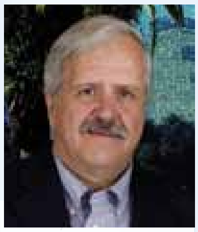
 SCIENCE FOR FORMULATORS

TONY O'LENICK[1]*, THOMAS O'LENICK[2]
*Corresponding author
1. Siltech L.L.C., 1625 Lakes Parkway, Suite N., Lawrenceville, GA 30043, USA
2. SurfaTech Corporation, 1625 Lakes Parkway, Lawrenceville, GA 30019, USA



Science for formulators
This is the first of a series of articles in a new approach to providing science to formulators. The concept is to provide the underlying science and to present it with formulation tips that make the information useful. Happy reading!

Tony O'Lenick is President of Siltech LLC. in Lawrenceville, Ga., a company he co- founded in 1989. Siltech is a silicone specialty company. Prior to that he held technical and executive positions at various surfactant and specialty chemicals companies including; Lambent Technologies, Alkaril Chemicals Inc, Henkel Corporation and Mona Industries. He has been involved in the surfactant and silicone industry for over 35 years. Tony has written 5 books in various aspects of surfactant, silicone and organic chemistry, also published over 70 technical articles in trade journals, contributed chapters to six books, and is the inventor on over 300 patents. He teaches a course in silicone chemistry, surfactant chemistry and patent law. He has received a number of awards for work including the 1996 Samuel Rosen Award given by the American Oil Chemists' Society, the 1997 Innovative Use of Fatty Acids Award given by the Soap and Detergents Association, and the Partnership to The Personal Care Award given by the Advanced Technology Group. Tony is a fellow in the Society of Cosmetic Chemists, and in the American Institute of Chemists. He was a member of several committees in SCC including the Committee on Scientific Affairs, the Continuing Education Committee, and has served the SCC National as Treasurer, Vice President Elect, Vice President and 2015 National President.

Why use silicone in personal care applications?
Part 1 – Dimethicone

KEYWORDS: Silicone, Dimethicone, formulation, hair care.

Abstract The formulator of personal care products is truly faced with a plethora of ingredients, some old, some new. Silicones can come in many forms with varying functionality, performance and solubility. This leaves the formulator in a strange situation, where they must ask "Why use silicone in Personal Care Applications?" This question is sometimes indeed very difficult to answer, partly because the information needed to choose suppliers do not provide the correct silicone. Formulations produced today are very complex, they are multi-ingredient compositions that must have synergy to produce the desired effect. This article will address the basic silicone polymer, that is one in which there is no organo-functionality, which is polymers that contain only CH3, Si, and O groups.

INTRODUCTION

Silicone polymers are a very diverse class of compound that encompass a wide variety of materials from traditional dimethicone to polymers that have many different functionalities in the polymer. Based on the functionality incorporated into the silicone polymer, a wide variety of properties can be obtained: water soluble silicone polymers, oil soluble silicone polymers, fluoro soluble silicone polymers. The problem is that the choice of silicone fluids is overwhelming due to the lack of a roadmap that will allow for the selection of the formulator friendly silicone for specific application. That roadmap is known, but rather guarded by those who understand it. The roadmap isn't an actual map hanging on the wall, instead it is an understanding of the silicone technology and the structure function relationship that exists within the technology base. The knowledge on this "roadmap" is no more complicated than the technology base used to make surfactants. While different, it is every bit as rich and flexible. It also needs to be clearly understood that if one considers a personal care formulation to be a gourmet meal, the silicone polymer will be the spice and not the meat or potatoes of the formulation. This means use them sparingly and to provide properties not available from other

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raw materials. Fortunately of the formulator, silicone polymers can be used at extremely low concentrations to get a desired performance. Performances shown in Table 1.

	Silicone Attributes
1.	Lowering surface tension to around 25 dynes/cm
2.	Providing unique skin feel, cushion and playtime
3.	Providing unique solubilities (are soluble in silicone, oil, water and fluoro compounds)
4.	Can provide emulsification with unique aesthetics (especially invert emulsions)
5.	Provide film/formation
6.	Provide water resistance
7.	Provide foaming for non-traditional formulations

Table 1. There are many characteristics that make Silicone such an interesting material for formulations.

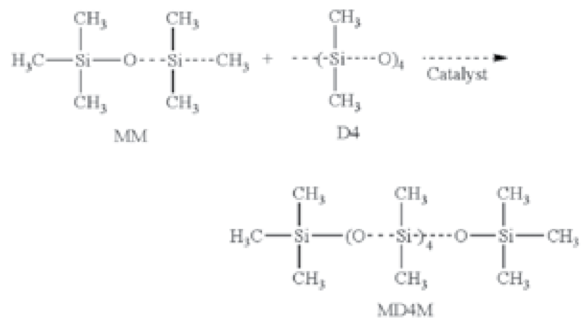
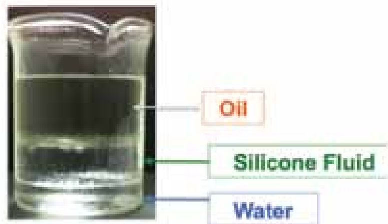


Figure 1. The synthesis of a dimethicone (MD_4D) starting with MM and D4. The typical reaction is conducted at room temperature for 12 hours, with sulfuric acid at 2% by weight as catalyst resulting in a mixture of about 10% free cyclic product and 90% linear fluid.

Figure 2. Separation of oil, dimethicone and water (3).



Today there are fewer formulators, making more complex formulations in a shorter time with more complex raw materials to meet the ever-changing market, regulatory and patent needs this requires that the formulator be more critical in including raw materials in the formulation and becoming more efficient in making products. With a good understanding of the basics of silicone chemistry, a formulators job can become a little easier.

Dimethicone

The "simplest" and oldest silicone polymer is commonly called silicone fluids or dimethicones. They contain a repeat unit of $(Si(CH_3)_2\text{-}O)$ and encompass a variety of forms from low viscosity fluids, to rubbery elastomers and brittle resins. Dimethicone polymers are synthesized by the equilibration reaction of hexamethyldisiloxane (MM) and cyclomethicone. The reaction is a ring opening reaction. Typical of the synthesis of fluids is the following reaction in which one MM is reacted with one octamethylcyclotetarsiloxane(D4) compound to make MD_4M, a simple dimethicone. The reaction may be run with either an acid or base catalyst. Typically, the reaction is conducted at room temperature for 12 hours, with sulfuric acid at 2% by weight as catalyst resulting in a mixture of about 10% free cyclic product and 90% linear fluid. It the catalyst is neutralized and the cyclic is stripped off, a stable fluid results. If the catalyst is not neutralized during stripping, the fluid will degrade back to MM and D4 (see *"Properties of Silicone Fluids"*, below).

A "finished dimethicone" fluid may be placed in contact with D4 and catalyst and re-equilibrated to make a higher viscosity fluid. Conversely, a "finished dimethicone" may be re-equilibrated with MM and catalyst to make a lower viscosity fluid. Finally, silicone rubber may be decomposed into MM, and D4 via stripping of the product in the presence of catalyst. This property of silicone polymers makes them decidedly different from organic compounds. Figure 2 shows the reaction.

The equilibration process is critical not only to produce stable dimethicone polymers, but as a means of introducing functional groups into the polymer. This will be discussed in more detail in the section on hydrosilylation, a process used to make organo-functional silicone compounds.

One of the most important factors to be understood about silicone polymers in which there are no organo-functionalities is that they are insoluble in both water and common oils. Figure 1 shows the separation of the three phases. It is this property that makes silicone polymers unique in their applications in personal care.

It is this lack of solubility in both oil and water that leads to some interesting characteristics of silicones. Along with hydrophilic and hydrophobic, materials can be siliphilic (silicone-loving) or siliphobic (silicone-hating).

Properties of silicone fluids

Dimethicone polymers, also called silicone oils, or simple silicone are sold by their viscosity and range from 0.65 cSt to 1,000,000 cSt. Typically the viscosity of the silicone fluid is supposed to be the molecular weight of the silicone polymer. The higher the viscosity, the higher the molecular weight. Unfortunately for formulating chemists, this is not always the case. The blending of two different molecular weight silicones can produce a viscosity that is in between the two molecular weights. If the silicone polymer is not a blend of silicones, the viscosity allows for an approximate calculation of the value of "n" (or DP) in Table 2.

Viscosity (25 °C, cSt.)	Molecular Weight$_{CAL}$	n$_{CAL}$
5	800	9
50	3,780	53
100	6,000	85
200	9,430	127
350	13,650	185
500	17,350	230
1,000	28,000	375
10,000	67,000	910
60,000	116,500	1,570
100,000	139,050	1,875

Table 2. Calculation of the "n" value (3).

The viscosity in non-blended Dimethicone polymers will be the salient characteristic, the viscosity will determine the cushion, play time, and spread achieved when applying the dimethicone to the skin or hair.

The silicone is generally added to the oil phase and is almost always used with other oils. This, not only increases the complexity of choosing the proper emulsifier, but also can decrease the stability of the resulting emulsion, since there are three phases in the emulsion (oil, silicone and water) not just two.
Dimethicones breakdown into several categories;

1. Volatile dimethicone
(linear, non-crosslinked silicone having a viscosity of less than 5 cSt)

Typical products
- 0.65 cSt (CAS# 107-46-0)(MM)
- 1cSt (CAS# 107-51-7)
- 3cSt (CAS # 63148-62-9)

Silicones of low molecular weight are considered to be volatile. Once the molecular weight increases past a certain point, the silicone polymer become non-volatile. Volatile silicones are commonly used as solvents, or for a "dry" feel on the skin. The biggest drawback of the volatile silicone is: many of them are considered to be Flammable and formulations that utilize a high content of volatile silicones will have to contain a red label. Since red-labeled products are not desirable in a manufacturing environment, care must be exercised to use raw materials that are not flammable.

INCI Name Common Name	Hexamethyl – disiloxane MM	Dimethicone 1 cSt fluid	Dimethicone 1.5 CSt fluid	Dimethicone 2 cSt fluid
Viscosity (cSt)	0.65	1	1.5	2
Molecular Weight	162	236	311	385
Specific Gravity	0.76	0.816	0.85	0.872
Refractive Index	1.375	1.382	1.387	1.389
Solubility Parameter	6.7	6.9	7.0	7.0
Flash Point (°C)	-3	34	56	87

Table 3. Characteristics of the volatile dimethicones.

These classes of products are used in a wide variety of antiperspirants, skin creams, skin lotions, suntan lotions, bath oils, and hair care products. They possess low surface tensions and exhibit excellent spreadability. Flammability is also an issue as one evaluates hydrocarbon replacements for D5, as most hydrocarbons suitable for this application are flammable while D5 is not. We suggest that in the future the use of D5 will be limited in the personal care market because there are more efficient materials that can be used in making products that now contain D5.

Formulation tip
There are many different "D-5 Replacements" being offered from hydrocarbons, branched hydrocarbons, esters and a plethora of organic substitutes. There are likewise a plethora of silicone replacements including 0.65 viscosity, and others that although dry in feel are flammable. As with any raw material asking "Why use this raw material" is the key to prudent selection.

2. Low viscosity dimethicone
(linear, non crosslinked dimethicone having a viscosity between 5 and 50cSt)
CAS# 63148-62-9

Typical products:
- 5cSt
- 10cSt
- 20cSt

Applications
Low viscosity Dimethicone polymers do not have as dry a feel as volatile fluids. This is because they do not evaporate, but spread on sin and hair. Primarily used is as an ingredient in a number of personal care products due to their high spreadability, low surface tension and subtle skin lubricity. These polymers are clear, tasteless, odorless and provide a non-greasy feel. They are used in a wide variety of skin creams, skin lotions, suntan lotions, bath oils, and hair care products

3. Regular viscosity dimethicone
(linear, non crosslinked dimethicone a having a viscosity of between 50 -1,000cSt)
CAS# 63148-62-9

Typical Products
- 50cSt
- 100cSt
- 200cSt
- 350cSt
- 500cSt
- 1,000cSt

These products have a cushion and play time when applied to the skin. The higher the viscosity, the higher the cushion and play time. These products are also blended with other oil phases and emulsified. Dimethicone polymers with a viscosity of 200 cSt and below are soluble in anhydrous alcohol, while higher viscosity products are insoluble.

4. High viscosity dimethicone
(linear, non crosslinked dimethicone viscosities between 10,000cSt -60,000cSt)
CAS# 63148-62-9

Typical Products;
- 10,000 cSt
- 60,000cSt

These Dimethicone polymers are very thick and provide a lot of cushion and play time in personal care products. They are rarely used as the primary oil phase in personal care products.

5. Ultra high viscosity dimethicone
(linear, non crosslinked dimethicone having a viscosity of over 60,000 cSt)
Typical products:
- 100,000 cSt
- 500,000 cSt
- 1,000,000 cSt

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These Dimethicone polymers are extremely thick. They are combines with lower molecular fluids and the combination can be used as a serum for hair.
Table 4 is provided to give meaning to the viscosity values, by relating them to everyday materials (7).

Table 4. Viscosity comparison refereed to common materials.

cSt	Reference
5	Water
10	Transformer oil
20	Kerosene
50	Sae 5 motor oil
1,000	Light syrup
2,500	Pancake syrup
10,000	Honey
25,000	Chocolate syrup
50,000	Ketchup
60,000	Molasses
100,000	Hot tar
250,000	Peanut butter

Dimethicone blends

Because the properties of Dimethicone polymers vary so much as the viscosity increases, it is common to blend Dimethicone polymers having different viscosities together to produce a bimodal blend that offers properties that neither dimethicone have alone. Blending dimethicone polymers is a very common, very important method to obtain products that cannot be accomplished with one polymer.
Before addressing the blending, it is important to note that all polymers are oligomeric compositions. This means that there are a series of different molecular weight polymers in the product. This is not a situation unique to silicone polymers. We see a Gaussian distribution of polymers in ethoxylates and many other common polymers.
A typical GPC[4] showing the distribution of polymeric species in a polymer is seen in Figure 3.

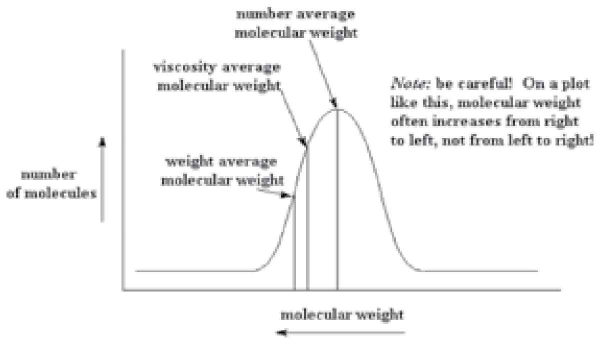

Figure 3. Polymer distribution.

The distribution shown in Figure 3 is not a bimodal distribution. It is one distribution. A bimodal distribution is the result of a blend. Figure 4 shows a bimodal distribution of two very different molecular weight silicon polymers.

Bimodal distribution

This type of bimodal distribution results from blending a low viscosity fluid with a high viscosity fluid. Consider a blend of a dimethicone having a viscosity of 100,000 cSt with a fluid having a viscosity of 50 cSt. The theoretical GPC, assuming that their hydrodynamic volumes are different in the mobile phase, of such bimodal fluid would look like the one shown in figure 4. Such a fluid would have very different functional properties that a single modal product made to the same viscosity as the resulting blend. Specifically, in the bimodal blend there are no molecules having the number average molecular weight, while in the mono-modal product, the concentration of the of polymer at the number average molecular weight is very high (Figure 3).

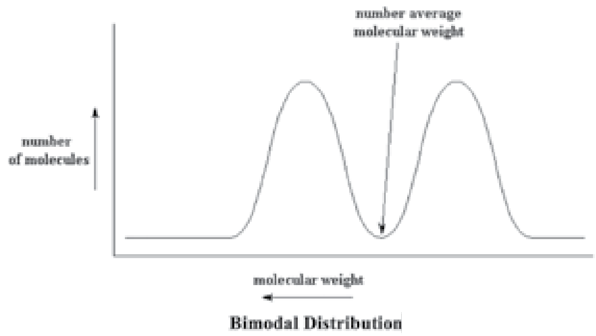

Figure 4. Bimodal Distribution.

From Table 4 one can see that the pure 100,000 cSt fluid would be as thick as hot tar. It would not spread well on the skin and would only be viable in a personal care product in a blend. The 50 cSt by itself product would be very thin and have little or no cushion. By blending the two a the 50 cSt fluid lowers the viscosity of the blend, provides for ease of spread and acts like a 'delivery' system for the high viscosity fluid. The desirable properties of the high viscosity fluid are delivered in a cosmetically elegant way. Also keep in mind that the 50 cSt, the 100,000 cSt and the blend all share the INCI name of dimethicone.
Making blends of different concentrations of different viscosity fluids results in an ability of the formulator, or silicone supplier to provide blends with highly unique properties in formulation. Consider blends that are much closer in viscosity, like a 25 cSt and a 50 cSt blend. The GPC of such a blend is presented in figure 5. This is indeed a strange GPC when compared to those shown above. It is a bimodal GPC that is poorly separated. The formulation properties of this type of blend when applied to cosmetic substrates would be very different that either the dimethicone that is mono-modal or the clear bi-modal one.

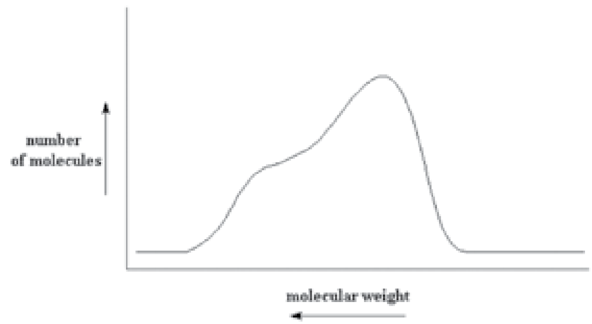

Figure 5. Blend of Dimethicone polymers – Similar in Viscosity.

The importance of understanding the blending of Dimethicone polymers in formulation will be shown in applications for hair in the next section.

Hair care

The use of dimethicone, in hair formulations, can be understood by looking at the type of damage that needs to be remediated. Dimethicone polymers can be delivered to hair in a variety of ways, a serum, a two-in-one shampoo (coacervate) or in a conditioner (generally a laminar gel network). Despite the variability in delivery mechanisms (which offer different formulation challenges), the function of the dimethicone, once delivered, remains the same.
the attempt of optimizing the cosmetic appeal of hair, we subject our hair to a lot of stress. Hair damage comes from a variety of sources including:

I. Sun damage
II. Mechanical damage
III. Heat damage
IV. Processing damage
V. Environmental (pollution) damage

Usually hair damage takes place gradually, stage by stage, as follows:
I. the hair is weakened;
II. the cuticle begins to break down;
III. the cuticle disappears, layer by layer;
IV. the cortex is exposed;
V. split ends appear;
VI. the hair breaks.

Dimethicone polymers have a low surface tension so they spread well on the hair. They are highly lubricious so they lubricate damaged dry hair. Dimethicone polymers lock in water and protect hair color from washing out. The problem is making a cosmetically acceptable product that is water based, like most shampoos, using a water insoluble dimethicone. Suspending agents are used commonly. Two in one shampoos based Dimethicone polymers are commonly not clear. Organo-functional materials are added for clear two in one products.

- Conditioners that contain dimethicone (a dimethicone compound deposit mainly at the edges of the cuticle scales - just where the damage happens most easily.
- Micro-fine droplets make the hair surface smooth and shiny (less 'fly-away')
- Dimethicone protects the hair from damage by reducing its resistance to brushing, combing and styling, when wet as well as when dry.

The difference between healthy hair and damaged hair is shown in figure 6 and figure 7. The ability of a dimethicone applied to the hair to spread on the hair and to deliver lubrication to the hair fiber is critical to performance. This means there are at least two functions, wetting/spreading and conditioning/lubrication. The scales shown on the damaged hair clearly show that the conditioning/lubrication must be preceded by wetting / spreading and that dimethicone of too high a viscosity will simply stay on top of the hair. The balance between the two properties needs to be balanced by both the viscosity and concentration of dimethicone blended.

Figure 6. Healthy hair is smooth and uniform.

Consider the application of a dimethicone serum to the damaged hair. For simplicity, assume the serum is optimized for this application at a viscosity of 800 cSt. By optimized is meant that the serum spreads on and wets the hair at a satisfactory rate of speed. The question now is which product or blends of products work best in the application? The simplest approach is to use mono-modal dimethicone having a viscosity of 800 cSt. This is indeed one approach, but remember the low viscosity dimethicone products spread and wet and the higher viscosity condition, so one would expect a blend would work best. There are in fact a number of blends possible. Table 5 shows eight of dimethicones (8).

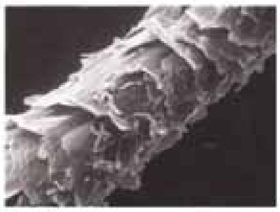

Figure 7. Damaged hair: has 'scales' visible and is degrading.

Example	High Viscosity Viscosity	High Viscosity %	Low Viscosity Viscosity	Low Viscosity %
1	1,000	93.0	50	7.0
2	1,000	68.0	500	32.0
3	1,000	96.0	5	4.0
4	1,000,000	42.0	5	58.0
5	10,000	52.0	50	48.0
6	100,000	36.0	50	64.0
7	1,000,000	28.0	50	72.0

Table 5. Dimethicone blending (targer viscosity: 800 cSt).

The high viscosity fluid in the blend ranges from 1,000 to 1,000,000 cSt, and the low viscosity fluid ranges from 5 to 500 cSt. The higher the concentration of the high viscosity fluid, the less of it is required to meet the target viscosity. This means more of the wetter / spreader viscosity is present. The ranking in the salon is shown in table 6 (Ranking is 1 is the best 8 is the worst).

Example	Ranking
800 CSt	7
1	6
2	8
3	5
4	1
5	4
6	3
7	2

Table 6. Ranking of hair serums. Ranking is from 1 (the best) to 8 (the worst).

As shown above the product judged best is the blend with a wide difference in viscosity between the high and low viscosity component and the one judged worst is the mono-modal fluid. This is an important factor to consider in the use of any silicone in formulation, would a blend of two different silicones with the same INCI name provide better performance than a single fluid? The answer is determined by first asking "Why use silicone?" then running the evaluation in the salon.

Formulation tip

When considering blends of dimethicones, ask what is the performance of a specific dimethicone. Then consider weather that silicone alone will work by itself or work better in the blend of at least or more silicones. Make sure to also consider if variations in the ratio of viscosity of the blends of choice of viscosities in blends would allow for product differentiation. It is quite conceivable that different ratios of

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ultra high and low viscosity blends might be altered for normal hair or for damaged hair. You might also ask what part does blending of dimethicone polymers play in heat protection of hair?

Dimethicone on substrate (9)
Oil introduced into water disrupts the hydrogen bonding between the water molecules. This disruption is accomplished only when the energy of mixing is sufficient to break the hydrogen bonds. When the mixing is stopped the oil is forced out of the water by the re-formation of the hydrogen bonds between water molecules. This phenomenon can be used to deliver of oil to a surface. Dimethicone fluids are delivered this fashion.

Improved adhesion properties of the dimethicones are obtained by creating molecules that operate by additional mechanisms including:

- Ionic Interactions: The charge on the molecule will also have an effect upon the delivery of the oil to substrate. An oil carrying a cationic charge will form ionic bonds with substrates that carry negative surface charges. The two opposite charges together forms a so-called ion pair bond. Since ionic charges are quite common existing on textile fabrics, fibers, glass and hair and skin this type of bonding becomes quite important.
- General Adhesion: If an oil delivered to a substrate penetrates and then polymerizes, an interlocking network of polymer will develop. Although not bonded directly to the substrate, this polymer network will adhere to the substrate.
- Specific Adhesion: If an oil is delivered to a substrate penetrates and then reacts with groups on these substrates, a chemical bond will b formed. This is the strongest and most permanent of the adhesion mechanisms.

Dimethicone polymers react almost exclusively by the mechanism related to hydrophobicity. To the extent the other mechanisms may be introduced, the more strongly and efficiently the conditioner can be delivered to substrate. Organo-functional Dimethicone polymers depend in large part on these additional mechanisms to provide thorough and efficient conditioning, lubrication and softness to the substrate.

Cosmetic usage of dimethicone polymers (10)

There are several cosmetic usages of Dimethicone polymers, as shown in Table 7.

Dimethicone polymers are used in low concentrations in many applications. The reason for this relates to their hydrophobicity, which results in their deposition on skin. This deposition has many desirable properties. Dimethicone polymers can be used to remove tacky organic products, like soaps from the skin, while providing lubrication, and a skin feel that is highly desirable.
While the mechanism of use is the same and independent of the viscosity chosen, the properties of the cosmetic product made with the different viscosity dimethicone polymers are quite different. The skin feel can be altered significantly by

Cosmetic Use of Dimethicone polymers		
Product Type	**Desired Effect**	**Use Level**
Skin Lotion	Desoaping Rub-out Protection Feel	0.1% 1 – 0.5% 1 – 30% 0.5 – 2 %
Skin Cleaner	Lubricity Wetting	0.1 – 0.5% 0.1%
Antiperspirant	Anti-whitening Detackification	0.5 – 2% 0.5 – 2%
Preshave Lotion	Lubricity	0.5 – 2%
Aftershave Lotion	Feel	0.5 – 2%
Makeup	Water Resistance	1 – 5%
Shaving Cream	Reduce Razor Drag	0.5 – 2%

Table 7. Cosmetic Use of Dimethicone polymers.

picking the proper dimethicone. Higher molecular weight products have an oilier feel on the skin, while lower viscosity products have a dryer feel on the skin.
One key limitation on the use of dimethicone polymers in formulation is the fact that the concentration needs to be kept very low. The very same insolubility that is a consequence of the hydrophobicity and drives the deposition places severe limitations on the useful concentrations. If too much dimethicone is used, it will result in a product that the will separate, have difficulty foaming, and will lack the most basic aesthetics for a personal care product. Skilled formulation helps, but modification of the product and introducing organo-functionality into the product is a major way to overcome the shortcomings of dimethicone polymers.

Formulation tip
Looking at table 7, which viscosity or viscosities of dimethicone might be used in which products? Specifically, what trade off of solubility, aesthetics and final product viscosity need to be made to make the commercially acceptable product?
Future articles will address the oil soluble and water-soluble dimethicone polymers and the distinct advantage they offer over dimethicone polymers in formulation personal care products.

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