Dr. Z Presents

Three Dimensional HLB

by

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Three Dimensional HLB

**Background**

Despite the fact that the basic HLB system has been around for about fifty years, the ability to predict the performance of specific emulsifiers in the preparation of emulsions remains a challenge to the formulator. The HLB system works best in predicting emulsification for alcohol ethoxylate that is surfactants based upon fatty alcohols that have been modified by reaction with ethylene oxide. The system is also most useful for emulsions in which water is a component.

Today there are a series of silicone-based surfactants, which contain no hydrocarbon-based hydrophobes, and silicone containing surfactants, which also contain hydrocarbon groups. It would be highly desirable to expand the HLB concept to these new surfactants and to apply it to anhydrous systems.

**Traditional Non-ionic Surfactants**

The selection of a surface-active agent for a specific emulsification application has been made simpler and more systematic by the development of the HLB System. The system was proposed by Griffin1 and has been widely promoted by ICI2, and over the years has proven to be a very valuable aide to the formulator. The basics of the system are as follows:

**HLB SYSTEM**

HLB, the so-called Hydrophile - Lipophile Balance, is the ratio of oil soluble and water-soluble portions of a molecule. The system was originally developed for
ethoxylated products. Listed in Table 1 are some approximations for the HLB value for surfactants as a function of their solubility in water. Values are assigned based upon that table to form a one-dimensional scale, ranging from 0 to 20.

We are using the generic term "hydrocarbon" to designate the oil soluble portion of the molecule. This generic term includes the more specific terms fatty, lipid, and alkyl.

<table>
<thead>
<tr>
<th>Solubility in Water</th>
<th>Table 1</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>HLB Value</td>
</tr>
<tr>
<td>---------------------------------------</td>
<td>-----------</td>
</tr>
<tr>
<td>Insoluble</td>
<td>4 - 5</td>
</tr>
<tr>
<td>Poorly dispersible (milky appearance)</td>
<td>6 - 9</td>
</tr>
<tr>
<td>Translucent to clear</td>
<td>10 - 12</td>
</tr>
<tr>
<td>Very soluble</td>
<td>13 - 18</td>
</tr>
</tbody>
</table>

There are two basic types of emulsions envisioned by the current HLB system. They are the oil in water (O/W) and the water in oil (W/O). The phase listed first is the discontinuous phase. That is it is the phase that is emulsified into the other. Bancroft\(^3\) postulated that upon mixing of the two phases with a surfactant present, the emulsifier forms a third phase as a film at the interface between the two phases being mixed together. He also predicted that the phase in which the emulsifier is most soluble will become the continuous phase. The continuous phase need not be the predominant quantity of material present. There are emulsions where the discontinuous phase makes up a greater weight percent than the continuous phase. A simple test is if the emulsion is readily diluted with water, water is the continuous phase.
It has been stated "The HLB system has made it possible to organize a great deal of rather messy information and to plan fairly efficient systematic approaches to optimize emulsion preparation. If one pursues the concept too far however the system tends to lose itself to complexities." We agree with this and believe that a system which provides direction in the selection of an emulsifier is the first objective. A mathematical model has been developed to allow for approximations of HLB.

**CALCULATION OF HLB**

The HLB system, in its most basic form, allows for the calculation of HLB using the following formulation:

\[
\text{HLB} = \frac{\% \text{ Hydrophilic by weight of molecule}}{5}
\]

Example: Oleyl alcohol 5 E.O.

M.W. Hydrophilic (5) (44) = 220

\[
\frac{220}{\text{Total M.W of molecule}} = 45.0\% 
\]

\[
\text{HLB} = \frac{45\%}{5} = 9.0 \quad \text{HLB} = 9.0
\]

**APPLICATION OF HLB**

One can predict the approximate HLB needed to emulsify a given material and make more intelligent estimates of which surfactant or combinations of surfactants are appropriate to a given application. When blends are used the HLB can be estimated by using a weighted average of the surfactants used in the blend.
### HLB NEEDED TO EMULSIFY

<table>
<thead>
<tr>
<th>Material</th>
<th>HLB</th>
<th>Material</th>
<th>HLB</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetophenone</td>
<td>14</td>
<td>Lanolin</td>
<td>12</td>
</tr>
<tr>
<td>Acid, Lauric</td>
<td>16</td>
<td>Lauryl amine</td>
<td>12</td>
</tr>
<tr>
<td>Acid, Oleic</td>
<td>17</td>
<td>Mineral spirits</td>
<td>10</td>
</tr>
<tr>
<td>Beeswax</td>
<td>9</td>
<td>Nonylphenol</td>
<td>14</td>
</tr>
<tr>
<td>Benzene</td>
<td>15</td>
<td>Orthodichlorobenzene</td>
<td>13</td>
</tr>
<tr>
<td>Butyl Stearate</td>
<td>11</td>
<td>Pine oil</td>
<td>16</td>
</tr>
<tr>
<td>Carbon Tetrachloride</td>
<td>16</td>
<td>Toluene</td>
<td>15</td>
</tr>
<tr>
<td>Castor oil</td>
<td>14</td>
<td>Xylene</td>
<td>14</td>
</tr>
<tr>
<td>Chlorobenzene</td>
<td>13</td>
<td>Kerosene</td>
<td>14</td>
</tr>
<tr>
<td>Cottonseed oil</td>
<td>6</td>
<td>Cyclohexane</td>
<td>15</td>
</tr>
<tr>
<td>Petrolatum</td>
<td>7</td>
<td>Chloronated paraffin</td>
<td>8</td>
</tr>
</tbody>
</table>

For those materials that are not listed above, it is recommended that the oil be tested using specific blends of known emulsifiers. This allows the formulator to calculate the HLB needed to emulsify the non-listed oil.

The appearance of the emulsion is dependant upon the particle size of the discontinuous phase. Particle size is listed in nanometers.

<table>
<thead>
<tr>
<th>Particle size</th>
<th>Appearance</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size</td>
<td>Appearance</td>
</tr>
<tr>
<td>--------------</td>
<td>------------</td>
</tr>
<tr>
<td>&gt; 1</td>
<td>White</td>
</tr>
</tbody>
</table>
Silicone / Polyoxyethylene Based Surfactants

In recent years, there has been a greater acceptance of silicone surfactants in the preparation of emulsions. The direct application of the HLB concept to these materials has resulted in an approximate value. Many manufacturers of silicone surfactants, rather than dealing with the differences between calculated and observed HLB, have dropped specific values and adopted the use of high, middle or low as a classification of HLB values. This approach simply begs the issue.

Silicone Surfactant / Fatty polyoxyethylene Surfactant Blends

Most formulations, which use silicone based surface-active agents, also have traditional hydrocarbon surfactants present. The presence of these blended systems has offered challenges for the predictability of emulsification properties. A recent paper on dealing with the difficulties in predicting behavior of silicone based surfactants when used in combination with hydrocarbon surfactants came to the conclusion that even when low molecular weight silicone compounds are evaluated there is "varying non-ideal behavior" depending upon the type and concentration of the surfactants used. This type of conclusion, while supported by the data, is not helpful to the formulator.

Silicone / Fatty / Polyoxyethylene Based Surfactants
To further complicate the situation there has been a virtual explosion of new silicone compounds on the market which combine polyoxyethylene (water soluble), silicone (silicone soluble) and hydrocarbon (oil soluble) portions into one molecule. The introduction of these types of molecules and the inability to fit them into the HLB concept, has resulted in confusion on how to use the compounds. Table 1 outlines the types of compounds available.

Table 1

**COMPARISON OF FATTY AND SILICONE DERIVATIVES**

<table>
<thead>
<tr>
<th>Hydrocarbon Based</th>
<th>Silicone Based</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>ANIONICS</strong></td>
<td></td>
</tr>
<tr>
<td>Phosphate Esters</td>
<td>Silicone Phosphate Esters$^6$</td>
</tr>
<tr>
<td>Sulfates</td>
<td>Silicone Sulfates $^7$</td>
</tr>
<tr>
<td>Carboxylates</td>
<td>Silicone Carboxylates $^8$</td>
</tr>
<tr>
<td>Sulfo succinates</td>
<td>Silicone Sulfo succinates $^9$</td>
</tr>
<tr>
<td><strong>CATIONICS</strong></td>
<td></td>
</tr>
<tr>
<td>Alkyl Quats</td>
<td>Silicone Alkyl Quats $^{10}$</td>
</tr>
<tr>
<td>Amido Quats</td>
<td>Silicone Amido Quats $^{11}$</td>
</tr>
<tr>
<td></td>
<td></td>
</tr>
<tr>
<td>------------------------</td>
<td>--------------------------</td>
</tr>
<tr>
<td><strong>Imidazoline Quats</strong></td>
<td><strong>Silicone Imidazoline Quats</strong></td>
</tr>
<tr>
<td><strong>AMPHOTERICS</strong></td>
<td></td>
</tr>
<tr>
<td>Amino Propionates</td>
<td>Silicone Amphoteric</td>
</tr>
<tr>
<td>Betaines</td>
<td>Silicone Betaines</td>
</tr>
<tr>
<td>Phosphobetaines</td>
<td>Silicone Phosphobetaines</td>
</tr>
<tr>
<td><strong>NONIONICS</strong></td>
<td></td>
</tr>
<tr>
<td>Alcohol Alkoxylates</td>
<td>Dimethicone Copolyol</td>
</tr>
<tr>
<td>Alkanolamides</td>
<td>Silicone Alkanolamides</td>
</tr>
<tr>
<td>Esters</td>
<td>Silicone Esters</td>
</tr>
<tr>
<td>Taurine Derivatives</td>
<td>Silicone Taurine</td>
</tr>
<tr>
<td>Isethionates</td>
<td>Silicone Isethionates</td>
</tr>
<tr>
<td><strong>COMPARISON OF FATTY AND SILICONE DERIVATIVES</strong></td>
<td></td>
</tr>
<tr>
<td>Hydrocarbon Based</td>
<td>Silicone Based</td>
</tr>
<tr>
<td><strong>FREE RADICAL POLYMERS</strong></td>
<td></td>
</tr>
<tr>
<td>PVP\Quats</td>
<td>Silicone Free Radical Quats</td>
</tr>
<tr>
<td>Polyacrylates</td>
<td>Silicone Polyacrylate</td>
</tr>
<tr>
<td></td>
<td>Copolymers</td>
</tr>
</tbody>
</table>
Three Dimensional HLB ("3D HLB")

The current HLB system has been used successfully for some time in helping to predict how to make water in oil and oil in water emulsions. It has not been less than satisfactory for predicting the performance of silicone based molecules and has been unsuccessful in predicting performance of surfactants having silicone, hydrocarbon and polyoxyalkylene portions present. Any attempt to expand the current HLB system must be able to assign meaningful values to the surfactants, which contain the three components, since each group is insoluble in the other. This mutual insolubility led us to consider an HLB system in which oil, water and silicone were all considered in the determination of the emulsion properties of surfactants. The fact that silicone, oil and water are the three components, which we consider, we have called our system "3 dimensional HLB". As you will see, the mathematics of the system, which evolved need only, handle two numbers, the third, silicone is done by difference.

Our first efforts to build a new HLB model attempted to use a cube, having an x, y and z coordinate. This type of system is difficult from a conceptual point of view and the mathematics are difficult to handle. Fortunately, experimental data solved this problem. As we made the many compounds, some of which are outlined in table 1-4 and evaluated their properties to make emulsions, we found that the system needed was not as complex as we envisioned. Since the original HLB system has proven over time to be very helpful, it was our desire to use it's concept and expand it to include a molecule with silicone present. In fact, we kept the scale used by the current system (0-20), and one of the calculations (% hydrophile /5). One of the lines in our triangle system is the current HLB system.
One of the major discoveries was that in a molecule which has oil, water and silicone present, we need only calculate two values, the third would be done by difference. This is because the calculations are based upon % by weight of oil soluble and % by weight of water-soluble portions of the molecule. If one subtracts the sum of those two values from 100 the % silicone soluble by weight is calculated. The same is observed in the current system, that is the calculation is the % by weight of water-soluble / 5. The calculation of % oil soluble is therefore (100 - % water soluble). We simply make one more calculation. Since our system is based upon the current HLB system, molecules which have no silicone present, can be easily accommodated. They fall on the hypotenuse of the triangle.

Our first attempt to develop a system was to evaluate an equilateral triangle with one of the corners representing oil, one water and the third silicone. While this was appealing in that it gave equal weight to each component, the experimental data did not support the equilateral triangle. The values, which were generated, were best accommodate by a right triangle. The hypotenuse representing the current HLB system, is the longest line. That line connects the oil and the water portions. The hypotenuse is labeled 0-20 like the other sides. However, the distance between units is longer than for the other two lines, which are of equal length. Initially this seemed unsettling. However, as we worked with the system, we discovered that this explains why the current HLB is not directly applicable to silicone-based surfactants. The physical significance of this is the fact that silicone and hydrocarbon compounds are not equally as hydrophobic per % by weight. As we considered this experimental observation, it became clear that this made sense. The right triangle (Figure 1-1) was generated using the experimental data and appears to best predict which molecules will give the indicated emulsions.

The basic triangle is shown:
CONCEPT: Top is old system - drops to be hypotenuse of new system
The line connecting points (0,0) and (0,20) is the silicone/water HLB line (AB). All points falling on this line have no fatty portion. Traditional dimethicone copolyol compounds fall on this line.

The line connecting points (20, 0) and (0, 20) is the traditional HLB line (CB). All points falling on this line have no silicone portion. Traditional surfactants fall on this line. This line represents the standard HLB line.

The last line connecting points (0,0) and (20,0) is the oil/silicone HLB line (AC). This line predicts that emulsifiers to allow for the emulsification of either silicone oil in fatty oil or visa versa is possible. This was a concept, which was not clear to us until the system was developed, and we anxiously looked to make molecules predicted to give these types of emulsions.

It is significant that the triangle used is not equilateral as we originally thought. As one looks at the right triangle that we propose, it is clear that the hypotenuse which represents the standard HLB is longer than the other two sides. This difference in length is not artifact. It helps explain why the predictions of HLB for dimethicone copolyol based upon the standard formula (% hydrophile /5) do not result in numbers, which match the observed values.

Our system addresses this problem.

The calculation of the x and y points within our system is as follows:

<table>
<thead>
<tr>
<th>X coordinate</th>
<th>Y coordinate</th>
</tr>
</thead>
<tbody>
<tr>
<td>% water soluble/5</td>
<td>% oil soluble/5</td>
</tr>
</tbody>
</table>

This calculation gives the two values that describe the point. Essentially the amount of silicone is the difference.

For example:
Examples

<table>
<thead>
<tr>
<th>% Oil Sol.</th>
<th>% Water Sol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>(A) Standard Fatty Surfactant</td>
<td>50.0 %</td>
</tr>
</tbody>
</table>

\[(x) \quad (y)\]

\[
\frac{50}{5} = 10.0 \quad \frac{50}{5} = 10.0
\]

Point is on line (CB).

<table>
<thead>
<tr>
<th>% Oil Sol.</th>
<th>% Water Sol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>(B) Standard Silicone Surfactant</td>
<td>0.0</td>
</tr>
</tbody>
</table>

\[(x) \quad (y)\]

\[
\frac{0}{5} = 0 \quad \frac{50.0}{5} = 10.0
\]

Point is on line (AB)

<table>
<thead>
<tr>
<th>% Oil Sol.</th>
<th>% Water Sol.</th>
</tr>
</thead>
<tbody>
<tr>
<td>(C) Three Dimensional Surfactant</td>
<td>30.0</td>
</tr>
</tbody>
</table>

\[(x) \quad (y)\]

\[
\frac{30.0}{5} = 6.0 \quad \frac{20.0}{5} = 4.0
\]

References

The corners of the graph are defined by the references below.
### Experimental Samples

The predictive value of the above graph was tested, by making a variety of surface-active agents. The predictions were then compared to the actual properties. The performance of the surfactants studied allowed for the definition of the boundaries of the system.

### Compound Tested

<table>
<thead>
<tr>
<th>Compound Tested</th>
<th>% Water Sol.</th>
<th>% Oil Sol.</th>
<th>x, y</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Dimethicone copolyol isostearate &quot;A&quot;</td>
<td>47.5/5</td>
<td>24/5</td>
<td>9.5, 4.8</td>
</tr>
<tr>
<td>2. Dimethiconol stearate</td>
<td>0/5</td>
<td>15/5</td>
<td>0.0, 3.0</td>
</tr>
<tr>
<td>3. Dimethicone copolyol iso-stearate &quot;B&quot;</td>
<td>32/5</td>
<td>20/5</td>
<td>8.0, 4.0</td>
</tr>
<tr>
<td>4. Cetyl Dimethicone</td>
<td>0/5</td>
<td>20/5</td>
<td>0.0, 4.0</td>
</tr>
<tr>
<td>5. Dimethicone copolyol amine</td>
<td>19/5</td>
<td>0/5</td>
<td>3.8, 0.0</td>
</tr>
<tr>
<td>6. Dimethicone copolyol isostearate &quot;C&quot;</td>
<td>55/5</td>
<td>10/5</td>
<td>11.0, 2.0</td>
</tr>
<tr>
<td>7. Dimethicone copolyol isostearate &quot;D&quot;</td>
<td>48/5</td>
<td>16/5</td>
<td>9.6, 3.2</td>
</tr>
<tr>
<td>8. Dimethicone copolyol amine &quot;B&quot;</td>
<td>27.5/5</td>
<td>5/5</td>
<td>5.5, 1.0</td>
</tr>
</tbody>
</table>
### Compound Tested

<table>
<thead>
<tr>
<th>Blends</th>
<th>% Water Sol.</th>
<th>% Oil Sol.</th>
<th>x</th>
<th>y</th>
</tr>
</thead>
<tbody>
<tr>
<td>11. Dimethiconol stearate stearth (2) alcohol</td>
<td>21.5/5</td>
<td>70/5</td>
<td>4.3</td>
<td>14.0</td>
</tr>
<tr>
<td>12. Dimethiconol stearate stearth (2) alcohol</td>
<td>6/5</td>
<td>55/5</td>
<td>1.2</td>
<td>11.0</td>
</tr>
<tr>
<td>13. Dimethiconol stearate stearth (2) alcohol</td>
<td>0/5</td>
<td>79/5</td>
<td>0.0</td>
<td>15.8</td>
</tr>
</tbody>
</table>

### Test Emulsions

The 3D System predicts six types of emulsions they are:

1. Silicone in water (S/W) (Formulation A)
2. Oil in water (O/W) (Formulation B)
3. Water in silicone (W/S) (Formulation C)
4. Water in oil (W/O) (Formulation D)
5. Oil in silicone (O/S) (Formulation E)
6. Silicone in oil (S/O) (Formulation F)
The emulsion systems for each was as follows:

**FORMULATION A**

Silicone in Water

<table>
<thead>
<tr>
<th>Material</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicone oil (350 visc)</td>
<td>15.0</td>
</tr>
<tr>
<td>Test Surfactant</td>
<td>5.0</td>
</tr>
<tr>
<td>Water</td>
<td>80.0</td>
</tr>
</tbody>
</table>

The test surfactant was added to the discontinuous phase under good agitation, for five minutes. The continuous phase was added slowly.

**FORMULATION B**

Oil in Water

<table>
<thead>
<tr>
<th>Material</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral oil</td>
<td>15.0</td>
</tr>
<tr>
<td>Test Surfactant</td>
<td>5.0</td>
</tr>
<tr>
<td>Water</td>
<td>80.0</td>
</tr>
</tbody>
</table>

The test surfactant was added to the discontinuous phase under good agitation, for five minutes. The continuous phase was added slowly.

**FORMULATION C**

Water in Silicone

<table>
<thead>
<tr>
<th>Material</th>
<th>%</th>
</tr>
</thead>
</table>
The test surfactant was added to the discontinuous phase under good agitation, for five minutes. The continuous phase was added slowly.

**FORMULATION D**

**Water in Oil**

<table>
<thead>
<tr>
<th>Material</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Water</td>
<td>15.0</td>
</tr>
<tr>
<td>Test Surfactant</td>
<td>5.0</td>
</tr>
<tr>
<td>Mineral oil</td>
<td>80.0</td>
</tr>
</tbody>
</table>

The test surfactant was added to the discontinuous phase under good agitation, for five minutes. The continuous phase was added slowly.

**FORMULATION E**

**Oil in Silicone**

<table>
<thead>
<tr>
<th>Material</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mineral oil</td>
<td>15.0</td>
</tr>
<tr>
<td>Test Surfactant</td>
<td>5.0</td>
</tr>
</tbody>
</table>
Silicone (350 Visc) 80.0

The test surfactant was added to the discontinuous phase under good agitation, for five minutes. The continuous phase was added slowly.

**FORMULATION F**

Silicone in Oil

<table>
<thead>
<tr>
<th>Material</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Silicone oil (350 visc)</td>
<td>15.0</td>
</tr>
<tr>
<td>Test Surfactant</td>
<td>5.0</td>
</tr>
<tr>
<td>Mineral oil</td>
<td>80.0</td>
</tr>
</tbody>
</table>

The test surfactant was added to the discontinuous phase under good agitation, for five minutes. The continuous phase was added slowly.

Emulsions were evaluated on a scale of 1-5. 5 being a stable emulsion, 0 being completely unstable.

Results

<table>
<thead>
<tr>
<th>Compound Tested</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
</tr>
</thead>
<tbody>
<tr>
<td>S/W O/W W/S W/O O/S S/O</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Compound Tested</td>
<td>A</td>
<td>B</td>
<td>C</td>
<td>D</td>
<td>E</td>
<td>F</td>
</tr>
<tr>
<td>-------------------------------------------------</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>1. Dimethicone copolyol iso-stearate &quot;A&quot;</td>
<td>4</td>
<td>5</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>2. Dimethiconol stearate</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>3</td>
<td>5</td>
<td>0</td>
</tr>
<tr>
<td>3. Dimethicone copolyol iso-stearate &quot;B&quot;</td>
<td>5</td>
<td>2</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>4. Cetyl Dimethicone</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>0</td>
<td>5</td>
<td>2</td>
</tr>
<tr>
<td>5. Dimethicone copolyol amine</td>
<td>0</td>
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<td>12. Dimethiconol stearate</td>
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Conclusions

1. The evaluation of the surfactant systems has resulted in a modification of the standard HLB system.

2. This modified systems appears to have applicability to a broad range of emulsifiers and emulsion types.

3. The prediction of the ability, and the ability to select emulsifiers to emulsify mineral oil and silicone oil with either being the continuous phase, absent water was unexpected by the authors.

4. The 3 dimensional HLB system predicts that there will be overlap in those materials, which make the two closely, related emulsions. For example surfactants that are on the cusp between oil in water and silicone in water emulsions, will have properties for both. This implies that these materials will be good emulsifiers for systems containing both oil and silicone co-emulsified in water.

References


7. U.S. Patent # 4,960,845.