From Concept to Application: Hydrated Grease for a more Sustainable Future

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Overview

- Section 1 Background: **H**igh **I**nverted **P**hase **E**mulsion (HIPE) Lubricant
  - Where we started and why
- Section 2 Hydrated Grease Product: First Generation
  - Change in approach and targeted a more ‘grease-like’ product
- **Section 3 Hydrated Grease Product: Second Generation (Current)**
  - **Addressed first generation challenges, expanded testing, stability focus**

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Section 1 Background: High Inverted Phase Emulsion (HIPE) Lubricant

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Section 1 - Background

- Lubrizol developed an emulsion technology that could allow the use of an **environmentally friendly grease-like lubricant** in place of standard mineral oil-based grease
- The initial concept was to help lower manufacturing **cost of goods (COGs)** and present a more bio-friendly product
- This technology was an oil continuous emulsion generally comprised of:
  - **Water**
  - **Vegetable or mineral oil**
  - **Performance additives**

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Section 1 - Background

- What did we hope to gain and target initially?
- **Original scope**
  - **Create a grease like product that can match the performance of a conventional lithium grease**
  - **Can a product like this compete in a marketspace held by conventional lithium products?**
- Grease performance from an emulsion-based lubricant – could this be achieved?
  - Reduced friction through proper, compatible additization
  - Enhanced cooling due to the presence of water
- The initial product was a **H**igh **I**nternal **P**hase water-in-oil **E**mulsion **(HIPE)**
  - 7+ components to make this work properly
- Desired physical characteristics
  - Semi-fluid NLGI 00 or 0 (likely)
  - Stiffer NLGI 2 or 3 (more difficult?)
    - Can we achieve stable stiffer emulsions?

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Section 1 – Performance Targets and Results

- Original goal - meet a cotton picker grease performance targets which was currently using a NLGI 00 to 0 conventional lithium grease with additives

Test	Description	Target Specification	HIPE Results	Comment
ASTM D217	Cone Penetration (W0)	00 (400-430 mm)	430 mm	Meets Target
ASTM D2509	Timken OK load	> 35 lb.	35 lb.	Meets Minimum Target
ASTM D2266	4-Ball Wear	<0.60 mm	0.40 mm	Exceeds Target
ASTM D1743	Corrosion Properties	Pass	Pass	Meets Target
ASTM D4048*	Copper Corrosion	1B or better	1A	Meets Target***
ASMT D5707**	SRV CoF	< 0.095	0.082	Meets Target
ASTM D5706	SRV EP Step Load	>500 N	1200 N	Exceeds Target
ASTM D2596	4-Ball Weld	For Information Only	126 kg Weld	For Information Only

Very Encouraging!

*Modified to account for water content. Duration was 24 h at 65 °C
**80 °C, 400 N, 10 Hz
***Results changed based on age of lubricant

Section 1 – Interim Review

What did we learn?

- By utilizing an emulsion-based approach and technology, *it is possible* to make an environmentally-friendly **grease-like** product from materials like vegetable oil and water which exhibits many of the features of a grease made with a mineral oil and solid thickeners
    - Initial design targets for a cotton-picking application were met!
- Passing specifications *does not* guarantee performance in the field
- Why didn't this product move forward?
    - Additional stress tests and evaluations were pursued

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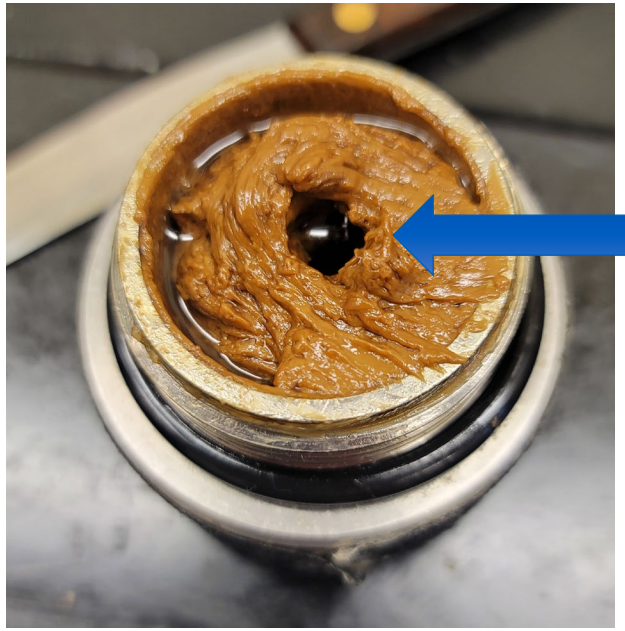
Section 1 - Challenges

- Exposure to any shear stress would start to break the emulsion
- ***Didn't 'feel' or look like a grease***



Emulsion grease after W60

Section 1 – Challenges and Conclusion

- Shear stability and keeping the emulsion intact was a challenge for the emulsion grease product. A desirable attribute of a lubricating grease is to not separate under stress
- Due to the high-water content, water was easily lost if stored higher than ambient temperature and the container was not properly closed. Compositional changes negatively affected it's grease like appearance and performance
- Stiffer grades needed *significant* HLB (Hydrophobic/Lipophilic Balance) adjustments to stay stable and viable
- Sulfur additives cannot be passivated easily
    - Copper corrosion worsened over time
- Corrosion testing (ASTM D6138, EMCOR) in saltwater was weak
- Longtime storage and stability was like a metalworking fluid – microbial growth was an issue
- Next steps…how to move forward?

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Section 2 Hydrated Grease Product: First Generation

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Section 2 – New Approach

- HIPE grease technology had some benefits and was possible but did not allow for much formulating variability or flexibility
- No discreet way to vary the NLGI grade, formulations were very sensitive to organic component viscosity grade
  - Unexpectedly, thicker base stock **did not** make a thicker product. ISO VG 100 oil components made grease-like materials with a similar thickness to an ISO VG 460 base oil
- High water content was originally desirable but very difficult to control over time and in different environments (containment, humidity, etc.)
- Moving back to a more traditional grease like material vs an HIPE emulsion was needed and was our next direction

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Section 2 – New Objectives

- New Scope:
  - design a protype grease product that contained >40% water content
  - Reduced water content from HIPE thought to help with stability and weeping
  - The target hydrated grease product was to be an NLGI#2 product with the possibility to be a NLGI#2, #1 or #0 depending on the amount of water or organic component contained therein
  - The concept desired the material to contain water and function in a ferrous system (bearings etc.)
- **This material was to compete in a marketspace held by any EP type grease, not a conventional lithium**
  - We extended the scope based on increasing the oil and organic content, moving away from the high invert phase emulsion
  - One step up in challenge

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Section 2 – New Product Proposal

- Product overview
  - >40% water at the time of manufacture*
  - >50% organic phase with select additives to bolster performance
- Multiple iterations of potential products were examined to provide a material that **appeared and presented as a lubricating grease**
  - HIPE product was grease-like but not like a grease
- Our proposal could be formulated to NLGI#2, #1 and #0 stiffness (picture)

*Due to the inclusion of water, it is assumed hydration level will change with usage

Section 2 – Generation 1 Performance Tests and Results

Test	Description	Result	HPM Core Requirement (truncated)
ASTM D6138	EMCOR, DI Water	**0,0 Rating**	**0,1 Rating**
ASTM D2265	Dropping Point	**>225 °C**	**No Requirement**
ASTM D2266	4-Ball Wear Scar	**0.57 mm**	**0.60 mm**
ASTM D2596	4-Ball Weld	**315 kg Weld**	**250 kg Weld**
ASTM D6184	Oil Bleed, 100 °C, 30 h	**0 wt%**	**7 wt%**
DIN 51805	Low Temperature Flow at 0 °C	**< 250 hPa**	**No Requirement in Basic, <1400 mBar @ -30°C for +LT (Low Temperature**

Improved performance over HIPE product

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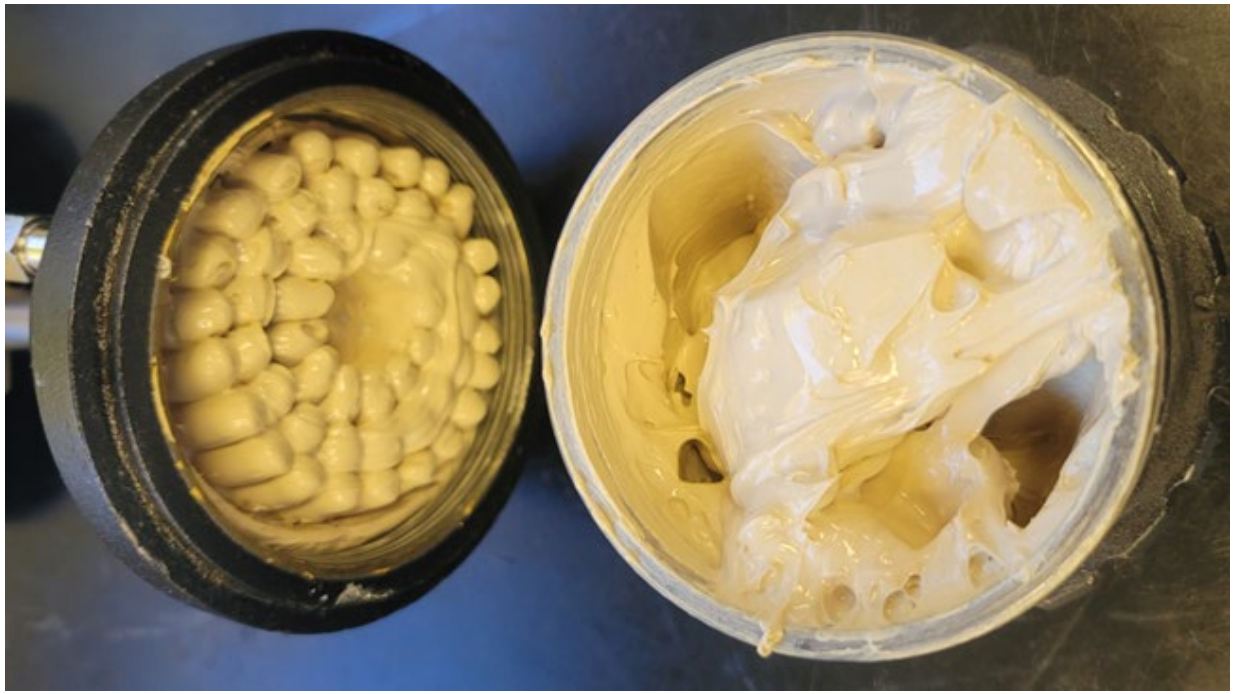
Section 2 – Shear Stability

- Primary drawback to the HIPE product was the ‘weeping’ observed under worked conditions or milling/processing
- The first-generation hydrated grease product performed much better and demonstrated no weeping (crying/tears) even after 100k strokes
- Regrettably, had a strong hydrocarbon odor



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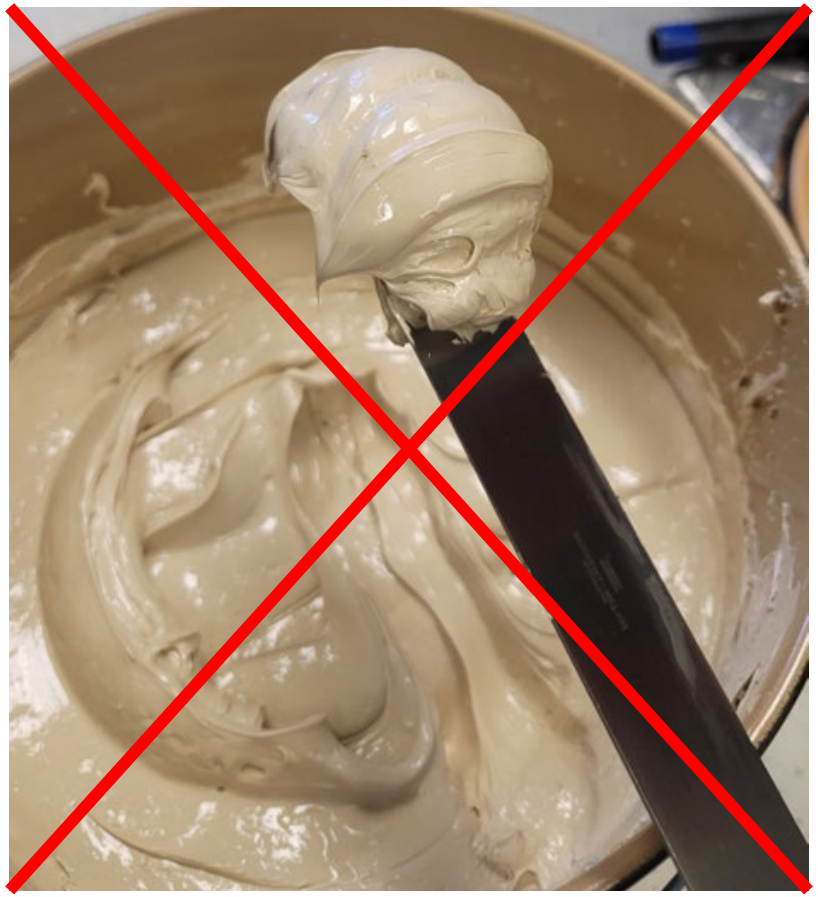
Section 2 – Challenges and Conclusion

- Scale up and transport from our North American grease lab to Hazelwood lab (overseas and several weeks in uncontrolled temperature or humidity) resulted in:
  - **Separation of water (low shear stress issues?)**
  - **Rust in container (unlined)**
  - **Microbial growth such the surface was black/green and smelled strong enough we could not take pictures**
- Unfortunately, a new set of challenges to produce a useable, hydrated grease product



Hydrated Grease product **before** shipment

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Section 3 Hydrated Grease Product: Second Generation (Current)

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Section 3 – Current Objectives

- Scope adjustments: Originally target a grease product that contained high water content; however, recent work has caused us to be more conservative at a lower water content
  - Reduced water further to help with stability and weeping
  - The target hydrated grease product was to be an NLGI#2 product with the possibility to be a NLGI#2, #1 or #0 depending on the amount of water or organic component contained therein
  - **We continue to target an EP type grease marketspace**
  - The concept still targets function in a ferrous system
- Physical bench tests were expanded for the new proposal along with some additional stability tests
  - Additional testing for robustness
  - Simulate transportation

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Section 3 - Product Changes

- New product overview
  - less water at the time of manufacture than Generation 1*
  - >50% organic with select additives to bolster performance
- **Primary change – move away from mineral oil and change to synthetic**

Generation 1 (GEN1) Emulsion (Mineral)

Generation 2 (GEN2) Emulsion (Synthetic)

*Due to the inclusion of water, it is assumed hydration level will change with usage

Section 3 - Basic Performance Tests + New Tests Review

- **ASTM D6138** – EMCOR – Why? Given the aqueous component, we needed to determine basic performance in a ferrous system
- **ASTM D2265** – Dropping Point – Why? With water present, what is the temperature at which the system will melt.
- **ASTM D2266** – 4-Ball Wear – Why? How much will water impact a basic performance parameter associated with bearing usage and life?
- **ASTM D2596** – 4-Ball EP – Why? One of the primary roles of this product that was communicated initially was to compete in EP applications. Can it carry load with water?
- **ASTM D6184** – Oil Bleed – Why? With water present – will it impact the amount of organic material lost during usage when at elevated temperatures?
- **DIN51805 @ 0 °C** – Low Temperature Flow – Why? We wanted to make sure that the system would not freeze and have some low temperature performance.
- **ASTM D1403/D217** – Cone Penetration – Immediate and extended – Why? QC test to indicate we are making the right grade of grease and extended to see how it compares to other grease products while being worked (standard development test)
- **ASTM D1742** – Oil Separation – Why? Added based comments on visual appearance after transport and shear stress
- **ASTM D1264** – Water Wash Out – Why? Added to see how the emulsion system hold us while exposed to a *new* water source
- **ASTM D1831** – Roll Stability – Why? Used to evaluate if and how much water might be lost under this type of stress
- **ASTM D4049** – Water Wash Off – Why? Added to see how the emulsion system hold us while exposed to a *new* water source

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Section 3 - Hydrated Grease Product GEN2 Test Results

ASTM Test	Description	Result	Typical
D1403	Cone Penetration	**267/265**	265-295
D217	Cone Penetration 100K	**312 (Δ47)**	Δ30 for HPM Core
D6138	EMCOR	**0,0**	0,1 for HPM Core
DIN 51805	Low Temp Flow	**0C = 150 mBar -5C = 200 mBar -10C = 225 mBar**	<1400 mBar at claim temperature
D1742	Oil Separation (Static)	**0**	<10% GC-LB, <5% HPM Core
D1264	Water washout	**21%**	<10% HPM Core
D1831	Roll Stability	**1.54%**	<15% HPM Core
D2265	Dropping Point	**>316° C without massive bubbling**	100 °C with water present (!)
D2266	4-Ball Wear	**0.52 mm**	0.6 mm HPM Core
D2596	4-Ball Weld	**400 kg**	250 kg HPM Core, 400 kg HPM +HL
D4049	Water Spray off	**71%**	<30% HPM – usually requires polymer
D6184	Bleed (Dynamic)	**0.6%**	<7% HPM Core

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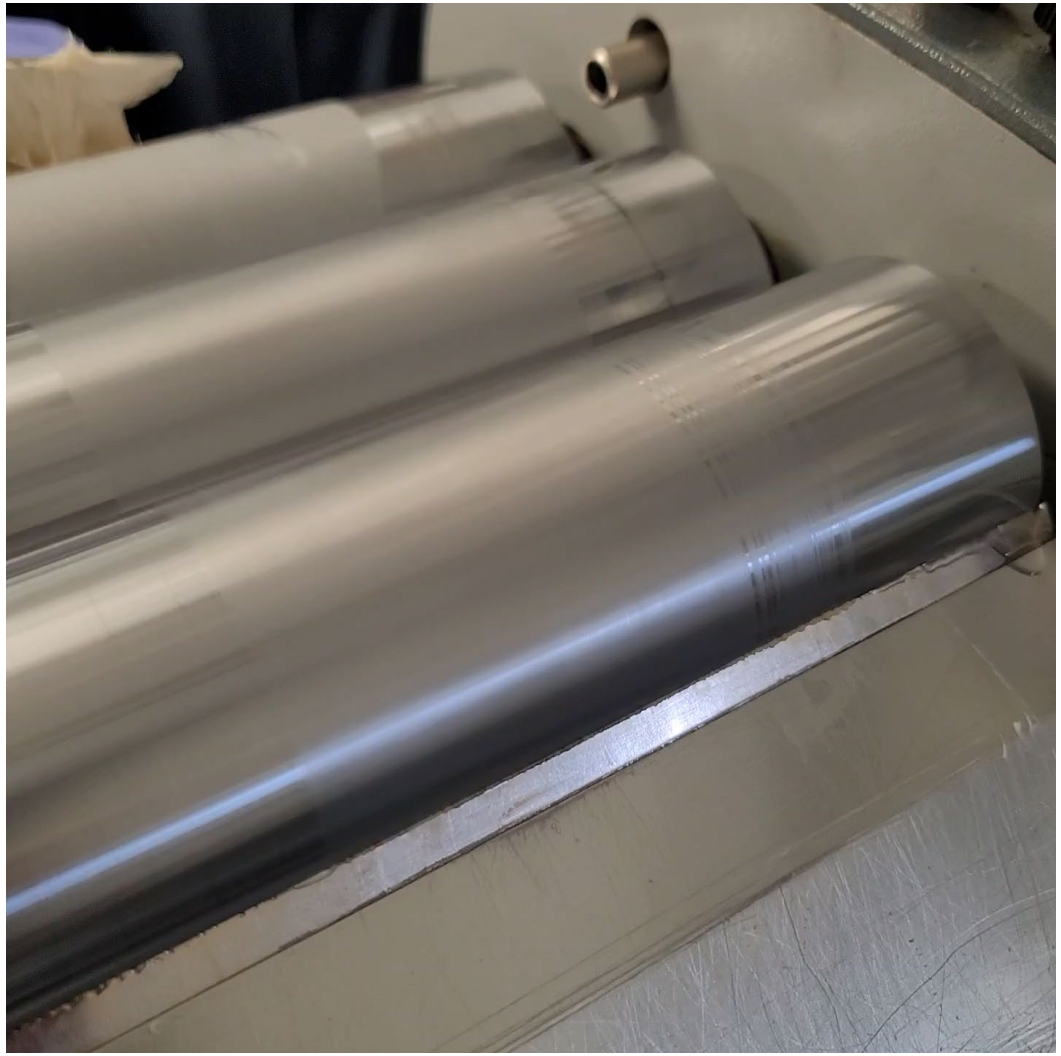
Section 3 - Additional Performance Tests

- Forced Bleed Test: <1 mil gap on EXACT Three Mill Roller
- Purpose – To stress the grease product and see if physical processing of the material at ambient temperatures causes any exit of the water component (HIPE product failed, GEN1 passed but did not survive transport)
- Result – The GEN2 material remains intact while being processed and milled multiple times



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Section 3 - Additional Performance Tests

- “Paint Shaker” Shipping Storage Stability Test for Emulsions used in explosives
- Purpose – To force water separation and simulate shipping low shear stress on the system (GEN1 failed shipping)
- Result – Typical test runs for 5 hours, and a successful candidate does not present any separation of water.

Sample after 5h stress

Section 3 – Life Cycle Assessment

- Disclaimer: LCA comparisons should be reviewed by an independent panel per ISO14040. This data has not been externally reviewed and is our initial assessment
- SimaPro/EcoInvent* was used for initial calculations with the general assumptions:
  - The underlying assumptions in this data are estimated to contribute to an uncertainty of ± 20%.
  - Global Warming kg CO2 eq is the category most grease customers have focused on currently
- Data is presented as **a percent relative to the highest kg CO2 eq**

Impact category	Unit	Synthetic Calcium Sulfonate Complex	Synthetic Lithium Complex EP	Hydrated Grease Product
Global warming (GWP100a)	kg CO2 eq	**100%**	**90%**	**61%**

- Note: 61% kg CO2 eq is roughly equivalent to the contributions from just the base oils in a grease formulation

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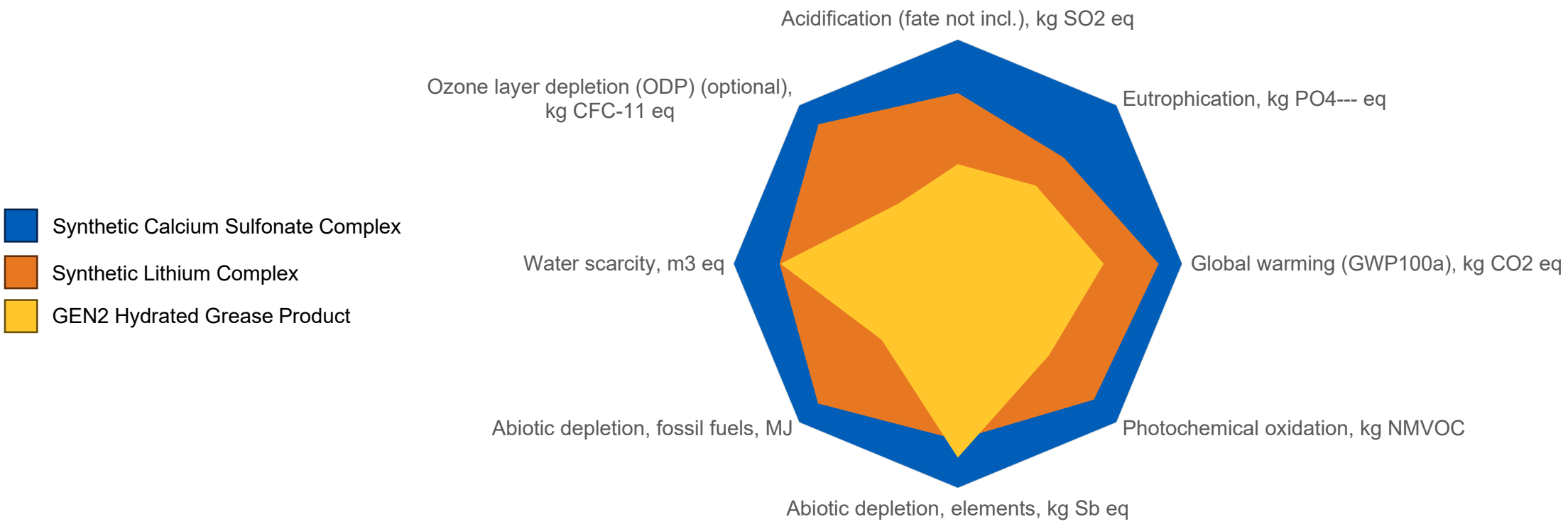
* EcoInvent is a product of SimaPro, a registered trademark of PRé Sustainability B.V.

Section 3 – Life Cycle Assessment

- Graphical representation of greases compared to each other – Hydrated Grease Product has a much smaller overall footprint with other common LCA considerations



Section 3 - Summary

- The extended bench tests and non-traditional stress tests have confirmed we have a more stable candidate for analysis than the HIPE product or GEN1 product
- Bench testing results are very good for an unprecedented product of this type, using a substance – water – that is typically deleterious to most lubricating grease applications and tests
- Odor profile of synthetic GEN2 improved significantly over mineral oil GEN1 version and HIPE product
- Biodegradability tests as next step

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Conclusions

Conclusions and Next Steps

- Current Gen2 product is promising
- Strong performance on bench tests; however, needs a field trial in a specific end use
- Looking for serious partners in this space to run field trials and complete complete technology development
- Questions?

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