Use of Fibrillated Fibers in Coatings Applications

Western Coatings Symposium
October 2015
Fibrillated Fibers

- Highly structured
- Created by refining or design
- Higher surface area
Process for Production of Fibrillated HDPE

Process patent by Crown Zellerbach – 1970s

HDPE $\xrightarrow{\text{Hexane}}$ HDPE solution in hexane

+ PVOH $\xrightarrow{}$ emulsified HDPE/hexane PVOH

Pressure through die $\xrightarrow{\text{vacuum}}$ base fiber produced

Refined to cut length $\xrightarrow{}$ Finished product
Synthetic Wood Pulp
### Grades Available

<table>
<thead>
<tr>
<th></th>
<th>ESS5F</th>
<th>ESS2F</th>
<th>E380F</th>
<th>N/A</th>
<th>E780F</th>
<th>E990F</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Standard Untreated Grades</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Treated Grades for Improved Dispersion</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Average Fiber Length</strong></td>
<td>~0.1</td>
<td>~0.6</td>
<td>~0.7</td>
<td>~0.9</td>
<td>~1.6</td>
<td>~2.0</td>
</tr>
<tr>
<td><strong>Expressed in millimeters (mm)</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Fiber Diameter</strong></td>
<td>5</td>
<td>5</td>
<td>15</td>
<td>15</td>
<td>25</td>
<td>20</td>
</tr>
<tr>
<td><strong>Expressed in microns (µ)</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Surface Area (m²/gm)</strong></td>
<td>12</td>
<td>12</td>
<td>8</td>
<td>8</td>
<td>8</td>
<td>8</td>
</tr>
<tr>
<td><strong>Measured by gas absorption</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Bulking Value</strong></td>
<td>Metric</td>
<td>1.04<del>1.10 litres per kg / .91</del>.96 kgs per litre</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Standard</td>
<td></td>
<td>0.1250<del>0.1319 gallons per lb / 7.580</del>7.997 lbs per gallon</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Specific Gravity</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>0.96 g/cm³</td>
<td></td>
</tr>
<tr>
<td><strong>Melting Point</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>275°F / 135°C</td>
<td></td>
</tr>
<tr>
<td><strong>Moisture Content</strong></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Less than 2.0%</td>
<td></td>
</tr>
</tbody>
</table>
Benefits in Many Applications

• Proven commercial replacement of mineral fibers in various applications
  – Improved particle suspension
  – Crack resistance - ductility
  – Contributes to water resistance
  – Imparts Fine Texture (shorter finer grades) to Medium / Heavy Build (coarser, longer fibers)
  – Viscosity control
Applications

- Roof Coatings
- Block Filler
- Textured Coatings
- Cement Coatings
- Putties

- Mastics
- Industrial Coatings
- Caulks
- Sealants
- Adhesives
Where do fibers work?

• **Water based systems**
  – Latex emulsion
  – Water reducible alkyds
  – Waterborne PU
  – Waterborne silicones

• **Solvent based systems**
  – Alkyds
  – PU
  – Epoxy
  – Silicones
Other Things to Consider

• High gloss
  – If fiber is thicker than the film, it will dull the finish

• Clear Finishes
  – May cause haze

• Water sensitive applications
  – Fiber has <2% moisture
  – Need to scavenge moisture in water sensitive applications
An Effective Suspending Agent for Pigments

Dense Pigments Sink

Short Stuff Lighter Than Water
Suspending Pigments

Pigments and Short Stuff
Entangle – Prevents Settling
An example of how fibrillated fibers will suspend particulate matter …
Examples of how fibrillated fibers will suspend particulate matter in **water**

play sand with 1.35% Medium Length Fiber in water

**TABLE 1: Play Sand Particle Size Distribution**

<table>
<thead>
<tr>
<th>Size, $S$ (µm)</th>
<th>% (w/w)</th>
</tr>
</thead>
<tbody>
<tr>
<td>$S \geq 300$</td>
<td>3.3</td>
</tr>
<tr>
<td>$212 \leq S &lt; 300$</td>
<td>92.7</td>
</tr>
<tr>
<td>$S &lt; 212$</td>
<td>4.0</td>
</tr>
</tbody>
</table>
Examples of how fibrillated fibers will suspend particulate matter in **water**

play sand with 1.35% Medium Length Fiber in water

**TABLE 2: Aqueous Short Stuff and Sand Formulation**

<table>
<thead>
<tr>
<th>Ingredient</th>
<th>Weight (g)</th>
<th>Dry Weight Fraction (%)</th>
<th>Wet Weight Fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>E380F Medium Length</td>
<td>1.9</td>
<td>2.75</td>
<td>1.35</td>
</tr>
<tr>
<td>Play Sand</td>
<td>67.2</td>
<td>97.25</td>
<td>47.80</td>
</tr>
<tr>
<td>Dry Ingredient, Subtotal</td>
<td>69.1</td>
<td>100.0</td>
<td>-</td>
</tr>
<tr>
<td>Water</td>
<td>71.5</td>
<td>-</td>
<td>50.85</td>
</tr>
<tr>
<td>Wet &amp; Dry Ingredient, Total</td>
<td>140.6</td>
<td>-</td>
<td>100.0</td>
</tr>
</tbody>
</table>

NOTE: play sand sp. gr. ≈ 2.6
Examples of how fibrillated fibers will suspend particulate matter in **water**

play sand with 1.35% Medium Length Fiber in water

Step 1: Add Fiber to tap water
Examples of how fibrillated fibers will suspend particulate matter in water

play sand with 1.35% Medium Length Fiber in water

Step 2: Shake vigorously for 1 minute
Examples of how fibrillated fibers will suspend particulate matter in water

play sand with 1.35% Medium Length Fiber in water

Step 3: Pour sand into the solution
Examples of how fibrillated fibers will suspend particulate matter in **water**

play sand with 1.35% Medium Length Fiber in water

Step 4: Shake vigorously for 1 minute

Note: This colloidal suspension is stable – no hard settling!
Two Studies on Fibrillated Fiber in Coatings

- Elastomeric Roof Paint Testing
  - Resin – Based on 100% Acrylic Resin

Test Thickeners
- HEC
- ESS5F
- E380F
- Adhesion to galvanized steel ASTM D4541
- Viscosity ASTM D562
- Sag Resistance – ASTM D4400
- Water vapor permeability ASTM D1653
- Tensile strength, psi ASTM D2370
- Elongation % ASTM D2370
- Dirt Pickup Lab Method – Lab Method
- Flexibility Conical mandrel ASTM D522
- Water & Alkali Absorption – Lab Method
- Permeability – ASTM D1653
- Impact Resistance ASTM D2794

- Interior / Exterior Flat Paint
  - Resin – Based on 100% Acrylic Resin

Test Thickeners
- HEC
- ESS5F
- E380F
- Viscosity, KU – ASTM D562
- Viscosity, ICI – ASTM D4287
- Viscosity, Brookfield – ASTM D2196
- Contrast Ratio, 3 Mils – ASTM D2805
- CIELAB Values (Untinted) – ASTM D2244
- Gloss (Sheen) – ASTM D523
- Flow & Leveling – ASTM D4062
- Sag Resistance – ASTM D4400
- Scrub Resistance – ASTM D2486
- Burnish Resistance – ASTM D6736
### Application Data

**Exterior Flat Paint**

**100% Acrylic Binder**

<table>
<thead>
<tr>
<th></th>
<th>0.22%</th>
<th>1%</th>
<th>1%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thickener</td>
<td>Cellulosic Thickener</td>
<td>ESS5F</td>
<td>E380F</td>
</tr>
<tr>
<td>Co-Thickener</td>
<td>Urethane Co-Thickener</td>
<td>Urethane Co-Thickener</td>
<td>Urethane Co-Thickener</td>
</tr>
<tr>
<td></td>
<td>1.70%</td>
<td>1.70%</td>
<td>1.70%</td>
</tr>
</tbody>
</table>

Brookfield #4 - 2.5 rpm - Poise:

<table>
<thead>
<tr>
<th></th>
<th>378</th>
<th>58</th>
<th>240</th>
</tr>
</thead>
<tbody>
<tr>
<td>KU</td>
<td>102</td>
<td>94</td>
<td>105</td>
</tr>
<tr>
<td>ICI</td>
<td>1.4</td>
<td>1.3</td>
<td>1.7</td>
</tr>
</tbody>
</table>
**Application Data**

**Exterior Flat Paint**

**100% Acrylic Binder**

<table>
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<td>Urethane Co-Thickener</td>
<td>Urethane Co-Thickener</td>
</tr>
<tr>
<td></td>
<td>1.70%</td>
<td>1.70%</td>
<td>1.70%</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Property</th>
<th>std.</th>
<th>=</th>
<th>=</th>
</tr>
</thead>
<tbody>
<tr>
<td>Opacity</td>
<td>=</td>
<td>=</td>
<td>=</td>
</tr>
<tr>
<td>Color, YI</td>
<td>=</td>
<td>=</td>
<td>=</td>
</tr>
<tr>
<td>Gloss</td>
<td>=</td>
<td>=</td>
<td>=</td>
</tr>
<tr>
<td>Sag mils</td>
<td>&gt;24</td>
<td>12</td>
<td>&gt;24</td>
</tr>
<tr>
<td>Flow &amp; Leveling</td>
<td>6</td>
<td>9</td>
<td>8</td>
</tr>
<tr>
<td>Burnish</td>
<td>160%</td>
<td>140%</td>
<td>127%</td>
</tr>
<tr>
<td>Stain Resist</td>
<td>std.</td>
<td>Slight +</td>
<td>+</td>
</tr>
</tbody>
</table>
# Application Data

## Elastomeric Roof Coating

### 100% Acrylic Elastomeric

<table>
<thead>
<tr>
<th></th>
<th>0.34% Thickener</th>
<th>1% Cellulosic Thickener</th>
<th>0.5% ESS5F</th>
<th>1.00% E380F</th>
</tr>
</thead>
<tbody>
<tr>
<td>Viscosity KU</td>
<td>103</td>
<td>102</td>
<td>108</td>
<td>124</td>
</tr>
<tr>
<td>Sag (mils)</td>
<td>45</td>
<td>25</td>
<td>30</td>
<td>&gt;60</td>
</tr>
<tr>
<td>Adhesion psi</td>
<td>300</td>
<td>325</td>
<td>325</td>
<td>300</td>
</tr>
<tr>
<td>Flexibility</td>
<td>pass 1/8”</td>
<td>pass 1/8”</td>
<td>pass 1/8”</td>
<td>pass 1/8”</td>
</tr>
<tr>
<td>Water Abs</td>
<td>14%</td>
<td>14%</td>
<td>12%</td>
<td>10%</td>
</tr>
<tr>
<td>Alkali Abs</td>
<td>14%</td>
<td>14%</td>
<td>13%</td>
<td>10%</td>
</tr>
</tbody>
</table>

**Thickener**
- Cellulosic Thickener
- ESS5F
- E380F

**Co-Thickener**
- Urethane Co-Thickener

**Minifibers, Inc.**
Study on Fibrillated Fibers for water-borne coatings application

Water resistance test

Immersion in water of 25 degrees Celsius for ten days

ESS-50F shows excellent resistance to water damage
Study on Fibrillated Fibers for water-borne coatings application

Bending Test

1. Preparation of coated sample

   Water-borne coating with additive
   Steel plate
   Drying at 50deg-C

2. Bending test using mandrel

   Steel plate with coating
   Diameter of Mandrel

3. Evaluation

   To check the surface situation of the coating changing mandrel with shorter diameter

   Diameter: 32mm -> 10mm -> 6mm -> 2mm

   Coatings without any crack with shorter diameter of mandrel are superior.
Study on Fibrillated Fibers for water-borne coatings application

Bending Test

⇒ Fibers provide excellent flexibility to the coating

Additive : HEC (0.1%)  
Diameter of Mandrel : 10 mm

Additive : HEC (0.1%) & FDSS-50 (1%)  
Diameter of Mandrel : 6 mm
Fibrillated Fibers
an effective
Rheology Modifier

Effectively Replaces Fumed Silica
In Epoxies
Effect of Agitation on Viscosity Versus Shear Rate
Short Stuff, Fumed Silica and Epoxy

Shear Rate $S^{-1}$
Viscosity $CP$

Pure Epoxy
Epoxy with 0.4% Short Stuff
Epoxy with 5% Fumed Silica

Gravitational SAG  Shear Rate $S^{-1}$  Agitated Tank
Hypothesis

Since fibrillated fibers give viscosity by physical means, might they be less prone to viscosity drop when used in deeply tinted paints?
Study

• Compare a range of polyurethane thickeners to the shortest fiber in a 100% acrylic exterior paint
  – Older PU and newest to market PU
  – All supplemented by a new range Newtonian PU for ICI gain
• Study viscosity loss on tinting
  – Brookfield, KU, and ICI
• Study flow and leveling and sag resistance
• Study color acceptance and color float
Viscosity Response

**Brookfield Viscosity**

- Initial Brookfield
- Tinted Brookfield

**KU Viscosity**

- Initial KU
- Tinted KU

**ICI Viscosity**

- Initial ICI
- Tinted ICI
Comparative Changes
Film Quality

Flow and Leveling

Sag (mils)
## Color Acceptance and Color Float

<table>
<thead>
<tr>
<th></th>
<th>Older PU</th>
<th>New PU1</th>
<th>New PU2</th>
<th>New PU3</th>
<th>Short Fiber</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Color Acceptance</strong></td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
<td>Pass</td>
</tr>
<tr>
<td><strong>Color Float</strong></td>
<td>Severe</td>
<td>Minimal</td>
<td>Minimal</td>
<td>Moderate</td>
<td>Severe</td>
</tr>
</tbody>
</table>
Conclusions

• It is a little early, but……
  – Fiber looks very good for
    • Viscosity maintenance
    • Good flow and leveling
    • Much better sag resistance

• Work remains to refine the application regarding color float
Processing Tips Fibrillated Fibers

• ADDED TO THE PRODUCT FIRST!
• Most applications use less than 2% loading by weight for desired effect.
• Fibers are robust and do not degrade under high speed dispersion shear
• Fine particle grades are used when a more smooth surface is desired.
• Medium to course grades are used when more of a textured surface is desired. Usually brushed and/or roll applied, but can be sprayed through nozzles if no screen is used.
Treated or Untreated??

- Utilize treated grades in water based systems
- Use untreated grades in solvent based systems
Summary

Synthetic product
No binder absorption
Does not absorb moisture (does not swell)
Does not absorb solvents
Not susceptible to mildew
Wets out clear / No color interference
Give excellent water resistance
Gives excellent film ductility
Contributes to good rheology control
No health hazards
Can be used in textured coating applications
Thank You!

www.minifibers.com