Dow Corning polysiloxanes can be readily formulated to oil-in-water micro or macro/mechanical emulsions. Micro emulsions are so called due to their very small average particle size (typically < 100nm). They are translucent. Macro/mechanical emulsions are white emulsions with typical average particle size 0.1 -> 1 micron.

Successful production of emulsions depends on choosing the correct HLB (Hydrophilic Lipophilic Balance) i.e. matching the HLB of the surfactant mixture with that of the polysiloxane which is being emulsified. The large number of surfactants available makes any trial and error system difficult and time consuming. The easiest way to begin to identify the HLB of a system is to use the ‘HLB method’. This involves preparing emulsions with a range of HLB values.

For micro emulsions for example, one response that can be measured, is particle size. The lower the particle size, the clearer the emulsion appearance.

A typical curve generated by the HLB method for a micro emulsion of 15% amino polysiloxane in water is:

This indicates optimum appearance is achieved at HLB 11.5-> 12.5

This method helps to narrow in on the required HLB value of the system being studied. In general, a mixture of surfactants (of high & low HLB) gives a more stable emulsion because:

(a) the spread of size of the lipophiles and/or hydrophiles allows more efficient packing at the oil/water interface
(b) the interface can be supplied with surfactant from both the oil (more lipophilic surfactant) and water (more hydrophilic surfactant) phases in more equal proportions rather than predominantly from one side, as would be the case with a single surfactant.
(c) a mixture of surfactants gives larger polydispersity to more effectively match the polydispersity of the oil phase.

The HLB method, however, is not predictive of emulsion stability and therefore different surfactant types should now be tested to achieve optimum emulsion stability e.g. linear/branched alcohols.

In general, non-ionic alcohol ethoxylates are employed as surfactants i.e. RO(CH₂CH₂O)ₓH , where R is an alkyl group e.g. C₉H₁₉/C₁₁H₂₃, i-C₁₃H₂₇, x is any positive integer, in order to allow compatibility with ionic auxiliaries (e.g. optical brighteners).

**A. PREPARATION OF OIL-IN-WATER MICRO EMULSIONS (15% AMINO POLYSILOXANE)**

Dow Corning aminofunctional polysiloxanes can be readily micro-emulsified to 15% siloxane minimum.

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dow Corning Amino polysiloxane</td>
<td>15.00</td>
</tr>
<tr>
<td>Surfactant(s)</td>
<td>7.50 - 9.00</td>
</tr>
<tr>
<td>Acetic acid, 40%</td>
<td>0.62</td>
</tr>
<tr>
<td>Water - thick phase - dilution</td>
<td>7.00</td>
</tr>
<tr>
<td>Biocide</td>
<td>as required</td>
</tr>
</tbody>
</table>

**Surfactants**

Non-ionic alcohol ethoxylates are generally used, at levels of 7.5 wt% to 9 wt%.

Trials using surfactants of:
(i) varying types (varying lipophile type), and
(ii) varying HLB
are necessary to identify the most stable formulation.

Micro emulsions of Dow Corning aminofunctional polysiloxanes can be prepared by:

(i) **THICK PHASE METHOD**

This method involves preparation of a viscous mixture of water, surfactant(s) and Dow Corning polysiloxane, which is diluted with water to the desired siloxane level.

1. To prepare the ‘thick phase’, mix amino polysiloxane, surfactants and ‘thick phase’ water until homogeneous.

The remainder of the water is used to dilute this ‘thick phase’.

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2. Add the dilution water in 3 separate and equal amounts, mixing well after each addition. Ensure mixture is homogeneous before proceeding.
3. Add acetic acid, 40%, and mix for 30 minutes.
4. Add selected biocide if required and mix for a further 15 minutes.

\textbf{NOTE:} For best results, it is important that homogeneity is achieved at each stage.

(ii) ‘Non-inversion’ Method
1. Mix surfactants and total water.
2. Add polysiloxane over a period of 1.5 to 2 hours, mixing at 300 to 400 rpm throughout addition, ensuring total incorporation of siloxane i.e. no free suspension.
3. Resulting mixture is white.
4. Add glacial acetic acid, 40%, and mix for 30 minutes. Mixture will turn clear.
5. Add selected biocide if required and mix for a further 15 minutes.

\textbf{NOTE:} Accurate identification of HLB is critical to achieve best results using this method.

B. Preparation of Macro/mechanical Emulsions of Dow Corning Polysiloxanes

Generally, macro emulsions containing 35% by weight of Dow Corning polysiloxanes can be prepared using the thick phase method of emulsification.

\textbf{Equipment:}
Production of macro emulsions requires a high shear device (e.g. colloid mill, sonolator, homogeniser) to reduce the particle size to that required for optimum emulsion stability i.e. average particle size less than 1 micron. Accurate HLB determination is also required to achieve optimum stability.

\textbf{Surfactants:}
Non-ionic alcohol ethoxylates are typically used.

Macro/mechanical emulsions of Dow Corning polysiloxanes can be prepared by:

(i) Using a Colloid Mill/Sonolator

\textbf{Typical formulation:}

<table>
<thead>
<tr>
<th>Material</th>
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</tr>
</thead>
<tbody>
<tr>
<td>Dow Corning Polysiloxane</td>
<td>35.00</td>
</tr>
<tr>
<td>Surfactant(s)</td>
<td>3 - 3.50</td>
</tr>
<tr>
<td>Acetic acid, 40%</td>
<td>1.45</td>
</tr>
<tr>
<td>Water - pre-shear stage - dilution</td>
<td>18.9 - 23.3 balance</td>
</tr>
<tr>
<td>Biocide</td>
<td>as required</td>
</tr>
</tbody>
</table>

The thick phase comprises water and Dow Corning polysiloxane in proportions to achieve a mixture containing 80 to 82.5% siloxane in water, and surfactant(s).
1. Mix surfactant(s) and water.
2. Add polysiloxane over approximately 90 minutes, mixing continuously at 200 to 300 rpm.
3. Continue to mix the resulting ‘thick phase’ at 200 to 300rpm for 1.5 to 2 hours. Average particle size now is in the range of 20 to 40 microns.
4. Pass the mixture through (i) a rotor/stator device (e.g. colloid mill) using the the minimum gap size, or (ii) a high pressure sonolator at 80 to 100 bar.

This shearing action reduces the average particle size of the thick phase to less than 1 micron.
5. Dilute this sheared mixture with water to 35% siloxane. The dilution water is added in 3 separate and equal amounts, mixing well after each addition.
6. Add acetic acid, 40%, and mix for a further 30 minutes.
7. Add selected biocide if required and mix for a further 15 minutes.

(ii) Using an Homogeniser

\textbf{Typical formulation:}

<table>
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</tr>
<tr>
<td>Biocide</td>
<td>as required</td>
</tr>
</tbody>
</table>

1. Mix Dow Corning polysiloxane with surfactant(s) and water to achieve a mixture containing 60 to 65% siloxane.
2. Pass this mixture through the homogeniser at maximum pressure (800bar). Several passes through the homogeniser may be necessary before achieving the required average particle size (less than 1 micron).
3. Dilute this sheared mixture with water to 35% siloxane. The dilution water is added in 3 separate and equal amounts, mixing well after each addition.
4. Add acetic acid, 40%, and mix for a further 30 minutes.
5. Add selected biocide if required and mix for a further 15 minutes.

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