A Summary of Proposed Changes to AASHTO M 295 Resulting from NCHRP Project 18-13 - Specifications and Protocols for Acceptance Tests of Fly Ash Used in Highway Concrete

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Acknowledgements

NCHRP 18-13 Specifications and Protocols for Acceptance Tests of Fly Ash Used in Highway Concrete

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Disclaimer

The research work presented herein was performed as part of the National Cooperative Highway Research Program Project 18-13 “Specifications and Protocols for Acceptance Tests of Fly Ash Used in Highway Concrete”. This presentation does not necessarily indicate acceptance by the National Academies, the Federal Highway Administration, or by the American Association of State Highway Officials of the findings, conclusions, or recommendations either inferred or specifically expressed herein.

Reference:
Objective - recommend potential improvements to specifications and test protocols to determine the acceptability of fly ash for use in highway concrete
• Characterization Study – evaluate existing specifications and classification methods for CFA
• Strength Test Study – investigate test methods for characterizing the strength activity of CFA
• Carbon Effects on Air Entrainment Study – develop test methods for characterizing the adsorption properties of residual carbon in CFA
• ASR Mitigation Study – examine test methods to evaluate use of CFA to mitigate alkali-silica reaction in concrete
Characterization Study

- Gathered data on 100+ CFA sources
- Surveyed the SHAs to determine common sources used
- Selected 30 for comprehensive analysis
  - 17 Class F, 13 Class C
  - Selected sources from the 30 best suited for the other testing performed
Summary of 30 Sources

- Sum of SiO$_2$, Al$_2$O$_3$, and Fe$_2$O$_3$: 51.8 to 92.7%
- Calcium oxide (CaO): 0.9 to 30.6%
- Na$_2$O$_e$: 0.3 to 7.9%
- LOI: 0.1 to 5.6%
- Fineness: 10 to 24.0%
- Strength Index (7-day test value): 75 to 112%
- Strength Index (28-day test value): 80 to 120%
- Water requirement: 93 to 100%
- Density: 2.1 to 2.8g per cubic-centimeter

Also made blends to achieve higher LOI
Characterization Study

- Characterized 30 sources using ASTM C311 methods
  - All AASHTO M 295 Required and Optional Chemical and Physical Properties
  - Pozzolanic Activity Index (PAI) using ASTM C311 methods
  - Qualitative XRD
- Quantitative XRD and TGA/DTA on 8 selected sources
Chemical Classification

![Graph showing chemical classification with data points and trend line]

- Class C
- Class F

$R^2 = 0.96$

**Variables:**
- CaO (%)
- Sum of Oxides ($\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$) (% wt.)
Chemical Classification

![Graph showing the relationship between CaO + MgO (%) and the sum of oxides (SiO₂+Al₂O₃+Fe₂O₃) (% wt.). The graph includes data points for Class C and Class F, with a correlation coefficient R² = 0.97.]
Chemical Classification

![Graph showing chemical classification based on CaO and Sum of Oxides (SiO₂+Al₂O₃+Fe₂O₃) (% wt.). The graph includes data points for Class C and Class F, with an R² value of 0.96.]
Available Alkali vs. Total Alkali

- Class C
- Class F
Strength Activity Index

![Graph showing the relationship between Strength Activity Index and Sum of Oxides (\(\text{SiO}_2+\text{Al}_2\text{O}_3+\text{Fe}_2\text{O}_3\)) (% wt.)]
Strength Test Study

• Strength Activity Index is questioned as it allows inert materials to pass

• Experiments performed with non-pozzolanic quartz filler

<table>
<thead>
<tr>
<th>Cement Type</th>
<th>Age (days)</th>
<th>100% Cement</th>
<th>20% Replacement</th>
<th>35% Replacement</th>
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<td>Strength (psi)</td>
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</table>
Strength Test Study

- Evaluated the Keil Hydraulic Index
- Replace an equal percentage of the control sample cement with an inert filler
- Evaluated different fillers, replacement levels, and cements

\[ \text{Keil Hydraulic Index} = \frac{a - c}{b - c} \times 100 \]

Where:

- \( a \) = the strength of 70% slag/30% portland cement at time \( t \);
- \( b \) = the strength of 100 percent portland cement at time \( t \);
- \( c \) = the strength of 70% ground quartz/30% portland cement at time \( t \)
## Keil Hydraulic Index

<table>
<thead>
<tr>
<th>ID-% Replace.</th>
<th>KHI - 7 days (%)</th>
<th>KHI - 28 days (%)</th>
<th>KHI - 56 days (%)</th>
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</thead>
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<tr>
<td></td>
<td>PC-1 PC-2 PC-3</td>
<td>PC-1 PC-2 PC-3</td>
<td>PC-1 PC-2 PC-3</td>
</tr>
<tr>
<td>FA-H-20</td>
<td>-31 4 -43</td>
<td>71 91 66</td>
<td>60 162 88</td>
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<td>FA-M-20</td>
<td>7 28 26</td>
<td>119 55 34</td>
<td>66 143 50</td>
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<tr>
<td>FA-O-20</td>
<td>10 -6 -24</td>
<td>7 73 39</td>
<td>57 84 26</td>
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<tr>
<td>FA-Q-20</td>
<td>53 44 26</td>
<td>135 102 109</td>
<td>120 185 121</td>
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<tr>
<td>FA-U-20</td>
<td>121 40 84</td>
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<td>FA-X-20</td>
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<td>FA-ZA-20</td>
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<td>FA-U-35</td>
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<td>FA-ZA-35</td>
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<td>124 46 114</td>
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<td>FA-ZC-35</td>
<td>140 45 39</td>
<td>83 75 82</td>
<td>102 99 96</td>
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</table>
Keil Hydraulic Index

Compressive Strength (ksi)

Keil Hydraulic Index

-50%  -40%  -30%  -20%  -10%  0%  10%  20%  30%  40%  50%

PC1-KI-7d
PC2-KI-7d
PC3-KI-7d
PC1-KI-56d
PC2-KI-56d
PC3-KI-56d
Strength Activity Index

![Graph showing the relationship between Strength Activity Index and Compressive Strength (kpsi). The graph includes data points for PC1-SA-7d, PC2-SA-7d, PC3-SA-7d, PC1-SA-56d, PC2-SA-56d, and PC3-SA-56d.]
Strength Test Study

• **Take Aways**
  
  – The Keil Hydraulic Index provided a test that identified strength contribution separate from “filler” effects
  
  – The test was sensitive to the cement used
  
  – Other evaluations of the existing strength activity index showed increasing the specification limit to 85% eliminated inert materials
  
  – Need to change the time required for testing to accommodate some Class F ash
Carbon Effects on Air Entrainment Study

- **Effect of Carbon on Air Entrainment**
  - The LOI test is adequate for estimating the total carbon but does not adequately identify if the carbon will affect air entrainment.
  - There is a need for a test to directly determine adsorption capacity.
  - The foam index test is useful at determining the interaction of the fly ash with air entrainment admixtures but has not been standardized and is not part of AASHTO M 295 or ASTM C311.
Carbon Effects on Air Entrainment Study

• Four tests evaluated:
  – Foam Drainage
  – Foam Index
  – Direct Adsorption Isotherm
  – Coal Fly Ash Iodine Number
Foam Index Test

• Evaluated 16 published versions
• Adopted the methodology of Harris with some modifications


Foam Index Test

- 2 g ash, 8 g cement
- 25 mL water
- Add AEA solution drop-wise
  - 5% vol. AEA / Water solution
  - (0.02 mL/drop)
- Shaken, not stirred
- Look for a stable foam
- Repeat...
Foam Index Test

- Vary Solution Strength
  - 2, 6, 10, 15 %vol. AEA
- Achieve uniform contact time
  - 12 to 18 minutes
- Determine total AEA added
  - Foam Index
Foam Index Test

- **Benefits**
  - Cheap & Easy

- **Issues**
  - Not achieving equilibrium
  - Not quantitative
  - Subjective
    - Agitation?
    - What is a stable foam?
Adsorption Based Tests

• Adsorption characterized by an adsorption isotherm

• Multiple adsorption models and isotherms

• Freundlich Isotherm

\[ q = K C^{1/n} \]

- \( q \) = mass of adsorbate adsorbed per unit mass of adsorbent, mg/g
- \( K \) = Freundlich isotherm capacity parameter, (mg/g) (L/mg)^{1/n}
- \( C \) = Solution concentration, mg/L
- \( 1/n \) = Freundlich isotherm intensity parameter, dimensionless
Freundlich Isotherm

Slope = $1/n$  
Intercept = $\log K$

![Graph showing Freundlich Isotherm with axes labeled Adsorption Capacity $q$ (mass adsorbate / mass adsorbent) and Solution Concentration. The graph includes data points and a line of best fit.]
Based on existing ASTM test method with modifications:

- Modified procedure for determining solution concentration
- COD test versus spectroscopic methods
- Needed to account for the contribution of cement
Direct Adsorption Isotherm determines AEA adsorption “capacity”
Direct Adsorption Isotherm

• Measures the adsorption capacity of the ash \textit{AND} the adsorption capacity of the AEA

• Can be used to estimate AEA dosage

• Simple execution
  – Scales
  – Beakers & Stir Plate & Filtration
  – COD Kits & Colorimeter
Direct Adsorption Isotherm

Vinsol resin

![Graph showing air content (% vol.) for different ash sources (FA-A, FA-G, FA-H, FA-J, FA-O, FA-T, FA-ZM, FA-ZN) with labels for Trial & Error, Isotherm Prediction, and Baseline Air Content 8.7%. The graph includes data points for % LCI (0.94, 2.32, 0.25, 1.59, 1.43, 0.45, 10.69, 3.41).]
Direct Adsorption Isotherm

Alpha olephin sulfonate
Coal Fly Ash Iodine Number

Designation: D4607 – 94 (Reapproved 2006)

Standard Test Method for Determination of Iodine Number of Activated Carbon

• Based on existing ASTM test method with modifications:
  – HCl treatment to acidify the ash and remove SO₃
  – Initial solution strengths modified (0.025 N vs 0.1 N)
  – Target concentration for determining capacity differs from published test method (0.01 N vs 0.02)
Coal Fly Ash Iodine Number

\[ y = 28.154x^{0.4371} \]

Fly Ash Iodine Number 9 (mg Iodine / g ash)
Coal Fly Ash Iodine Number

![Graph showing the relationship between Iodine Number (mg iodine/g CFA) and LOI (% wt.). The graph includes data points and a trend line with an R² value of 0.947.](Image)
Coal Fly Ash Iodine Number

![Graph showing the relationship between LOI (% wt.) and Iodine Number (mg iodine/g CFA).]
Iodine Number vs. Capacity

![Graph showing Iodine Number vs. Capacity](image-url)
Coal Fly Ash Iodine Number

• Measures the adsorption capacity of the ash
• Does not account for the adsorption capacity of the AEA
• Simple execution
  – Scales
  – Beakers & Stir Plate & Filtration
  – Titration
Carbon Effects on Air Entrainment Study

- **Take Aways**
  
  - Publish a standardized version of the foam index test that provides a uniform test time and mechanical agitation
  
  - Specify use of the coal fly ash iodine number to evaluate ash adsorption potential
  
  - Specify use of the direct adsorption isotherm test to evaluate fly ash – air entrainer combinations
ASR Mitigation Study

- Evaluate protocols for applying existing test methods (ASTM C1567 and ASTM C1293)
- Evaluate the Alkali Leaching Test (Shehata and Thomas, 2006) and correlate with the results of ASTM C1293 and ASTM C1567
  - The alkali leaching test is used to determine the free alkalis available to be leached from a particular combination of cement and fly ash
ASTM C1293

Prism expansion at 24 months (%)

0.141% Expansion for Control @ 24 Months

Percent replacement of cement with fly ash (% wt)

20  30  40
ASTM C1567 – 14 days

Expansion at 14 days (%)

0.35
0.30
0.25
0.20
0.15
0.10
0.05
0.00

FA-H
FA-M
FA-O
FA-Q
FA-U
FA-X
FA-ZA
FA-ZC

0.23% Control @ 14 Days

Percent Replacement of Cement with CFA (% wt.)
ASTM C1567 – 28 days

Expansion at 28 days (%) vs. Percent Replacement of Cement with CFA (% wt.)

- FA-H
- FA-M
- FA-O
- FA-Q
- FA-U
- FA-X
- FA-ZA
- FA-ZC

0.39% Control @ 28 Days
ASR Mitigation Study

• Take Aways
  – Confirmed the AASHTO PP-65 limits of 0.1% expansion @ 14 days for ASTM C1567
  – Provided data showing a 28-day limit on ASTM C1567 does not correlate with ASTM C1293
  – Alkali Leaching Test – no clear threshold of alkali release was identified that correlated with a 0.04% ASTM C1293 expansion
Recommended Changes to AASHTO M 295

• Add a maximum sum of the oxide limit (i.e., 70%) to the Class C classification

• Report CaO, MgO, Na$_2$O, and K$_2$O

• Adopt the use of the Iodine Number Test and the Direct Adsorption Isotherm Test under Optional Chemical Requirements
Recommended Changes to AASHTO M 295

- Raise the Strength Activity Index to 85% of control BUT allow the material to be qualified at 7, 28, OR 56 days

- Delete the available alkali limit

- Delete use of ASTM C441 (Pyrex Glass Test) and adopt ASTM C1567 with a 14 day limit of 0.1%
Questions?

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