



**STABLE, LOWER PARTICLE SIZE  
MULTIFUNCTIONAL SILANOLS**

1995

Joint Venture of  
Hoechst and  
Bayer Textile  
Dyes, Mitsubishi



2000

Joint Venture with BASF  
(incl. ICI / Zenecco dyes) &  
Mitsui



2004 - 06

Acquisition of Yorkshire Americas,  
Rotta Group & Boehme Group



2002

Acquisition of Ceter Solutions



2007

Acquisition of  
Texanlab



2010

Jointly acquired by:  
Longsheng Group &  
Kiri Industries



2016

**Emerald Performance Materials**  
Hilton Davis

Acquisition of Emerald Hilton  
Davis LLC, Emerald Carolina  
Chemical LLC, & Emerald Foam  
Control LLC

2013

Acquisition of Lenmar  
Chemical Corporation



OUR HISTORY

DyStar®



# DYSTAR'S APPROACH » SILOXANE EMULSIONS

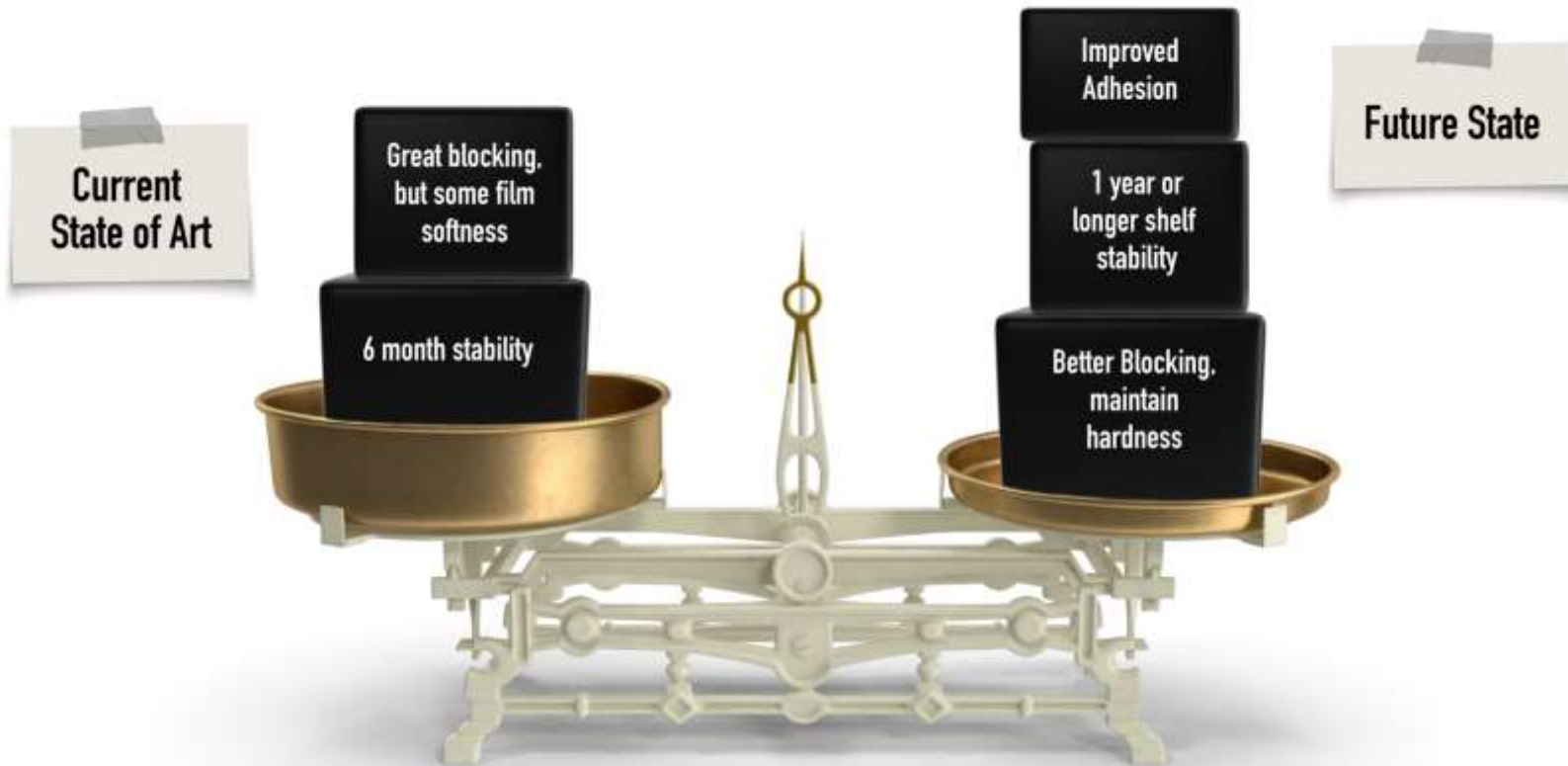
VOC REGULATIONS AND CONSUMER PREFERENCES HAVE PUSHED COATINGS MANUFACTURERS TO INNOVATE USING LOW TG RESINS, BUT WITH NEGATIVE TRADE-OFFS

- » BLOCK
- » TACKINESS
- » IMPRINTING
- » DIRT PICK UP
- » OPEN TIME
- » EXTERIOR GLOSS LOSS
- » STAIN RESISTANCE



ADDITIVES CAN HELP, BUT THEY CAN BE *EXPENSIVE, INCOMPATIBLE, OR CREATE OTHER ISSUES*

# DYSTAR'S APPROACH » SELF-CROSSLINKING SILANOL EMULSIONS





# SYNTHESIS AND EMULSIFICATION OF THE MULTIFUNCTIONAL SILANOLS



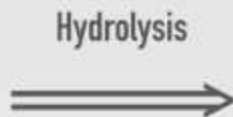
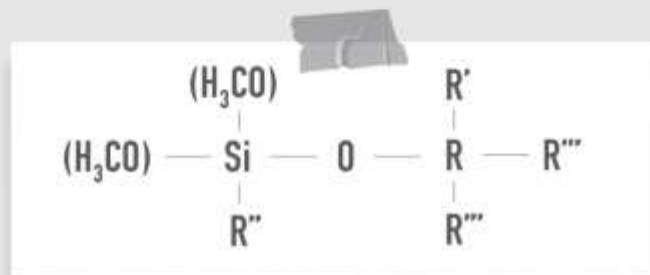
# GENERAL REACTION SCHEMATICS FOR HYDROLYSIS

## » Hydrolysis



# GENERALIZED STRUCTURES OF SILANES STUDIED

( R = silane repeat units / R' = alkyl or hydroxyl / R'' = alkyl/functional / R''' = alkyl )



**Idealized Structure**  
Multi-functional  
alkoxy silanes

**Silicone R'' variations studied:**

- » Methyl
- » Octyl
- » Phenyl
- » Epoxy functional

# EMULSIFICATION CONSIDERATIONS



- » **pH** (generally 4-7 needed for WB dispersion)
- » **Water** sufficient quantity (< 20% active)
- » **Temperature** (heat can cause unwanted reaction)
- » **Degree of Functionality & Molecular Weight** (as increases, becomes more difficult to stabilize as water-based dispersion)
- » **Anionic, Cationic, Amphoteric, Nonionic**
- » **Means to Emulsify** (rotor-stator, high pressure, etc)
- » **Surfactant HLB** (we used 12)
- » **Surfactant Selection** critical to stabilization due to the organic/inorganic nature of the product





# GENERALIZED STRUCTURES OF SURFACTANTS

» Linear Alcohol Ethoxylates  
(LAE)



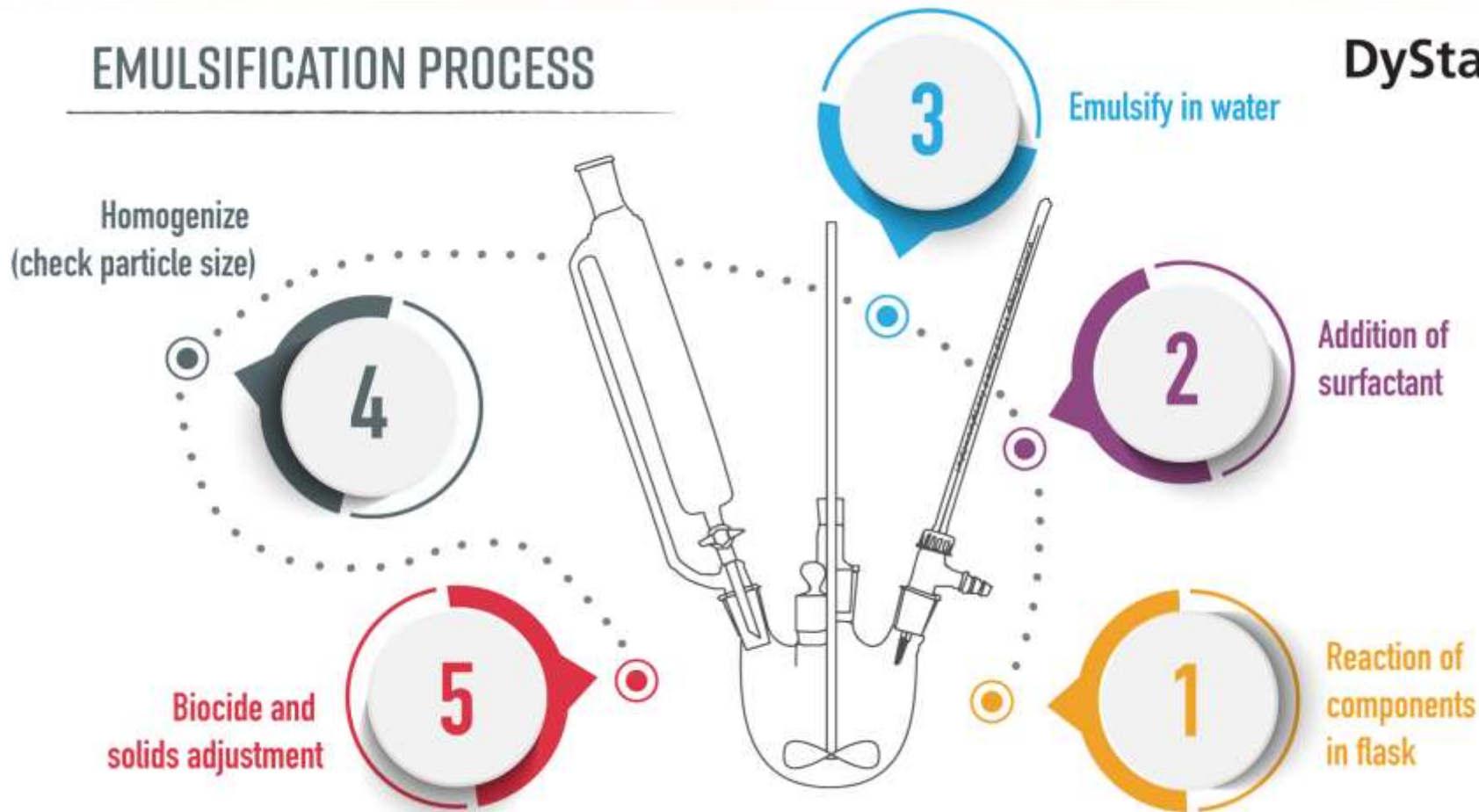
» Silicone Polyether higher MW  
(HMWSP)



» Silicone Polyether lower MW  
(LMWSP)



# EMULSIFICATION PROCESS



## WET PROPERTIES »

SAMPLE ITEM	LAE	HMWSP	LMWSP
% Solids (130°, 60min, 0.3 - 0.4 g samp, 2 ml of H <sub>2</sub> O)	49.4	47.2	47.8
pH	6.11	6.43	6.25
Viscosity, cps (RVT, #3 spin, 100 RPM, 25°C)	49.0	55.0	40.0
Comments	Smooth, white opaque, homogeneous	Smooth, white opaque, homogeneous	Smooth, white opaque, homogeneous

## 2 MIL WET FILMS » ON ALUMINUM



**Linear Alcohol Ethoxylates  
( LAE )**



**Silicone Polyether higher MW  
( HMWSP )**



**Silicone Polyether lower MW  
( LMWSP )**



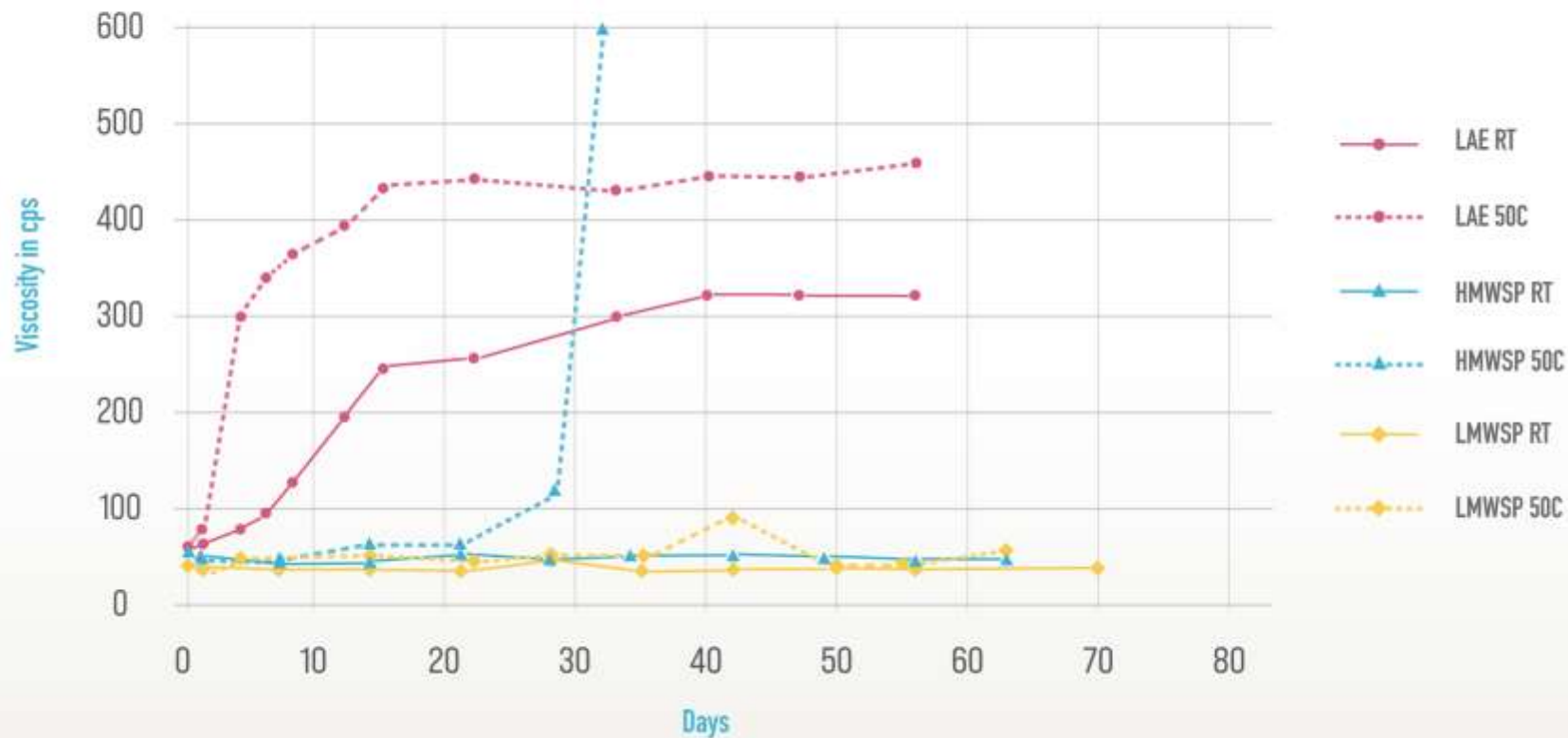
**STABILITY**

**STUDY OF THE EMULSION**

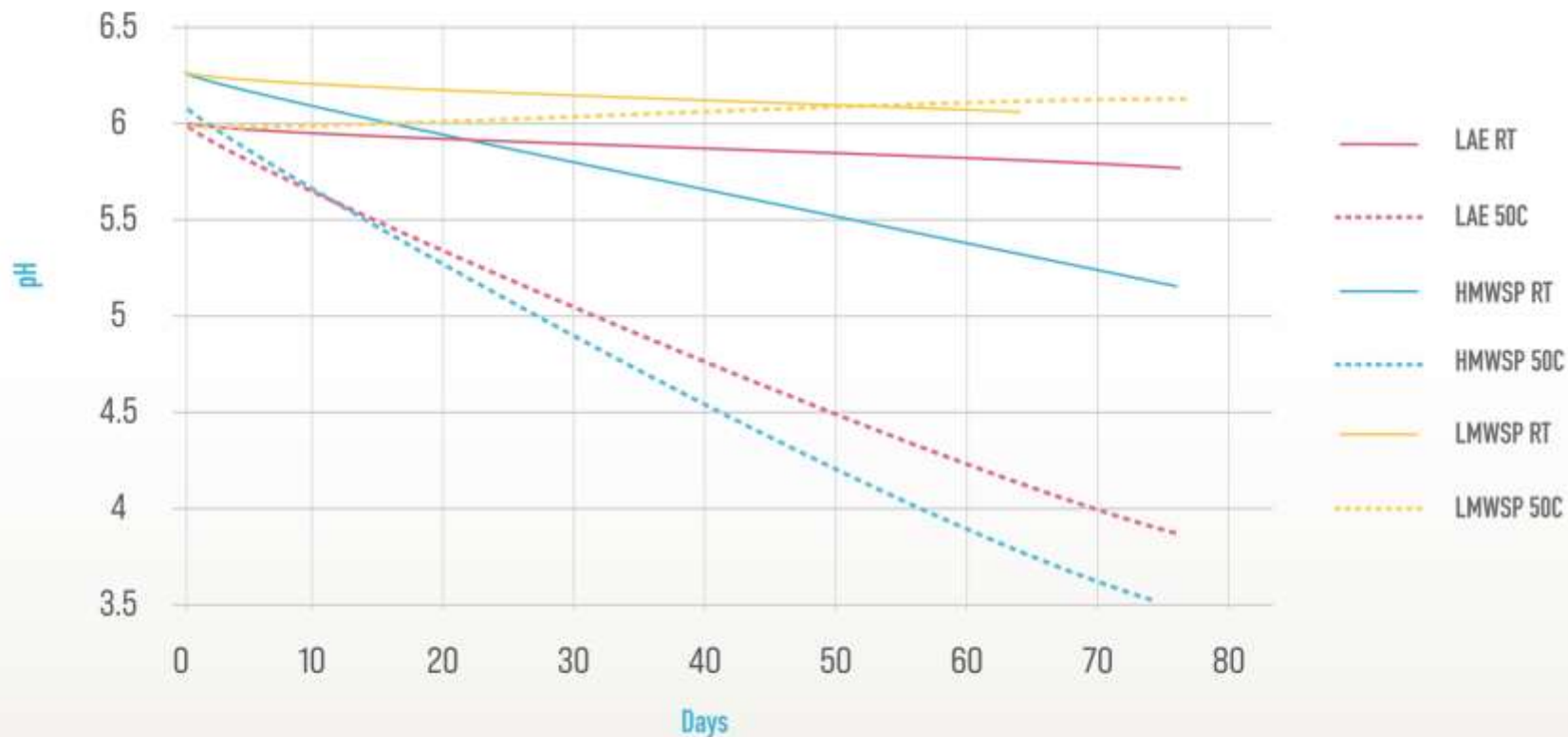




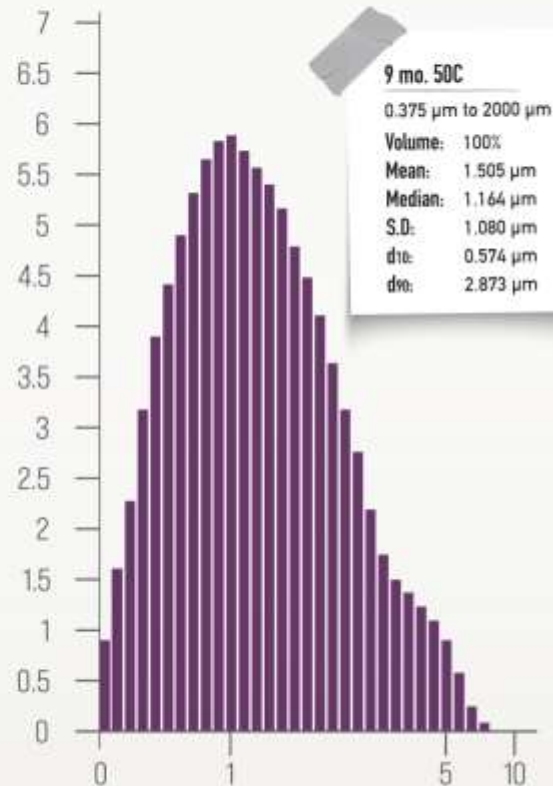
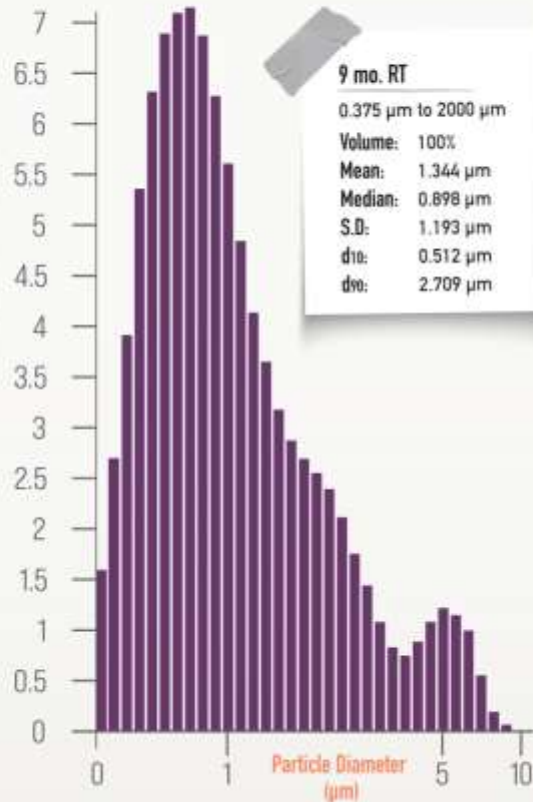
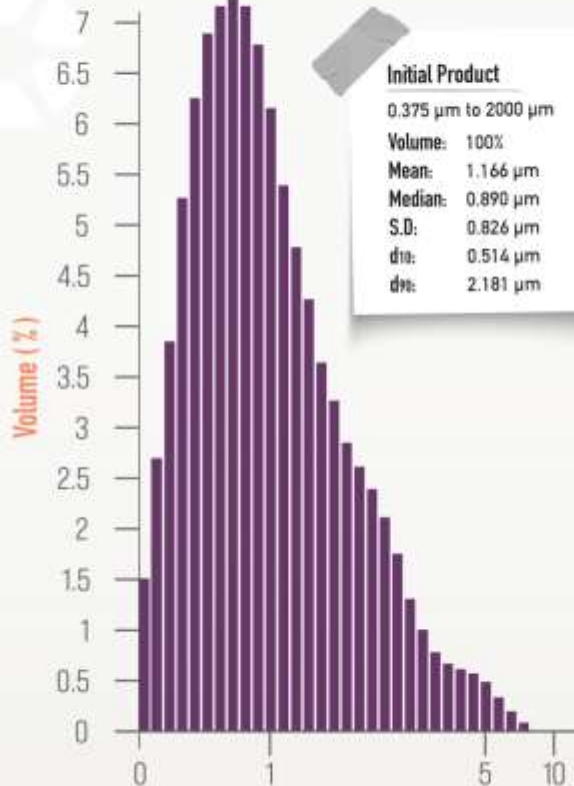
# SURFACTANT STUDY » VISCOSITY vs TIME



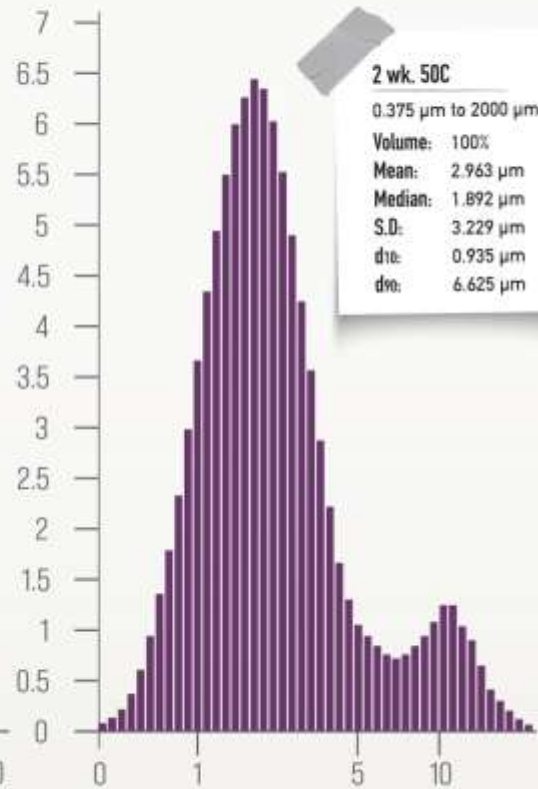
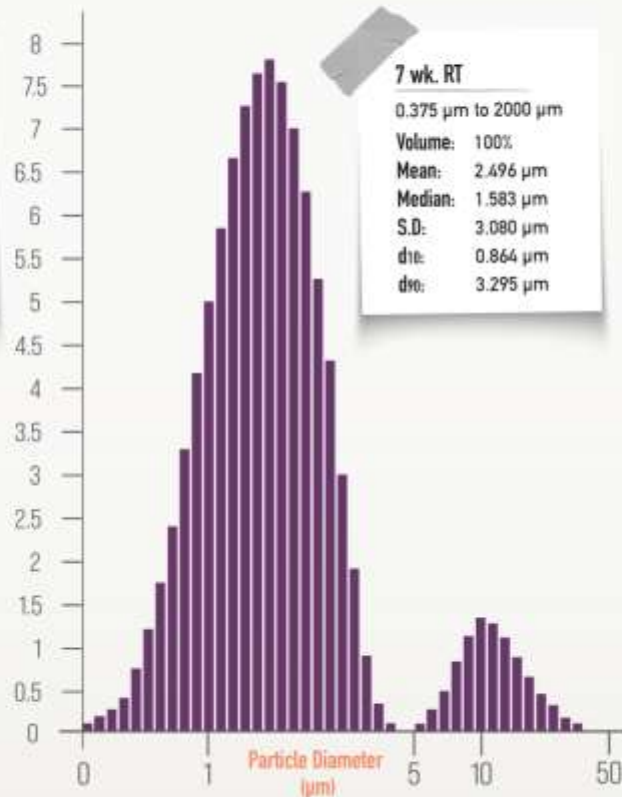
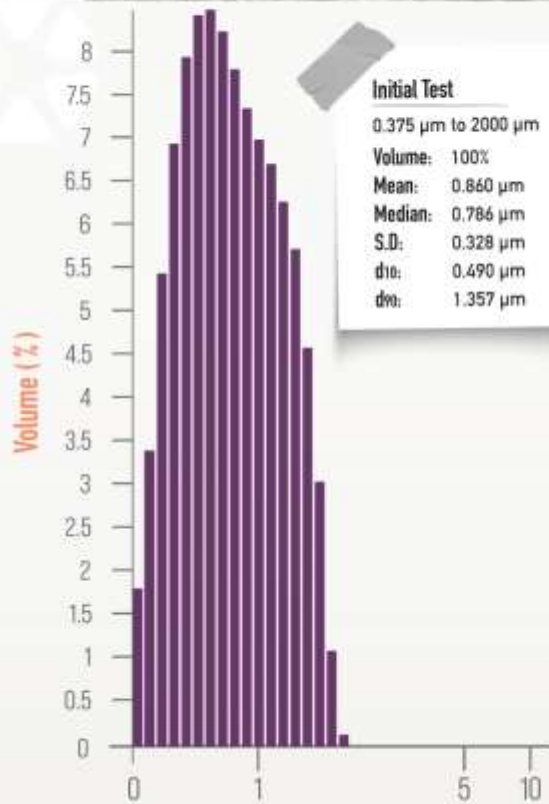
# SURFACTANT STUDY » pH vs TIME



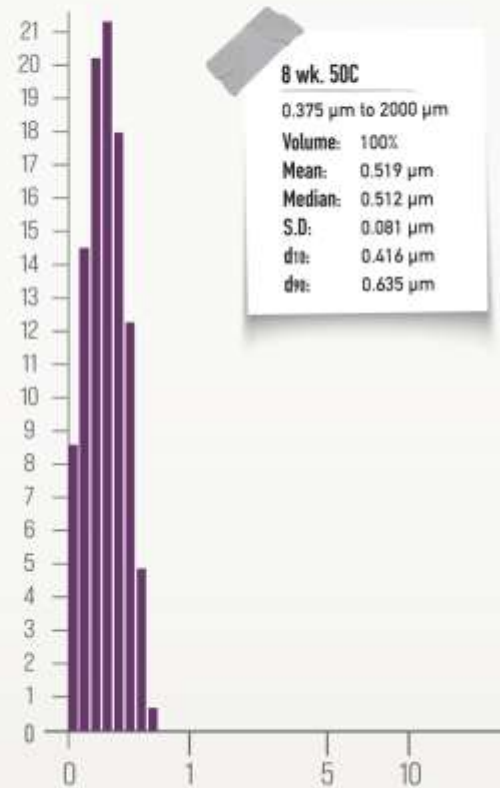
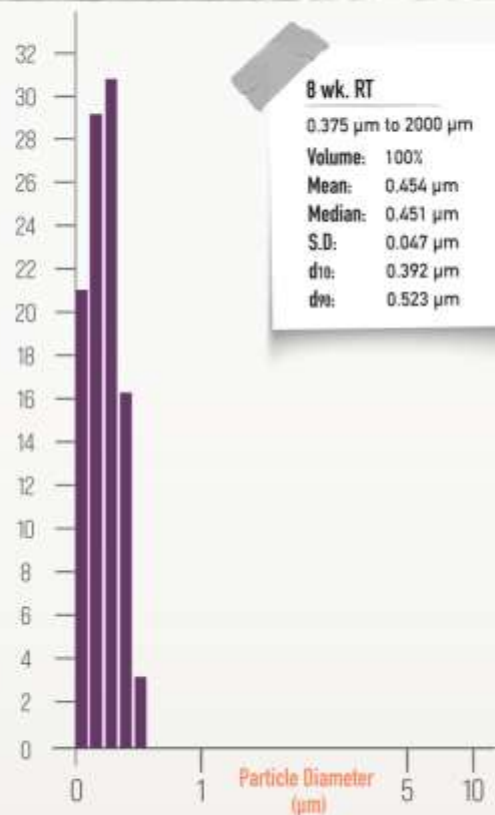
# PARTICLE SIZE » LAE STABILITY



# PARTICLE SIZE » HMWSP STABILITY



# PARTICLE SIZE » LMWSP STABILITY





# STABILITY » 6 months ( RT )



LAE

HMWSP

LMWSP



**TESTING OF THE EMULSION**  
in various Applications

# APPLICATION » PAINT FORMULATION



	FUNCTION	- 1 % ADDITIVE	- 2 % ADDITIVE	- 3 % ADDITIVE
GRIND	Water	13.80	13.80	13.80
	Coalescing aid	1.56	1.56	1.56
	Biocide	0.20	0.20	0.20
	Antifoam	0.20	0.20	0.20
	Dispersing Aid	0.20	0.20	0.20
	Pigment	23.40	23.40	23.40
	Rheology thickener	0.49	0.49	0.49
	Water	1.66	1.66	1.66
	Rheology thickener	0.20	0.20	0.20
PRE-LETDOWN	Water	2.93	2.93	2.93
	Binder	45.90	44.92	43.95
	Additive	0.98	1.95	2.93
	Water	2.34	2.34	2.34
LETDOWN	Antifoam	0.15	0.15	0.15
	Water	3.91	3.91	3.91
	Rheology thickener	2.00	2.00	2.00
	pH adjustment	0.10	0.10	0.10



# LADDER STUDY » APPLICATION TESTING



White Paint ( Pigment )	Control ( No Additive )		1% Additive with LAE		2% Additive with LAE		3% Additive with LAE	
	1 day	7 day	1 day	7 day	1 day	7 day	1 day	7 day
<b>Block (on Leneta)</b>								
1000g, 30min, 40C, 1/2hr cooling. (10 is best), (% removed at 7 days)	Rating 2 40% Removed		Rating 2 25% Removed		Rating 2 50% Removed		Rating 8 Very Slight Tack	
500g, 24hrs, 25C, (10 is best), (% removed at 7 days)	Rating 8 Very slight tack		Rating 9 Trace tack		Rating 10 Perfect		Rating 10 Perfect	
<b>Surface Testing (on Leneta)</b>								
20 Deg	15.7	15.2	13.6	12.1	11.2	11.3	10.8	11.1
60 Deg	55.7	55	53.3	51.6	49.2	49.7	48.6	49.6
85 Deg	88	86.9	86.9	85.3	82.8	82.3	84	83.7
Surface tension, Dynes Pens (7 Day)	44		38		36		36	
Dirt Pick-Up Test (14 Day) color strength	133.04		122.13		116.57		110.40	
Paint Stain Testing (3 Day)	42		36		34		37	
<b>Film Testing (on Aluminum)</b>								
Pencil Hardness	F	F	HB	HB	B	B	5B	4B
Crosshatch Adhesion (% loss of material)	2B - 20	2B - 15	2B - 15	2B - 10	2B - 30	2B - 10	2B - 30	2B - 20
Koenig Hardness 3"	16	16	14	15	13	14	12	14
Contact Angle (7 Day)	75.7		80.1		85.4		82.1	

## BLOCK » APPLICATION TESTING OK LNETA

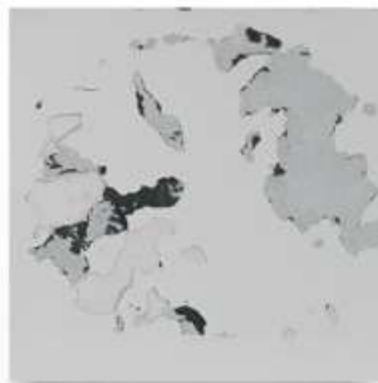
1000g, 30 min, 40°C, 1/2 hr cooling



**CONTROL**



**1%**



**2%**



**3%**



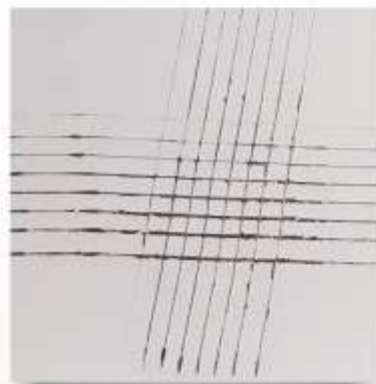
# CROSS HATCH » APPLICATION TESTING



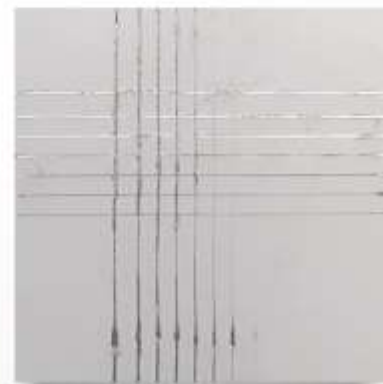
**CONTROL**



**1%**



**2%**



**3%**



# SURFACTANT COMPARISON » APPLICATION TESTING



White Paint ( Pigment )	Control ( No Additive )		1% Additive with LAE		1% Additive with LMWSP	
	1 day	7 day	1 day	7 day	1 day	7 day
<b>Surface Testing (on Leneta)</b>						
20 Deg	15.7	15.2	13.6	12.1	12.7	12.7
60 Deg	55.7	55	53.3	51.6	52.3	52.3
85 Deg	88	86.9	86.9	85.3	83.2	82.1
Surface tension, Dynes Pens (7 Day)	44		38		42	
Dirt Pick-Up Test (14 Day) color strength	133.04		122.13		114.20	
<b>Film Testing (on Aluminum)</b>						
Pencil Hardness	F	F	HB	HB	B	HB
Crosshatch Adhesion (% loss of material)	2B - 20	2B - 15	2B - 15	2B - 10	2B - 25	2B - 15
Koenig Hardness 3"	16	16	14	15	12	14
Contact Angle (7 Day)	75.7		80.1		75.6	

# WATER-BASED SEALER » APPLICATION TESTING

After stain application

After wash, rinse, & dry



1 hr



4 hr



CONTROL

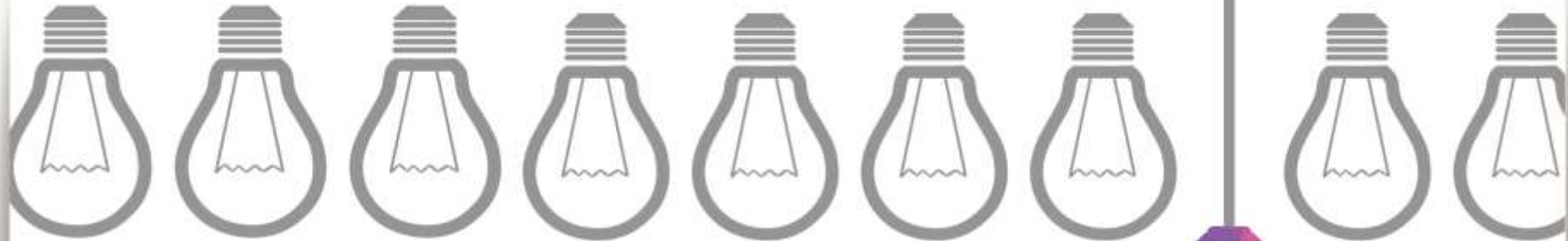
LMWSP

CONTROL

LMWSP

**ANTI GRAFFITI**

**APPLICATION TEST**



# CONCLUSIONS

- **Successful Production** of multifunctional self-crosslinking emulsion possible using novel surfactant package
- **Particle Size** significantly smaller than current technology
  - Opens up application space
    - Architectural and industrial coatings
    - Coatings and sealers for porous substrates
    - Future? Digital inks (sub-micron)
  - Allows for better distribution of additive throughout paint films
    - Improved blocking at lower levels possible
    - Improved DPUR at lower levels possible
- **Stability**/shelf-life significantly improved



# PLEASE **CONTACT** US WITH ANY QUESTIONS:

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