“Technology and Innovation in the use of Micronized Rubber Powder in today’s Green World”

11 April, 2013
• Setting the Stage and What we are Learning

• Who is Lehigh Technologies

• Technical Presentation

• What Does it All Mean in Terms of Green?
Millions of End-of-Life Tires Generated Each Year

Energy Recovery
Civil Engineering
Landfill Stockpiled
Data Not Available

USA: 292
Canada: 30
Brazil: 250
Europe: 112
South Africa: 80
China: South Korea: Japan: Malaysia: Australia: New Zealand:

Lehigh Technologies Inc.
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The First Chemical Revolution

- **1800s**
  - dyes/pigments
  - metals
  - soaps

- **1850-1900**
  - oil and gas discoveries
  - carbohydrates

- **1900-1930**
  - cracking/refining
  - synthetic chemistry
  - atomic theory and the chemical bond
The World Today – 3 Challenges

- 4lbs /person/day
- over 200 million tons per year
- oil prices over $80 and spiking to >$100/bbl
- borrow-buy-burn is US energy strategy
- world population 7B people
- 1 billion in the developed world consume as much energy as the other 6B.
The Second Chemical Revolution

infinite cycles of use
- Bury or burn is not a solution
- Vast resource pools available
- Huge technology challenge

sustainable production of building blocks
- Must be waste based.
- Amyris, Kior, Renmatix, Genomatica
- Small companies leading

efficient use of existing carbon sources
- Principles of Green Chemistry
- Chemical companies leading
Micronized Rubber Powder Industry – Lehigh Experience

- **Image of industry:**
  - Reliability of supply-process safety; quality
  - Scale—not capable of supporting global applications
  - Capitalization
  - “Tried it 15 years ago – didn’t work”

- **Absence of technical knowledge:**
  - No shortage of conjecture
  - Very little data—insufficient experimental rigor
  - Potential customers “on their own”
  - No technical knowledge = no adoption

- **Long sales cycles:**
  - All performance markets have long development cycles—testing, optimization, re-testing, production trials, optimization-launch—that’s why stuff works!
  - All markets need foundational knowledge and technical support-customers rarely have sufficient resources to do this—mindset matters.
Infinite Uses – The Next Steps

- **Change in mindset:**
  - Move from “getting rid of waste material” to “supplier of specialty materials”.
  - Good science and technical support drive adoption.
  - This changes the way everything is done.

- **Raw material type and structure:**
  - Different rubber compositions and tire components behave differently in complex formulations-tire compounds, asphalt systems, plastics etc.
  - Only limited ability to provide discrete raw materials for specific uses.
  - This requires “demand” and “capability”.

- **History suggests that the value proposition enables adoption not subsidies:**
  - Chemical building blocks have always delivered performance at economic cost
  - No customer pays more unless the material delivers more
  - Bio-based feedstocks, solar cells etc are burdened with this issue
  - We must drive to provide value and performance-
Lehigh Technologies: Overview

**Key Facts**
- Headquartered in Tucker, GA
- Founded in 2003
- 75 employees
- Blue Chip Investors:
  - KPCB
  - Index Ventures
  - NGP Energy Technology Partners

**Proven Technology**
- +140 million tires manufactured utilizing Lehigh’s PolyDyne™
- 45,000MT of annual manufacturing capacity
- 5 out of top 10 tire manufacturers companies are Lehigh customers
- Up-cycle post-consumer scrap
- ISO 14001 / 9001 Certified
- PolyDyne / MicroDyne

Lehigh Technologies is a technology-driven green materials manufacturer that turns end-of-life tires and other post-industrial rubber into sustainable powders that are used in a wide range of high-value industrial and consumer applications.
Lehigh Technologies Particle Sizes Range from 40 to 300 Mesh – Clean: Metal & Fiber Free

Expanded Product-Line to Include EPDM, Nitrile, Butyl & Natural Rubber Powders in Certain Sizes
Optimization of Rubber Compounds: Incorporating Sustainable, Micronized Rubber Powders
Agenda

- Introduction
- Experimental
- Results and Discussion
  - Addition Point of Micronized Rubber Powder (MRP)
  - Sulfur-Accelerator Optimization
  - Particle Sizes
- Summary of Findings and Recommendations
Introduction

- **The status of End-of-Life Tires**
  - On a global basis each year, one billion tires become unusable and are classified as end-of-life.
    - In the European Union, the practice of land filling tires was banned in 2006.
- **The need for the work**
  - The rubber rheological and physical properties are adversely affected when MRP is added to the mix
    - Cure rate and viscosity increases
    - General physical property decline
    - Reduced performance in abrasion, higher heat build-up and compression set, and increased hysteresis
  - Many rubber article manufacturers do not modify their base recipes when incorporating MRP.

1. ETRMA source document
Introduction

Many researchers have offered explanations for the reported losses in rheological and physical properties when incorporating MRP into new rubber\textsuperscript{1-9}

- MRP particles in new cured rubber are discontinuities and act like stress-raising flaws.\textsuperscript{4}
- Scanning electron microscope (SEM) photographs showing improper bonding of MRP to the new rubber matrix.\textsuperscript{6}
- The decreased modulus is caused by sulfur in the new rubber matrix migrating into the MRP causing a lower cross-link density in the final product.\textsuperscript{2,3,9}
- The shorter scorch times and faster cure rates which are explained by the migration of accelerator fragments from the MRP into the new rubber matrix.\textsuperscript{3}
- Trouser tear resistance actually improves slightly and this effect was explained by the theory of crack tip blunting.\textsuperscript{4}
- Reviews and summaries were presented previously by this author elsewhere.\textsuperscript{7,8}
# Experimental – Base Compound

<table>
<thead>
<tr>
<th>Addition Sequence</th>
<th>Ingredients</th>
<th>Base Compound</th>
</tr>
</thead>
<tbody>
<tr>
<td>First Pass</td>
<td>ESBR1500 (Non-oil extended)</td>
<td>70.00</td>
</tr>
<tr>
<td>First Pass</td>
<td>High Cis Polybutadiene Rubber</td>
<td>30.00</td>
</tr>
<tr>
<td>First Pass</td>
<td>Micronized Rubber Powder (MRP) From Whole Tire</td>
<td>As Per Studies</td>
</tr>
<tr>
<td>First Pass</td>
<td>N339 Carbon Black</td>
<td>65.00</td>
</tr>
<tr>
<td>First Pass</td>
<td>Heavy Naphthenic Process Oil</td>
<td>25.00</td>
</tr>
<tr>
<td>First Pass</td>
<td>Homogenizing Agent</td>
<td>1.00</td>
</tr>
<tr>
<td>First Pass</td>
<td>Alkyl Phenol Formaldehyde Novolak Tack Resin</td>
<td>3.00</td>
</tr>
<tr>
<td>First Pass</td>
<td>6PPD Antidegradant</td>
<td>2.50</td>
</tr>
<tr>
<td>First Pass</td>
<td>TMQ Antidegradant</td>
<td>1.50</td>
</tr>
<tr>
<td>First Pass</td>
<td>Microcrystalline and Paraffin Wax Blend</td>
<td>2.50</td>
</tr>
<tr>
<td>First Pass</td>
<td>Zinc Oxide Dispersion (85% ZnO)</td>
<td>3.53</td>
</tr>
<tr>
<td>First Pass</td>
<td>Stearic Acid</td>
<td>2.00</td>
</tr>
<tr>
<td>Finish Pass</td>
<td>TBBS Accelerator</td>
<td>1.00</td>
</tr>
<tr>
<td>Finish Pass</td>
<td>Sulfur Dispersion (80% Sulfur)</td>
<td>2.50</td>
</tr>
<tr>
<td>Finish Pass</td>
<td>Retarder N-(cyclohexylthio) phthalimide</td>
<td>0.10</td>
</tr>
</tbody>
</table>

Total PHR Finish Batch: 209.63
Density kg/l: 1.126
Experimental Procedure

- All mixes were performed in a 1.6L Farrel Banbury internal mixer.
  - Mixing was conducted either as a two or three pass mix
- Milling was performed on a KSB 2-roll mill 33 cm x 15 cm.
- In all studies for each operation, weighing, mixing, curing, and testing, a unique randomized sequence was employed to reduce or eliminate bias scatter of the data.
- Some of the experimental designs used replication of batches, and some of the designs with replicated batches used a procedure of blending the master batches for reducing variation.

- MDR2000 Rheometer ASTM D 5289 @ 160°C
- Tensile, Elongation, Modulus ASTM D 412, unaged & oven aged
- Trouser tear resistance, ASTM D 624 T, unaged & oven aged
- Hardness tested with Rex Digital Durometer, ASTM D 2240 Type A on rebound specimens
- BF Goodrich Flexometer ASTM D 623, Method A
- Zwick Rebound ASTM D 7121
- Zwick Rotary Drum Abrader ASTM D 5963, Method A
- Static Outdoor Exposure (20% Strain) ASTM D 518, Method A
Introduction

MDR Rheometer 160°C Tread with 177 Micron RRP
Normalized Time to Stated Property

Normalized Physical Properties of Tread
with 177 Micron MRP

Flexometer Heat Build-Up and Compression Set
Tread with 177 Micron MRP

Normalized Abrasion Resistance Index and Rebound
@ 60°C Tread with 177 Micron MRP
Addition Point of MRP

Which addition point in the mix for the MRP gives better physical properties?

- In the first master pass with the following
  - Polymers
  - Carbon black
  - Chemicals

- In the finish pass with curatives
  - Take advantage of any remaining bonding capability?\(^1\)

<table>
<thead>
<tr>
<th>Mix Pass</th>
<th>First Pass</th>
<th>Second Pass</th>
<th>Finish Pass</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Mix Time</td>
<td>0</td>
<td>1</td>
</tr>
<tr>
<td></td>
<td>Material</td>
<td>BMB</td>
<td>½ CB</td>
</tr>
<tr>
<td></td>
<td>Addition</td>
<td>Polymers</td>
<td>½ MRP</td>
</tr>
<tr>
<td></td>
<td></td>
<td>MRP</td>
<td>Oil</td>
</tr>
</tbody>
</table>

1. Private conversations and data.
Results of the addition point in the mix for the MRP

- Earlier in the first pass for optimum tensile and modulus
- Later in the first pass mix for optimum elasticity
Sulfur-Accelerator Optimization

- The study design used the base recipe shown earlier in a two pass mix version.
- The 177 µm MRP from whole tire was used from 2% to 14% by weight loading.
- Sulfur loading from 1.5 phr to 4.0 phr.
- TBBS loading from 0.2 phr to 1.4 phr.
- The DOE used was a central composite design, response surface method, with the aid of Design Expert® software published by Stat-Ease, Inc.
Performance From Actual Mix

177 Micron MRP 10% Loading Sulfur Optimization Actual Data

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>177 µm</th>
<th>177 µm+S</th>
</tr>
</thead>
<tbody>
<tr>
<td>MRP %</td>
<td>0.0</td>
<td>10.0</td>
<td>10.0</td>
</tr>
<tr>
<td>Sulfur phr</td>
<td>2.0</td>
<td>2.0</td>
<td>2.5</td>
</tr>
<tr>
<td>TBBS phr</td>
<td>1.0</td>
<td>1.0</td>
<td>1.0</td>
</tr>
</tbody>
</table>
Particle Size Study

Project Description

- MRP of 400, 300, 177, and 105 µm (commercially from whole tire)
- MRP loadings of 3%, 6%, and 10%
- Standard tread with SBR
- Two batches of control compound and one mix each of the MRP batches made
- Control master batches blended together before mixing the next pass
- Randomization used for each step
- All MRP added with the carbon black in the first pass
- Three pass mixing used with sulfur and accelerator optimization
- Researchers in the UK estimated the intrinsic flaw size of carbon black filled SBR, without MRP, to be 130 µm

Effect of Particle Size on Physical Properties

![Normalized Unaged Tensile vs. Normalized Unaged E@B](image)
Effect of Particle Size on Physical Properties

Normalized Unaged Tensile vs. Normalized Unaged 300M

- PD40+
- PD80+
- PD84+
- PD140+

Legend:
- Control
- 400 µm 3%
- 300 µm 3%
- 177 µm 3%
- 105 µm 3%
- 400 µm 6%
- 300 µm 6%
- 177 µm 6%
- 105 µm 6%
- 400 µm 10%
- 300 µm 10%
- 177 µm 10%
- 105 µm 10%
Summary of Findings & Recommendations

- For optimum tensile and modulus, mix MRP’s into the first pass, either with the polymers or the carbon black. For optimum E@B, mix MRP’s later in the first pass with the chemicals.

- Increasing the sulfur and slightly decreasing the accelerator levels can recover many of the physical properties to nearly match the same compound without MRP.

- There appears to be no antidegradants migrating from MRP’s into the new rubber matrix when the MRP’s have been made from end-of-life tires.

- For products with demanding performance requirements, such as tires, the largest particle size MRP to use at 10% loading while maintaining basic physical properties is the 105 μm product.
Micronized Rubber Powder Applications – cont’d

<table>
<thead>
<tr>
<th>Tires</th>
<th>Waterproofs, Improves Traction</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Better Cost Management, Sustainable Materials without Negative Performance Impact</strong></td>
<td><strong>Waterproofs, Sound Dampener, Insulator</strong></td>
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<tr>
<td><strong>Waterproofs, Sound Dampener, Insulator</strong></td>
<td><strong>Polypropylene Applications</strong></td>
</tr>
<tr>
<td><strong>Polyurethane Foam</strong></td>
<td><strong>HDPE Applications</strong></td>
</tr>
<tr>
<td><strong>Waterproof Membranes</strong></td>
<td><strong>Sustainable Material, Lowers Costs</strong></td>
</tr>
<tr>
<td><strong>Polyurethane Foam</strong></td>
<td><strong>Sustainable Material, Lowers Costs</strong></td>
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Environmental Benefits of Lehigh’s Sustainable Micronized Rubber Powder

Lehigh’s Micronized Rubber Powder eliminates waste from going to landfills: end-of-life tires and other post-industrial rubber.

Every kg of Lehigh’s Micronized Rubber Powder saves 6.70 liters of oil*  
*Same amount of oil needed to fuel a passenger car for 18 km

Every pound of Lehigh’s Micronized Rubber Powder saves approximately 10kWh of energy*  
*Same amount of energy needed to run a medium window-unit AC for 10 hours

Micronized Rubber Powder releases nearly half the CO₂ required to manufacture synthetic rubber.

*vs. synthetic rubber

Claims developed by Sustainable Design and Manufacturing Program at Georgia Institute of Technology
Global Tire Companies Using MRP –

*Increasing their Efforts to Produce a Cost Effective, Sustainable Tire*

“We have a number of efforts going on in that [sustainability] direction; one of them is to use recycled rubber.” “We are already using recycled rubber.” - Goodyear Chief Technical Officer, Jean-Claude Kihn

Source: TireBusiness.com

ECOPIA EP422 (ECO)

**Recycled Ground Rubber**

Made from ground-up post consumer tires, it contributes to 5% of the tread compound

Source: Bridgestonetire.com

Percent usage of recycled rubber doubled between 2008 – 2010

Source: Yokohama 2011 CSR Report
Thank You