## **Draft Technical Memorandum**

## Title: Ultra-High-Performance Concrete (UHPC) Use in Florida Structural Applications

## FDOT Contract Number: BDV31 977-105

## Task 2: Acquisition and Characterization of Materials for UHPC Specimen Fabrication

Submitted to The Florida Department of Transportation Research Center 605 Suwannee Street, MS 30 Tallahassee, FL 32399

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Prepared in cooperation with the State of Florida Department of Transportation and the U.S. Department of Transportation.

Approximate Conversions to SI Units (from FHWA)										
Symbol	When You Know	Multiply By	To Find	Symbol						
Length										
in	inches	25.4	millimeters	mm						
ft	feet	0.305	meters	m						
yd	yards	0.914	meters	m						
mi	miles	1.61	kilometers	km						
		Area								
in <sup>2</sup>	square inches	645.2	square millimeters	mm <sup>2</sup>						
ft <sup>2</sup>	square feet	0.093	square meters	m <sup>2</sup>						
yd <sup>2</sup>	square yard	0.836	square meters	m <sup>2</sup>						
mi <sup>2</sup>	square miles	2.59	square kilometers	km <sup>2</sup>						
	Volume									
fl oz	fluid ounces	29.57	milliliters	mL						
gal	gallons	3.785	liters	L						
ft <sup>3</sup>	cubic feet	0.028	cubic meters	m <sup>3</sup>						
yd <sup>3</sup>	cubic yards	0.765	cubic meters	m <sup>3</sup>						
	NOTE: volumes greater th	an 1000 L sha	ll be shown in m <sup>3</sup>							
	]	Mass								
OZ	ounces	28.35	grams	g						
lb	pounds	0.454	kilograms	kg						
	Temperatur	e (exact degre	ees)							
°F	Fahrenheit	5 (F-32)/9 or (F-32)/1.8	Celsius	°C						
	Illumination									
fc	foot-candles	10.76	lux	lx						
fl	foot-Lamberts	3.426	candela/m <sup>2</sup>	cd/m <sup>2</sup>						
	Force and P	ressure or Str	·ess							
lbf	pound-force	4.45	newtons	N						
lbf/in <sup>2</sup>	pound-force per square inch	6.89	kilopascals	kPa						

# I. Activities Performed During Period project start to 07/15/19

# TASK 2 – Acquisition and Characterization of Materials for UHPC Specimen Fabrication

Status: This task is complete

# 1 Aggregate Properties

Sand was obtained for the project from Edgar Minerals. Because of the improved behavior associated with small particle sizes, a fine masonry sand was selected. No coarse aggregate was selected for this project due to its lack of use in UHPC mixes. The sand was tested according to ASTM C128 [1] to determine the specific gravity and absorption. These results are shown in Table 1. Particle size distribution was determined using ASTM C136 [2]. These results are shown in Table 2. The sand was shown to have a fineness modulus of 1.40.

Table 1 Fine aggregate relative density and absorption

Relative Density (Specific Gravity) (Oven Dry)	2.63
Relative Density (Specific Gravity) (Saturated Surface Dry)	2.64
Apparent Relative Density (Specific Gravity)	2.66
Absorption	0.20 %

Sieve Size	Percent Retained	Cumulative Percent Retained
No.4	0.0	0.0
No. 8	0.1	0.1
No. 16	0.4	0.5
No. 30	3.7	4.2
No. 50	37.1	41.4
No. 100	52.7	94.1
No. 200	5.6	99.7
Pan	0.3	100.0

Table 2 Fine aggregate particle size distribution

# 2 Fibers

Two different steel fibers were ordered. Steel was selected because this is the predominant material used in UHPC mixes. Polypropylene fibers are rarely used as the only fiber in a UHPC

mix due to its relatively low modulus of elasticity. The properties of the steel fibers used are presented in Table 3. Depending on results, different fiber lengths or materials may be ordered in the future for this project.

	Bekaert Straight Fibers	Helix Twisted Fibers
Diameter	0.0078 in. (0.2 mm)	0.0197 in. (0.5 mm)
Length	0.5 in. (13 mm)	0.5 in. (13 mm)
Coating	brass	uncoated
Shape	straight	twisted
Aspect Ratio	65	26
Strength	377.1 ksi (2600 MPa)	246.5 ksi (1700 MPa)

Table 3 Fiber	properties
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# 3 <u>Cementitious Materials</u>

Cement and supplementary cementitious materials were obtained for use in the project. Because of the focus on designing non-proprietary UHPCs from materials local to Florida, an ASTM C595 [3] type IL cement was selected, along with an ASTM C618 [4] Class F fly ash and an ASTM C989 [5] slag cement. An ASTM C618 metakaolin and an ASTM C1240 [6] silica fume were also procured. Additional cementitious materials may be procured in the future based on test results and new materials becoming available.

All five of the cementitious materials were analyzed using X-ray fluorescence (XRF) at the University of Florida. Specimens were prepared by Raid Alrashidi and Megan Voss, and the analyses were performed by Dr. Ann Hetherington. The standards used are shown in Table 4. The results of the XRF analyses are listed in Table 5.

	С	CRL #19	90	CCRL #175		AGV-1			Slag BFS SL-1			Silica Stone R405			
%	Standard Average	Measured	% difference	Standard Average	Measured	% difference									
SiO <sub>2</sub>	19.60	19.91	1.60	18.71	19.08	1.99	59.88	60.04	0.27	35.73	34.63	-3.07	98.03	98.09	0.06
TiO <sub>2</sub>	0.25	0.25	-0.40	0.33	0.32	-2.42	1.07	1.05	-1.50		0.36		0.02	0.03	45.00
$Al_2O_3$	4.74	4.80	1.33	5.60	5.66	3.11	17.46	17.50	0.20	9.63	9.39	-2.49	1.07	1.02	4.58
Fe <sub>2</sub> O <sub>3</sub>	3.03	3.03	0.07	2.53	2.48	-1.94	6.88	6.84	-0.63	1.18	1.03	-12.54	0.05	0.08	64.00
MnO	0.07	0.09	22.86	0.10	0.10	-2.00	0.09	0.14	56.67		0.87		0.00	0.06	
MgO	4.58	4.49	-2.03	3.96	3.85	-2.83	1.56	1.83	17.50	12.27	12.68	3.36	0.02	0.07	230.00
CaO	63.51	63.48	0.04	64.87	63.76	-1.71	5.03	4.85	-3.68	32.48	37.58	0.25	0.03	0.03	-10.00
NaO	0.26	0.25	-5.77	0.35	0.30	-14.86	4.34	4.32	-0.41		0.34		0.06	0.10	63.33
K <sub>2</sub> O	0.85	0.77	-9.53	0.85	0.77	-9.06	2.94	3.02	1.96		0.50		0.71	0.78	9.86
$P_2O_5$	0.10	0.11	13.00	0.24	0.25	2.92	0.50	0.51	1.60		0.01		0.00	-0.01	
SO <sub>3</sub>	3.32	3.34	0.51	3.50	3.08	0.98	0.11			3.15	3.03	-3.94	0.00	0.05	

Table 4 References used for XRF

Table 5 Cementitious material composition as measured by XRF

	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	LOI
Material	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
Cement IL	18.82	0.22	4.79	3.10	0.06	0.80	62.85	0.08	0.25	0.41	3.02	5.45
Slag	34.79	0.64	13.17	0.78	0.32	4.66	43.71	0.19	0.41	0.04	3.00	0.02
Class F Fly Ash	57.31	1.03	22.59	6.76	0.06	2.02	3.37	0.54	2.27	0.24	0.49	2.8
Silica Fume	80.45	0.02	0.48	4.78	0.44	10.43	0.95	0.18	0.77	0.03	0.07	2.93
Metakaolin	47.41	1.51	41.14	0.44	0.05	0.00	0.05	0.20	0.18	0.04	0.04	0.68

The type IL cement was analyzed using X-ray diffraction (XRD). This method detects the crystalline phases of the cement, and Rietveld refinement was used to determine the percentages of each phase present in the cement. The PANalytical X'Pert powder diffraction machine at the University of Florida Nanoscale Research Facility was used. The scan was performed over a 20 angle range of 8° to 80°. A step size of 0.008 was used with each step lasting 10 seconds. The voltage was set to 45kV, and the current was 40 mA. Soller slits of 0.04 radians were used for the X-ray tube. Divergence slits and anti-scatter slits, each at 1°, were used. A fixed incident beam mask was also used. The software Profex was used for the Rietveld refinement analysis. Results are presented in Table 6.

Phase	Percent
Alite	44.26
Belite	23.19
Orthorhombic Aluminate	3.47
Cubic Aluminate	0.65
Ferrite	11.19
Bassanite	0.49
Gypsum	5.10
Calcite	11.65

Table 6 Cement composition from X-ray diffraction

Thermogravimetric analysis (TGA) was used to measure the calcite content of the cement. The mass loss between approximately 700°C and 800°C was attributed to CO<sub>2</sub> released from decomposition of calcite, and the mass loss was used to back-calculate the original mass of calcite. The calcite was determined to be 8.95% of the cement by weight.

The particle size distribution of the cementitious materials was determined using the HORIBA laser particle size analyzer (LPSA) at the State Materials Office of the Florida Department of Transportation. Each sample was run multiple times with increasing levels of ultrasonic treatment available in the laser particles size analyzer. The silica fume was sonicated with an external ultrasonic probe that provided significantly more power than the sonicator in the LPSA. This was done because laser particle size analysis done on densified silica fume often gives results showing larger particles than those actually present because the densified (intentionally agglomerated for safer handling) silica fume particles are hard agglomerates that are difficult to disperse into the individual crystallites that make up the densified particles. Sonication helps to break up the clumps of silica particles and give a more realistic distribution [7]. The final silica fume particle size distribution was measured after the densified particles were sonicated for 7.5 minutes with the external ultrasonic probe.

The median particle size of each material is shown in Table 7. Particle size distributions are shown in



Figure 1. Cumulative particle size distribution graphs are found in Figure 2. As seen in Figure 1, both metakaolin and silica fume have bimodal particle size distributions. For silica fume, it is likely that the peak in the higher particle size range is actually because of agglomerates of smaller silica fume particles that were not separated during sonication.

Material	D <sub>50</sub> (micrometers)
Type IL Cement	10.6
Fly Ash	17.7
Slag	9.24

Table 7 Median particle size of cementitious materials

Silica Fume	0.329
Metakaolin	4.56



Figure 1 Particle size distribution measured by laser particle size analysis



IL Cement ······· Fly Ash ---· Metakaolin - · - Silica Fume - - Slag Cement

Figure 2 Cumulative particle size distribution measured by laser particle size analysis

The densities of the cementitious materials were measured using a helium pycnometer at the Nanoscale Research Facility at the University of Florida. A Quantrachrome Ultrapyc 1000 gas pycnometer was used in this analysis. The material density results are presented in Table 8.

Material	Average Specific				
	Gravity				
Type IL Cement	3.11				
Fly Ash	2.39				
Slag	2.89				
Silica Fume	2.48				
Metakaolin	2.62				

Table 8 Material specific gravities

# 4 Planned testing

The materials described will be used to design non-proprietary UHPC mixes. These concretes will then be tested to characterize their durability and mechanical properties. In addition to comparing mechanical and durability properties of different concrete mixes, this research will also test the mechanical and durability properties resulting from different curing regimens. Three different curing regimens will be designed: lab curing in a fog room, steam curing, and simulated field curing.

Freeze-thaw resistance will be tested for the UHPC mixtures using ASTM C666 (ASTM C666). Two samples per mixture and curing regimen will be tested in ASTM C666 procedure A. Sample resonant frequency will be measured at least every 36 cycles for 300 cycles of freeze-thaw. Samples will be tested in low-temperature calorimetry at the FDOT State Materials Office to determine the temperature at which water freezes in UHPC and the material pore size distribution.

The porosity of the concretes will be tested using multiple methods. The low volume and connectivity of micro pores in UHPC makes it difficult to use certain durability tests that are commonly adopted for normal concrete. Concrete bulk and surface resistivity will be tested and compared with some more specialized laboratory techniques, such as mercury intrusion porosimetry (MIP). In addition, other methods that could easily be implemented in industry, such as NT Build 492 rapid chloride migration test and a new water pressure absorption test, will be performed to determine their correlations.

Resistance to fresh chloride penetration will be studied by testing the UHPC with varying levels of chlorides mixed into the concrete. Long-term durability will also be tested in the field by placing samples in the University of Florida's durability site at Seahorse Key.

Mechanical properties of UHPC and very high performance concrete (VHPC) will also be tested. The mixes will be designed to have compressive strengths ranging from 10 ksi up to above 21 ksi. In addition to testing compressive strength, creep will also be measured. Creep is currently estimated using a concrete's compressive strength, but because of the high compressive strengths and widely unknown behaviors of UHPC, the relation between creep and other properties will be investigated to provide a recommendation for designers. Eighteen UHPC samples will be creep-tested using size modifications recommended in ASTM C1856 [9]. However, the load at which creep will be tested will be determined following upcoming consultations with FDOT.

Tensile properties of UHPC will be determined using the direct tension testing procedure as designed and described in phase 1 of this project. The results of the direct tension tests will be compared to the results of simpler tests to characterize the tensile strength and ductility of the mixes. The purpose of this study is to develop an easily-executed test that can be used for quality control of non-proprietary UHPC mixes. Among the tests evaluated will be a simple flexure test, with a set-up similar to ASTM C1399 [10]. In this test, the deflection and load will be measured. This could be implemented by requiring certain loads to be met or exceeded at different deflections. The double-punch test, or Barcelona method, will also be used and compared to the direct tensile test. It is important that, in addition to finding the maximum tensile strength of a mix, the strain hardening behavior, or toughness, be measured. In addition to running these tests on mixes with different compressive strengths, these methods will also be tested on mixes using different fiber percentages as well as different fiber shapes. This will ensure that the different tests are compared over a wide range of tensile strengths and with varying post-cracking behaviors.

#### References

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- [10] ASTM C1399, "Standard Test Method for Obtaining Average Residual-Strength of Fiber-Reinforced Concrete," ASTM International, 2015, 6 pp.