Surfactants for Emulsion Polymerization

Presented by Bruno Dario
June 20th, 2024

AGENDA

Surfactants for Emulsion Polymerization

OUR PURPOSE

We enhance our relationships

Reimagining Chemistry Together to Create A Better World

Constant search for innovation

Value creation through sustainability

Purpose word choice explanation:

Why we exist

Over the last 20 years, IVL has **acquired** and **integrated more than 50** companies into its business

One of the world's leading producers of PET with

Presence in
35 countries

Consolidated revenues of
US$ 18.7 bi
(end of 2022)

Manufacturing Units
147

Employees
+ 26k

Purpose
REIMAGINING CHEMISTRY TOGETHER TO CREATE A BETTER WORLD

Combined PET*

Global leader in the production of PET and r-PET

*Combined PET - Feedstock, PTA, PET, Packaging and Recycling

FIBERS

Polyester fibers and yarns grouped into five categories: Home, Apparel, Hygiene and Medical, Automotive, and industrial/ technical.

INDOVINYA

Leader in the production of non-ionic surfactants in the Americas
Leader in EO production in the Americas
Second largest ethoxylation company globally

WHERE WE ARE

- **18** industrial units
- **8** countries
- **9** R&D centers
- **3,700** employees

Origin and Sustainability

Emulsion Polymerization

Definitions

When we talk about **EMULSION POLYMERIZATION** it is important to remember that…

EMULSION

- **Liquid dispersed in another liquid**
  - E.g., Mayonnaise
- **Monomers emulsified in water**

AS WE POLYMERIZE

- Monomers will create a polymer, which is a solid stabilized in water. A **DISPERSION** or a **COLLOID**
  - E.g., Natural Latex

SO, TODAY

You will hear me talking about:

- Latex (synthetic)
- Emulsion Polymer
- Emulsion
- Resin
- Binder
- Dispersion

Origins and Importance

Emulsion Polymerization

World War II – shortage of natural rubber

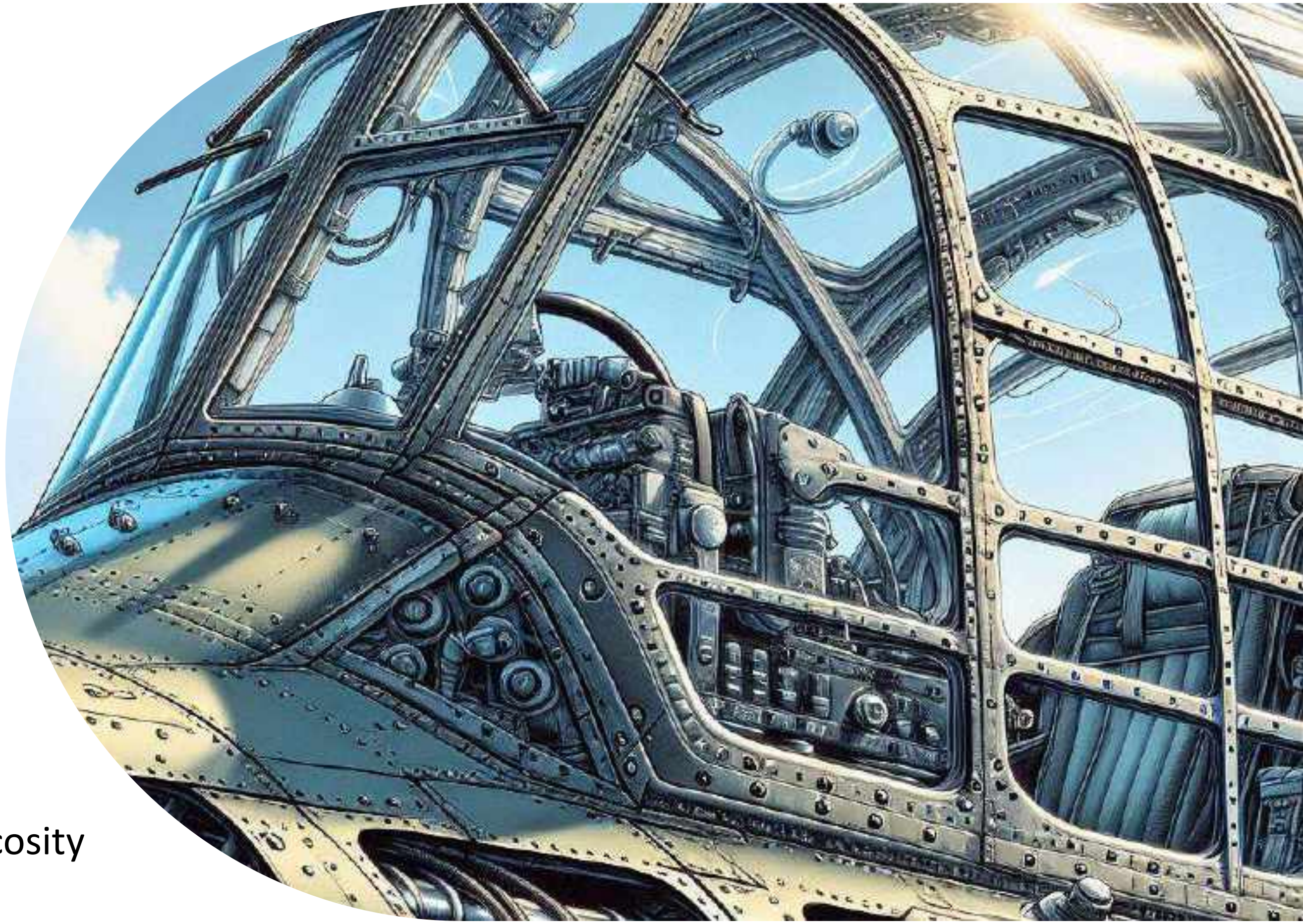
- Solution: free radical polymerization

Versatility

- Several types of polymers that can be made
- Wide variety of applications

Advantages:

- Remove the heat of polymerization
- High control of particle size and particle morphology
- High polymer concentration and high M.W. with low viscosity
- Environmental regulations: low VOC capabilities



Sustainability Key Drivers

CASE Market

Reduce **environmental** and **social impact**	Efficient use of **resources**	Enhance **durability** of **materials**
Reduce VOC emissions	**Energy efficiency** in **production processes**	**Enhance** short & long-term performance
Biobased alternatives	High **performance** in the **application**	
HSE friendly formulations		

Polymers and Process

Emulsion Polymerization

General Formulation

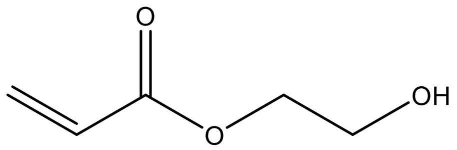
Emulsion Polymerization

	% Weight	
Ingredients	**Minimum**	**Maximum**
Water	50.0	55.0
Surfactant	0.5	5.0
Monomer	40.0	50.0
Initiator	0.2	0.5

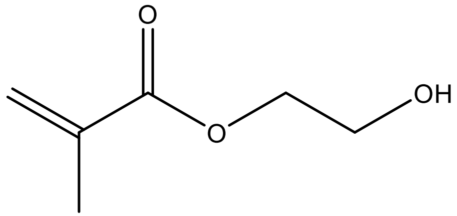
Typical Monomers

Emulsion Polymerization

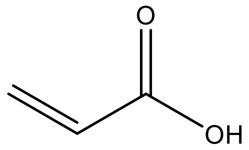
Monomer	Tg (°C)	Affinity for Water
2-Ethylhexyl acrylate	-64	Hydrophobic
N-butyl acrylate	-54	Hydrophobic
Methyl acrylate	8	Hydrophobic
Vinyl acetate	32	Hydrophilic
Styrene	100	Hydrophobic
Methyl methacrylate	106	Hydrophilic



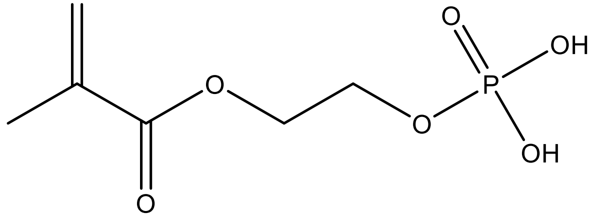
2-Hydroxyethyl acrylate



2-Hydroxyethyl methacrylate



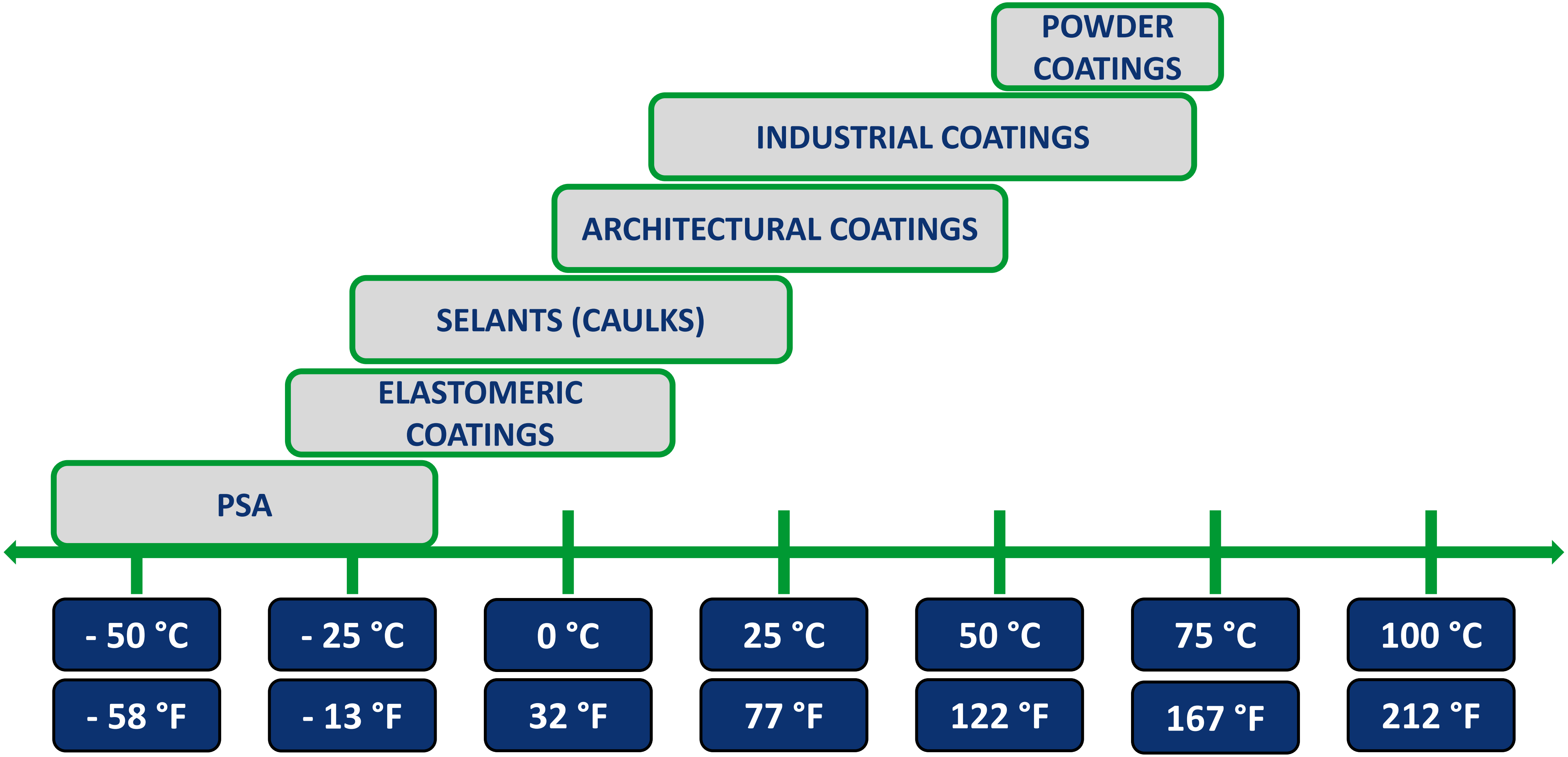
Acrylic Acid



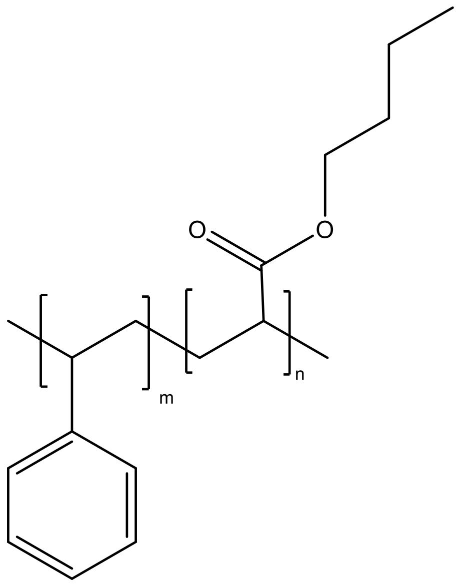
HEMA Phosphate

Application and Tg

Acrylic polymers

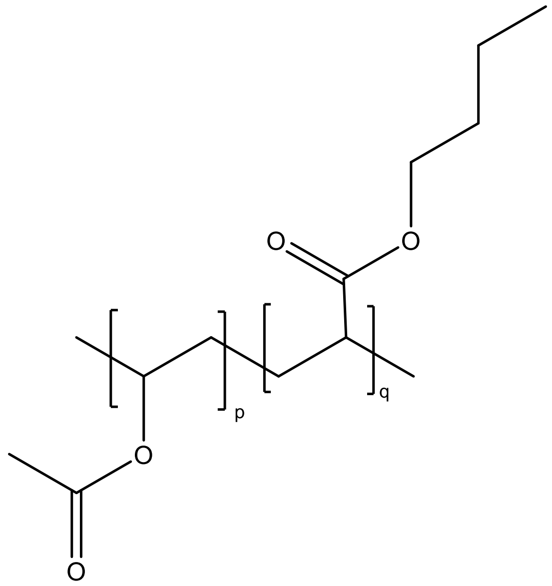


Common Polymers applied in Architectural/Industrial Coatings



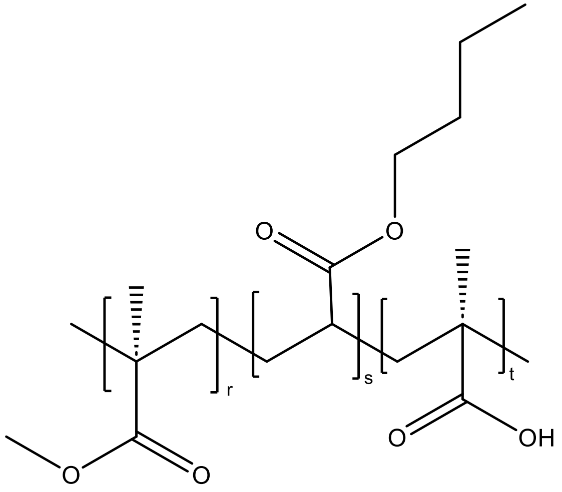
Styrene-acrylic

- Excellent hydrophobicity
- Excellent adhesion to metal/plastic
- Cost competitive
- Poor UV / durability



Vinyl-acrylic

- Low-VOC capable (hydroplasticization)
- Good scrub resistance
- Good balance of performance and cost
- High solubility of vinyl-acetate



All-acrylic

- Premium resins
- Excellent durability / weatherability
- High chemical and oil resistance
- Can be more expensive

Diverting a little bit from the topic…

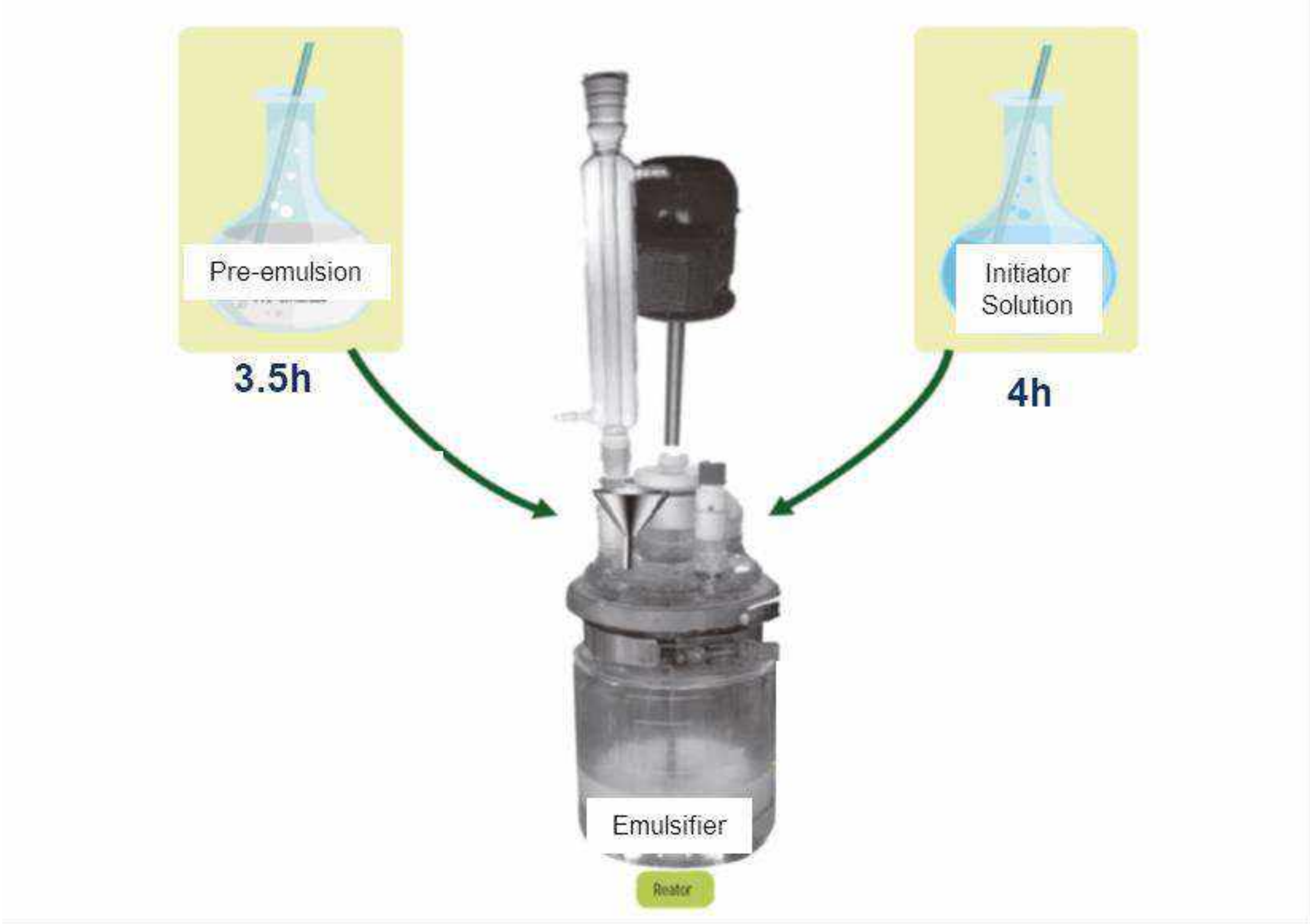
How do acrylics compare to other polymers applied in Architectural / Industrial Coatings?

Resin	UV Resistance	Chemical Resistance	Hardness	Flexibility	Heat Resistance	Cost
Air Dry Alkyd	G to VG	P to G	G to VG	P to G	P to G	VG to E
Alkyd Melamine	G to VG	G to VG	G to VG	G	G to VG	G to VG
Alkyd Urethane	VG	G to VG	G to VG	G	P to G	P to G
Polyester Melamine	VG to E	G to VG	G to VG	VG to E	G to VG	G to VG
Polyester Urethane	E	G to VG	G to VG	VG to E	P to G	P to G
PUD	VG to E	P to G	G to VG	VG to E	P to G	P to G
Acrylic Latex (1K)	VG to E	G to VG	G to VG	P to G	P to G	VG to E
Acrylic Melamine	VG to E	G to VG	VG to E	G to VG	G to VG	G to VG
Acrylic Urethane	VG to E	VG to E	VG to E	VG to E	G to VG	P to G
Epoxy Amide	P	E	E	P to G	G to VG	G to VG

P= Poor, G=Good, VG=Very Good, E=Excellent

Emulsion Polymerization

Simplified setup



All-Acrylic Recipe Example

1. Prepare **Initial charge** and add to the reactor.
2. Heat up the reactor to 80-85°C under N2 atmosphere.
3. Charge 5 wt.% of **Monomer** Pre-emulsion in the reactor.
4. After this, add the **First Initiator Batch** in the reactor.
5. Keep the reaction for 30 minutes at 80-85°C.
6. Add 95 wt.% of **Monomer Pre-emulsion** for 3.5 hours.
7. Add 95 wt.% of **Initiator Batch** for 3.5 hours.
8. After finishing the addition of **Monomer Pre-emulsion** and **Initiator Batch**, keep the reaction for 30 **minutes at 80-85°C.**
9. Lower the temperature of reaction medium to 60°C.
10. Add **Post-oxidizer** and **Post-reducer** simultaneously for 30 minutes in order to consume the free monomer.
11. Lower the temperature of reaction medium to 50°C.
12. Adjust the pH to 8-9.
13. Discharge the latex from the reactor and filter in a 200 Mesh sieve.

Parameters and Properties of Emulsion Polymers

Emulsion Polymerization

Common Process Issues

- Clot dispersed in the latex
- Polymer buildup on reactor surface
- Conversion time
- …

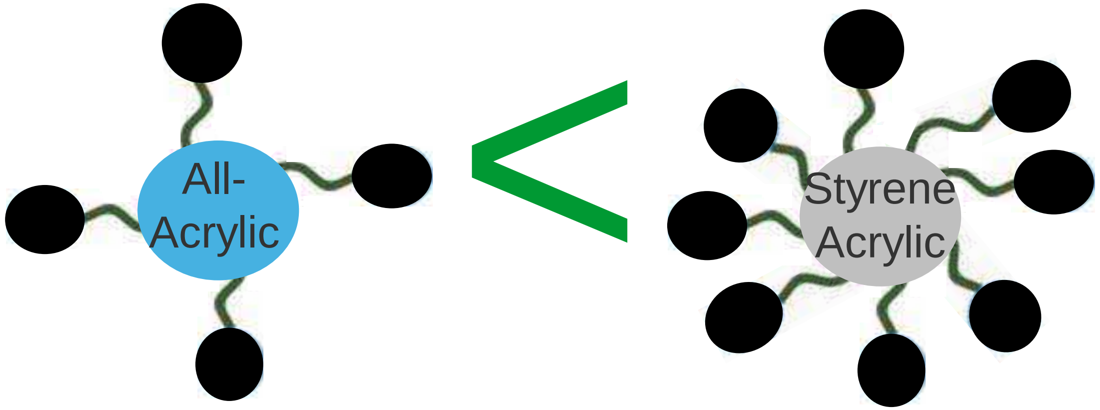
Emulsion Polymer Properties

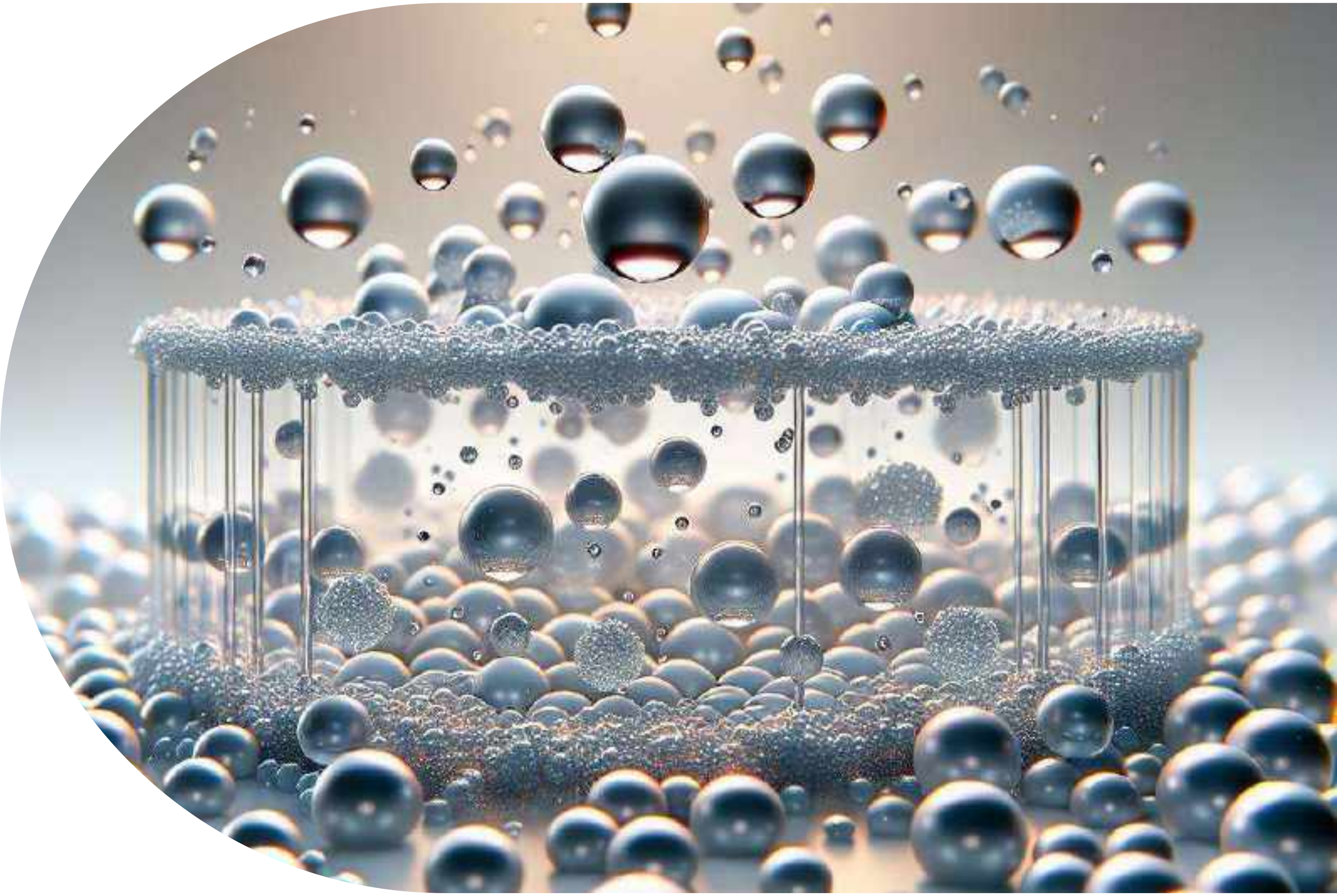
- Residual Monomer
- pH
- Solid Content
- Particle Size
- Viscosity
- Molecular Weight
- Tg
- Zeta Potential
- Mechanical Stability
- Electrolytic Stability
- …

Stability and Surfactants

Emulsion Polymerization

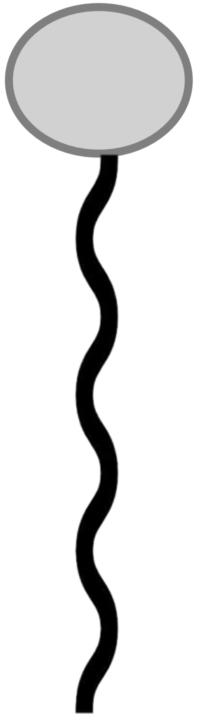
Colloidal Stability





Source: M. El-Aasser and P. Lovell, Emulsion Polymerization and Emulsion Polymers, Wiley, West Sussex, 1997

Surfactants – SURFACE ACTIVE AGENTS

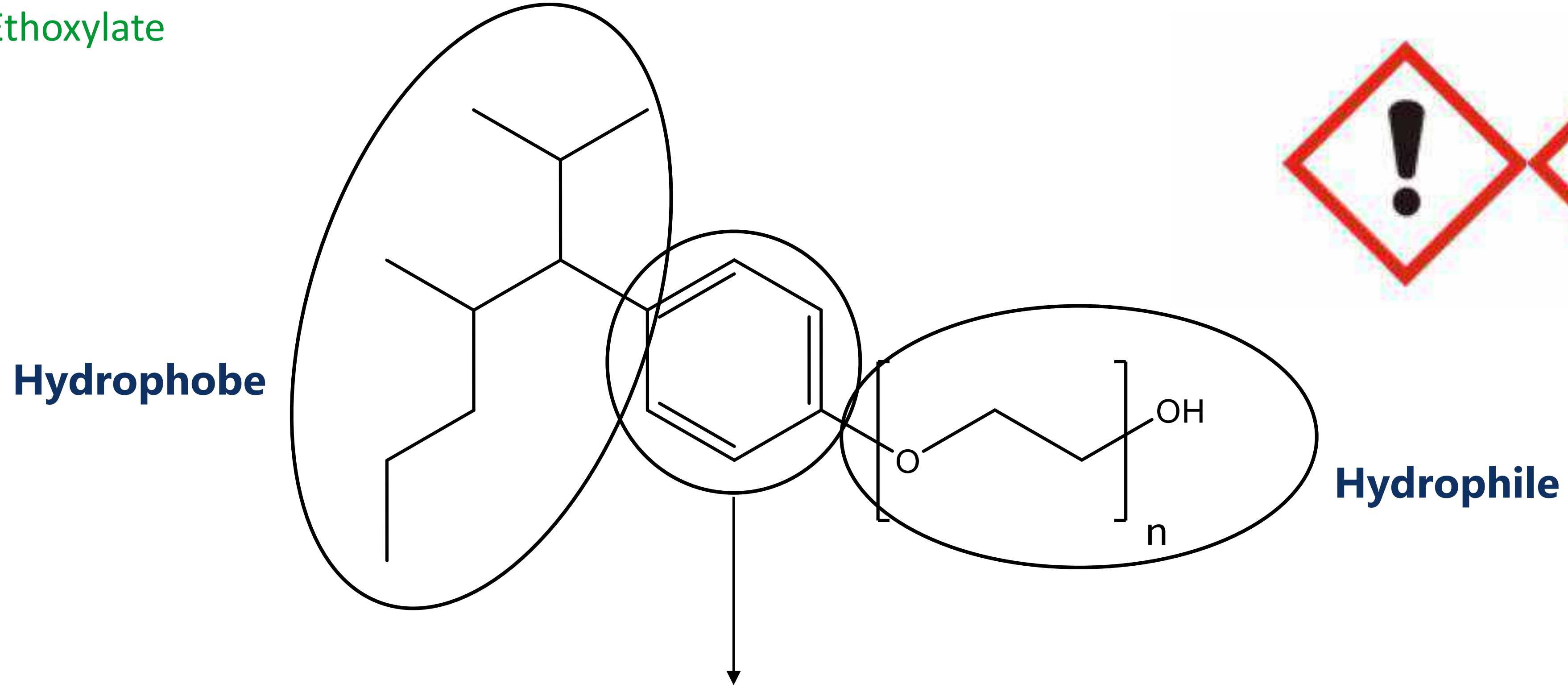


- **Hydrophilic Polar Head (Affinity with water)**
  - Charged
    - Anionic
    - Cationic
  - Neutral
- **Hydrophobic Nonpolar Tail (No affinity with water)**
  - Aromatic
  - Aliphatic

*Linear or Branched

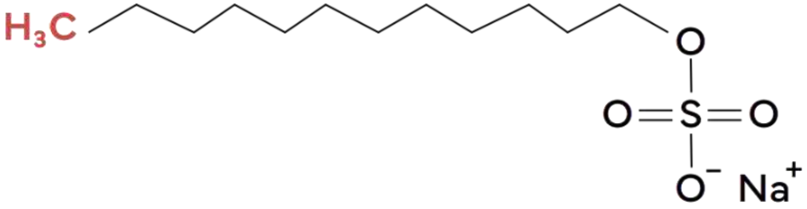
When talking about surfactants... The most "famous" one:

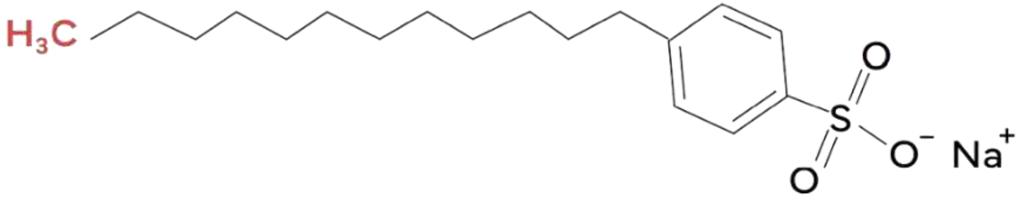
Alkylphenol Ethoxylate

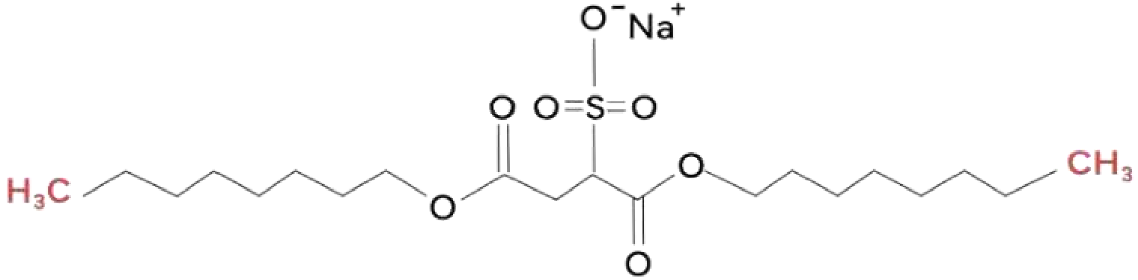


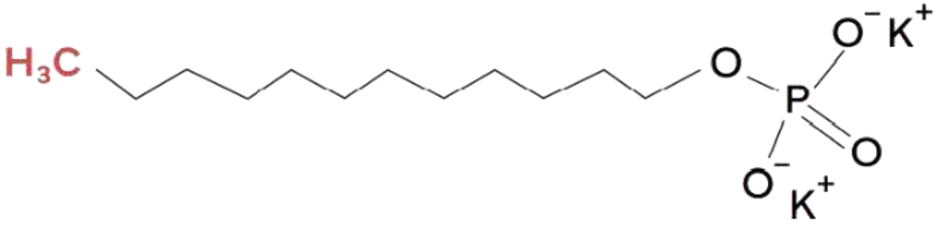
- Polarizable Aromatic Ring
- Dense electronic cloud
- Creates a soft transition from hydrophobic to hydrophilic

Anionic Surfactants

(A) 

(B) 

(C) 

(D) 

Function:

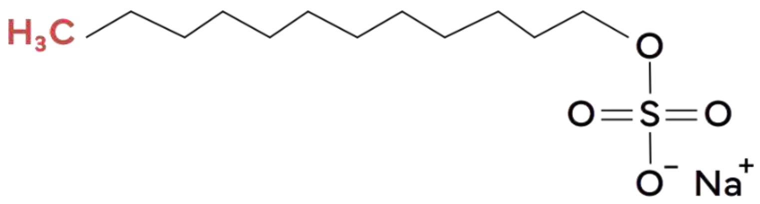
- Monomer emulsification
- Particle Nucleation

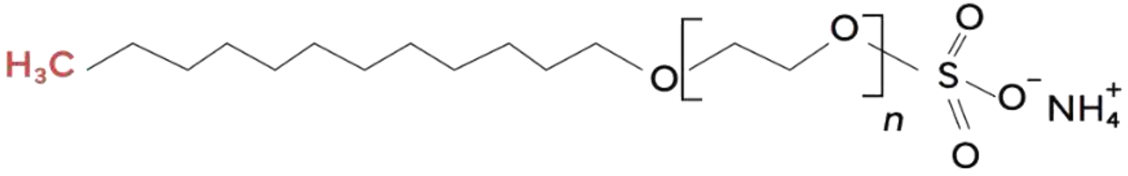
  $Nc \propto [Surfactant]^{0.6}$
- Polymerization speed
- Particle size distribution
- Clot formation
- Shelf-life stability
- Sensitive to salt concentration

(A) *Sodium salt of sulfated lauryl alcohol*
(B) *Sodium dodecylbenzene sulfonate*
(C) *Sodium dioctyl sulfosuccinate*
(D) *Potassium salt of phosphate lauryl alcohol*

APE-free Anionic Surfactants

Tailoring surfactants to fit our customer's needs

(A) 

(B) 

(A) *sodium salt of sulfated lauryl alcohol*

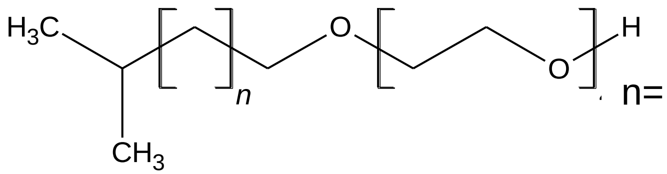
(B) *ammonium salt of ethoxylated lauryl alcohol*

PROPERTIES	OXIMULSION® 1228	OXIMULSION® 1328 APH	OXIMULSION® 11230
EO Content*	Low	Low	Medium
Appearance	Liquid	Liquid	Liquid
Solids, wt.%	28	28	28
CMC, g/L	0.27	0.28	0.34
S.T., 0.1%, mN/m	35	38	46

* ***Low***: *0-10 EO*
Medium: *11-24 EO*
High: *> 25 EO*

Nonionic Surfactants

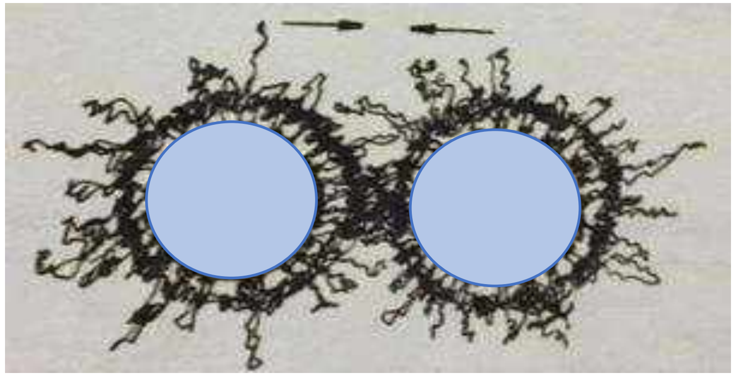
n= 9 - 40



n= 9 - 40

Function

- Electrolytic Stability
- Stability during neutralization
- Mechanical Stability
- Freeze-thaw Stability
- Shelf-life



Higher steric repulsion is obtained when:

- Thick and dense layers
- Strong adsorption
- Complete coverage

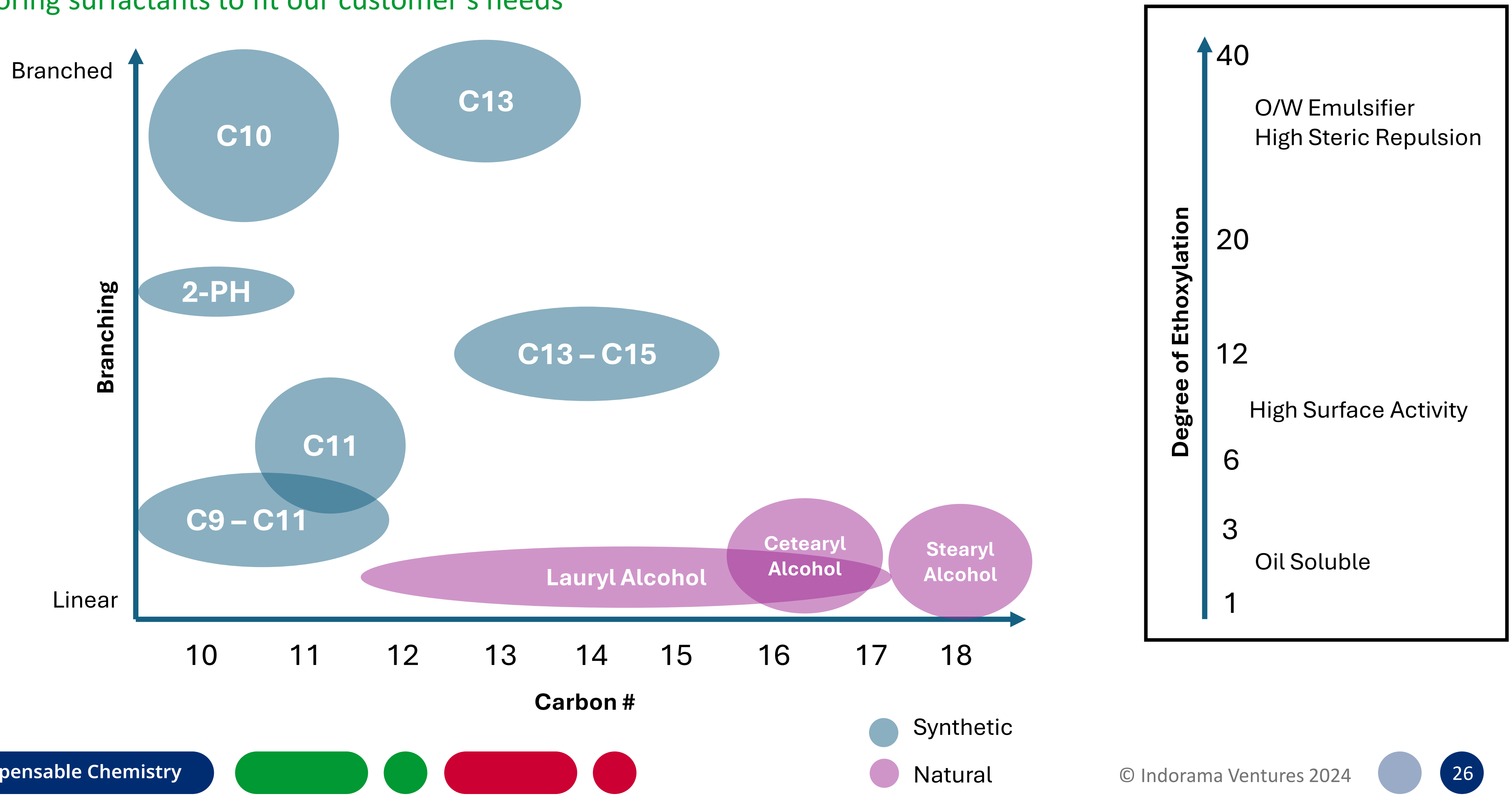
APE-free Nonionic Surfactants

Tailoring surfactants to fit our customer's needs

Hydrophobe	Type	Size	Structure	Distribution	Sourcing
• Fatty Alcohols • Synthetic Alcohols • Sorbitan esters	• EO • PO • EO/PO	• # of Carbons • EO units • PO units	• Linear • Branched	• Broad range • Narrow range	• Natural • Synthetic

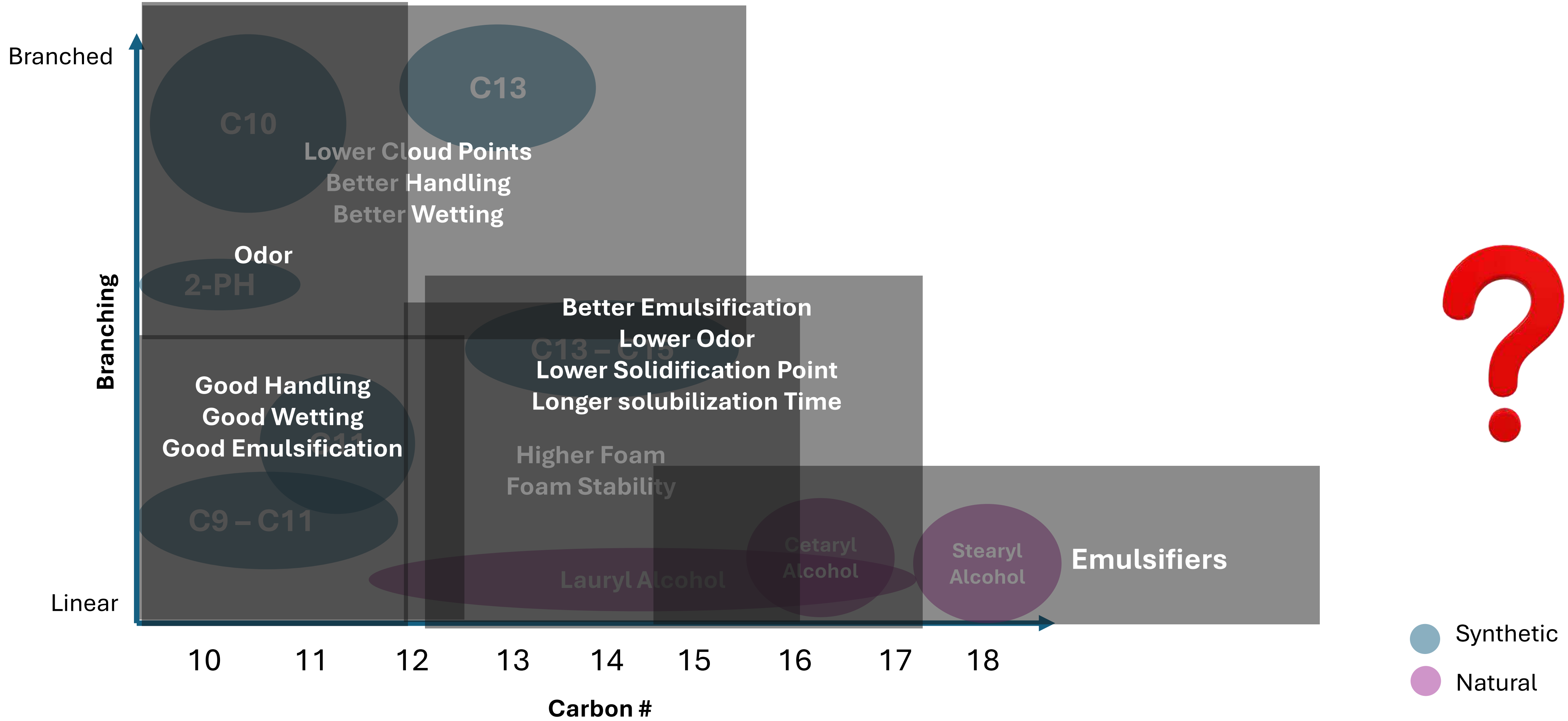
Alcohol Ethoxylates

Tailoring surfactants to fit our customer's needs



Alcohol Ethoxylates

Tailoring surfactants to fit our customer's needs



What to consider when choosing surfactants

Performance

- Clot Formation
- Particle Size and Control during the process

↑ Electrolyte Stability

↑ Mechanical Stability

↑ Water Resistance

Sustainability Attributes

↑ BIODEGRADABILITY

↓ TOXICITY

↑ [Renewable Content]

↑ Ease of processing / energy savings

Anionic Surfactants

Emulsion Polymerization

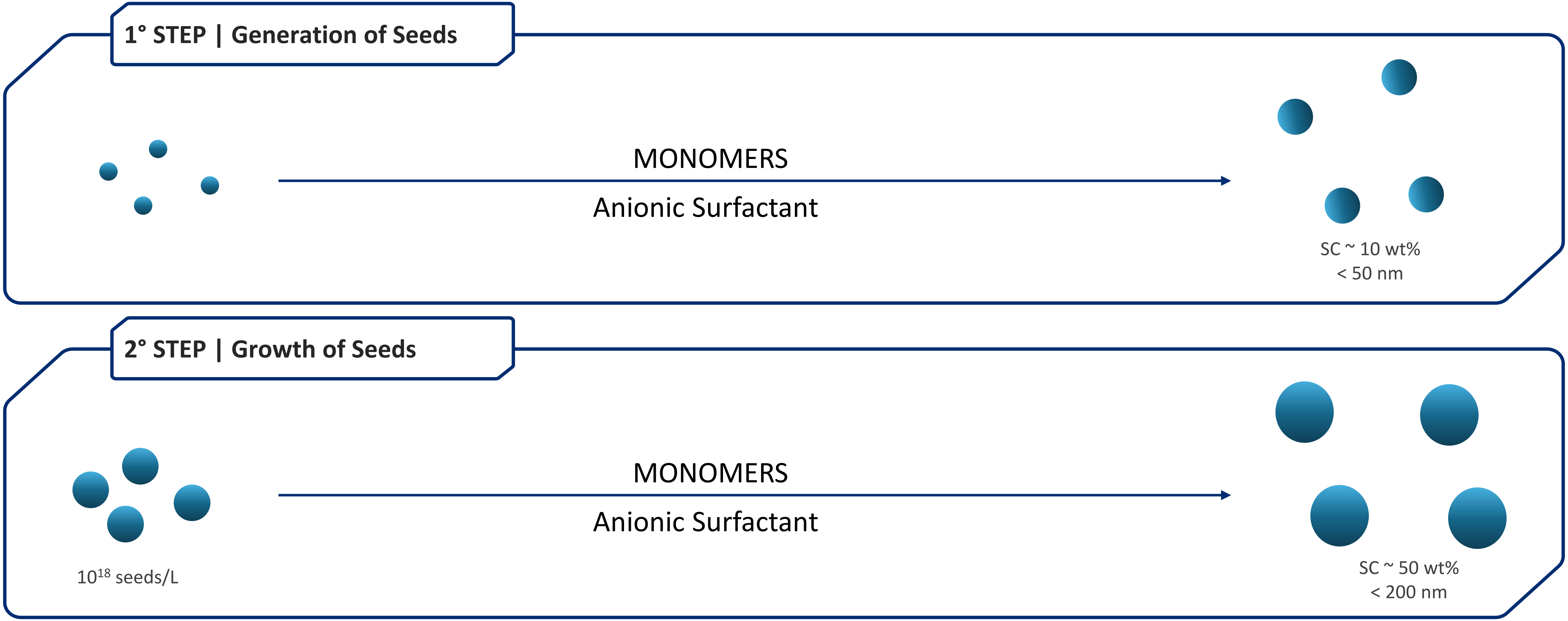
Anionic Surfactants

PROPERTIES	OXIMULSION® 1228	OXIMULSION® 1328 APH	OXIMULSION® 11230	OXIMULSION® 2742
Description	Sulfated	Sulfated	Sulfated	Phosphate
EO content*	Low	Low	Medium	Low
Appearance	Liquid	Liquid	Liquid	Liquid
Solid Content, wt.%	28	28	28	> 95
Solvent	Water	Water	Water	Water
pH	7	11	8	3
CMC, g/L	0.27	0.28	0.34	0.26
Surface tension, 0.1%, mN/m	35	38	46	28

** **Low**: 0-10 EO*
***Medium**: 11-24 EO*
***High**: > 25 EO*

Two Step Process

Emulsion Polymerization

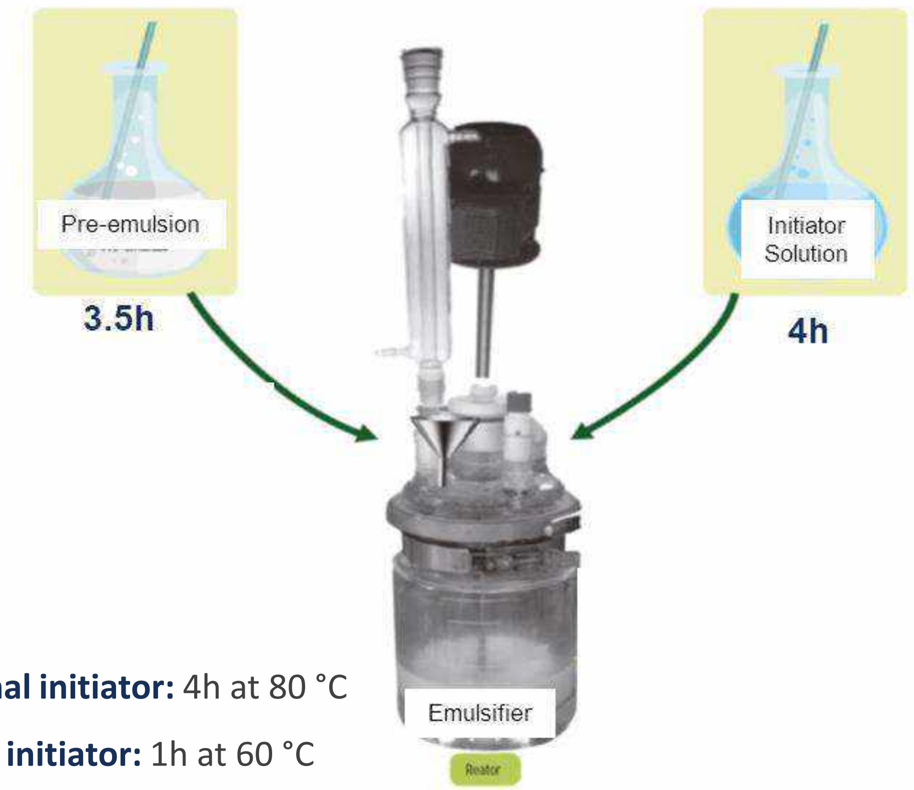


Source: Asua et. Al., Langmuir 2003, 19, 3212 - 3221.

Formulations

Emulsion Polymerization

Components (phm)	Two Step Process	
	Step 1	Step 2
Methyl methacrylate	50	50
Butyl acrylate	48	48
Methacrylic acid	1	1
APE-free anionic surfactant	**25**	**2**
Buffer	0.7	0.7
Ammonium persulfate	0.6	0.6
Oxidizing agent	---	0.04
Reducing agent	---	0.04



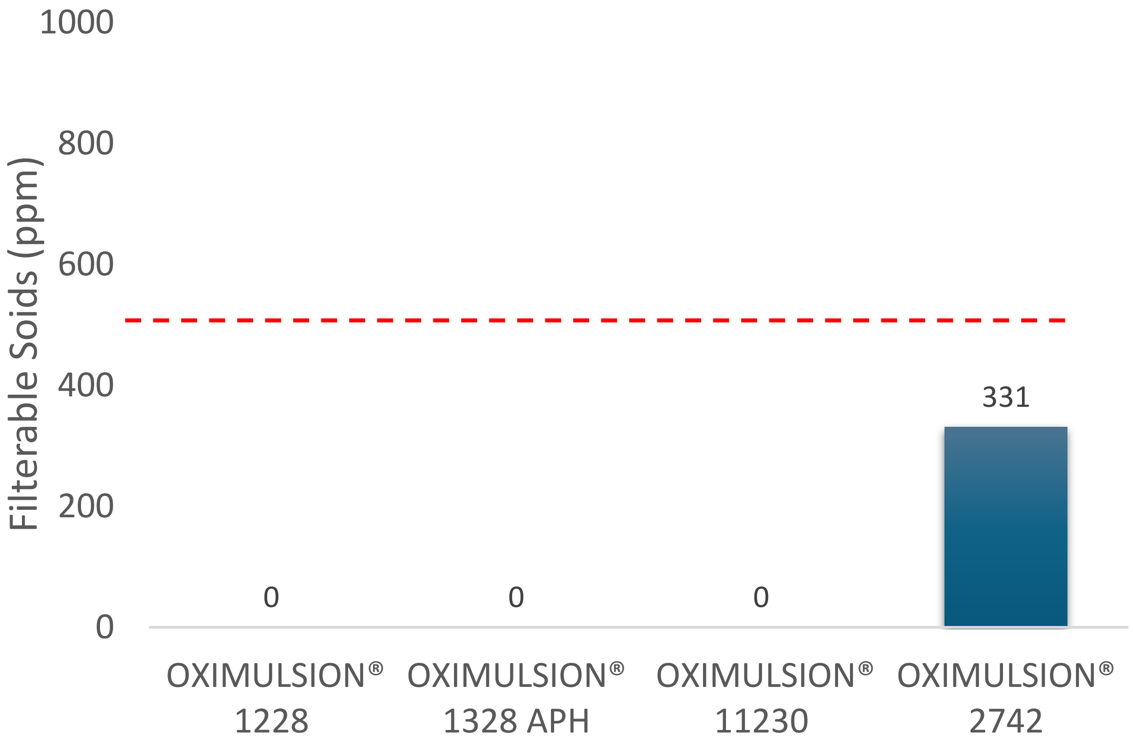
- **Thermal initiator:** 4h at 80 °C
- **Redox initiator:** 1h at 60 °C
- **Neutralizer:** NH_4OH

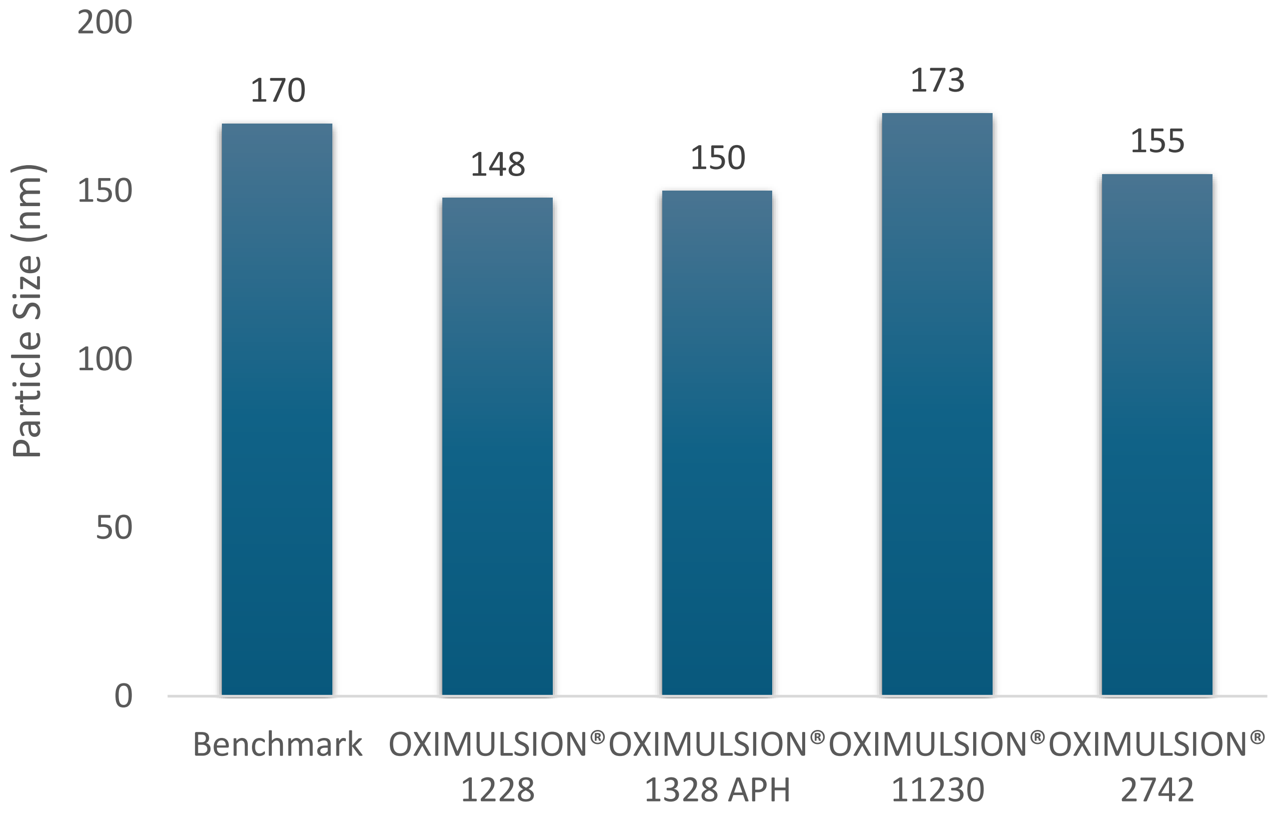
Formulations

Emulsion Polymerization

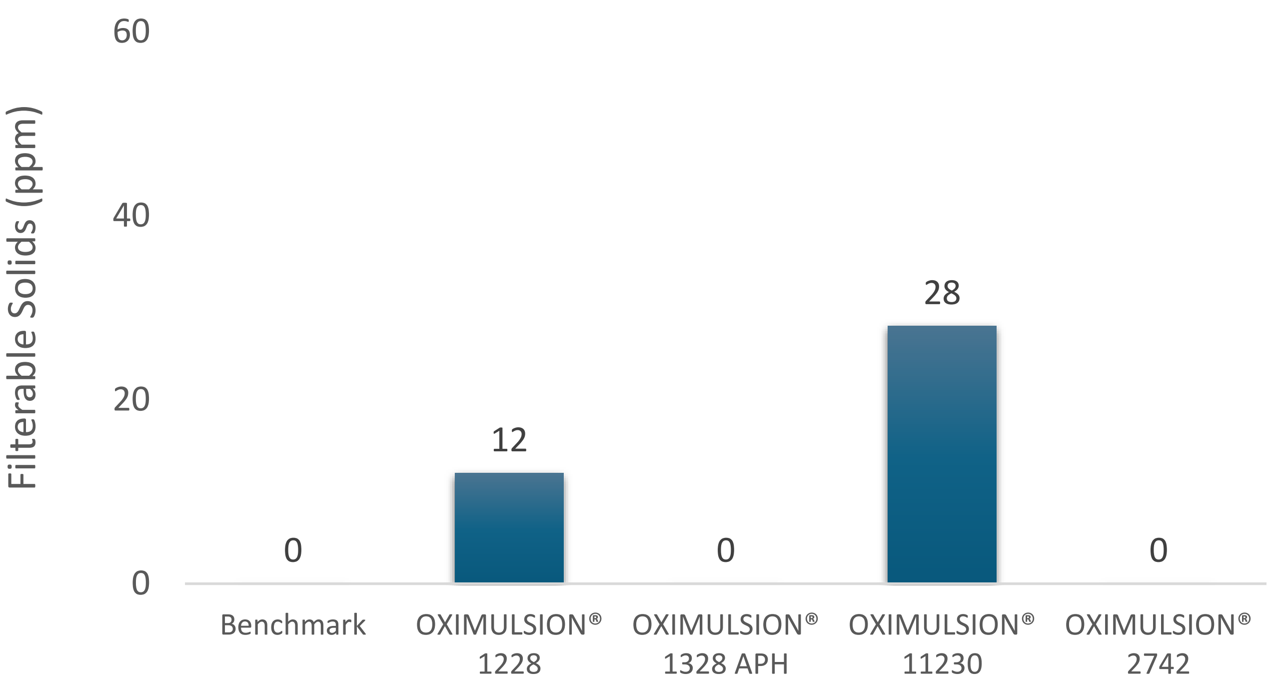
		Seeded Process			
Properties	**Benchmark**	**OXIMULSION® 1228**	**OXIMULSION® 1328 APH**	**OXIMULSION® 11230**	**OXIMULSION® 2742**
Solid Content, wt.%	50	50	50	50	50
pH	9	9	9	9	9
Viscosity, 25°C, cP	300	281	235	186	185
Zeta Potential, mV	-77	-60	-59	-58	-56
Freeze-thaw stability (Cycles)	5	5	5	5	5

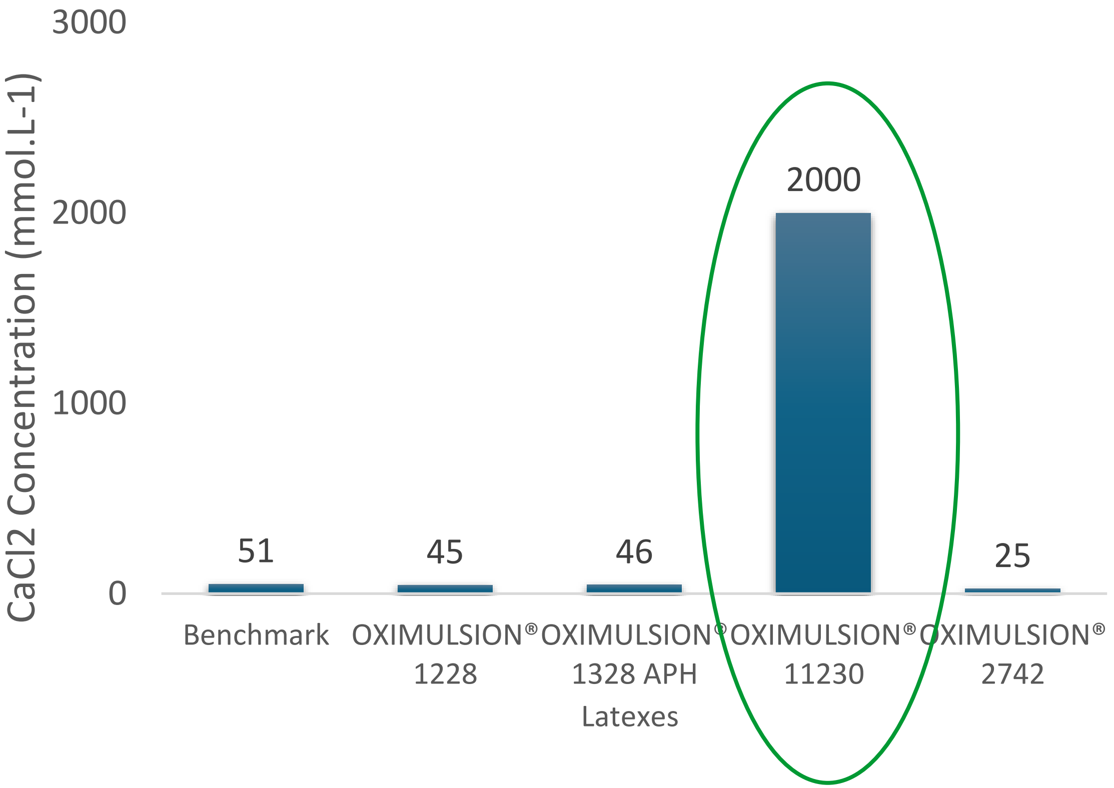
Effect of surfactant on coagulum formation in the reactor





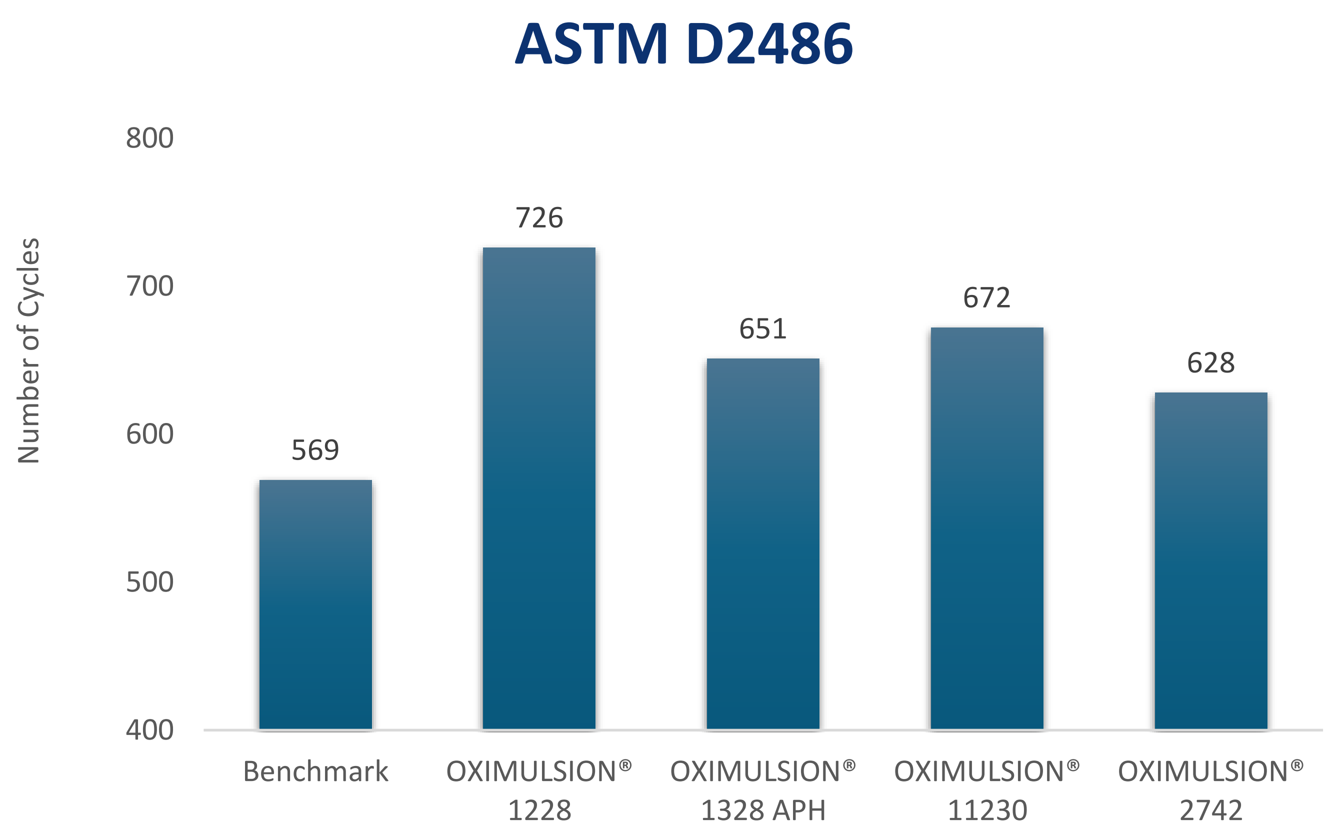
Effect of surfactant on mechanical stability





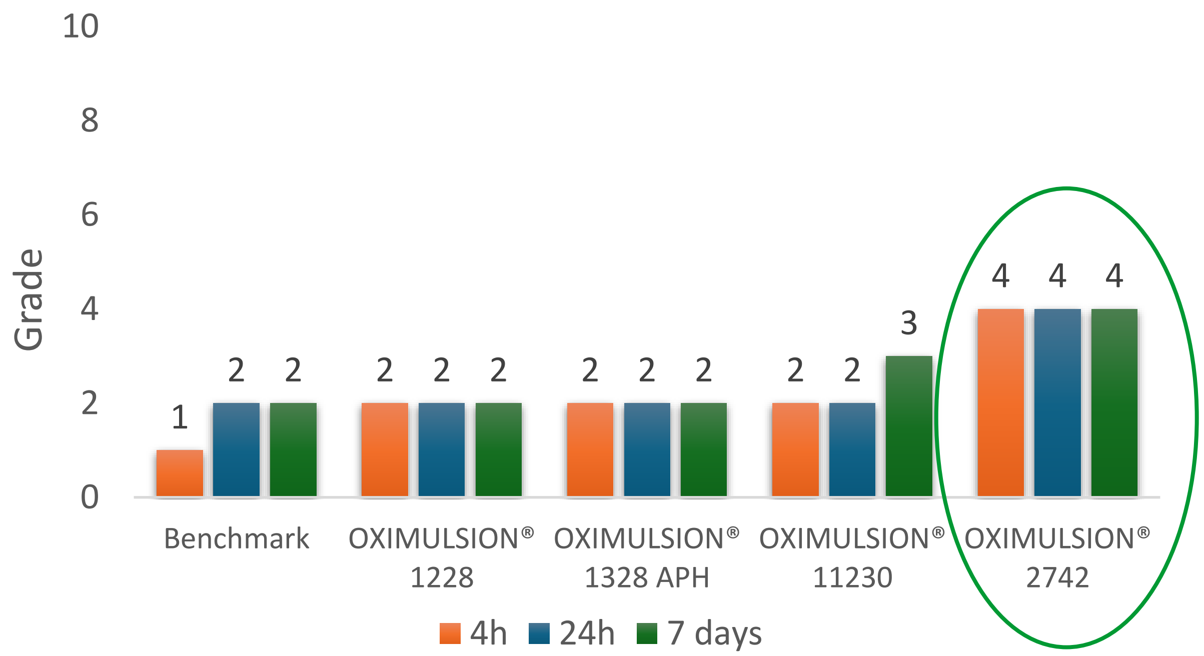
Effect of surfactant on wet scrub resistance of 30% PVC paint

Emulsion Polymerization

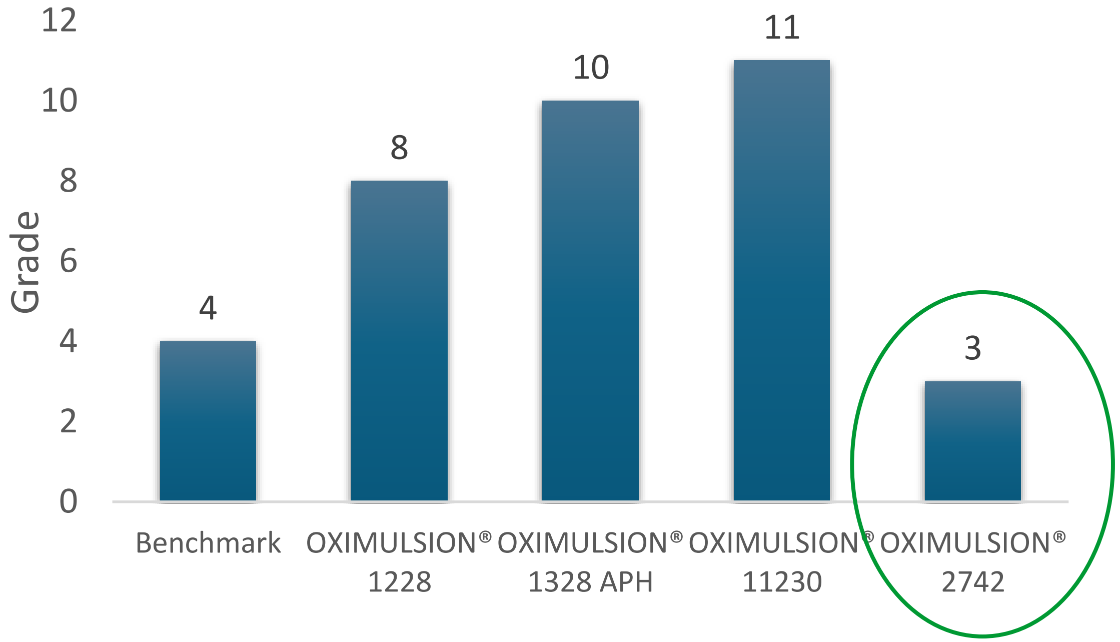


Effect of surfactant on blocking evolution of 30% PVC paint

ASTM D4946



Dirt Pickup
Rating: 1-11 / (1=Best)



Nonionic Surfactants

Emulsion Polymerization

Vinyl-Acrylic Emulsion

SURFONIC® NB-407

Physical-Chemical Properties

- Nonylphenol ethoxylated
- 40 EO
- HLB of 17.8
- 70 wt.% of solid content
- CMC of 0.76 g/L
- Surface tension of 40 mN/m (0.1 wt.%, 25°C)

OXITIVE® 7110

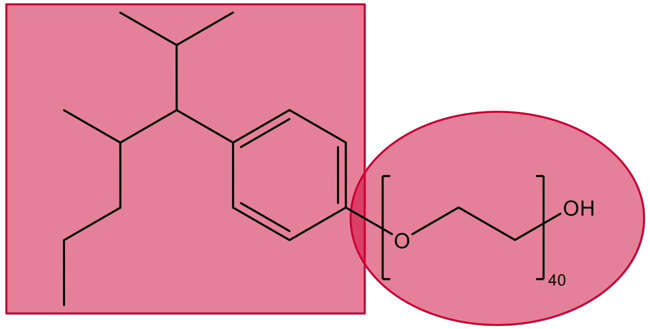
Physical-Chemical Properties

- Fatty alcohol ethoxylated
- Medium degree of ethoxylation
- HLB of 16.9
- 60 wt.% of solid content
- CMC of 0.19 g/L
- Surface tension of 40 mN/m (0.1 wt.%, 25°C)

OXITIVE® 7140

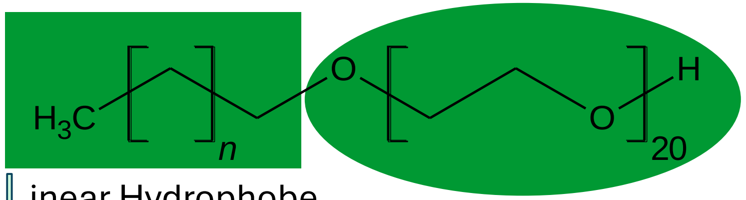
Physical-Chemical Properties

- Synthetic alcohol ethoxylated
- High degree of ethoxylation
- HLB of 18.0
- 70 wt.% of solid content
- CMC of 1.37 g/L
- Surface tension of 37 mN/m (0.1 wt.%, 25°C)



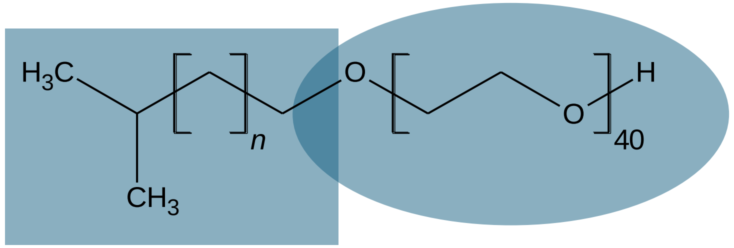
4-Nonylphenol

40 Ethylene Oxide repetitions



Linear Hydrophobe

20 Ethylene Oxide repetitions



Branched Hydrophobe

40 Ethylene Oxide repetitions

Vinyl-Acrylic Emulsion

SURFONIC® NB–407

Components	Weight (%)
Initial Charge	
Water	34.6
Buffer	0.2
HEC	0.2
ALKOPON® NNP 940 HS	0.2
SURFONIC® NB-407	3.4
Monomer Pre-emulsion	
Vinyl Acetate	42.4
Butyl Acrylate	10.6
First Initiator Batch	
Water	1.6
Potassium Persulfate	0.06
Reducer Batch	
Water	2.6
Sodium Metabisulfite	0.06
Post-Oxidizer	
Water	0.6
t-BHP	0.05
Post-Reducer	
Water	0.6
Sodium Metabisulfite	0.04
Total	100

OXITIVE® 7110

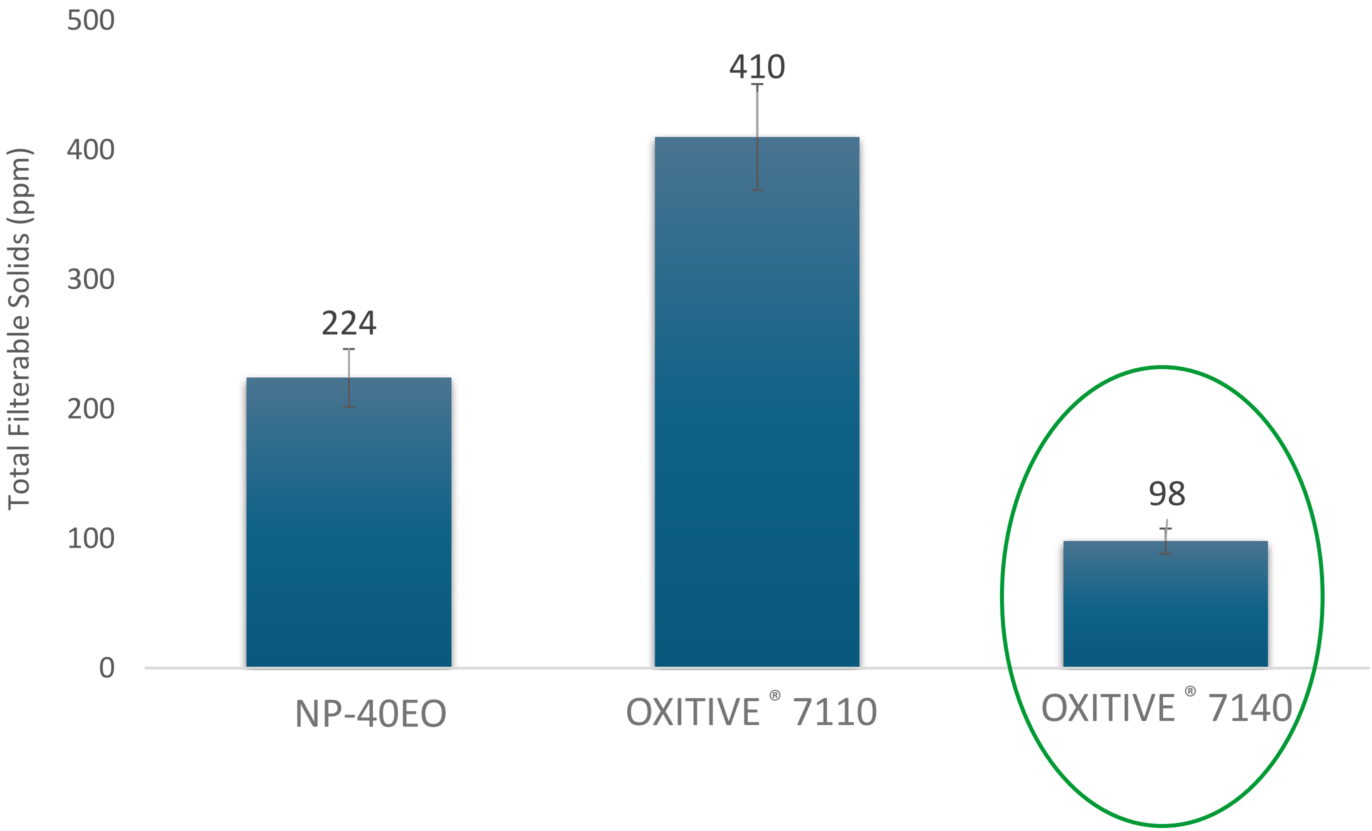
Components	Weight (%)
Initial Charge	
Water	34.6
Buffer	0.2
HEC	0.2
OXIMULSION® 1128	0.3
OXITIVE® 7110	4.0
Monomer Pre-emulsion	
Vinyl Acetate	42.4
Butyl Acrylate	10.6
First Initiator Batch	
Water	1.6
Potassium Persulfate	0.06
Reducer Batch	
Water	2.6
Sodium Metabisulfite	0.06
Post-Oxidizer	
Water	0.6
t-BHP	0.05
Post-Reducer	
Water	0.6
Sodium Metabisulfite	0.04
Total	100

OXITIVE® 7140

Components	Weight (%)
Initial Charge	
Water	34.5
Buffer	0.2
HEC	0.2
OXIMULSION® 1228	0.3
OXITIVE® 7140	3.4
Monomer Pre-emulsion	
Vinyl Acetate	42.4
Butyl Acrylate	10.6
First Initiator Batch	
Water	1.6
Potassium Persulfate	0.06
Reducer Batch	
Water	2.6
Sodium Metabisulfite	0.06
Post-Oxidizer	
Water	0.6
t-BHP	0.05
Post-Reducer	
Water	0.6
Sodium Metabisulfite	0.04
Total	100

Effect of different nonionic surfactants

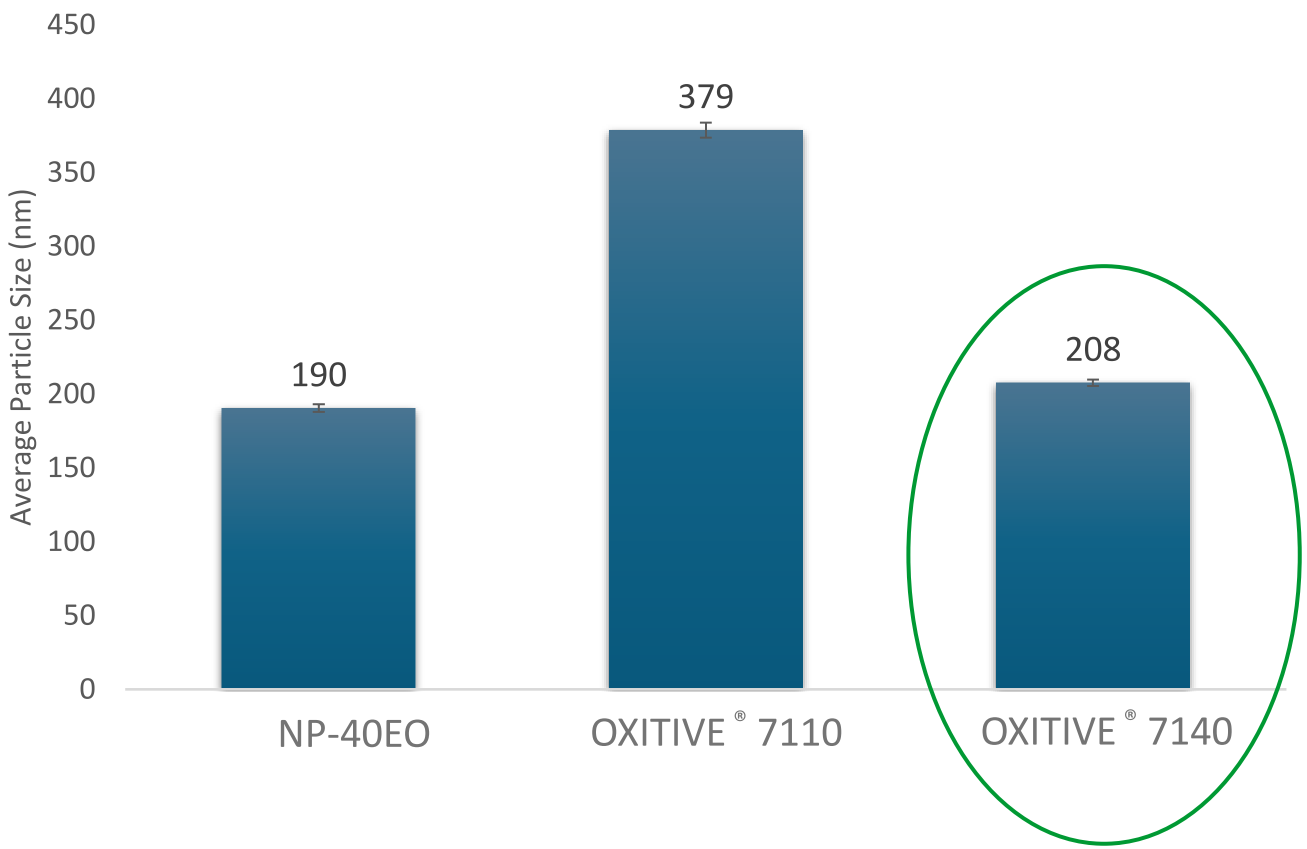
Reactor Cleanliness



- **Clot formation < 500 ppm** for all formulations.
- **Longer ethylene oxide chain** in the nonionic **improved** the performance even compared against reference.

Effect of different nonionic surfactants

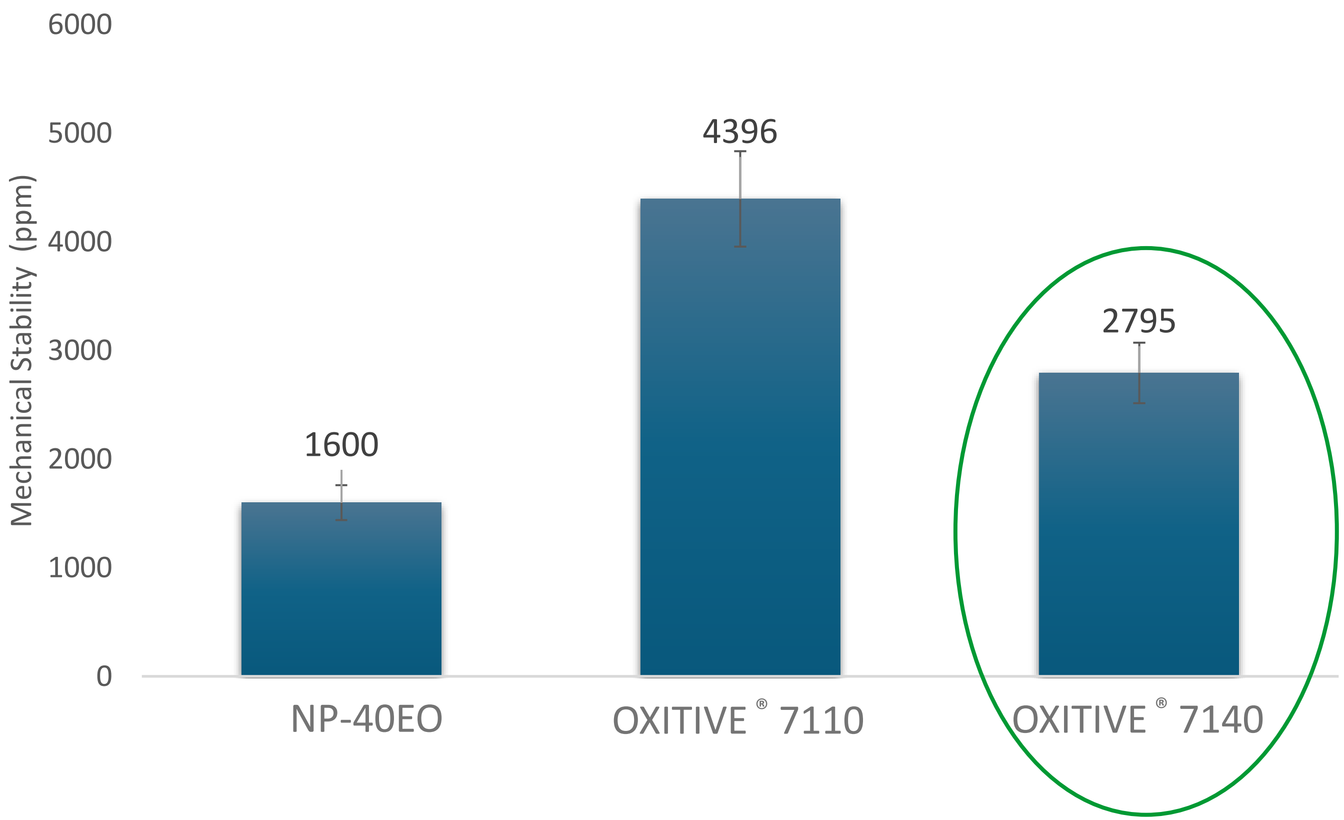
Particle Size



- Acceptable particle size range is **200 – 400nm.**
- **Longer ethylene oxide chain in the nonionic surfactant** tends to decrease the particle size.

Effect of different nonionic surfactants

Mechanical Stability



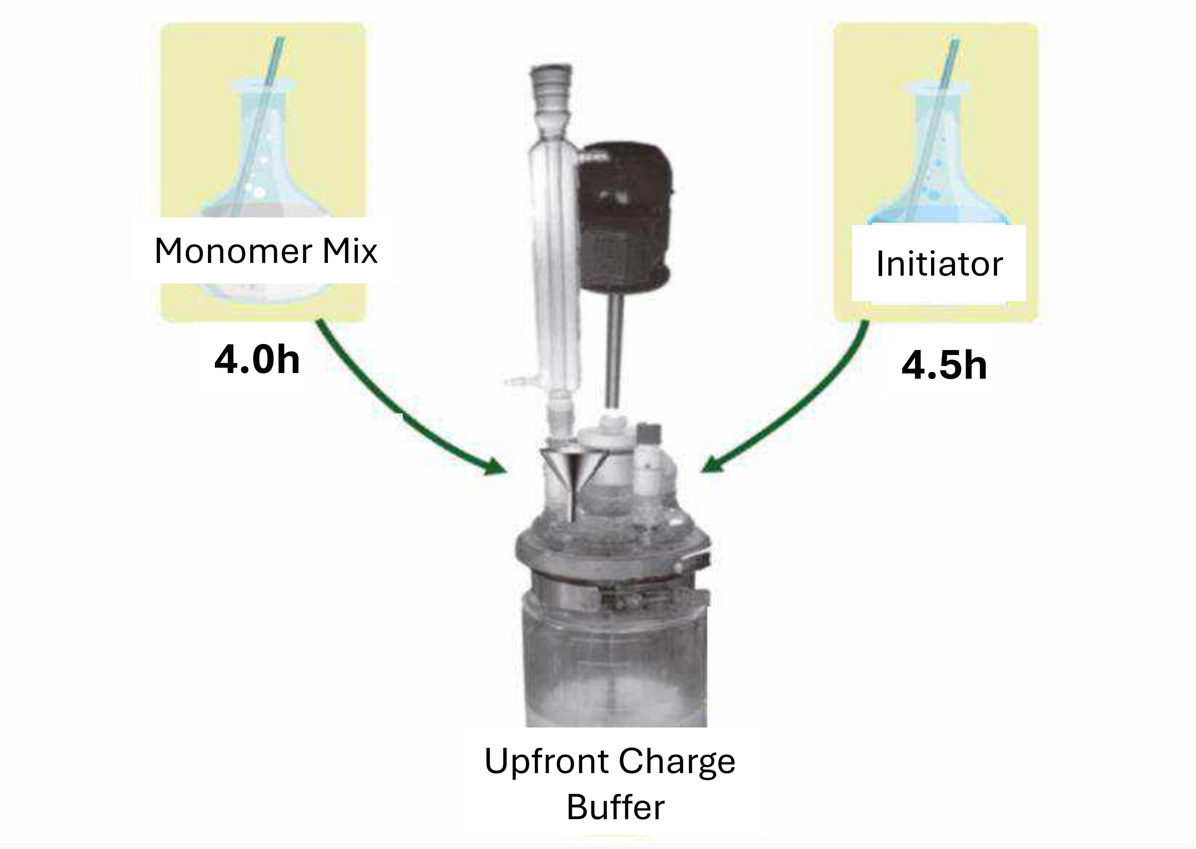
- **Longer EO chain in the nonionic** improved the mechanical stability;

Emulsion Polymer Formulation

Goal: to evaluate different ratios of nonionic to anionic surfactants:

95:5 / 85:15

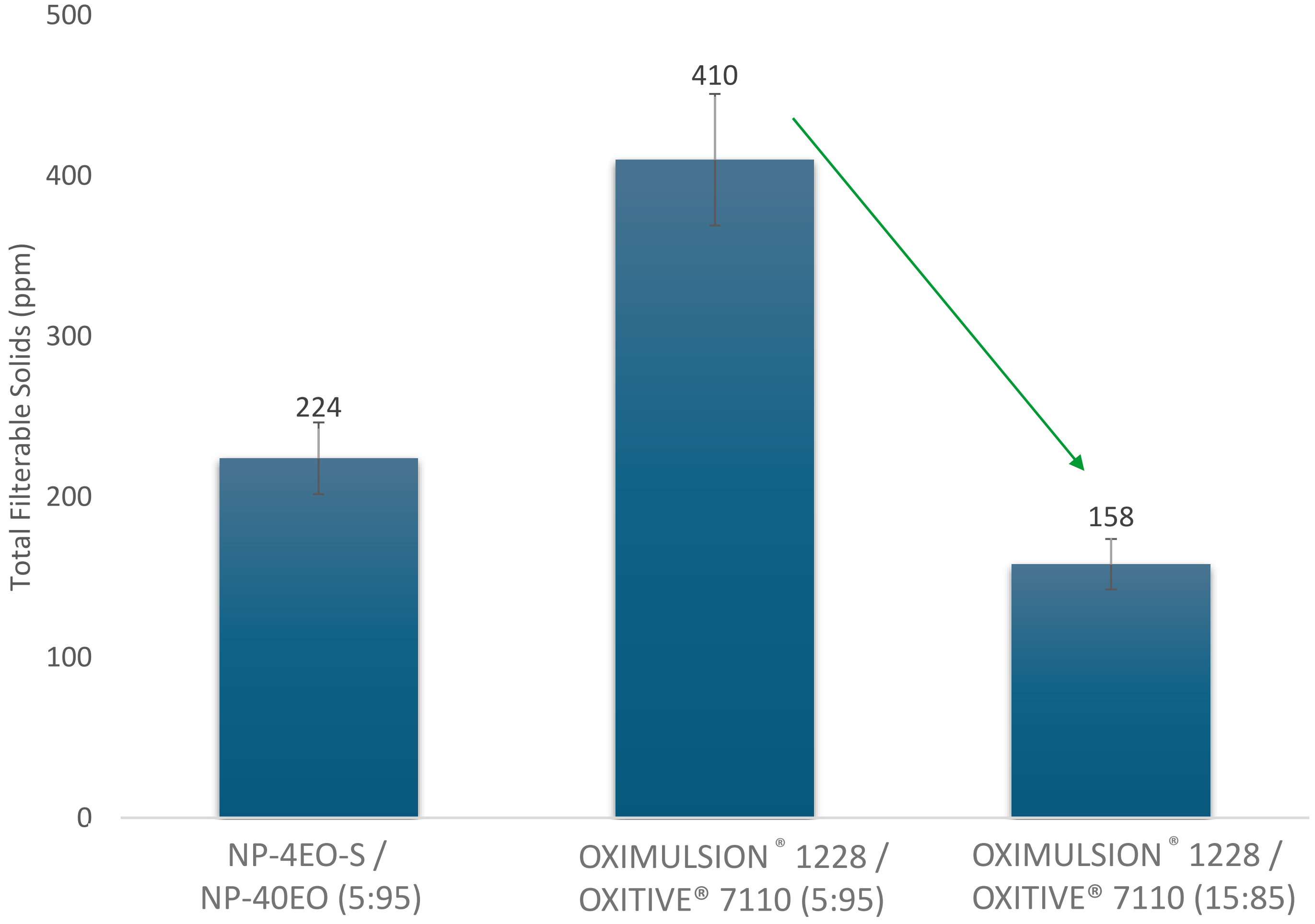
Components (phm)	Formulation 1
Vinyl Acetate	80 %
Butyl acrylate	20 %
	Active Content (phm)
OXITIVE® 7110	3.74 – 4.18
OXIMULSION® 1228	0.22 – 0.66
Persulfate initiator	
Chase Redox	



- **Thermal initiator:** 4.5h at 70 °C
- **Solid content:** 55 wt.%

Effect of different ratios of nonionic to anionic

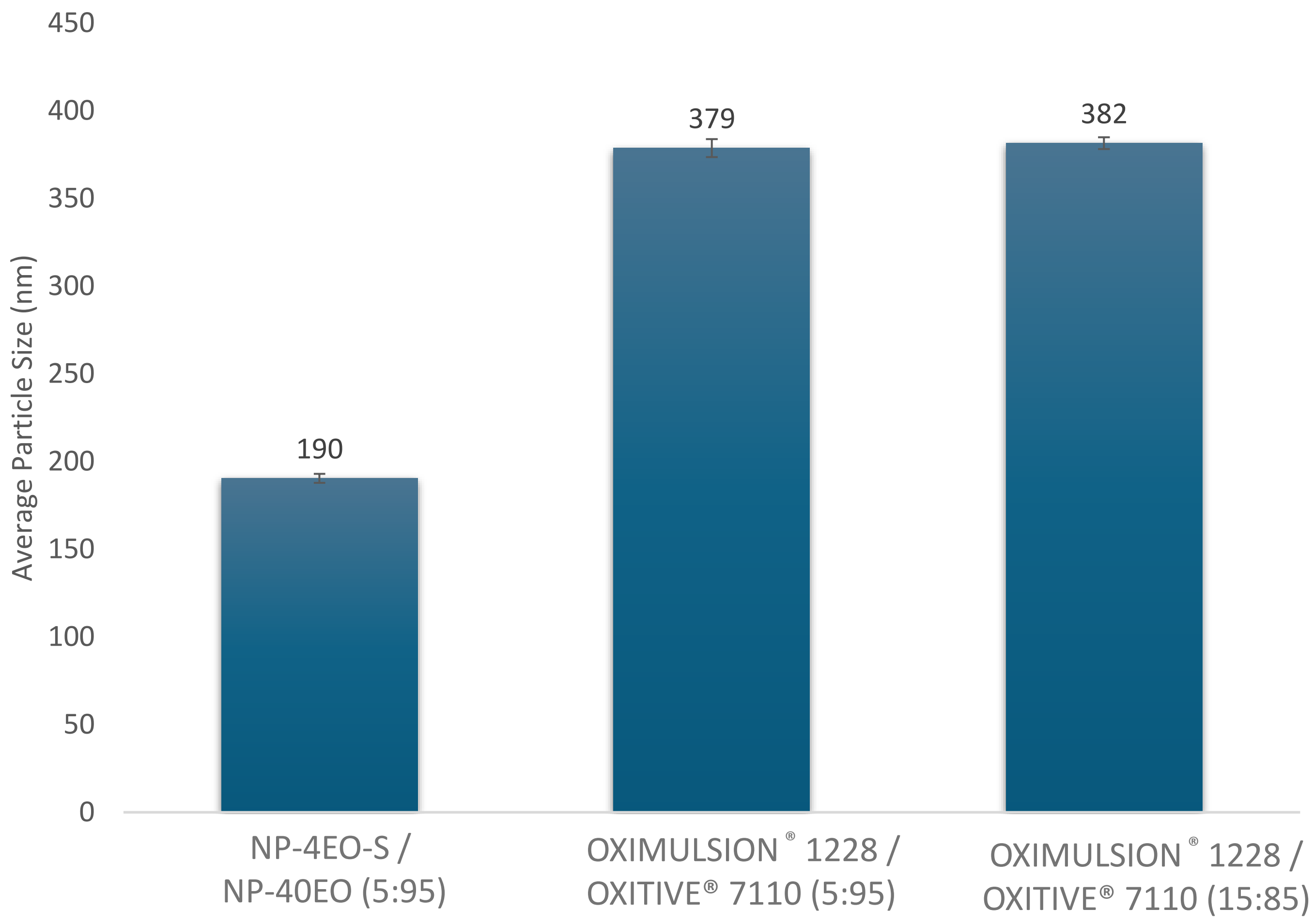
Reactor Cleanliness



- **Clot formation < 500 ppm** for all formulations.
- New ratio of anionic to nonionic **reduced** clot formation.

Effect of different ratios of nonionic to anionic

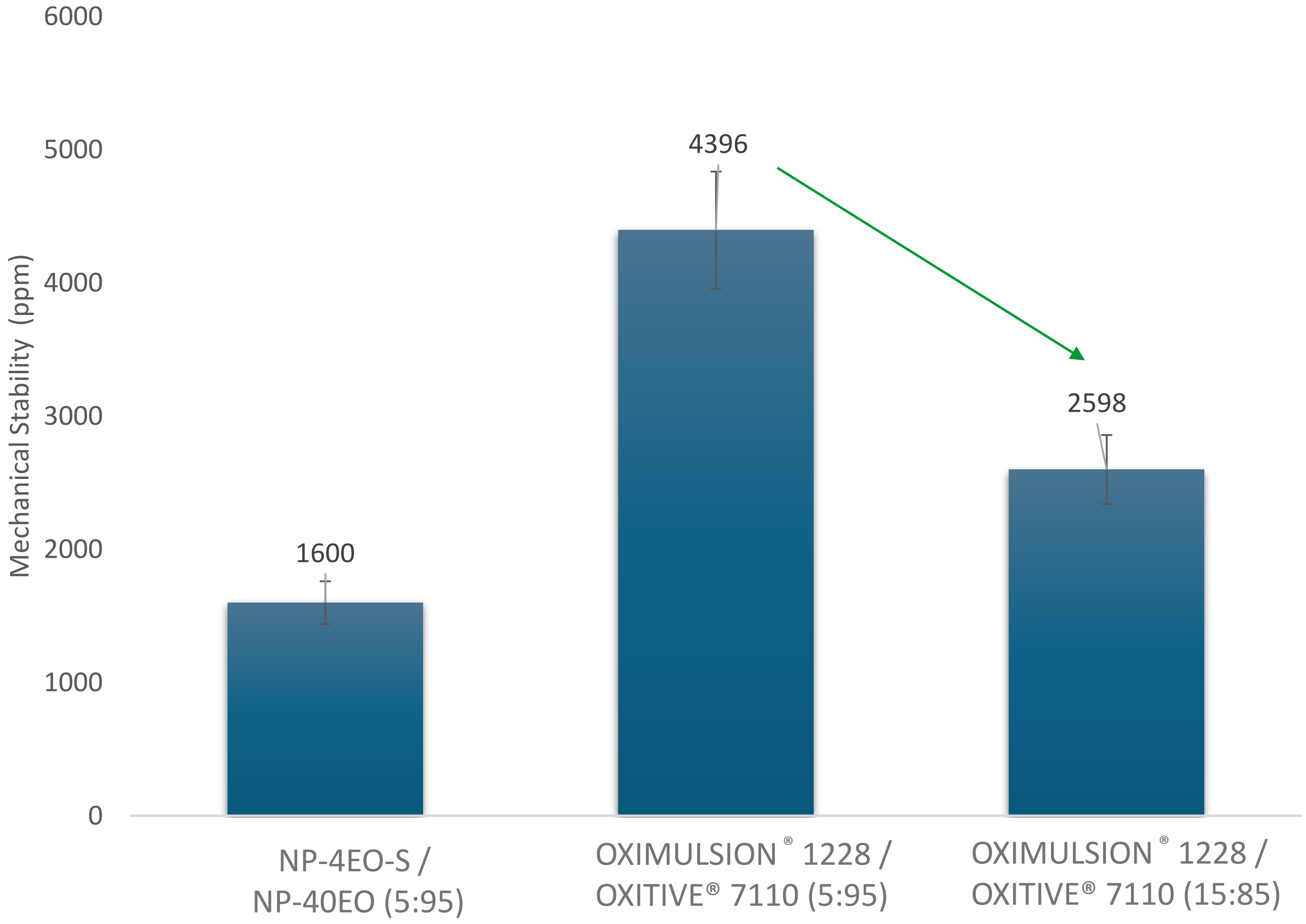
Particle Size



- Acceptable particle size range is **200 – 400nm.**
- **Low impact** in particle size.

Effect of different ratios of nonionic to anionic

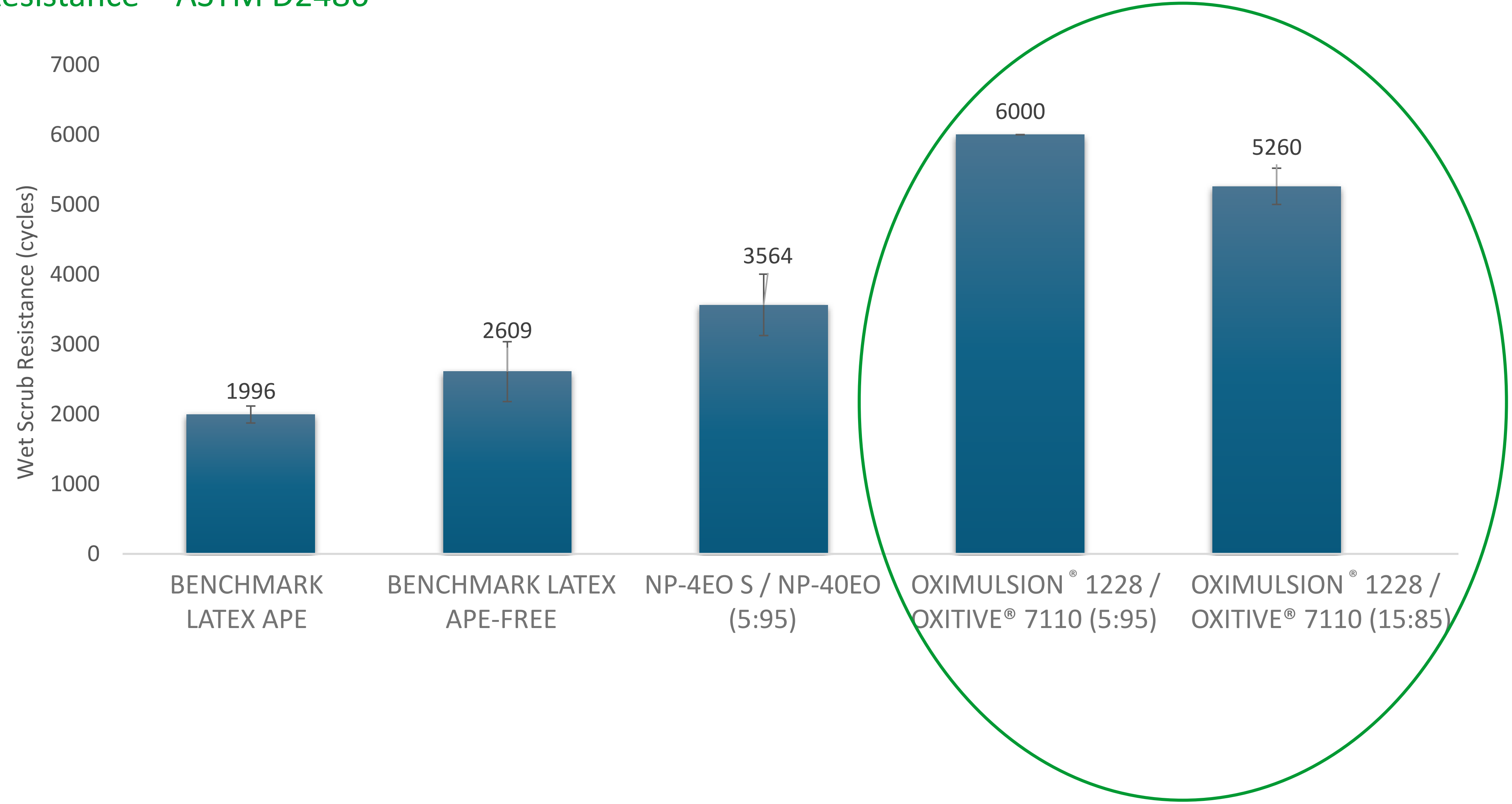
Mechanical Stability



- **Less than 1.0% clot** formed under shear stress.
- New ratio of anionic to nonionic **reduced** clot formation after mechanical stress.

Vinyl-Acrylic Paint

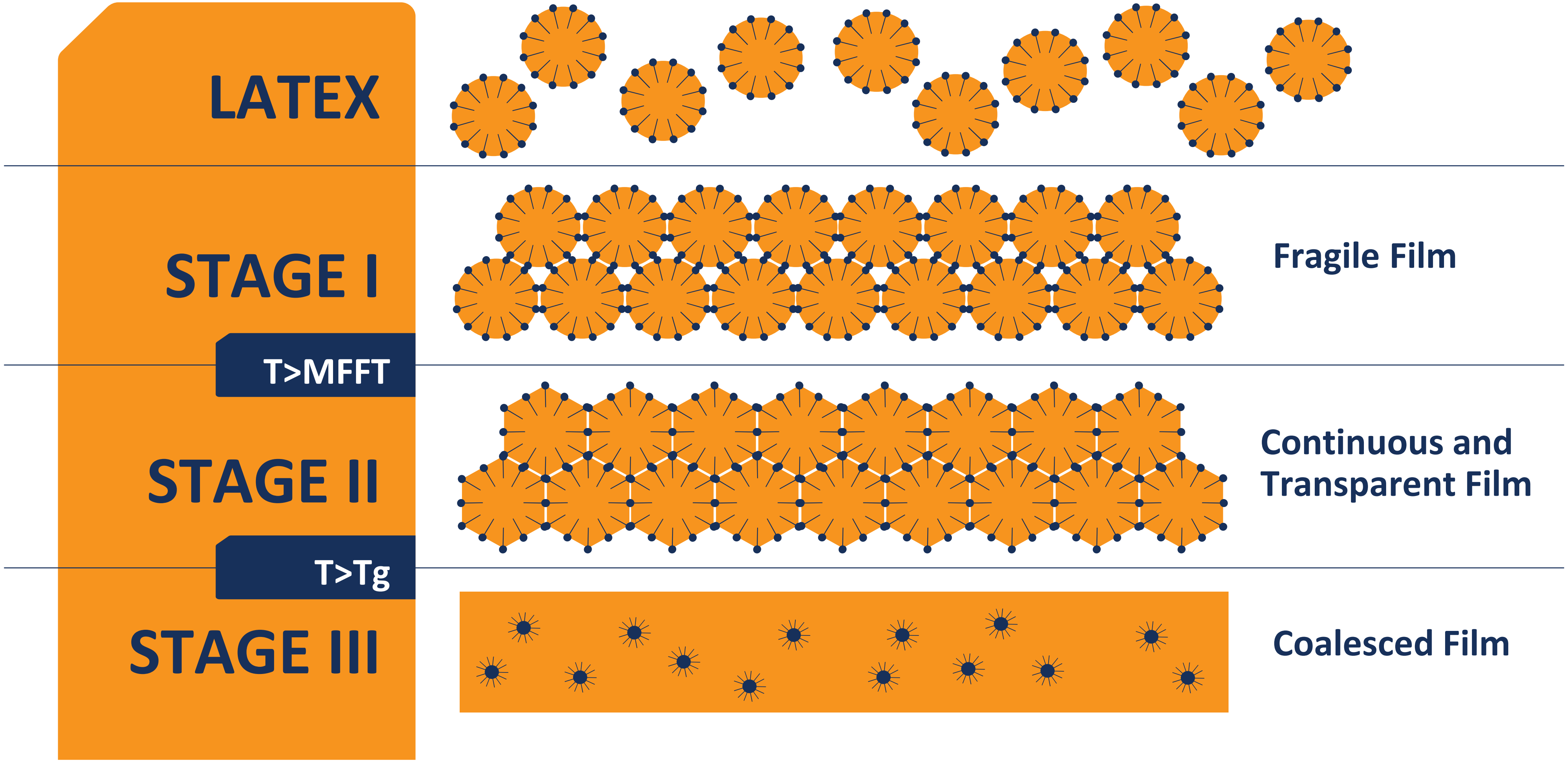
Scrub Resistance – ASTM D2486



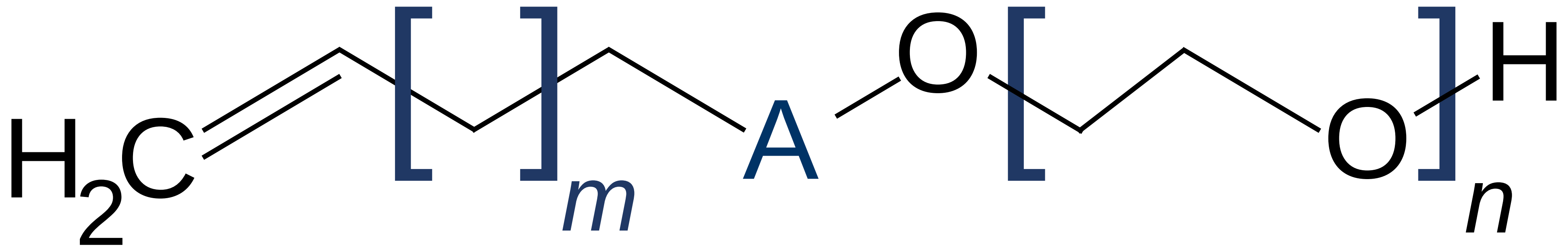
Reactive Surfactants

Emulsion Polymerization

Why does it happen?



OXIMULSION REACT® N1



APEO-free

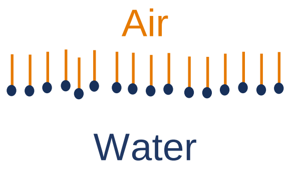
Partially Bio-based

Concentrated Product
Solid content >99 wt.%

VOC ≤ 5 g/L

Liquid

HLB of **15**



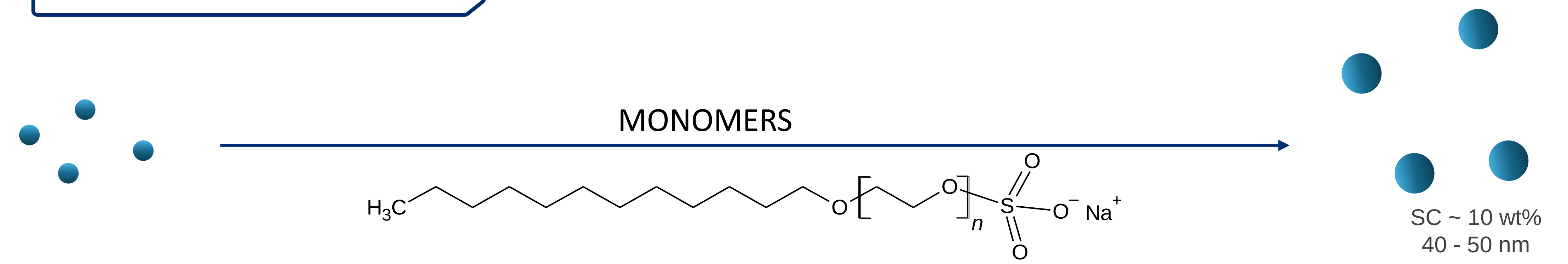
Behave as **conventional** surfactants

Compatible with all latexes

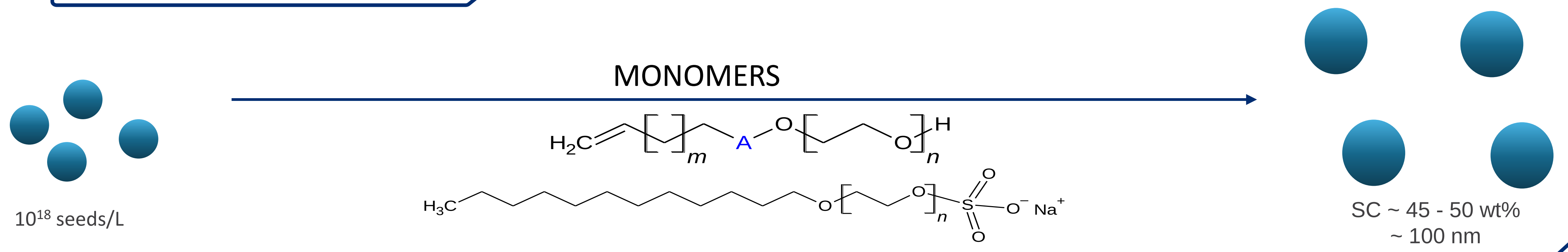
Two Step Process

Emulsion Polymerization

1° STEP | Generation of Seeds



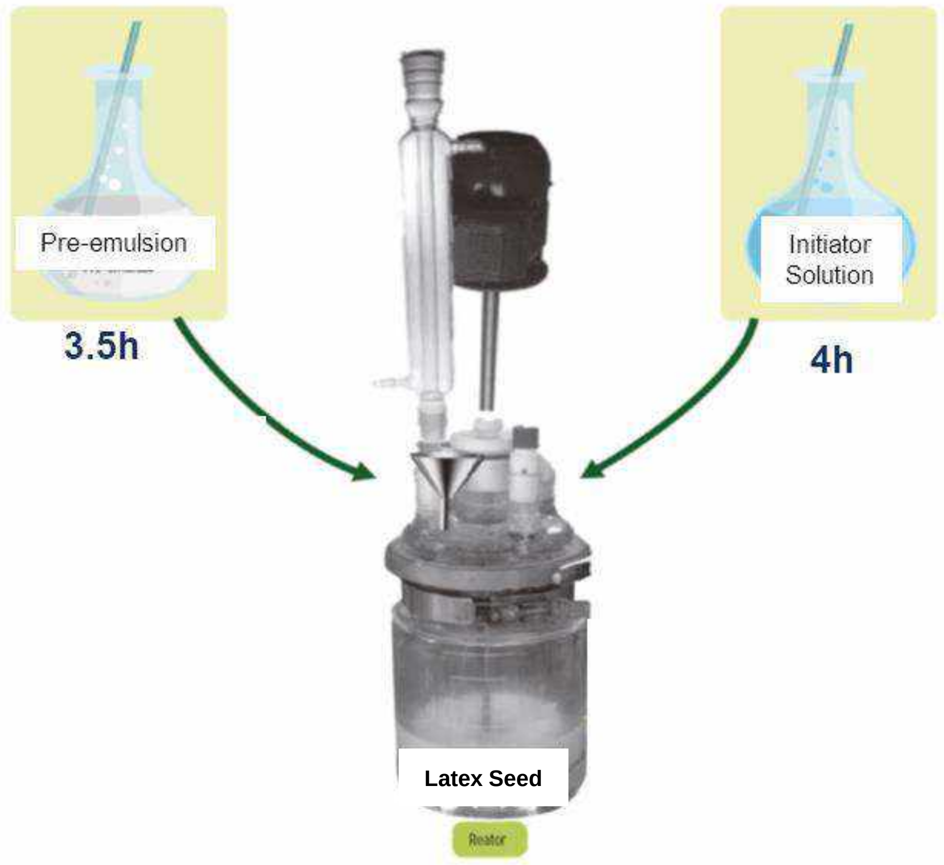
2° STEP | Growth of Seeds



Source: Asua et. Al., Langmuir 2003, 19, 3212 - 3221.

Emulsion Polymerization

SEEDED SEMI-BATCH PROCESS



Starting Formulations

	Components	w/w
Latex seed (Previously prepared)	Methylmethacrylate (MMA)	0.75
	Butyl acrylate (BA)	0.72
	Methacrylic acid	0.03
	Anionic surfactant*	0.38
	Ammonium persulfate	0.004
Pre-emulsion	Methylmethacrylate (MMA)	23
	Butyl acrylate (BA)	22
	Methacrylic acid	1
	Anionic surfactant*	TBD
	REACT® N1	TBD
Thermal Initiator	Ammonium persulfate	0.15
Ox-redox Initiator	Oxidizing Agent	0.02
	Reducing Agent	0.02

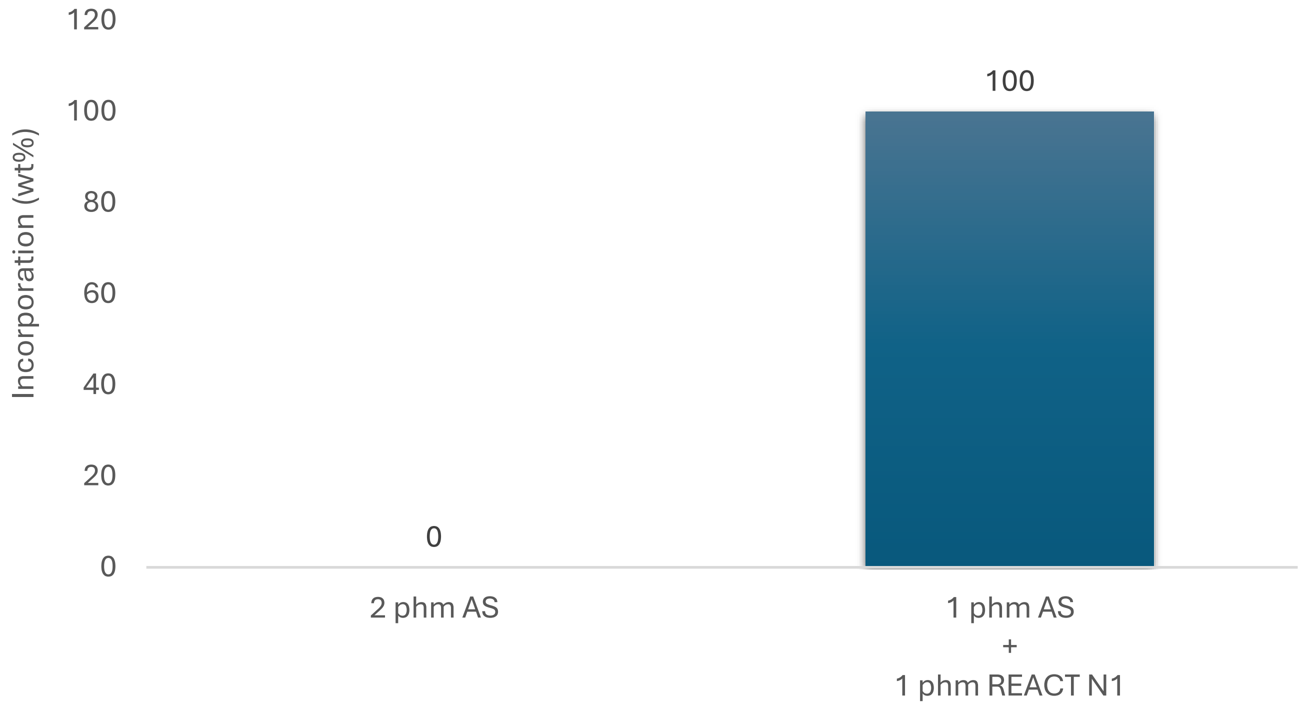
** Sodium salt of lauryl ether sulfate*

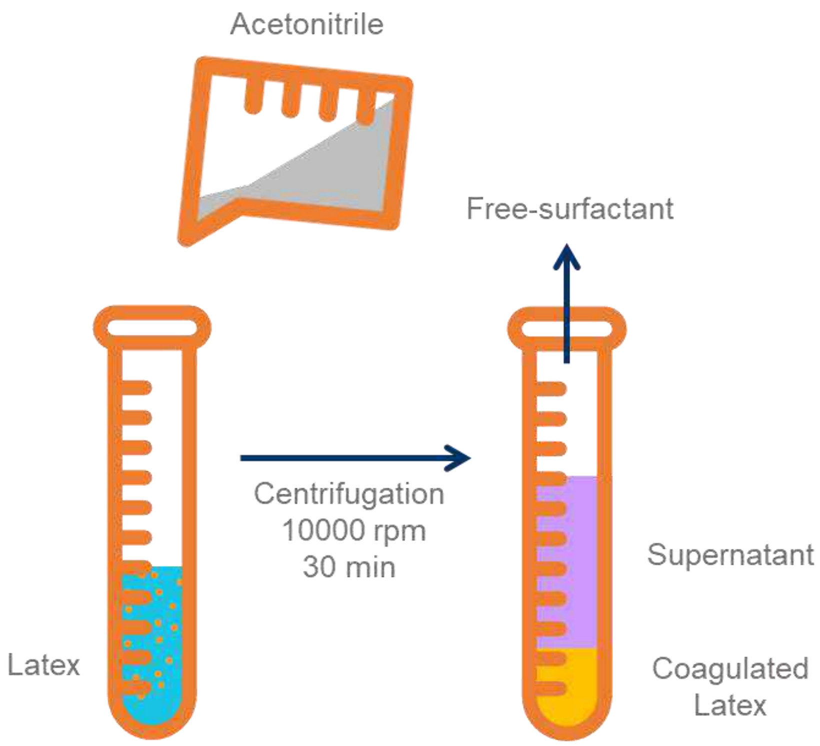
Effect of Surfactant Composition on Coagulum Formation in EP

	75 wt% REACT® N1 25 wt% Anionic Surfactant	67 wt% REACT® N1 33 wt% Anionic Surfactant	50 wt% REACT® N1 50 wt% Anionic Surfactant
Coagulum in Reactor Thermocouple Impeller			
Dispersed coagulum in latex	800 ppm	834 ppm	805 ppm

Incorporation of Reactive Nonionic Surfactant

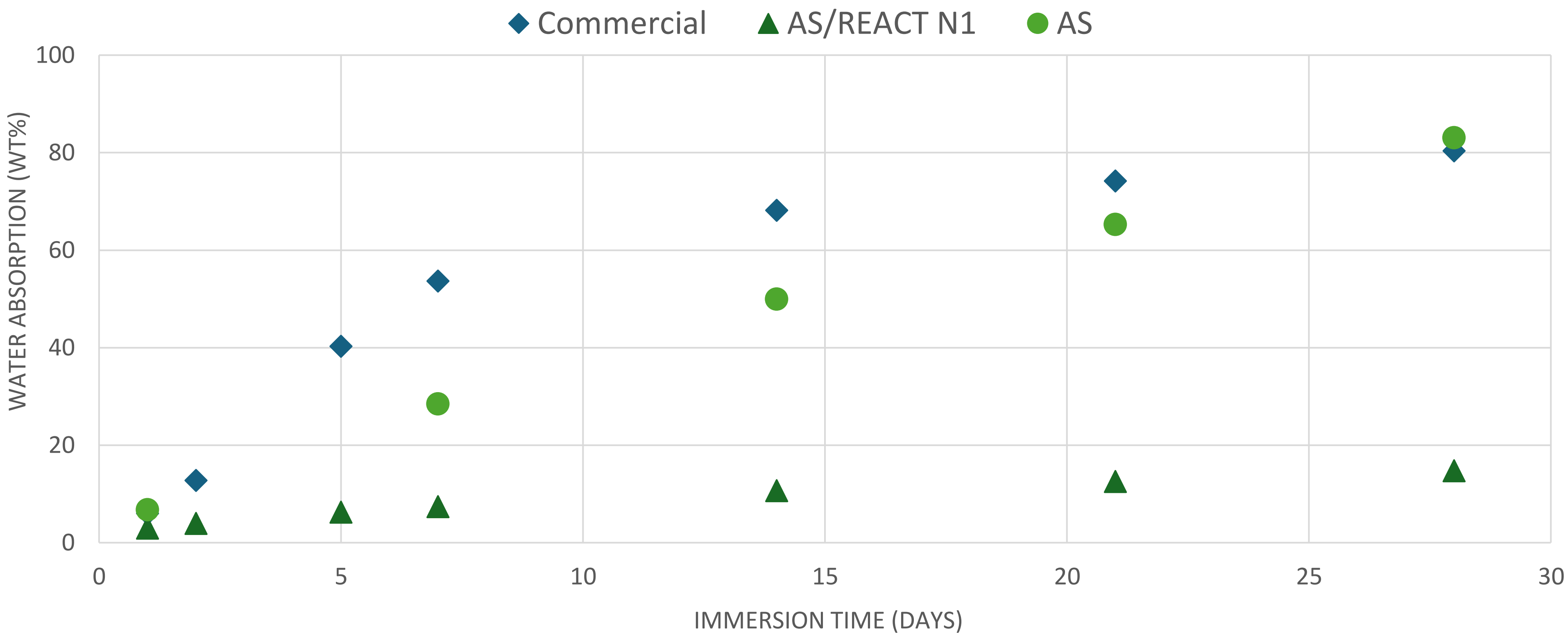
Surfactant Incorporation =
Total Surfactant - Free Surfactant





Water Absorption

Latex films with 2 phm of coalescent immersed in water at 25°C



WHITENING

Latex films with 2 phm of coalescent immersed in water at 25°C after 4 days

Commercial

AS / REACT® N1 (1:1)

Transition to APE-free Surfactants

Emulsion Polymerization

APE-FREE Alternatives to APE-based Nonionic Surfactants

Emulsion Polymerization

APE-FREE Alternatives	HLB	APE-based Nonionic	HLB
OXITIVE® 7254	13.4	SURFONIC® N-95	13.1
OXITIVE® 7110	16.9	SURFONIC® NB-307	17.1
OXITIVE® 7130	17.4	SURFONIC® NB-307	17.1
OXITIVE® 7140	18.0	SURFONIC® NB-407	17.8
OXITIVE® 7240	18.0	SURFONIC® NB-407	17.8

Replacement of APE-based for APE-FREE Surfactants

Emulsion Polymerization

Replacement of APE for APE-FREE Surfactants in Vinyl-Acrylic Latex

APE-based	APE-free solution
NP 9.5 EO SULFATED + NP 30 EO	OXIMULSION® B1000 + OXITIVE® 7110 OXIMULSION® 1228 + OXITIVE® 7130 OXIMULSION® 1228 + OXITIVE® 7140 OXIMULSION® 1228 + OXITIVE® 7240
NP 4 EO SULFATED + NP 40 EO	OXIMULSION® 1228 + OXITIVE® 7140 OXIMULSION® 1228 + OXITIVE® 7240 OXIMULSION® 11230 + OXITIVE® 7110

Replacement of APE for APE-FREE Surfactants in Styrene-Acrylic Latex

APE-based	APE-free solution
NP 9.5 EO SULFATED + NP 23 EO	OXIMULSION® B1000 OXIMULSION® 1228 + OXITIVE® 7110
NP 25 EO SULFATED + NP 30 EO	OXIMULSION® 1228 + OXITIVE® 7110 OXIMULSION® 11230

Replacement of APE for APE-FREE Surfactants in All-Acrylic Latex

APE-based	APE-free solution
NP 9.5 EO SULFATED	OXIMULSION® 1228 OXIMULSION® 1328
NP 4 EO SULFATED	OXIMULSION® 11230 OXIMULSION® B 1000 OXIMULSION® 2742

Q&A Session

Surfactants for Emulsion Polymerization

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