SUPERLINK™ CROSS-LINKABLE ROTOMOULDING RESINS

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February 2019
Ingenia Polymers In Rotomoulding

• Ingenia is a global supplier to the rotomolding industry with a track record of developing innovative products

• Superlink™ cross-linkable polyethylenes exemplify this commitment to excellence in rotomoulding.
SUPERLINK™ XLPE

• Traditional linear materials do not meet the requirements of high performance applications including fuel and hydraulic oil tanks:
  • High impact and/or low temperature impact
  • Improved ESCR
  • Improved temperature resistance

• Ingenia Superlink™ XLPE can meet these requirements either as a single layer rotomoulded product or as part of a multilayer solution for applications requiring low fuel permeation.
## Property Comparison

<table>
<thead>
<tr>
<th>Resin Type</th>
<th>Superlink XLPE</th>
<th>Typical LLDPE/HDPE</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>0.943</td>
<td>0.932-0.942</td>
</tr>
<tr>
<td>Impact resistance (ARM test, -40 °C) (ft-lbs)</td>
<td>&gt;180</td>
<td>80-160</td>
</tr>
<tr>
<td>Brittle failure (%)</td>
<td>0</td>
<td>85%-100%</td>
</tr>
<tr>
<td>Tensile elongation (%)</td>
<td>800</td>
<td>865-1000</td>
</tr>
<tr>
<td>Flexural Modulus (psi)</td>
<td>110,000</td>
<td>88-110,000</td>
</tr>
<tr>
<td>ESCR (hrs)</td>
<td>&gt;1000</td>
<td>20-400 typical</td>
</tr>
<tr>
<td>HDT (°C) ASTM D648 at 66psi</td>
<td>67</td>
<td>49-57</td>
</tr>
<tr>
<td>Chemical Resistance</td>
<td>Excellent</td>
<td>Fair</td>
</tr>
<tr>
<td>Moldability</td>
<td>Excellent</td>
<td>Good</td>
</tr>
</tbody>
</table>
Impact Strength vs Density

- Linear PE resins show a negative correlation between impact strength and increasing density.
- Superlink™ XLPE shows highly improved impact strength relative to density as compared to linear PE.
- Besides improvements in impact strength, crosslink resins offer improvements in several properties...
What is Crosslinking?

- Crosslinking is a chemical reaction.
- It is initiated by heat or temperature.
- Rate is time and temperature dependent.
- It changes the composition of PE from a collection of linear thermoplastic polymer chain to a thermoset network chain structure.
Measuring Crosslinking

• Crosslinking of Superlink resins is initiated by decomposition of peroxide
• A torque rheometer can measure crosslinking characteristics
  • Viscosity and torque will increase as crosslinking proceeds
  • The time to reach a percentage (50 or 90%) of maximum torque is indicative of the rate of cross-linking. Shorter times = higher rates
Measuring Crosslinking via Torque Rheometer

- **M<sub>HF</sub>** – Plateau Torque
- **M<sub>L</sub>** – Minimum Torque
- **t<sub>90</sub>** – 90% cure time
- **t<sub>50</sub>** – 50% cure time
- **t<sub>s2</sub>** – time to reach 2 in-lb above M<sub>L</sub> (Scorch Time)
Rotomolding Process and Crosslinking

- Linear PE
  - Loading of powder
  - Heating of mold and powder
  - Fusion of resin
  - Cooling
  - Removal of part

- Cross-linkable PE
  - Loading of powder
  - Heating of mold and powder
  - Fusion of resin
  - Chemical crosslinking
  - Cooling
  - Removal of part
Rotomolding process and Crosslinking

- XLPE requires a higher peak internal air temperature (PIAT) as compared to linear PEs during rotomolding
Impact properties vs. cycle time

- Linear PEs are known to degrade if the rotomoulding cycle is extended. The resin ‘overcooks’, and undergoes thermal and oxidative degradation. Impact properties are severely negatively affected.
- However XLPEs maintain their impact properties if the rotomoulding cycle is extended beyond the full gel point.
Peroxide decomposition and crosslinking process

- Measuring gel content of finished product confirms complete crosslinking.
- The gel content is measured by dissolving the PE in recirculating hot xylene. Linear PEs will completely dissolve. XLPE will have a content of undissolvable ‘gel’.
- A typical gel content of 70-84% indicates full cross-linking and fully developed physical properties.
  - Increased toughness
  - Increased stress crack resistance
  - Increased notch crack resistance
  - Increased abrasion resistance
Due to the nature of the rotomoulding process – where heat is applied from the outside – the part thickness has a gradient in heat history and corresponding gel level. → the gel content will be higher on the outside wall.
Property Comparison

• Superlink has been tested extensively and has a proven track record of 20+ years
• Superlink XLPE outperforms linear materials
• Superlink has also been evaluated against industry competitive XLPEs and demonstrates superior processability and properties
• Differences between XLPE resins can be attributed to differences in:
  • Base resin
  • Crosslinking system (peroxide type, concentration and use of co-additives)
  • Antioxidant system
  • UV additives
  • Pigments
Comparison of Industry XLPEs

- Ingenia regularly benchmarks its Superlink™ XLPE versus industry competitors
## Property Comparison

<table>
<thead>
<tr>
<th>Resin Type</th>
<th>SL110NA</th>
<th>Competitive 1</th>
<th>SL110CA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lot#</td>
<td>113218 (Natural)</td>
<td>Natural</td>
<td>112376 (Black)</td>
</tr>
<tr>
<td>Density</td>
<td>0.942</td>
<td>0.941</td>
<td>0.942</td>
</tr>
<tr>
<td>Impact resistance (ARM test, -40 °C, ¼&quot;) (ft-lbs)</td>
<td>183</td>
<td>181</td>
<td>186</td>
</tr>
<tr>
<td>Tensile Strength at Yield (psi)</td>
<td>2921</td>
<td>2806</td>
<td>2935</td>
</tr>
<tr>
<td>Tensile Strength at Break (psi)</td>
<td>899</td>
<td>904</td>
<td>860</td>
</tr>
<tr>
<td>Flexural Modulus (psi)</td>
<td>113,000</td>
<td>101,200</td>
<td>111,800</td>
</tr>
<tr>
<td>ESCR (hrs)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>100% Igepal</td>
<td>&gt;1000</td>
<td>&gt;1000*</td>
<td>&gt;1000</td>
</tr>
<tr>
<td>10% Igepal</td>
<td>&gt;1000</td>
<td>&gt;1000*</td>
<td>&gt;1000</td>
</tr>
<tr>
<td>HDT (°F)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ASTM D648 at 66 psi</td>
<td>152</td>
<td>141*</td>
<td></td>
</tr>
<tr>
<td>264 psi</td>
<td>104</td>
<td>98*</td>
<td></td>
</tr>
</tbody>
</table>

*from datasheet. All other properties measured
Comparison of Industry XLPEs

- Rheological studies show that Ingenia Superlink has a longer crosslinking time (t50 and t90) at lower temperatures and a similar crosslinking time at higher temperatures to competitive XLPE.
- A lower reactivity at low temperatures allows Superlink to fully fill the mold, fill out detail, and fully fuse without entrapped air bubbles, before crosslinking.
- A higher reactivity at higher temperatures allows Superlink to fully crosslink without extended heating cycles.
Comparison of Industry XLPEs

- Crosslinking time is inversely proportional to reactivity.
- Superlink is less reactive at lower temperatures and has increased reactivity at higher temperatures.
- This provides good moldability:
  - Good fill and detail
  - Complete melting and fusion prior to crosslinking
  - Efficient molding times
Comparison of Industry XLPEs

- Rheological studies show that Superlink has a higher maximum torque than industry competitive XLPE.
- Maximum torque is an indication of crosslink density and gel content.
Comparison of Industry XLPEs

- Ingenia has carried out extensive long term testing of Superlink and competitive XLPE.
- Ingenia Superlink has superior QUV resistance to industry competitive XLPE.
- All XLPEs have comparatively low UV resistance compared to well stabilized linear resins.
Comparison of Industry XLPEs

- The UV performance of Superlink was confirmed by Xenon Arc Weatherometer, which is considered more representative to real world conditions than QUV.
Comparison of Industry XLPEs

• Materials have also been evaluated for resistance to thermal aging
Comparison of Industry XLPEs

- SuperlinkTM shows equivalent performance to industry competitive XLPE in thermal aging tests
Long Term Testing of Superlink™

- HYDROSTATIC PRESSURE TESTING (ASTM D1998/D2837)
- LTHS: LONG TERM HYDROSTATIC STRENGTH
  - Samples are exposed to pressurized high temperature water for an extended period of time
  - Property that resists stress in the hoop direction generally due to hydrostatic forces in applications such as pipe, pressure vessels or upright storage tanks
  - LTHS improves as MW increases (this is why pipe is extruded from high to very high MW resins)
  - Roto XLPE is generally 30% higher LTHS vs LLDPE of similar density
  - Critical for large tanks (i.e. wall thickness/material usage, process window for thick walled parts)
Long Term Hydrostatic Testing

- Preparation of samples for LTHS
- Samples are tested until failure
Long Term Hydrostatic Testing

- Testing at different pressure and temperature allows extrapolation to extended times at lower temperatures.
WHAT DOES ALL OF THIS MEAN TO THE MOLDERS?

• Superlink offers improved properties over linear materials for high performance applications including large chemical tanks and fuel tanks
• Performance has been demonstrated in extensive laboratory and field testing and supported by industry certifications:
  • UNECE-R34 TUV certificate for wheel vehicle part application;
  • UL94 HB burning test certificate,
  • Open fire resistance testing certificate per ABYC H24 specification for marine fuel tank application (provided on request)
• Processability and performance are improved versus industry competitive XLPE.
Molders observations
  • Lower scrap rate
  • Faster cycle time
  • Fewer defects and rejects (Pinholes, Voids, Blemishes)
XLPE applications

- Fuel tanks for gasoline and diesel
  - (single layer or part of a multilayer solution depending on local fuel permeability requirements)
- Large chemical tanks
- Road barriers
- Waste bin covers
- Go-cart bodies
- High impact doors
XLPE Grades

SL110 Series: High performance XLPE resin with wide processing window.

SL120 Series: ‘Low odor’ product

SL114 Series: High flow, high productivity grade for molding challenging product design at very high oven temperatures, short oven times with moderated gel content.

SL230 series: High adhesion grade for bonding to metal. Used for lining metal tanks in order to provide excellent chemical resistance.
Processing Guidelines for SUPERLINK™

• Oven temperature ~510F—600F (265C to 315C)
• PIAT target:  SL110 series----195C to 210C  
  SL120 series----195C to 205C  
  SL114 series----205C to 215C  
  SL230 series----195C to 210C  
• We suggest the use of AXEL WB 4606 mold release for all Superlink™ resins
• 2” vent opening.
• Cycle time: ~18 to 28 minutes depending on part thickness and design, mold specifics and oven temperature.
Benefits of Superlink™

- Greatly improved stiffness - toughness balance as compared with LLDPE and HDPE:
- Improved temperature resistance
- Improved abrasion resistance
- Improved stress crack resistance
  - propagation failure

Superlink™ was developed to meet the demanding requirements for applications requiring outstanding durability, improved abrasion and scratch resistance and improved ESCR.