FUNCTIONAL PRODUCTS INC.
Innovative Chemistry for Lubricants

Polymers to Enhance the Performance of Inorganic Greases

2016 NLGI

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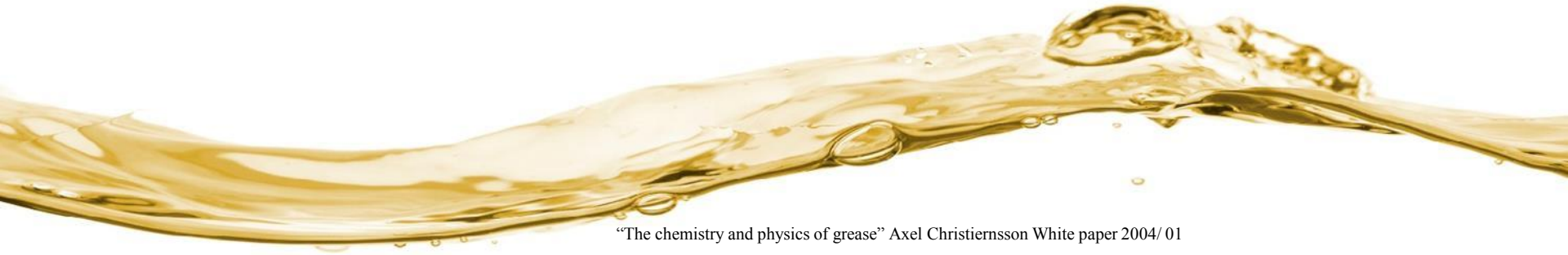
Outline

- The Structure of Grease
- Polymer Structure
- Benefits of polymers - Stiffness and Water Resistance
- Performance Tests – Cone Penetration and Water Spray-off
- Data and Results
  - » Lithium Complex
  - » Aluminum Complex
  - » Calcium Sulfonate
  - » Polyurea
  - » Vegetable Oil Based Grease
  - » Inorganic Greases

Grease Structure

Grease composition

- Base fluid: 70-90%
- Thickener: 5-25%
- Other additives



"The chemistry and physics of grease" Axel Christiernsson White paper 2004/ 01

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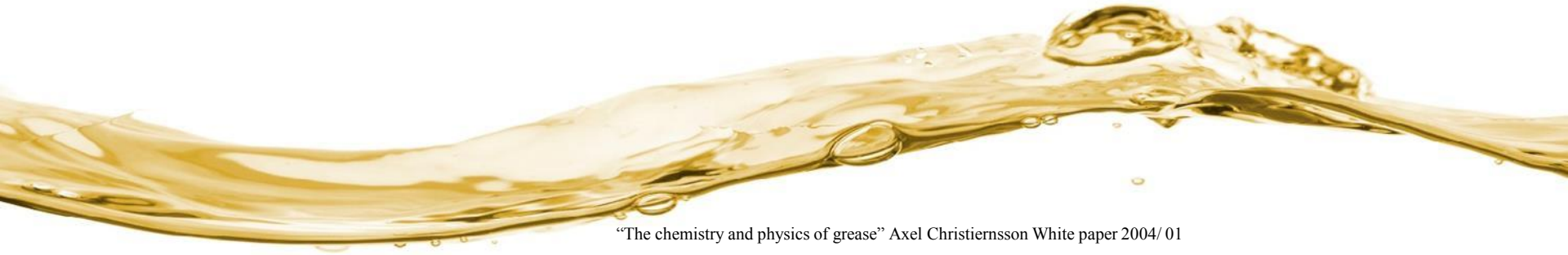
Thickener

- Soap thickeners



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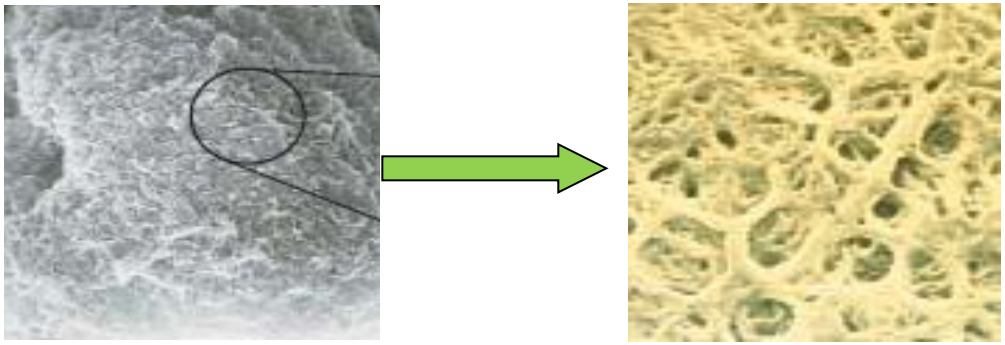
Thickener

- Soap thickeners

Multi-phase structure of grease

The solid phase thickener provides a physical matrix to hold the liquid phase base fluid in a semi-solid structure until operating conditions.
The soap thickeners possess interlocked fibrous structures.



"The chemistry and physics of grease" Axel Christiernsson White paper 2004/ 01

Grease Structure

Grease composition

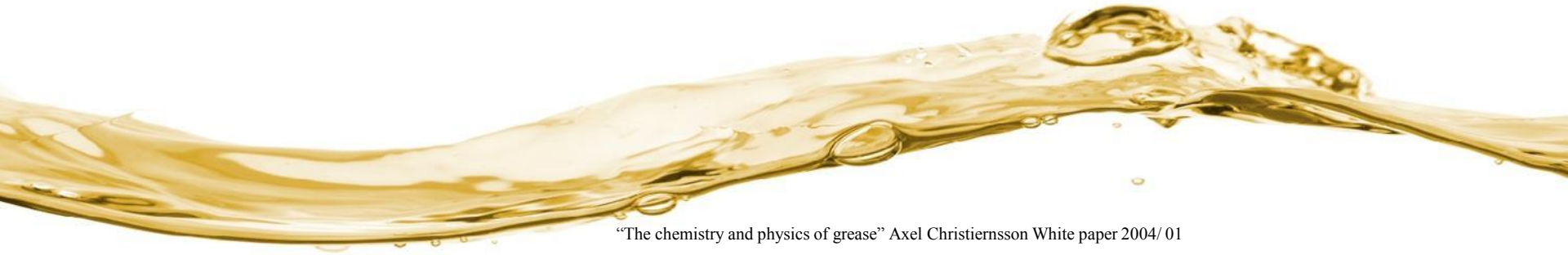
- Base fluid: 70-90%
- Thickener: 5-25%
- Other additives

Thickener

- Soap thickeners
- Non-soap thickeners
  - Organic thickener (polymer)
  - Inorganic thickener (clay, silica gel, etc.)

Multi-phase structure of grease

The solid phase thickener provides a physical matrix to hold the liquid phase base fluid in a semi-solid structure until operating conditions.



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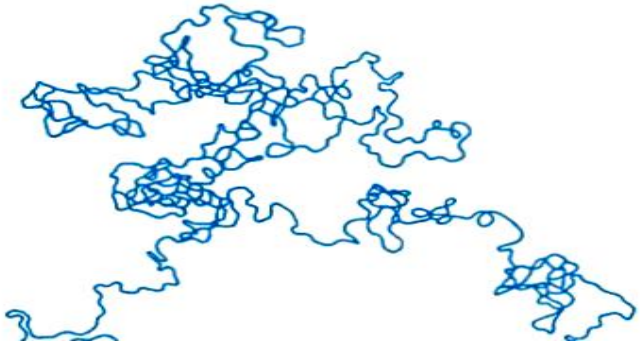
Benefits of Polymer in Grease

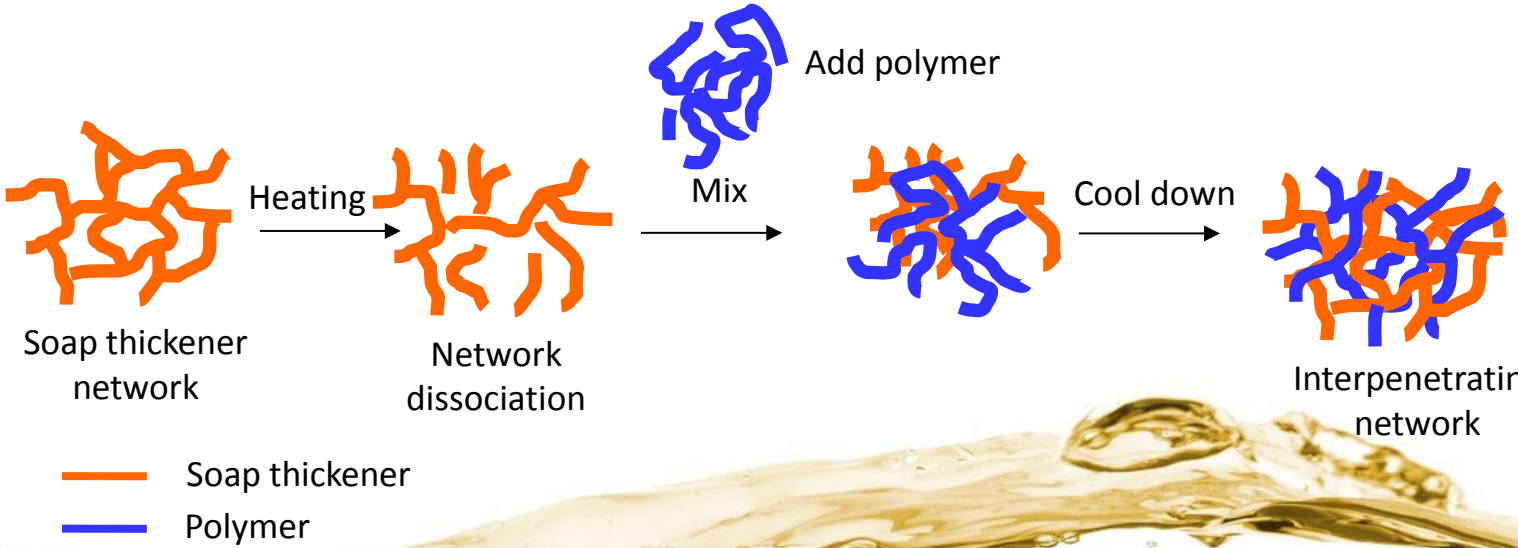
- Due to long chain nature of polymer grease show:
  - **G**reater adhesive tack
  - **R**educed bleeding
  - **E**levated cohesive tack
  - **A**dded yield
  - **S**uperior shear resistance
  - **E**nhanced water resistance



http://capolight.files.wordpress.com/

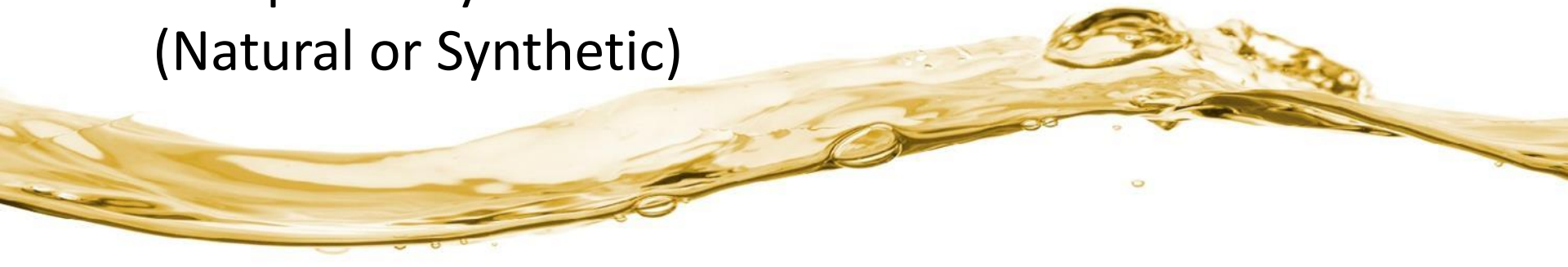
Mechanism

- In order to enhance the structure and the mechanical properties of grease, the polymer must form a 3D network with the soap
- Both grease and polymer networks are flexible and may be reversible
- The grease soap and polymer networks entangle to form an interpenetrating network (IPN) as shown for a simple lithium grease:



Polymer Selection Criteria

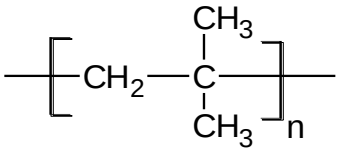
- Ability to form physical networking structure
  - Semi-crystalline polymer (e.g. polyolefin copolymer with high ethylene loading)
  - Temperature sensitive solubility (e.g. polyolefin/styrene block copolymer)
  - Hydrogen bonding (e.g. polyurea or polyolefin with amide/anhydride groups)
  - Long chain entanglement (e.g. high molecular weight polyolefin)
- Compatibility with base oil – Mineral or Ester (Natural or Synthetic)



Grease Polymers

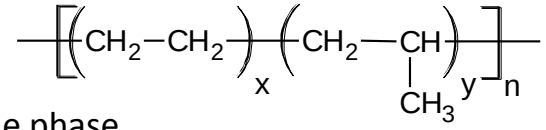
Polyolefins:

Polyisobutylene (PIB)



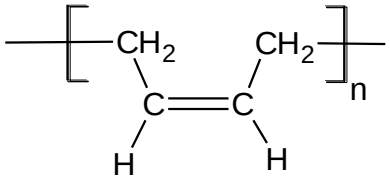
Saturated hydrocarbon

Ethylene/propylene copolymer (OCP)



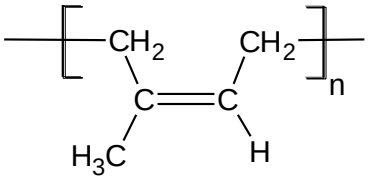
Saturated hydrocarbon; High ethylene loading in OCP can form crystalline phase

Polybutadiene (PB)



Unsaturated hydrocarbon

Polyisoprene (PIP)



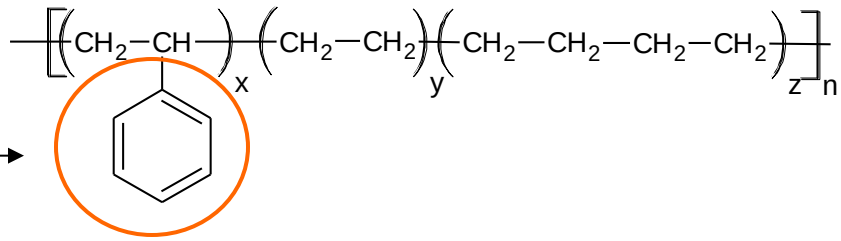
Unsaturated hydrocarbon

Grease Polymers

Modified Polyolefins:

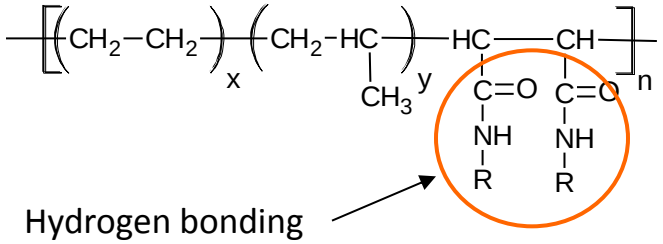
Styrene-ethylene-butylene copolymer (SEBS/SEBCP)

Temperature sensitive solubility →



Ethylene/propylene copolymer grafted with amide/anhydride (OCP-A)

Saturated hydrocarbon with polar amide or anhydride group



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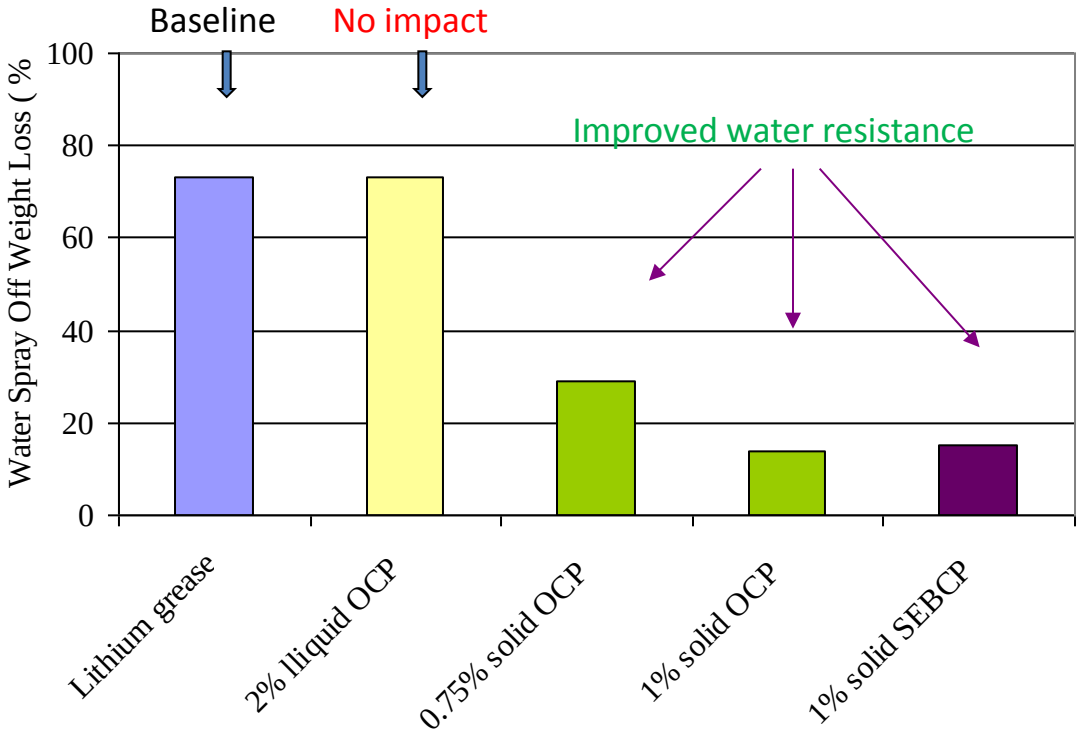
Polymer in Mineral Oil-based Lithium Grease

Water spray-off (ASTM D4049)



Lithium Complex Greases

- Dominant grease by sales in North America. Robust and very popular.
- High temperature operating conditions are prominent. Dropping point 50 C than lithium grease.

Table 5 shows the improvement in water spray-off properties when polymers are added.

Polymer Type	Polymer concentration, wt%	% Water spray-off	Worked cone penetration value
None	0	63	275
OCP	0.5	29	272
OCP	1.0	14	265
SEBCP	0.5	48	263
SEBCP	1.0	15	262

Aluminum Complex

Aluminum Complex Greases

The water spray-off ability of aluminum complex type greases is usually very good but is dependent on the amount of aluminum soap content. The aluminum being tri-valent can form a tighter or stronger network of fibers within the grease network.

Table 6

Polymer Type	Polymer concentration, wt. %	% Water spray-off	Worked cone penetration value
None (Baseline)	0	65	284
OCP	0.25	34	275
OCP	0.5	31	270
OCP	1.0	13	266

Polyurea Grease

Polyurea Greases

This type of grease is typically used where elevated temperatures and high running speeds are encountered. They have a unique ability to offer low noise characteristics to grease. Water resistance is typically okay but can be improved by the addition of polymers.

Table 8

Polymer Type	Polymer concentration, wt. %	% Water spray-off	Worked cone penetration value
None (Baseline)	0	47	192 – Grade 4
PIB	0.05	38	193
OCP	0.75	10	144
OCP	1.0	12	130

Calcium Sulfonate

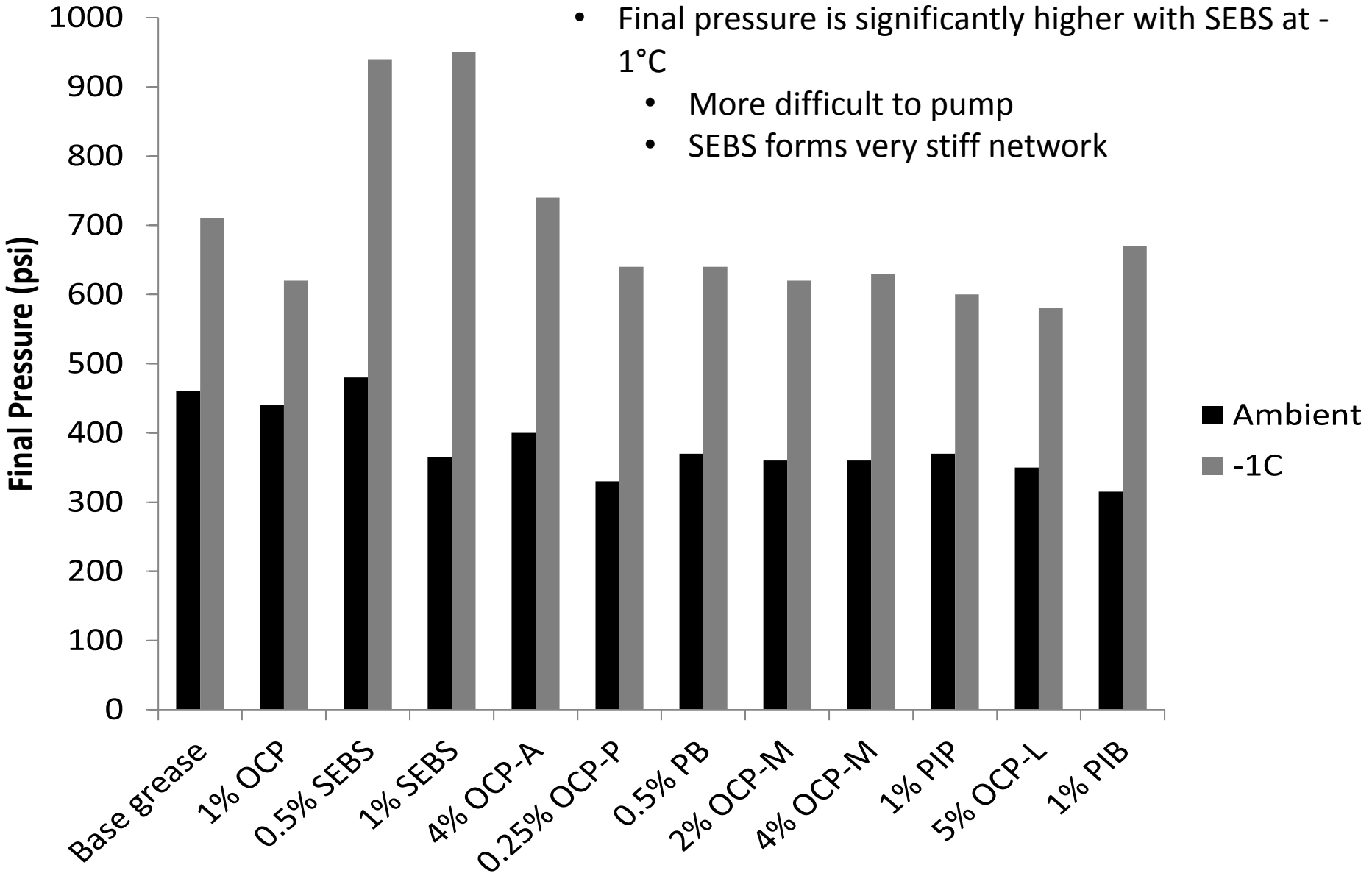
- Good all-purpose type grease.
- Very good oxidation and corrosion resistance,
- Excellent thermal and mechanical stability
- Good water resistance and high load carrying capability.
- Applications- marine applications and in food machinery.

Table shows the improvement in water spray-off properties when polymers are added.

Polymer Type	Polymer concentration, wt%	% Water spray-off	Worked cone penetration value
None	0	67	290
OCP-A	2.0	Not dissolved	
OCP	0.5	48	281
OCP	1.0	37	273
OCP	2.0	12	268
SEBCP	2.0	Not dissolved	

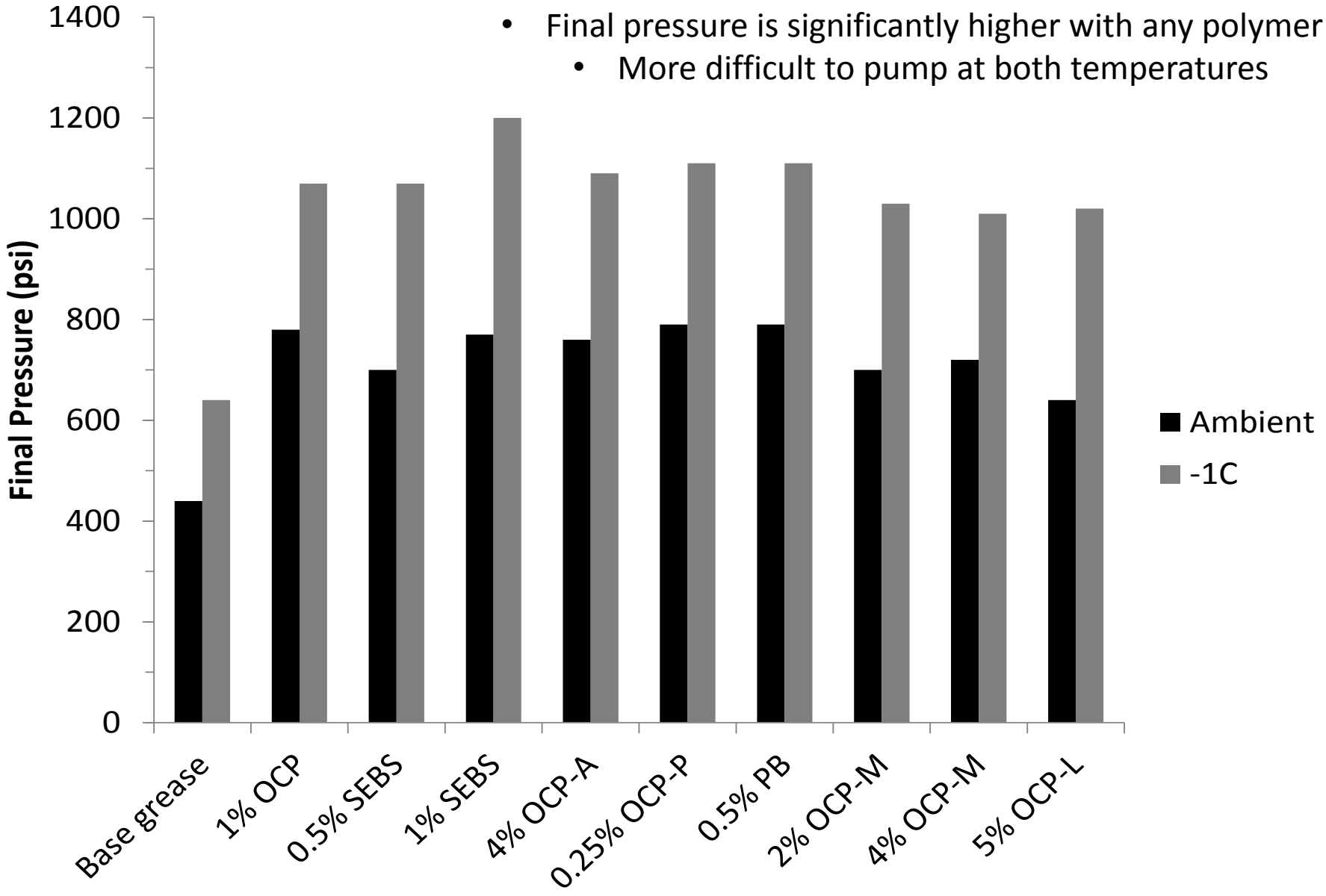
Low Temperature Properties: Li Complex

- Final pressure is significantly higher with SEBS at -1°C
  - More difficult to pump
  - SEBS forms very stiff network



Low Temperature Properties: Ca Sulfonate Complex

- Final pressure is significantly higher with any polymer
  - More difficult to pump at both temperatures



Inorganic Greases: Clay

Clay, %w	Base Oil	NLGI Grade	Additives
8.5	ISO 460 Paraffinic Bright Stock	2	0.5% Antioxidant

	Cone Penetration ($^{1}/_{10}$ mm)	Water Spray-off (%)	Water Washout (%)	US Steel Mobility (g/s)
Base grease	294	49	0.007	0.053
+1% PIP	286	10	29.35	0.009
+1% PIB	339	57	-2.03	0.012
+4% OCP-M	335	12	-2.21	0.111
+5% OCP-L	350	48	1.28	0.034

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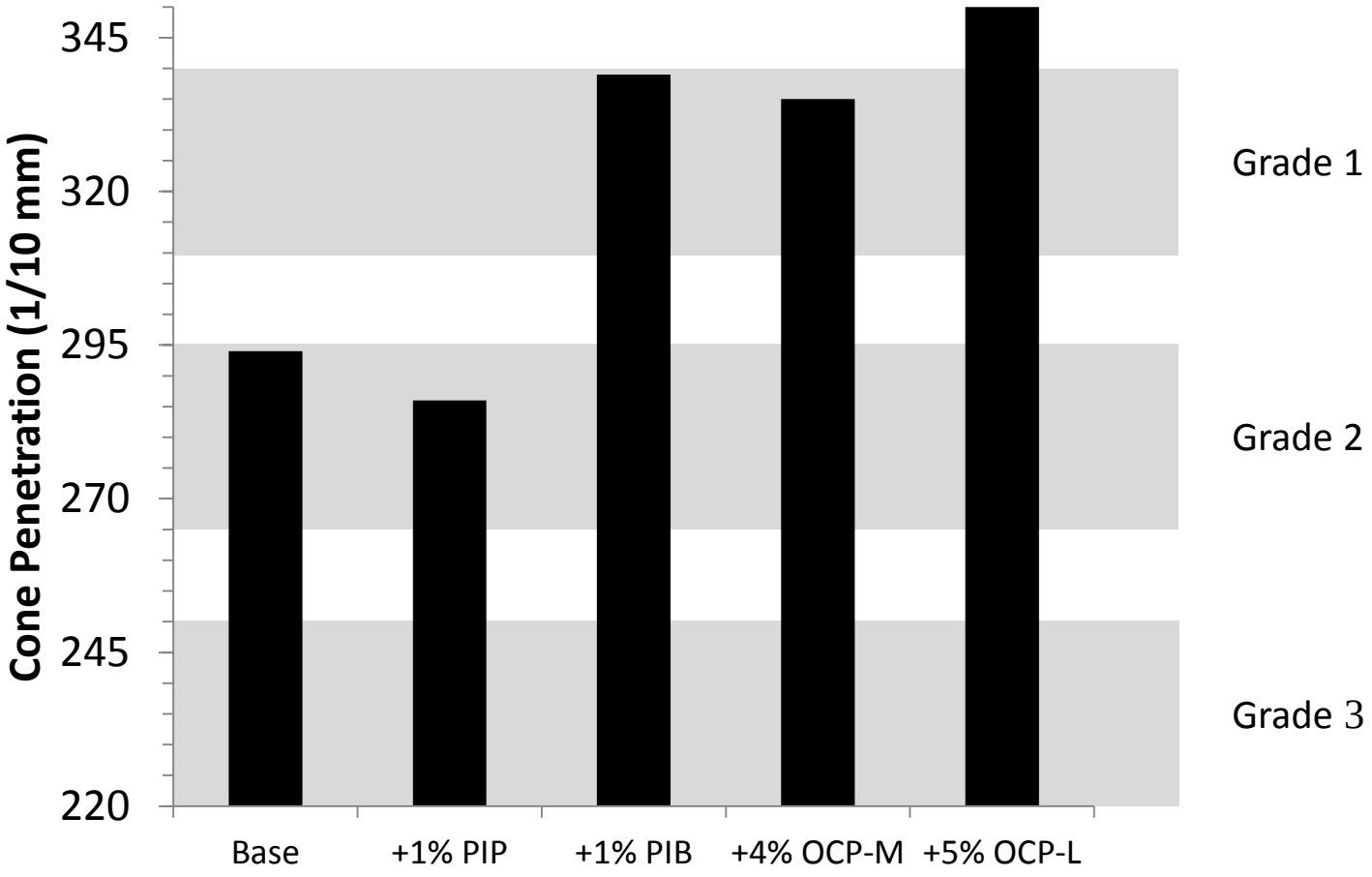
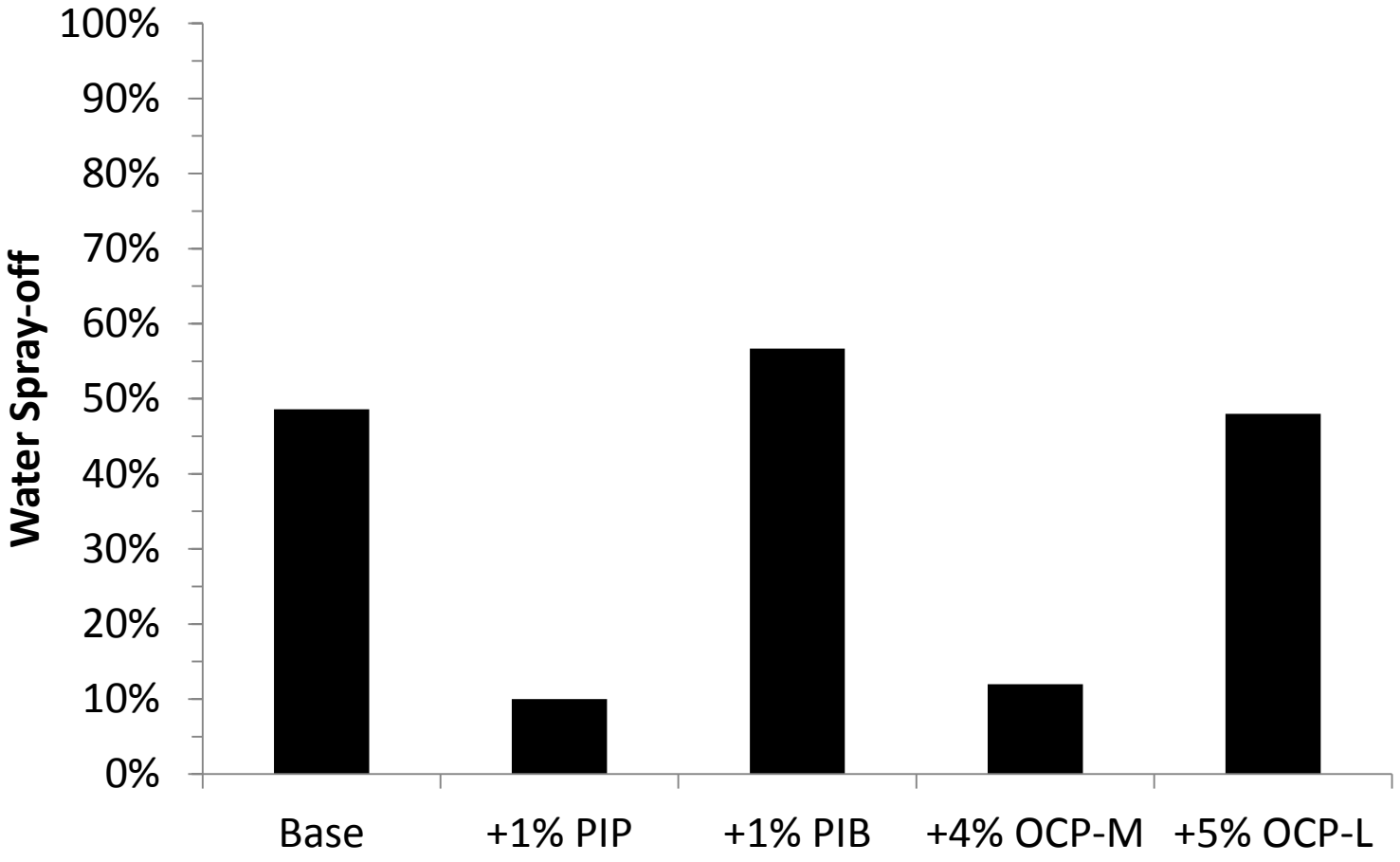
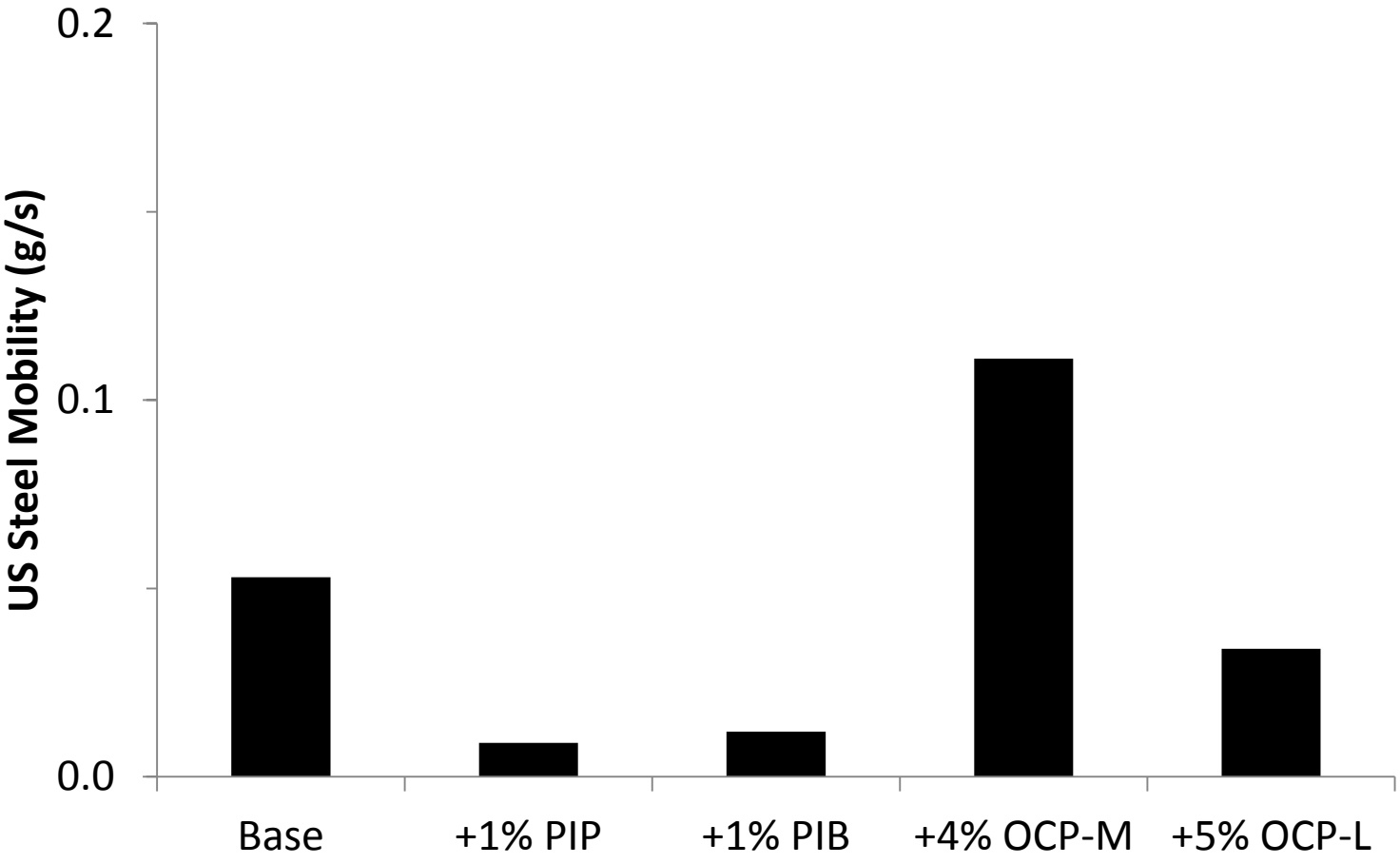
Clay Grease: Cone Penetration



Clay Grease: Water Spray-off



Clay Grease: US Steel Mobility

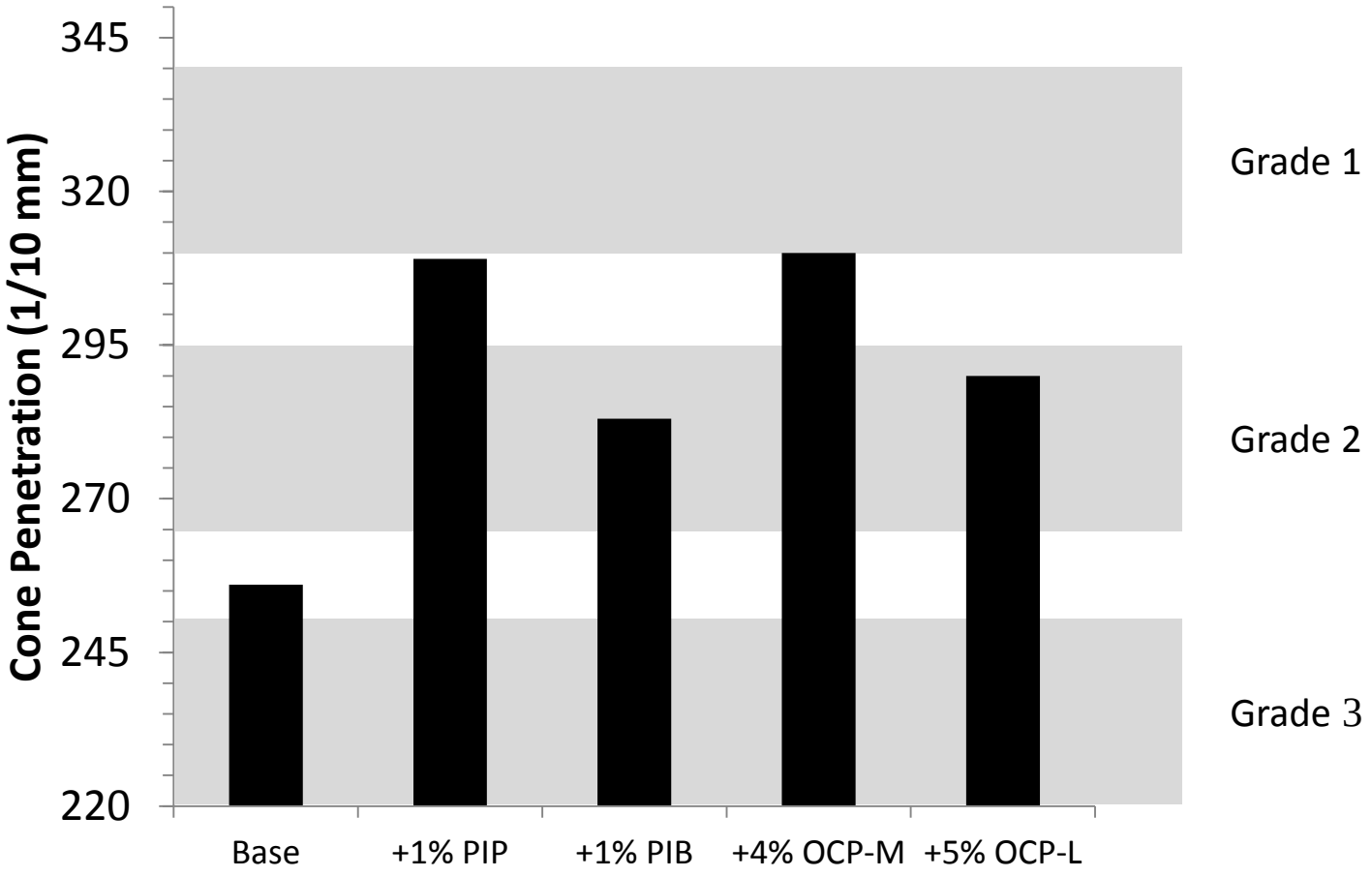


Inorganic Greases: Silica

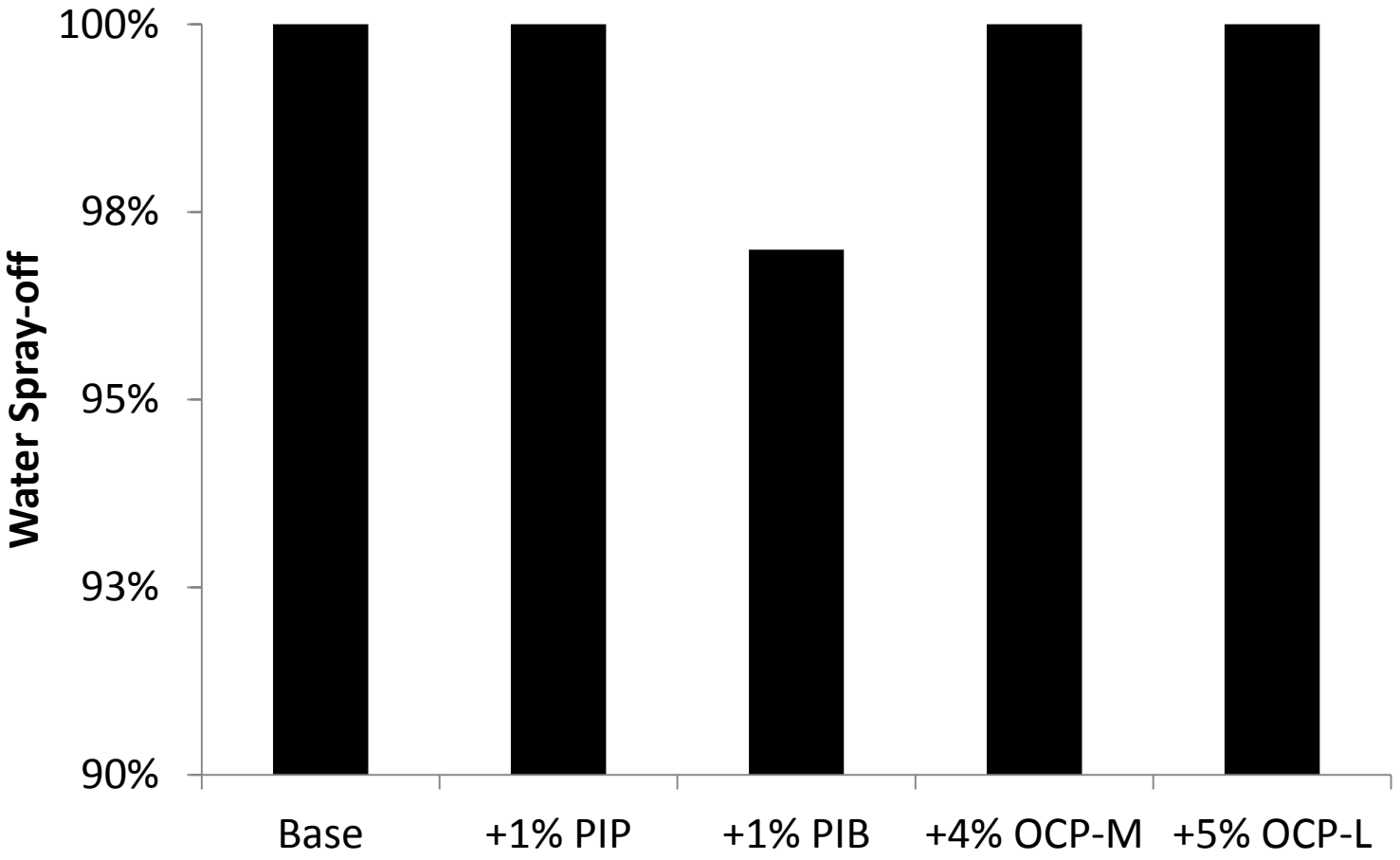
Silica, %w	Base Oil	NLGI Grade	Additives
9.2	ISO 120 Group I paraffinic	2	None

	Cone Penetration ($^{1}/_{10}$ mm)	Water Spray-off (%)	Water Washout (%)	US Steel Mobility (g/s)
Base grease	256	100	--	1.019
+1% PIP	309	100	--	0.165
+1% PIB	283	97	--	0.187
+4% OCP-M	310	100	--	0.339
+5% OCP-L	290	100	--	0.077

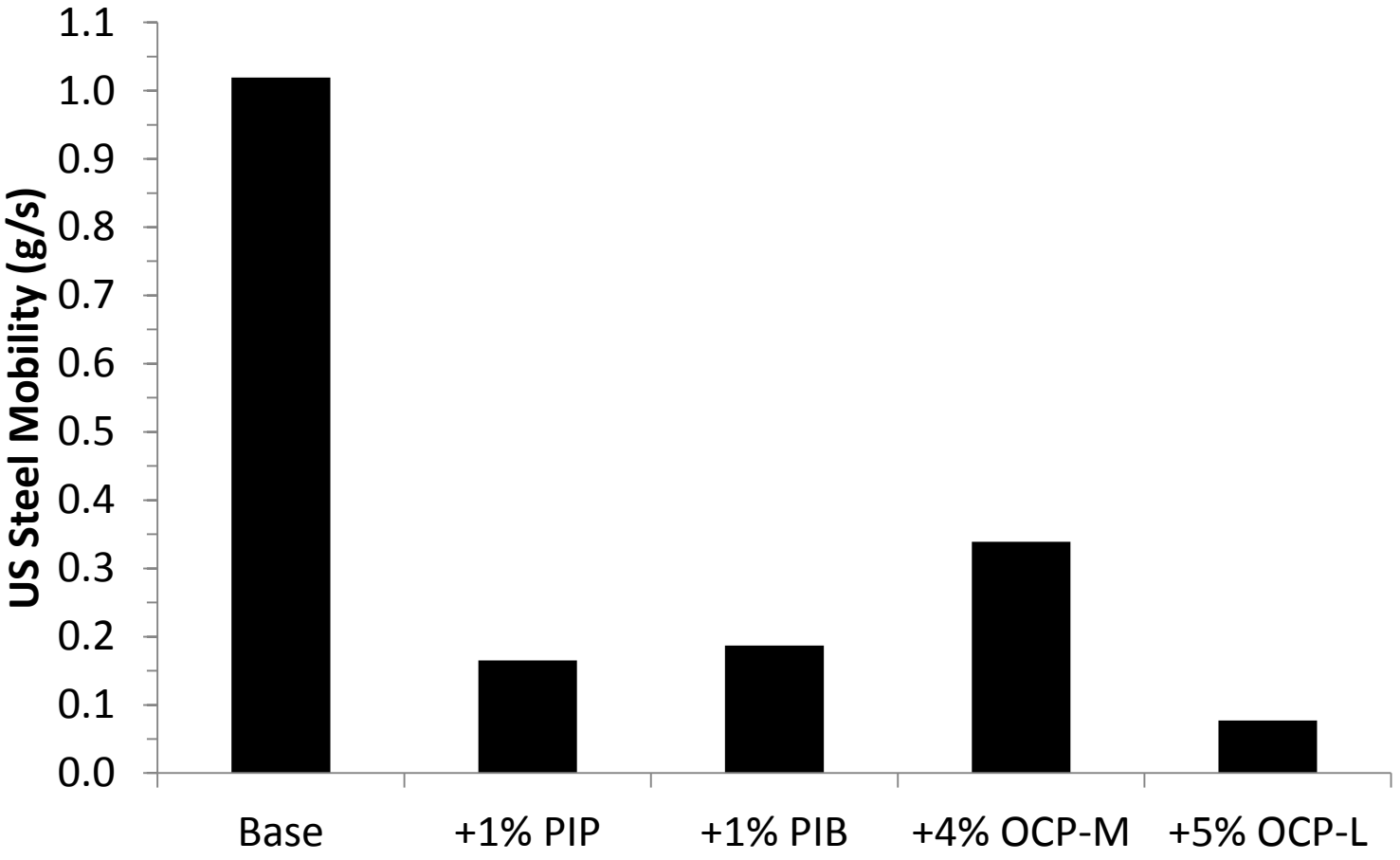
Silica Grease: Cone Penetration



Silica Grease: Water Spray-off



Silica Grease: US Steel Mobility



Conclusions

- Clay and Silica thickeners lack the ability to form a network structure compared to soap based greases
  - Added polymers cannot form an interpenetrating network with the thickener
- For a clay grease, polymers with functionality that can adsorb or orient themselves onto a metal surface (OCP-M) provides the best water spray-off performance
  - Polymers once adsorbed may function as a friction modifier allowing the bulk of the grease to have greater flow
- For a silica grease none of the polymers tested were effective

Acknowledgements

We are grateful to Battenfeld Grease & Oil Corporation of New York for supplying the clay base grease and Lubrication Engineers, Inc. for supplying the silica base grease.

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