High performing silicone microemulsion in hair care

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Today's hair care industry is a highly competitive and segmented market place wherein formulators need to respond to a range of hair types, cultures, regions and ethnicities. A savvy youth market, emerging economies and an expanding group of sophisticated consumers need to be accommodated.

To meet these requirements, the industry must continuously develop creative solutions. Specialty silicones are able to create hair care products that help to strengthen and protect hair from environmental stress, colour fade, heat associated damage and grooming effects. Elsewhere they are able to add volume, control hair frizziness and help repair split ends. Silicone plays a key role, whether in conditioners, shampoo, colourant or styling products.

Individualism in hair care

The concept of customised hair care products is gaining popularity as customers intend to portray their individuality and handle specific grooming challenges based on hair type and its desired style and condition.

Specialty silicones can provide specific solutions for individual needs based on customer's specific hair care requirements. For example, hair care products are extensively designed for fine, dry, kinky or curly hair, damaged or coloured hair, and styles from long to short, voluminous to sleek. The range of conditioning levels is from intense to light with alternatives for heat and colour protection as well as frizz control.

Specialty silicone for hair moisturisation

UV radiation, curling irons, and chemicals from colouring, perming and straightening – even frictional forces from daily combing and styling – all conspire to hair damage. In the case of coloured hair, regular washing can lead to discolouration. Ageing along with the changes of hormone and sebum production can lead to dry, dull and grey hair.

Hair moisturisation is one of the most popular claims in hair care products nowadays. In fact, silicone does not provide moisturisation to hair. But, when silicone is added to leave on or rinse off hair products, it gives the slippery texture that makes your hair feel it is being moisturised. The definition of hair moisturisation is different from skin moisturisation. Hair moisturisation is expressed by hair that is easy to comb when wet as well as dry, has healthy-looking shine, and fibres that align more easily to provide a sleek look.

With the aid of specialty silicones, it is possible to formulate hair care products that can improve hair appearance. For example, incorporating specialty silicones in shampoos and conditioners can improve shine and help maintain the colour-treated hair.

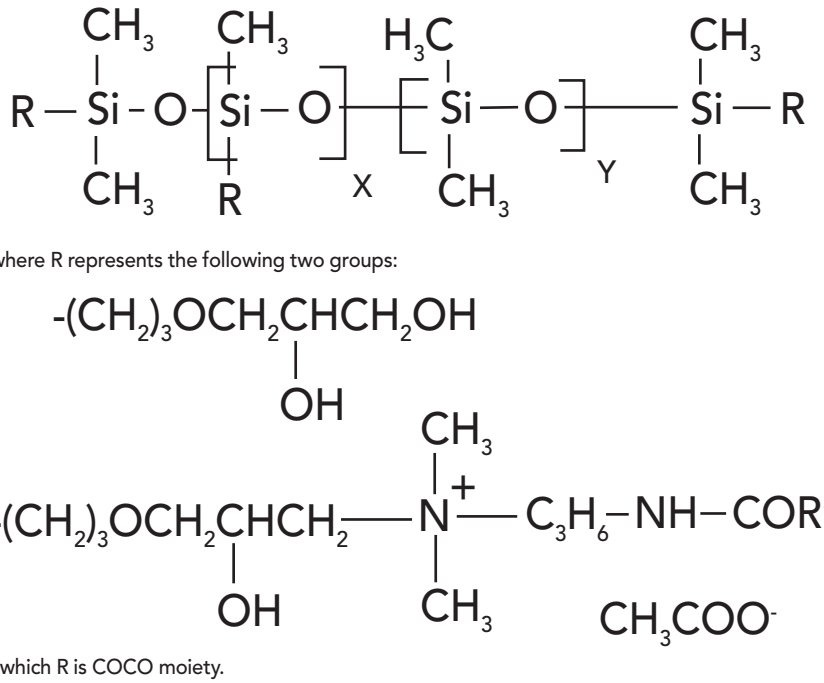

Figure 1: Molecular structure of Silicone Quaternium-17.

Silicone Quaternium-17 in microemulsion

High molecular weight silicones are well known for hair conditioning and protecting properties but remain a challenge to formulate successfully due to their hydrophobic nature and high viscosity. However, when delivered as emulsion, small droplets of the highest molecular weight silicone can be easily incorporated into hair care formulations. This is more convenient and saves on costs for manufacturing time (shorter) and manufacturing cost (energy and time saving) including productivity as well.

The development of thermodynamically stable microemulsions with very fine silicone droplets is very beneficial to formulators. As the droplets are smaller than the wavelength of visible light, they are usefully transparent. This approach, based on such fine droplets was used to develop a microemulsion based on high molecular weight Silicone Quaternium-17 (Fig 1).

The microemulsion A1 was then processed by emulsifying Silicone Quaternium-17 with emulsifier of Trideceth-

7 and Trideceth-5, delivering a product does not contain polyether. The active silicone content in the microemulsion is approximately 15%.

Meanwhile microemulsion B2 was developed with shine boosting Phenyl Trimethicone and, cationic polymer, Silicone Quaternium-17 together with emulsifier of Laureth-4 and Laureth-23. This system presents itself as a preservative and polyether free product. The active silicone content in the microemulsion is approximately 17%.

These new silicone microemulsions are simple to incorporate into hair care products with cold, low shear blending, delivering a good sensory effect, superior conditioning, plus excellent wet and dry combing properties. They are ideal for use in clear, leave-in and rinse-out products performing with positive effect on all hair types. Various tests were conducted to demonstrate the performance benefit of such microemulsions.

Performance testing

Combing force measurement

These studies show the significant conditioning performance benefits of silicone mircroemulsion A (INCI: Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5) and silicone microemulsion B (INCI: Phenyl Trimethicone (and) Laureth-4 (and) Silicone Quaternium-17 (and) Laureth-23) based on ease of combing in wet and dry conditions. Combing force measurement was taken before and after application of shampoo (Formula 1) and conditioner (Formula 2) using MTT175 tensile tester (Dia-Stron). Tests were conducted on virgin brown Caucasian hair tresses which had been pre-double bleached to ensure chemical substances were removed from hair before measurements. Results of combing force measurements are shown in Figure 2.

Both microemulsion A and microemulsion B significantly reduced the combing forces in wet and dry conditions after shampoo and conditioner application, compared to the control.

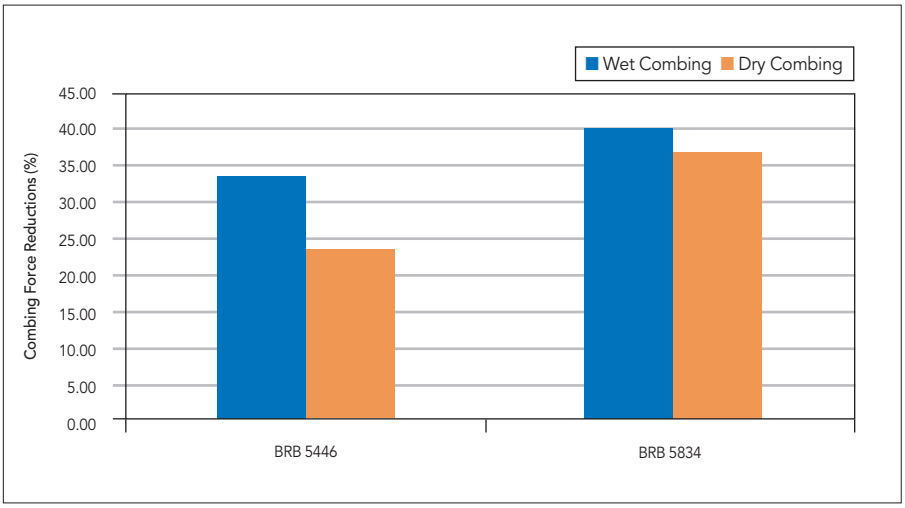

Figure 2: Ease of combing of wet & dry condition with Dia-Stron tensile tester.

$$\% \text{ Force reduction} = \frac{\text{TCW (untreated)} - \text{TCW (treated)}}{\text{TCW (untreated)}}$$

TCW = Total Combing Work (in joules)

Thermal protection

Thermal hair damage easily results from the frequent use of heat-based styling equipment, including hair blow-dryers, straightening irons and curling tongues. These heat treatments are conducted around 180-230°C, sufficient to thermally damage hair's keratinous structure. If the use of hot styling tools is done improperly, the moisture will evaporate from hair, destroying all its natural structure and chemistry. The oil and protein basis will be damaged leading to dull, brittle, dry and fragile hair. Therefore, thermal protection is essential in order to prevent heat damage. Specialty silicones can be used to protect hair from heat due to its film forming properties and low thermal conductivity.

After 15 Cycles of washing:
I: Color reference (not treated)
II: Control (no silicone)
III: Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5
IV: Non-silicone ingredients

Figure 4: Colour protection with silicone mircoemulsion.

Figure 3: Thermal protection with flat iron test.

Figure 5: Improve hair shininess with silicone microemulsion.

Tresses of virgin blonde Caucasian hair were pre-double bleached prior to performance testing. These hair tresses were treated with heat protecting spray, formulated with silicone microemulsion A (Formula 3) and followed by 50 cycles of flat iron at 230°C. Results are shown in Figure 3.

Figure 3 shows less damage in those tresses treated with silicone emulsion A compared to the untreated control. Silicone Quaternium-17 actively protects from heat damage, preventing cuticle cracking. The hair fibres require less force to detangle, break less when combing and look and feel healthier. A good result in thermal protection is achievable with the deposition of Silicone Quaternium-17 from a microemulsion format.

Colour protection

Modern consumers often use hair colourants with variable degrees of longevity in order to change or enhance their natural hair colour. These permanent hair dyes, also referred to as oxidative dyes, are peroxide based alkalising agents that swell the hair fibre, enabling the dye precursors and a catalysing agent to reach the cortex. It is observed that hair treated with red hues are particularly prone to fading after UV exposure and shampooing. Studies found that red coloured hair tresses loose colour to a larger extent compared to other dyes, such as black, brown and blond. Thus, colour fading of the hair dye, whilst a key performance characteristic of the dye formulation, is also a useful consideration when formulating targeted rinse off or leave on products.

The hair colour retention test was conducted on virgin blond Caucasian hair tresses. The tresses were treated with commercial red colour dye. Then the tresses were washed with shampoo (Formula 4) followed by rinse off conditioner (Formula 5). The tresses were washed in 15 cycles. Observation and measurements were recorded at an interval of 5, 10, 15 washes. Hair Tress I is the untreated swatch, directly after dying; hair tresses II was treated 15 times with control shampoo and conditioner; hair tress III was treated 15 times with shampoo and conditioner using silicone microemulsion A; hair tress IV was treated 15 times with shampoo and conditioner using non-silicone raw-materials.

The results in Figure 4 demonstrate a clear improvement in colour fading on hair tresses treated with silicone microemulsion A after 15 cycles of washing. Silicone microemulsion maintains colour vibrancy by forming a hydrophobic surface on hair.

Hair shininess

Physical measurements show that phenyl group substituted silicones can significantly increase shine on hair. In particular, Phenyl Trimethicone is recognised for its ability to enhance hair shine and gloss along with

Formulation 1: Shampoo for Combing Force Measurement

Phase	Ingredients	INCI	A (%)	B (%)
A1	DI Water	Aqua	To 100	To 100
A2	Tetrasodium EDTA	Tetrasodium EDTA	0.10	0.10
A3	Viscolam CK1	Acrylates Copolymer	3.00	3.00
A4	SLES 70%	Sodium Laureth Sulfate	15.00	15.00
A5	TEA 99%	Triethanolamine	q.s.	q.s.
B1	Amido Betaine C	Cocamidopropyl Betaine	10.00	10.00
B2	Comperlan CDE	Cocamide DEA	2.00	2.00
C1	DMDMH	DMDM Hydantoin	0.10	0.10
C2	Salt	Sodium Chloride	1.00	1.00
C3	BRB 5446	Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5	8.00 *(1.2% Si active)*	–
C4	BRB 5834	Phenyl Trimethicone (and) Laureth-4 (and) Silicone Quaternium-17 (and) Laureth-23	–	7.06 *(1.2% Si active)*

Procedure:

Add A1-A4 one by one into bulk. Mix well after each addition. Adjust pH to 6.5 using A5. Add B1 & B2 into bulk and mix well. Add C1 – C4 into bulk and mix well.

Formulation 2: Conditioner for Combing Force Measurement

Phase	Ingredients	INCI	A (%)	B (%)
A1	DI Water	Aqua	To 100	To 100
A2	Glycerin	Glycerin	2.00	2.00
A3	Tylose HS 100,000	Hydroxthethylcellulose	1.10	1.10
B1	Euxyl PE 9010	Phenoxyethanol (and) Ethylhexylglycerin	0.80	0.80
B2	Citric Acid	Citric Acid	q.s.	q.s.
B3	BRB 5446	Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5	8.00 *(1.2% Si active)*	–
B4	BRB 5834	Phenyl Trimethicone (and) Laureth-4 (and) Silicone Quaternium-17 (and) Laureth-23	–	7.06 *(1.2% Si active)*

Procedure:

Add A1-A3 one by one into bulk. Mix well after each addition. Add B1 into bulk. Mix well. Add B2 into bulk to adjust pH to 4-5. Add B3 & B4 into bulk. Mix well.

Formulation 3: Heat Protecting Spray

Phase	Ingredients	INCI	(%)
A1	DI Water	Aqua	To 100
A2	Alcohol denat.	Alcohol Denat.	20.00
A3	Palmera G995E	Glycerin	2.00
A4	Propylene glycol	Propylene Glycol	2.00
A5	Natura-Tec Aminosens Keratin Sol 25%	Aqua (and) Hydrolyzed Keratin	2.00
A6	BRB 1288	Amodimethicone (and) Trideceth-12 (and) Cetrimonium chloride	1.50
A7	BRB 5446	Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5	2.00

Procedure:

Add one by one all of the ingredients of phase A to water whilst stirring until obtaining a homogenous phase. Check final pH.

adding softness, manageability and smoothness to the abraded hair cuticle.[5] Benefits of Phenyl Trimethicone include improved organic compatibility, shine and gloss, spreadability and reduced tackiness.

Virgin blonde Caucasian hair tresses were pre-double bleached before performance testing. The hair tresses were treated with 'whipped cream conditioner' formulated with silicone microemulsion B (Formula 6). Hair tresses I treated with control 'whipped cream conditioner' without silicone microemulsion; hair tresses II were treated with 'whipped cream conditioner' formulated with 3% silicone mircroemulsion B.

The results in Figure 5 clearly indicated that hair tresses II, treated with silicone microemulsion, provide notably higher gloss levels. The shine of the hair is much improved demonstrating healthy-looking hair, a common claim of many hair care products.

Application

The silicone microemulsions A and B are low viscosity, pourable liquids and therefore very easy to incorporate into hair care formulations. They are fully soluble in water, cold processable and can be added into formulation at the end of process with gentle mixing. They are particularly recommended for use in clear rinse off or leave on hair care products.

Conclusion

Fast developing, global cosmetic trends help create a growing demand for high performance hair care products offering the multifunctional, claimable effects. Functionally modified silicone microemulsions are raw materials specially developed in response to meet this demand for customized hair conditioning treatments.

Both silicone microemulsion A (Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5) and silicone microemulsion B (Phenyl Trimethicone (and) Laureth-4 (and) Silicone Quaternium-17 (and) Laureth-23) are multifunctional hair conditioning agents, delivering measurable improvements, providing hair with an enhanced natural look and feel. PC

References

1. Silicone Emulsion A: Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5
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4. Boonme P, Pakpayat N, Yotmanee K, Kunlawijitrungsee S, Maneenuan D. Evaluation of Shampoos Containing Silicone Quaternary Microemulsion. *Journal of Applied Pharmaceutical Science* 2011; **01** (01): 59—63
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Formulation 4: Shampoo for Colour Protection Test

Phase	Ingredients	INCI	II (%)	III(%)	IV (%)
A1	DI Water	Aqua	To 100	To 100	To 100
A2	Iselux-LQ-CLR	Sodium Lauroyl Methyl Isethionate	30.00	30.00	30.00
B1	Amido Betaine C	Cocamidopropyl Betaine	15.00	15.00	15.00
B2	Corum 6130	Disodium Laureth Sulfosuccinate	3.00	3.00	3.00
B3	Euxyl PE 9010	Phenoxyethanol (and) Ethylhexylglycerin	1.00	1.00	1.00
B4	BRB 5446	Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5	–	6.67 (1.0% Si active)	–
B5	Polyquaternium-11	Polyquaternium-11	–		1.00
B6	Citric acid	Citric Acid	q.s.	q.s.	q.s.

Procedure:

Add A1 & A2 into bulk. Mix well. Add B1-B5 one by one into bulk. Mix well after each addition. Add B6 to adjust pH to 5.0-6.0.

Formulation 5: Conditioner for Colour Protection Test

Phase	Ingredients	INCI	II (%)	III(%)	IV (%)
A1	DI Water	Aqua	To 100	To 100	To 100
A2	Glycerin	Glycerin	3.00	3.00	3.00
A3	Tylose HS 100000	Hydroxyethylcellulose	1.00	1.00	1.00
B1	Cetearyl Alcohol	Cetearyl Alcohol	4.00	4.00	4.00
B2	Tewax TC-65	Glyceryl Stearate (and) PEG-100 Stearate	1.00	1.00	1.00
C1	BRB 5446	Silicone Quaternium-17 (and) Trideceth-7 (and) Trideceth-5	–	*6.67 (1.0% Si active)*	–
C2	Polyquaternium-11	Polyquaternium-11	–	–	1.00
C3	Euxyl PE 9010	Phenoxyethanol (and) Ethylhexylglycerin	1.00	1.00	1.00
C4	Citric Acid	Citric Acid	q.s.	q.s.	q.s.

Procedure:

Add phase A into tank and mix well. Heat to 70-75°C. In a separate beaker heat phase B to 70-75°C. Add step 2 into step 1. Homogenize well. Cool bulk to 45°C. Add C1-C3 one by one into bulk. Mix well after each addition. Adjust pH to 3.5-4.5 with C4.

Formulation 6: Whipped Cream for Hair Shininess Test

Phase	Ingredients	INCI	I (%)	II(%)
A1	Tego Alkonol 1618	Cetyl Alcohol	1.00	1.00
A2	Stearic acid	Stearic Acid	0.50	0.50
A3	Tego Alkanol L 4	Laureth-4	1.00	1.00
B1	DI Water	Aqua	To 100	To 100
B2	Genamin KDMP	Behentrimonium Chloride	0.30	0.30
B3	Tequart Cetac 29	Cetrimonium Chloride	3.00	3.00
B4	Propylene Glycol	Propylene Glycol	1.00	1.00
C1	BRB 5834	Phenyl Trimethicone (and) Silicone Quaternium-17 (and) Laureth-4 (and) Laureth-23	–	3.00
C2	Dhyton PK 45	Cocoamidopropyl Betaine	1.00	1.00
C3	Aminoacid Complex	Aminoacid Complex	1.00	1.00
D1	Euxyl PE 9010	Phenoxyethanol (and) Ethylhexylglycerin	0.40	0.40

Procedure:

Heat and mix A1-A3 to 80-82°C. In a separate beaker, heat B1-B2-B4 to 80-82°C and then added B3. Add step 1 into step 2. Homogenize well. Cool bulk to 40°C. Add C1-C3 into bulk one by one. Mix well after each addition. Add D1 into bulk. Mix well. Checked pH.