



Long-term strength and durability performance of eco-friendly concrete with supplementary cementitious materials

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Abstract

Research has shown that adding supplementary cementitious materials (SCMs), such as fly ash (FA) and slag (SL), to concrete improves its mechanical and durability properties up to certain limits. However, the long-term performance of concrete made with FA and SL is not fully known. This study investigates the impact of FA and SL on the long-term (up to 900 days) performance of concrete. The concrete specimens were made with six replacement percentages (0, 10, 20, 30, 45 and 60 by weight) of ordinary Portland cement (OPC). The short-term fresh and hardened properties of all concrete mixes were assessed after 14, 28, 60, and 90 days of water curing. After 120, 365, 730, and 900 days of water curing, the long-term performance was investigated for 100% OPC (control), 30% FA, and 30% SL concretes. At 28 days, no significant difference in strength development was observed for the concrete mixes containing up to 30% FA and 30% SL than the control concrete (100% OPC). In contrast, a remarkable enhancement in strength development was registered for all mixes containing up to 30% FA and 30% SL at 60 and 90 days of tests. Likewise, 30% FA and 30% SL showed the lowest porosity and water absorption than the control. The mechanical strength of concrete prepared with 30% FA and 30% SL gradually rises over time (from 14 to 900 days) compared to the control concrete. With increasing concrete age, a reduction in porosity and capillary water absorption was seen (up to 900 days).

Keywords Fly ash and slag · Concrete · Mechanical properties · Porosity · Water absorption · Microstructure

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Introduction

The usage of supplementary cementitious materials (SCMs), such as powdered fuel or fly ash, ground granulated blast furnace slag (GGBS), and silica fume, either mixed with Portland cement or blended with it, is of necessity in developing sustainable cement-based materials [1–4]. The addition of SCMs to cement-based composites up to certain thresholds is known to enrich its properties through their pozzolanic reaction and filler effect [5, 6]. These SCMs are the by-products collected directly from industrial wastes or after processing from the agricultural wastes [7–9]. As one of the main components of concrete, ordinary Portland cement (OPC) production accounts for an estimated 5–7% of global CO₂ emissions [9, 10]. The production of OPC involves the clinkering process, where the raw materials (e.g., limestone and clay) are ground into a very fine powder and prepared slurry and heated to very high temperatures in a rotary kiln, resulting in a hard material called clinker, which is ground with gypsum to make OPC. Conversely, the production of the industrial SCMs does not involve any

additional clinkering process like OPC. Thus, a significant amount of CO₂ emission can be cut down when SCMs are partially used to replace cement in concrete production. As a result, the environmental impact of the cement and concrete industries is significantly reduced when clinker cement is blended with SCMs [8].

Globally, between 450 and 530 million tons of fly ash (FA) and GGBS are produced annually, with only 25% of FA and 60% of slag being utilized for various uses [8, 11–13]. Thus, the application of these by-products in concrete can also preserve the landfill site, eliminate the disposal cost and protect the environment. Besides the environmental and economic benefits, the intrinsic physical and chemical properties of various types of SCMs have shown not only the improvement of the engineering properties of concrete, but also promising results in developing concrete for different applications, including energy storage systems [3], concrete structures and infrastructures exposed to fire [14–16].

SCMs like slag and FA are popular in the concrete industry due to good pozzolanic activity, which generates lower heat, better workability, and diminishes the possibility of bleeding and segregation. The spherical FA's ball-bearing effect improves particle packing and creates a dense structure in the concrete [17]. Studies have reported high-volume use of FA for cement in concrete, 50–70%, and at the lower level, up to 30% [18–20]. In some special high-performance materials, such as strain-hardening cementitious composite (SHCC), FA content was used 1.5 times higher than the cement [21]. GGBS is a by-product of iron in blast furnaces, which is rich in lime, silica, and alumina and thus depends on the raw materials used in iron production. Powdered slag is a valuable SCM source in concrete and can also be used up to 60% as a replacement for OPC in concrete production [22].

Numerous types of SCMs have been used in concrete by researchers, focusing on their physical (i.e., size and surface area) and chemical (i.e., oxide and phase compositions) properties and amorphous content. These properties determine the pozzolanic reactivity, interaction with the product of cement hydration, and water demand [23]. For example, high surface area and SCM fineness of silica fume may demand more water for concrete workability. On the other hand, the smooth surface and round shape of FA could enhance better workability. The concrete slump increased as the percentages of FA increased in the concrete mixes, which was 33%, 39%, and 48% for FA content of 50%, 70%, and 90%, respectively [8]. A similar trend was also reported in other studies [6, 11]. With GGBS, the literature shows conflicting reports. At the replacement levels of 50%, 70%, and 90%, the slump of concrete was reported to decrease by 33%, 52%, and 55%, respectively [8]. This reduction was thought to be because GGBS mixes have a low capillary pore volume, which traps water from the mix. This behavior

was inconsistent with other studies where the slump was reported to have increased as the GGBS content increased in the concrete mixes [6, 11]. At 20%, 40%, and 60% of GGBS, the slump increased by 10%, 70%, and 100%, respectively, compared to the control concrete [6, 11]. The authors concluded that the spherical shape of GGBS increases slump because it minimizes friction at the fine aggregates-paste interface. Hence, the physical characteristics of SCMs affect the characteristics of fresh concrete.

Regarding the mechanical strength, it is observed that the compressive strength of concrete at 28 days was 38% and 47% less when 30% and 50% of OPC were substituted by FA, respectively [24]. With the inclusion of 30% and 50% slag in the mix, the reduction in concrete strength was about 13% and 22%, respectively. The decrease in strength for FA mixed concrete was ascribed to the lack of lime content, which reduced the hydration rate [17]. The slower heat of hydration of FA also led to low early strength of concrete. Conversely, with a longer curing age, the ultimate strength of concrete made with FA was relatively higher than the reference concrete. Xu et al. [25] found that the compressive strength of concrete was reduced by 17%, 36%, and 46% when 40%, 55%, and 70% of cement were replaced by FA at 28 days. For the same replacement level for slag, the reduction in strength was 6%, 19%, and 23%, respectively. A similar strength reduction trend was also reported for the splitting tensile strength test of concrete with FA and slag. It was found that the strength development was better for the concrete made with slag compared to FA. The reduction in strength for FA and slag concrete can be attributed to their different degrees of hydration and hydration products' morphology. The scanning electron microscopy (SEM) analysis showed that in FA concrete, the hydration products are villous-like, whereas for slag, they looked like interweaving thick needles, which may be responsible for higher strength [25].

The porosity and absorption (determined by the water sorptivity test) of concrete at different FA and slag contents (0, 30, 50, 70, and 90%) were investigated by Karahan [26]. Both porosity and absorption decreased as the percentages of FA content increased. Compared to the reference concrete, porosity and absorption decreased by a maximum of 4% and 11% in concrete with 50% FA. In the pozzolanic reaction of SCMs, the calcium hydroxide (Ca(OH)₂) is transformed into a secondary calcium silicate hydroxide (C-S-H) gel. This formation of secondary C-S-H gel may likely affect the pore structure by transforming coarser pores into finer ones [6]. The greater the concentration of Ca(OH)₂ in a hydrated matrix, the greater the volume of continuous pores [27].

The durability of concrete in the marine environment is a crucial concern for concrete structures and infrastructures, as concrete structures can easily deteriorate due to physical salt attacks and corrosion of steel bars induced

by chloride penetration from seawater [28]. This ingress of chemicals can dramatically decay the mechanical and durability properties of concrete, especially for a longer period. Chakraborty et al. [29] reported that the compressive strength decreased by 12.4% after exposure to the chloride solution of concrete at the age of 200 days. The decay of compressive strength was explained by the leaching out of Ca^{2+} ions through the formation of the soluble salts associated with the drop in pH caused by the presence of chloride ions. In addition, the volume expansion induced by the building of secondary ettringite and gypsum induced by the sulfate attack decreased the strength. Sičáková et al. [30] performed the compressive strength and water absorption of cement-based composites incorporating different percentages of natural zeolite at 28, 365, 730, and 1095 days. The authors stated that adding natural zeolite enhanced the compressive strength by 16.4% compared to the control concrete. Another study carried out by Sičáková and Špak [31] revealed that the compressive strength augmented by 72.2% and 67.6% at the age of 365 days of the concrete incorporated with recycled concrete and brick powder as micro-filler compared to the control mix. In contrast, flexural strength was enhanced by 44.8% and 65.7% after 365 days of water curing than the control concrete. Menhosh et al. [32] found that compressive strength improved by 16% at 545 days of concrete containing 15% metakaolin compared to the control concrete. Furthermore, the carbonation rate and chemical attack were the lowest compared control mix at 180 days due to the formation of secondary hydrated products caused by pozzolanic reactions between metakaolin and cement hydration products, resulting in dense microstructures.

It has become a convention to include SCMs in concrete to achieve sustainability and lower carbon footprint; therefore, optimum content and physical and chemical properties must be adequately scrutinized. In a recent article, it is acknowledged that information on the durability of high-volume FA concrete is sparse [33]. Several studies

have concentrated on the mechanical and durability performances of concrete using SCMs/high-volume SCMs for up to 90 days. The formation of secondary C-S-H gels and densification of microstructures due to reduced capillary pore volume of concrete is well-understood for the short-term properties of concrete. Also, several studies deal with concrete’s long-term mechanical and durability properties, mainly containing OPC and some filler materials. In contrast, investigations on the long-term properties of concrete made with fly ash and slag as a replacement for OPC are still essential, especially up to 900 days; hence, the behavior at such an extended period is important.

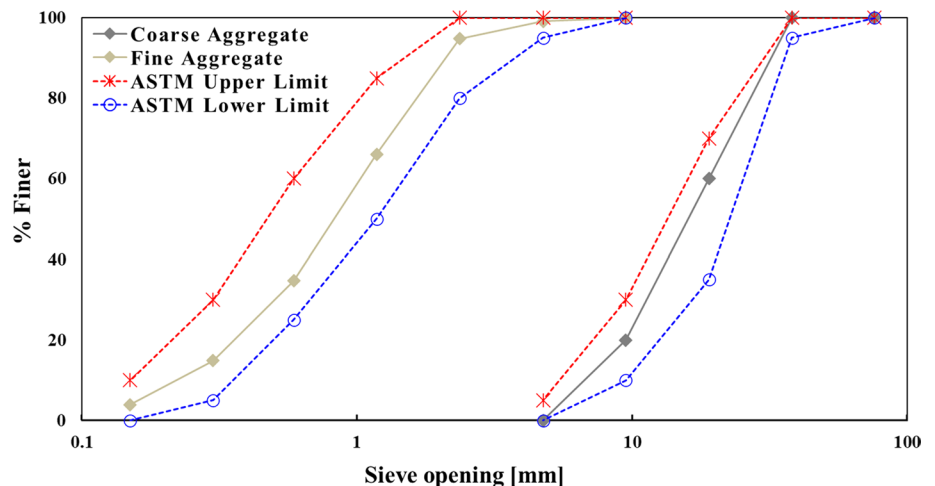
A comprehensive experimental research was carried out to test the fresh, mechanical (compressive and splitting tensile), and durability (porosity and capillary water absorption) properties of concrete by replacing OPC with FA and slag (GGBS) at six replacement contents of 0, 10, 20, 30, 45, and 60% (by wt. of cement). The properties were tested at 14, 28, 60, and 90 days. Thereafter, the long-term performances were investigated at 120, 365, 730, and 900 days on concrete made with 100% OPC (control), 30% FA, and 30% slag since these mixes provide the optimal performances.

Materials and methods

Materials and mixtures

A binder (CEM I 42.5 N, FA and slag-SL), water, natural crushed stone (CS) as coarse aggregate, and natural sand (NS) as fine aggregate were used to produce concrete specimens. The ASTM C136 [34] standard sieves were used to sieve the aggregates, and the limits of the ASTM C33 [35] standard were used to depict the distribution of particle sizes (PSD) in Fig. 1. Per ASTM requirements for coarse and fine aggregates, the physical properties were investigated and presented in Table 1. For coarse aggregate, the specific

Fig. 1 PSD of fine and coarse aggregates and presented them with the ASTM limits provided by the ASTM C33 [35] standard



gravity, unit weight, and absorption capacity were 2.74, 1550 kg/m³, and 1%, respectively, whereas for fine aggregate, the values were 2.56, 1530 kg/m³, and 5.90%. The Los Angeles abrasion resistance of coarse aggregate was 12%.

In contrast, the fineness modulus of coarse and fine aggregates was 6.35 and 2.90, respectively. Figure 2 depicts the SEM examination of the microscopic features of both aggregates. Indeed, the microstructure of the aggregates and surface texture play a key role in developing the strength and durability of concrete. The SEM images of crushed stone and natural sand revealed that they are highly angular with a rough surface texture, which could provide a better bond with the cement mortar. This behavior will eliminate the bond failure around the stone aggregate and offer higher strength due to the contribution of carrying the load by mortar and aggregate, thus ensuring to reach the target strength (more than 40 MPa) of the control concrete.

The OPC, FA, and SL were collected from a local cement company. X-ray fluorescence (XRF) analysis was used to determine the chemical composition of OPC, FA, and SL. The results are shown in Table 2. The results show that the CaO is significantly lower in FA than OPC and SL, which could provide slower heat of hydration of FA. This low CaO content of FA (1.39%, as reported in Table 2) reveals that the FA is in class F, which is pozzolanic, and may have little or

Table 2 Chemical composition of OPC, FA, SL, CS, and NS

Chemical composition	OPC (%)	FA (%)	SL (%)	CS (%)	NS (%)
CaO	65.01	1.39	52.86	12.32	0.85
SiO ₂	21.47	61.14	31.57	50.32	84.56
Al ₂ O ₃	5.03	22.36	8.10	11.81	4.55
Fe ₂ O ₃	3.76	6.61	1.43	13.44	4.15
MgO	0.92	0.76	1.53	5.53	0.37
SO ₃	2.04	0.24	1.96	0.15	–
TiO ₂	–	3.34	1.19	2.02	0.31
K ₂ O	–	2.53	0.40	0.69	4.18
P ₂ O ₅	–	1.08	–	0.29	0.13
Na ₂ O	–	0.19	0.40	3.11	0.82
MnO	–	0.06	0.39	0.17	0.04
ZrO ₂	–	0.12	0.03	–	–
SrO	–	0.06	0.11	0.06	0.02
Cr ₂ O ₃	–	0.07	–	0.07	–

no cementitious properties. This could result in lower early strength of concrete, as reported by Saha [17]. In contrast, the SiO₂ and Al₂O₃ are significantly high in FA than OPC and SL, which agrees with the literature [6, 36]. The chemical composition of natural crushed stone (CS) and NS is also presented in Table 2.

Due to rapid urbanization caused by a higher population compared to the land needed to build houses, especially in Asia, like Bangladesh, the construction of high-rise buildings is growing dramatically. Generally, the commonly used concrete design strength in some Asian countries, such as Bangladesh, is about 20–28 MPa [37], which is not used for high-rise buildings. Therefore, this study attempts to fabricate low-cost and highly durable high-strength concrete (more than 40 MPa, i.e., 6000 psi) using slag (SL) and FA, which can be used in high-rise buildings, satisfies

Table 1 Physical properties of crushed stone (CS) and natural sand (NS)

Properties	Unit	CS	NS
Fineness modulus	–	6.35	2.90
Unit weight (SSD)	kg/m ³	1550	1530
Specific gravity (SSD)	–	2.74	2.56
Absorption capacity	%	1.00	5.90

* SSD: saturated surface dry

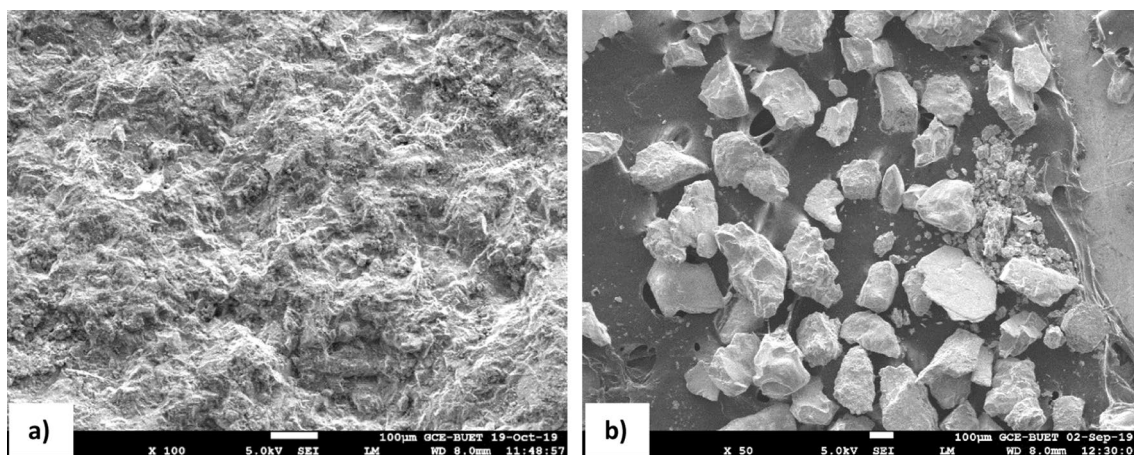


Fig. 2 SEM images of natural crushed stone (a) and natural sand (b)

sustainability, and can contribute to the circular economy. Therefore, the target strength of control concrete at 28 days was 42 MPa (≈ 6000 psi), which was achieved with an OPC content of 500 kg/m^3 after some initial trial mix tests at the laboratory. Studies were conducted on eleven concrete mixtures with a water-to-binder (w/b) ratio of 0.35. Among all mixes, ten mixes (five mixes for both FA and SL) were designed with five different replacement percentages of FA and SL at 10, 20, 30, 45 and 60 by weight of OPC. The mix IDs of concretes containing FA and SL are denoted as 10% FA, 20% FA, 30% FA, 45% FA and 60% FA and 10% SL, 20% SL, 30% SL, 45% SL and 60% SL. In contrast, the control mix was designed with 100% OPC. Although the w/b ratio was low and air voids had to be avoided in the concrete samples, a chemical additive of 0.5% wt. of cement was utilized to improve the workability of fresh concrete. The overview of the mixture proportions of the concrete mixes is given in Table 3. Using the slump test, the workability of the fresh concrete mixtures was determined. Furthermore, the fresh concrete temperature for all mixes was monitored using a digital thermometer, giving an idea of the fresh concrete mixtures' temperature generation (i.e., hydration) for different OPC replacement levels.

Sample preparation and test procedures

Mechanical properties

All concrete mixes were tested for mechanical strength (compressive and splitting tensile) using cylindrical specimens with a 100-mm diameter and a 200-mm height. Three equal layers of fresh concrete were poured into the steel mold, each with 25 tamps. The concrete samples were then given 24 h in the laboratory to harden before being covered with a plastic sheet to prevent water evaporation. Before the age of mechanical testing, the concrete samples were

afterward demolded and cured underwater ($20 \pm 2 \text{ }^\circ\text{C}$). The water curing was carried out for the specimens test at 14, 28, 60, and 90 days. In contrast, after 28 days of water curing, the specimens for the long-term strength and durability tests were kept at the ambient atmospheric temperature in the laboratory and tested at 120, 365, 730, and 900 days. The compressive and splitting tensile strength tests were carried out at 14, 28, 60, and 90 days to investigate the strength development of concrete mixes according to the ASTM C39 [38] and ASTM 496 [39]. The specimens were tested in compression and tension at 120, 365, 730, and 900 days to investigate the role of the FA and SL in the long-term strength of concrete. Since the concrete made with 30% FA and 30% SL provide the best performances, three mixes were investigated for long-term performance: (i) the concrete made with 100% OPC (control), (ii) 30% FA, and (iii) 30% SL. Three samples were evaluated for compressive and splitting tensile strength in each mix and curing age, and the mean of the three samples was used to determine the average strength. Within the scope, 336 cylinder specimens were tested for the mechanical test.

Furthermore, the dry density of hardened concrete specimens was measured on the cylindrical specimens before compressive strength tests by measuring the volume and mass of the specimens. However, to better understand the mechanical performance of concretes, SEM observations were conducted on control, 30% FA, and 30% SL concrete at 730 days to investigate possible cracking at the interface between the cement/mortar matrix and aggregate. JSM-7600F was used to examine the microstructure of concrete samples. All the samples were extracted from the concrete cylinders that failed after the compression test. Then, the surface of the concrete samples was ground with silicon carbide abrasive papers. The successful smooth surface was achieved by polishing using a diamond paste of finer grades. Then, the SEM test was conducted.

Table 3 Mix design of concrete mixes made with different FA and SL percentages (kg/m^3)

Mix ID	OPC	FA	SL	CS	NS	Water
Control	500	0	–	974	745	175
10% FA	450	50	–	974	745	175
20% FA	400	100	–	974	745	175
30% FA	350	150	–	974	745	175
45% FA	275	225	–	974	745	175
60% FA	200	300	–	974	745	175
10% SL	450	–	50	974	745	175
20% SL	400	–	100	974	745	175
30% SL	350	–	150	974	745	175
45% SL	275	–	225	974	745	175
60% SL	200	–	300	974	745	175

Durability of concrete mixes

In order to assess the durability performance of concrete containing FA and SL as a partial replacement for OPC, the porosity and capillary water absorption were examined. The porosity of concrete mixes was performed through water absorption porosity following French standard NFP18-459 [40], while the capillary water absorption tests were conducted as recommended in AFPC-AFREM [41]. The porosity of eleven concrete mixes (control, and 10%, 20%, 30%, 45%, and 60% for both FA and SL) was carried out at 14, 28, 60, and 90 days, while the porosity at the more extended period was investigated at 120, 365, 730, and 900 days on concrete made with 100% OPC (control), 30% FA, and 30% SL. In contrast, capillary water absorption tests were performed at 90 days for all eleven mixes and at 730 days only for concrete made with control, 30% FA, and 30% SL. For each mix and curing age in both tests, three specimens were utilized to check the repeatability of the tests.

Experimental results and discussion

Fresh properties of concrete mixes

The workability of eleven concrete mixes is presented in Fig. 3. It has been demonstrated that adding FA and SL has a considerable impact on the workability of fresh concrete. The workability of fresh concrete increases as the percentage of FA and SL increases, and it was more pronounced for the higher FA and SL content. The slump of concrete mixes prepared with FA and SL was 10%–37% and 5%–32% higher than the control mix, respectively. It implies that the incorporation of FA and SL reduced the water demand for concrete and enhanced the workability of the fresh concrete.

These higher slump values in the mixes made with FA are linked with the smooth surface area and spherical shape

of FA particles [6], which lends a ball-bearing type effect on the concrete mixture. Likewise, the higher slump values of SL mixes could be due to a smooth and dense surface and better binding particle dispersion of SL than OPC [6, 11]. Indeed, a higher slump of the mix provides higher pumpability and compactability, which leads to lower voids in the concrete specimens and higher strength and durability. However, the fresh concrete temperature during placing could influence the slump values of concrete mixes. The control concrete was found to have a higher temperature than the concrete mixes prepared with FA and SL (Fig. 3). A decrease in the temperature of the fresh concrete was noticed as the percentage of FA and SL was increased due to the lower cumulative heat of hydration (i.e., heat evolution) of SCMs. It is also reported that the thermal evolution of concrete containing FA decreased dramatically with the percentages of FA increased in the mix [42, 43]. This lower heat evolution can significantly reduce the thermal stress and thermal cracking of the concrete and reveal that the FA and SL can potentially eliminate the thermal cracking in mass concrete production. Hence, this higher temperature of control concrete (made with 100% OPC) could accelerate the hydration of the cement paste, thus reducing the workability of freshly mixed control concrete.

Dry density of hardened concrete

Table 4 presents the average dry density of eleven concrete mixtures, and the normalized dry density of hardened concrete mixes measured at different ages is reported in Fig. 4. It was discovered that the dry density of concrete mixtures rose by up to 45% when FA and SL were used to replace OPC. The concrete prepared with 30% FA and 30% SL has a density that was approximately 9% and 11% more than the control concrete (Fig. 4). Contrarily, the density of the concrete prepared with 60% FA and 60% SL compared to the control mix was largely similar or slightly decreased.

Fig. 3 Slump and temperature of fresh concrete made with different percentages of FA and SL

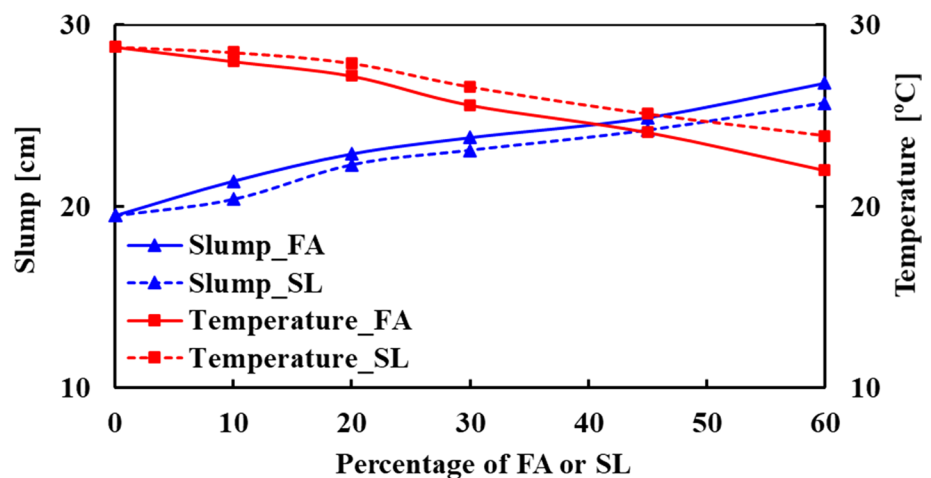
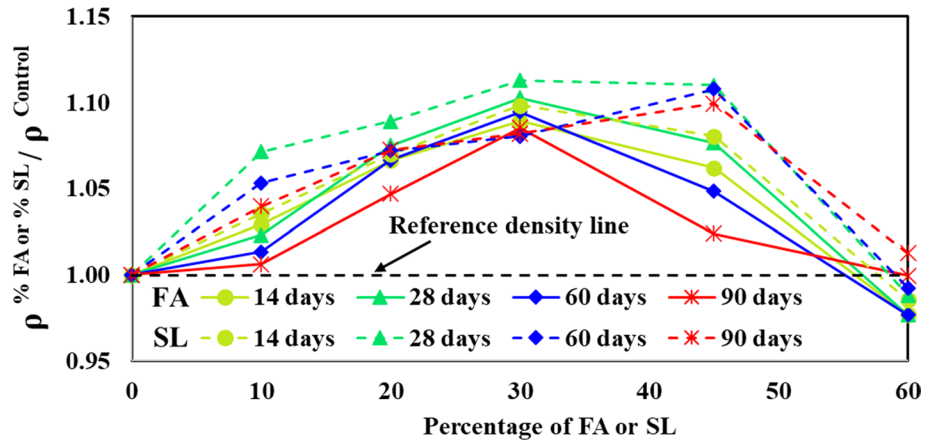


Table 4 Average dry density (ρ) of concrete mixes with different FA and SL percentages

		Control	10%	20%	30%	45%	60%
14 days	ρ [Kg/m ³] of FA	2063	2123	2199	2246	2191	2015
	ρ [Kg/m ³] of SL	2063	2137	2207	2266	2229	2033
28 days	ρ [Kg/m ³] of FA	2218	2269	2385	2446	2388	2167
	ρ [Kg/m ³] of SL	2218	2377	2416	2469	2462	2192
60 days	ρ [Kg/m ³] of FA	2318	2349	2472	2537	2430	2264
	ρ [Kg/m ³] of SL	2318	2441	2485	2504	2568	2300
90 days	ρ [Kg/m ³] of FA	2392	2406	2504	2596	2448	2391
	ρ [Kg/m ³] of SL	2392	2487	2565	2588	2629	2422

Fig. 4 Normalized dry density (ρ) of hardened concrete mixes measured at different ages



The higher density of concrete made with FA and SL could be linked to the filling of the micropores due to the smaller particle size of FA and SL than the clinker (OPC), which significantly reduces the pore size and provides a dense matrix. This could also be explained by the higher workability of fresh concrete containing FA and SL than OPC, thus minimizing/removing the air voids due to its better compacting. Further, C-S-H gels are produced by the pozzolanic reaction of FA and SL when they interact with calcium hydroxide (Ca(OH)₂). These new extra C-S-H gels would fill the micropores (dense microstructure and lower porosity), increasing density.

In contrast, the increase in density of concrete containing 60% SL and FA compared to the other mixes could be due to significantly higher porosity and capillary water absorption induced by higher voids and well-connected pores in the matrix and lower mechanical strength caused by the formation of lower C-S-H gels. These properties offer poor microstructures, resulting in a decrease in the density of concrete. The evidence of higher porosity and water absorption and poor mechanical strength of concrete containing 60% SL and FA were observed and discussed in Sects. “Mechanical properties” and “Durability of concrete mixes.”

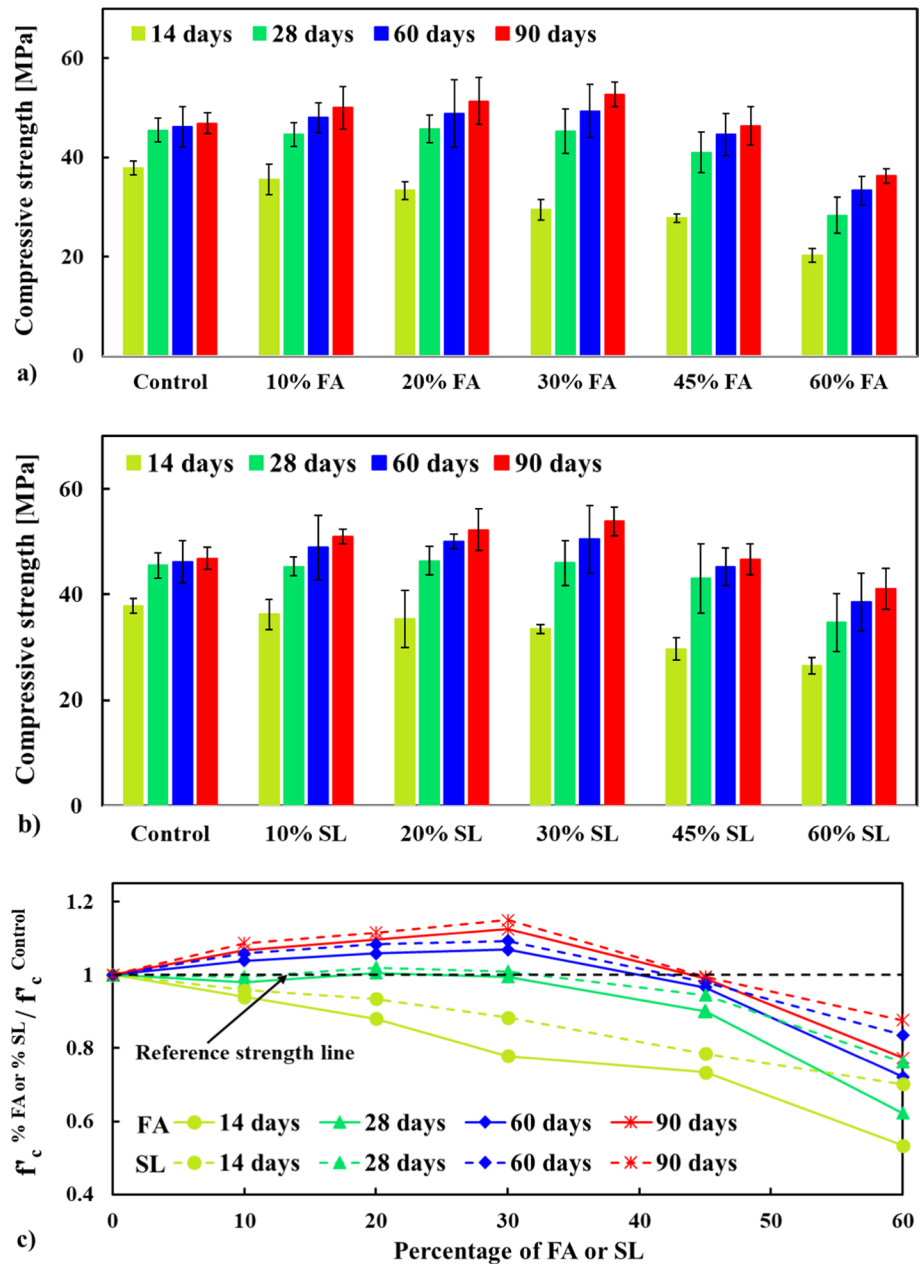
Mechanical properties

Compressive strength of concrete mixes

Figure 5a and 5b shows the average compressive strength of concrete mixes incorporating FA and SL that were tested at 14, 28, 60, and 90 days, whereas Fig. 5c displays the normalized compressive strength (i.e., $f_c^{\text{FA or SL}} / f_c^{\text{Control}}$). The schematic diagram of the cross sections of concrete mixes made with OPC and FA/SL is presented in Fig. 6a and b, while the effect of cement hydration, micro-filler, and pozzolanic effect on the strength development of concrete mainly due to FL/SL is reported in Fig. 6c. The target strength of control concrete at 28 days was 42 MPa, which has been achieved, as shown in Fig. 5a. Compressive strength was unchanged in control concrete up to 30% FA and 30% SL at 28 days, but at the age of 14, it decreased as the proportion of FA and SL increased; after that, the strength decreased beyond 30% of FA and SL. At an early age, the lower strength development could be due to the slower rate of pozzolanic reaction within FA/SL and lime produced by OPC hydration [6].

The results of 30% FA and 30% SL at 28 days reveal that it can save the cost of producing 30% clinker (i.e., economic) and sustainable green construction material since producing

Fig. 5 Compressive strength (f'_c) of concrete mixes made with different replacement percentages of OPC by fly ash (a) and slag (b), and normalized f'_c performed at 14, 28, 60, and 90 days, respectively



a clinker produces a significant amount of CO₂ (environmental damage and pollution are minimized). Therefore, our findings suggest that a 30% OPC replacement by FA and SL is the optimal amount, which is consistent with the conclusion made by Johari et al. [6].

While in a more extended curing period (60 and 90 days), a significantly higher compressive strength was observed for concrete made with 30% FA and 30% SL than for control concrete. At 60 days, the control concrete and those with 30% FA and 30% SL have compressive strengths of 46.19 MPa, 49.35 MPa, and 50.48 MPa, respectively. At 90 days, those values became 46.86 MPa, 52.74 MPa, and 53.87 MPa. In 60 and 90 days, the enhanced strength was

around 7–9% and 13–15% higher than the control concrete. The combination of micro-filler effect and extra C-S-H gels formed by the pozzolanic reaction of FA and SL may be responsible for the higher strength of concrete made with these two materials (Fig. 6). The finer particles of FA and SL would fill the microvoids (decreasing porosity, as mentioned in Section “Porosity”), resulting in a denser microstructure and an improved interfacial transition zone (ITZ) between the cement paste and the aggregate. This leads to higher compressive strength since the failure path occurred through the aggregate and the mortar (i.e., combined failure, Fig. 6b).

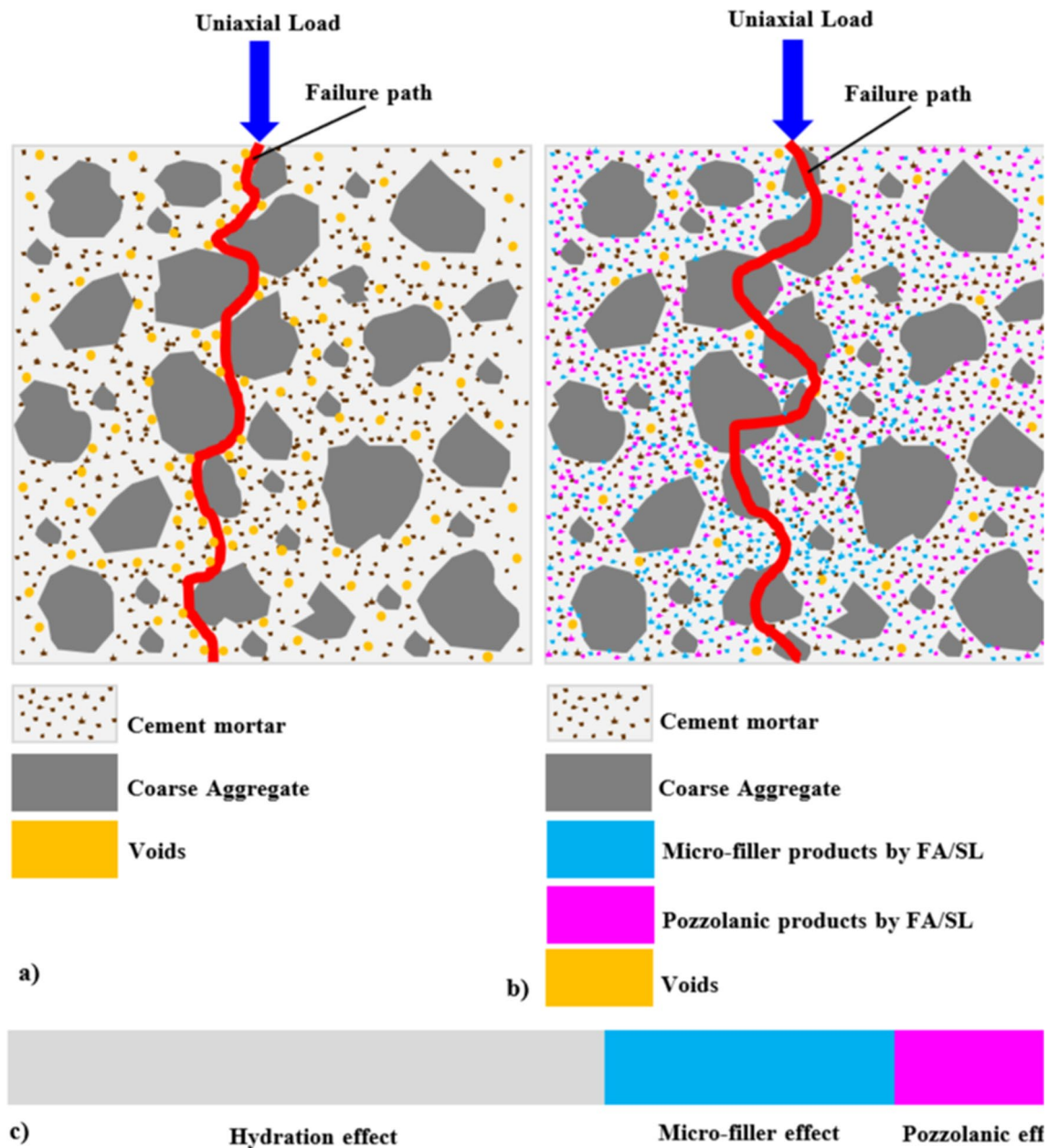
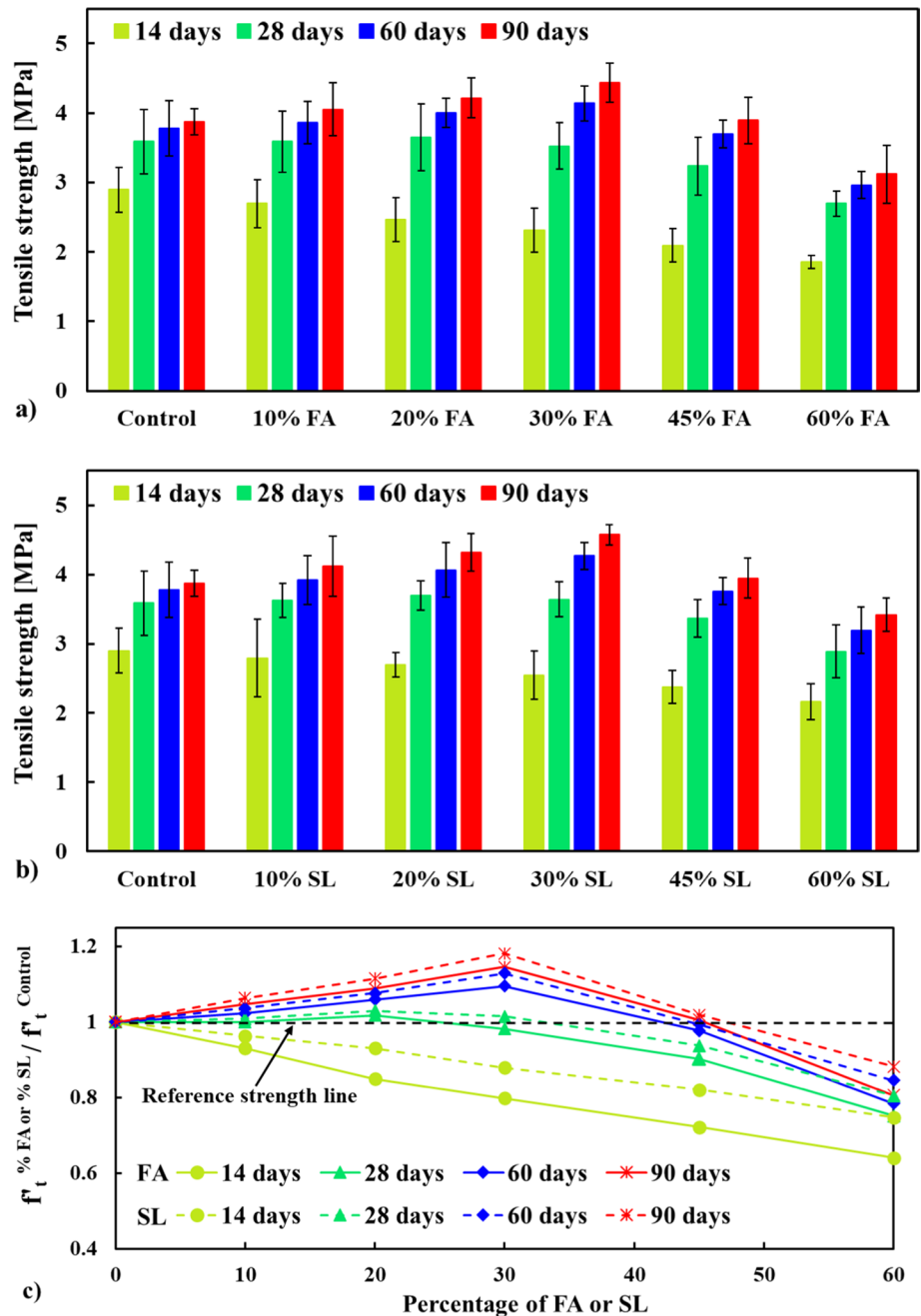


Fig. 6 Schematic diagram of the cross sections of concrete mixes made with OPC (a) and FA/SL (b), and the effect of cement hydration, micro-filler, and pozzolanic effect on the strength development of concrete (c), respectively (dimensions of the objects in the figures are not actual)

In reality, the ITZ is the most vulnerable path for concrete failure under mechanical loading. The lower strength of control concrete could be owing to its larger porosity compared to the concrete made with FA and SL (described in Section “Porosity”), which provides a weak ITZ, and the failure path would occur at the interface of the ITZ and cement paste (Fig. 6a). Nonetheless, at an extended curing period, the FA and SL would react with the hydrated products of clinker ($\text{Ca}(\text{OH})_2$) in the presence of water

and would produce additional C–S–H gels [6] (Fig. 6b, reducing porosity, resulting in stronger ITZ), which contribute to the improvement of the mechanical strength of the concrete mixes as it is considered the primary source of the strength-giving compound in cementitious materials. While these additional C–S–H gels would not occur in the control concrete, except for the hydration reaction (Fig. 6c) of clinker, resulting in higher porosity, weaker ITZ around the aggregates leads to a lower mechanical strength.

Fig. 7 Splitting tensile strength (f_t') of concrete mixes made with different replacement percentages of OPC by fly ash (a) and slag (b), and normalized f_t' tested at 14, 28, 60, and 90 days, respectively



Tensile strength of concrete mixes

The splitting tensile strength of eleven concrete mixes is presented in Fig. 7a–b, and the normalized tensile strength (i.e., $f_t'^{\%FA\text{or}\%SL} / f_t'^{\text{Control}}$) is depicted in Fig. 7c. As seen in compressive strength tests, at 14 days, a decreasing tensile strength trend was observed for the increasing percentage of FA and SL. A maximum decrease was observed for 60% FA and 60% SL concrete, about 36% and 25%, respectively, with lower tensile strength than the control concrete. The results are consistent with the compressive strength test results at 28 days, and the tensile strength

of the concrete prepared by FA and SL with up to 30% replacement OPC is remarkably similar to the control concrete.

Conversely, for 60 and 90 days, an increase in tensile strength was seen up to 30% replacement of OPC by FA and SL, and after that point, the tensile strength declined. At 60 days and 90 days, the average tensile strengths of the control, 30% FA, and 30% SL concretes were 3.78 MPa, 4.14 MPa, and 4.27 MPa and 3.78 MPa, 4.44 MPa, and 4.57 MPa, respectively. Increases in tensile strength of roughly 10–13% and 15–18% over control concrete are observed after 60 and 90 days, respectively.

It was found that the inclusion of FA and SL in OPC as a binder exhibited a higher increase in tensile strength than the compressive strength in all curing ages. As previously discussed, the reason behind such an increment in strength is directly associated with the combined micro-filler effect and formation of additional C-S-H gels by the pozzolanic reaction of FA and SL (Fig. 6). In more explicitly, the smaller particle size of FA and SL than clinker (OPC) would fill the micropores in the concrete matrix (amount of void is reduced, i.e., lower porosity, Fig. 6b and Section “Porosity”), thus provide dense microstructure and better bonding with cement paste around aggregates (i.e., stronger ITZ). Meanwhile, the pozzolanic interaction between FA and SL can

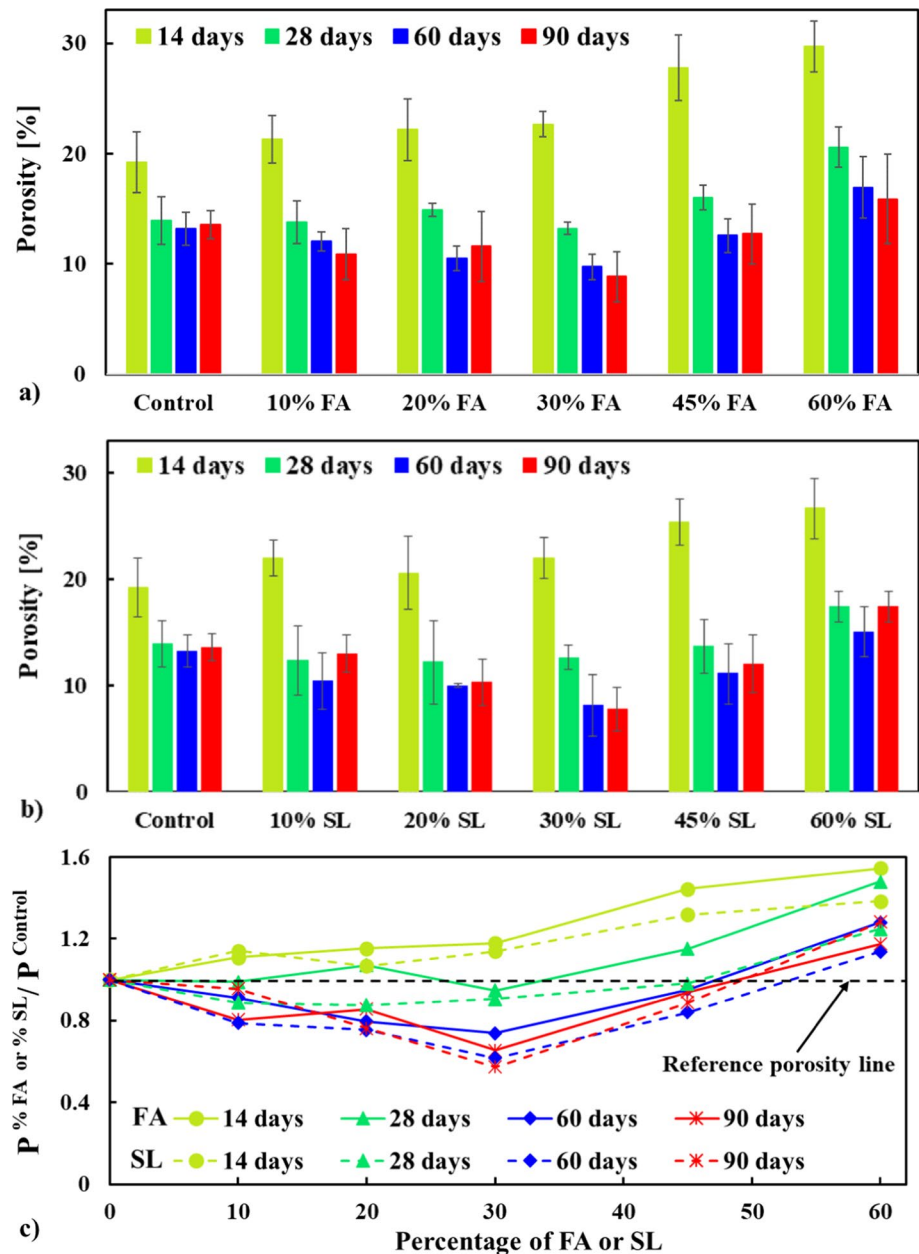
produce further strength-giving C-S-H gel, filling the pores, densifying the microstructure of concrete, and ultimately increasing its tensile strength (Fig. 6b).

Durability of concrete mixes

Porosity

Eleven different concrete mixtures were examined for total porosity at 14, 28, 60, and 90 days of curing (Fig. 8a–b). The experimental results demonstrate that the proportion of FA and SL tested at 14 days enhances the porosity of concrete. With OPC replacement, porosity was reduced up to 30%

Fig. 8 Apparent porosity (P) of concrete mixed made with different replacement percentages of OPC by FA (a) and SL (b), and normalized P tested at 14, 28, 60, and 90 days, respectively



and increased above that after 28 days. The compressive and tensile strengths of the identical concrete mixtures exhibit a similar pattern as the porosity (Figs. 5 and 7). Indeed, concrete porosity increases when mechanical strength (compressive and tensile) declines. This behavior was more evident at the more extended curing for the concrete made with up to 30% replacement of OPC by both FA and SL, i.e., the porosity was decreased up to 30% replacement and following increased with the increased replacement level. At 60 days, the average porosity of the control, 30% FA, and 30% SL concrete was 13.22%, 9.75%, and 8.16%, and at 90 days, it was 13.54%, 8.87%, and 7.82%, respectively. Compared to the control concrete, the decrease in porosity for concrete prepared with 30% FA and 30% SL was approximately 34% and 43% (Fig. 8c), respectively. As indicated in Section 4.3, the reduction in porosity of concrete prepared with FA and SL during a longer curing period might be attributed to a greater reduction in micropores (Fig. 6b) due to the finer particle size of FA than clinker.

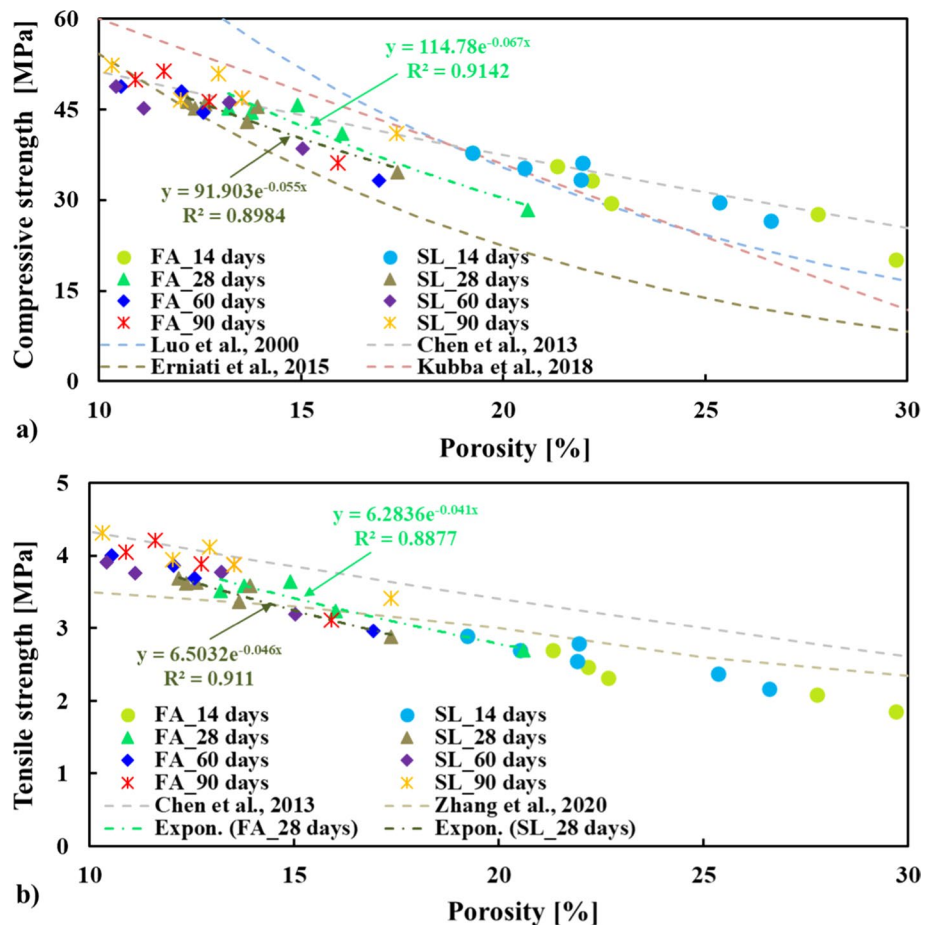
Further fill-up of the microvoids could be occurred by the additional C-S-H gels produced by the pozzolanic reaction (lower connectivity of pores, resulting in lower permeability), thus leading to lower porosity of the concrete mixes.

Indeed, permeability directly links with porosity [44, 45]. Therefore, the lower porosity and better ITZ of concrete made with FA and SL could provide lower permeability. Perhaps, this is why the concrete made with 30% FA and 30% SL shows lower water absorption than control concrete (discussed in Section “Capillary water absorption”), resulting in a lower risk of corrosion and higher durability.

Porosity is closely related to the mechanical strength of quasi-brittle materials such as concrete and mortar [46]. Hence, an attempt has been made to comprehend the relationship between the porosity and strength of concrete mixtures made with FA and SL, which is not well-known. The strength and porosity relation is shown in Fig. 9, and the results were plotted with relationships available in the literature [47–51]. It has been demonstrated that the compressive and tensile strengths of concrete decrease with increasing porosity. The relationship between concrete strength and porosity was significantly linear, and the pattern was consistent with the relationships described in the literature (Fig. 9).

Equations (Eqs. 1–2) are proposed using the best-fitted exponential curves to compute the compressive and tensile strength of concrete containing fly ash and slag at 28 days once the porosity is known or vice versa.

Fig. 9 Relation between porosity and compressive strength (a) and splitting tensile strength (b) of concrete mixes, and plotted with different relationships available in the literature [45–50]



$$f'_c = 114.78e^{-0.067 * P}, \quad R^2 = 0.9142 \quad (\text{Compressive strength at 28 days}) \tag{1}$$

$$f'_t = 6.2836e^{-0.041 * P}, \quad R^2 = 0.8877 \quad (\text{Tensile strength at 28 days}) \tag{2}$$

where f'_c is compressive strength, f'_t is tensile strength, and P is porosity.

Capillary water absorption

At 90 days, the influence of FA and SL on the capillary water absorption of concrete mixtures was evaluated. The outcomes are depicted in Fig. 10. For up to 8 h, the water absorption curves of all concrete mixtures exhibited a steep slope. The water absorption increased with time, followed by a steady absorption slope until around 216 min. At around 300 h, the slope of the curves becomes almost zero (i.e., nearly saturation). It was noticed that the water absorption was decreased up to 30% replacement levels of OPC and increased at the higher replacement level. This water absorption trend agrees with the porosity of the concrete (Fig. 8) and compressive and tensile strength (Figs. 5 and 7). The average water absorption measured at 300 h of concrete made with 100% OPC, 30% FA, and 30% SL was 8.41 kg/m², 6.30 kg/m² and 6.60 kg/m², respectively, which

decreased by about 25.11% for 30% FA and 21.52% for 30% SL than the control concrete. As early mentioned, this could be attributed to the significantly lower porosity (i.e., lower volume of permeable voids for 30% FA and 30% SL concrete than the control one), denser microstructure in the concrete matrix, and strong ITZ of concrete made with 30% FA and 30% SL than control concrete, which prevent/slow down the migration of water through the concrete specimens. This result reveals that the concrete made with 30% FA and 30% SL would provide better protection against steel bar corrosion, leading to higher durability of concrete. It is noted that above 30% replacement of OPC by FA and SL increased the capillary water absorption of concretes, which is consistent with the porosity and mechanical strength of concretes.

Long-term performances of concrete mixes

Mechanical strength

Since the concrete made with 30% FA and 30% SL provides the best performance, the long-term strength was investigated at 120, 365, 730, and 900 days, and the results were compared with the control. The compressive and tensile strength of control concrete, 30% FA and 30% SL, are presented in Fig. 11. It is clearly shown that as the age of

Fig. 10 Capillary water absorption of concrete mixed made with different replacement percentages of OPC by fly ash (a) and slag (b) measured at 90 days, respectively

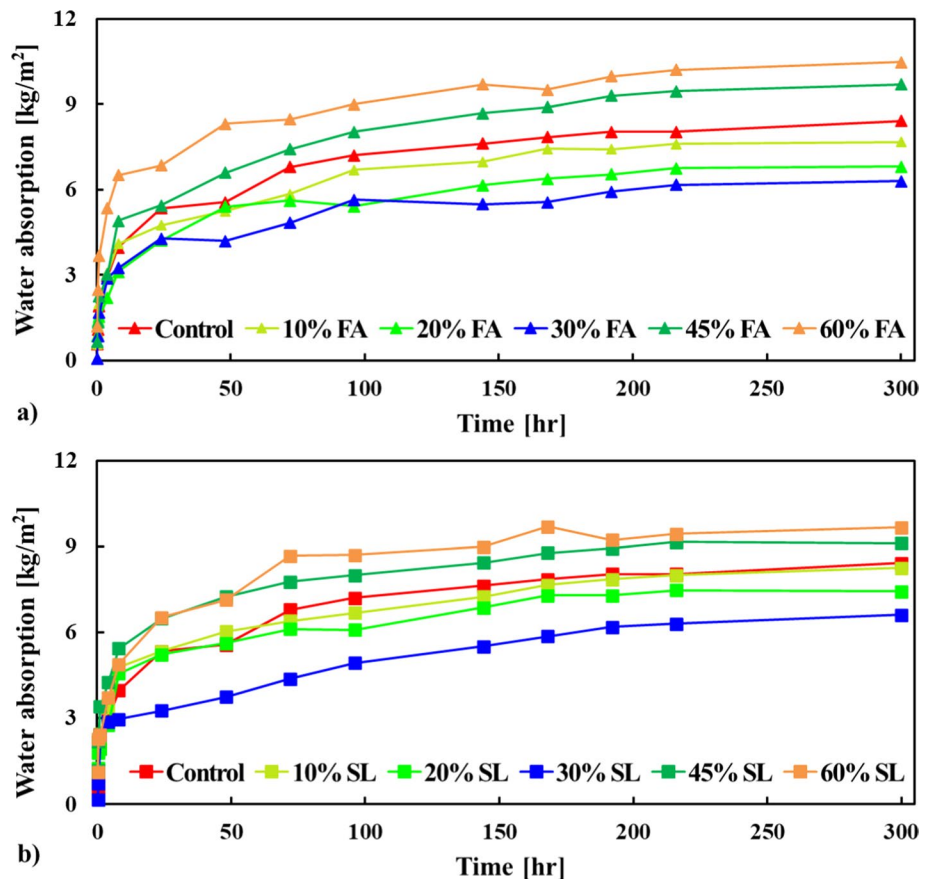
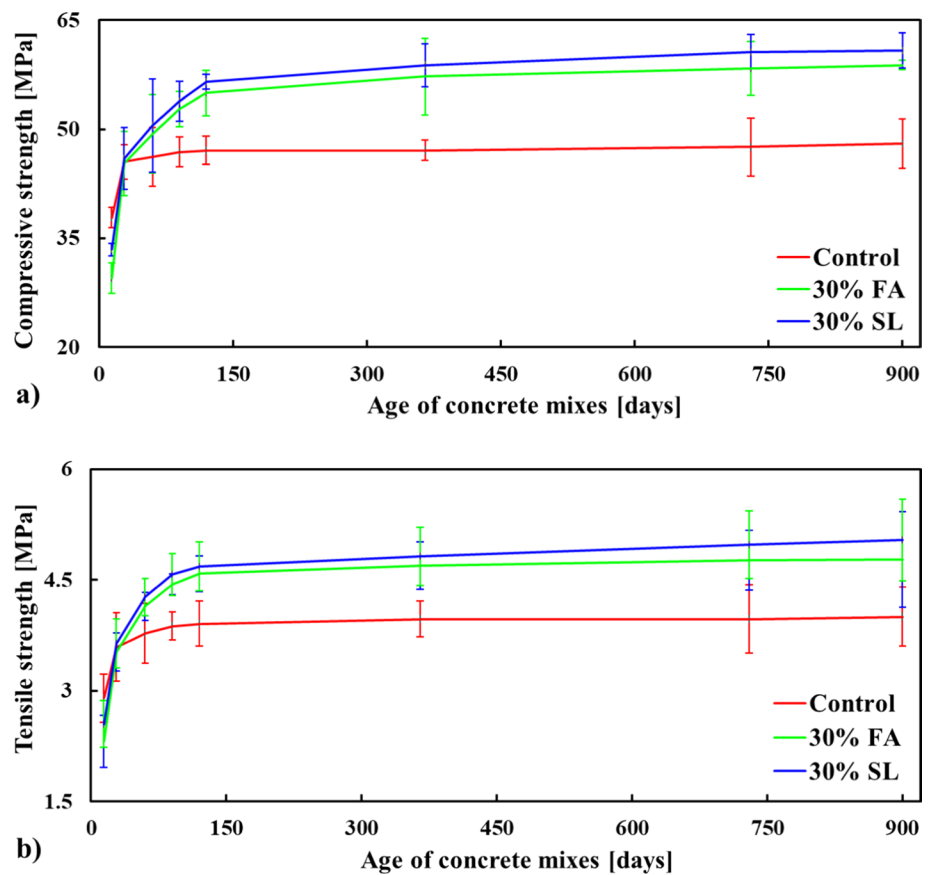


Fig. 11 Long-term compressive and splitting tensile strength of concrete mixes made with 100% OPC (control), 30% FA, and 30% SL performed for up to 900 days



concrete increased from 14 to 900 days, the compressive and tensile strength of concrete made with 30% FA and 30% SL increased, while almost no increment was observed for the control concrete. The average strength of the control concrete, 30% FA, and 30% SL tested at 900 days was 48.0 MPa, 58.80 MPa, and 60.90 MPa, respectively, for compressive strength (Fig. 11a), and 4.0 MPa, 4.77 MPa, and 5.04 MPa, accordingly, for the tensile strength (Fig. 11b). The increase in strength was 23% for FA and 27% for SL for compressive strength and 19% for FA, and 26% for SL for tensile strength than control concrete. This higher strength of concrete made with 30% FA and 30% SL is attributed to the micro-filler effect (lower porosity and permeability) and the evidence of continued pozzolanic reaction in the mixes. As shown in Fig. 6, the micropores are filled due to the significantly lower particle size of FA and SL compared to the clinker (OPC), providing a denser microstructure.

Because of the reduced particle size, FA and SL have enough specific surface area to support pozzolanic reactions. At an extended curing period, in the presence of water, the hydrated clinker products (calcium hydroxide, $\text{Ca}(\text{OH})_2$) combine with FA and SL to generate secondary C-S-H gels ($\text{Ca}(\text{OH})_2 + \text{FA/SL} + \text{H}_2\text{O} = \text{C-S-H gel}$). This higher C-S-H gel content fills the microvoids (dense microstructure) and densifies the ITZ, resulting in higher mechanical strength

than control concrete. However, this pozzolanic reaction produces new C-S-H gels and reduces the $\text{Ca}(\text{OH})_2$ content in the mix, which is more porous (i.e., reduces the volume of continuous pores) and useless material from the point of view of mechanical strength and durability. Indeed, the higher the content of $\text{Ca}(\text{OH})_2$ available in the mix, the higher the chances of forming calcium carbonate (CaCO_3 , i.e., carbonation of concrete), resulting in lower durability [52]. It was noticed that the concrete containing SL has a bit higher mechanical strength at all curing ages than the concrete containing FA. This lower strength of concrete made with FA could be due to significantly lower calcium oxide content (CaO : 1.39% for FA and 52.86% for SL, Table 2) compared to SL, which reduced the hydration rate and strength-giving product, especially at an early age [16].

Scanning electron microscopy (SEM) observations were conducted at the interface between cement paste and aggregates (i.e., at ITZ). The SEM images show that the control concrete has more precise and large cracks through the cement paste and ITZ (Fig. 12a). This is because of the higher porosity in the cement paste (coarser particle than FA and SL) and ITZ of control concrete, i.e., weak ITZ, which induces higher cracks. These higher and thicker cracks should be the main reason for the lower mechanical strength of control concrete. This is

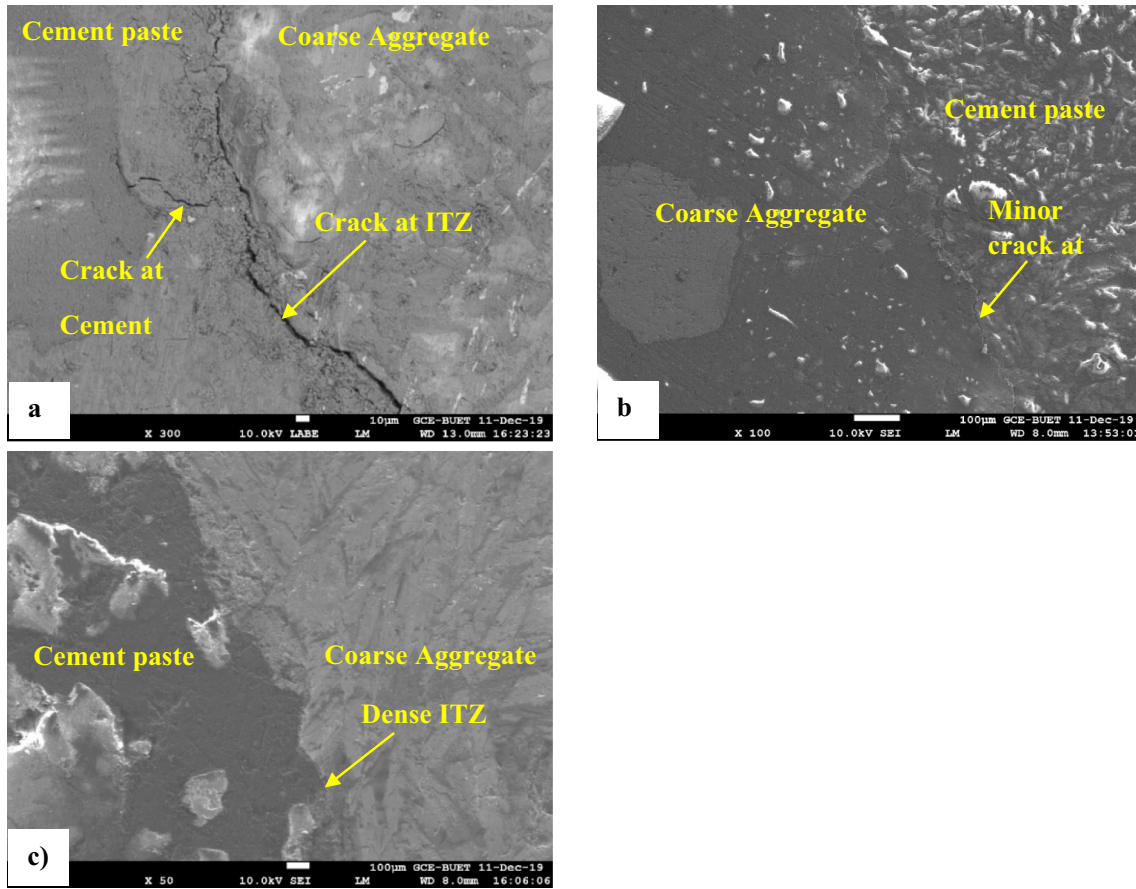


Fig. 12 SEM observations at the interface between cement paste and aggregate: **a** 100% OPC, **b** 30% fly ash, and **c** 30% slag, respectively, tested at 730 days

consistent with the increased porosity and water absorption (Sects. “Durability of Concrete mixes” and 3.5.2) of this concrete compared to 30% FA and 30% SL concrete.

On the contrary, a dense ITZ was observed for both concretes made with 30% FA and 30% SL, especially for 30% SL concrete (Fig. 12b, c). This is due to the decreased porosity in the cement paste and ITZ of FA and SL concrete, which is a result of the micro-filler effect and pozzolanic reaction that improves the microstructure of the concrete by creating secondary C-S-H gel (reducing the pore size and distribution and also reduces the microcracks at the transition zone, i.e., lower permeability). As a result, higher mechanical resistance of ITZ thus led to higher mechanical strength.

Durability

The porosity test was conducted at 120, 365, 730, and 900 days to investigate the durability of control concrete, 30% FA, and 30% SL concrete, while the water absorption test was performed at 730 days. The results are presented in Figs. 13 and 14. The porosity of concrete made with 30% FA and 30% SL was found to decrease as the curing ages increased from 14 to 900 days. In contrast, almost no significant decrease in porosity was observed for the control concrete from 90 to 900 days.

The average porosity of control concrete, 30% FA and 30% SL concrete tested at 900 days were 13.51%, 7.62%, and 6.92% (Fig. 13), respectively, which decreased about 44% for FA and 49% for SL than the control concrete. Likewise, a decreasing trend of water absorption was observed for all mixes as the curing age increased. It should be noted that 30% FA and 30% SL concrete absorbed significantly less

Fig. 13 Long-term porosity of concrete mixes made with 100% OPC (control), 30% FA, and 30% SL performed up to 900 days

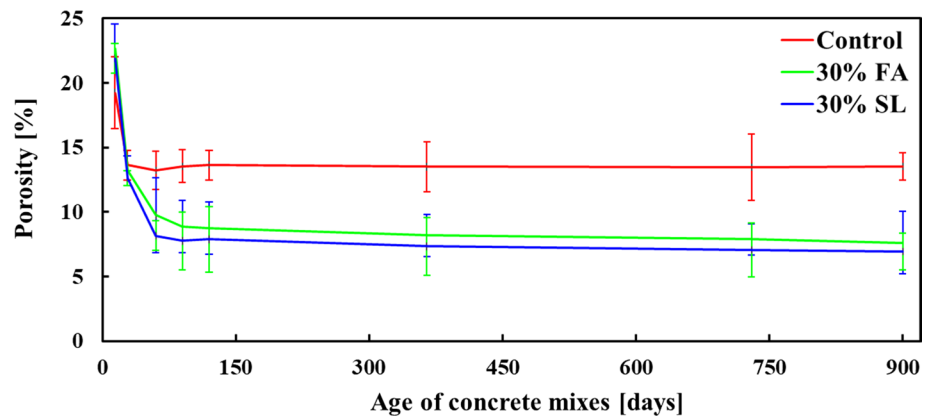
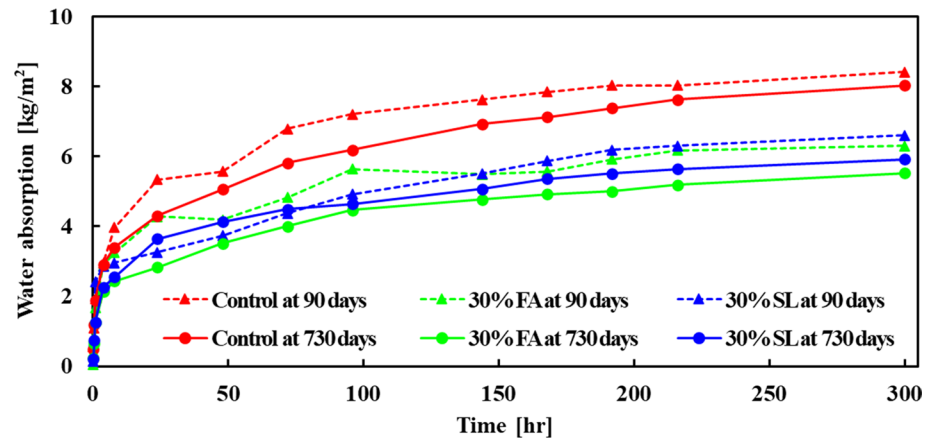


Fig. 14 Capillary water absorption of concrete mixed made with 100% OPC (control), 30% FA, and 30% SL performed at 90 days and 730 days



water than the control concrete. This trend in water absorption is consistent with the porosity test results. The average water absorption of 730-day-old control concrete, 30% FA, and 30% SL concrete measured at 300 h was 8.04 kg/m², 5.53 kg/m², and 5.92 kg/m², respectively, which decreased by about 31.22% for 30% FA and 26.29% for 30% SL than the control concrete. This is mainly due to the fill-up of the microvoids and strengthening of the ITZ (lower porosity and permeability) due to the formation of secondary C-S-H gels induced by the pozzolanic reaction in FA and SL concrete, which does not occur for control concrete. This lower porosity and water absorption of concrete made with FA and SL ensure its long-term durability, which is a crucial concern for structures and infrastructures.

Conclusions

This study studied the effects of including FA and SL in OPC on the long-term mechanical and durability performance of concrete (up to 900 days). The concrete specimens were prepared with six different replacement percentages (0, 10, 20, 30, 45, and 60 by weight) of OPC by both FA and SL. The workability, mechanical strength (compressive and

splitting tensile), and durability (porosity and capillary water absorption) were performed at 14, 28, 60, and 90 days for all mixes. The long-term performances were investigated at 120, 365, 730, and 900 days on concrete made with 100% OPC (control), 30% FA, and 30% SL since this percentage provides the best performances. The outcome of this research are summarized as follows:

- i. A significantly higher slump was observed for the concrete made with FA and SL than the control, which increased as the content of FA and SL increased.
- ii. The dry density increased up to 45% replacement of OPC by both FA and SL, and beyond that replacement, the density decreased.
- iii. At 14 days, the mechanical strength (compressive and tensile) of concrete made with FA and SL was lower than the control concrete, while at 28 days, no strength difference was observed, up to 30% replacement of OPC by FA and SL. Conversely, concrete prepared with 30% FA and 30% SL exhibited a significantly better mechanical strength than control concrete throughout a longer curing period (at 60 and 90 days).
- iv. The concrete prepared with 30% FA and 30% SL had much less porosity and capillary water absorption

than the control concrete, which is in accord with the mechanical strength of these concretes.

- v. As the mechanical strength (compressive and tensile) of the concrete decreases, the porosity increases, which is in good agreement with the literature, and the relationship is quite linear.
- vi. It was found that the strength of concrete produced with 30% FA and 30% SL increased as the age of the concrete increased from 14 to 900 days, whereas nearly no significant change in strength was detected for control concrete. The SEM observation confirmed this, which shows a dense ITZ for concrete made with 30% FA and 30% SL, while large and thick cracks were observed through the cement paste and ITZ of control concrete.
- vii. The concrete prepared with 30% FA and 30% SL had a constant decrease in porosity and capillary water absorption over a longer length of time (up to 900 days), which is consistent with the concrete's mechanical strength at these ages.

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Declarations

Conflicts of interest The authors declare no conflict of interest.

Ethical approval This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent For this type of study, formal consent is not required.

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