Fly Ash Derived Ceramic-Polyurethane Foam Composite Insulation

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Polyurethane Foam Insulation

- One of the best insulating materials on the market
- For rigid foam panels, different thicknesses are available to suit different applications
- Spray foam polyurethane provides both insulation and air sealing, as it expands to fill gaps and cracks
- Easy to install

<table>
<thead>
<tr>
<th>Insulating Material</th>
<th>R-value/inch (Market Share)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cellulose</td>
<td>3.7 (14.3%)</td>
</tr>
<tr>
<td>Fiberglass</td>
<td>3.0 (52.1%)</td>
</tr>
<tr>
<td>Mineral Wool</td>
<td>3.0 (6.8%)</td>
</tr>
<tr>
<td>Expanded Polystyrene</td>
<td>4.0 (10.5%)</td>
</tr>
<tr>
<td>Extruded Polystyrene</td>
<td>5.0 (1.2%)</td>
</tr>
<tr>
<td>Rigid Polyisocyanurate</td>
<td>6.0 (11.7%)</td>
</tr>
<tr>
<td>Polyurethane Foam (Closed Cell)</td>
<td>7.0 (3.4%)</td>
</tr>
</tbody>
</table>
Polyurethane Foam Insulation

• Has a high oxygen index
• Manufactured from petroleum derivatives
• Untreated and exposed to high temps or flame, the foams will burn vigorously and spread
• Burning characteristics require manufacture with fire retardant for optimally safe application
Polyurethane Foam Insulation Fire Retardants

• Halogenated fire retardants form a slow burning char on the foam

• No longer considered desirable due to environmental considerations –
  • Possible leaching into the environment
  • Accumulating in birds, mammals, and fish
  • Accumulating in soil and sediment
Alternatives to Chemical Fire Retardants

- Addition of ultrafine fly ash-derived ceramic particles to polyurethane foam allows for multiple benefits, including:
  - Lower flammability of the PU foam
  - Higher strength and toughness
  - Reduced overall cost
- Fine particles also form an inorganic crust on the foam

SEM image of ultrafine fly ash-derived ceramic particles recovered from coal combustion ash.
-Materials/Techniques Used

• Ultrafine ceramics (D$_{50}$ = 5 microns) recycled from discarded coal combustion ash from:
  • Kingston Fossil Plant (Tennessee Valley Authority; Kingston, Tennessee)
  • Kyger Creek Power Plant (Ohio Valley Electric Corporation; Cheshire, Ohio)
  • Ghent Power Plant (Kentucky Utilities; Carroll County, Kentucky)

• To fabricate composite foams, fly-ash derived ceramics were blended into commercially available polyurethane foam resins:
  • 3-lb density; U.S. Composites, West Palm Beach, Florida
  • Low density, low strength, high insulating capacity
-ASTM C303 – Standard Test Method for Dimensions and Density of Preformed Block and Board-Type Thermal Insulation

- Density of 5 blocks were measured at the same time:
  - Width and length of each block were measured to nearest mm in 2+ locations
  - Thickness of each block measured 4+ times
  - Blocks were weighed
  - Densities were calculated

\[
\rho = \frac{m}{v}
\]
ASTM C303 – Standard Test Method for Dimensions and Density of Preformed Block and Board-Type Thermal Insulation

- Density calculation were also used to compare composite foams:
  “Bench-top Mold vs. “Pilot-scale Mold”

- Results:
  - ADC increased weight of PU foams when compared to Control foam
  - Regardless of source, iron, or coupling agent, ADC added similar weight
  - Exception is Kingston with Iron for “pilot-scale”, due to large amount of iron-rich material

- Compressive Strength was tested:
  - Test specimens (2 in. x 2 in. x 2 in.) were cut
  - Samples tested in direction of expansion (longitudinal) and perpendicular to expansion (traverse) – anisotropy

• Results:
  • Addition of 30% (by weight) ADC improved strength of PU foam
  • Improvement was dependent on ash source
  • Ghent and Kingston are predominately Class F (high aluminum/low calcium)
  • Kyger Creek closely resembles Class C (high calcium)

• Results:
  • Foams with ceramic products with iron-rich particles were also produced and tested
  • Iron-rich particles did not adversely affect PU foams, but strengthened them more than ceramics alone

• Silane (SiH₄) coupling agents are used to promote adhesion between additives and matrix materials during composite production.

• Silane, in solution form, was blended with the ADC.
  • Silanol (Si-O-H) was fixed onto the surface of ADC via hydrogen bonding with surface hydroxyl groups (-OH) on the ceramics.

• Results:
  • Addition of silane strengthened the composite foams beyond the benefit of adding untreated ceramics
  • It was anticipated that ADC without iron particle would perform better

• Water absorption is an important aspect of insulation, especially if it’s installed below ground level, or can come into contact with moisture

• Water absorption can lead to:
  • Mildew
  • Mold growth
  • Rot
  • Diminished performance

• Closed cell foams (PU) usually provide a vapor barrier, but important to test the addition of ADC

• Specimens were cut (3 in. x 3 in. x 0.5 in.) and weighed

• 5 specimens per formulation were completely immersed in water (73 ± 5°F)

• After 24 hours:
  • Specimens removed from water
  • Shaken to remove surface water
  • Weighed
  • Percent weight increase calculated

- Decrease in amount of water absorbed during 24 hour immersion was generally observed
- Iron-rich materials were shown to perform worse
- Silane detracted slightly overall
- With the exception of Kingston with Fe, ADC’s performed as good or better than Control
FMVSS 302 – Flammability of Materials Used in the Interior of Motor Vehicle Occupant Compartments Test

• Primary Objective – determine the feasibility of use ADC’s as a flame retardant in PU foam for insulation

• Rectangular panels (4 in. x 12 in. x 1 in.) were cut from larger composite foam panels (0%, 15%, 30%, and 45% (by weight) ADC)

• Govmark test:
  • Mounted specimens vertically
  • 38 mm flame from a Bunsen burner was placed 19 mm below center of bottom edge of specimen for 15 seconds
  • Distance the flame traveled and burn time were recorded
  • Burn rate was calculated if flames traveled at least 38 mm from edge
- FMVSS 302 – Flammability of Materials Used in the Interior of Motor Vehicle Occupant Compartments Test

<table>
<thead>
<tr>
<th>Specimen #</th>
<th>Time to Reach 38 mm (sec)</th>
<th>Time Beyond 38 mm (sec)</th>
<th>Time Beyond 38 mm (SPF)</th>
<th>Burn Rate (mm/minute)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>85</td>
<td>158</td>
<td>194</td>
<td>100</td>
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<tr>
<td>2</td>
<td>18</td>
<td>357</td>
<td>184</td>
<td>70</td>
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<tr>
<td>3</td>
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<td>322</td>
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<td>4</td>
<td>4</td>
<td>176</td>
<td>294</td>
<td>87</td>
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<td>7</td>
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<tr>
<td>8</td>
<td>54</td>
<td>384</td>
<td>294</td>
<td>70</td>
</tr>
</tbody>
</table>

**Control (0wt% ADC):**

- All 8 specimens ignited and flames traveled beyond the 38mm threshold
- On average, controls burned at a rate of 79.8 mm/min
- All 8 = B grade
  - Specimen ignites, burning progress more than 51 mm beyond the 38mm timing start line
### FMVSS 302 – Flammability of Materials Used in the Interior of Motor Vehicle Occupant Compartments Test

#### 15wt% ADC:
- All 8 specimens ignited
- Only 6 remained lit past time line
- Burn rate was calculated for only 5 of 8 specimens
- Average burn rate was 57.6 mm/min
- 5 specimens = B grade
- 2 specimens = SE grade
  - Self-extinguishing – specimen ignites, but does not burn to the timing zone
- 1 specimen = SE/NBR grade
  - Specimen ignites, burning progresses to timing line, extinguishes within 51 mm beyond start line

<table>
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<tr>
<th>Specimen</th>
<th>150 mm Time to Heat Release (s)</th>
<th>T5 Burning Time (mm)</th>
<th>Burn Rate (mm/min)</th>
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<td>158</td>
<td>224</td>
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<td>68</td>
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</tbody>
</table>
- FMVSS 302 – Flammability of Materials Used in the Interior of Motor Vehicle Occupant Compartments Test

- 30wt% and 45wt% ADC:
  - All specimens = SE grade
    - If the specimen ignited at all, the flames extinguished before reaching the 38 mm line

- Results suggest ADC’s have potential as flame retardants, or at least have the potential to be able to slow the burn rate
NFPA 286 – Standard Methods of Fire Tests for Evaluation Contribution of Wall and Ceiling Interior Finish to Room Fire Growth Test

• Known as the Full Room Test
• Used to determine the contribution of interior finish materials to room fire growth during specific fire exposure conditions
• Measures:
  • Extent of fire growth
  • Rate of heat release
  • Total heat release
  • Time to flashover
  • Time to flame extension
  • Total heat flux incident to the floor
  • Upper level gas temperature
  • Smoke obscuration
  • Production of carbon monoxide
  • Emissions of other combustion gases
- NFPA 286 – Standard Methods of Fire Tests for Evaluation Contribution of Wall and Ceiling Interior Finish to Room Fire Growth Test

• Full Room test has two phases:
  • First – preliminary test of two panels put together to form a “room corner”
  • Second – consists of six full-sized panels forming two walls and a ceiling

• Preliminary “room corner” test is necessary to ensure that a smaller amount of material can withstand the intensity of the flame without:
  • Dripping flaming material
  • Damaging the test apparatus
  • Creating a fire that risks spreading beyond containment
-NFPA 286 – Standard Methods of Fire Tests for Evaluation Contribution of Wall and Ceiling Interior Finish to Room Fire Growth Test

- Trimmed panels were mounted vertically at juncture of two 8 ft. x 12 ft. test walls
- Panels received full test exposure of 40 kW flame for 5 minutes, followed by 160 kW flame for 10 minutes
- Panels are observed during the test – if flames spread to edges of panels or ceiling, or if material dripped flames, test is stopped
-NFPA 286 – Standard Methods of Fire Tests for Evaluation Contribution of Wall and Ceiling Interior Finish to Room Fire Growth Test

• First attempt:
  • Two panels were sent bare, leaving PU foam fully exposed to the flame
  • These panels survived for 1 minute, 20 seconds of the lesser flame intensity (40 kW) before the flames reached the ceiling of the testing apparatus
-NFPA 286 – Standard Methods of Fire Tests for Evaluation Contribution of Wall and Ceiling Interior Finish to Room Fire Growth Test

• It was determined that PU foams should be tested with a gypsum wallboard covering

• Test requires materials of interest be installed in test apparatus as they would be in building application

• Second attempt:
  • 45wt% composite foam blended and spread on back of a 4 ft. x 8 ft. panel of ½ in. gypsum wallboard
NFPA 286 – Standard Methods of Fire Tests for Evaluation Contribution of Wall and Ceiling Interior Finish to Room Fire Growth Test

• Results:
  • Improved from first attempt
  • Panels faced with gypsum wallboard survived first 5 minutes of the 40 kW flame exposure
  • Flame intensity was increased to 160 kW, panels survived an additional 4 minutes, 58 seconds
  • Although panels did not receive a passing grade, the results were very promising

• Primary objective of test was to directly measure the thermal conductivity of ADC PU foam composites

• Services of Precision Measurements and Instruments Corporation (PMIC; Corvallis, OR) were used
  • Specialize in measuring:
    • Coefficient of thermal expansion
    • Coefficient of moisture expansion
    • Thermal Conductivity

• Results:

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Nominal Temperature (°C)</th>
<th>Tested Temperature (°C)</th>
<th>Conductivity (W/m-K)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Polyurethane Foam 1A &amp; 1B Tested in Dry Nitrogen Gas</td>
<td>-17.7</td>
<td>-18.0</td>
<td>0.0296</td>
</tr>
<tr>
<td></td>
<td>23.0</td>
<td>23.0</td>
<td>0.0338</td>
</tr>
<tr>
<td></td>
<td>48.9</td>
<td>48.0</td>
<td>0.0487***</td>
</tr>
</tbody>
</table>

- 2 square specimens (6 in. x 6 in. x 1 in.) were cut – 30wt%

- Erratic temperatures were observed while testing at highest temps
- Thermal conductivity values at -18°C and 23°C were typical of closed-cell PU foam materials
- Calculated R-values are good indicator ADC PU foams would compete very well with current insulating materials
-Conclusions

• ADC PU foam have shown a marked increase in efficiency and performance when compared to current PU foam

• ADC’s:
  • Show potential as flame retardant
  • Increase compressive strength
  • Decrease water absorption

• Overall, ADC’s are a very promising material, with great potential for ADC PU foam composites
Thank you!