Semi-Continuous Manufacturing of Personal Care Liquids

Prepared for the
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October 20-24, 2013
Castelldelfei, Spain
ABSTRACT

In many personal care product liquids applications, batch manufacturing has been replaced by continuous or semi-continuous processes. When moving from batch to continuous processing, coordination between formulation development, process development, supply chain and manufacturing quality control is mandatory. R&D commitment to support the semi-continuous platform is a prerequisite for wide spread adoption and long term progression.

Unilever has adopted and adapted semi-continuous processing for its personal care liquids manufacturing. With multi channel systems from 4 liters/minute to 400 liters/ min., we have installed, commissioned and validated units in 10 countries. A review of scale-up practices, machine design (including hygienic considerations) and practical considerations are outlined in the presentation.

ABOUT THE AUTHOR

Peter A. Divone, Sr., P.E., is Unilever’s Global Skin Care and Cleansing Process Development Director. With over 30 years of experience across several consumer goods companies, Peter has worked in R&D Process Development for most of his career.

Peter has pioneered Unilever’s semi-continuous manufacturing platform for skin care and cleansing liquids (creams, lotions and personal wash liquids). The technology has been globally transferred at the pilot and plant levels. Peter is based in Unilever’s R&D center in Trumbull, Connecticut, USA.
Agenda

- Introduction: Overview of Technology
- Batch vs. Continuous: Benefits
- R&D Integration & Scale-up
- Plant Design
- Summary
Introduction: Overview of Technology

- Flow Diagram
- Details of in-line mixer
Semi-Continuous Processing of Personal Care Liquids

Typical 3 stream Skin Lotion schematic
High Pressure Cavitation Mixer – Principle of Operation: Provides Instantaneous Emulsification

Details of In-line Mixer

- **Water Ingredient**
  - Premix

- **Oil Ingredients**
  - Premix

- **Minor**
  - Premix

- **Orifice**

Pressure: 13-22 bars

PRODUCTION: 180 to 270 kg/min
Benefits: Semi-Continuous vs. Batch Mixing

- Cycle Time Comparison
- Cost Comparison
- Experience to Date
Cycle Time Comparison

- Phase preparation – 35 to 50 minutes
- Emulsification – 0 (Instant)
- Cooling – 0
- Final mix – 0
- Discharge – 30 to 40 minutes
- Total cycle time – 65 to 90 minutes (within 1.5 hours)

- Phase preparation – 35 to 50 minutes
- Emulsification – 30 to 60 minutes
- Cooling – 60 to 120 minutes
- Final mix – 15 to minutes
- Discharge – 30 to 40 minutes
- Total cycle time - 170 to 290 minutes (3 to 5 hours)

Typical HBL semi-continuous vs and batch process (based on 6.5 ton run)
Benefits : Semi-Continuous vs Batch

Cost Comparison

BATCH Process
- Capacity – 45 kgs/min
- Capital - $1.2M (0.8M EU)

SEMI-CONTINUOUS Process
- Capacity - (270 kgs/min)
- Capital - $1.9M (1.3M EU)

BATCH REQUIRES ~ 4X THE INVESTMENT TO DELIVER THE SAME CAPACITY AS SEMI-CONTINUOUS MIXING
Experience to date:

- 70% Capacity increase
  - Allows the use of concentrated premixes for further capacity increase (3X for HBLs and up to 10X for shampoos)

- Less energy / Kg consumption

- Reduced waste - both effluent and product

- Quality control
  - Tighter droplet size distribution
  - Better run to run consistency

- Innovation enabler – tailored product characteristics
  - Controlled droplet size distribution

- Smaller Footprint

- Easier to Clean and Changeover
R&D Integration and Scale-Up

- Formulation Breakdown
- Pilot Plant Data Acquisition and Measurement Tools
- Understanding Hydrodynamic Cavitation
- Pilot Plant Models
Formula Development: O/W Skin Cream Emulsion

Oil Phase

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<th>Material</th>
<th>%</th>
<th>Kgs</th>
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<td>Pristerene</td>
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Total: 4000 kgs

Temperature 73°C

Bomb No.2

% 12.1910

Water Phase

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Total: 4000 kgs

Temperature 32°C

Bomb No.1

% 87.8090

Specifications @ 25°C

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<tr>
<td>Color</td>
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<tr>
<td>Odor</td>
<td>STD</td>
</tr>
<tr>
<td>Appearance</td>
<td>STD</td>
</tr>
<tr>
<td>pH</td>
<td>5.85 - 6.05</td>
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<tr>
<td>Visc. Initial 34°C</td>
<td>6000 - 12000</td>
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<tr>
<td>Overnight 25°C</td>
<td>16000 - 22000</td>
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<tr>
<td>Espec. Grav.</td>
<td>0.990 - 1.025</td>
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Premix before addition to Water Phase

87.8090 3512.3600

SCALE UP CONDITIONS DOVE LOTIONS

<table>
<thead>
<tr>
<th>Phase</th>
<th>Temperature</th>
<th>Pressure</th>
<th>Flow Rate</th>
<th>Orifice</th>
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<tbody>
<tr>
<td>Oil Phase</td>
<td>73°C</td>
<td>180 PSI  / 12 BAR</td>
<td>140 KG/MIN</td>
<td>0.096 IN²</td>
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<tr>
<td>Water Phase</td>
<td>32°C</td>
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</table>
Pilot Plant DAQ & Measurement Tools

Dove Nourishing pilot trials

WP Temp = 105F, P = 250 psi

Shear Rate (1/s)

Stress (Pa)

orifice = 0.0037 in²

orifice = 0.0059 in²
Hydrodynamic flow equation: $Q = kA(P)^{0.5}$

<table>
<thead>
<tr>
<th>SI Units</th>
<th>US Units</th>
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<tbody>
<tr>
<td>$Q =$ Throughput water like fluid (L/min)</td>
<td>$Q =$ Throughput water like fluid (gal/min)</td>
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<tr>
<td>$k =$ SI Orifice Coefficient (67)</td>
<td>$k =$ US Orifice Coefficient (30)</td>
</tr>
<tr>
<td>$A =$ orifice area (cm$^2$)</td>
<td>$A =$ orifice area (in$^2$)</td>
</tr>
<tr>
<td>$P =$ Pump-to-orifice Pressure (bar)</td>
<td>$P =$ Pump-to-orifice Pressure (PSI)</td>
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<tr>
<td>$Q^* =$ Throughput water like fluid SG=1 (kgs/min)</td>
<td>$Q^* =$ Throughput water like fluid SG=1 (lbs/min)</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>$k$</th>
<th>67</th>
<th>SI Coefficient</th>
<th>30</th>
<th>US Coefficient</th>
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<tr>
<td>$A$</td>
<td>0.0381 cm$^2$</td>
<td>0.0059 in$^2$</td>
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<tr>
<td>$P$</td>
<td>19.65 bar</td>
<td>285.00 PSI</td>
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<tr>
<td>$Q$</td>
<td>11.31 L/min</td>
<td>2.99 gal/min</td>
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<tr>
<td>$Q^*$</td>
<td>11.31 kgs/min</td>
<td>24.94 lbs/min</td>
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</table>

1 in = 2.54 cm
1 bar = 14.5 psi
1 gal = 3.785 L
1 gal = 8.345 lbs (SG=1)
1 kg = 1.000 kgs (SG=1)
1 kg = 2.205 lbs
Pilot Plant Models
Plant Design

- Controls & Software
- Layout Considerations
- Maintenance and Repair
- Hygienic Valves and CIP
Semi-Continuous kit location

- locate pumps within close proximity to pre-mix tanks
- minimize length of oil line (Lotion/Cream)
Plant Design: Layout Considerations

- Location with Adequate Space
  - adequate room to work on wet end of pumps
Progressive Cavity Metering pumps
- replacement parts include rotors, stators, seals, boots
- stators represent largest expense
- stators made from Viton have provided best performance
- average annual cost (3 pumps) = $9,000 USD
Plant Design: Valves & CIP

- Check-valves on all streams leading into mixing chamber
- CIP around in-line mixer
- Pressure relief valves (Bardiani or Tuchenhagen Q) or rupture discs
14 SEMI-CONTINUOUS SYSTEMS GENERATE 30% OF ALL THE PCL TONNAGE PRODUCED BY UNILEVER, OVER 200 BATCH PLATFORMS PRODUCE THE REST OF THE VOLUME

By 2016, we expect to move this to 75% of all Personal Care Liquids Production.

Semi-continuous systems are now used to make almost all PCL products including Hand & Body Lotions, Face Creams, Shampoos, Conditioners, Personal Wash Liquids

Semi-continuous systems are now located in 10 R&D pilot plants around the world.
It takes Dedicated Teamwork and Focus to move from batch to continuous processing! Thank you