Development of UHPC Using Ternary Blends of Ultra-Fine Class F Fly Ash, Meta-kaolin and Portland Cement

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Abstract:

The influence of binary and ternary blends of pozzolans on properties of UHPC were studied in this investigation. The pozzolans included in this study include ultra-fine class F fly ash (UFA), Meta-kaolin (MK), and silica fume (SFU). The properties of UHPC investigated include workability, time of set, autogenous shrinkage, compressive strength and drying shrinkage of cementitious paste with water-to-cementitious materials ratio at 0.20. The test results on paste containing binary blend of pozzolans and cement showed that the increase in the SFU content resulted in significant decrease in the workability, decrease in the autogenous shrinkage and increase in the drying shrinkage of paste. The increase in the UFA content resulted in significant retardation in the setting behavior of paste, decrease in the 1-day compressive strength and increase in the drying shrinkage of paste. The increase in the MK content resulted in significant decrease in the workability and time of set, improved 1-day compressive strength, increase in the autogenous shrinkage and decrease in the drying shrinkage of paste. The test results on paste containing ternary blend of MK, UFA and cement showed that the combined use of MK and UFA compensated the reduction in the 1-day compressive strength and increase in the drying shrinkage of paste due to the use of UFA, and compensated the reduction in the workability of paste due to the use of MK. Ultra-high performance concrete was prepared by using properly proportioned ternary blend of MK, UFA and cement that achieved comparable performance with UHPC containing SFU alone.

Keywords:

UHPC, meta-kaolin; fly ash; silica fume; ternary blend

1. Introduction

Ultra-high performance concrete (UHPC) is characterized by very low water-to-cementitious materials (w/cm) ratio (often less than 0.2), high cementitious materials content (often higher than 1000 kg/m^3 or 1682 lb/yd^3) and use of reinforcing fiber. Silica fume (SFU) is the most frequently used supplementary cementitious material (SCM) to improve the compressive strength and durability of UHPC, which is attributed to its micro-filler effect and pozzolanic effect (Tafraoui et al.; Randl et al.; Li et al.; Wille and Boisvert-Cotulio; Russell and Graybeal; Graybeal; Wille, Naaman and Parra-Montesinos). Considering the limited availability and high cost of quality SFU, other types of SCM, such as ultra-fine fly ash (FA) and meta-kaolin (MK), have gained attention as replacement for SFU, particularly in high-performance concretes. (Yu, Spiesz and Brouwers; Randl et al.; Tafraoui et al.).

It is well known that fly ash (FA) reduces the water demand and improves the workability of the fresh concrete through the ball bearing effect and the electrostatic effect (Mindess, Young and Darwin). One of the disadvantages of using FA is that it causes the reduction in compressive strength at early ages (Bai et al.; Mindess, Young and Darwin; Guneyisi and Gesoglu). Meta-kaolin (MK) contains reactive alumina-silicate. It can densify the microstructure and improve the mechanical properties and durability of concrete (Khatib and Wild; Poon, Kou and Lam; Zhang and Malhotra; Poon et al.; Wild, Khatib and Jones). The use of MK has been found to accelerate the cement hydration (da Cunha et al.; Frías, de Rojas and Cabrera; Lagier and Kurtis). However, using MK can reduce the workability and increase mixing time of fresh mixture (Guneyisi and Gesoglu; Tafraoui et al.). Studies on the combined use of FA and MK in normal strength concrete has shown that the slower strength development in concrete at early ages due to the use of FA can be addressed by the use of MK, and the reduction in workability of fresh concrete resulting from the use of MK can be limited by the use of FA (Guneyisi and Gesoglu; Bai et al.).

The present study explored the possibility of developing UHPC using MK and FA. The experiment was divided into three parts. In the first part, the effects of different types of SCMs (SFU, MK and FA) on the properties of paste fraction of UHPC were studied. The investigated properties included workability, setting time, autogenous shrinkage, compressive strength and drying shrinkage of paste. In the second part, the properties of paste using ternary blend of MK, FA and cement were studied. In the third part, UHPC mixtures were developed by proportioning proper amount of fine aggregate and steel micro fibers (SMF) into the selected paste mixtures.

2. Experimental Program

2.1. Materials

A Type III portland cement meeting ASTM C150 specification was used for this study. The specific gravity and Blaine's surface area of the cement were 3.15 and 540 m^2/kg (293 yd^2/lb), respectively. The fine aggregate was semi round natural siliceous sand meeting the gradation specification in ASTM C33 for fine aggregates. The percent passing values through each of the standard sieves for the fine aggregate used in this study are as follows (1 inch =25.4 mm): 9.5-mm sieve – 100%, 4.75-mm sieve – 99.8%, 2.36-mm sieve – 97.1%, 1.18-mm sieve – 82%, 600-µm sieve – 41.9%, 300-µm sieve – 14.0%, 150-µm sieve – 0.5% and 75-µm sieve – 0.1%. The specific gravity, water absorption, and fineness modulus of the sand were 2.63, 0.3% and 2.65, respectively. The steel micro fibers were approximately 13 mm in length and 0.2 mm in diameter. Their specific gravity and ultimate tensile strength were 7.8 and 2000 MPa (290 ksi), respectively. A polycarboxylic ester based high-range water-reducing admixture (HRWRA) in a powder form was used to improve the workability.

Three types of SCM were used for this study, which were class F ultrafine FA with an average particle size of 3 microns, high reactive MK with an average particle size of 1.4 microns, and SFU with an average particle size of 0.15 microns. The chemical compositions and physical properties of the portland cement and the SCMs are given in Table 1.

Table 1. Physical and Chemical Properties of Cementitious Materials

	SiO_2 (%)	Fe_2O_3 (%)	Al_2O_3 (%)	CaO (%)	MgO (%)	Na_2O_{eq} (%)	SO_3 (%)	LOI (%)	Specific gravity
Cement	20.4	3.5	6	64.4	1	0.49	3.5	1.34	3.15
SFU	95.5	0.3	0.7	0.4	0.5	1.4	--	0.89	2.2
FA	54.1	8.01	27.8	1.34	0.9	2.13	0.16	2.39	2.49
MK	50.4	0.45	42.6	0.02	0.16	0.22	0	1.63	2.2

2.2. Mixture Proportions

A total of 14 paste mixtures were investigated to study the properties of pastes using SFU, MK and FA as SCM. The investigated SCM/c ratio included 0.0, 0.05, 0.1, 0.2, 0.3 and 0.4. For the entire investigation, the w/cm (mass ratio) was fixed at 0.2, and the HRWRA dosage was fixed at 1% by mass of total cementitious materials. The relative mixture proportions of the 14 paste mixtures by mass are shown in Table 2.

Table 2. Relative Proportions of Materials in Paste (by mass)

Paste ID	c^a/c^a	SFU/c^a	MK/c^a	FA/c^a	SCM^b/c^a	$Water/cm^c$	$HRWRA/cm^c$ (%)
C	1.00	0	0	0	0.00	0.20	1.0
S1	1.00	0.10	0	0	0.10	0.20	1.0
S2	1.00	0.20	0	0	0.20	0.20	1.0
S3	1.00	0.30	0	0	0.30	0.20	1.0
M1	1.00	0	0.05	0	0.05	0.20	1.0
M2	1.00	0	0.10	0	0.10	0.20	1.0
M3	1.00	0	0.20	0	0.20	0.20	1.0
F1	1.00	0	0	0.10	0.10	0.20	1.0
F2	1.00	0	0	0.20	0.20	0.20	1.0
F3	1.00	0	0	0.30	0.30	0.20	1.0
MF1	1.00	0	0.05	0.15	0.20	0.20	1.0
MF2	1.00	0	0.10	0.10	0.20	0.20	1.0
MF3	1.00	0	0.05	0.25	0.30	0.20	1.0
MF4	1.00	0	0.10	0.20	0.30	0.20	1.0

Note: [a] cement; [b] supplementary cementing materials: silica fume alone or meta-kaolin + fly ash; [c] cementitious materials: cement + SCM

As Table 2 shows, paste C was pure portland cement paste. The next nine pastes contained binary blends of pozzolan and cement. The last four pastes were prepared by using ternary blends of MK, FA and cement. The levels of SCM/c investigated include 0.05, 0.1, 0.2, 0.3 and 0.4.

Selected paste mixtures with good performance were used to produce UHPC by adding sand and SMF. The sand-cementitious materials (s/cm) ratio was 1.25. The SMF content was 2% by volume of the UHPC mixture.

2.3. Specimens Preparation

Fresh pastes were prepared by a UNIVEX M20 planetary mixer. The mixing procedure was divided into three stages, as some of the mixtures took much longer time to reach fluid state than others, in particular when MK content was high. As the first step, the cementitious materials and the HRWRA were dry mixed for about 4 min. at low speed (100 RPM). Then the mixing water was added to the dry mixture. The mixing continued at low speed until the dry mixture started to behave as a fluid. The time (Tc) needed for the dry mixture to reach fluid state was recorded. In the final mixing stage, the fluid mixture was mixed for another 3 min at medium speed (300

RPM). The entire mixing process lasted for 8 to 39 min. depending on the materials and their proportions used in the pastes. Flow tests were conducted immediately after mixing.

The fresh paste was cast into molds without vibration. After casting, the specimens were kept in the moist room maintained at 100% relative humidity and 23 ^{o}C (73 ^{o}F) in accordance with ASTM C511. For the study of the compressive strength of paste, the specimens were de-molded at 24 hr. after casting, and stored in the moist room until the testing age. For the study of the drying shrinkage of paste, the specimens were de-molded at 48 hr. after casting due to the slow development in early age strength of some paste mixtures, particularly when FA content was high. The drying shrinkage specimens were stored following the procedures in ASTM C596.

2.4. Test Methods

Workability of paste was determined using a procedure similar to ASTM C1437. The fresh paste was allowed to spread freely on a level plastic plate without being dropped, instead of using a flow table. When the mixture stopped spreading (about 5 min after the removal of the flow mold) the diameter of the mixture was measured for calculating the flow value as described in the ASTM C1437 method.

The setting time of the paste was determined using a Vicat needle apparatus as described in ASTM C191.

Both autogenous and drying shrinkage of the paste specimens was determined in this study. Autogenous shrinkage of the UHPC paste was determined following the method described in ASTM C1698. The drying shrinkage was determined in accordance with ASTM C596 test procedure using standard length comparator measurements as described in ASTM C157.

Compressive strength was determined on 50 mm x 50 mm x 50 mm (2 in. x 2 in. x2 in.) cube specimens. Three specimens for each mixture were tested at 7 and 28 days following the procedures in ASTM C109.

Loss-On-Ignition (LOI) test was conducted to determine the bound water content calculated from the mass ratio of bound water to portland cement in paste at the ages of 1 and 28 days. A portion of paste from the middle of each of the three broken cubic specimens for the compressive strength test was collected, crushed and sieved. Particles passing through #4 sieve and retained on #8 sieve were collected as a sample. The sample was soaked in propanol for 5 hr. at ambient temperature to dissolve any free water and stop the hydration. Then the sample was dried at 105 ^{o}C (221 ^{o}F) for 24 hr. in an oven. The oven-dried sample was heated up to 1000 ^{o}C (1832 ^{o}F) at a rate of 200 ^{o}C/hr. (392 ^{o}F/hr.) in a muffle furnace and then held at that temperature for a period of 1 hr. The weight change between 105 ^{o}C (221 ^{o}F) and 1000 ^{o}C (1832 ^{o}F) was recorded to determine the bound water content. A correction was made in order to account for the weight change due to the raw materials such as cement, SFU, MK, and FA.

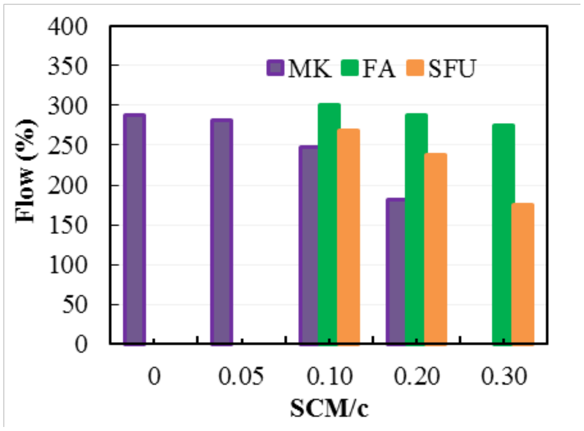
The specimens used for the drying shrinkage study of paste were used to investigate the volume of permeable voids of hardened paste following the procedures described in ASTM C642. The two ends of the specimen were sawn to remove the metal studs on each end of the specimen.

3. Results

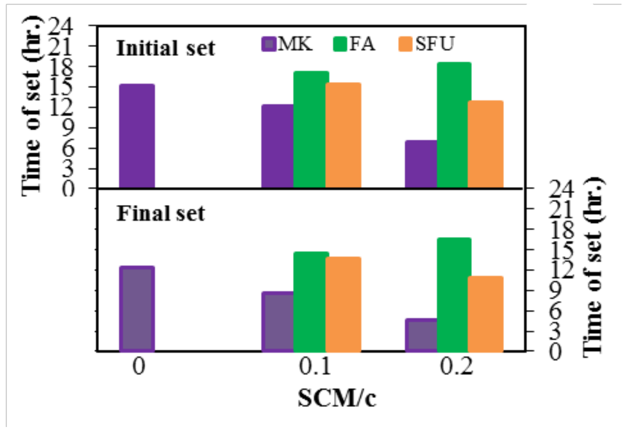
3.1. Paste Using Binary Blend of SCM and Cement

3.1.1. Fresh State Properties

The workability and setting time of paste is shown in Figure 1.



(a) Workability



(b) Time of Set

Figure 1. Fresh State Properties of Paste Using Binary Blend of SCM and Cement

As shown in Figure 1a, the flow increased with the FA content up to SCM/c=0.1, after which the flow slightly decreased with the FA content. The increase in the SFU or MK content continuously decreased the flow value.

As shown in Figure 1b, the increase in the FA content significantly increased both the times of initial and final set. However, the increase in the MK content decreased both the times of initial and final set. For pastes containing binary blends of cement and SFU, there appeared to be a threshold SFU content of SCM/c=0.1. When the SFU content increased from SCM/c=0 to SCM/c=0.1, the time of initial set increased by 12%, and the time of final set increased by 2%. When the SFU content increased from SCM/c=0.1 to SCM/c=0.2, the time of initial set decreased by 21%, and the time of final set decreased by 17%.

3.1.2. Autogenous Shrinkage

The autogenous shrinkage of paste is shown in Figure 2.

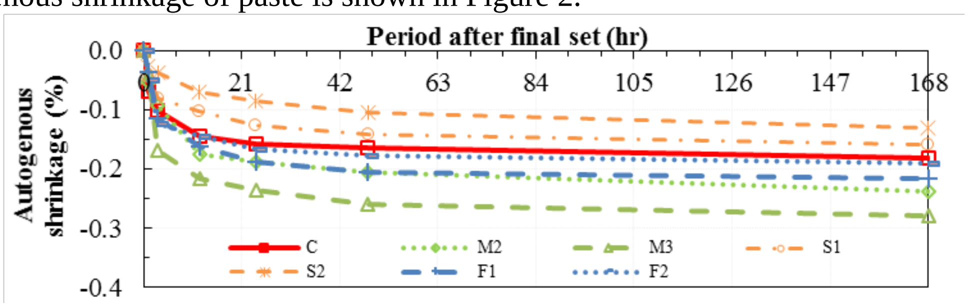
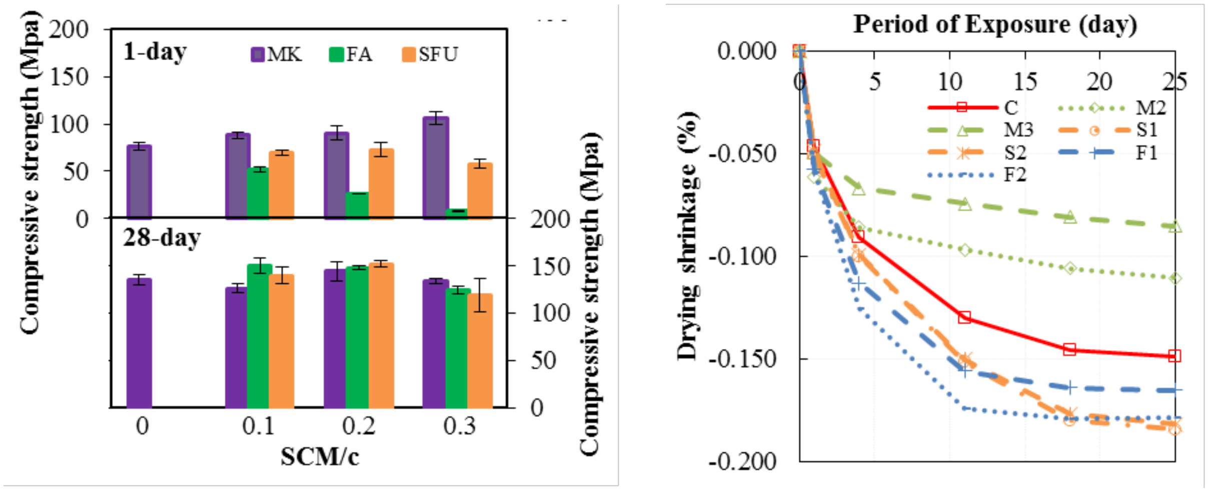


Figure 2. Autogenous Shrinkage of Paste Using Binary Blend of SCM and Cement

As shown in Figure 2, the development of autogenous shrinkage of pastes containing different types of SCM started to flat out at 48 hours after the final set of paste. The increase in the SFU content decreased the autogenous shrinkage of paste. However, the increase in the MK content increased the autogenous shrinkage of paste. The use of FA did not have a significant effect on the autogenous shrinkage of paste.

3.1.3. Hardened State Properties

The compressive strength and drying shrinkage of paste is shown in Figure 3.



(a) Compressive Strength (b) Drying Shrinkage

Figure 3. Hardened State Properties of Paste Using Binary Blend of SCM and Cement

As shown in Figure 3a, at the same level of SCM/c, the difference in the compressive strength of paste using different types of SCMs was very significant at the age of 1 day. The increase in the FA content continuously decreased the 1-day compressive strength, while the increase in the MK content continuously increased the 1-day compressive strength. For pastes containing binary blends of cement and SFU, the 1-day compressive strength was not significantly affected up to SCM/c=0.2, after which the 1-day compressive strength was decreased. At the age of 28 days, the use of SCM at proper proportions improved the 28-day compressive strength of paste, compared with the control. For pastes containing binary blends of FA and cement, the 28-day compressive strength of paste increased with the increase in the FA content up to SCM/c=0.1, after which the 28-day compressive strength of paste decreased with the increase in the FA content. For pastes containing binary blends of SFU and cement, the 28-day compressive strength of paste increased with the increase in the SFU content up to SCM/c=0.2, after which the 28-day compressive strength of paste decreased with the increase in the SFU content. The effect of MK on the 28-day compressive strength was not significant.

As shown in Figure 3b, the use of FA or SFU increased the drying shrinkage of paste. However, the increase in the MK content decreased the drying shrinkage of paste significantly.

3.2. Development of UHPC Using Ternary Blend of MK, FA and Cement

Four paste mixtures (MF1, MF2, MF3 and MF4) using ternary blends of MK, FA and cement were prepared. Their properties are presented in Table 3, as well as the properties of pastes using binary blends of SCM and cement.

Table 3. Properties of Paste Using Ternary Blend of MK, FA and Cement

	MF1	MF2	MF3	MF4	M3	F2	S2	S3
Flow (%)	269	239	258	239	181	289	238	175
1-day compressive strength (MPa)	61	59	38	32	106	26	72	58
28-day compressive strength (MPa)	145	145	149	136	134	147	152	119
25-day drying shrinkage (%)	-0.1557	-0.1490	-0.1570	-0.1407	-0.0853	-0.1787	-0.1820	-0.1680

As shown in Table 3, the investigated properties of pastes MF1 and MF2 fell into the middle of that of pastes M3 and F2, which illustrated that the use of ternary blend of MK, FA and cement addressed the shortcomings of using binary blend of MK and cement or binary blend of FA and cement. Among these four paste mixtures, pastes MF2 and MF3 exhibited good workability, high 1-day and 28-day compressive strength and relatively low drying shrinkage. They were selected to develop UHPCs. Paste S2 was also used to produce UHPC as a reference. The sand-cementitious materials (s/cm) ratio was 1.25. The SMF content was 2% by volume of the UHPC mixture. The mixture proportions for 1 m^3 of UHPC are shown in Table 4.

Table 4. Quantities of materials used for 1 m^3 of UHPC

UHPC ID	Constituents (kg/m^3)							
	Cement	SFU	MK	FA	Sand	SMF	Water	HRWRA
UHPC1 (MF2 [a])	808	0	81	81	1212	156	194	9.7
UHPC2 (MF3 [a])	743	0	37	186	1208	156	193	9.7
UHPC3 (S2 [a])	804	161	0	0	1206	156	193	9.6

[a] The parent paste mixture ID, see Table 2

The properties of UHPC are presented in Table 5.

Table 5. Properties of UHPC

	UHPC1	UHPC2	UHPC3
Flow (%)	144	150	106
1-day compressive strength (MPa)	66	58	74
28-day compressive strength (MPa)	142	150	160
25-day drying shrinkage (%)	-0.0390	-0.0520	-0.0692

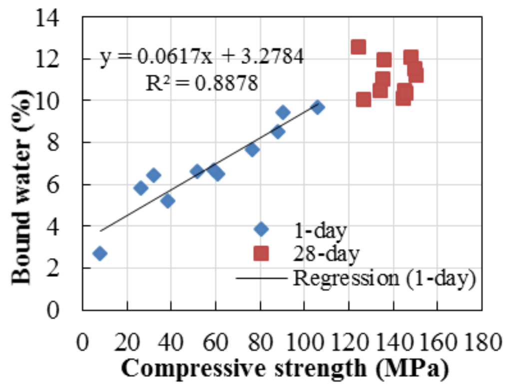
As shown in Table 5, at the same SCM content, UHPC prepared with ternary blend of MK, FA and cement exhibited slightly lower 1-day and 28-day compressive strength than UHPC prepared with binary blend of SFU and cement, which was evident by comparing UHPC1 and UHPC3. However, UHPC1 exhibited significantly higher workability and lower drying shrinkage than UHPC3. It should be noted that UHPC 2 exhibited 28-day compressive strength of 150 MPa (21750 psi), which fell into the typical range of compressive strength of UHPC.

4. Discussions

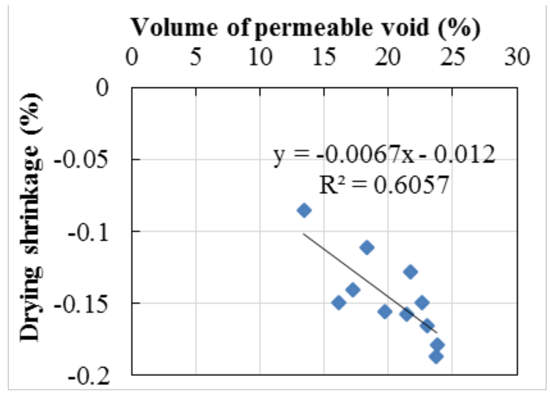
4.1. Cementitious Paste Containing MK and FA

As shown in this study, SFU is still the desirable SCM in the UHPC formulation than MK and FA, from the consideration of achieving high 28-day compressive strength. Among 14 paste mixtures investigated, the highest 28-day compressive strength of 152 MPa (22040 psi) was achieved by using SFU at dosage of SCM/c=0.2. The use of FA and the use of MK have distinct effects on the properties of cementitious paste with very low w/cm, particularly on the time of set, 1-day compressive strength and drying shrinkage of paste. The use of FA in paste did not have significant effect on the workability and autogenous shrinkage of paste. However, it significantly prolonged the time of set, resulted in a decrease in the 1-day compressive strength and an increase in the drying shrinkage of paste. The use of MK decreased the workability and time of set of paste. It also increased the autogenous shrinkage of paste. However, the use of MK improved the 1-day compressive strength and decreased the drying shrinkage of paste. The combined use of MK and FA addressed the reduction in the early age compressive strength, prolonged time of set and increased drying shrinkage of paste due to the use of FA, and addressed the reduction in the workability of paste due to the use of MK.

To understand the behavior of compressive strength and drying shrinkage of pastes containing MK or FA, the bound water content of paste and the volume of permeable voids (V_P) of pastes C, F1, F2, F3, M1, M2, M3, MF1, MF2, MF3 and MF4 were investigated. The correlation between compressive strength and bound water content of pastes and the correlation between 25-day drying shrinkage and V_P of pastes are presented in Figure 4.



(a) Compressive Strength vs. Bound Water Content



(b) 25-day Drying Shrinkage vs. V_p

Figure 4. Correlation between Properties of Paste

As showed in Figure 4a, the 1-day compressive strength of paste had a strong correlation with the 1-day bound water content, which indicated that the 1-day compressive strength of paste was significantly affected by the degree of cement hydration. As reported in the previous literature, the reduction in the 1-day compressive strength of paste due to the use of FA was attributed to the low pozzolanic reactivity of FA at early ages (Guneyisi and Gesoglu; Fajun, Grutzeck and Roy; Brooks, Johari and Mazloom; Frías, de Rojas and Cabrera), and the increase in the early age compressive strength of paste due to the use of MK was attributed to the promoted cement hydration in the presence of MK (da Cunha et al.; Frías, de Rojas and Cabrera; Guneyisi and Gesoglu). In this study, it was considered that proportions of MK and FA that could promote the cement hydration increased the 1-day compressive strength of pastes.

However, the 28-day compressive strength of paste did not have significant correlation with the 28-day bound water content. It was considered that at fixed w/cm as this study, the 28-day compressive strength of paste was affected more by factors such as the packing of the component materials' particles and the C-S-H gel characteristic, rather than by the degree of cement hydration.

As shown in Figure 4b, the drying shrinkage of paste at the period of exposure of 25 days had a good correlation with V_P. The possible explanation for this behavior could be that the less permeable micro-structure of paste reduced the moisture loss from the paste to the external environment, which resulted in less drying shrinkage. Proportions of MK and FA that could reduce the volume of permeable voids decreased the drying shrinkage of paste.

4.2. Development of UHPC Containing MK and FA

UHPC mixture (i.e. UHPC 2) with a high early age compressive strength was prepared by using ternary blend of MK, FA and cement. Such mixture presented higher workability and lower drying shrinkage, but slightly lower 28-day compressive strength than UHPC 3. This indicates that at the cost of slightly sacrificing the 28-day compressive strength, a UHPC using ternary blend of MK, FA and cement can be prepared with higher workability and lower drying shrinkage than UHPC using binary blend of SFU and cement. The use of MK and FA in producing UHPC also has sustainability and environmental significance than the use of SFU.

5. Conclusions

In this study, the effects of supplementary cementitious materials on several properties of the paste fraction of UHPC were studied. UHPC mixtures were developed using MK and FA. Based on the materials and proportions used in this study, the following conclusions are drawn:

- The workability of paste decreased with the increase in the MK content or SFU content of the paste when MK or SFU was used alone as SCM. However, the use of FA alone as SCM in paste did not significantly affect the workability of paste.
- Both the times of initial and final set of paste decreased with the increase in the MK content or decrease in the FA content when MK or FA was used alone as SCM. SFU did not have a significant effect on the time of initial set and time of final set.
- The autogenous shrinkage of paste increased with the increase in the MK content, and decreased with an increase in the SFU content, when MK or SFU was used alone as SCM. However, the use of FA alone as SCM in the paste did not significantly affect the autogenous shrinkage of paste.
- The drying shrinkage of paste increased with increase in the SFU or the FA content when SFU or FA was used alone as SCM. However, an increase in the MK content significantly reduced the drying shrinkage of paste, when MK was used alone as SCM.
- The 1-day compressive strength of paste was not significantly affected by SFU content up to SCM/c=0.2, beyond which the 1-day compressive strength decreased. The increase in the MK content resulted in significant increase in the 1-day compressive strength of paste. However, the increase in the FA content resulted in significant decrease in the 1-day compressive strength.
- SFU was the more desirable SCM than MK and FA in the UHPC formulation from the consideration of achieving high 28-day compressive strength.
- The combined use of MK and FA as SCM in paste could address the reduction in the 1-day compressive strength and increase in the drying shrinkage due to the use of FA alone as SCM in the paste, and counter the reduction in the workability due to the use of MK alone as SCM in the paste.
- Proportions of MK and FA that improved the early age cement hydration increased the 1-day compressive strength of paste; however, the 28-day compressive strength of paste did not follow significant correlation with the degree of cement hydration. Moreover, proportions of MK and FA that reduced the volume of permeable voids of hardened paste resulted in a decrease in the drying shrinkage of paste as a dense microstructure of paste reduced the loss of moisture from within the hardened paste into the external environment.
- Based on this research it can be concluded that UHPC mixtures can be prepared by combined use of MK and FA as SCM, instead of using SFU alone as SCM, to address the drying shrinkage issue.

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