Encapsulation

Introduction

The encapsulation methods reported in the literature\textsuperscript{1-7} for the production of microcapsules are generally achieved using one of the following techniques:

1. Phase separation
2. Spray drying, spray congealing
3. Solvent evaporation
4. Coating

The choice of particular method and shell material will depend on the physiochemical properties of the active substance, the desired particle size and release characteristics. From a technological point of view, the successful selection of a preparation method will be determined by the ability to achieve high loadings with the active substance (high encapsulation efficiency), high product yields, and the potential for easy scale-up.

Battelle Technology

Battelle encapsulation technology is based on a particle forming polymerization approach. This method is amenable to encapsulate both solid and liquid active ingredients. Particles from nanometer to micron size are obtained using our encapsulation process. The idealized representation of our technology is given in Figure 1. In our process, the active ingredients are suspended in the medium using a stabilizer along with the desired shell forming monomer and initiator. The initial polymerization occurs in solution and as the molecular weight increases the polymer precipitates onto the active ingredients. The key to the success of dispersion polymerization is the choice of stabilizers, and type of monomer and solvents. If the active ingredients are water insoluble, either aqueous dispersion polymerization or other particle forming polymerization such as emulsion and suspension polymerization could be employed.
Figure 1. Mechanism of particle formation (a) homogeneous polymerization medium (b) Nucleation stage and (c) Stabilization of polymeric particles by the added stabilizer
Note: In the presence of active ingredients (AI) the AI acts as nucleus for the polymer growth and eventually the polymer shell will be formed around the AI core.

We have demonstrated the efficacy of our process in encapsulating a wide variety of active ingredients. Some examples are highlighted in the following Figure 2.

Polyurethane microspheres filled with agrochemicals
Release Mechanism: Diffusion

Polyacrylate microspheres filled with liquid catalyst
Release Mechanism: Temperature

Hydrogels filled with solid reactive active materials
Release Mechanism: pH

Microspheres filled with Self healing materials
Release Mechanism: Corrosion

Flow of self healing materials into the crack site

Figure 2: Battelle Encapsulation Capabilities

Battelle also has expertise and capabilities in conventional physical encapsulation methods such as phase separation, spray drying solvent evaporation and coating methods. Based on the performance requirement, Battelle will identify the most suitable encapsulation methodology.
The Battelle Smart Corrosion Detector® bead

The Battelle Smart Corrosion Detector® bead is a microscopic bead that can detect corrosion forming on a metal substrate, deliver a payload to heal damage caused by the corrosion, and provide an early warning sign that corrosion is present. The beads, 30- to 50-μm diameter spherical capsules filled with a liquid healing agent, resemble a fine, whitish powder in bulk and are designed to be mixed into coatings that protect critical infrastructure from corrosion.

These self-healing smart beads detect and reveal corrosion forming on metal before it is visible to the naked eye. When corrosion is present, the beads’ surfaces undergo a chemical reaction (Figure 3) that causes them to fluoresce (which can be detected with an ultraviolet [UV] light or Terahertz imaging), then break apart and release a healing agent. The fluorescence is a prompt indicator to maintainers that corrosion has initiated and provides them with the opportunity to mitigate the underlying problem early on, while the healing agent immediately repairs the corrosion damage and slows the corrosion process. The timely discovery and remediation of corrosion can result in significant time and cost savings as well as improved structural reliability.

- When ferrous or non-ferrous substrates corrode, positive metal ions are formed:
  \[
  \text{Al}^{(s)} \rightarrow \text{Al}^{3+} \]
  &
  \[
  \text{Fe}^{(s)} \rightarrow \text{Fe}^{3+} \]
- When 8-hydroxyquinoline (HQ) compound is present, the metal ions complex with the HQ:
  \[
  3 \text{OH}_2\text{N} \quad + \quad \text{Al}^{3+} \quad \rightarrow \quad \text{Al}([\text{HQ}]_3

![Terahertz Imaging of Corrosion]

Figure 3: The Battelle Smart Corrosion Detector® bead’s mechanism of action
The Battelle Smart Corrosion Detector® bead is a single-component microcapsule technology comprised of functional microspheres with storage stable healing agent. The key features of our product are compared with competing technology in the following table and schematically described in Figure 4.

<table>
<thead>
<tr>
<th>Battelle Smart Corrosion Detector® bead</th>
<th>Competing Technology</th>
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<tr>
<td>Functional microcapsules capable of sensing corrosion byproducts and releasing healing agent when corrosion occurs.</td>
<td>Microcapsules are not functionalized and do not sense corrosion. Releases the healing agent only due to structural damage of the coatings.</td>
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<tr>
<td>Single component system, does not require additional microcapsules to cure the healing agent. The healing agent is cured by the corrosion byproducts.</td>
<td>Requires two capsules (generally catalyst) or favorable environment such as oxygen or water to cure the healing agent.</td>
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<tr>
<td>Moisture and oxygen permeability does not affect the stability of the healing agent. Therefore the healing agent is very stable and will not cure inside the microcapsules.</td>
<td>Limited shelf life and application as the healing agents used in the competing technology are highly unstable as they readily react with oxygen or moisture.</td>
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</tbody>
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![Figure 4: The Battelle Smart Corrosion Detector® bead’s technology differentiation](image-url)
References:

1. US 2003138557, "Composition and method for the encapsulation of water-soluble molecules with polymers into nanoparticles", (2003), Assignee PR Pharmaceuticals, Inc., USA.


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