Polymer interactions in formulations

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ABSTRACT

The development of efficient cosmetic formulations is a complicated process. Since all of the ingredients in the formulation interact with each other, the careful evaluation of the complete formulation is critical and often difficult. One of the more interesting effects that can be achieved by blending silicone polymers is the formation that is both rub and transfer resistant film by blending two different polymers together. The entanglement of the two different polymers results in a film with both properties.

FORMULATOR TIP

The preparation of a good formulation always includes taking advantage of interactions between ingredients in the formulation. Always look for interactions, not only are they the basis of superior formulations, they are the creative step in many patents.

This article will investigate an example of synergistic interactions. In this case the two ingredients are a linear polyurethane polymer and a Q resin. These materials together in a formulation that contains a volatile solvent interact and form a network which provide both transfer resistance and rub resistance in formulations.

FILM FORMATION

Reactive mixtures

The concept of entanglement is an important on in the generation of films. There are two kinds of film formation mechanisms that are used in cosmetic formulations. The first is a blend of two reactants that react with each other chemically and provide a film. An example is a silanic hydrogen (Si-H) compound reacting with a vinyl compound (CH_2=CH-.) This reaction has become important in the formation of a film on the skin that can be peeled off. Two reactive phases are mixed and react on the skin to form this type of film (1-4). Figure 1 shows the reaction scheme for the hydrosilylation reaction.

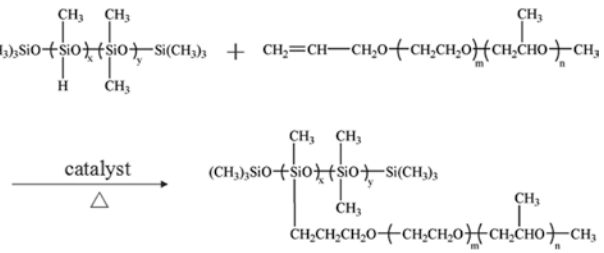

Figure 1. Hydrosilylation Reaction.

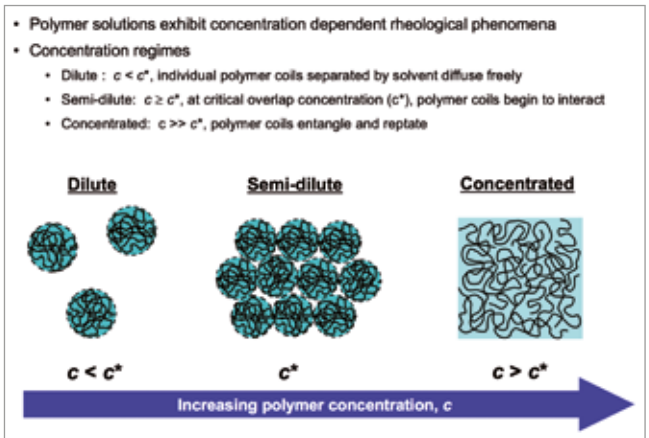

Figure 2. Polymer solution concentration regimes (5).

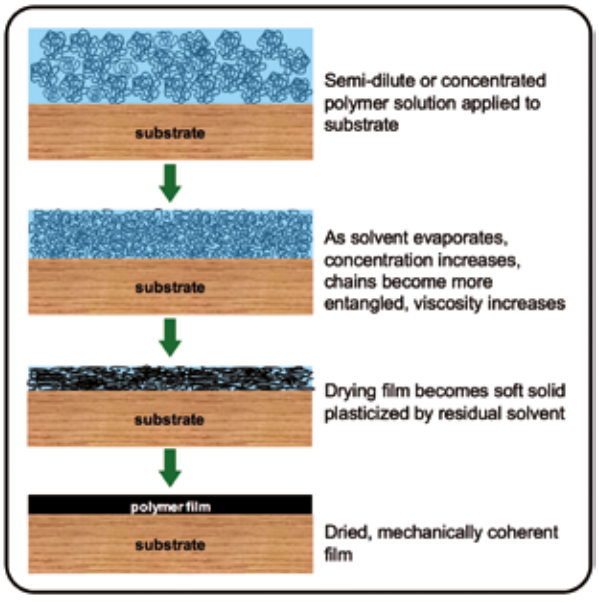

Figure 3. Solution-borne polymer film formation (6).

Concentration of Entanglement
The second mechanism of film formation is the result not of a reaction, but of the evaporation of the solvent. As the solvent evaporates the polymer chains entangle in each other and form an entangled mesh. Figure 2 shows what happens as the concentration of polymer is increased from dilute, to semi dilute to a concentration that exceeds the concentration of entanglement. The concentration increase can be caused by evaporation of the solvent. The resulting mass is made up on entangled polymers that have a film integrity due to the entanglement.

Figure 3 shows the progression of a film formed by solvent evaporation on a substrate.

The nature of the polymers that become entangled has a profound effect upon the properties of the film.

Formulator Tip
Keep in mind that since entanglement is not a reaction, various concentrations of one to the other need to be evaluated. Reactions require stoichiometric concentrations.

Formulator Tip
The formulator must keep in mind that there are several different times in the use of a formulation that stability, aesthetics and performance need to be evaluated. Firstly, when the initial formulation is made, secondly, over time in the bottle, thirdly when delivered to the skin, and finally when the film has formed. All are critical and many times a compromise needs to be made to get the most "nearly perfect" performance is all the evaluations.

POLYMERS EVALUATED

Silicone Elastomer (7)
Silmer G162 is 13% Dimethicone Vinyl Dimethicone Crosspolymer in D5.

Urethane polymer
Silmer UR5050 is 45% Bis Hydroxypropyl Dimethicone/ SMDI Copolymer 45% in isododecane.

Q Resins (8)
Silmer Q9 is 70% Trimethylsiloxysilicate in Isododecane.

The silicone urethane polymer is linear; the silicone elastomer is principally linear therefore when mixed together and after solvent evaporation were not expected to provide both rub resistance and transfer resistance. Rub resistance was thought to require additional entanglement. In order to improve entanglement a Q resin which is primarily crystalline was added.

Method of Evaluation
To achieve a polymer film that has both rub resistant and transfer resistance, a series of blends were made using a silicone elastomer, silicone urethane and an MQ resin.

SCREENING SIMPLE BLENDS FOR TRANSFER RESISTANCE

A. Blend of Bis Hydroxypropyl Dimethicone/ SMDI Copolymer. and Dimethicone Vinyl Dimethicone Crosspolymer

Bis Hydroxypropyl Dimethicone/ SMDI Copolymer.	Dimethicone Vinyl Dimethicone Crosspolymer
1*	1*
2	1
3	1
4	1
1	4
1	3
1	2

*ratio was measured by weight of product as described.

2 g of each blend above was weighted in aluminum tray respectively and then they were put in oven over 24 hours (at 105 °C) and after dried down all the films formed by the above blends were all sticky.

B. Blends of Bis Hydroxypropyl Dimethicone/ SMDI Copolymer and Trimethylsiloxysilicate

Blend	Bis Hydroxypropyl Dimethicone/SMDI Copolymer	Trimethylsiloxysilicate
A	1**	1**
B	2	1
C	3	1
D	1	2
E	1	2.5
F	1	3

*ratio was measured by weight

2 g of each of the above blends was weighted in aluminum tray respectively and then they were put in oven over 24 hours (at 105 °C), after dried down the films of the blends A/B/C were all sticky. The blend D was little bit tacky, blend E was smooth, blend F was brittle, therefore only blend E with BIS HYDROXYPROPYL DIMETHICONE/SMDI COPOLYMER / TRIMETHYLSILOXYSILICATE = 1:2.5 was chosen to be tested in color cosmetic finished products.

FORMULATION 1 - EYE STAY

Ingredients	FM630Control w/Isododecane	FM630A w/Blend E
Part A	Wt/Wt%	Wt/Wt%
Bentone Gel VS-5 PC V	38.00	38.00
Isododecane	**31**	**0**
Bis Hydroxypropyl Dimethicone/ SMDI Copolymer/ TRIMETHYLSILOXYSILICATE (1:2.5)	**0**	**31**
Pentaerytherityl Tetraisostearate	5.00	5.00
Phenonip	0.80	0.80
Bentone 38V	5.00	5.00
Silmer G162	10.00	10.00
Silquat J2-8B	5.60	5.60
Part B		
IRIS91-TRI-77891 (White)	1.20	1.20
IRIS-Y-77492 (Yellow)	0.10	0.10
IRIS-R-77491 (Red)	0.20	0.20
IRIS-B-77499 (Black)	0.10	0.10
Talc	3.00	3.00
Timiron Splendid Violet	2.20	2.20
Colorona Camine Red	4.97	4.97
Timiron Splendid Blue	2.28	2.28
D&C Red No.28 Al Lake	0.10	0.10
Total	100.00	100.00

Procedure:

1. Combine all the ingredients of Part A in a clean and sanitized container, mix well.
2. Add ingredients of Part B one by one into Part A under mixing.
3. Check pigment dispersion and keep mixing until uniform
4. Pour into mold.

Analysis of Eye Stay

Analysis	FM630 Control w/Isododecane	FM630A w/Blend E
S.G.	0.916	0.920
pH	7.00	7.00
Appearance	Pink Cream/Gum	Pink Cream/Gum
Stability @ RT/45C	Good/Good	Good/Good
Feel (1-10, 10 the best)	9.0	9.1
Compatibility	Good	Good

FORMULATION 2 – LIPSTICK

Ingredients	FL515 Control w/Isododecane	FM515A w/Blend E
Part A	**w/w%**	**w/w%**
Isododecane	**62.00**	**26.00**
Bis Hydroxypropyl Dimethicone/SMDI Copolymer/ TRIMETHYLSILOXYSILICATE (1:2.5)	**0**	**36.00**
Part B		
Cetyl Alcohol	2.69	2.69
Ozokerite	9.73	9.73
Candelilla Wax	8.91	8.91
Carnauba Wax	6.10	6.10
Part C		
D&C Red 28	1.71	1.71
Brown Iron Oxide	2.00	2.00
Part D		
Colorona Red Gold	0.73	0.73
Colorona Camine Red	0.51	0.51
BPD-500	3.16	3.16
DC 9701	2.46	2.46
Total	**100.00**	**100.00**

Procedure:

Into a cleaned and sanitized vessel equipped with a propeller mixer, add all the ingredients of Part A+B, then heat up to 80 ~ 85 °C. Once melted, add Part C, blend well and check dispersion. Pour into mold at 70 °C.

Analysis of Lipstick

Analysis	FL515 Control w/Isododecane	FM515A w/Blend E
S.G.	0.906	0.912
Appearance	Penny Candy bullet-like	Penny Candy bullet-like
Melt Point	43-51	46-55
Hardness	50	54
Stability @RT/45 °C	Good/good	Good/good
Feel (1-10, 10 the best)	8.8	9.0
Compatibility	Good	Good

Lipstick with isododecane is too soft, lipstick with Blend E is good.

FORMULATION 3 – LIPGLOSS

Ingredients	FG110 Control w/Isododecane	FG110A w/Blend E
Part A	**W/W%**	**W/W%**
Polyisobutene	10.00	10.00
Silwax D0-MS	5.00	5.00
Bentone Gel ISD-V	27.86	27.86
Bentone 38V	6.94	6.94
D5	1.49	1.49
Pentaerytherityl Tetraisostearate	5.00	5.00
Isododecane	**32.29**	**0**
Bis Hydroxypropyl Dimethicone/ SMDI Copolymer/TRIMETHYLSILOXYSILICATE (1:2.5)	**0**	**32.29**
Silquat J2-8B	5.74	5.74
Part B		
Talc	3.00	3.00
Suncroma D&C Red 28 Al Lake	0.15	0.15
Gemtone Tan Opal G005	0.20	0.20
Colorona Carmine Red	1.37	1.37
Part C		
Citric Acid	0.10	0.10
Vitamin C Palmitate	0.05	0.05
Microcare OHB	0.10	0.10
Tinogard TT	0.10	0.10
Tinogard AS	0.10	0.10
Total	100.00	100.00

Procedure:

Combine Part A and heat up to 80 °C; keep mixing for 10 minutes until homogeneous. Add Part B under mixing and then cool down to 50 °C, slowly add Part C under mixing, keep mixing for another 10 minutes, cool down to room temperature.

Analysis of Lipgloss

Analysis	FG110 Control w/Isododecane	FG110A w/Blend E
S.G.	0.918	0.926
Gloss*	68.1	70.1
Appearance	Smooth Shiny semi-solid	Smooth Shiny solid
Melting Point	40-43	41-44
Stability @RT/45 °C	Stable/stable	Stable/stable
Feel (1-10, 10 the best)	8.9	9.0
Compatibility	Good	Good

*Gloss was tested by a BYK Micro-gloss (60°) Gloss-meter. The maximum value is 100

FORMULATION 4 - MASCARA (W/O)

Ingredients	FM625.1 Control w/Isododecane	FM625.1A w/Blend E
Part A	W/W%	W/W%
D.I. Water	14.89	14.89
Disodium EDTA	0.10	0.10
Potassium Sorbate	0.20	0.20
Methyl Paraben	0.10	0.10
Part B		
Nylon-12	2.50	2.50
IRIS91-B-77499	4.76	4.76
Brown Iron Oxide	4.04	4.04
Isododecane	**36.70**	**0**

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The general theme of the conference focusses on defining and maintaining healthy skin and safe-guarding against premature skin ageing. The effects of environment al insults, genetics and the benefits of active skin care products and nutrition will be discussed.

2018 PROGRAMME

Tuesday 5 June

Session 1: The science of skin health and skin ageing

Keynote: Ten years older but can we prevent ageing?
Professor Christopher Griffiths, University of Manchester, UK

The role of vitamin D in skin protection
Dr Bianca McCarthy, University of Sydney, Australia

Life style and youthful looks
David Gunn, Unilever R&D, UK

Keynote: The molecular anatomy of dry and photodamaged facial stratum corneum
Prof Anthony Rawlings, AVR Consulting, UK

Keynote: Mapping skin ageing
Dr Frédéric Flament, L'Oréal Paris, France

Session 2: Performance testing for innovative claims

Development, validation and application of an image analysis method for in vivo measurement of skin visual roughness
Di Qu, Ph.D., Amway R&D, USA

What is an Anti-Polluageing Claim and how can it be supported?
Dr Stephan Bielfeldt, ProDERM Munich, Germany

Cutaneous ageing due to environmental pollutants
Jessen Curpen, CIDP, Mauritius

Keynote: The Democracy and Diversity of Ageing in Our Global Village
Prof Paul Matts, Procter and Gamble, UK

Wednesday 6 June

Session 3: Creating effective skin care for healthy younger looking skin

Optimizing anti-aging efficacy using a rotational treatment regimen
Lisa DiNatale, Avon, USA

Network Pharmacognosy™ - accelerating discovery of natural active ingredients for younger looking skin
Susanne Fabre PhD, Oriflame Cosmetics, Sweden

The importance of touch in skincare
Katerina Steventon, Independent Skincare Consultancy Ltd, UK

Session 4: Functional skin care and physiological effects: the regulatory challenges and interfaces

Regulatory control of Anti-ageing skin care claims
Speaker to be confirmed

Pick your wavelength! Interactions between EMR and skin ageing
Karl Lintner, KALidees, France

Third Wave Skincare: Restoring the Microbiome
Kit Wallen-Russell & Nick Wallen, JooMo, UK

An innovative satellite-based technology for the prevention of skin photoageing
Emilio Simeone, siHealth, UK

Skin & Ageing: How will things look in 2028?
Steve Barton, Skin Thinking, UK

Scientific programme organiser:
Jack Ferguson, Skinnovation, UK

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Bis Hydroxypropyl Dimethicone/ SMDI Copolymer/ TRIMETHYLSILOXYSILICATE (1:2.5)	**0**	**36.70**
Silquat J2-8B	7.00	7.00
Silmer G162	7.00	7.00
Silube 316	3.00	3.00
Part C		
Carnuba Wax	6.00	6.00
Candelilla Wax	6.00	6.00
Ozokerite Wax	2.60	2.60
Beeswax	3.50	3.50
Propyl paraben	0.10	0.10
Part D		
Ammonium Hydroxide (29% Ammonia)	0.56	0.56
Part E		
Phenonip	0.65	0.65
Aloe Vera Oil	0.10	0.10
Ginseng Extract	0.10	0.10
Green Tea Extract	0.10	0.10
Total	100.00	100.00

Procedure:
1. Combine Part A and heat up to 75~80 °C.
2. Combine Part B and heat up to 75 ~ 80 °C, blend well.
3. Add part A into Part B at 75 ~ 80 °C under mixing, keep the temperature for 5 minutes, then cool down to room temperature.
Add part C, Part D and Part E one by one under mixing.

Analysis for Mascara

Analysis	FM625.1 Control w/Isododecane	FM625.1A w/Blend E
S.G.	0.944	0.956
pH	6.91	6.89
Appearance	Black brown Cream	Black brown Cream
Stability @ RT/45C	Stable/stable	Stable/stable
Feel (1-10, 10 the best)	8.8	9.0
Compatibility	Good	Good

FORMULATION 5 – FOUNDATION

Ingredients	FM628Control w/Isododecane	FM628A w/Blend E
Part A	W/W%	W/W%
D.I. Water	46.43	46.43
Glycerine	3.00	3.00
Sodium Chloride	1.00	1.00
Part B		
Silube 316	5.00	5.00
Isododecane	**31.00**	**0**
Bis Hydroxypropyl Dimethicone/ SMDI Copolymer/ TRIMETHYLSILOXYSILICATE (1:2.5)	**0**	**31.00**
Argan Oil	2.00	2.00
Siltech CE-2000	2.98	2.98
Unipure White LC981 AS	3.68	3.68
Unipure Yellow LC182 AS	0.74	0.74
Unipure Red LC381 AS	0.22	0.22
Unipure Black LC989 AS	0.07	0.07
Intermer IPA 13-6 Polymer	1.20	1.20
Excel Mica JP-2	1.48	1.48
Bentone 38V	1.20	1.20
Phenonip	q.s.	q.s.
Total	**100.00**	**100.00**

Procedure: Combine Part A and mix well, heat up to 75~80 °C. Combine Part B and blend well then heat up to 75~80 °C. Add Part A into Part B while agitating quickly to obtain a homogeneous mixture; continue to mix for 10 minutes. Cool batch down to room temperature.

Analysis of W/O Foundation

Analysis	FM628 w/Isododecane	FM628A w/Blend E
Viscosity (cps)	28,000	42,000
pH	5.70	5.70
Appearance	Ivory Makeup	Ivory Makeup
Stability @RT/45°C	Good/good	Good/good
Feel (1-10, 10 the best)	9.0	9.1
Compatibility	Good	Good

FORMULATION 6 – SUNSCREEN

Part ID	Ingredients	FS423Control w/Isododecane	FS423A w/Blend E
A		**Wt/Wt %**	**Wt/Wt %**
	D.I. Water	54.30	54.30
	Propylene Glycol	5.0	5.0
	NaCl	1.0	1.0
	Na_2EDTA	0.10	0.10
B	**Isododecane**	**32.00**	**0**
	Bis Hydroxypropyl Dimethicone/SMDI Copolymer/TRIMETHYLSILOXYSILICATE (1:2.5)	**0**	**32.00**
	Silmer TTRIMETHYLSILOXYSILICATE-30T	**0**	**0**
	Silmer TTRIMETHYLSILOXYSILICATE-40T	**0**	**0**
	Silube 316	6.00	6.00
	$C_{12\text{-}15}$ Alkyl Benzoate	5.00	5.00
	$C_{10\text{-}30}$ Alkyl Acrylate	3.50	3.50
	Titanium Dioxide	2.00	2.00
	Octyl Methoxycinnamate	7.00	7.00
	Benzophenone-3	5.00	5.00
	Avobenzone	2.00	2.00
	Phenonip	0.60	0.60
C	Fragrance	q.s.	q.s.
Total		100%	100%

Procedure: 1. Combine ingredients in Part B and heat up to 70°C, blend well using Bamix mixer until homogeneous, then heat up to 75 ~ 80 °C. 2. Combine Part A mix well and then heat up to 75 ~ 80 °C. 3. Add Part A into Part B slowly under mixing, keep mixing for 5 minutes after finish addition, then cool down to 45 °C and high shear for 1 minute at 20,000 rpm. 4. Add Part C into batch and mix well.

Analysis of W/O Sunscreen

Analysis	FS423 w/Isododecane	FS423A w/Blend E
Viscosity (cps)	16,000	21,000
pH	6.20	6.20
Appearance	White Cream	White Cream
Stability @ RT/43°C	Good/Good	Good/Good
Feel (1-10, 10 the best)	9.0	9.1
Compatibility	Good	Good

FORMULATION 7 - CONCEALER

Ingredients	FM629 Control w/Isododecane	FM629A w/Blend E
Part A	W/W%	W/W%
Silmer Q20	3.69	3.69
Silwax D02	0.92	0.92
Lanolin Oil	0.65	0.65
Silwax J219M	4.61	4.61
Isododecane	**31.52**	**0**
Bis Hydroxypropyl Dimethicone/SMDI Copolymer/TRIMETHYLSILOXYSILICATE (1:2.5)	**0**	**31.52**
Silmer TTRIMETHYLSILOXYSILICATE-30T	**0**	**0**
Silmer TTRIMETHYLSILOXYSILICATE-40T	**0**	**0**
Siltech F100	1.00	1.00
Hydrogenated Polyisobutene	2.02	2.02
Dicaprylyl Ether	3.27	3.27
White Ozokerite 164/170	3.41	3.41
Beeswax	10.14	10.14
Microcrystalline Wax	9.22	9.22
Aloe Vera Oil	0.10	0.10
Part B		
TiO2	21.03	21.03
Yellow Iron Oxide	2.95	2.95
Red Iron Oxide	0.77	0.77
KMP-590	4.61	4.61
Part C		
VE Acetate	0.04	0.04
BHT	0.05	0.05
Total	**100.00**	**100.00**

Procedure:
Combine Part A mix well then heat up to 75 °C under mixing. Add Part B, keep mixing for 10 minutes and then blend well until homogeneous, check pigment dispersion until no particles can be seen from two glass slides. Then cool down to 50 °C, slowly add Part C under mixing, keep mixing for another 5 minutes, pour batch into jars.

Analysis of Concealers

Analysis	FM629 Control w/Isododecane	FM629A w/Blend E
S.G.	0.925	0.931
pH	6.71	6.72
Appearance	Warm Beige Paste	Warm Beige Paste
Stability @ RT	Good	Good
Stability @ 45 °C	Good	Good
Feel (1-10, 10 the best)	8.9	9.0
Compatibility	Good	Good

TEST METHOD FOR NON-TRANSFER PROPERTY TEST FOR PERSONAL CARE PRODUCTS

Method: (Adapted from US Patent 20120276034A1)
A. 1 mm thin uniform film of color cosmetic product was applied on a 2×3 inch leather sheet, the cosmetic samples on the leather sheet were allowed to rest at ambient conditions for about one hour. Using a pipette, drop three drops of vegetable oil onto samples located on the right side of the leather sheet. Using another pipet, drop three drops of water onto the left side of the leather sheet.
B. Samples on the right side are used to determine the oil transfer resistance while samples on the left side are used to determine the water transfer resistance of the sample cosmetic composition.
C. Separately for the oil and water sections, distribute the oil or water evenly over the surface of each cosmetic film sample using cosmetic brush applicators, brushing lightly.
D. Allow the solvent to remain on the film undisturbed for about 15 minutes.
E. Using a lint-free wiper, carefully blot excess solvent from the

surface of each cosmetic film sample. Apply as little pressure as possible during this step.
F. Cut two disks from a clean, white Styrofoam dinner plate using a 1.0 inch diameter circular punch. The surface and edges of each disk should be smooth and even.
G. Firmly attach with double-sided adhesive tape each disk from step (F) to the bottom surface of a 2 lbs weight.
H. Set the weight on top of the cosmetic samples applied to the leather surface from step (A) above so that a first disk is in contact with the oil section of the film (i.e., the right side of the leather surface) and a second disk is in contact with the water section of the film (i.e., the left side of the leather surface). It is important to position the weight gently so that excess force beyond 2 lbs is not applied.
I. Grasping the top of the 2 lbs weight, carefully rotate each disk through 360 degrees while maintaining the 2 lbs force on the film. Do not lift or press the weight into the film during the rotating motion to the weight. The entire 360 degree rotation is preferably completed within a time interval between 3 and 5 seconds.
J. Lift the weight straight up off the film surface and carefully remove the disk from the weight avoiding damage to the disk.
K. Color transfer on each disk is visually assessed. The negative control using control samples (with Isododecane), which means it has minimum non-transfer property. The scale used is from 1 to 10 and 10 is the best, which means totally non-transfer.

CONCLUSIONS

1. Blending BIS HYDROXYPROPYL DIMETHICONE/SMDI COPOLYMER and TRIMETHYLSILOXYSILICATE results in a considerable improvement of the formulation in terms of rub resistance and transfer resistance.
The suggested ratio of BIS HYDROXYPROPYL DIMETHICONE/SMDI COPOLYMER at 1 part as provided to 2.5 parts of TRIMETHYLSILOXYSILICATE.

Evaluation of Long Wearing/Transfer Resistance for Cosmetics Products

Sample	Water Transfer Resistance	Oil Transfer Resistance
Eye Stay w/Isododecane	4.5	2.5
Eye Stay w/ BIS HYDROXYPROPYL DIMETHICONE/SMDI COPOLYMER /TRIMETHYLSILOXYSILICATE (1:2.5)	9.0	8.5
Lipstick w/Isododecane	5.0	3.5
Lipstick w/BIS HYDROXYPROPYL DIMETHICONE/SMDI COPOLYMER /TRIMETHYLSILOXYSILICATE (1:2.5)	8.5	8.0
Lipgloss w/Isododecane	5.5	4.5
Lipgloss w/UR 5050/TRIMETHYLSILOXYSILICATE (1:2.5)	8.5	8.0
Foundation w/Isododecane	4.5	4.0
Foundation w/UR 5050/TRIMETHYLSILOXYSILICATE (1:2.5)	8.5	8.0
Mascara w/Isododecane	6.0	2.0
Mascara w/UR 5050/TRIMETHYLSILOXYSILICATE (1:2.5)	9.0	4.0

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- Animal Health & Feed (new)
- Finished Dosage Formulation
- Laboratory Equipment
- Pharma Logistics

New in 2018

Excipients

Pharma Excipients event

Brazilian Pavilion

Polish Pavilion

Quick Facts: Expected in 2018

45,000+ pharmaceutical professionals

3,000+ exhibiting companies

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