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Science for Formulators

This is the second 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!

The first one was published in H&PC Today Vol. 11(1) pag. 54-59

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Why Use Silicones in Personal Care Products Part 2 - Water Soluble Silicone Polymers

KEYWORDS: Silicone, Dimethicone, formulation, hair care.

Abstract This is the second of three articles on the use of different types of silicone polymers in personal care formulations. The question "Why Use Silicones in Personal Care Products?" is an important question all formulators need to consider. There are a growing number of silicone polymers available to the formulator, which is a testimony to the creativity of the polymer synthesis chemist. While this is a good thing for the formulator in that there is a large pallet of silicones from which to prepare outstanding formulations that must be cost effective, meet consumer expectations and the marketing requirements of the companies selling the formulation., it can also be the source of some problems with making the correct choice of polymers in a specific formulation. Properly formulated personal care formulations are like fine gourmet meals in which proper choice for the silicone polymers is to function as the spice of the meal, not the meat or potatoes. These versatile polymers need to be used at low concentrations to provide formulations that cannot achieve the overall effect without them. The first article in the series dealt with silicone polymers that contained no functional groups to alter the solubility. This article will deal with silicone polymers that contain water-soluble groups.

INTRODUCTION

Silicone Polymers are chosen for formulations to fulfill a basic need that cannot be achieved using other chemistries. Silicone polymers have a handful of salient properties that make them value able in formulation. They are outlined in Table 1 (1).

The formulator must first decide which of these properties is required in a given formulation then evaluate the proper type of silicone. We suggest minimally disruptive

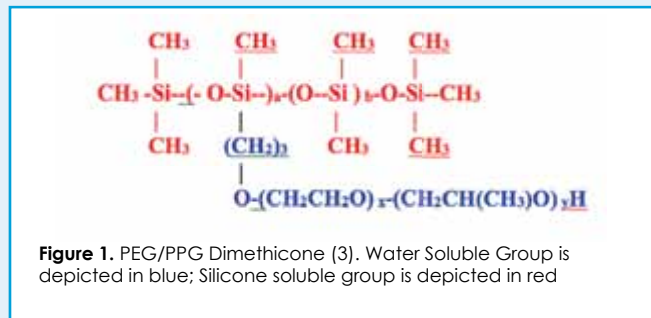
A properly closed silicone polymer can provide the following attributes to formulations;

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

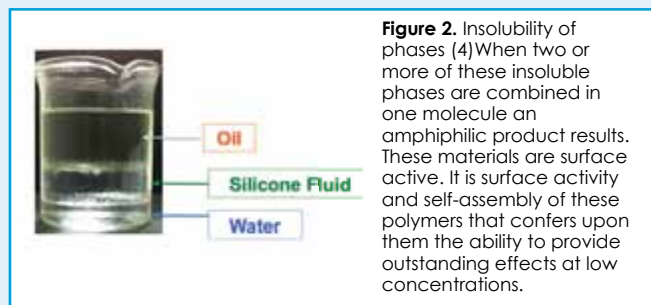
Table 1. Silicone attributes.

formulation technique to evaluate several silicone polymers in formulation. This powerful approach makes changes to formulations in which the silicone is present in low concentrations with other silicone polymers to evaluate each in an established chassis, allowing for fine tuning of the silicone polymer for the effect desired by the formulator.

The silicone polymers we examine are referred to as PEG / PPG Dimethicone compounds. Formerly, these materials were referred to as Dimethicone copolyols. Regardless of the INCI designation these materials conform to the structure shown in Figure 1 (2).

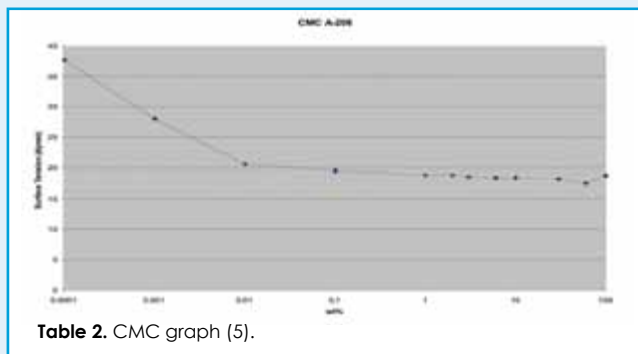


What distinguishes these polymers from dimethicone polymers is the fact that these materials have a water-loving group in them. It must be clearly understood that water, oil, silicone and furor compounds that have only one group present on them will not be soluble in the others. Figure 2 shows that insolubility.



We clearly understand the addition of standard fatty surfactants to water and the effect the addition has on the solution. The addition of sodium lauryl sulfate to water results firstly in a decrease in surface tension. As the concentration increases, the surface becomes saturated with surfactant and there is no additional decrease in surface tension. This is the so-called critical Michelle concentration or CMC. The presence of a properly chosen surface active agent at the CMC will provide wetting as a result of surface tension lowering. The proper selection of PEG/PPG dimethicone will provide not only surface tension reduction, but have a dramatic effect upon skin feel. As the concentration continues to be increased micelles form. Detergency, emulsification, and related properties result from use of a properly chosen silicone surfactant.

Silicone surfactants display a CMC, (critical micelle concentration) as shown in Table 2. They get to the surface and lower surface tension then agglomerate once a critical concentration is reached. A-208 is a PEG-8 Dimethicone with a molecular weight of around 611.



The trick is to choose the proper surfactant for your formulation by picking the proper polymer in terms not only solubility but also surface tension, wetting, and aesthetics. All this requires a knowledge of structure / function of the polymers one uses.

Solubility

The very first thing the formulator needs to consider is which silicone polymer is soluble in which phase of my formulation? A silicone polymer that is not soluble in a particular phase will provide no meaningful property alteration to that phase. Stated a different way, if the silicone polymer is not soluble in the phase in which you want to see the benefit of the addition it will be of no value to that phase. When a product is a one phase system, this is easy. Table 3 shows the approach for single phase formulations (solutions including oil and silicone based strums, and water soluble products). Table 3 shows the type of additive that is needed for modification of a formulation having only one phase.

Phase	Additives
Water	PEG / PPG Dimethicone, Silicone quaternary compounds
Oil	Alkyl Dimethicone
Silicone	Dimethicone or Dimethicone blends

Table 3. Single phase products.

If a formulation is an emulsion, a silicone polymer that goes into each phase will be a possible candidate, altering the properties of the phase in which the silicone is soluble. For example an oil in water emulsion will likely benefit from/the addition of one or more silicone surfactants in each phase, depending upon which phase you desire to alter and if you are looking for surface tension reduction or aesthetic modification, Each phase should be evaluated for a silicone polymer addition in a minimally disruptive technology approach to determine the effect upon the formulation. Generally speaking the silicone will be in the formulation at 2% or less by weight. We recommend looking at a wide range of silicone polymer in the different phases to learn how additions of each will affect the final properties.

Solubility is the first requirement, but just because a silicone polymer is soluble in a formulation does not mean it will provide the desired benefit in the most efficient way. Solubility is the first requirement, but certainly not the only requirement.

Micro emulsions (6)

In essentially all rinse off applications, the formulator wants the silicone to end up on the hair or skin, rather

than down the drain. This means if the silicone polymer is too water soluble it simply does not remain on the hair. If however, the silicone polymer is too insoluble, adding it to an aqueous formulation will result in separation and defoaming. The solution is to design the silicone to spontaneously form a micro emulsion. Micro emulsions are very interesting since it is the surfactant that provides a micro emulsion that has the best combination of solubility, deposition and clarity.

Micro emulsions are clear to slightly blue, thermodynamically stable, isotropic liquid mixtures. In contrast to ordinary emulsions, micro emulsions form upon simple mixing of the components and do not require the high shear conditions generally used in the formation of ordinary emulsions. The IUPAC definition is in Table 4 (7).

The ability to regulate the structure of a silicone polymeric PEG / PPG surfactant to provide a micro emulsion in formulation, provides a mechanism that can be used to maximize deposition. Simply put, if PEG / PPG Dimethicone is too water soluble, it will wash down the drain with rinse off and provide little or no additional benefit to the formulation. If you want to add them to efficient PEG / PPG Dimethicone to a formulation for deposition and providing long term effect in a system in which the PEG / PPG Dimethicone is clear and soluble, use a PEG / PPG Dimethicone that provides a micro emulsion in the formula tested.

The most simple way to do that is to alter the D / D* ratio (the ratio of a:b). In so doing the solubility is altered and a product, which is the least soluble without insolubility, is added making the lowest free energy on the substrate being deposited on the substrate. This means if you want to get the best delivery on a substrate and still maintain clarity for the formulation, deliver the silicone polymer as a micro emulsion. Table 4 shows the effect of altering the D / D* ratio in PEG Dimethicone (8).

In fact it is quite easy to use a formula shown in Table 4 to effectively determine the number of PEG 8 units in a silicone polymer to make a micro emulsion.

Formula to determine soluble dimethicone copolyol

$$\text{"Minimum needed "b" units} = (0.1499)(\text{actual number of "a" units}) + 1"$$

b is the subscript "b" in the structure above; "a" is the subscript "a" in the above structure. This means that if the number of "b" units is above the value calculated an appreciable concentration of insoluble oligomer would not be present. If the value of "b" in the polymer is lower than the value of "b" requires in the formula for the number of "a" subunits, a soluble product is impossible since there will be an appreciable amount of insoluble polymer present in the oligomer mixture.

Choosing a D: D* ratio that provides a silicone polymer that forms a micro emulsion is the most simple, most cost effective method to improve the efficiency of a PEG / PPG dimethicone in formulation. The INCI name is even the same!

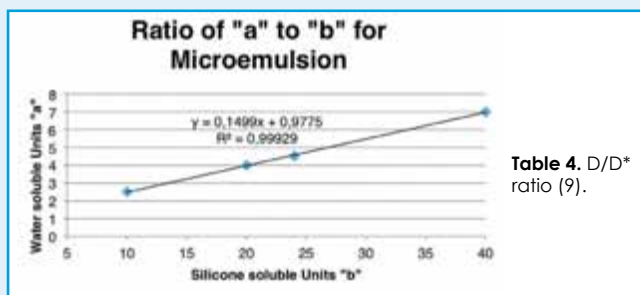


Table 4. D/D* ratio (9).

The above formula was determined by preparing evaluating a variety of dimethicone copolyol products with eight moles of ethylene oxide (INCI: PEG 8 dimethicones). The results are shown in Table 5.

a	b	Predicted* (Formula Below)	Observed (Lab Synthesized)
5	1.0	Insoluble	Insoluble
5	2.0	Soluble	Soluble
10	2.0	Insoluble	Insoluble
10	2.5	Micro	Micro
10	3.0	Soluble	Soluble
20	3.5	Insoluble	Insoluble
20	4.0	Micro	Micro
20	4.5	Soluble	Soluble
24	4.0	Insoluble	Insoluble
24	4.5	Micro	Micro
24	5.0	Soluble	Soluble
40	6.0	Insoluble	Insoluble
40	7.0	Micro	Micro
40	8.0	Soluble	Soluble

Table 5. Solubility (10% Water).

* using the micro emulsion formula:
Minimum needed "b" units = (0.1499)(actual number of "a" units)+1

Formulation Tip

The preparation of any formulation always has to do with balance. The selection of the easiest raw material to incorporate into the formulation is almost always the selection of a less efficient approach, particularly if the raw material is old or comes from another industry. Always challenge the raw material supplier to provide the most efficient product. Always consider why the raw material is in the formulation and how it may be modified to improve the efficiency it provides. Remember solubility is generally the enemy of deposition, and that silicone down the drain simply wastes money with no benefit.

Surface Tension Reduction

Once solubility is established in the formulation, in whatever phase the benefit is desired, the ability to lower surface tension is the next most critical factor in efficient formulations. If a silicone polymer cannot lower surface tension of the formulation, it will be of limited value in the formulation. We recommend always looking at surface tension of the formulation with and without the silicone polymer being added. Surface tension reduction is key to performance. Surface tension is a key analysis for formulation with silicones.

Just because a silicone polymer lowers surface tension does not assure it will provide the desired effect upon the formulation. Many patents lump together all PEG / PPG Dimethicone polymers into one class regardless of structure. This is just incorrect. Since PEG / PPG Dimethicone polymers cover a wide range of ratio to silicone portion to water portion, it is not logical to assume all of them will function the same way in formulation.

Consider the polymers in Table 6. They all have the same backbone, but differ in the amount of PEG and PPG in the molecule.

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Product	PEG PPG Ratio	Surface Tension
6-1	8 PEG / 0 PPG	25.3
6-2	18 PEG / 6 PPG	30.8
6-3	16 PEG /16 PPG	32.0

Table 6. Effect of PEG / PPG Ratio to surface tension.

The polymers in table 6 all having the same backbone with differing amounts of PEG and PPG have very different surface tension values. Product 6-3 would function, from a surface tension point of view, not as a silicone surfactant but as a fatty surfactant that contained silicone!

The reason for this is the fact that the reduction of surface tension using silicone polymers is determined by what is seen at the interface. Specifically, when the amount of PEG / PPG in the molecule is large, it overcomes any surface active effects of the silicone present. Put another way the surface tension is determined by the methylene groups (-CH₂-) in the PEG / PPG and not the methyl groups present in the silicone (-CH₃). The addition of a silicone polymer at a surface tension of 32 dynes / cm would have little or no effect upon feel, and wetting but may offer minimal emulsification effects.

Formulation Tip

Just because there is silicone in the polymer you purchase does not mean the advantage of silicone you seek is conferred on the formulation in which you add that particular polymer. A molecule with 0.1% silicone and 99.9% fatty will act like a fatty compound. Surface tension tells you what is happening at the surface and if you silicone is able to get to the surface to provide the desired benefit.

Surface Tension Reduction of Traditional Surfactants

Thus far we have limited discussion to silicone polymers in water, not in formulation. Interactions between components in solution and silicone polymers is a very real consideration and is key to optimization in formulation. The understanding and quantification of interactions in the average cosmetic formulations between ingredients is complicated enough for awarding a PhD for each successful formulation.

The interaction between PEG 8 Dimethicone and SLS and SLES 2 is shown in Table 7.

	Cocamidobetaine % weight	PEG-8 Dimethicone % weight	Surface Tension Dynes/cm
Example 7.1	100	0	31.3
Example 7.2	75	25	26.0
Example 7.3	50	50	23.1
Example 7.4	25	75	21.6
Example 7.5	0	100	20.1

Table 7. Interaction between PEG 8 Dimethicone and SLS and SLES 2.

Table 8 shows that when two or more surfactants are present in aqueous solution interactions could result that alter the expected CMC graph shown in Table 2. This alteration makes the calculation of CMC difficult as there is no clear break point.

In order to evaluate the efficiency of the ability of a silicone surfactant to decrease surface tension in a mixed surfactant system a new measurement needed to be created. This measurement is the concentration needed to reduce the surface tension to 25 dynes/cm. It is referred to as RF50.

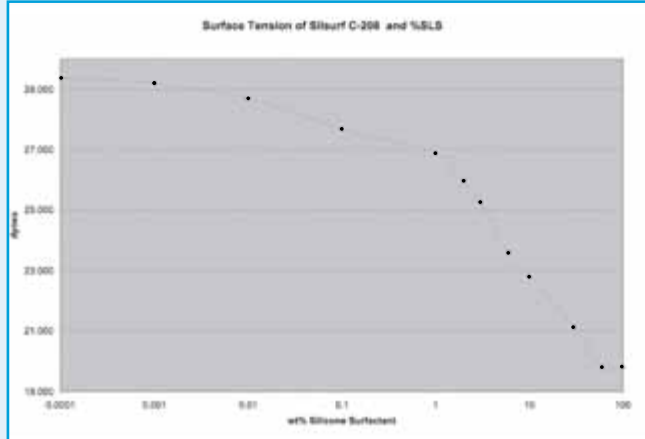


Table 8. Surface tension of Silsurf C-208 and % SLS (10).

Table 9 shows the effect of increasing the molecular weight of the silicone surfactant in its interaction with SLS and SLES-2. The low molecular weight polymer shows little difference in the RF-50 when added to either SLES-2 or SLS. We attribute that to the ability of the lower molecular weight product to get to the surface with minimal interaction. When the molecular weight is increased, the interaction becomes more important. In this instance the interaction of the higher molecular weight silicone surfactant and the ethoxylated sodium lauryl sulfate results in a higher RF-50, that is more silicone surfactant is needed to reach the 25 dynes/cm.

Silicone Surfactant	Fatty Surfactant	MW of Silicone Surfactant	RF-50
10-1	SLS	611	1.2
10-1	SLES-2	611	1.2
10-2	SLS	1398	1.5
10-2	SLES-2	1398	3.5

Table 9. The effect of differing molecular weight PEG-8 dimethicone polymers in two different fatty sulfate solutions.

Wetting

Wetting is a property of silicone polymers that is related to structure. Good wetting agents for water based systems must (a) be soluble in water; (b) lower surface tension and (c) must have a low enough hydrodynamic volume (size) to get to the interface. Properties of several PEG 8 dimethicone polymers are shown in Table 10.

Product	MW	Surface Tension	Wetting Time (second)
11-2	632	22.0	7
11-2	855	23.5	8
11-3	1398	27.3	10
11-4	2105	28.1	16
11-5	2706	28.4	27
11-6	6334	30.8	88

Table 10. PEG 8 Dimethicone Wetting Times = Draves Wetting (1% in DW) (11).

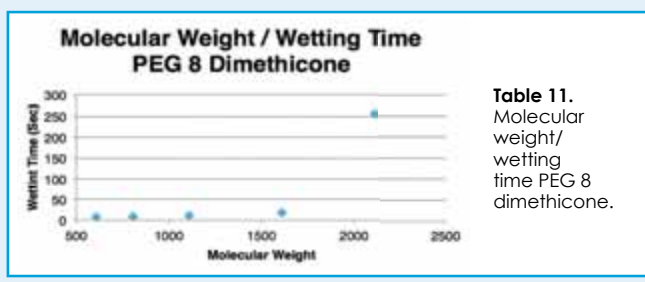


Table 11. Molecular weight/ wetting time PEG 8 dimethicone.

Formulation Tip

Remember that silicone polymers that get to the surface and lower the surface tension of the formulation have different properties. Some function at the air formulation interface and these generally contribute wetting, which in turn helps spreadability. Others function above the CMC and emulsify, and condition. Rarely does one specific polymer do both well. Always consider a blend of polymers with the same INCI and add them together for the label declaration.

Minimally Disruptive Formulation (12)²

We have begun using a concept we refer to as minimally disruptive formulation (MDF) as an effective approach to product development. This approach depends upon the ability of personal care formulators to provide products that have consumer perceptible differences that meet a market need. Since product aesthetics are a key attribute of personal care products, the ability to alter product aesthetics to provide a different consumer perception with minimal change to the formulation is a very cost effective way to develop new products.

The fact is that silicone polymers, properly chosen at a concentration of 10% or less, will provide to the formulation a lowering of surface tension, an alteration of feel, an altering of cushion and play time, a change in gloss, and a perception to a customer the product is different from the formulation to which the additive has not been made. This makes silicone polymers quite valuable at low concentrations on formulation to make 'new products'. I have often said that if a personal care product is compared to a gourmet meal, silicone additives will be the spice, not the meat or potatoes. This means that small amounts of silicone polymer added to great formulas will bring out desired properties to a consumer, that will amaze and delight. This approach will allow the formulator to make small but major modifications to formulations in a very efficient way by modifying well known formulations to provide new products with different aesthetics. Additions of a properly chosen PEG/PPG dimethicone polymers can be added using the guidelines given above to improve the efficiency of many products. We will discuss a 2 in 1 shampoo.

SELECTION OF SILICONE

Product: Clear Conditioning Shampoo

Based upon the data presented in this article, the silicone needs to be chosen based upon the desired function in the particular formulation. Since we are dealing with a conditioning shampoo, conditioning is certainly a priority and wetting is another, since it will allow for the increased spreadability on the hair and improved wet comb. For purposes of this formulation are we have chosen to work with PEG 8 dimethicone, but which one?

PEG-8 Dimethicone

Product 1 is a commonly available PEG-8 dimethicone. It would be the most likely product a formulator would get if the request was simply for PEG-8 dimethicone.

Product 2 is a higher molecular weight product, having the same D:D*. The higher molecular weight would lead to somewhat better deposition, but the product is not a micro emulsion.

Product 3 makes use of the formula to make a micro

emulsion product of Product 2. In order to make a micro emulsion for a silicone polymer having 20 "D" units (the "b" value or silicone soluble portion), requires 4 water soluble groups having 8 moles of EO.

Product 4 is a silicone wetting agent (low molecular weight).

Product	"a" Value(D*)	" b" Value (D)	D*:D	Description
1	4	8	1:2	Standard product
2	10	20	1:2	Higher molecular weight same D:D* Ratio
3	4	20	1:4	Higher molecular weight micro-emulsion D:D* ratio
4	2	2	1:1	Wetting agent

Table 12. 4 different PEG-8 dimethicone products.

	PHASE	INGREDIENT	A	B	C	D	E
1	A	WATER	48.60	48.60	48.60	48.60	48.60
2	A	DISODIUM EDTA	0.10	0.10	0.10	0.10	0.10
3	A	PRODUCT 1	2.00	-	-	-	-
3	A	PRODUCT 2	-	2.00	-	-	-
3	A	PRODUCT 3	-	-	2.00	-	-
3	A	PRODUCT 4	-	-	-	2.00	-
3	A	PRODUCT 3	-	-	-	-	1.5
3	A	PRODUCT 4	-	-	-	-	0.5
4	B	AMMONIUM LAURYL SULFATE	20.00	20.00	20.00	20.00	20.00
5	B	SODIUM LAURETH SULFATE	15.00	15.00	15.00	15.00	15.00
6	B	PEG-120 METHYL GLUCOSE DIOLEATE	1.50	1.50	1.50	1.50	1.50
7	C	COCAMIDOPROPYL BETAINE	7.00	7.00	7.00	7.00	7.00
8	D	COCAMIDE MEA	3.00	3.00	3.00	3.00	3.00
9	E	QUATERNIUM-15	1.50	1.50	1.50	1.50	1.50
10	F	RED#33	0.60	0.60	0.60	0.60	0.60
11	G	SODIUM CHLORIDE	0.50	0.50	0.50	0.50	0.50
12	H	CITRIC ACID					
		Total			100.00		

Table 13. Clear Gel 2:1 shampoo. Formulation used for evaluation.

Hair evaluation:

All products were evaluated on 10 inch Virgin Brown Hair. Two x 2gm. swatches were used for each material tested, all from the same lot. All swatches were wet with 25°C water and one gram of test material was used for each swatch. Swatches were washed and then rinsed for at least one minute per swatch. Wet Comb Evaluation was then performed. No blow drying of hair was done. All swatches air-dried then the Dry Comb Evaluation was performed once hair was completely dry. The addition of the PEG-8 dimethicone with the best wetting properties (product 4) improved the wet comb properties.

Property	Product A	Product B	Product C	Product D
Dry Comb	1	2	4	5
Feel	2	3	5	5
Fly Away	2	3	4	4
Static Control	2	3	5	5
Feel	2	2	4	4

Table 14. The wet comb properties. Scale used is 1 to 5, 5 being the best.

Property	Formula A	Formula B	Formula C	Formula D
Wet Comb	1	3	3	5
Clean Feel	5	4	4	4
Residual Feel	2	3	4	5

Table 15. he dry comb properties. Scale used is 1 to 5, 5 being the best.

The addition of the PEG-8 Dimethicone chosen for its ability to make a micro emulsion provided the best dry comb conditioning. Product D offered both the best wet comb and dry comb properties, using two different polymers both with the name PEG 8 dimethicone.

CONCLUSIONS

PEG/PPG dimethicone polymers are a diverse class of compounds, even though many of them have the same INCI name. This explains why copying a formulation from the list of INCI materials contained therein is often so frustrating.

As with any silicone polymer, always ask "Why use silicone", then determine which silicone offers the best properties in your formulation.

The availability of a variety of silicone polymers for use in personal care formulations is a double edge sword. While the ability to work with a large number of materials offers the ability to provide many alternate functional properties to formulations, it also will require a logic toward the evaluation and a testing protocol that makes such selection possible.

We recommend determining the meat and potatoes of the formulation first, that is to determine the chassis and then to begin adding the spice of the formulation, that is the silicone polymer.

1. The silicone polymer will be added to the phase in which the benefit is desired, Add water soluble silicone polymers to the aqueous phase, oil soluble silicone polymers to the oil phase or fluoro soluble silicone polymers to the fluoro based phase. Oil soluble silicone polymers are called alkyl dimethicone polymers, water soluble silicone polymers are called PEG / PPG dimethicone polymers and Fluor soluble ill ones are called fluoro silicones. There are many polymers in each family, the challenge is to pick the proper one based upon the desired function,
2. Make sure you pick a product that is clear in the formulation but if possible one that is capable of forming a micro emulsion in the formulation. Remember, if the silicone polymer is too soluble in the desired phase it will not deposit.
3. Pick the silicone by the function you desire, wetting agents

tend to have low molecular weight, aesthetic modifiers, emulsifier ad conditioning agrees tend to have higher molecule weight,.

4. Utilize minimally disruptive formulation techniques, replacing only the silicone and observing effects.
5. Remember there is a silicone polymer available to alter the property of each phase and using one or more in each phase at low concentrations can result in very unexpected properties.

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