Understanding Microcapsule Properties for Developing Consumer Products

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### Biological and Non-biological Materials (1μm - 1mm)

<table>
<thead>
<tr>
<th>Biological Materials</th>
<th>Non-Biological Materials</th>
</tr>
</thead>
<tbody>
<tr>
<td>animal cells</td>
<td>microcapsules (encapsulates)</td>
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<tr>
<td>yeasts</td>
<td>microspheres</td>
</tr>
<tr>
<td>Bacteria</td>
<td>agglomerates</td>
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<tr>
<td>fungal hyphae</td>
<td>granules</td>
</tr>
<tr>
<td>plant cells</td>
<td>textile fibres</td>
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<tr>
<td>cell and debris flocs</td>
<td>ice crystals</td>
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<tr>
<td>skin cells and chondrocytes</td>
<td>bubbles</td>
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<tr>
<td>starch granules</td>
<td>structured liquids</td>
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<tr>
<td>pollen grains</td>
<td></td>
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<tr>
<td>biofilms and food fouling deposits</td>
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</tbody>
</table>
Formulation and Characterisation of functional microcapsules

- Pressure-sensitive materials (e.g. melamine formaldehyde perfume capsules; capsules for self healing materials)
- Carriers of speciality chemicals (e.g. peroxide, antimicrobial agents, herbicide)
- Drugs (water soluble, non-soluble, protein)
- Probiotic cells (bacteria and yeast)
- Nutraceutical enzyme (Nattokinase)
http://www.scienceinthebox.com/laundry-perfumes-provide-fresh-scents
Properties of Capsules

- Core and wall chemical compositions
- Morphology, size and wall thickness
- **Mechanical strength**
- Pore size, structure of wall materials and **release rate**
- Surface charge
- **Adhesion on surface**
- Functionality of active ingredient
Formation of a melamine formaldehyde (M-F) wall on the surfaces of oil droplets.

Sun and Zhang (2001) *J Microencapsulation*
Transmission electron microscopy (TEM) image of melamine formaldehyde microcapsules.

Schematic diagram of the micromanipulation rig

Zhang, Saunders & Thomas (1999) *J Microencapsulation*
Force versus displacement for compression of a microcapsule to rupture.

Sun and Zhang (2001) *J Microencapsulation*
Micromanipulation to measure the rupture force of single encapsulates and finite element modelling (FEM) to determine their intrinsic mechanical property parameters.
Determination of the Elastic Modulus ($E$):

- MF encapsulates are known to be elastic at small fractional deformations $\varepsilon < 0.15$

- The force profile depends on $h/r$ at small fractional deformations

- We can estimate $h/r$ using the shape of the force profile

Once $h$ is known we can estimate $Eh$

Compare experimental force curve with FEM results at the appropriate $h/r$

**FEA results:**

\[
\frac{F}{E rh} = a \varepsilon^2 + b \varepsilon + c
\]

$0.03 < \varepsilon < 0.1$

The experimental $Eh$ is calculated at different fractional deformations

\[
Eh_\varepsilon = \frac{F_\varepsilon/r}{a \varepsilon^2 + b \varepsilon + c}
\]
**MF Encapsulates – Elastic shell – Estimate $E_h$**

$E_h \sim 355 \pm 60 \text{ N/m}$

$h \sim 0.2 \mu\text{m}$

$E \sim 1.8 \pm 0.3 \text{ GPa}$

$E_h$ is independent of the encapsulate size.
• At high deformations (e.g. \( \varepsilon > 0.1 \)), MF microcapsules deform plastically
• Consider the simplest plasticity scenario: Perfect plasticity

Mercadé-Prieto et al. (2011b)
FEM – Determination of rupture parameters

\[ \sigma_B \approx 325 \text{ MPa} \]

\[ \sigma_Y \approx 165 \text{ MPa} \]

\[ E \approx 7 \text{ GPa} \]

\[ T \approx 1 \text{ GPa} \]

Mercadé-Prieto et al. (2012) *AIChEJ*
Schematic diagram of the nano-manipulation device in an ESEM

ESEM (LHS) and TEM (RHS) images of the MF, ripened NP CaCO₃ and double shell composite microcapsules

Percentage leakage of the core oil from the MF, ripened NP CaCO3 and double shell composite microcapsules over 24 hours.
Schematic diagram of the release of the inner perfume oil through the microcapsule shell.

Mercadé-Prieto et al. (2012) J. Microencapsulation

\[ J = \frac{D}{h} (c_{in(s)} - c_{out(s)}) = \frac{P}{h} (c_{in} - c_{out}) \]
Saturation concentration \( (c_s) \) of hexyl salicylate in different water-solvent solutions at 22\(^{\circ}\)C
The corresponding mean P/h values obtained using different cosolvents.
Is there any relationship between the fracture strength and oil release rate?

The fracture strength is mainly determined by the macro-structure.

\[
\frac{F}{Erh} = a\varepsilon^2 + b\varepsilon + c \quad 0.03 < \varepsilon < 0.1
\]

The oil release rate is dominated by the fine structure, particularly for small molecules.

\[
J = \frac{D}{h} (c_{in(s)} - c_{out(s)}) = \frac{P}{h} (c_{in} - c_{out})
\]

Shell thickness \( h \) affects both the fracture strength and oil leakage rate!
Schematic of an AFM set up.
SEM image showing an encapsulate (11.9 μm) was attached to a tipless cantilever

Adhesion Investigation by AFM

Schematic representations of steps during a typical force interaction between an encapsulate and a cellulose film.

He et al. (2014) *J Microencapsulation*
Adhesion Force between encapsulates and Cellulose Films

Mean adhesion between 5 encapsulates and a cellulose film before and after being modified with chitosan solution.
Fabric care R&D in Procter & Gamble

Laundry Liquid Detergents (HDL)

Fabric Enhancers

Laundry UnitDose
Conclusions

• Perfume microcapsules are required to have non/low permeability, strong adhesion on fabric surface and optimum mechanical strength.

• Functional perfume capsules with different size, structure, surface property, mechanical strength and permeability have be prepared using various formulation and processing conditions to meet industrial needs.

• Micromanipulation has been demonstrated to be a very powerful tool to characterise the mechanical properties of capsules and to infer their structure, and their mechanical strength can be used as a trigger to control the release of core materials, e.g. perfume.
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