Adhesive & Sealants

ENGINEERED CELLULOSE FIBERS

Lightweight, High Bulk Reinforcing Fiber for the Chemical Processing Industries.
Properly formulated Interfibe cellulose fibers are one of the most cost effective and unique fillers and thixotropes available to the Adhesive and Sealant industry.
Gel-Cel W90 in epoxy adhesives

Dr. Dave Dunn, March 2006
Dr. Dave Dunn

- Former VP & Director of R&D and New Business Development, Loctite Corporation
- Managed New Business Development and R&D for all Loctite’s automotive and consumer business in the US
- Member of the Editorial Advisory Board of Adhesives and Sealants Industry Magazine and writes monthly Q&A column entitled “Ask Dr. Dave”
- Recently acted as historical consultant and on-screen presenter for the History Channel TV program “Modern Marvels – Glue”
Epoxy adhesives

Epoxy adhesives are widely used in industrial, automotive, construction and consumer markets.

Consumption of two component epoxies in the US is over 30 million lbs
Components of Epoxy adhesive

- Epoxy resin for basic properties
- Hardener for specific properties
- Diluents and Additives for fine-tuning
- MIX

Mix 2 components
In practice a typical two-component epoxy adhesive often comprises:

<table>
<thead>
<tr>
<th>Components of Epoxy adhesive</th>
<th>Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy resin</td>
<td>Epon 828, DER 331</td>
</tr>
<tr>
<td>Hardener</td>
<td>DEH 20, Capcure 3-800</td>
</tr>
<tr>
<td>Versamids, GPM-800</td>
<td></td>
</tr>
<tr>
<td>Fillers</td>
<td>Talc, Ca carbonate, barytes</td>
</tr>
<tr>
<td>Accelerators</td>
<td>Versamines</td>
</tr>
<tr>
<td>Reactive diluent</td>
<td>Glycidyl ethers</td>
</tr>
<tr>
<td>Toughening agents</td>
<td>CTBN rubber</td>
</tr>
<tr>
<td>Thixotropes</td>
<td>Usually fumed silica</td>
</tr>
</tbody>
</table>
Examples – epoxy adhesives
# Five Minute Consumer adhesive

<table>
<thead>
<tr>
<th>Resin</th>
<th>Percent</th>
<th>Mixed 1:1 with</th>
<th>Hardener</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>DER 331 epoxy</td>
<td>78</td>
<td></td>
<td>Polymercaptan</td>
<td>90</td>
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<tr>
<td>DER 732 epoxy</td>
<td>20</td>
<td></td>
<td>Ancamine K-64</td>
<td>8</td>
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<tr>
<td>Fumed silica</td>
<td>2</td>
<td></td>
<td>Fumed silica</td>
<td>2</td>
</tr>
</tbody>
</table>
# Five Minute Metallic Paste

<table>
<thead>
<tr>
<th>Resin</th>
<th>Percent</th>
<th>Mixed 1:1 with</th>
<th>Hardener</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>DER 331 epoxy</td>
<td>45</td>
<td></td>
<td>Polymercaptan</td>
<td>35</td>
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<tr>
<td>Barytes</td>
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<td></td>
<td>DEH-20</td>
<td>8</td>
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<tr>
<td>Ca carbonate</td>
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<td></td>
<td>Weston PTP</td>
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<tr>
<td>Iron oxide</td>
<td>14</td>
<td></td>
<td>Barytes</td>
<td>10</td>
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<tr>
<td>Fumed silica</td>
<td>1</td>
<td></td>
<td>Kaolin</td>
<td>6</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Ca carbonate</td>
<td>35</td>
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<tr>
<td></td>
<td></td>
<td></td>
<td>TiO₂</td>
<td>3</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Fumed silica</td>
<td>1</td>
</tr>
</tbody>
</table>
Thixotropes for epoxies

Fumed silicas:

- **Hydrophilic**
  - Cabosil M5
  - Aerosil 200

- **Hydrophobic**
  - Cabosil TS-720
  - Aerosil 202

Thixatrol – castor oil derivative - requires heat and shear for activation.

Organo clays – require polar additive. Sometimes cause crystallization

Kevlar pulp – very expensive

Interfibe cellulose fibers
Thixotropes for epoxies

The low cost hydrophilic fumed silicas like Cabosil M5 and Aerosil 200 are rarely used with new epoxy adhesive formulations these days because they tend to lose viscosity on aging and most people are using expensive treated hydrophobic silicas like Aerosil 202 or Cabosil TS-720.
Comments from manufacturers

Why do you use thixotropes in your adhesives?

• “Need predictable stable viscosity”
• “Need sag resistance in some industrial products”
• “We want anti-settling in filled products”
• “We need to get the resin and hardener to look and feel the same to get the best mixing. Also need to have good thixotropy to flow well onto the surfaces but then thicken up so we don’t get squeeze-out from the bond”
Comments from manufacturers

How satisfied are you with the performance of your current fumed silica?

Very satisfied 14%
Somewhat satisfied 86%
Unhappy 0
Comments from manufacturers

Are there any negatives associated with using your current fumed silica?

Handleability: Dust is major problem – all respondents say this

Cost: Pay $2-3/lb for hydrophilic, $5-6/lb for hydrophobic grades
Comments from manufacturers

Other comments:

“Viscosity sometimes increases over time”

“M5 is used in old products where we have no problems. In products or products where there are viscosity problems we use TS720 and also for new products.”

“M5 doesn’t maintain viscosity on aging. If you over-shear the fumed silicas into epoxies, it takes a lot of time for viscosity to stabilize (ie. thicken) so there is a temptation to correct low viscosity batches by adding more fumed silica but this causes high viscosity problems in the future on shelf aging”
Comments from manufacturers

Other comments:

“Fumed silica not nearly as good as asbestos but not as dangerous either!”
Comparison of W90 and Cabosil TS-720 in epoxies

Two systems studied:

1. In base epoxy resin Epon 828

1. In filled Epon 828 – 30% calcium carbonate
Unfilled systems in Epon 828

Thixotropes Cabosil TS-720 and Interfibe™ W90 were added to base resin and mixed with high shear for 15 minutes
Initial viscosity in Epon 828

- **W90**
  - Loading %: 1
  - Viscosity cps: 56000
  - Viscosity cps: 84000
  - Viscosity cps: 124000

- **TS-720**
  - Loading %: 1
  - Viscosity cps: 20000
  - Viscosity cps: 33000
  - Viscosity cps: 57000
Cabosil TS720 room temp in Epon 828

4 WEEKS AGING
Filled systems in Epon 828

Thixotropes Cabosil TS-720 and Interfibe™ W90 were added to Epon 828 and mixed with high shear for 20 mins. Then Omyacarb UF added and mixed for a further 40 mins.)
Initial viscosity in filled Epon 828

Viscosity cps

Thixotrope Loading %

0% 0.5% 1% 2% 3%

TS720

W90

interfibe™
Cabosil TS720 in filled Epon 828

4 WEEKS AGING

Filler only

Initial

After 4 weeks

Viscosity cps

0

2% 1% 0.5%

0 5000000 1000000 1500000 2000000 2500000 3000000

interfibe™
Cabosil TS720 in filled Epon 828

Viscosity cps

Initial | After 4 weeks | After 19 weeks

Filler only

2%
1%
0.5%

19 WEEKS AGING
Cabosil TS720 in filled Epon 828

19 WEEKS AGING

Viscosity cps

0 2000000 4000000 6000000 8000000

Initial After 4 weeks After 19 weeks

0.5% 1% Filler only
Interfibe W90 in filled Epon 828

4 WEEKS AGING
Interfibe W90 in filled Epon 828

19 WEEKS AGING

Viscosity cps

Filler only

Initial | After 4 weeks | After 19 weeks

3% | 2% | 1% | 0.5%

0 | 500000 | 1000000 | 1500000 | 2000000 | 2500000 | 3000000
Conclusions

- Cabosil TS-720 is a widely used, but not perfect thixotrope. In unfilled epoxy resin, viscosity decreases with time.

- Interfibe™ W90 is much more effective than hydrophobic fumed silica at thickening unfilled epoxy resins.
Conclusions

• In filled systems, Cabosil TS-720 is an effective thickener but viscosity becomes very sensitive to loading at loadings over 1%
• Interfibe™ W90 is not as efficient in thickening filled systems but is much more controllable than fumed silica at loadings over 1%
• Both thixotropes show increases in viscosity on aging
• Interfibe™ W90 is much easier to handle than fumed silica – no dust and incorporates very easily