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Concrete Trials 3

CCUS Innovation 2.0

Key Knowledge Deliverable 3.4

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Key Knowledge Deliverable Cover Sheet

This Key Knowledge Deliverable (KKD) has been produced by Imperial College London as part of the Department for Energy Security and Net Zero £1bn Net Zero Innovation Portfolio (NZIP) - CCUS Innovation 2.0 programme. The document is reflective of the status of the project at the time of writing. The material presented could have been subject to change as the project matured. These documents should not be considered a full representation of the final project.

Project Description

This project seeks to further develop and scale a new carbon sequestration process which transforms waste CO_2 gas from industrial facilities into valuable construction products. Sequestered CO_2 through this process is cheaper than conventional approaches that rely on purification, liquification and offshore or geological storage. The CO_2 is stored in the form of a stable mineral which ensures they will be no leakage over time.

The patent-pending technology involves taking globally abundant magnesium silicate minerals and splitting this into magnesia and silica components. Through simple chemical processing two products of high purity are created: a) an amorphous silica that can be used as supplementary cementitious material (SCM) to facilitate low-carbon concrete and b) a concentrated magnesium solution in which CO_2 from industrial flues can be sequestered to produce other construction materials.

This CCUS Innovation 2.0 award will be used to increase our technology and commercial readiness level by de-risking and facilitating the development of a pilot facility, in order to demonstrate that the technology is economically viable and deployable at scale.

Description of KKD

The KKD reports on the durability of concrete incorporating amorphous precipitated silica (APS) derived from olivine as a supplementary cementitious material (SCM). Four concrete mixes were prepared including a control mix made of normal concrete (CO), a fly ash-blended mix (CFA), a blended APS concrete mix (C20), and a ternary blend with APS and limestone (CLS). Mixes were tested for workability, compressive strength, water absorption, sorptivity, electrical resistivity, chloride permeability and carbonation. Results indicate that APS contributes to improved durability by reducing permeability and enhancing pore structure refinement. The work shows that APS is a promising SCM for producing durable, low-carbon concrete, suitable for general construction while contributing to environmental sustainability.

KKDs to be released in full

- D3.4 – Concrete Trials 3
- D4.4 – Product Optimisation 2

KKDs to be released after redactions

- D1.1 – Flue Gas Recovery and Testing 1
- D1.2 – Dissolution Procurement
- D1.3 – Dissolution Operation
- D1.4 – Flue Gas Recovery and Testing 2 & Carbonation Procurement
- D1.5 – Carbonation Operation
- D2.3 – Reagent Regeneration Procurement
- D2.4 – Reagent Regeneration Operation
- D3.2 – Concrete Trials 1
- D3.3 – Concrete Trials 2
- D4.2 – Process Optimisation
- D4.3 – Product Optimisation 1
- D5.2 – Business Development 2 (Supply Chain)
- D5.3 – Business Development 3 (Business Planning)
- D5.4 - Business Development 4 (Commercial Readiness)
- D6.1 – Year 1 Report
- D6.2 – Year 2 Report

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Contents

Introduction

Climate change driven by greenhouse gas emissions from anthropogenic activities is drawing the attention of the scientific communities. The built environment is responsible for nearly 40% of the total CO_2 emissions and energy-related environmental pollution [1]. Concrete with Portland cement a key component is the main contributor to global warming among all construction materials used in the built environment [2]. That stems from the large volume of concrete used worldwide as compared to other construction materials. One of the concrete decarbonization pathways involves the use of less cement in concrete. Blended cement is gaining attention as cement substitution with secondary materials can reduce global emissions by up to 1.3 gigatons per year [3]. However, the most commonly used SCM, (fly ash and Slag) are meant to be limited availability because of the transition measure in the energy and steel sectors. The Amorphous precipitated silica (APS) derived from acid digestion of olivine,(a globally abundant magnesium silicate mineral) is a promising SCM as reported in the previous deliverable reports 3.1, 3.2 and 3.3. Apart from the environmental benefit it possesses unique characteristics that help improve the cement hydration and enhance the pozzolanic reaction. This deliverable aims to assess the durability performance of concrete mixes produced from APS.

Methodology

Mix design and concrete preparation

The chemical composition of OPC, Silica (APS), Fly ash and limestone used and their physical characteristics are in Table 1. Silica was prepared as described by Shanks et al. [4] and reported in the deliverable report 3.3. A commercial grade gypsum ($CaSO_4 \cdot 2H_2O$, 99 % purity) from VWR was used to regulate hydration as reported in the previous deliverable report 3.3. Due to the high specific surface area of silica compared to CEM I, a PCE-based superplasticizer (ViscoFlow3000, Sika) was added at 1 wt.% to obtain a workable concrete mix with silica. River sand with 40% passing 600µm sieve and a specific gravity of 2.56 g/cm³ was used as fine aggregate. Natural rock with a maximum grain size of 20 mm and a specific gravity of 2.6 g/cm³ was used as coarse aggregate.

The summary of the four concrete mix compositions is presented in Table 2. Further details are as follows: The design of the normal concrete mix was done following the method described by BRE [5]. Ordinary Portland cement CEM I, 52.5R was used as the binder. The mix parameters include compressive strength class C20/25 and a slump range of 60-180 mm with a water content of 195 kg/m³. These specifications were chosen to accommodate the high fineness of silica that might cause the resulting concrete mixing to be very dry. In addition, the water correction as described in the BRE method was carried out to maintain the free water-to-cementitious materials ratio at 0.55 in all the mixes. The CFA mix was made with blended cement composed of 80 wt.% cement and 20 wt.% fly ash and used as a blended cement benchmark to compare the performance of the concrete mixes with silica. The C20 mix was made of blended cement composed of an OPC/silica mass ratio of 80/20 replaced by 3 wt.% of gypsum.CLS concrete mix was made with a ternary cement blend composed of an OPC/silica mass ratio of 80/20 replaced by 5 wt.% limestone and 3 wt.% of gypsum. Additionally, 1wt.% superplasticizer is used to enhance the workability of the mix.

The concrete samples were prepared by mixing all the raw materials in a 30L pan mixer for 3 min followed by casting in cylinder molds of 200 mm height and 100 mm diameter. The cast samples were vibrated for 3 min, covered with a plastic bag and left in a storage room at 99% relative humidity for 24h. Then they were demolded and cured under water until further tests were performed at a specific age. Concrete samples of 50 mm height and 100 mm diameter were also cast for durability tests.

The image of the concrete samples and curing conditions are depicted in Figure 1.

Table 1 Chemical composition and Physical characteristics of raw materials.

Oxides (wt. %)	SiO_2	Al_2O_3	MgO	CaO	Fe_2O_3	SO_3	Cr_2O_3	K_2O	TiO_2	LOI	Total
CEM I	20.29	6.08	1.31	62.05	2.67	3.49	-	-		1.94	97.83
APS (Silica)	73.14	1.07	3.35	0.49	1.89	4.00	0.85	-	-	15	99.79
FA	50.36	33.03	0.68	4.34	5.73	1.08	0.03	1.33	1.39	-	97.97
Limestone	2.32	1.3	0.44	94.65	0.64	0.15	-		0.07	-	99.69

Physical properties	d_{10} (μm)	d_{50} (μm)	d_{90} (μm)	BET-SSA (m^2/g)	Specific gravity (g/cm^3)
CEM I	2.5	14.3	48.3	1.14	3.15
APS (Silica)	6.01	28.9	86.9	146.1	2.14
FA	4.68	17.9	66.3	-	2.31
Limestone	2.88	15.3	92.2	0.79	2.79

Table 2 Concrete mix design composition.

	Mix proportion concrete (Kg/m^3)									
Mix ID	Cement (52.5R)	FA	Silica	Limestone	Gypsum	Fine aggregate	Coarse aggregate	Water	SP	Free W/CM
Co	355	0	0	0	0	812.7	993.3	195	0	0.55
CFA	284	71	0	0	0	796.5	973.5	195	0	0.55
C20	275.5	0	68.9	0	10.7	792.9	969.1	195	7.1	0.55
CLS	264	0	66.1	17.4	7.1	792	968	195	5.3	0.55

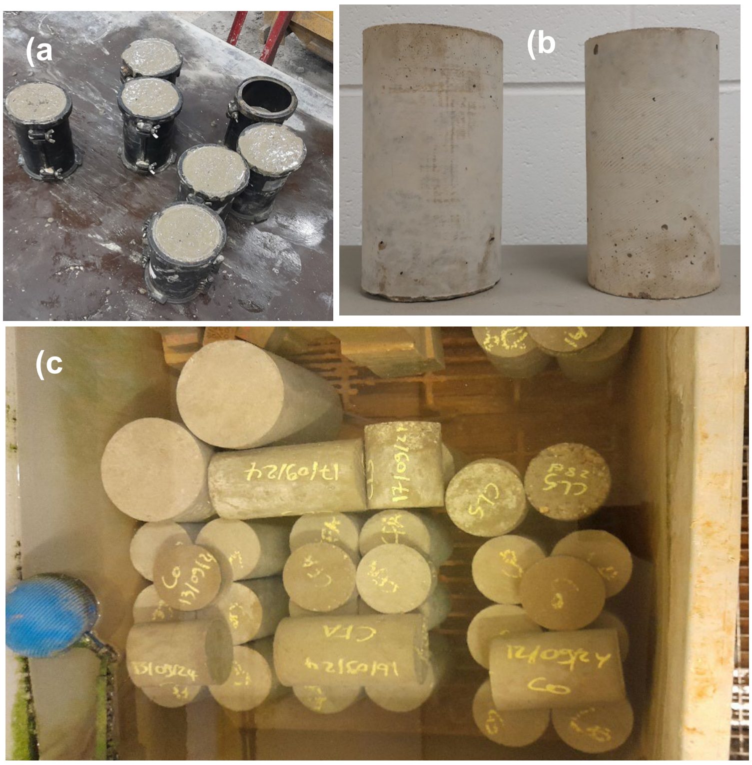

Figure 1. Concrete sample in the mould (a) after demolding (b) and in the water curing tank (c).

Workability and compressive strength

The concrete slump test was carried out to ensure that the amount of water added to the mixes was correct to achieve the slump range targeted for the normal concrete mix. This was carried out following the procedure described in BS EN 12350-2.

Compressive strength testing was performed on 28 and 90-day-old concrete samples using an Instron 5984 universal testing machine operated at a loading rate of 0.03 kN/s. Three samples were tested at each age and the average was considered.

Capillary and water adsorption

The test was carried out on concrete samples after 28 and 90 days by measuring the mass gain at different time intervals over 4h using the test set-up described in ASTM C1585-20. Initially, the weight was measured every 5 min for 60 min as the water penetration was fast, then at 10 min intervals for the next 60 min and 30 min intervals for the rest of the test. Before the test, samples were cured at 105 °C in an oven for 48h. Thereafter the lateral faces were sealed with waterproof sealer tape so that only the bottom faces were exposed to water.

The water absorption was measured following the ASTM standard C642-21. For this test, the samples the initial mass of the sample cured in water for 28 and 90 days was recorded (saturated mass), then cured at 105 °C in the oven for 24h. After removal from the oven, they were cooled down to 25 °C and the dry mass was recorded. Three samples were tested at each age and the average was considered.

Electrical resistivity

The bulk electrical resistivity was measured according to ASTM C1760-12 to get information on the connectivity of the pores structure of the concrete samples. The test was carried out on a bulk resistance meter (AIM LCR Databridge 401) on saturated surface dry concrete cylinders (100 mm diameter and 50 mm height) after 28 and 90 days. The device is equipped with two square plate electrodes (150 mm size), between which the sample is placed and has two conductor wires to connect the control box to the plates. The two flat surfaces of the concrete sample were first recovered with an electrode gel to ensure a good electrical connection between the sample and the plate electrodes. Then an alternative current with a frequency of 1 kHz was applied through the specimen by the control box and the data was recorded after the signal stabilized for 60s. Three samples were tested at each age and the average was considered.

Rapid chloride permeability test (RCPT)

The RCPT was conducted according to ASTM C1202 to measure the concrete's ability to resist the penetration of chloride ions and to determine its permeability class. The test measures the total electrical charge passed through a saturated concrete specimen when applying an electrical potential of 60 V for 6h, applied to two cell compartments containing each 3wt.% NaCl solution and 0.5N NaOH solution respectively. This was done on samples aged 28 and 90 days.

Results and discussion

Workability and compressive strength

Workability is an interesting test that measures the slump of a fresh concrete mix to demonstrate its cohesiveness and how it can be placed and consolidated with minimal loss of homogeneity. Fig 2 shows the change in the slump of the fresh concrete prepared. All concrete mixes show a true slump which means they substantially keep their shape as described in the BS EN12350-2 and demonstrate the homogeneity of the mix [6]. This also indicates the conformity of the mix-design and it is easy to place on site. As expected the normal concrete mix achieved a slump of 62 mm which is within the targeted slump range (60- 180 mm) and corresponds to a slump class S2 according to the BS 8500 [7,8]. The slump of the CFA mix used as a benchmark is 32 mm corresponding to a slump class S1. The slump of concrete mixes C20 and CLS is 0 and 5 mm respectively, and also correspond to slump class S1. It is noteworthy that despite this very low slump of the mixes C20 and CLS, they become more workable when placed in the mould under vibration as depicted in Fig 1a. This observation indicates that water trapped in the pore structure of silica is freed when vibrating. So the high fineness and porous structure of silica fixes the water and reduces the slump. This slump class make concrete mixes with silica suitable for general construction work, ensuring ease of placement without compromising strength.

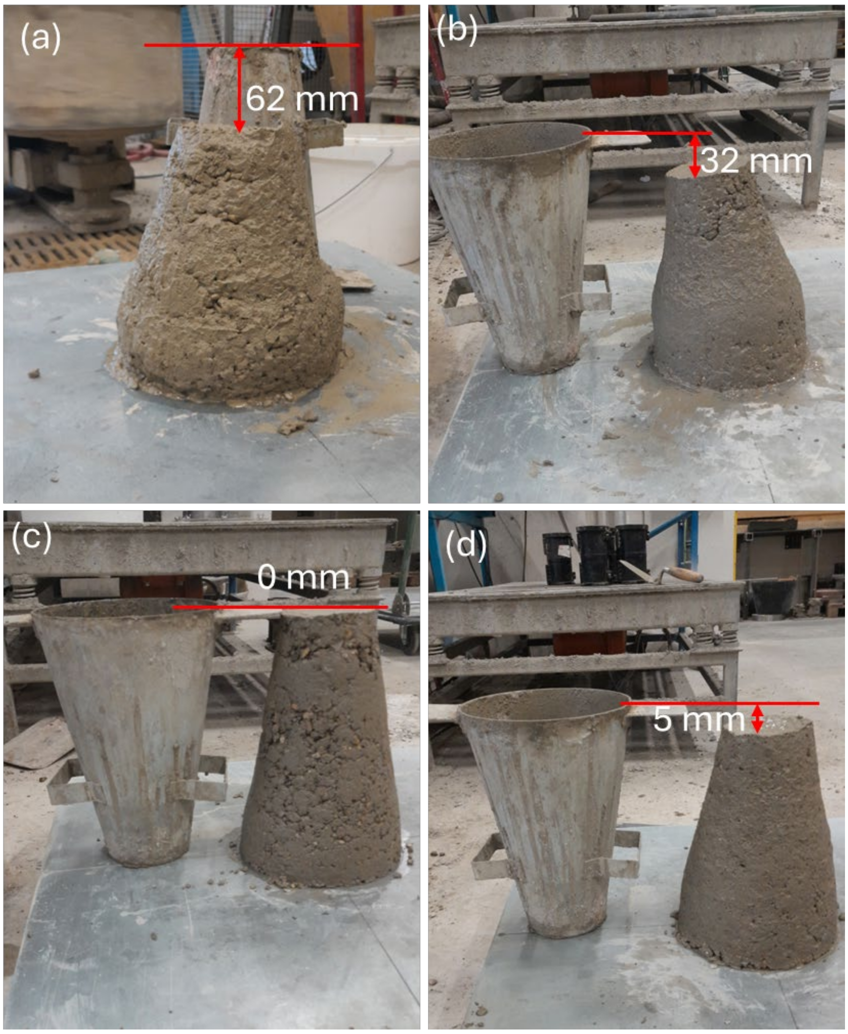

Figure 2. Slump test of fresh concrete (a) Co (b) CFA (c) C20 and (d) CLS

Fig 3 shows the compressive strength of the different concrete mixes at 28 and 90 days. As expected the 28-day strength is between the targeted class C20/25 corresponding to 20 MPa on cylinder samples. There is no considerable change in compressive strength at 28 days. The 90-day compressive strength of C0, CFA and CLS does not change considerably though higher than at 28 days strength. However, the comprehensive strength of C20 does not change considerably from 28 to 90 days. The latter agrees with the finding reported and discussed in deliverable 3.2 where limited strength change was observed. That is ascribed to the rapid reaction of silica that is maximum at 28 days.

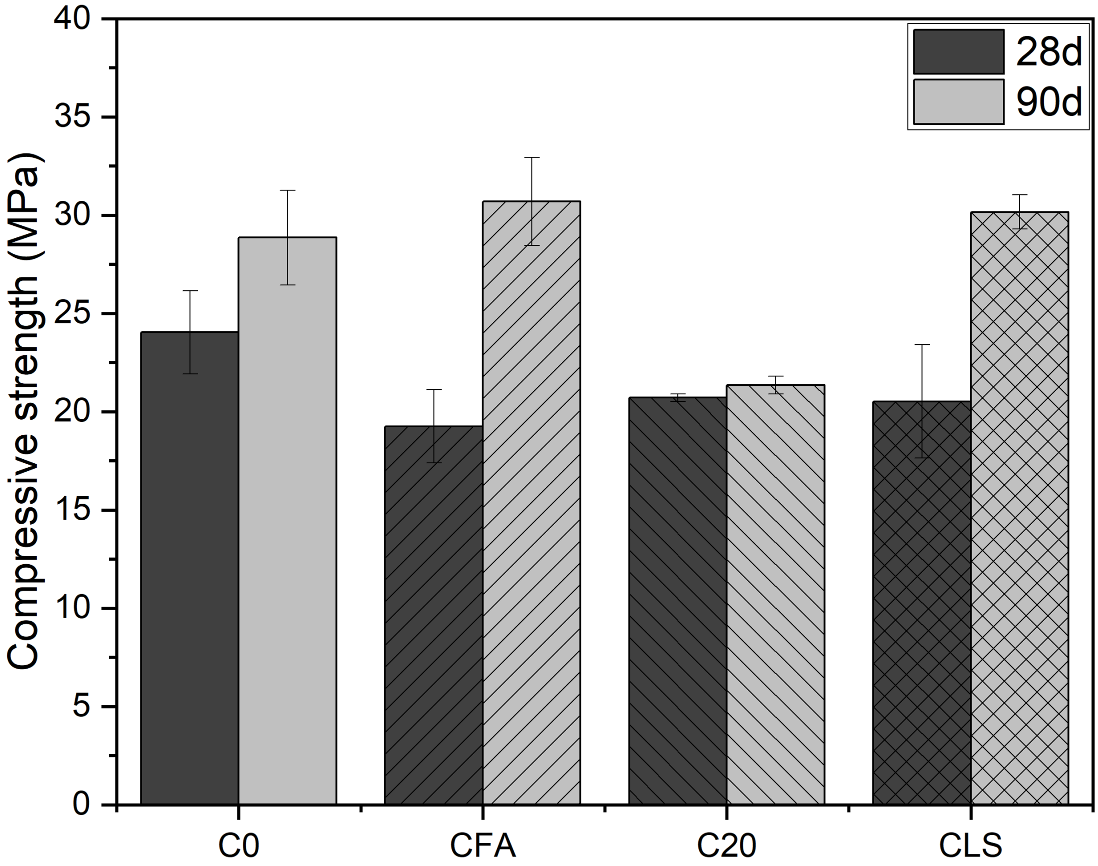

Figure 3. 28 and 90-day compressive strength of concrete samples.

Capillary and water adsorption

Fig 4 presents the change in sorptivity also known as capillary water suction measured on 28 and 90 days of concrete samples. All the mixes show a different rate of water suction over time. At 28 days It is higher for normal concrete, while C20 has the lowest rate. At 90 days, CLS initially has the highest rate which later on crosses the normal. The capillary water suction rate of C20 is the lowest at 90 days and has slightly changed when compared to 28 days. These observations demonstrate the microstructural characteristics especially the pore structure of each concrete mix. That is because despite the similar water absorption the samples absorb water at different rates. An explanation will be the refined pores size and less connectivity which explain the slowing down of the capillary water absorption rate. This is fostered by the presence of silica especially in C20 due to its fineness and large specific surface area.

Fig 5 presents the water absorption of 28 and 90-day-old concrete blocks. This measures the total water uptake in concrete after immersion. As observed there is no significant change in the water absorption among the different mixes at 28 and 90 days. However, the water absorption slightly decreases from 28 to 90 days for all the mixes except C20 which remains equal. This agrees with the compressive strength trend and indicates that the microstructural characteristics of concrete C20 do not evolve after 90 days. One can also notice that the filler effect of limestone contributes to the reduction in porosity as marked by the major drop in water absorption observed among all the concrete mixes.

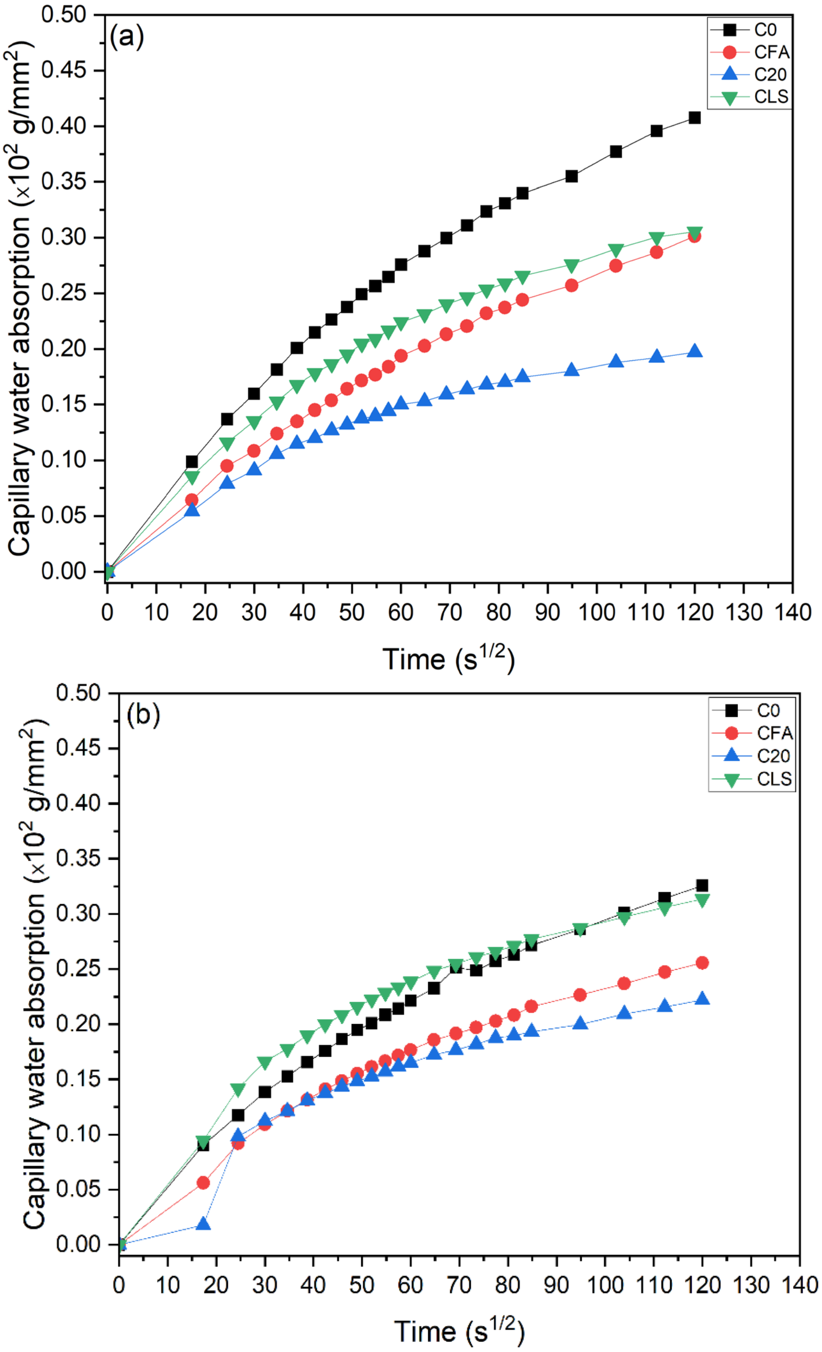

Figure 4. 28 and 90-day capillary water sorptivity of concrete samples

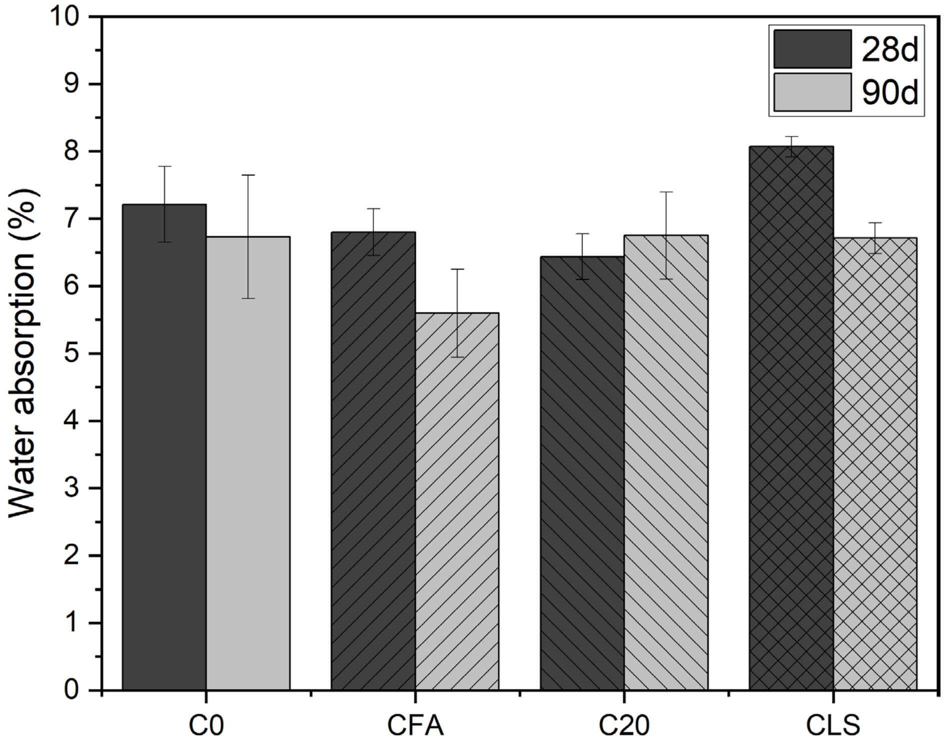

Figure 5. 28 and 90-day water absorption of concrete samples

Electrical resistivity and chloride permeability

The electrical resistance helps to measure the pore connectivity within a concrete sample. Fig 6 shows the changes in electrical resistance over the age of concrete samples. It increases with the age of the sample as a consequence of the pore de-perlocation related to the densification of the concrete. Sample C20 shows the highest electrical resistance at 90 days followed by CLS, while the normal concrete and the CFA have comparable resistance. This agrees with previous results and supports that the inherent characteristics of silica contribute to reducing pore connectivity. The reduction of the electrical resistance of CLS-containing limestone filler indicates the increase of the permeability as compared to C20. This agrees with literature that also proved the reduced permeability with the addition of inert filler to concrete [9]. The evolution of the electrical resistance is consistent with the chloride permeability test result presented in Fig 7. It shows that the permeability class of samples C20 and CLS at 90 days is very low while the one of normal concrete and CFA is moderate and low respectively. That chloride permeability has decreased from 28 to 90 days for all the mixes except C0 the normal concrete whose permeability class remain the same. All these findings mean that the use of silica as SCM is suitable for enhancing the durability performance of concrete.

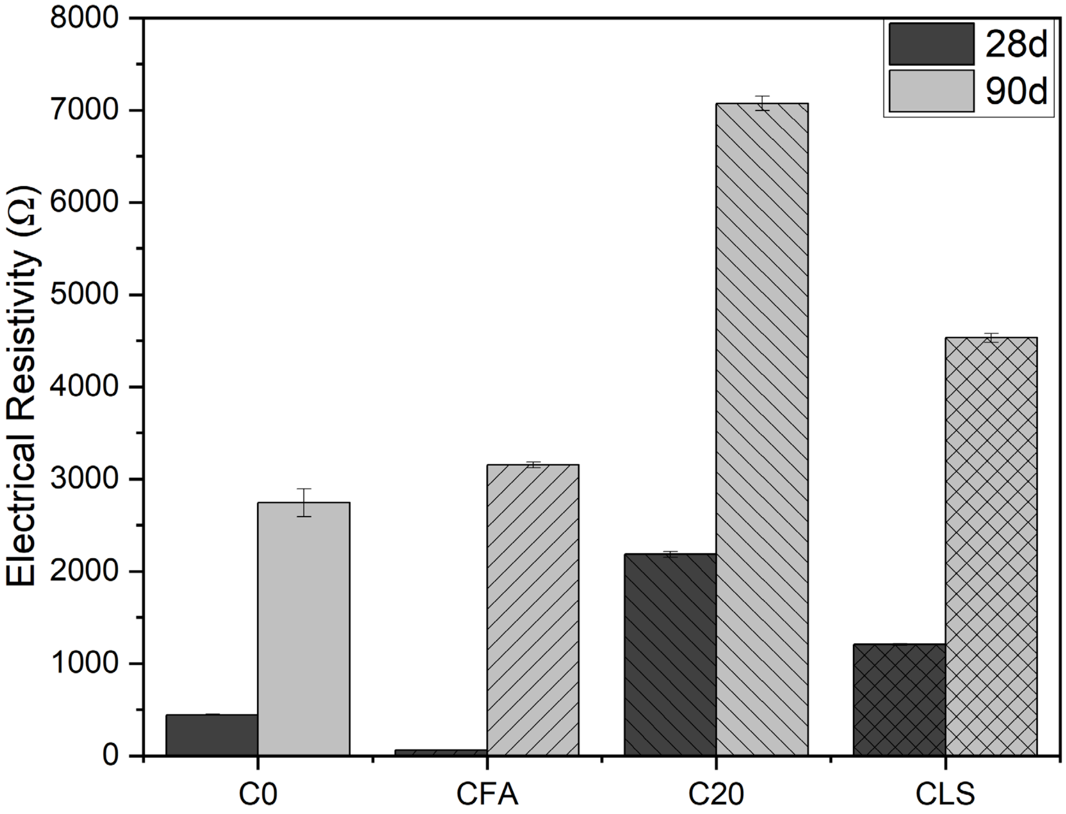

Figure 6. 28 and 90-day electrical resistivity of concrete samples.

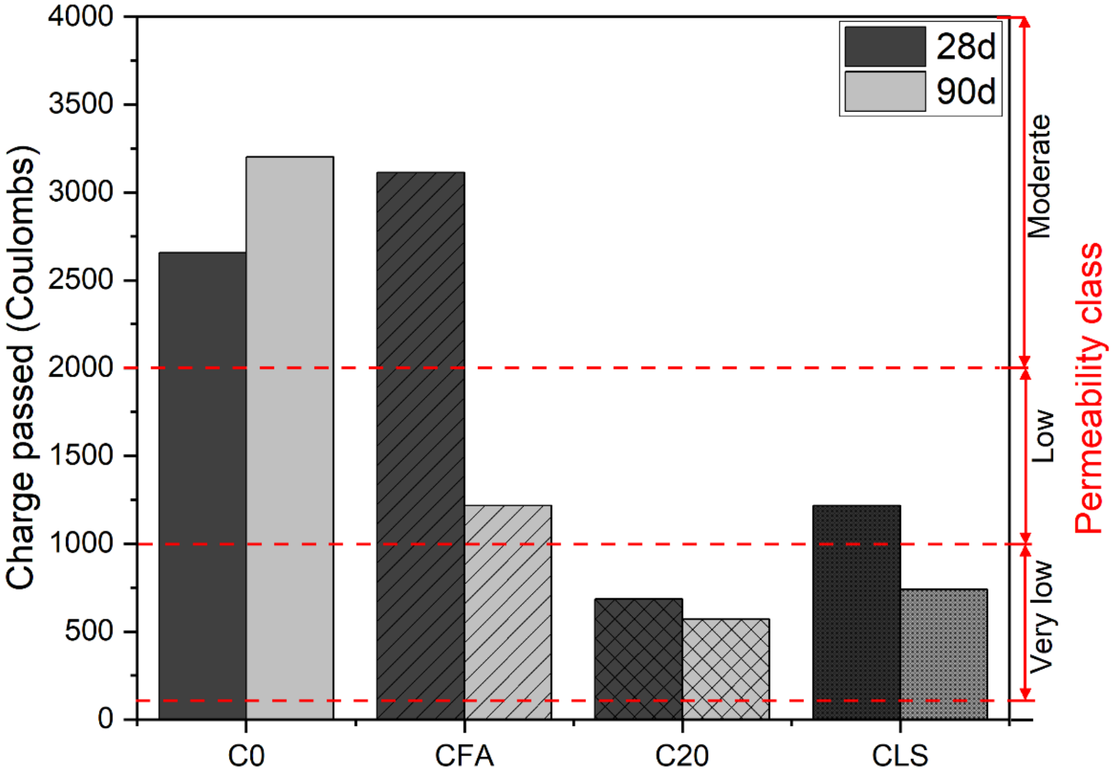

Figure 7. 28 and 90-day chloride resistance and permeability class of concrete samples.

Conclusions

The study demonstrates that amorphous precipitated silica (APS) derived from olivine is a viable supplementary cementitious material (SCM) for enhancing the durability of concrete while reducing its environmental impact. The experimental results indicate that APS effectively improves concrete performance by refining pore structure, reducing permeability, and maintaining compressive strength over time. Despite lower initial workability due to its high fineness and water absorption capacity, APS-based mixes showed good cohesiveness and improved durability properties.

Water absorption and sorptivity tests revealed that APS contributes to decreased porosity, with the C20 mix exhibiting the lowest capillary water absorption rate. The electrical resistivity and rapid chloride permeability tests further confirmed that APS enhances concrete durability by reducing pore connectivity and increasing resistance to chloride penetration. Compared to conventional concrete and fly ash-blended mixes, APS-based concrete showed superior resistance to long-term deterioration, making it a suitable material for sustainable construction.

Overall, the findings support the use of APS as an eco-friendly alternative to traditional SCMs like fly ash and slag, whose availability is declining due to industry shifts. This research highlights APS’s potential in producing durable, low-carbon concrete, reinforcing its role in advancing sustainable building practices and reducing CO_2 emissions in the construction sector.

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