New Oil Soluble Polyalkylene Glycols (OSP) for Grease Manufacture

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The Dow Chemical Company
Introduction

- New OSP technology offers many benefits in grease formulations
- OSPs offer options to upgrade hydrocarbon oils, naphthenic oils and synthetic base fluids to boost solvent power and improve additive compatibility
- **OSP-based Premium Lithium complex Grease provides Significantly higher temperature performance**
- The flexibility of PAG chemistry provides a huge amount of space for innovation and providing solutions to specifications and standards of the future
What is Grease?

- Grease is a combination of oil, additives and a thickener
- The oil and additives serve the same function as in a lubricating oil
- The thickener converts the liquid lubricant to a semi-solid lubricant
- A grease can’t be any better than its base oil
Evolution to OSP™ Technology

Current Applications

- Refrigeration oils
- Water glycol fluids
- Gear lubricants
- Bearing oils
- Hydraulic oils
- Air Compressor fluids

Water Soluble PAGs

Base Fluids

Polymer design space

Application Expansion

- Base fluid or co-base fluid for crankcase oils.
- Greases & Industrial oils.
- Transmission fluids.
- Metal rolling oils.
- Surfactants.

EO/PO

EO

PO

PO/BO

Bo/EO

OSP™ Technology
Traditional PAG Polymerization Technologies

Ethylene oxide (EO) | Propylene oxide (PO)

\[
\begin{align*}
\text{H}_2\text{C} & \text{O} \text{CH}_2 \\
\text{H}_2\text{C} & \text{O} \text{CH\text{--CH}_3}
\end{align*}
\]

Initiator

\[
\begin{align*}
\text{R'O} & \text{CH}_2\text{--CHO} \text{CH}_2\text{--OH} \\
\text{R} & \text{H, CH}_3
\end{align*}
\]

CLASSICAL POLYMER STRUCTURES BASED ON EO & PO

- Initiators are typically monols, diols or triols (for example butanol, propylene glycol, glycerol)
- Polymers can be designed having a wide range of viscosities (10-20,000 cSt at 40°C)
- Extremely versatile and can be tailored designed to have many specific functionalities

Initiator

PO block

EO block

1 EO

1 PO

Block Polymerization

Random Polymerization

Inverse block Polymerization
Synthetic Options in Designing Oil Soluble PAGs

**Oil miscibility of Homo-polymers**

**Increasing the carbon to oxygen ratio in PAGs improves oil miscibility**

- **Ethylene oxide**
- **Propylene oxide**
- **Butylene oxide**

**PO Homo-polymers are partially oil miscible**

**EO Homo-polymers are water soluble and not oil miscible**

**Carbon/oxygen ratio**

Oil miscibility in reference to Group I-IV Hydrocarbon oils
Oil Soluble Polyalkylene Glycols – Benefits

**BENEFITS**

✔ Availability in a very wide range of viscosity grades with design flexibility
✔ High viscosity index
✔ Good low temperature properties
✔ Excellent deposit control and equipment cleanliness
✔ Hydrolytic stability
✔ Safe to Use

**NEW BENEFITS**

△ Oil Miscibility (Compatibility)
△ Low Aniline Point
△ Solvency Provider
△ Excellent Capability for Additive Solubility
What is Different?

CHEMICAL COMPOSITION: POLYALKYLENE GLYCOL VS. HYDROCARBON OILS

Mineral Oil – Petroleum-based Hydrocarbon Group I Base stock

Non-Polar
Heavily additized for oxidation stability and has poor solvency

PAO- Poly Alpha Olefin- Synthetic Hydrocarbon Oil Group IV

PAG – Poly Alkylene Glycol Base Fluid Group V

Polar
Inherently thermo-oxidatively stable with high VI
## OSP Typical Properties

<table>
<thead>
<tr>
<th>OSP Grade</th>
<th>Aniline point temperature, °C</th>
<th>Viscosity at 40°C, cSt</th>
<th>Viscosity at 100°C, cSt</th>
<th>Viscosity Index</th>
<th>Pour point, °C</th>
<th>Flash point (COC), °C</th>
<th>Fire point, °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>OSP-32</td>
<td>&lt; -30.0</td>
<td>32</td>
<td>6.5</td>
<td>146</td>
<td>&lt; -43</td>
<td>216</td>
<td>242</td>
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<tr>
<td>OSP-46</td>
<td>&lt; -30.0</td>
<td>46</td>
<td>8.5</td>
<td>164</td>
<td>&lt; -43</td>
<td>210</td>
<td>240</td>
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<tr>
<td>OSP-68</td>
<td>&lt; -30.0</td>
<td>68</td>
<td>12</td>
<td>171</td>
<td>&lt; -40</td>
<td>218</td>
<td>258</td>
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<tr>
<td>OSP-150</td>
<td>&lt; -30.0</td>
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<td>23</td>
<td>186</td>
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<td>OSP-220</td>
<td>-26.0</td>
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<td>32</td>
<td>196</td>
<td>-34</td>
<td>226</td>
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<tr>
<td>OSP-680</td>
<td>ND</td>
<td>680</td>
<td>77</td>
<td>196</td>
<td>-30</td>
<td>243</td>
<td>260</td>
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### Comparison Typical Properties of OSP’s / PAO’s

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<tr>
<th>UCON Grade</th>
<th>Viscosity at 40°C, cSt</th>
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<tbody>
<tr>
<td></td>
<td>ASTM D445</td>
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<td>ASTM D2270</td>
<td>ASTM D97</td>
</tr>
<tr>
<td>PAO-6</td>
<td>31</td>
<td>5.1</td>
<td>138</td>
<td>-57</td>
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<td>PAO-8</td>
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<td>8.0</td>
<td>139</td>
<td>-48</td>
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<td>PAO-10</td>
<td>66</td>
<td>10</td>
<td>137</td>
<td>-48</td>
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<tr>
<td>PAO-40</td>
<td>396</td>
<td>29</td>
<td>147</td>
<td>-36</td>
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<tr>
<td>PAO-100</td>
<td>1240</td>
<td>100</td>
<td>170</td>
<td>-30</td>
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</table>
## Miscibility of OSPs in Common Base Oils

### Typical Miscibility Features

<table>
<thead>
<tr>
<th>Chemistry</th>
<th>OSP/Base Oil 10/90 w/w</th>
<th>OSP/Base Oil 50/50 w/w</th>
<th>OSP/Base Oil 90/10 w/w</th>
</tr>
</thead>
<tbody>
<tr>
<td>Group I Mineral oils</td>
<td>Miscible</td>
<td>Miscible</td>
<td>Miscible</td>
</tr>
<tr>
<td>Group II and III Mineral oils</td>
<td>Miscible</td>
<td>Miscible</td>
<td>Miscible</td>
</tr>
<tr>
<td>PAO-4, 6, 8</td>
<td>Miscible</td>
<td>Miscible</td>
<td>Miscible</td>
</tr>
<tr>
<td>Diesters &amp; Polyol esters</td>
<td>Miscible</td>
<td>Miscible</td>
<td>Miscible</td>
</tr>
<tr>
<td>Naphthenics</td>
<td>Miscible</td>
<td>Miscible</td>
<td>Miscible</td>
</tr>
<tr>
<td>PAG’s - PO homopolymers</td>
<td>Miscible</td>
<td>Miscible</td>
<td>Miscible</td>
</tr>
</tbody>
</table>

Miscibility defined as clear homogeneous solutions before and after storing at ambient temperature and 80°C for 168 hours.
Non-Polar Fluid + High MW Polar By-Products = Agglomeration of Soft Contaminates

Polar Fluid + Low MW Polar By-Products = Homogeneous Solution

High MW molecules grow, forming insoluble varnish that adheres to surfaces

Additional degradation may occur, but by-products remain in solution

Temperature, System, & time

Mineral & Synthetic Hydrocarbon Oil vs. Polyalkylene Glycols
Tribological Properties - MTM Curves of OSP vs. Hydrocarbon Oil

Steel Ball on Steel Disc using MTM, GPa 1.08, at 130°F and 160°F
Benefits OSP-based Premium Lithium complex Grease

- Significantly higher temperature performance
  Oxidation Induction Time
- Significantly increased dropping point
  313°C versus 200°C of benchmark greases
- Significantly reduced energy and time to produce
  Excellent Solvating Power (Aniline Point)
Aniline Point for Various Base Stocks

- **Group I (MO)**
  - VI 95

- **Group II (MO)**
  - VI 102

- **Group III (MO)**
  - VI 135

- **Group IV (PAO)**
  - VI 136

- **Group V (PAG)**
  - VI >200

- **Group V (Napthenic oil)**
  - VI 45

Solvating Power:
- Poor
- Strong
OSP as Solvency Provider

- Aniline Point (°C)
- OSP + T
  - \( \text{OSP} + \text{T} - 22 \)
  - \( \text{Spec 6} - 6 \)

Bar graph showing:
- OSP + Spec-6
- T-22
- OSP + T-22

Y-axis: Aniline Point (°C)
- 0
- 20
- 40
- 60
- 80
- 100
- 120
- 140

X-axis: Solvating Power
- Poor
- Strong
## Dow OSP 220 Grease Typical Properties

<table>
<thead>
<tr>
<th>Property</th>
<th>Method</th>
<th>Result</th>
</tr>
</thead>
<tbody>
<tr>
<td>Color</td>
<td>Visual</td>
<td>Light Beige</td>
</tr>
<tr>
<td>Appearance</td>
<td>Visual</td>
<td>Smooth</td>
</tr>
<tr>
<td>Po, mm/10</td>
<td>ASTM D217</td>
<td>265</td>
</tr>
<tr>
<td>P60, mm/10</td>
<td>ASTM D217</td>
<td>275</td>
</tr>
<tr>
<td>NLGI Grade</td>
<td>ASTM D217</td>
<td>2</td>
</tr>
<tr>
<td><strong>DP °C</strong></td>
<td><strong>ASTM D2265</strong></td>
<td><strong>313</strong></td>
</tr>
<tr>
<td>Oil Separation, 24h, 100°C, %</td>
<td>ASTM D6184</td>
<td>0.00</td>
</tr>
<tr>
<td>Evaporation, 24h, 100°C, %</td>
<td>CTM</td>
<td>0.75</td>
</tr>
<tr>
<td><strong>Water Washout, 79 °C, %</strong></td>
<td><strong>D 1264</strong></td>
<td>13.6</td>
</tr>
<tr>
<td>PDSC, 175°C, minutes</td>
<td><strong>D 5483</strong></td>
<td>&gt;120 (see graph)</td>
</tr>
<tr>
<td>Cu Corrosion, 24h at 100°C</td>
<td>ASTM D4048</td>
<td>1A</td>
</tr>
<tr>
<td>Four Ball Wear, mm</td>
<td>ASTM D2266</td>
<td>0.54</td>
</tr>
<tr>
<td><strong>Oxidation Test @ 100 hr, psi</strong></td>
<td><strong>ASTM D942</strong></td>
<td><strong>1.8</strong></td>
</tr>
<tr>
<td>Low Temperature Apparent Viscosity at -29.5°C, m Pa.s</td>
<td>CTM</td>
<td>0.4 x 10$^6$ mPa.s</td>
</tr>
<tr>
<td><strong>Low Temp Torque, -40 ºC</strong></td>
<td><strong>ASTM D 4693</strong></td>
<td><strong>3.88 (see graph)</strong></td>
</tr>
<tr>
<td>Four Ball EP, Weld Load, Kg Load wear Index</td>
<td><strong>ASTM D 2596</strong></td>
<td><strong>200 62</strong></td>
</tr>
<tr>
<td>Roll Stability, P60 Change</td>
<td><strong>ASTM D 1831</strong></td>
<td>+30</td>
</tr>
</tbody>
</table>

Soap Content: 9.5%
OSP-220: VI 196
Pressurized Differential Scanning Calorimetry (PDSC) Method

Determination of the OIT on a thermal curve

<table>
<thead>
<tr>
<th>Parameter Assessed</th>
<th>Heat Flow vs. Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>Reported Data</td>
<td>Oxidation Induction Time (OIT)</td>
</tr>
</tbody>
</table>
| Gas                | Gas Composition: Oxygen  
                      Gas Flow: 6 l/h  
                      Gas Pressure: 3.5MPa |
| Sample Size        | 2.0 mg |
| Pans               | Solid Fat Index (SFI), AL |
| Temp Programme     | Isothermal, between 155 & 210 °C |
PDSC Thermal Curve: OIT of OSP 220 Grease and Lithium Grease
Low Temperature Torque – ASTM D 4693

Sample: Grease 220  
Lab#: 10102102  Maximum Torque = 3.88 N-m  
Test Temperature: -40 °C  Date: 11/3/10  
60 Second Reading: 3.14 N-m
Conclusions

• The formulators and researchers have another option for using PAGs as a “tool” for solving some of our industry problems.
• Equipment conversions from hydrocarbon oils to Oil Soluble PAGs is simpler and less problematical
• OSPs offer options to upgrade hydrocarbon oils & synthetics to boost additive solubility for robust grease formulations
• OSPs can provide improved aniline point when used as co-base oil in grease formulations.
• The flexibility of PAG chemistry provides a huge amount of space for innovation and providing solutions to specifications and standards of the future
THANK YOU
STLE 2011 Annual Meeting & Exhibition
May 15-19, 2011
Atlanta, GA
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