Influence of supplementary cementitious materials on sulfate resistance of ultra-high-performance concrete

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Abstract: The study presented herein investigated the effect of supplementary cementitious materials (SCMs) on the sulfate resistance of ultra-high-performance concretes (UHPCs) made with ASTM Type III and Type V cement. Three types of UHPCs were prepared for each cement type, namely, one with 100 % cement (C100) and two others with 20 % cement replaced by fly ash (FA20) and a combination of silica fume and fly ash (FA15SF5). These UHPCs were exposed to a 5 % sodium sulfate solution for 365 days to evaluate their sulfate resistance. In addition to the sulfate-induced expansion, the compressive strengths of the studied UHPCs were evaluated under two curing conditions: continuous water curing and curing under exposure to the 5 % sulfate solution. The Rapid Sulfate Permeability Test (RSPT) was also conducted at 28 and 90 days to assess rapid sulfate ions penetration into the studied UHPCs. The results revealed that the addition of supplementary cementitious materials (SCMs) significantly enhanced sulfate resistance in both Type III and Type V cement UHPCs. However, Type V cement UHPCs consistently showed reduced sulfate-induced expansion by approximately 20.4 % compared to Type III cement UHPC, primarily due to its lower C_3A content. The Rapid Sulfate Permeability Test (RSPT) results revealed that, at both 28 and 90 days, Type V UHPCs consistently displayed lower penetrability compared to Type III UHPCs. Furthermore, Type V cement UHPCs incorporating SCMs exhibited an average surface penetration reduction of about 6 % relative to their Type III cement counterparts.

1 Introduction

~~Sulfate attack is a critical durability i~~ssue for concrete, particularly in environments with high sulfate concentrations such as marine areas, swamps, and sulfate-rich groundwater [1, 2]. This deterioration occurs when sulfate ions chemically react with the cement paste, forming expansive compounds like ettringite and gypsum that lead to cracking, spalling, and structural damage [3, 4]. Factors influencing sulfate attack include sulfate concentration, concrete permeability, cement type, and the use of supplementary cementitious materials (SCMs) [5, 6]. Although ultra-high-performance concrete (UHPC) exhibits superior mechanical and transport properties than conventional concrete, it remains susceptible to sulfate attack under certain conditions [7, 8]. The severity of sulfate attack depends on the chemical and physical mechanisms involved. Sulfate ions interact with calcium hydroxide and aluminate phases in the cement paste, forming expansive sulfate salts that generate internal stresses and reduced mechanical strength [9], [10], [11]. High permeability accelerates the ingress of sulfate ions, worsening the damage [5, 6]. Additionally, the type of cement and sulfate (e.g., sodium, magnesium, or ammonium) influences the reaction kinetics and resulting deterioration [7].

SCMs such as fly ash, silica fume, ground granulated blast-furnace slag (GGBS), and metakaolin have been shown to enhance sulfate resistance by reducing concrete porosity and promoting pozzolanic reactions. These reactions consume calcium hydroxide, forming additional calcium silicate hydrate (C-S-H) gel, which densifies the microstructure and hinders sulfate ion ingress [12, 13]. Studies indicate that the effectiveness of SCMs varies depending on their type, size and grain size distribution and proportion, and cement compatibility [12, 14]. This study examined the effect of SCMs on the sulfate resistance of UHPCs made with ASTM Type III and Type V cement. Three UHPC types were prepared for each cement: one with 100 % cement (control), and two with 20 % cement replaced by fly ash (FA20) and a combination of fly ash and silica fume (FA15SF5). These UHPCs were exposed to a 5 % sodium sulfate solution for 360 days to evaluate sulfate-induced expansion and compressive strength. The compressive strength was assessed under continuous water curing and sulfate exposure, while the Rapid Sulfate penetration Test (RSPT) was performed on 28- and 90-day moist-cured.

2 Experimental program

2.1 Materials

The UHPC mixtures were produced using ASTM Type III and Type V Portland cement (ASTM C150), along with class F fly ash (ASTM C618) and silica fume (ASTM C1240) as pozzolanic materials. The chemical and physical characteristics of the two types of cement and

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SCMs are provided in Table 1. Locally sourced fine aggregates with sizes ranging from 0.075 mm to 4.75 mm were used, and their grading was optimized using the modified Andreasen and Andersen model [14]to achieve maximum packing density at a distribution modulus of 0.21. The combined fine aggregates had a specific gravity of 2.80 and an absorption rate of 0.45 %. A polycarboxylate-based high-range water-reducing admixture (HRWRA) was incorporated to ensure adequate workability, with dosages adjusted to maintain a uniform flow across the studied UHPCs.

Table 1. Chemical and physical characteristics of cementitious materials.

Composition	**V (%)**	**III (%)**	**FA (%)**	**SF (%)**
SiO_2	20.9	20.4	59.93	94.72
CaO	62.3	63.5	4.67	-
Al_2O_3	4.1	5.5	22.22	-
Fe_2O_3	3.8	1.2	5.16	-
MgO	3.0	2.7	-	-
SO_3	2.1	3.3	0.38	0.23
*$Na_2O + K_2O$	0.53	0.44	1.29	0.47
(LOI)	2.0	1.2	0.32	2.82
Tricalcium silicate (C_3S)	55	56	-	-
Dicalcium silicate (C_2S)	21.5	19.6	-	-
Tricalcium aluminate (C_3A)	4.0	13	-	-
Tetracalcium aluminoferrite (C_4AF)	12.2	3.7	-	-
Physical Properties	V	III	FA	SF
325 Mesh (45 μm), % passing	98.3	99.0	85.4	97.12
Blain fineness (m^2/kg)	389	541	-	-

NOTATION: V – Type V cement; III – Type III cement; FA – Fly Ash; SF – Silica Fume; LOI – Loss on ignition. * $Na_2O + K_2O$; the combination of sodium and potassium oxide

2.2 Mixture proportion and design

Three UHPCs were studied: a control mix (C100) containing 100 % cement, a binary mix with 20 % fly ash (FA20), and a ternary mix with 15 % fly ash and 5 % silica fume (SF15FA5). The mixture proportions for these UHPCs are presented in Table 2. All UHPCs maintained a constant water-to-cementitious materials ratio (w/cm) of 0.21. HRWRA dosages ranged from 10.1 to 14.3 kg/m³, that ensured a uniform flowability for all studied UHPCs.

Table 2. Mixture constituents and proportions of UHPCs (kg/m^3).

Mix ID	**C**	**SF**	**FA**	**W**	**HRWRA**	**Sand**
C100	1089	-	-	239	14.2	1128
FA20	871	-	161	206	10.3	1128
FA15SF5	871	39	121	206	10.4	1128

NOTATION: C - Cement; SF - Silica Fume; FA - Fly Ash; W -Water; HRWRA - High Range Water Reducing Admixture.

2.3 Mixing, Sampling, Curing, and Testing

The production of UHPC requires prolonged mixing durations and higher energy input due to the high concentration of fine particles and low water-cementitious materials ratio (w/cm). The mixing process began with dry blending of the cementitious materials for five minutes in a Hobart-type mixer, followed by the gradual incorporation of fine aggregates and an additional five minutes of dry mixing. Subsequently, 90 % of the mixing water was added and mixed for five minutes, followed by the remaining water and HRWRA. This ensured homogeneity in the UHPCs. For each testing mixture, four expansion bars were prepared using prism dimensions of 25 mm × 25 mm × 290 mm according to ASTM C1012 standards. Three cylindrical specimens of 50 mm × 100 mm were prepared for compressive strength testing, while three disk-like samples of 100 mm × 50 mm dimension were prepared for the Rapid Sulfate Penetration Test (RSPT). After casting, specimens were sealed in plastic wrap and stored at room temperature (21 ± 3) °C for 24 hours, followed by three days of water curing to achieve the target compressive strength of (20 ± 1.0) MPa (2900 ± 145) psi per ASTM C1012 requirements. Following initial curing, half of the cylindrical specimens and the expansion bars were submerged in 5 % sodium sulfate (Na_2SO_4) solution, while the remaining cylindrical samples continued curing in a water environment for 360 days. The sulfate solution tanks maintained a solution-to-mortar volume ratio of 3:1, with consistent circulation via submersible pumps. The pH of the solution was adjusted daily to 7.0 ± 1 using 0.5 N H_2SO_4 during the first six months and weekly thereafter to ensure consistent sulfate ion availability.

3 Results and discussion

3.1 Compressive strength of Type V & III cements UHPCs under water and sulfate curing

The compressive strength results of Type V and Type III cement UHPCs cured under water and sulfate solution for 360 days are presented in Figures 1 and 2. The incorporation of supplementary cementitious materials (SCMs), significantly enhanced the compressive strength performance of UHPCs across both cement types. Even under sulfate exposure, SCM-incorporated UHPCs consistently outperformed their control counterparts, demonstrating the effectiveness of SCMs in mitigating sulfate-induced deterioration.

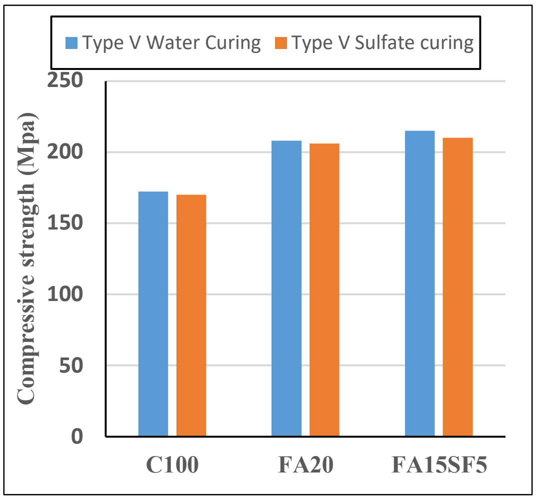

Fig 1. Compressive strength of Type V cement specimens under water and sulfate curing.

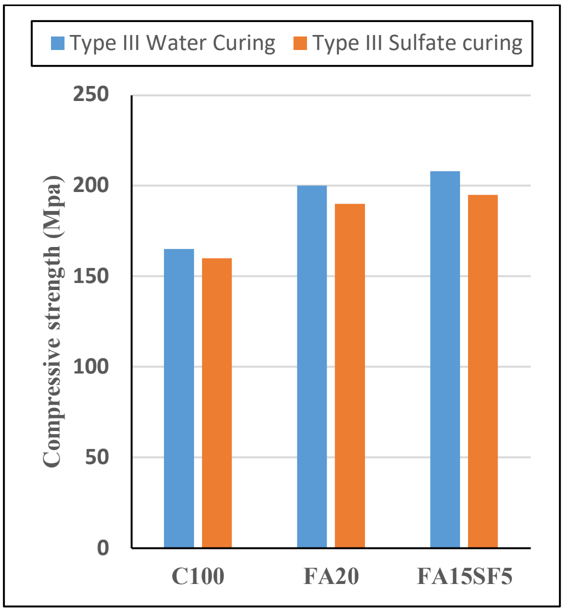

Fig 2. Compressive strength of Type III cement specimens under water and sulfate Curing

For Type V cement UHPCs, the binary FA20 UHPC exhibited compressive strengths of 208 MPa under water curing and 206 MPa under sulfate curing, marking a 21 % strength improvement over the control UHPC (C100). The ternary FA15SF5 UHPC showed an enhanced strength, reaching 215 MPa under water curing and 210 MPa under sulfate curing, reflecting 25 % and 23 % increases, respectively, over the control UHPC.

A similar trend was observed for Type III cement UHPCs, where FA20 achieved 200 under water curing and 190 MPa under sulfate curing, resulting in a 21 and 18.7 % increase over the control UHPC, respectively. The FA15SF5 ternary attained 208 under water curing and 195 MPa under sulfate curing, translating to 26 and 22 % strength improvements over the control UHPC.

Comparing the binary and ternary UHPC, FA15SF5 ternary UHPC demonstrated a slightly higher compressive strength over FA20 in Type V cement UHPCs, recording 3.4 % and 1.9 % higher compressive strengths under water and sulfate curing, respectively. Similarly, in Type III cement UHPCs, the ternary blend exhibited marginally higher compressive strength under both curing conditions by an average of 2.6 %, emphasizing the advantage of incorporating silica fume. The fineness of silica fume, as shown in Table 1, contributed to improved packing density and enhanced pozzolanic activity, facilitating greater C-S-H formation. This may have likely increased the matrix densification, to result in improved compressive strength of the ternary UHPC.

When examining the effect of sulfate curing on both types of cement UHPCs, Type III cement UHPCs consistently exhibited lower strength reductions compared to Type V cement UHPCs as seen in Fig 2. The control UHPC using Type V cement experienced only a 1.16 % strength reduction, decreasing from 172 MPa to 170 MPa, whereas the Type III cement UHPC showed a 3.03 % reduction, dropping from 165 MPa to 160 MPa. Similarly, in the binary FA20 UHPC, Type V UHPC recorded less than 1 % decrease in compressive strength, declining from 208 MPa to 206.8 MPa under sulfate curing. In contrast, Type III FA20 UHPC showed a 5.3 % reduction, falling from 200 MPa to 190 MPa, which is 5 times more than the strength loss observed in its counterpart Type V cement UHPCs. For the ternary FA15SF5 UHPC, Type V cement UHPC exhibited a 2.4 % reduction, with compressive strength decreasing from 215 MPa to 210 MPa, whereas Type III cement ternary UHPC showed 6.25 % reduction, dropping from 208 MPa to 195 MPa.

The observed reduction in compressive strength for both cement types UHPCs was marginal at best. Type III UHPCs showed a higher reduction in compressive strength that may be partially attributed to the softening of the concrete matrix due to the formation of gypsum ($CaSO_4 \cdot 2H_2O$).

Although ternary UHPCs exhibited higher overall compressive strengths, they also showed a slightly greater strength reduction under sulfate curing, particularly in Type III cement UHPCs. This may be due to the higher surface area of silica fume (SF), which, while beneficial for densification, also accelerated early reactions, potentially amplifying the effects of sulfate-induced degradation over extended exposure. Despite this, ternary UHPCs still outperformed their binary counterparts in absolute strength values, demonstrating that the refined microstructure and reduced permeability provided by the combined SCMs helped to offset the potential for sulfate deterioration.

3.2 Expansion due to sulfate exposure

Tables 3 and 4 present the expansion measurements of Type III and V cement UHPCs over a 52-week immersion period in a 5 % sulfate solution. Expansion was evident from the first week of exposure, as the Type V cement control UHPC expanded by 0.0012 %, while the Type III control UHPC exhibited a higher expansion of 0.0020 %, reflecting a 67 % increase. As exposure progressed, expansion steadily increased in both UHPCs types, though Type III cement consistently exhibited a greater expansion percentage.

Table 3. Type III UHPCs expansion measurements at key test periods (%).

Type III UHPCs			
	C100	**FA20**	**FA15SF5**
1 week	0.002	0.0022	0.0027
4 weeks	0.009	0.0077	0.0065
8 weeks	0.0124	0.0083	0.0069
12 weeks	0.0128	0.0094	0.0073
15 weeks	0.0133	0.0105	0.0082
20 weeks	0.0148	0.0122	0.009
25 weeks	0.0157	0.0128	0.0096
30 weeks	0.0165	0.0134	0.0108
35 weeks	0.0177	0.014	0.0111
40 weeks	0.0184	0.0146	0.0125
45 weeks	0.0188	0.0154	0.0132
52 weeks	0.0195	0.0165	0.0138

Table 4. Type V UHPCs expansion measurements at key test periods (%).

Type V UHPCs (%)			
	C100	**FA20**	**FA15SF5**
1 week	0.0012	0.001	0.0017
4 weeks	0.0076	0.0066	0.005
8 weeks	0.0084	0.0071	0.0059
12 weeks	0.0095	0.008	0.0065
15 weeks	0.0118	0.0094	0.0074
20 weeks	0.0135	0.0118	0.008
25 weeks	0.0142	0.0125	0.0088
30 weeks	0.0148	0.0134	0.0095
35 weeks	0.0153	0.0138	0.011
40 weeks	0.0157	0.0141	0.0117
45 weeks	0.016	0.0142	0.0126
52 weeks	0.0162	0.0145	0.013

By 52 weeks, the Type V control UHPC reached 0.0162 % expansion, whereas Type III cement UHPC expanded further to 0.0195 %, representing a 21 % greater. The observed differences in expansion between Type V and III cement UHPCs are primarily attributed to the varying tricalcium aluminate (C_3A) content in the cements. Type V cement is formulated for high sulfate resistance and contains a maximum C_3A content of 5 %, whereas Type III cement is designed for high early strength and typically has a higher C_3A content (13% for this study). The higher abundance of aluminates in Type III cement as shown in Table 1, also led to a more aggressive rate of sulfate-induced expansion [12].

Without a pozzolan to physically reduce porosity and chemically-bind calcium ions released from calcium hydroxide ($Ca(OH)_2$), the high C_3A content of Type III cement UHPC proved to be less resistant to sulfate attack compared to the control Type V cement UHPC.

The incorporation of SCMs significantly influenced sulfate-induced expansion in both Type V and Type III UHPCs, with ternary UHPCs exhibiting the highest resistance. In Type V UHPCs, FA20 demonstrated a steady reduction in expansion, measuring 16 % lower than the control UHPC at 1 week, 12 % lower at 25 weeks, and 11 % lower at 52 weeks. Meanwhile, the ternary FA15SF5 UHPC initially expanded 125 % more than the control at 1 week, but over time, it exhibited a 38 % lower expansion at 25 weeks and a 20 % reduction at 52 weeks. Similarly, in Type III cement UHPCs, FA20 expanded 21 % more than the control at 1 week but showed a 19 % reduction by 25 weeks and a 16 % decrease at 52 weeks, indicating improved sulfate resistance compared to the control UHPC. The ternary FA15SF5 UHPC exhibited the lowest overall expansion, starting at 39% below control UHPC at 1 week, with a 29% decrease by 25 weeks and a 15% reduction at 52 weeks.

The enhanced performance of SCMs-replaced UHPCs is primarily due to their pozzolanic activity. This activity led to the formation of additional calcium silicate hydrate (C-S-H), which reduced the porosity of the concrete matrix. In addition, substituting a portion of the cement with SCMs effectively reduced the tricalcium aluminate (C_3A) content in the mixture [15]. Since C_3A readily reacts with sulfates to form expansive compounds such as ettringite, lowering its concentration diminishes the potential for deleterious reactions that can compromise concrete resistance to external sulfate attack.

The ternary FA15SF5 UHPC demonstrated the best performance in mitigating sulfate-induced expansion compared to binary FA20 UHPC, regardless of cement type. This is due to the incorporation of 5 % silica fume (SF), owing to its higher fineness relative to fly ash (FA) as shown in Table 1, which provided a larger reactive surface area. This increased surface area facilitated the rapid formation of additional calcium silicate hydrate, thereby reducing porosity and penetrability into capillary pores. Consequently, the ingress of sulfate ions was impeded, thereby enhancing the concrete's resistance to sulfate attack. This finding aligns with the hypothesis that mixtures with higher fineness cement and appropriate dosages of pozzolans exhibit reduced expansion under sulfate exposure [13].

Comparing the two cement types over the 52-week period, Type III UHPC consistently exhibited greater sulfate-induced expansion than Type V UHPC. At 1 week, the Type III control UHPC expanded 67 % more than the Type V control, while at 25 weeks, it showed a 11 % higher expansion and 20.4 % expansion by 52 weeks. For binary FA20 UHPC, the Type III mix initially expanded 120 % more than Type V at 1 week, but the gap gradually narrowed, with a 14 % higher expansion in Type III by 52 weeks. Ternary FA15SF5 UHPC exhibited 59 % more expansion at 1 week, which reduced to 6.2 % at 52 weeks.

The observed differences in expansion between Type V and Type III cement UHPCs can be attributed to the formation of ettringite compounds, a hydrous calcium aluminium sulfate mineral with the chemical formula $Ca_6Al_2(SO_4)_3(OH)_{12}\cdot 26H_2O$. Ettringite forms during the hydration of Portland cement when tricalcium aluminate (C_3A) reacts with gypsum ($CaSO_4\cdot 2H_2O$) in the presence of water:

The higher C_3A content in Type III cement led to increased formation of ettringite compounds when exposed to sulfate ions, resulting in more expansion compared to Type V cement. The sulfate-induced expansion is also influenced by cement Blaine fineness.

Type III cement, with a higher Blaine fineness of 541 m²/kg was more susceptible to sulfate attack due to its increased fineness which accelerated hydration, leading to rapid strength gain but also a higher propensity for expansion due to the formation of expansive ettringite compound. In contrast, Type V cement had a lower Blaine fineness of 347 m²/kg and a C_3A content of 4 %, resulting in slower hydration and reduced formation of ettringite compounds, which facilitated its higher resistance to sulfate-induced expansion. This aligns with findings that cement mortars made with lower Blaine fineness and low tricalcium aluminate content exhibited less expansion due to sulfate attack compared to those with higher Blaine fineness [16].

3.3 Rapid Sulfate Permeability Test

The results of the Rapid Sulfate Permeability Test (RSPT) using test samples water-cured at 28 and 90 days for both Type V and Type III cement UHPCs are presented in Fig. 3 and 4. These results offer valuable insights into the sulfate resistance of the studied UHPCs and are consistent with findings of section 3.2 on expansion behavior over extended periods. The lower RSPT coulomb values observed for Type V cement UHPC correlated well with their enhanced performance against sulfate-induced expansion over the 52-week testing period.

At 28 days, the control UHPC (C100) exhibited the highest penetration for both cement types. Type V cement C100 showed a value of 1500 Coulombs, while Type III cement C100 recorded 1574 Coulombs, which is an increase of 5% higher penetrability. Type V cement binary fly ash replaced UHPC (FA20), showed reduced penetrability compared to its Type III cement counterpart at both 28 and 90 days.

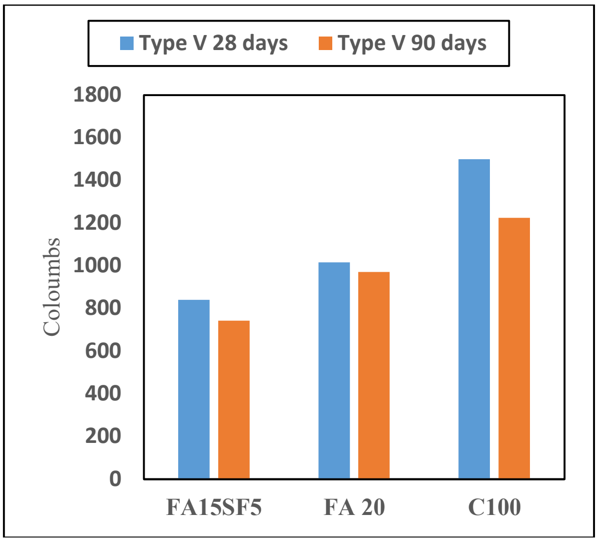

Fig. 3. RSPT results for Type V cement UHPCs.

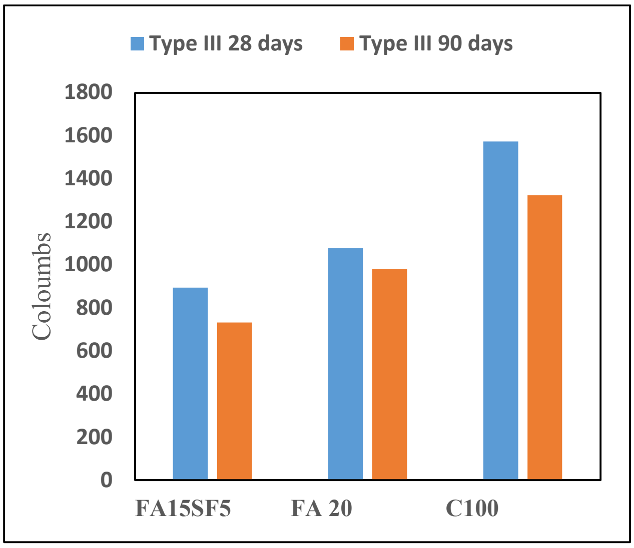

Fig. 4. RSPT results for Type III cement UHPCs.

At 28 days, Type V cement FA20 UHPC exhibited an RSPT value of 1016 Coulombs, approximately 6% lower than Type III FA20 UHPC 1079 Coulombs. At 90 days, with Type V cement FA20 UHPC maintaining a lower RSPT value of 971 Coulombs compared to 982 Coulombs in Type III. Similarly, at 28 days, FA15SF5 in Type V achieved an RSPT value of 840 Coulombs, which was approximately 6% lower than its Type III counterpart (894 Coulombs). By 90 days, the difference reduced to 1.5%, with Type V FA15SF5 recording 732 Coulombs in comparison to 743 Coulombs when Type III cement was used. These findings strongly correlate with expansion data, where ternary Type V blends exhibited the least sulfate-induced expansion at 52 weeks due to their refined pore structure. The RSPT results support the conclusion that, despite having lower Blaine fineness, Type V cement provided superior sulfate resistance compared to Type III cement, as evident by its lower sulfate penetration and reduced expansion in sulfate environments. The combination of optimized C_3A content, higher C_4AF, and slower hydration rates also contributed to the enhanced sulfate resistance of Type V cement UHPCs, particularly in the UHPCs incorporating supplementary cementitious materials such as fly ash and silica fume.

4 Conclusions

The UHPCs prepared from Type V cement consistently outperformed those of Type III cement in terms of sulfate-induced expansion and compressive strength. The addition of SCMs further enhanced these properties, leading to improved resilience against external sulfate attack.

1. The impact of cement type on sulfate-induced expansion was noticeably greater than on compressive strength. Type V cement UHPCs consistently exhibited lower sulfate-induced expansion over time, whereas Type III cement UHPCs showed a higher rate of expansion. Although Type III cement UHPCs experienced a reduction in compressive strength relative to Type V UHPCs, this

reduction was marginal in comparison to the pronounced effect on expansion.

2. Independent of cement types and SCMs, the compressive strength of studied UHPCs was barely impacted after one year of exposure to sulfate solution. The control UHPCs experienced the most loss in strength, while those incorporating supplementary cementitious materials (SCMs) showed an overall enhancement in strength due to improved microstructural characteristics. Type V and III cement UHPCs consistently outperformed control and binary UHPCs in terms of absolute compressive strength values but demonstrated a slight strength reduction in terms of percentage comparison under sulfate curing.
3. Regardless of cement types and SCMs, the studied UHPCs exhibited extremely low sulfate-induced expansion. Type V cement UHPCs demonstrated lower sulfate-induced expansion compared to Type III cement UHPCs, with SCMs effectively reducing expansion across both cement types. After 52 weeks of sulfate exposure, Type III cement UHPC showed 20.4 % expansion more than the control Type V UHPC. Similarly, binary FA20 and ternary FA15SF5 Type III cement UHPCs showed higher sulfate-induced expansions compared to their Type V cement counterparts.
4. UHPCs containing supplementary cementitious materials showed a trend of reduced sulfate ion penetrability, particularly in Type V cement. Under both curing ages, Type V C100 UHPC consistently showed lower sulfate ion penetration compared to Type III C100 UHPC. Similarly, both FA20 and FA15SF5 Type V cement UHPCs showed reduced RSPT values relative to their Type III cement counterparts.

Acknowledgement: The authors are grateful to the manufacturers who provided the materials used in this study.

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