Presentation Overview

- Introduction to Innospec
- Structured Liquid Systems (SLS)
- Advantages of using SLS in Personal Care
- Current Market Technology and Products
- How to produce a SLS
- Determination and Characterisation
- Example Formulation
Innospec – At a Glance

- Focused on Specialty Chemicals
- 1000 employees
- 23 countries
- NASDAQ listed
- UK HQ in Ellesmere Port
- Turnover 960$m in 2014
Innospec consists of three market sectors:

- Performance Chemicals
- Fuel Specialties
- Octane Additives

Innospec Specialty Chemicals
Performance Chemicals’ markets

- Personal Care
- HI & I
- Plastics and Polymers
- Fragrances
Introduction to structured liquid systems

- Systems where the surfactants are in liquid crystalline phases, particularly of interest lamellar phase
- Typically formed at high surfactant concentrations
- They are used in a variety of applications
  - Personal care
  - Liquid laundry detergents
  - Pharmaceutical

Schematic of Lamellar Phase
Surfactant Phases – packing diagram
Advantages of SLS Formulations

- Allows the suspension of particles (e.g. beads)
- Allows high levels of oil to be incorporated
- Improved deposition of oils and fragrances on to hair and skin compared to micellar systems\(^1,2,3\)
- Stable under high temperature conditions
- Incorporation of usually incompatible materials e.g. UV filters
- Easy pumping and delivery from packaging

1. D. Bendejaque, Cosmetics and toiletries, Vol 125, No: 11, p22-29
2. C. Mabille, Cosmetics and toiletries, Vol 128, No :1, p34-41
3. US2007155638A1 (Figure 1)
# Incorporated Oil levels in SLS Systems

<table>
<thead>
<tr>
<th>Cosmetic Oil</th>
<th>Formulation typical values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dimethicone</td>
<td>1-5</td>
</tr>
<tr>
<td>Dimethiconol</td>
<td>1-5</td>
</tr>
<tr>
<td>Rapeseed Oil</td>
<td>5-50</td>
</tr>
<tr>
<td>Sunflower Oil</td>
<td>5-50</td>
</tr>
<tr>
<td>C12-C15 Alkylbenzoate</td>
<td>5-15</td>
</tr>
<tr>
<td>Dipropylene glycol dibenzoate</td>
<td>5-15</td>
</tr>
<tr>
<td>Argan Oil</td>
<td>1-5</td>
</tr>
<tr>
<td>Avocado Oil</td>
<td>1-5</td>
</tr>
<tr>
<td>Coconut Oil</td>
<td>5-50</td>
</tr>
<tr>
<td>Olive Oil</td>
<td>5-50</td>
</tr>
</tbody>
</table>
Key Personal Care Applications

► Shower gels
  • Enhanced (24 hour) moisturization (high oil levels with foaming)
  • Visual effects are desired e.g. layers
  • Incompatible ingredients can be an issue

► Skin cleanser
  • Suspend objects for either visual or performance (beads)
  • Incompatible ingredients can be an issue

► Shampoos
  • Improved deposition of conditioners on the hair
  • Good foaming properties
  • Incompatible ingredients can be an issue
  • Visual effects are desired
Example products on the market

Aqua, Sodium Lauryl Methyl Isethionate, Sodium Lauroamphoacetate, Cocamide MIPA, Oryza Sativa (Rice) Bran Oil, Glycerin, Dimethicone, Xanthan Gum, Cellulose Gum, Guar Hydroxypropyltrimonium Chloride, Citric Acid, Butyrospermum Parkii Butter Extract (Shea Butter), Carica Papaya Seed Oil, Parfum (Fragrance), Citrus Sinensis Peel Oil Expressed, Laureth-8, Succinoglycan, Methylchloroisothiazolinone, Methylisothiazolinone, Sodium Benzoate

Aqua, Glycerin, Helianthus Annuus Seed Oil, Hydrogenated Soybean Oil, Cocamidopropyl Betaine, Sodium Hydroxypropyl Starch Phosphate, Sodium Laureth Sulfate, Sodium Cocoyl Glycinate, Lauric Acid, Parfum, Sodium Lauryl Isethionate, Sodium chloride, Stearic Acid, Guar Hydroxypropyltrimonium Chloride, Citric acid, Sodium Palmitate, BHT, Sodium Isethionate, Sodium Stearate, Tetrasodium EDTA, Sodium Palm Kernelate, Zinc Oxide, Tetrasodium Etidronate, Alumina, DMDM Hydantoin, Sodium benzoate, Benzyl alcohol, Butylphenyl Methylpropional, Citronellol, Hexyl Cinnamal, Limonene, Linalool, CI 77891
Water, Petrolatum, Mineral Oil, Sodium Trideceth Sulfate, Sodium Lauryl Sulfate, Sodium Lauroamphoacetate, Sodium Chloride, Trideceth-3, Fragrance, Prunus Amygdalus Dulcis (Sweet Almond) Oil, Citric Acid, Guar Hydroxypropyltrimonium Chloride, Acrylonitrile/Methacrylonitrile/ Methyl Methacrylate Copolymer, Isopentane, Xanthan Gum, Sodium Benzoate, PEG-90M, Disodium EDTA, Methylchloroisothiazolinone, Methylisothiazolinone, Sodium Hydroxide, Red 7

Aqua (Water), Helianthus annuus (Sunflower) seed oil, Sodium trideceth sulphate, Butyrospermum parkii (Shea butter), Glycerin, Sodium chloride, Sodium lauroamphoacetate, Cocamide MEA, Parfum (Fragrance), Tocopheryl acetate, Guar gum, Guar hydroxypropyltrimonium chloride, Citric acid, Benzyl salicylate, Linalool, Benzyl benzoate, Hexyl cinnamal, Limonene, Disodium EDTA, Magnesium nitrate, Methylisothiazolinone, Methylchloroisothiazolinone, Magnesium chloride
Formulating Structured Surfactant Systems

- Determine the phase properties/diagram of surfactant system.
- Surfactant system needs to be in lamellar phase
- Lamellar phase when sheared can deform to form multi lamellar vesicles/spherulites
- Some surfactant systems which can form spherulites:
  - Sodium tetradecyl sulfate/Sodium lauroamphoacetate/cocamide MEA
  - Sodium lauroyl methyl isethionate/sodium lauroamphoacetate/cocamide MEA
  - Sodium bis (2-ethylhexylsulfosuccinate) /SDS/Salt
  - Poly(oxyethylene alcohol)/water
  - Didecylpyrroolidium bromide
Typical Phase Diagram

- M-Phase - rods
- 2 Phases
- Anionic surfactant
- Lamellar Phase
- Micellar Phase
- Electrolyte (salt)
- Amphoteric Surfactant
Surfactant Phase Transition In SLS Formulations

- **Micellar state**
- **M-phase, gel**
- **Sheer thinning Lamellar state**

Transition to structured state with mixing

- **Dilution upon use in shower**
- **Foaming**
- **Cleansing**
- **Deposition**

Structured system

Spherulite

Micellar state
Structured surfactant formulation

- Insoluble Or Incompatible Materials
How do I know I have a SLS?

► Rheology
  • High zero shear viscosity/yield point - shear thinning

► Polarised Light Microscopy
  • Lamellar phases are bierfrigent and give characteristic patterns when viewed using polarised light.

► SAXS
  • Lamellar phases can be determined by characteristic d line spacing and broad peak at 4.5Å.

► Electron Microscopy
  • Images show round spheres of spherulites in cryo SEM or layers in cryo TEM
Rheology

Rheology of lamellar phase and spherulites can be studied using rheology.

Oscillatory methods
- linear visco-elastic region
  - Poking a Jelly

Flow methods
- Equilibrium shear rate ramp
- Linear region at low shear rate (Zero Shear viscosity)
Polarised Light Microscopy

- Lamellar phase surfactant systems are bierfringent materials
- Refractive index dependant on polarisation of light
- Defects in lamellar phase cause characteristic Maltese cross patterns
SAXS Analysis

- Definitive characterisation technique
- Determination of d spacing distance between layers
- Expensive and limited research institutions where it can be done
Electron Microscopy

Electron Microscopy one of the most definitive characterisation techniques

Cryo SEM with high resolution see spherical structures

Cryo TEM high resolution see layers in 2D structure

Expensive and equipment availability
Outsourced either in academia or test institutes
Classical Formulation Procedure

- Water phase, conditioners, chelants
- Rheology modifiers
- Oil phase
- Amphoteric surfactant
- Anionic and non-ionic surfactants
- Fragrance
- Form lamellar/spherilitic phase (salt or pH adjust)
Structured Liquid System Concentrates

- Ease of formulation (lamellar phase already formed)
- SLS surfactant concentrates developed:
  - Based on sulfates
    - Sodium trideceth sulfate, Sodium lauroamphoacetate, cocamide MEA
  - Based on sulfate-free technology (Innospec technology)
    - Sodium lauroyl methyl isethionate, Sodium lauroamphoacetates, Cocamide MEA
Benefits of Sodium Lauroyl Methyl Isethionate vs Sodium Trideceth sulfate in personal care

<table>
<thead>
<tr>
<th>Property</th>
<th>Sodium Lauroyl Methyl Isethionate*</th>
<th>Sodium Trideceth Sulphate</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sulfate Free</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>1,4 Dioxane Free</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Ethylene Oxide Free</td>
<td>Yes</td>
<td>No</td>
</tr>
<tr>
<td>Biodegradability</td>
<td>Readily</td>
<td>Inherently</td>
</tr>
<tr>
<td>Hydrophobe Origin</td>
<td>Natural</td>
<td>PetroChemical</td>
</tr>
<tr>
<td>Foam Type</td>
<td>Tight/Small</td>
<td>Larger/ More Open</td>
</tr>
<tr>
<td>Mildness</td>
<td>Mild As Is</td>
<td>Mildness Must Be &quot;Created&quot;</td>
</tr>
</tbody>
</table>

*Sodium Lauroyl Methyl Isethionate is available from Innospec under the tradename Iselux®*
# Innospec Formulation – Make up Remover

<table>
<thead>
<tr>
<th>INCI Ingredient</th>
<th>% w/w Active</th>
</tr>
</thead>
<tbody>
<tr>
<td>A) Aqua</td>
<td>q.s to 100</td>
</tr>
<tr>
<td>Sodium Chloride</td>
<td>4.00</td>
</tr>
<tr>
<td>Citric Acid</td>
<td>Trace</td>
</tr>
<tr>
<td>B) Guar Hydroxypropyltrimonium Chloride</td>
<td>0.20</td>
</tr>
<tr>
<td>C) Disodium Cocoamphoacetate</td>
<td>2.85</td>
</tr>
<tr>
<td>Sodium Methyl Cocoyl Taurate</td>
<td>2.3</td>
</tr>
<tr>
<td>D) Cyamopsis Tetragonoloba Guar Gum</td>
<td>0.5</td>
</tr>
<tr>
<td>Glycerine</td>
<td>1.00</td>
</tr>
<tr>
<td>E) Sodium Lauroyl Methyl Isethionate</td>
<td>9.6</td>
</tr>
<tr>
<td>Cocamide MEA</td>
<td>3.50</td>
</tr>
<tr>
<td>Cocamidopropyl Betaine</td>
<td>3.0</td>
</tr>
<tr>
<td>F) Rapeseed Oil</td>
<td>10.00</td>
</tr>
<tr>
<td>C12-C15 Alkyl Benzoate</td>
<td>2.00</td>
</tr>
<tr>
<td>G) Preservative, dye, fragrance</td>
<td>q.S</td>
</tr>
<tr>
<td>H) Citric Acid (50% w/w soln)</td>
<td>q.s to pH 5.0-6.0</td>
</tr>
</tbody>
</table>
Structured Liquid Systems in personal care can give:

- High oil incorporation
- Good foaming properties
- Enhanced deposition of actives, oils and fragrance
- Can be formed with using a range of surfactants
- Improved temperature stability
- Surfactant concentrates can be used to simplify manufacture
Thank you for your attention

www.innospécinc.com