DRAGONITE™

Halloysite – The Natural Reinforcement for Elastomeric Systems

Presented by: Dr. Chris DeArmitt – CTO
Silicone Elastomers 2011
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Agenda

- Introduction to fillers
- What is Halloysite?
- Mechanical properties
- Flame retardance
- Sustained release of actives
- Availability and pricing
- Conclusions
Markets Addressed

**Plastics**
- Productivity +20%
- Mechanicals +20%
- Flame retardance

**Environment**
- Oil clean-up
- Soil remediation
- Water purification

**Coatings & Adhesives**
- UV cure speed +20%
- Mechanicals +20%
- Improved adhesion

**Elastomers**
- Reinforcement
- Flame retardance
- Thermal stability
Markets Addressed

- Additive for plastics, elastomers and coatings

- Global fillers for plastics market is > $13 BN (> 9 Million tons)\(^1\)
  - Elastomers 50%
  - Thermoplastics 35%
  - Thermosets 15%

- Global market for fillers in coatings > $1.8 BN\(^2\)

- Global controlled release market > $21 BN (for drugs alone)\(^3\)

- Global soil remediation market $25-30 BN\(^4\)

2. World Inorganic Filler Market, Acmite Market Intelligence 2009
## Reinforcements Market

<table>
<thead>
<tr>
<th>Filler</th>
<th>Millions of Tons</th>
<th>Billions of Euros</th>
</tr>
</thead>
<tbody>
<tr>
<td>Carbon Black</td>
<td>4.5</td>
<td>3.96</td>
</tr>
<tr>
<td>Natural CaCO$_3$</td>
<td>2.3</td>
<td>0.17</td>
</tr>
<tr>
<td>Precipitated CaCO$_3$</td>
<td>0.2</td>
<td>0.12</td>
</tr>
<tr>
<td>Precipitated Silica</td>
<td>0.3</td>
<td>0.3</td>
</tr>
<tr>
<td>Al(OH)$_3$</td>
<td>0.3</td>
<td>0.17</td>
</tr>
<tr>
<td>Talc</td>
<td>0.3</td>
<td>0.14</td>
</tr>
<tr>
<td>Kaolin</td>
<td>0.2</td>
<td>0.03</td>
</tr>
<tr>
<td>Others</td>
<td>0.8</td>
<td>0.12</td>
</tr>
<tr>
<td><strong>Total</strong></td>
<td><strong>8.9</strong></td>
<td><strong>5.01</strong></td>
</tr>
</tbody>
</table>

Source: Rothon Consultants
Halloysite is a natural aluminosilicate clay with a hollow tubular morphology.

- Naturally exfoliated morphology means no need to chemically separate particles and makes for easy dispersion.

- Halloysite nanotubes typically have diameter ~50nm with lengths ranging from 1 to 2 microns giving an aspect ratio of ~20.

- Traditional uses include fine china, fillers in paints and paper, food extenders, catalysts and molecular sieves.
Halloysite Property Overview

- Aluminosilicate mineral: \( \text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot \text{nH}_2\text{O} \)
- Molecular weight: 294.19
- CAS: 1332-58-7
- Density: 2.70 ± 0.03 gcm\(^{-3}\)
- Refractive index at room temperature: 1.534, dried at 100°C 1.548
- Specific heat capacity: 0.92 kJkg\(^{-1}\)K\(^{-1}\)
- Thermal conductivity: 0.092 WK\(^{-1}\)m\(^{-1}\)
- Thermal diffusivity: 5.04 x 10\(^{-4}\) cm\(^2\) sec\(^{-1}\)
- CTE: 10.0 ± 1.5 perpendicular to the layer, 6.0 ± 2.0 parallel
- Colorless and UV transparent
- pH in water 6.4-7.2
- Particle shape: 1-2 microns long, 50nm across, 15nm diameter hole
- Surface area: 65-120 m\(^2\)g\(^{-1}\)
- Dragonite™ purity: 95-100%
High aspect ratio

- Reinforcement of plastics, elastomers, coatings etc.

High surface area

- Catalysts, adsorbents, carrier, elastomers, immobilization, nucleation of crystal growth and foam cell formation

Hollow

- Controlled release, thermal insulation, light-weighting, wicking, membranes, reverse osmosis

Bound water

- Fire retardance, temperature indicator, foaming agent
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Isotropic fillers retain impact but do not reinforce

Reinforcing fillers ruin impact resistance and elongation to break

Halloysite reinforces and retains or improves impact and elongation

This is possible due to shape, surface area and easy dispersibility
Halloysite in EPDM Rubber

Halloysite in EPDM Rubber

### Halloysite in EPDM Rubber

<table>
<thead>
<tr>
<th>Material</th>
<th>M100 Modulus (MPa)</th>
<th>Tensile Yield Strength (MPa)</th>
<th>Elongation to Break (%)</th>
<th>UL 94 Classification</th>
</tr>
</thead>
<tbody>
<tr>
<td>EPDM Unfilled</td>
<td>1.07</td>
<td>1.32</td>
<td>151</td>
<td>Unclassified</td>
</tr>
<tr>
<td>+5 phr Halloysite</td>
<td>1.16</td>
<td>1.94</td>
<td>223</td>
<td>Unclassified</td>
</tr>
<tr>
<td>+10 phr Halloysite</td>
<td>1.21</td>
<td>2.21</td>
<td>241</td>
<td>Unclassified</td>
</tr>
<tr>
<td>+15 phr Halloysite</td>
<td>1.24</td>
<td>2.85</td>
<td>299</td>
<td>Unclassified</td>
</tr>
<tr>
<td>+30 phr Halloysite</td>
<td>1.52</td>
<td>4.19</td>
<td>361</td>
<td>V-2</td>
</tr>
<tr>
<td>+50 phr Halloysite</td>
<td>1.85</td>
<td>5.42</td>
<td>400</td>
<td>V-1</td>
</tr>
<tr>
<td>+70 phr Halloysite</td>
<td>2.22</td>
<td>8.15</td>
<td>506</td>
<td>V-1</td>
</tr>
<tr>
<td>+100 phr Halloysite</td>
<td>2.99</td>
<td>12.86</td>
<td>613</td>
<td>V-0</td>
</tr>
</tbody>
</table>

### Halloysite with Natural Rubber

<table>
<thead>
<tr>
<th>Material</th>
<th>200% Modulus (MPa)</th>
<th>300% Modulus (MPa)</th>
<th>Tensile Strength (MPa)</th>
<th>Elongation to Break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Natural Rubber</td>
<td>0.83</td>
<td>1.12</td>
<td>14</td>
<td>1496</td>
</tr>
<tr>
<td>+10 phr Silica</td>
<td>1.03</td>
<td>1.03</td>
<td>12</td>
<td>996</td>
</tr>
<tr>
<td>+10 phr Halloysite</td>
<td>1.12</td>
<td>1.54</td>
<td>18</td>
<td>1270</td>
</tr>
<tr>
<td>+10 phr Halloysite + silane</td>
<td>1.24</td>
<td>2.18</td>
<td>15</td>
<td>1050</td>
</tr>
</tbody>
</table>

# Halloysite with SBR

<table>
<thead>
<tr>
<th>Material</th>
<th>300% Modulus (MPa)</th>
<th>Tensile Strength (MPa)</th>
<th>Elongation to Break (%)</th>
<th>Permanent Set (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SBR</td>
<td>1.33</td>
<td>2.4</td>
<td>610</td>
<td>4</td>
</tr>
<tr>
<td>+10 phr Halloysite</td>
<td>2.27</td>
<td>3.4</td>
<td>580</td>
<td>6</td>
</tr>
<tr>
<td>+20 phr Halloysite</td>
<td>3.02</td>
<td>4.7</td>
<td>590</td>
<td>8</td>
</tr>
<tr>
<td>+30 phr Halloysite</td>
<td>3.57</td>
<td>6.6</td>
<td>600</td>
<td>16</td>
</tr>
<tr>
<td>+40 phr Halloysite</td>
<td>5.09</td>
<td>8.4</td>
<td>580</td>
<td>20</td>
</tr>
<tr>
<td>+50 phr Halloysite</td>
<td>4.81</td>
<td>9.3</td>
<td>600</td>
<td>30</td>
</tr>
</tbody>
</table>

Morphology, interfacial interaction and properties of styrene-butadiene rubber/modified Halloysite nanotube nanocomposites

1:1 molar mixture of hexamethylenetriamine and resorcinol as coupling agent
### Halloysite with FKM Fluoroelastomer

<table>
<thead>
<tr>
<th>Material</th>
<th>100% Modulus (MPa)</th>
<th>$(E'<em>0 - E'</em>\infty)$ Modulus (MPa)</th>
<th>Tensile Strength (MPa)</th>
<th>Elongation to Break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FKM gum</td>
<td>2.5</td>
<td>7.45</td>
<td>4.5</td>
<td>197</td>
</tr>
<tr>
<td>+5 phr Halloysite</td>
<td>3.0</td>
<td>8.50</td>
<td>6.2</td>
<td>217</td>
</tr>
<tr>
<td>+10 phr Halloysite</td>
<td>3.4</td>
<td>13.91</td>
<td>6.8</td>
<td>205</td>
</tr>
<tr>
<td>+20 phr Halloysite</td>
<td>3.6</td>
<td>15.48</td>
<td>7.3</td>
<td>200</td>
</tr>
<tr>
<td>+30 phr Halloysite</td>
<td>2.6</td>
<td>11.97</td>
<td>5.6</td>
<td>254</td>
</tr>
</tbody>
</table>

Dispersibility of Halloysite and Polarity

- Halloysite has been shown to disperse well in all types of system, from apolar to very polar.
- Wetting through the tubes gives mechanical bonding even in cases where no specific chemical interaction takes place.
- In thermosets, thermoplastics and elastomers, effective reinforcement is reported even without dispersants or coupling agents.
- Dispersants and coupling agents may also be used.

<table>
<thead>
<tr>
<th>Polarity</th>
<th>System</th>
</tr>
</thead>
<tbody>
<tr>
<td>Very low polarity</td>
<td>FKM (~20 mJm⁻²)</td>
</tr>
<tr>
<td>Low polarity</td>
<td>PE, PP (~35 mJm⁻²)</td>
</tr>
<tr>
<td>Medium polarity</td>
<td>PVC, PA12 (~40 mJm⁻²)</td>
</tr>
<tr>
<td>High polarity</td>
<td>PET, PA6 (~45 mJm⁻²)</td>
</tr>
<tr>
<td>Very high polarity</td>
<td>Water (~70 mJm⁻²)</td>
</tr>
</tbody>
</table>
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Dragonite Thermal Stability by TGA

![Graph showing weight loss at different temperatures. At 100°C, there is a 1.0% weight loss. At 400°C, there is a 1.9% weight loss. By 700°C, the weight loss is 12.8%.](image)
## Flame Retardancy:
**Dragonite XR vs. MDH - Magnesium Hydroxide**

<table>
<thead>
<tr>
<th></th>
<th>Control</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Dragonite XR</strong></td>
<td>0</td>
<td>0</td>
<td>20</td>
<td>30</td>
<td>40</td>
<td>60</td>
</tr>
<tr>
<td><strong>Magnesium Hydroxide (ST)</strong></td>
<td>0</td>
<td>60</td>
<td>40</td>
<td>30</td>
<td>20</td>
<td>0</td>
</tr>
<tr>
<td><strong>PP 20 MFI</strong></td>
<td>100</td>
<td>40</td>
<td>40</td>
<td>40</td>
<td>40</td>
<td>40</td>
</tr>
<tr>
<td><strong>Flexural Modulus tangent (Kpsi)</strong></td>
<td>207</td>
<td>432</td>
<td>467</td>
<td>464</td>
<td>521</td>
<td>557</td>
</tr>
<tr>
<td><strong>Flexural Modulus 1% (Kpsi)</strong></td>
<td>212</td>
<td>373</td>
<td>391</td>
<td>392</td>
<td>440</td>
<td>461</td>
</tr>
<tr>
<td><strong>Flexural Strength (psi)</strong></td>
<td>6517</td>
<td>5131</td>
<td>5350</td>
<td>5347</td>
<td>5666</td>
<td>6200</td>
</tr>
<tr>
<td><strong>Tensile Modulus (Kpsi)</strong></td>
<td>150</td>
<td>277</td>
<td>275</td>
<td>285</td>
<td>300</td>
<td>294</td>
</tr>
<tr>
<td><strong>Tensile Strength (psi)</strong></td>
<td>5180</td>
<td>3242</td>
<td>3182</td>
<td>3189</td>
<td>3650</td>
<td>3818</td>
</tr>
<tr>
<td><strong>Notched Izod Impact (ft-lb/in)</strong></td>
<td>0.44</td>
<td>0.44</td>
<td>0.54</td>
<td>0.5</td>
<td>0.45</td>
<td>0.43</td>
</tr>
<tr>
<td><strong>Smoke</strong></td>
<td>low</td>
<td>low</td>
<td>very low</td>
<td>very low</td>
<td>low</td>
<td></td>
</tr>
<tr>
<td><strong>UL 94 Rating</strong></td>
<td>V2</td>
<td>V1</td>
<td>V1</td>
<td>V1</td>
<td>V1</td>
<td>V1</td>
</tr>
</tbody>
</table>
Advantages of Dragonite-XR™
Reduced Flammability

Heat release rate of neat PP and PP/Halloysite composites

Source: 2010 Society of Chemical Industry: Du, Guo, Jia
PET FR Development

- Reinforcing, halogen free flame retardant
- Good mechanicals in combination with glass fiber
- High water release temperature >400°C means Dragonite™ is ideally suited to polymers processed at high temperature
- Char strength boosted with Dragonite™ plus glass fiber
- Synergistic fluxing effect under investigation
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Halloysite Tubules

100 nm
Controlled-Release

0.5 nm  7 nm  15 nm

Glucose  Hemoglobin Protein  Dragonite™
180 Da   60 kDa
## Loading the Tubes

<table>
<thead>
<tr>
<th>Property</th>
<th>Density</th>
<th>Weight % Oil</th>
<th>Volume % Oil</th>
<th>Loading %*</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dragonite</td>
<td>2.666</td>
<td>0.0</td>
<td>0.0</td>
<td>0.0</td>
</tr>
<tr>
<td>1 Wintergreen</td>
<td>2.503</td>
<td>5.2</td>
<td>11</td>
<td>58</td>
</tr>
<tr>
<td>2 Wintergreen</td>
<td>2.583</td>
<td>2.7</td>
<td>5.9</td>
<td>31</td>
</tr>
<tr>
<td>3 Wintergreen</td>
<td>2.111</td>
<td>21</td>
<td>37</td>
<td>190</td>
</tr>
<tr>
<td>4 Winter &amp; Cedar</td>
<td>2.479</td>
<td>5</td>
<td>12</td>
<td>63</td>
</tr>
<tr>
<td>5 Tinuvin 292</td>
<td>2.467</td>
<td>5.2</td>
<td>13</td>
<td>68</td>
</tr>
<tr>
<td>6 Tinuvin 292</td>
<td>2.448</td>
<td>5</td>
<td>13</td>
<td>68</td>
</tr>
<tr>
<td>7 Tinuvin 292</td>
<td>2.466</td>
<td>5.2</td>
<td>13</td>
<td>68</td>
</tr>
<tr>
<td>8 Mustard</td>
<td>2.620</td>
<td>1</td>
<td>2.6</td>
<td>14</td>
</tr>
</tbody>
</table>

*Based on Halloysite having 19% Lumen volume  
Cedar Oil 0.95 gcm⁻³ Oil of Wintergreen 1.17 gcm⁻³ Tinuvin 292: 0.99 gcm⁻³  
50:50 Cedar + Wintergreen assumed 1.06 gcm⁻³ Mustard 1.00 gcm⁻³
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...the production capacity for all carbon nanotubes, nanofibers, graphenes, fullerenes and nanodiamonds was 4,065 tons in 2010, and is expected to exceed 12,300 tons in 2015. The actual production was less than 25% of the capacity in 2010 and about 50% of the capacity in 2015. Total production value is estimated at about $435 million in 2010 and is expected reach a value of $1.3 billion in 2015.

- Production capacity of the Dragon Mine exceeds the global capacity of all those materials combined

- Furthermore, Dragonite is safe, natural and is less than 1/100\textsuperscript{th} the cost of even the cheapest carbon nanotubes
Availability and Pricing

- Dragonite™ brand high-purity Halloysite is commercially available from Applied Minerals
- Dragonite™ is shipped directly from the Dragon Mine in Utah, USA
- Masterbatch concentrates are available as well as neat powder
- Supply is plentiful (>30ktons) to support large-scale applications
- Pricing is in the $1-3 / lb range
- Samples are available to interested parties
- Technical support is also available
Conclusions

- Nanotubular materials have long held great promise
- High cost, lack of availability and other factors have slowed progress until now
- Dragonite™ is 100% natural, safe, cost-effective and abundant enough to support large-scale commercial applications
- Due to high aspect ratio, surface area and easy dispersibility, Halloysite provides effective reinforcement to plastics and elastomers
- Water release at high temperature give a halogen-free FR alternative for high temperature and engineering polymers
- The hollow tubes provide a controlled release effect
Thank You For Your Time

Q&A
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