corrosion by acting as corrosion inhibitor and as self-healing agent.

![Figure 2: Schematic representation of polymeric nanocomposite coatings](image)

### Research Objectives

- **Synthesis** and performance evaluation of polymeric nanocomposite epoxy coatings.
- **Encapsulation** of the self-healing agent (NMTU) inside the Halloysite nanotubes.
- **Structural characterization** of the encapsulated Nano containers and the developed epoxy coatings (SEM, XRD, FTIR, TGA).
- **Evaluation** of corrosion inhibition performance of the developed epoxy coatings by EIS technique with Gamry instrument.

### Materials and Equipment

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<tr>
<th>Materials</th>
<th>Equipment</th>
<th>Testing equipment</th>
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<tbody>
<tr>
<td>N-Methyl Thionine (NMTU)</td>
<td>Magnetic stirrer</td>
<td>SEM</td>
</tr>
<tr>
<td>halloysite nanotubes/Nanocrystals (HNT)</td>
<td>Vacuum chamber</td>
<td>FTIR</td>
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<td>Epoxy resin</td>
<td>Centrifuge</td>
<td>TGA</td>
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<td>carbon steel substrate</td>
<td>polisher</td>
<td>XRD</td>
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<tr>
<td>Hardener</td>
<td>Sonicator</td>
<td>GAMRY</td>
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![Figure 3: diagram showing materials, equipment and testing equipment](image)

### Methodology

#### Research Procedure:

The procedure included 3 processes:

1. **Loading process**
   - NMTU + NANTOBES = LOADED PRODUCT
   - a. Mixing 2 g of NMTU and 1 g of nanotubes in 10 ml of water and stirring them by magnetic stirrer
   - b. Placing the sample in the vacuum chamber to help encapsulation of NMTU into nanotubes for 24 hours
   - c. Centrifuge for 15 min. using centrifuge device to separate the loaded product
   - d. Drying the sample in room temperature for 24 hours

2. **coating process**
   - a. Polishing of the steel substrate using polisher machine to remove any impurities from the surface and to keep it smooth
   - b. Mix 10 g of epoxy with 2.5 g of hardener and add 1.25 g of loaded product in it
   - c. Removing bubbles from the epoxy resin using ultrasonic bath to ensure homogenous mixture of epoxy
   - d. Coating the steel substrate with the synthesized epoxy and coating thickness is controlled by doctor blade
   - e. The previous steps are repeated for another sample of steel substrate but without adding the loaded product, this is called the reference coating

3. **Testing process**
   - a. Structural characterization using SEM, FTIR, TGA and XRD
   - b. Corrosion testing using Gamry 3000 device to measure the corrosion resistance by Electrochemical Impedance Spectroscopy (EIS) analysis

![Figure 4-6: vacuum chamber, polishing step, coating process](image)

### Data and Information Analysis

1. **Scanning Electron Microscope- SEM**
   - TheSEM results showsthe tubular smooth surface of the nanotubes which ensures the successful encapsulation of the self-healing material into the Halloysite nanotubes.

![Figure 8: picture of SEM results](image)

2. **Fourier Transform Infrared Spectroscopy- FTIR**
   - It was performed to confirm the loading of inhibitor/self-healing agent (NMTU) inside the HNTs. The change in the characteristic peaks observed for loaded product that shows the loading of the inhibitor in HNTs.

![Figure 9: picture of FTIR results](image)

3. **X-ray diffraction-XRD**
   - The XRD results provide the structural details of products and sharp peaks show the crystalline nature of product. New sharp peak appeared around 29°-30° for loaded product with NMTU. That confirms the loading of product inside HNTs.

![Figure 10: picture of XRD results](image)

4. **Thermogravimetric Analyzer-TGA**
   - TGA analysis provides the thermal stability and loaded capability of HNTs. The initial weight loss till 100 °C is due to moisture content. Around 5 wt.% product of NMTU is successfully loaded inside HNTs.

![Figure 11: picture of TGA results](image)

5. **Electrochemical Impedance Spectroscopy**
   - A scratch was made on the reference and modified coating in order to check their anti-corrosive property. The coated samples were tested in 3.5 wt.% of NaCl solution (corrosive medium) for 120 hrs. The modified coatings shows the increase in corrosion resistance with time as compared to reference coating.

![Figure 12-13: EIS results of reference coating and modified coating](image)

### Results Summary

- Structural characterization using SEM, FTIR, TGA and XRD ensured successful encapsulation of NMTU inside the Halloysite nanotubes.
- The EIS results showed the corrosion resistance of reference coatings decreases with time that can be shown in bode and phase angle plot in figure (12).
- The EIS results showed the corrosion resistance of modified coatings increases with time that can be shown in bode and phase angle plot in figure (13).
- From EIS results, the increase in corrosion resistance for modified coating is due to better corrosion inhibition and self-healing coatings.

### References