### Enhancing Sun Care Using Bentone® Hectorite Clay Technology

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### **Abstract**

Recent studies have shown that Bentone<sup>®</sup> hectorite clay technology offers multiple benefits for today's sun care formulations beyond just thickening. Data has proven its benefits for both formulators and consumers, such as improved product physical stability and rheological properties, enhanced SPF protection and water resistance and modified product texture with good spreadability. Representative formulations demonstrated that hectorite clay technology provides formulation versatility and is easy to use in various sun care products.

#### Introduction

UV protection is now widely applied in various personal care formulations. Among different product forms, emulsions continue to be the most popular of all formulation vehicles for sun care products. Organic and inorganic actives are being used separately and in combinations as desired. Protection from both UVB and UVA has become the new standard. Besides sunscreen actives, product rheology is of great importance in sunscreens.

Driven by new and differentiated performance claims, sun care product manufacturers have been continuously working on product innovations. However, some fundamental sun care formulation needs still remain. No matter what innovative product is being created, the most basic but critical formulation need is to ensure product physical stability. Achieving elevated temperature stability can sometimes be a challenge. Suspending inorganic actives is essential with the increased use of physical sunscreens.

A main formulation need is to enhance SPF with the least amount of actives. It is generally believed that formulation rheology has a major effect on the SPF, and high product performance relies on uniform and thick distribution on the skin. In addition, for better UV protection, most sun care products require certain levels of SPF water resistance.

For sunscreens, improving product application properties is a formulation need directly related to product performance. Improved

product texture is always desired. Immediate protection upon applying a sunscreen is a goal to achieve. Good skin spreadability is necessary for more instant and even protective films.

The Bentone<sup>®</sup> Hectorite Clay Technology presented in this paper is proved to be able to enhance sun care formulations by responding to the formulation needs described above.

### **Hectorite Clay Technology**

Hectorite clay is a naturally occurring material with the structural formula  $Na_{0.33}[Mg_{2.67}Li_{0.33}]Si_4O_{10}[OH]_2$ . Bentone® hectorite clay technology is based on this unique swelling clay. The hydrophilic clays Bentone® MA, EW, and LT contain highly refined hectorite clay. These products can be delaminated in water and form a gel network structure. They are suitable for use in the aqueous phase of sun care formulations.

By reacting hydrophilic clay with quaternary ammonium compounds, Bentone® organoclays are produced for use in the oil phase. The organoclay Bentone® 38 VCG (INCI name: disteardimonium hectorite) is for organic systems with low to medium polarity, while Bentone® 27 VCG (INCI name: stearalkonium hectorite) is for medium to high polarity systems. These organoclay powders need to be properly dispersed and activated in organic solvents before incorporating them into formulations.

The Bentone Gel® products are optimally dispersed and activated pre-dispersions of hectorite organoclays. They are more suited for sunscreen formulations than the organoclay powders due to ease of use and optimal performance results. Bentone Gel® is typically comprised of one or more organic solvents, a hectorite organoclay (Bentone® 38 VCG or Bentone® 27 V CG) and an organic activator. A wide range of Bentone Gel® products made from various commonly used cosmetic oils, esters and solvents can be selected based on each formulation need.

Fully activated hectorite clay and organoclays form gels with an open, three dimensional network of clay platelets. Such gel structure is shown in Figure 1. The average dimensions of a clay platelet are 0.08 x 0.8 x 0.001  $\mu m$ . Bentone® products exhibit shear thinning and thixotropic flow behaviour (Figure 2). At low shear rates, the hectorite platelets are in a three-dimensional gel network structure, thus creating a high viscosity medium. With the increase in shear rate, the structure is disturbed and the platelets orientate in the direction of flow, causing a decrease in viscosity. On removal of shear, the reformation of the gel structure occurs at a reduced rate than the deformation, thus producing a system with thixotropic flow behaviour.

Hectorite and Bentonite are both swellable smectite clays. But they have major differences in composition, as well as clay platelet size and shape. Hectorite clay based products exhibit multiple advantages over Bentonite, such as lower iron content, lighter colour, higher swelling capacity, greater viscosity, excellent skin feel and no crystalline silica.

### **Formulation Study**

Bentone® hectorite clay and organoclays are suitable for various sun care applications, including creams, lotions, sticks and ointments, etc. Formulations shown in Table 1 to Table 7 demonstrate the applications in common sun care emulsion based formulations. These examples represent products with low, medium and high SPF values including W/O and O/W emulsions, organic actives, inorganic actives, organic and inorganic active combinations, UVA and UVB filtres.

A Bentone Gel® product is added into the oil phase, usually at a use level ranging from 2 to 15%. During the formulation process, it is preferred to first mix Bentone Gel® with most compatible component, if possible. Before further addition or emulsification, homogeneity is required by applying medium to high shear mixing.

Ingredient         % w/w           Phase A           Bentone Gel® IPM V (Isopropyl Myristate and Stearalkonium Hectorite and Propylene Carbonate)         2.5           Parsol MCX (Ethylhexyl Methoxycinnamate)         6.0           Tegin 4100 Pellets (Glyceryl Stearate)         2.5           Tegosoft OS (Ethylhexyl Stearate)         13.5           Lexol IPM-NF (Isopropyl Myristate)         5.0           Isohexadecane         8.0           Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone)         2.5           isolan GO 33 (Polyglyceryl 3-Oleate)         1.75           Phase B         3.0           Nanox™ 200 (Zinc Oxide)         10.0           Phase C         3.0           Magnesium Sulphate Heptahydrate         0.75           Sodium Chloride         0.75           Deionized Water         43.55           Phase D         3.0           1, 3-Butylene Glycol         3.0           Phenoxetol (Phenoxyethanol)         0.2           Mixing Procedure         1. Heat Phase A to 75°C and add Phase B with mixing.           2. Heat Phase C to 75° C.         3. Add Phase A + B into Phase C with mixing.           4. Cool to 35°C and add Phase D.	Formula 1 - Water in Oil Suncream (In-Vitro SPF 45)				
Bentone Gel® IPM V (Isopropyl Myristate and Stearalkonium Hectorite and Propylene Carbonate) Parsol MCX (Ethylhexyl Methoxycinnamate) 1	Ingredient	% w/w			
Stearalkonium Hectorite and Propylene Carbonate) 2.5   Parsol MCX (Ethylhexyl Methoxycinnamate) 6.0   Tegin 4100 Pellets (Glyceryl Stearate) 2.5   Tegosoft OS (Ethylhexyl Stearate) 13.5   Lexol IPM-NF (Isopropyl Myristate) 5.0   Isohexadecane 8.0   Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone) 2.5   fsolan GO 33 (Polyglyceryl 3-Oleate) 1.75   Phase B   Nanox™ 200 (Zinc Oxide) 10.0   Phase C   Magnesium Sulphate Heptahydrate 0.75   Sodium Chloride 0.75   Deionized Water 43.55   Phase D   1, 3-Butylene Glycol 3.0   Phenoxetol (Phenoxyethanol) 0.2   Mixing Procedure 1. Heat Phase A to 75°C and add Phase B with mixing.   2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Phase A				
Parsol MCX (Ethylhexyl Methoxycinnamate)  Tegin 4100 Pellets (Glyceryl Stearate)  Tegosoft OS (Ethylhexyl Stearate)  13.5  Lexol IPM-NF (Isopropyl Myristate)  Isohexadecane  Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone)  15 solan GO 33 (Polyglyceryl 3-Oleate)  Phase B  Nanox™ 200 (Zinc Oxide)  Phase C  Magnesium Sulphate Heptahydrate  0.75  Sodium Chloride  0.75  Deionized Water  1, 3-Butylene Glycol  phenoxetol (Phenoxyethanol)  Nanox™ Procedure  1. Heat Phase A to 75°C and add Phase B with mixing.  2. Heat Phase C to 75° C.  3. Add Phase A + B into Phase C with mixing.	Bentone Gel® IPM V (Isopropyl Myristate and				
Tegin 4100 Pellets (Glyceryl Stearate)  Tegosoft OS (Ethylhexyl Stearate)  Lexol IPM-NF (Isopropyl Myristate)  Isohexadecane  Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone)  Salan GO 33 (Polyglyceryl 3-Oleate)  Phase B  Nanox™ 200 (Zinc Oxide)  Phase C  Magnesium Sulphate Heptahydrate  Magnesium Sulphate Heptahydrate  O.75  Sodium Chloride  O.75  Deionized Water  1, 3-Butylene Glycol  Phase D  1, 3-Butylene Glycol  Phenoxetol (Phenoxyethanol)  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing.  2. Heat Phase C to 75° C.  3. Add Phase A + B into Phase C with mixing.	Stearalkonium Hectorite and Propylene Carbonate)	2.5			
Tegosoft OS (Ethylhexyl Stearate)  Lexol IPM-NF (Isopropyl Myristate)  Isohexadecane  Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone)  Solan GO 33 (Polyglyceryl 3-Oleate)  Phase B  Nanox™ 200 (Zinc Oxide)  Phase C  Magnesium Sulphate Heptahydrate  Magnesium Sulphate Heptahydrate  Deionized Water  Deionized Water  1, 3-Butylene Glycol  Phenoxetol (Phenoxyethanol)  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing.  2. Heat Phase C with mixing.	Parsol MCX (Ethylhexyl Methoxycinnamate)	6.0			
Lexol IPM-NF (Isopropyl Myristate) Isohexadecane 8.0 Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone) 2.5 Îsolan GO 33 (Polyglyceryl 3-Oleate) 1.75  Phase B Nanox™ 200 (Zinc Oxide) 10.0  Phase C  Magnesium Sulphate Heptahydrate 0.75 Sodium Chloride 0.75 Deionized Water 43.55  Phase D  1, 3-Butylene Glycol 2, 3.0 Phenoxetol (Phenoxyethanol) 0.2  Mixing Procedure 1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75°C. 3. Add Phase A + B into Phase C with mixing.	Tegin 4100 Pellets (Glyceryl Stearate)	2.5			
Isohexadecane 8.0 Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone) 2.5 Îsolan GO 33 (Polyglyceryl 3-Oleate) 1.75  Phase B Nanox™ 200 (Zinc Oxide) 10.0  Phase C  Magnesium Sulphate Heptahydrate 0.75 Sodium Chloride 0.75 Deionized Water 43.55  Phase D  1, 3-Butylene Glycol 3.0 Phenoxetol (Phenoxyethanol) 3.0 Phenoxetol (Phenoxyethanol) 0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Tegosoft OS (Ethylhexyl Stearate)	13.5			
Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone)  \$\hat{1}{2}\text{solan GO 33 (Polyglyceryl 3-Oleate)}\$ \$1.75\$  Phase B  Nanox™ 200 (Zinc Oxide)  Magnesium Sulphate Heptahydrate  Sodium Chloride  0.75  Sodium Chloride  0.75  Deionized Water  43.55  Phase D  1, 3-Butylene Glycol  3.0  Phenoxetol (Phenoxyethanol)  0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing.  2. Heat Phase C to 75° C.  3. Add Phase A + B into Phase C with mixing.	Lexol IPM-NF (Isopropyl Myristate)	5.0			
isolan GO 33 (Polyglyceryl 3-Oleate)  Phase B  Nanox™ 200 (Zinc Oxide)  Phase C  Magnesium Sulphate Heptahydrate Sodium Chloride O.75 Sodium Chloride O.75 Deionized Water 43.55  Phase D  1, 3-Butylene Glycol 7, 3-Butylene Glycol 9henoxetol (Phenoxyethanol) 0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Isohexadecane	8.0			
Phase B Nanox™ 200 (Zinc Oxide) 10.0  Phase C  Magnesium Sulphate Heptahydrate 0.75 Sodium Chloride 0.75 Deionized Water 43.55  Phase D  1, 3-Butylene Glycol 3.0 Phenoxetol (Phenoxyethanol) 0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Abil EM 90 (Cetyl PEG/PPG 10-1 Dimethicone)	2.5			
Nanox™ 200 (Zinc Oxide)  Phase C  Magnesium Sulphate Heptahydrate  Sodium Chloride  Deionized Water  Phase D  1, 3-Butylene Glycol  Phenoxetol (Phenoxyethanol)  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing.  2. Heat Phase C to 75° C.  3. Add Phase A + B into Phase C with mixing.	Îsolan GO 33 (Polyglyceryl 3-Oleate)	1.75			
Phase C  Magnesium Sulphate Heptahydrate 0.75 Sodium Chloride 0.75 Deionized Water 43.55  Phase D  1, 3-Butylene Glycol 3.0 Phenoxetol (Phenoxyethanol) 0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Phase B				
Magnesium Sulphate Heptahydrate 0.75 Sodium Chloride 0.75 Deionized Water 43.55  Phase D  1, 3-Butylene Glycol 3.0 Phenoxetol (Phenoxyethanol) 0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Nanox™ 200 (Zinc Oxide)	10.0			
Sodium Chloride  Deionized Water  43.55  Phase D  1, 3-Butylene Glycol Phenoxetol (Phenoxyethanol)  3.0  Phenoxetol (Phenoxyethanol)  0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing.  2. Heat Phase C to 75° C.  3. Add Phase A + B into Phase C with mixing.	Phase C				
Deionized Water 43.55  Phase D  1, 3-Butylene Glycol 3.0 Phenoxetol (Phenoxyethanol) 0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Magnesium Sulphate Heptahydrate	0.75			
Phase D  1, 3-Butylene Glycol 3.0 Phenoxetol (Phenoxyethanol) 0.2  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Sodium Chloride	0.75			
1, 3-Butylene Glycol 2, 3.0 Phenoxetol (Phenoxyethanol) 3.0  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing. 2. Heat Phase C to 75° C. 3. Add Phase A + B into Phase C with mixing.	Deionized Water	43.55			
Phenoxetol (Phenoxyethanol)  Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing.  2. Heat Phase C to 75° C.  3. Add Phase A + B into Phase C with mixing.	Phase D				
Mixing Procedure  1. Heat Phase A to 75°C and add Phase B with mixing.  2. Heat Phase C to 75° C.  3. Add Phase A + B into Phase C with mixing.	1, 3-Butylene Glycol	3.0			
<ol> <li>Heat Phase A to 75°C and add Phase B with mixing.</li> <li>Heat Phase C to 75°C.</li> <li>Add Phase A + B into Phase C with mixing.</li> </ol>	Phenoxetol (Phenoxyethanol)	0.2			
<ul><li>2. Heat Phase C to 75° C.</li><li>3. Add Phase A + B into Phase C with mixing.</li></ul>	Mixing Procedure				
3. Add Phase A + B into Phase C with mixing.	· · · · · · · · · · · · · · · · · · ·				
4. Cool to 35°C and add Phase D.					
	4. Cool to 35°C and add Phase D.				

Table 1



Formula 2 – Oil in Water Inorganic Sunsci Ingredient Phase A	reen ( <i>In-Vitro</i> SP % w/w	F 15) % w/w	% w/w
Bentone Gel® IHD V (Isohexadecane and Disteardimonium			
Hectorite and Propylene Carbonate)	3.0		
Bentone Gel® OP V (Ethylhexyl Palmitate and Stearalkonium			
Hectorite and Propylene Carbonate)		3.0	
Bentone Gel® TN V (C12-15 Alkyl Benzoate and Stearalkonium			
Hectorite and Propylene Carbonate)			3.0
Tegosoft TN (C12-15 Alkyl Benzoate)	10.0	10.0	10.0
Tegosoft Liquid (Cetearyl Ethylhexanoate)	5.0	5.0	5.0
Tego Alkanol (Cetyl Alcohol)	3.0	3.0	3.0
Tego SMS (Sorbitan Stearate)	2.2	2.2	2.2
Beeswax	0.5	0.5	0.5
Phase B			
Nanox <sup>™</sup> 200 (Zinc Oxide)	7.0	7.0	7.0
T805 (Titanium Dioxide)	1.2	1.2	1.2
Phase C			
Deionized Water	56.6	56.6	56.6
Vegetable Glycerin	8.0	8.0	8.0
Crillet 3 Super (Polysorbate 60)	3.3	3.3	3.3
Phase D			
Phenoxetol (Phenoxyethanol)	0.2	0.2	0.2
Mixing Procedure			
1. Heat Phase A together to 75°C, and add Phase B with mixing.			
2. Heat Phase C to 75°C.			
3. Add Phase A+B into Phase C with mixing.			

Table 2

Bentone® MA, EW, or LT can be used in the water phase either alone or in combination with Bentone Gel®, at a typical use level of 0.2 to 1.0%. The hydrophilic hectorite clay is first dispersed in de-ionized water with high shear mixing. Other ingredients may then be combined into the water phase.

4. Cool to 35°C and add Phase D.

Both hydrophilic hectorite clay and organoclays may be processed at room temperature or at elevated temperatures. Therefore, Bentone® hectorite clay technology allows both cold process formulation conditions and common emulsion production conditions with heat.

All formulations shown in Tables 1-7 passed three months stability testing at room temperature and at elevated temperatures. All invitro SPF values presented in this article were measured using the Optometrics SPF290S Ultraviolet Transmittance Analyser. Vitro-Skin was used as the substrate.

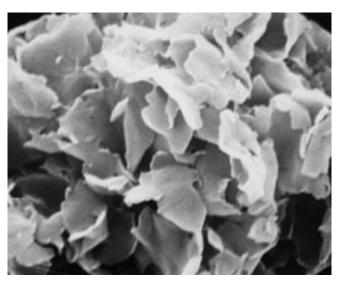


Figure 1. Gel Structure



Formula 3 – Water in Oil Inorganic Sunscreen	(In-Vitro S	PF 23)			
Ingredient	% w/w	% w/w	% w/w		
Phase A					
Bentone Gel® IHD V (Isohexadecane and Disteardimonium					
Hectorite and Propylene Carbonate)	8.0				
Bentone Gel® OP V (Ethylhexyl Palmitate and Stearalkonium					
Hectorite and Propylene Carbonate)		8.0			
Bentone Gel® TN V (C12-15 Alkyl Benzoate and Stearalkonium					
Hectorite and Propylene Carbonate)			8.0		
Tegosoft OS (Ethylhexyl Stearate)	13.5	13.5	13.5		
Isohexadecane	8.0	8.0	8.0		
Tegosoft M (Isopropyl Myristate)	5.0	5.0	5.0		
Abil EM 90 (Cetyl PEG/PPG-10-1 Dimethicone)	1.8	1.8	1.8		
Isolan GO 33 (Polyglyceryl 3-Oleate)	1.75	1.75	1.75		
Phase B					
Nanox™ 200 (Zinc Oxide)	13.5	13.5	13.5		
T805 (Titanium Dioxide)	2.35	2.35	2.35		
Phase C					
Magnesium Sulphate Heptahydrate	0.75	0.75	0.75		
Sodium Chloride	0.75	0.75	0.75		
Deionized Water	41.4	41.4	41.4		
Phase D					
1,3-Butylene Glycol	3.0	3.0	3.0		
Phenoxetol (Phenoxyethanol) 0.2 0.2 0					
Mixing Procedure					
1. Heat Phase A to 75°C and add Phase B with mixing.					
2. Heat Phase C to 75°C and add to Phase A+B.					
3. Cool to under 35°C and add Phase D.					

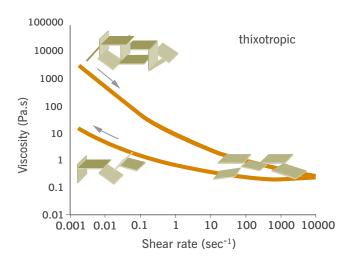


Figure 2. Hectorite Clay Flow Behaviour

Table 3

Bentone® materials are found to be compatible with typical sunscreen ingredients, such as commonly used organic and inorganic sunscreen actives, emulsifiers, emollients, film formers, fragrances, preservatives, etc.

Bentone® hectorite clay technology is suggested for sun care formulations due to several reasons. Bentone® hectorite clay offers a unique combination of properties, such as thixotropic flow, viscosity building, suspending capability, unique texture and light and smooth skin feel. As mentioned above, it also exhibits multiple advantages compared to bentonite clay products. The benefits of choosing hectorite clay products over other commonly used thickeners are also evident. For sun care, typical oil phase thickeners such as waxes, or long-chain fatty alcohols, usually have a heavy skin feel with no rheological properties like



Formula 4 – Inorganic Sunscreen ( <i>In-Vitro</i> SPF 14) Ingredient Phase A	% w/w
Bentone Gel® TN V (C12-15 Alkyl Benzoate and Stearalkonium	
Hectorite and Propylene Carbonate)	10.0
Tegosoft CT (Caprylic/Capric Triglyceride)	5.0
Cithrol GMS A/S PAST (Glyceryl Stearate and PEG-100 Stearate)	4.0
Tegosoft TN (C12-15 Alkyl Benzoate)	4.0
Tego Alkanol 1618 (Cetearyl Alcohol)	1.2
Paratexin P (Propyl Paraben)	0.1
Phase B	
Nanox <sup>™</sup> 200 (Zinc Oxide)	5.5
T 805 (Titanium Dioxide)	2.5
Phase C	
Bentone® LT (Hectorite and Hydroxyethylcellulose)	0.24
Deionized Water	63.36
Phase D	
Paratexin M (Methylparaben)	0.1
Propylene Glycol	4.0
Mixing Procedure	
1. Heat Phase A to 75°C and add Phase B with mixing.	
2. Mix Phase C and add to Phase D. Heat to 75°C.	
3. Add Phase A + B to Phase C + D.	
4. Cool with stirring.	

Table 4

Bentone<sup>®</sup>. Thixotropic flow is desired for many personal care products, while most common aqueous phase thickeners do not show the thixotropic flow behaviour offered by Bentone<sup>®</sup> hectorite clay products.

# **Performance Benefits in Sun Care Formulations**

### Organoclays improve physical stability of sunscreens

The product stability improvement offered by a Bentone Gel® product has been proven in many formulation examples.

Stability testing for an organic sunscreen (Formula 5, Table 5) at  $40^{\circ}$ C after 1 month showed that the addition of 3% Bentone  $Gel^{\$}$  improved the emulsion stability significantly (Figure 3). We observed obvious phase separation for Formula 5 without Bentone  $Gel^{\$}$ . In comparison, the incorporation of organoclay in a Bentone  $Gel^{\$}$  product resulted in a stable formulation.

Rheological data confirmed that organoclays improve the formulation stability due to the stronger internal structure. We



Figure 3. Formula 5 Stability Test

carried out rheological oscillation testing for Formula 5 with and without Bentone Gel® EUG V, as shown in Figure 4. The storage modulus (G') and the loss modulus (G") were measured with variable frequency and constant amplitude values using Physica



Formula 5 – Sprayable Organic Sunscreen ( <i>In-Vitro</i> SPF 39) Ingredient Phase A	% w/w
Bentone Gel® EUG V (Octyldodecanol and Disteardimonium	
Hectorite and Propylene Carbonate)	3.0
Tegosoft CT (Caprylic/Capric Triglyceride)	4.0
Eutanol G (Octyldodecanol)	2.5
Phase B	
Dow Corning 345 Fluid (Cyclomethicone)	10.0
Parsol MCX (Ethylhexyl Methoxycinnimate)	5.5
Multiwax W-835 (Microcrystalline Wax)	3.0
Dow Corning 5200 Formulation Aid (Laurylmethicone Copolyol)	2.0
Tegosoft TN (C12-15 Alkyl Benzoate)	2.0
Parsol 1789 (Butyl Methoxybenzoylmethane)	1.6
Phase C	
Deionized Water	57.3
Sodium Chloride	2.0
Vegetable Glycerin	7.0
Phase D	
Phenoxetol (Phenoxyethanol)	0.1
Mixing Procedure	
1. Mix Phase A and warm to 40°C.	
2. Add Phase A to Phase B. Heat to 70°C.	
3. Mix Phase C and heat to 70°C.	
4. Add Phase A + B to Phase C.	
5. Cool to under 35°C and add Phase D.	

Frequency Sweeps Pa 

Table 5

Angular Frequency

Figure 4. Formula 5 Oscillation Curve

1/s

Sprayable Organic Sunscreen no Bentone Gel<sup>®</sup> EUG V
 Sprayable Organic Sunscreen with Bentone Gel<sup>®</sup> EUG V

## Sun Screens & UV Protection

Formula 6 – Organic Suncream ( <i>In-Vitro</i> SPF 30)				
Ingredient	% w/w			
Phase A				
Bentone Gel® TN V C 12-15 Alkyl Benzoate and Stearalkonium				
Hectorite and Propylene Carbonate	3.0			
Cosmowax EM5483 PAST (Cetearyl Alcohol and Ceteareth-20)	7.5			
Eusolex 2292 (Ethylhexyl Methoxycinnimate and BHT)	7.5			
Cithrol GMS A/S PAST (Glyceryl Stearate and PEG-100 Stearate)	7.0			
Tegosoft CT (Caprylic/Capric Triglyceride)	6.0			
Tegosoft TN (C12-15 Alkyl Benzoate)	4.0			
Eusolex 4360 (Benzophenone-3)	3.0			
Phase B				
Deionized Water	57.9			
Phase C				
Propylene Glycol	4.0			
Phenoxetol (Phenoxyethanol)	0.1			
Mixing Procedure				
1. Heat Phase A to 75°C.				
2. Heat Phase B to 75°C.				
3. Add Phase A to Phase B				
4. Cool to under 35°C and add Phase C.				

Table 6

Formula 7 – Sunscreen with Bentone Gel® HSO V (In-Vitro SPF 14)				
Ingredient	% w/w			
Phase A				
Bentone Gel® HSO V (Helianthus Annuus Seed Oil and				
Disteardimonium Hectorite and Propylene Carbonate)	12.0			
Tegosoft Liquid (Cetearyl Ethylhexanoate)	7.5			
Polarwax NF (Cetearyl Alcohol and Polysorbate 60)	4.2			
Tegosoft M (Isopropyl Myristate)	3.0			
Eusolex 2292 (Ethylhexyl Methoxycinnimate and BHT)	2.5			
Parsol 1789 (Butyl Methoxybenzoylmethane)	0.8			
Phase B				
Deionized Water	66.8			
Vegetable Glycerin	3.0			
Phase C				
Phenoxetol (Phenoxyethanol)	0.2			
Mixing Procedure				
1. Heat Phase A to 75°C.				
2. Heat Phase B to 75°C.				
3. Add Phase A to Phase B.				
4. Cool to under 35°C and add Phase C.				

Table 7



MCR 300 Rheometer. We examined the formulation's short-term behaviour, simulated by rapid movements (at high frequencies), and long-term behaviour, simulated by slow movements (at low frequencies).

As we know, G' represents the elastic component (storage modulus), and G" represents the viscous component (loss modulus) for a real viscoelastic material. Higher G' indicates a more structured system and better stability. Higher G" indicates better flow but a more loose structure thus a less stable system. For Formula 5 with Bentone Gel® EUG V, G' is greater than G" in the low angular frequency range, indicating the formulation's strong internal structure and better stability (Figure 4). In comparison, for the same formula without Bentone Gel® EUG V, G" is constantly greater than G' indicating a stability problem within the formulation. These rheological study results not only confirmed our formulation stability test results, but also explained

how a Bentone Gel® product improves stability from the internal structural level.

Another water in oil (W/O) sunscreen containing inorganic actives (Formula 3, Table 3) was also studied to provide additional evidence for the stability improvement effect of Bentone® organoclay.

Formula 3 without the use of any Bentone Gel® failed stability testing at 40°C after 1 month by showing obvious phase separation (Figure 5). Three different Bentone Gel® products, Bentone Gel® TN V, Bentone Gel® OP V and Bentone Gel® IHD V, were added separately into the same formulation base. Here it was clearly seen that the addition of any Bentone Gel® contributed to an improvement in physical stability.

We further investigated the thermostability of Formula 3 by measuring the temperature dependent structural behaviour in

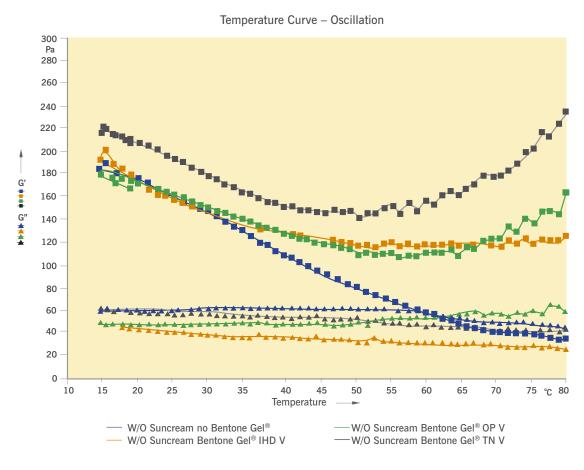


Figure 6. Formula 3 Oscillation Curve Over Temperature Range





Figure 5. Formula 3 Stability Test

the oscillation mode using the Physica MCR 300 Rheometer. As shown in Figure 6, G' is constantly greater than G" for the sunscreens with a Bentone Gel® product over the temperature range. This result indicated that the sunscreens have a stronger elastic behaviour than viscous behaviour even at higher temperatures, thus good thermostability. For the same sunscreen base without any Bentone Gel®, G' continuously decreases to a value below G" at around 60°C, indicating decreased system stability as temperature increases. The dramatically different results further proved that the organoclay improves the internal structure and formulation thermostability.

In addition to emulsion stability, inorganic sunscreen actives, or other particulates, need to remain suspended in the formulations. We studied the suspending capability of organoclays in sunscreens and used Formula 2 (Table 2) as an example. Formula 2 is an O/W inorganic sunscreen containing both titanium dioxide and zinc oxide.

The suspending capability test was carried out at 40°C for 3 months by observing Formula 2 containing Bentone Gel® IHD V, Bentone Gel® OP V, or Bentone Gel® TN V, separately, as well as Formula 2 without any Bentone Gel® or organoclay. At the end of this test, the sample without organoclay showed obvious TiO2 and ZnO separation, while the samples with either Bentone Gel® IHD V, Bentone Gel® OP V, or Bentone Gel® TN V remained uniform and stable.

An explanation on why organoclays improve the suspending capability is provided by the rheological data shown in Figure 7. From a similar oscillation test (frequency sweep) as described earlier, we found that Formula 2 with a Bentone Gel® product exhibits a higher G', as well as a greater difference between G' and G", compared to the same formula without any Bentone Gel®.

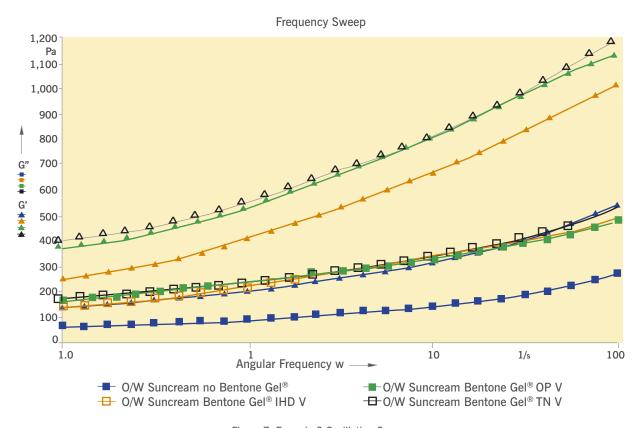


Figure 7. Formula 2 Oscillation Curve



These results suggest that the formulations with an organoclay are relatively undisturbed under gravity and during shipping/handling, thus provide better suspending capability.

## Organoclays enhance SPF values and water resistance

Organoclays are typically used as rheological additives, and the above study results have already demonstrated their performance for improving the physical stability of sunscreens. To identify the impact of organoclays to the UV protection performance of sunscreens, we measured the in-vitro SPF for all the formulations listed in Tables1-7 with and without organoclay.

Our measurement results are compared in Table 8. This data repeatedly showed a definite general trend of higher SPF values with the use of organoclays, no matter which formulations were used. The sun protection enhancement we observed is certainly an added benefit of the Bentone® technology. These results also

suggest that the increase in SPF value due to the organoclay should not be considered as a dramatic SPF boost. Whether a significant SPF increase can be obtained depends on the formulation base.

SPF water resistance performance was further evaluated by following the Very Water Resistance *In-Vitro* SPF protocol as outlined below.

Firstly the Vitro-Skin® substrate was hydrated for 16 hours in a closed hydration chamber. A sunscreen sample of 2mg/cm² was applied to the substrate and rubbed evenly onto the substrate for 20-30 seconds. The treated Vitro-Skin® substrate was then conditioned in a hydration chamber for 15 minutes. The substrate was removed and measurements were taken from different sites on the substrate. Following this initial reading, the treated substrate was immersed in an ambient temperature water bath for 80 minutes with stirring. The Vitro-Skin® substrate

Organoclay enhances sunscreen SPF values							
	Formula 1 W/O organic + inorganic Bentone Gel® IPM V	Formula 2 O/W inorganic Bentone Gel® IHD V	Formula 3 W/O inorganic Bentone Gel® TN V	Formula 4 inorganic Bentone Gel® TN V	Formula 5 Sprayable organic Bentone Gel <sup>®</sup> EUG V	Formula 6 O/W organic Bentone Gel® TN V	Formula 7 O/W organic Bentone Gel® HSO V
In-Vitro SPF Formula with organoclay (Bentone Gel®)	44.7	15.5	23.3	14.1	38.9	30.4	14.2
In-Vitro SPF Formula with no organoclay	41.9	9.4	17.9	11.7	32.5	26.6	12.8

Table 8. SPF Test Results

Organoclay can improve sunscreen SPF water resistance					
Formulations		In-Vitro SPF (static) Before water immersion	In-Vitro SPF (VWR) After 80 minutes water immersion		
Formula 2 O/W Inorganic	No organoclay (with Finsolv TN)	12.2	1.6		
Suncream	With organoclay (Bentone Gel® TN V)	13.2	11.9		
Formula 6 Organic Suncream	No organoclay (with Finsolv TN)	26.6	6.0		
	With organoclay (Bentone Gel® TN V)	30.4	19.4		

Table 9. SPF Water Resistance Test Results



was then allowed to air-dry for one hour at room temperature and then re-conditioned in the hydration chamber for 2 hours. SPF measurements were taken using the Optometrics SPF290S Ultraviolet Transmittance Analyzer, and the SPF value is calculated as an average. These measurements are compared to a blank for each Vitro-Skin® substrate with no test material applied.

The SPF water resistance test results of an organic sunscreen (Formula 2, Table 2) and an inorganic sunscreen (Formula 6, Table 6) are shown in Table 9. We compared the SPF values before and after 80 minutes water immersion for each formula with and without the organoclay.

For Formula 2, the in-vitro SPF without organoclay dropped from 12.2 to 1.6, which is almost a full loss of sun protection after water immersion. In comparison, only 3% Bentone Gel® TN V in the same formula contributed to the excellent SPF retention after water immersion, from an initial SPF value of 13.2 to 11.9.

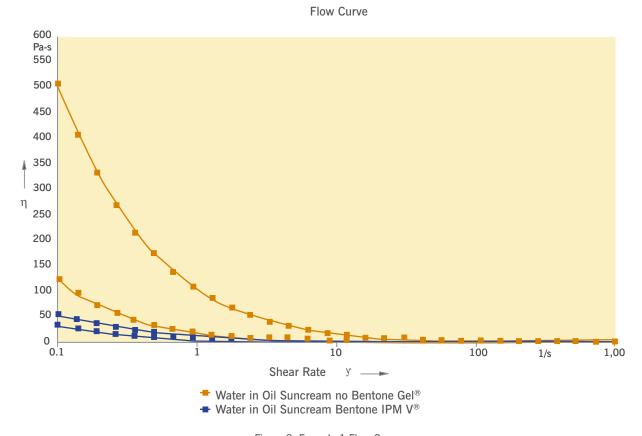
A similar trend was found when studying Formula 6. Again, we saw the significant SPF loss (from 26.6 to 6.0) without the use of organoclay. However, with the addition of 3% Bentone Gel<sup>®</sup>

TN V, a SPF value of 19.4 remained after 80 minutes water immersion, which is significantly higher than a SPF 6.0 from the same formula base without organoclay.

Organoclays are not designed to be a substitute for film formers in sun care products. Improving SPF water resistance can simply be an extra benefit from the Bentone® hectorite clay technology.

### Organoclays improve application properties

The rheological properties provided by the Bentone® technology also have a positive impact on the sun care product application properties. Rheological data illustrated in Figure 8 explained such benefits. We measured the flow curves of Formula 1 with and without Bentone Gel® to evaluate the effects of increasing and decreasing shear forces. With Bentone Gel® IPM V, the formulation viscosity decreases dramatically with increased shear rate, and rebuilds at an impaired rate with reduction in shear. The formula without Bentone Gel® shows an almost Newtonian flow with very little thixotropy. The comparison of these flow curves suggests that the organoclay imparts viscosity, shear thinning and thixotropic rheological properties to a sunscreen







formulation. The significantly reduced viscosity under higher shear, which is similar to the action of applying sunscreen on skin, makes the product easy to apply and spread. The thixotropic properties help to ensure that the sunscreen spreads into an even protective film.

Emulsions in general and creams in particular, can achieve the highest SPFs attainable. However, lotions are more popular than creams due to their easier spreadability on the skin and dispensability from bottle (Ref 1). By applying Bentone® technology, formulators can create a cream product by taking advantage of its viscosity building character in formulations and making the cream spread like a lotion using its shear thinning and thixotropic properties. Therefore, the use of Bentone® products makes it possible to have a cream vehicle for the highest SPF and to get easy spreadability and dispensability, simultaneously.

In addition, all the studied formulations containing Bentone® products exhibit a rich and unique texture, which is a highly desirable formulation property. For sunscreen products, skin feel is critical for consumers. We found that the Bentone® technology allows excellent product aesthetics with a smooth, non-greasy, and non-tacky skin feel.

### Conclusion

Bentone<sup>®</sup> hectorite clay technology is proved to be suitable for various sun care formulations. It is easy to use in different cold and heated formulation processes. It also shows good compatibility and formulation versatility. Most importantly, this technology provides proven multiple benefits for sun care formulations.

For sun care product consumers, it means enhanced UV protection with SPF water resistance, unique product texture

and skin aesthetics, as well as easy spreadability allowing more immediate and even sun protection.

For product formulation and processing, Bentone® technology helps to stabilize products, even at elevated temperatures. For sunscreens with inorganic actives, it efficiently suspends the particulates. It provides oil and aqueous phase viscosity building, rheology modification and thixotropic flow. Its rheological properties allow the sunscreen manufacturers to create products giving thick and even skin coverage. Furthermore, because of the use of Bentone® rheological additives, formulators may achieve enhanced SPF values and SPF water resistance.

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### **Primary Author's Biography**

Hongjie Cao is Global Technical Manager for Consumer Products department at Elementis Specialties. In this role, she is responsible for global technical support for existing products, as well as new application development and new product development efforts. Hongjie has been working in the cosmetics industry for 12 years. Before joining Elementis, Hongjie worked for National Starch and Chemical Company (ICI), where she established the new technology screening program for personal care and served as Manager for new technology screening and development. Hongjie started her career as a Senior Scientist at Nu Skin International, where she researched and developed various personal care products. Hongjie Cao received her PhD. in Physical Chemistry from Brigham Young University, an M.S. and a B.S. in Chemistry from Nankai University.

