Characterization of Encapsulated Corrosion Inhibitors for Environmentally Friendly Smart Coatings

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NASA Kennedy Space Center Corrosion Technology Laboratory
Corrosion

• Worldwide corrosion cost: $2.2 trillion (2010)
• US cost: ~$1 trillion (2013)
• Replace current corrosion inhibitors with environmentally friendly alternatives
  – Coating compatibility issues
  – Solubility issues

http://philipmarshall.net/Images/corrosion_hyperphysics.gif
Delivery System

Inhibitor

Coating compatibility
Inhibitor solubility

Corrosion Protection

Coating
KSC Approach

- “Smart coating” for corrosion sensing and control
  - Autonomous
  - pH controlled
  - Universal

Microcapsule containing pH indicator (inhibitor, self healing agents)

The shell of the microcapsule breaks down under basic pH (corrosion) conditions

pH indicator changes color and is released from the microcapsule when corrosion starts
Release Video
RELEASE STUDIES
Inhibitor Release

• Determine release of inhibitor with time
  – 2-Mercaptobenzothiazole (2-MBT)
  – Nitrite
  – Molybdate

• Method
  – Immersion of particles into 0.01 M base
  – Sampling at regular intervals
MF: 2-MBT Short-term Release

Percent 2-MBT Released vs Microparticle Mass

Time (hours)

0 50 100 150 200 250 300 350 400

Standard MFPTT Formula
No PTT
Higher Formaldehyde
Highest Formaldehyde
Higher Melamine
Highest Melamine
MF: 2-MBT Long-term Release

![Graph showing the percent 2-MBT released vs microparticle mass over time for different formulas.]

- Standard MFPTT Formula
- No PTT
- Higher Formaldehyde
- Highest Formaldehyde
- Higher Melamine
- Highest Melamine

The graph indicates the cumulative release of 2-MBT over time, with varying levels of formaldehyde and melamine influencing the release rate.
Inorganic: 2-MBT

![Graph showing the release of 2-MBT over time for different samples.](image-url)
Inorganic: Nitrite

![Graph showing percent nitrite released vs microparticle mass over time. The graph has three lines representing different samples: SiNO2-C13.5 I, SiNO2-C70 I (50%), and SiNO2-C65 I. The x-axis represents time in hours, ranging from 0 to 2500, and the y-axis represents the percent nitrite released versus microparticle mass, ranging from 0% to 6%. The graph shows varying trends for each sample over time.]
Inorganic: Molybdate

Percent Molybdate Released vs Microparticle Mass

Time (h)

SiMo-C13.5 Mo I
SiMo-C13.5 Cl- Mo I
Release Studies

• Successful encapsulation and release of inhibitor

• Organic particles
  – Inhibitors can react with particle material
  – Slower, longer-term release

• Inorganic particles
  – Can incorporate a variety of inhibitors, including highly water soluble ionic compounds
  – Quicker, higher amount release
ELECTROCHEMICAL CORROSION TESTING
Accelerated Corrosion Testing

• Carbon steel in 3.5% NaCl solution
• Electrochemical measurements
• Salt immersion
  – Phenylphosphonic acid (PPA)
  – 8-Hydroxyquinoline (8-HQ)
  – 2-MBT & Sodium 2-Mercaptobenzothiazole (2-MBTNa)
Corrosion Potential Increase

![Graph showing the increase in corrosion potential for different pH levels and substances.](image)
Polarization Resistance

- Control pH 2
- Control pH 4
- Control pH 7
- Control pH 9
- 0.1% PPA
- 0.1% 8-HQ
- 0.1% 2-MBT
- 0.1% MBTNa
- 0.1% PPA
- 0.1% 8-HQ
- 0.1% 2-MBT
- 0.1% MBTNa
- 0.1% PPA
- 0.1% 8-HQ
- 0.1% 2-MBT
- 0.1% MBTNa
- 0.1% PPA
- 0.1% 8-HQ
- 0.1% 2-MBT
- 0.1% MBTNa
- 0.1% PPA
- 0.1% 8-HQ
- 0.1% 2-MBT
- 0.1% MBTNa
- 0.1% PPA
- 0.1% 8-HQ
- 0.1% 2-MBT
- 0.1% MBTNa
SALT IMMERSION TESTING
### Pure Inhibitor: PPA

<table>
<thead>
<tr>
<th>Time</th>
<th>Control</th>
<th>0.1% PPA</th>
<th>0.1% PPA and 0.1% 8-HQ</th>
<th>0.1% PPA and 0.002% 2-MBT</th>
<th>0.1% PPA and 0.1% NaMBT</th>
<th>0.1% PPA, 0.1% 8-HQ and 0.002% 2-MBT</th>
<th>0.1% PPA, 0.1% 8-HQ and 0.1% NaMBT</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td><img src="image1" alt="Initial Control" /></td>
<td><img src="image2" alt="Initial 0.1% PPA" /></td>
<td><img src="image3" alt="Initial 0.1% PPA and 0.1% 8-HQ" /></td>
<td><img src="image4" alt="Initial 0.1% PPA and 0.002% 2-MBT" /></td>
<td><img src="image5" alt="Initial 0.1% PPA and 0.1% NaMBT" /></td>
<td><img src="image6" alt="Initial 0.1% PPA, 0.1% 8-HQ and 0.002% 2-MBT" /></td>
<td><img src="image7" alt="Initial 0.1% PPA, 0.1% 8-HQ and 0.1% NaMBT" /></td>
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<td>5 hour</td>
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<td><img src="image11" alt="5 hour 0.1% PPA and 0.002% 2-MBT" /></td>
<td><img src="image12" alt="5 hour 0.1% PPA and 0.1% NaMBT" /></td>
<td><img src="image13" alt="5 hour 0.1% PPA, 0.1% 8-HQ and 0.002% 2-MBT" /></td>
<td><img src="image14" alt="5 hour 0.1% PPA, 0.1% 8-HQ and 0.1% NaMBT" /></td>
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<td>6 day</td>
<td><img src="image15" alt="6 day Control" /></td>
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<td>After Wash</td>
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## Pure Inhibitor: 8-HQ

<table>
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<tr>
<th>Time</th>
<th>Control</th>
<th>0.1% 8-HQ</th>
<th>0.1% 8-HQ and 0.002% 2-MBT</th>
<th>0.1% 8-HQ and 0.1% NaMBT</th>
<th>0.1% PPA and 0.1% 8-HQ</th>
<th>0.1% PPA 0.1% 8-HQ and 0.002% 2-MBT</th>
<th>0.1% PPA 0.1% 8-HQ and 0.002% NaMBT</th>
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<td>After Wash</td>
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## Pure Inhibitor: 2-MBT

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<th>0.1% PPA and 0.002% 2-MBT</th>
<th>0.1% 8-HQ and 0.002% 2-MBT</th>
<th>0.1% PPA, 0.1% 8-HQ and 0.002% 2-MBT</th>
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<tr>
<td>1 day</td>
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<td><img src="image14.png" alt="Image" /></td>
<td><img src="image15.png" alt="Image" /></td>
</tr>
<tr>
<td>Steel Piece</td>
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<td><img src="image17.png" alt="Image" /></td>
<td><img src="image18.png" alt="Image" /></td>
<td><img src="image19.png" alt="Image" /></td>
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# Pure Inhibitor: 2-MBTNa

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<th>Time</th>
<th>Control</th>
<th>0.1% NaMBT</th>
<th>0.1% PPA and 0.1% NaMBT</th>
<th>0.1% 8-HQ and 0.1% NaMBT</th>
<th>0.1% PPA, 0.1% 8-HQ and 0.1% NaMBT</th>
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# Particles: PPA

<table>
<thead>
<tr>
<th>Time</th>
<th>Control</th>
<th>0.3% PPA Particles</th>
<th>0.3% PPA Particles 0.25% 8-HQ Particles</th>
<th>0.3% PPA Particles 0.004% 2-MBT Particles</th>
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<tr>
<td>Initial</td>
<td><img src="image" alt="Initial Control" /></td>
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<tr>
<td>1 day</td>
<td><img src="image" alt="1 day Control" /></td>
<td><img src="image" alt="1 day 0.3% PPA Particles" /></td>
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<td>6 day</td>
<td><img src="image" alt="6 day Control" /></td>
<td><img src="image" alt="6 day 0.3% PPA Particles" /></td>
<td><img src="image" alt="6 day 0.3% PPA Particles 0.25% 8-HQ Particles" /></td>
<td><img src="image" alt="6 day 0.3% PPA Particles 0.004% 2-MBT Particles" /></td>
<td><img src="image" alt="6 day 0.3% PPA Particles 0.25% 8-HQ Particles 0.004% 2-MBT Particles" /></td>
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<tr>
<td>Before Wash</td>
<td><img src="image" alt="Before Wash Control" /></td>
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</table>
# Particles: 8-HQ

<table>
<thead>
<tr>
<th>Time</th>
<th>Control</th>
<th>0.25% 8-HQ Particles</th>
<th>0.3% PPA Particles</th>
<th>0.25% 8-HQ Particles</th>
<th>0.25% 8-HQ Particles</th>
<th>0.3% PPA Particles</th>
<th>0.25% 8-HQ Particles</th>
<th>0.004% 2-MBT Particles</th>
<th>0.3% PPA Particles</th>
<th>0.25% 8-HQ Particles</th>
<th>0.004% 2-MBT Particles</th>
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<tr>
<td>Initial</td>
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<td><img src="Image2" alt="Image" /></td>
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<td><img src="Image10" alt="Image" /></td>
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<tr>
<td>1 day</td>
<td><img src="Image12" alt="Image" /></td>
<td><img src="Image13" alt="Image" /></td>
<td><img src="Image14" alt="Image" /></td>
<td><img src="Image15" alt="Image" /></td>
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<td>Before Wash</td>
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### Particles: 2-MBT

<table>
<thead>
<tr>
<th>Time</th>
<th>Control</th>
<th>0.009% Inorganic 2-MBT Particles</th>
<th>0.3% PPA Particles 0.004% 2-MBT Particles</th>
<th>0.25% 8-HQ Particles 0.004% 2-MBT Particles</th>
<th>0.3% PPA Particles 0.25% 8-HQ Particles 0.004% 2-MBT Particles</th>
</tr>
</thead>
<tbody>
<tr>
<td>Initial</td>
<td><img src="image1" alt="Control" /></td>
<td><img src="image2" alt="0.009% Inorg 2-MBT Part" /></td>
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<td>Before Wash</td>
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</tbody>
</table>
Conclusion

- Successful encapsulation of various inhibitors into organic & inorganic microparticles
- Release of inhibitor monitored over long periods of time → short- and longterm controlled release
- Corrosion protection of pure materials confirmed through electrochemical testing
- Particles effective at preventing corrosion in salt immersion testing
- Inhibitors combinations showing high corrosion inhibition efficiency
Synthesis: Organic Particles

Melamine + Formaldehyde $\rightarrow$ Methylol melamine (MM) $\rightarrow$ Base $\rightarrow$ Melamineformaldehyde (MF)

Base $\rightarrow$ Melamineformaldehyde Pentaerythritol tetrakis (MF-PTT)

Melamine + Methylol melamine (MM) $\rightarrow$ Melamineformaldehyde (MF)

2-Mercaptobenzothiazole (2-MBT) + Melamineformaldehyde (MF) $\rightarrow$ MNH$_2$N-C=S-S-NH$_2$
Pure Inhibitor Solution pH

<table>
<thead>
<tr>
<th>pH Before Polarization</th>
<th>Control pH 2</th>
<th>Control pH 4</th>
<th>Control pH 7</th>
<th>Control pH 9</th>
<th>0.1% PPA</th>
<th>0.1% PPA</th>
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pH Change during Polarization

![Graph showing pH change during polarization](image)

- **Anodic Polarization**
- **Cathodic Polarization**

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<th>Condition</th>
<th>pH 2</th>
<th>pH 4</th>
<th>pH 7</th>
<th>pH 9</th>
<th>0.1% PPA</th>
<th>0.1% 8-HQ</th>
<th>0.1% 2-MBT</th>
<th>0.1% MBTNa</th>
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