Microencapsulation Studies for Mass Production of IFE Targets

B. Vermillion, G. Besenbruch, L. Brown, D. Goodin, B. McQuillan, M. Takagi, T.Woo

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Goals of Experimental Study

Overall

• Examine scale-up opportunities utilizing chemical engineering principals and apparatus
• Study methods to reduce costs associated with mass production

Specific

• Produce 4.6 mm/250 micron wall polystyrene targets
• Produce a stock of targets for additional coating studies
• Design and test apparatus for mass production of targets
Microencapsulation utilizes a triple orifice generator

- Three Phases
  - Inner aqueous phase (DI water)
  - Polymer phase (polystyrene in solution with fluorobenzene)
  - Outer aqueous phase (poly acrylic acid in DI water)
Mass Production of IFE Targets differs from Lab Scale Production

- Desire to scale-up production of polymer targets to at least 500,000/day
  - Liquid ratio assumptions
    - Ratio = Volume of solution plus targets/volume of targets
    - Lab scale 200 : Production scale assumes 10
  - Need for recycle: >3,000 L of outer aqueous phase per 100,000 target production run
  - Methods to remove contaminants
  - Parametric study to determine flowrates
    - Operating in a different regime for IFE target production
  - Optimizing cure times
    - Surface defects, low mode
      - Controlling the Marangoni effect
    - Rate of fluorobenzene removal

\[
M = \frac{\frac{dL}{dc}}{D}
\]
Target Specification influence scale-up decisions

- Possible specifications for larger targets affect scale-up design and cost
  - OD: 4600 +/- 5%
    - Over and Under: Acceptable Yield?
  - Wall: 250 +/- 10%
  - OOR: <1% of radius

If one assumes OOR scales with $R^3$:

$$\frac{3\text{mm}}{0.36\%} = \frac{4.6\text{mm}}{0.86\%}$$

- NC: < 1% to 5% of wall thickness
- Surface Finish: 20-200 nm RMS

Polystyrene targets: 3 mm diameter/30 micron walls
Exploring Scale-Up Opportunities

- Laboratory Scale
  - Droplet Generation
  - Prototype Gas Agitated Contactor
- Mass Production
  - Rotary Contactor

Los Alamos National Laboratory
IFE Shell-Making Laboratory is in Operation

### Process Conditions

<table>
<thead>
<tr>
<th>Run #</th>
<th>O1 Composition and Flowrate</th>
<th>W1 Composition and Flowrate</th>
<th>W2 Composition and Flowrate</th>
<th>Drop Rate, shells/s</th>
<th>Inner Needle, mm</th>
<th>Outer Needle, mm</th>
<th>Filtered Solutions</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>8.0 wt% PS/ FB, 20 cc/h</td>
<td>DI, 20 cc/h</td>
<td>0.3 wt% PVA, 2400 cc/h</td>
<td>1</td>
<td>0.2 ID</td>
<td>0.51 OD</td>
<td>no</td>
<td>Shakedown Run</td>
</tr>
<tr>
<td>2</td>
<td>8.0 wt% PS/ FB, 20-40 cc/h</td>
<td>DI, 20-40 cc/h</td>
<td>0.3 wt% PVA, 2400-3600 cc/h</td>
<td>1 to 6</td>
<td>0.2 ID</td>
<td>0.51 OD</td>
<td>no</td>
<td>First Run</td>
</tr>
<tr>
<td>3</td>
<td>18 wt% PS/ FB, 14 cc/h</td>
<td>DI, 40 cc/h</td>
<td>0.05 wt% PAA, 3750 cc/h</td>
<td>1.7</td>
<td>0.2 ID</td>
<td>0.51 OD</td>
<td>no</td>
<td>Second Run</td>
</tr>
<tr>
<td>4</td>
<td>18 wt% PS/ FB, 17 cc/h</td>
<td>DI, 48 cc/h</td>
<td>0.05 wt% PAA, 2400 cc/h</td>
<td>1</td>
<td>0.5 ID</td>
<td>0.8 OD</td>
<td>yes</td>
<td>Larger shells with filtered solutions</td>
</tr>
<tr>
<td>5</td>
<td>18 wt% PS/ FB, 17 cc/h</td>
<td>DI, 48 cc/h</td>
<td>0.05 wt% PAA, 2400 cc/h</td>
<td>1</td>
<td>0.5 ID</td>
<td>0.8 OD</td>
<td>yes</td>
<td>GAC and DI water wash</td>
</tr>
</tbody>
</table>

### Results

<table>
<thead>
<tr>
<th>Specifications</th>
<th>Run 3</th>
<th>Run 4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Estimated Diameter</td>
<td>2,300 micron</td>
<td>3,003 micron</td>
</tr>
<tr>
<td>Estimated Wall Thickness</td>
<td>24 micron</td>
<td>30 micron</td>
</tr>
<tr>
<td>Diameter +/- 5%</td>
<td>2,387 micron, StDev 24</td>
<td>3,013 micron, StDev 8.2</td>
</tr>
<tr>
<td>Wall +/- 10%</td>
<td>23 micron, StDev 0.9</td>
<td>32 micron, StDev 1.3</td>
</tr>
<tr>
<td>OOR, &lt; 1.0% of radius</td>
<td>0.44%</td>
<td>0.36%</td>
</tr>
<tr>
<td>NC, &lt;1% to 5%</td>
<td>NA</td>
<td>1.4 +/- 1.6 %</td>
</tr>
<tr>
<td>Number of shells</td>
<td>~ 2,000</td>
<td>~1,000</td>
</tr>
</tbody>
</table>

- Run 5 to be characterized
Potential new method for curing, extractions, and nucleation

• Gas Agitated Contactor to possibly replace rotary contactor
• More suitable for mass production; eminently scaleable without repeating parametric studies; adaptable to continuous process
• Functional Attributes
  • Bubbles agitate targets and remove fluorobenzene, (FB)
  • Controlled FB to air concentration to optimize curing time
• Status: Design complete; initial run complete
Gas Agitated Contactor Provides Uniform Mixing

Gas Agitated Contactor initial run: 3 mm PS targets in 0.05 wt% PAA solution
Gas Agitated Contactor Requires Foam Minimization

• Three washing solutions tested for foaming
  0.05 wt% PAA: Foaming inconsequential
  0.3 wt% PVA: Foaming moderate, drops of IPA broke apart bubbles
  3.0 wt% PVA: Foaming excessive, IPA drops did not alleviate

• Therefore, desire to identify a new washing solution with dual roles:
  • Ability to wash PAA off targets without foaming
  • Can be utilized in a continuous process without excessive cost
  • Several other washing solutions have been suggested:
    DI Water
    Isopropanol
    Ethylene Glycol
    Sodium Hydroxide
    Acetic Acid
Fluorobenzene/polystyrene interacts with Teflon frit

- Fluorobenzene wets Teflon at a < 90 degree contact angle

- Altering frit material of construction to aluminum

FB in 0.05 wt% PAA, contact angle < 90

PS target material stuck to Teflon frit

FB in 0.05 wt% PAA contact angle ~180

Teflon strip

Aluminum strip
Microencapsulation work near term

- Redesign of droplet generator
  - 5 mm wet target diameters
  - Redesign glass needle as mass production will require greater reproducibility
- Flowrates for larger targets; Double the flowrate, double the targets?
- Examine alternative wash solutions
- Heating of targets to decrease drying time
- Explore solvent removal rates to optimize cure time
  - Flame ionization detector to characterize FB concentration in purge stream

![Graph showing conceptual optimization of curing time]

Maximum Removal Rate

Time

Conceptual optimization of curing time
ICF characterization techniques/equipment are insufficient for IFE targets

- Desired characterizations may determine which system is best for measurements
  - Inferometry: Stage size, optical focus
  - New automated optical system: layered targets, thick target walls
More scale-up development is needed

- Examine methods for earlier characterization of shells
  - FID, process control
  - Optical, line scan cameras for target
- Explore pulsing as an addition to droplet generator
  - Increase droplet generation to >200 Hz; Reduce debris in process stream
- Explore other methods for reducing drying time
  - Nucleation (in 100 % ethanol), Forced convection

\[
Time = \frac{1}{6} \left[ \frac{L^2}{D_{AB}} \right]
\]

2 mm PS target, ~1,000 frames per second
Microencapsulation Studies Recap

Examine scale-up opportunities utilizing chemical engineering principals and apparatus; study methods to reduce costs associate with mass production

- Gas Agitated Contactor design and implementation
- Study of alternative washing solutions including DI water
- Further literature review and theoretical work

Produce 4.6 mm/250 micron wall polystyrene targets

- Experiments are approaching larger diameters, already formed 3 mm targets, limit of current droplet generator design
- Work still needed to determine process flowrates for larger targets and greater production rates

Produce a stock of targets for additional coating studies

- Targets are being produced now which can be utilized for further studies