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# Potential Causes of Color Shift in Pigmented Emulsions

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# Results

- 1) Color Shift
- 2) Unpredictable results
- 3) Stability issues especially pigmented emulsions (W/Si).

# Areas of Potential Problems

- 1. Treated Pigment
  - Bonded
  - Non-bonded
- 2. Emulsifier
  - Homogeneous
  - Non-homogeneous
- 3. Interactions
  - Immediate
  - Age
- 4. Testing
  - Product Development
  - QC

# Treated Pigment

- Why Treat?
  - Make pigment more hydrophobic
  - Make pigment more compatible with oil phase
  - Make pigment less likely to agglomerate
  - Make emulsion more stable

# Types of Treated Pigments

- Reacted
  - All of these materials are based upon reactive coating materials. These can include silicones, silanes and free radical products.
- Chemisorbed
  - All of these provide coating by physical bonding an oil to a pigment making it hydrophobic.

# Chemisorbed Pigments

- Transient Coatings
  - That is the coating will be removed by emulsifiers over time to result in the lowest free energy
  - The time frame is determined by the nature if the coating
  - Can be long time frame
- Accelerated by temperature, emulsifier and overall energetics of the system.

# Treated Pigments

- Reacted Coatings
  - The reaction of the coating to the surface of the pigment results in improved permanence of the coating.
  - Depends upon the reaction and how well it is run each batch
  - QC?

# Example 1

- A pigment is coated with oleic acid in a ribbon blender. The result is an organo modified pigment on which the oleic acid is roughly uniform.
- There is no reaction. The oleic acid stays on the pigment simply by wetting it out and assuming the lowest free energy.

# Example 1 p.2

- The oleic pigment is placed in an invert emulsion along with an emulsifier, an oil, water and other additives, including thickener.
- Under normal conditions the emulsifier would remove the acid and the acid would end up in the oil phase, that is the phase in which it is most stable.

# Example 1 p.3

- Since the formulation is thickened and the emulsion has stability the whole process is delayed!
- A separation that would normally take minutes to occur could take weeks.
- Heat accelerates the effect.

# Example 1
# What does the formulator see?

- “Bad emulsion”.
- Color Shift
  - Coated pigment
  - Naked pigment
- Lack of predictability
- Corrective Steps
  - Change emulsifier
  - Change process for emulsion

# Reacted Pigments

- To the extent the coating process is not correctly run, there can be batch to batch variation in the pigment and to greater or lesser extent the problem outlined as example 1 occurs.

# Reacted Pigments

- This problem then is one of randomness in the lack of performance and is worse in many ways since it is unpredictable batch to batch.
- Drives production and QC people crazy!

# Pigments
# What to do?

- Get full disclosure
  - Reacted or chemosorb
  - Type of coating
  - QC of coating
- Impose QC Test
  - Extraction prior to use
  - Require test from vendor as part of QC.

# Soxhlet Extractor

# Extraction

- A **Soxhlet extractor** is a type of laboratory glassware invented in 1879 by Franz von Soxhlet. It was originally designed for the extraction of lipid from a solid test material, but can be used whenever it is difficult to extract any compound from a solid.

# Extraction

- Typically, dry test material is placed inside a "thimble" made from filter paper, which is loaded into the Soxhlet extractor. The extractor is attached to a flask containing a solvent (commonly diethyl ether or petroleum ether) and a condenser. The solvent is heated, causing it to evaporate. The hot solvent vapor travels up to the condenser, where it cools and drips down onto the test material. The chamber containing the test material slowly fills with warm solvent until, when it is almost full, it is emptied by siphon action, back down to the flask. This cycle may be allowed to repeat many times. During each cycle, a portion of the lipid dissolves in the solvent. However, once the lipid reaches the solvent heating flask, it stays there. It does not participate in the extraction cycle any further. This is the key advantage of this type of extraction; only clean warm solvent is used to extract the solid in the thimble. This increases the efficiency of the extraction when compared with simply heating up the solid in a flask with the solvent

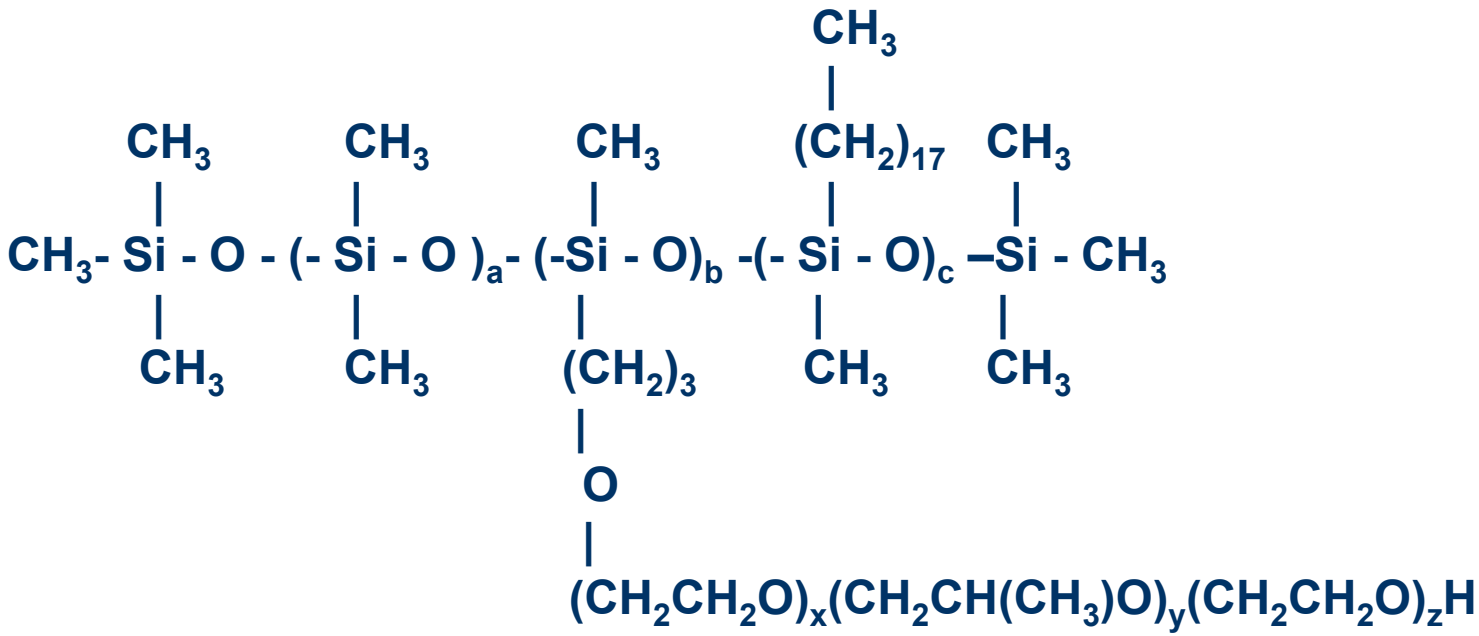
# Emulsifiers

# Emulsifiers

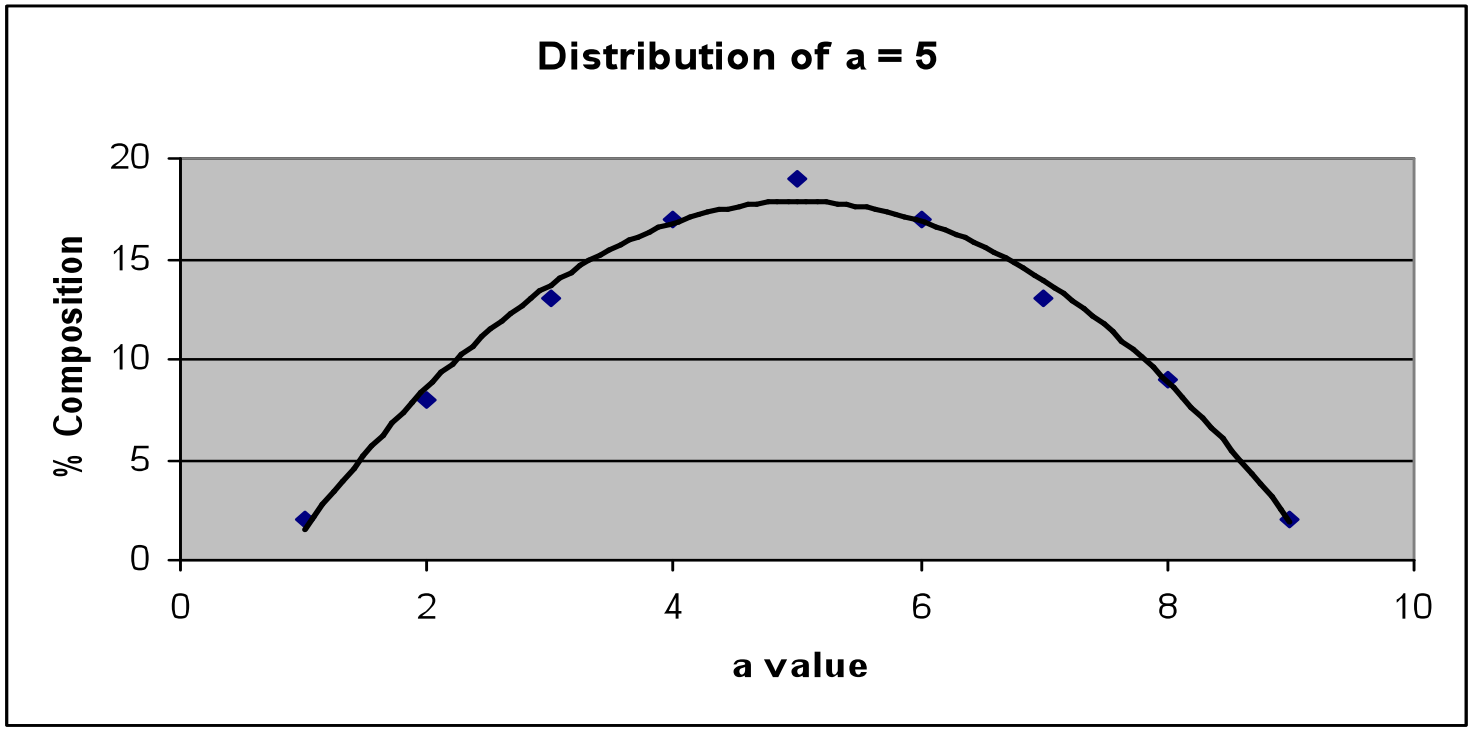
- Emulsifiers are complex oligomeric mixtures of products used with a plethora of other ingredients by the cosmetic chemist in making invert emulsions.

- Invert emulsions are by far the most complicated single phase emulsion in personal care line.

# Alkyl Dimethicone Copolyol



# Distribution



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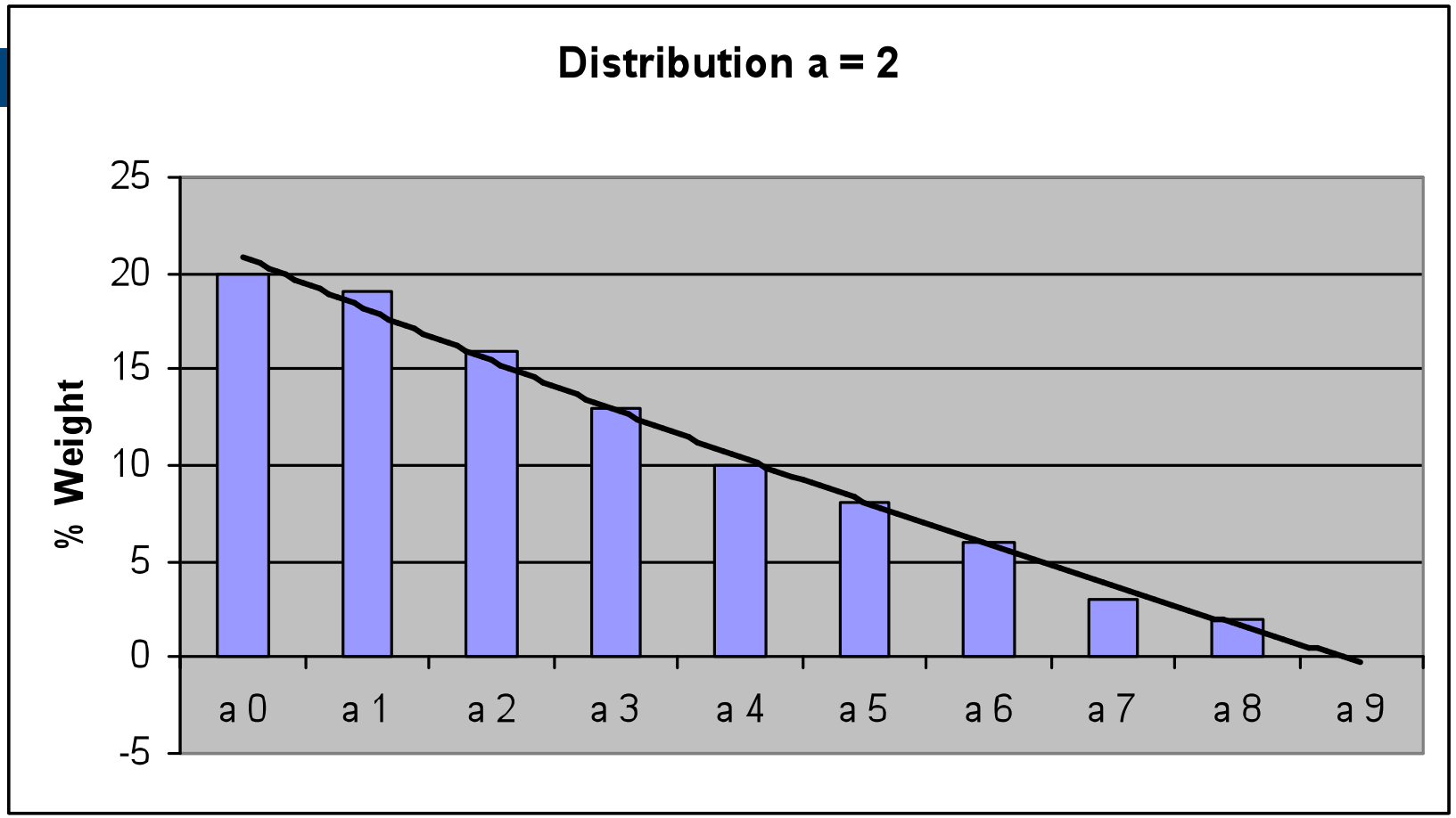

# Alkyl DMC Properties

**These materials are used as emulsifiers in the preparation of both water in silicone and silicone in water systems. These products provide advantageous over traditional hydrocarbon chemistries since they can be used in the preparation of emulsions without heat. These silicone polymers can be used to prepare products that contain little wax, contain a large concentration of water and have a light spreadable feel on the skin. These products possess a water-soluble, an oil soluble, and a silicone soluble portion of the molecule, and consequently the 3D HLB concept applies to these materials. This tool allows for the systematic selection of an emulsifier containing all three parts in one molecule.**

| | % | | 3D HLB | |
|---|---|---|---|---|
| | EO | Alkyl | x | y |
| | | | %EO/5 | % Alk/5 |
| **ADMC J208-212** | 48 | 6 | 9.6 | 1.2 |
| **ADMC J208-412** | 39 | 13 | 7.8 | 2.6 |
| **ADMC J208-612** | 28 | 22 | 5.6 | 4.4 |
| **ADMC J208-812** | 16 | 32 | 3.2 | 6.4 |

## Solubility:

| | Water | | IPA | | Min. spirits | | Min. oil | | Aromatics | | Cyclics | | F350 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 1 % | 10 % | 1% | 10 % | 1 % | 10 % | 1 % | 10 % | 1 % | 10 % | 1 % | 10 % | 1 % | 10 % |
| J208-212 | S | S | S | S | I | I | D | D | S | S | D | D | D | D |
| J208-412 | D | D | S | S | D | D | D | D | S | S | D | D | D | D |
| J208-612 | I | I | S | S | S | S | S | D | S | S | D | D | D | D |
| J208-812 | I | I | S | S | S | S | S | S | S | S | S | S | D | D |

I - insoluble; D - dispersible; S - soluble

# Case Study

**The names have been changed to protect the guilty**

- Emulsion from R&D is robust giving acceptable product every time
- First few production batches are fine

# Case Study

- Soon product is not working well.
- Production complaints product is not performing each time

# Case Study

- Production becomes convinced that R&D has a haunted formula.
- R&D is convinced that production is haunted.

# What is happening?

- It is finally realized that all batches made in the lab were with pre-warmed one phase emulsifier. (a=2).
- Production batches come in metal drums. Operators do not have X ray vision. So they do not know if separated material is in the drum.

# What is happening?

- Separation could well happen in the warehouse if it gets cold.
- Problems of a random type.

# Correction?

- Buy an emulsifier that does not split when cold. (a=5).

# What is happening?

- It is finally realized that all batches made in the lab were with pre-warmed one phase emulsifier. (a=2).
- Production batches come in metal drums. Operators do not have X ray vision.

# Combination Problems?

- Erratic Emulsifier
- Unpredictable Pigment coating

# Very bad situation.

- Correct one situation at a time.
- Kit Approach

| | % | | 3D HLB | |
|---|---|---|---|---|
| | EO | Alkyl | x | y |
| | | | %EO/5 | % Alk/5 |
| **ADMC J208-212** | 48 | 6 | 9.6 | 1.2 |
| **ADMC J208-412** | 39 | 13 | 7.8 | 2.6 |
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# Kit Approach

1. Leave out Pigment
2. Check Emulsion with different emulsifiers
3. Optimize for oil
4. Run pigment extraction test
5. Put pigment back in
6. Re check


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***Questions ?***

***Discussions?***

***Next Step?***