Developing Fundamental Understanding of High-Performance Nano-silica Admixture on Concrete Curing

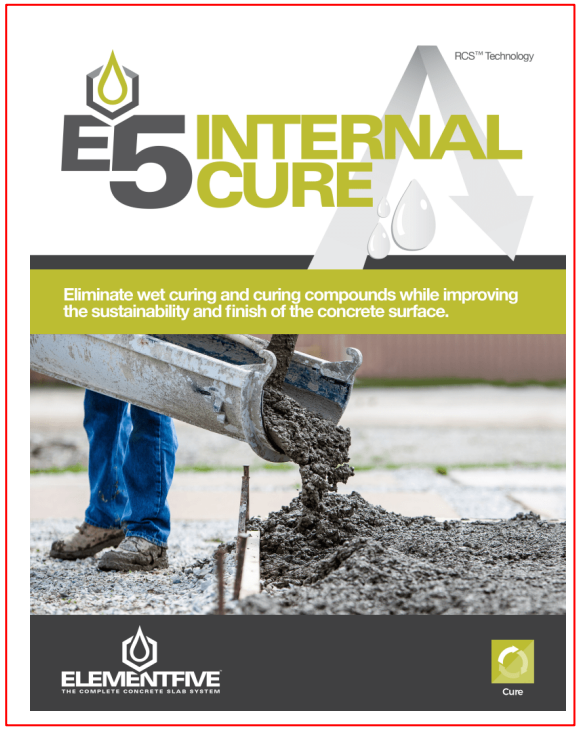
Cihang Huang Yen-Fang Su
Purdue University

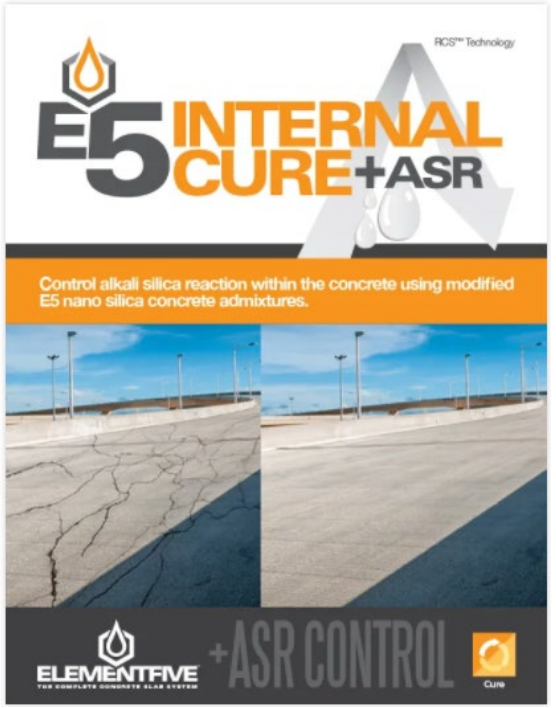
PURDUE UNIVERSITY | Lyles School of Civil Engineering

CENTER FOR INTELLIGENT INFRASTRUCTURES

Objective

- This project aims to develop a comprehensive and systematic understanding of Nano-silica admixture (E5^{TM} Admixture) on the concrete curing and related properties.





Specification Products

Works have been done

02/2020-12/2020

- **Properties of E5**
  - ❑ E5-Internal cure SEM observation
  - ❑ E5-Internal cure water retention capacity
- **Effect of E5 on concrete performance**
  - ❑ Isothermal calorimetry (IC) and thermal gravimetric analysis (TGA) (Twice)
  - ❑ Viscosity evaluation with E5-Internal cure
  - ❑ Lab mechanical testing for concrete (Twice)
  - ❑ Slump, density and air content evaluation
  - ❑ Concrete sample preparation-Rapid Chloride Permeability Test (RCPT)
- **Large slab pouring and testing**

Works have been done

01/2021-10/2021

- **LFA testing**
  - ❑ Compressive test
  - ❑ Drying shrinkage
  - ❑ TGA and IC
  - ❑ SEM
  - ❑ ASR
- **INDOT bridge mixture with E5**
  - ❑ Compressive test
  - ❑ Water sorptivity test
- **Durability of concrete with E5 (w/c=0.42)**
  - ❑ Rapid chloride permeability test (RCPT)
  - ❑ Flexural testing
  - ❑ Micro-CT
  - ❑ Concrete corrosion test (in progress)
  - ❑ Electrical resitivity
- **Evaluate the internal curing effect of E5 (w/c =0.47)**
  - ❑ Internal humidity measurement
  - ❑ Drying shrinkage evaluation
  - ❑ TGA and IC
  - ❑ SEM
  - ❑ Water retention test
  - ❑ Sorptivity test
- **SHCC-E5 design and sample testing**
  - ❑ Mechanical testing (Compressive, tensile and flexural)
  - ❑ Self-healing evaluation
  - ❑ Bonding strength evaluation
  - ❑ TGA
  - ❑ SEM

Schedule

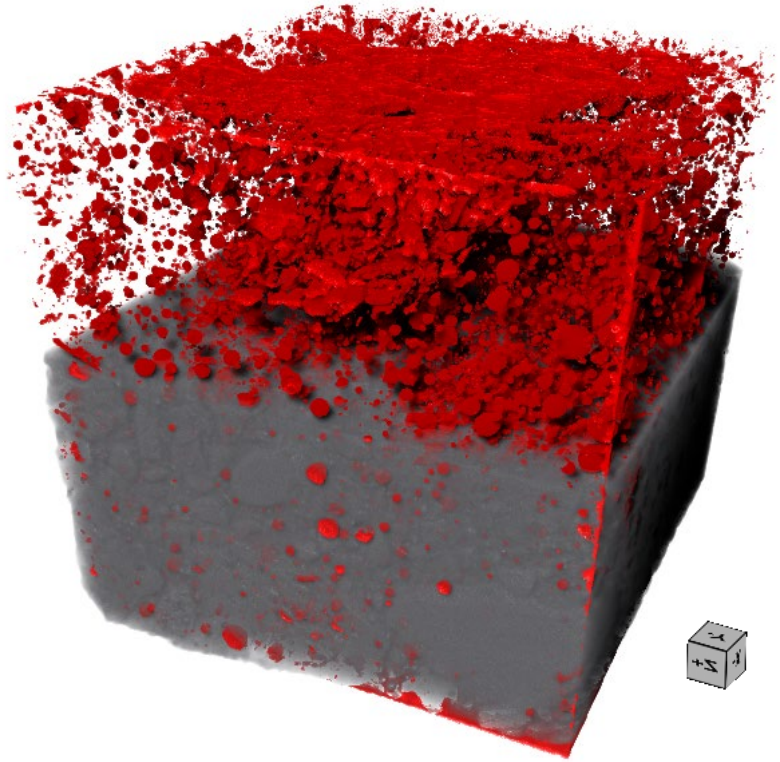
Tasks	Year 1	Year 2	Year 3
Task 1 – Mechanical Testing			
Task 2 – EMI testing for early age properties			
Task 3 – Understand the hydration effect			
Task 4 – Understand the concrete rheology effect			
Task 5 – Physio-chemical and microstructure			
Task 6 – Durability testing			
Task 7 – Interfacial shearing strength			
Task 8 – Large scale testing			

- In progress

Progress update

Micro-CT for Concrete w/c=0.42

Reference sample

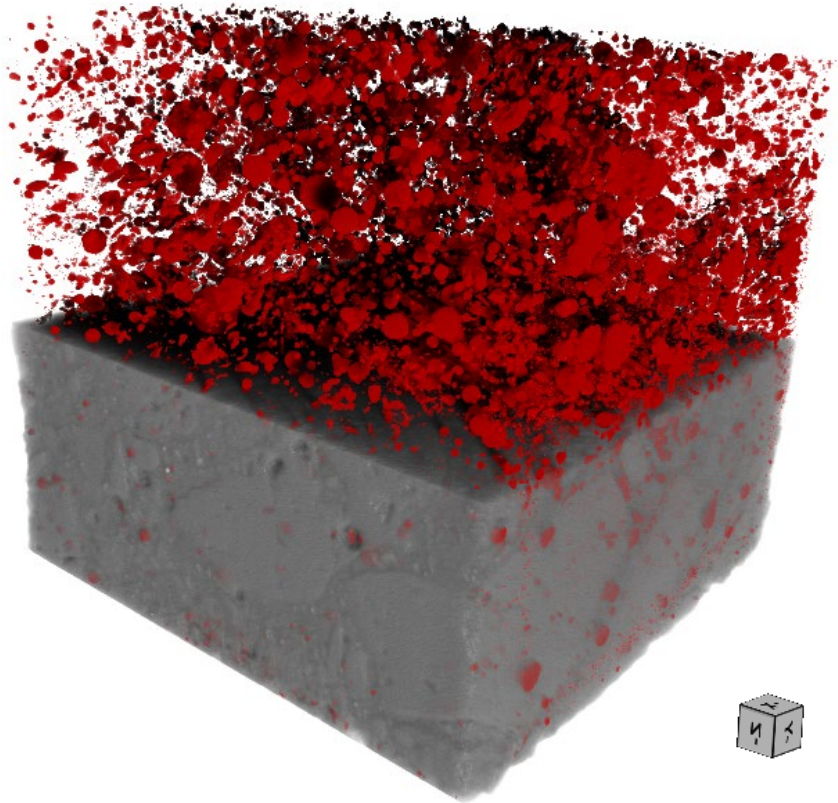


Pore volume: 2,664 mm³

Sample Volume: 53,008 mm³

Porosity: 5.03 %

0.6%E5 sample



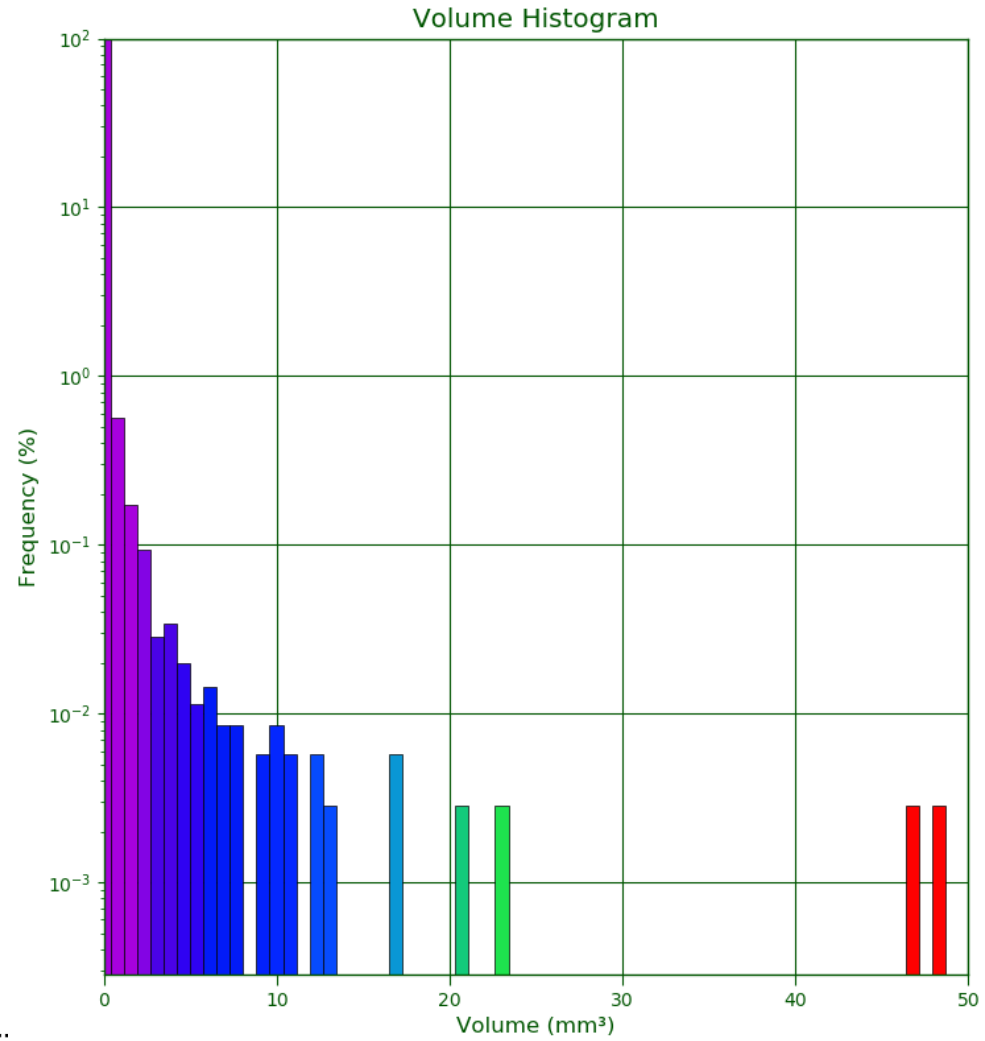
Pore volume: 1,822 mm³

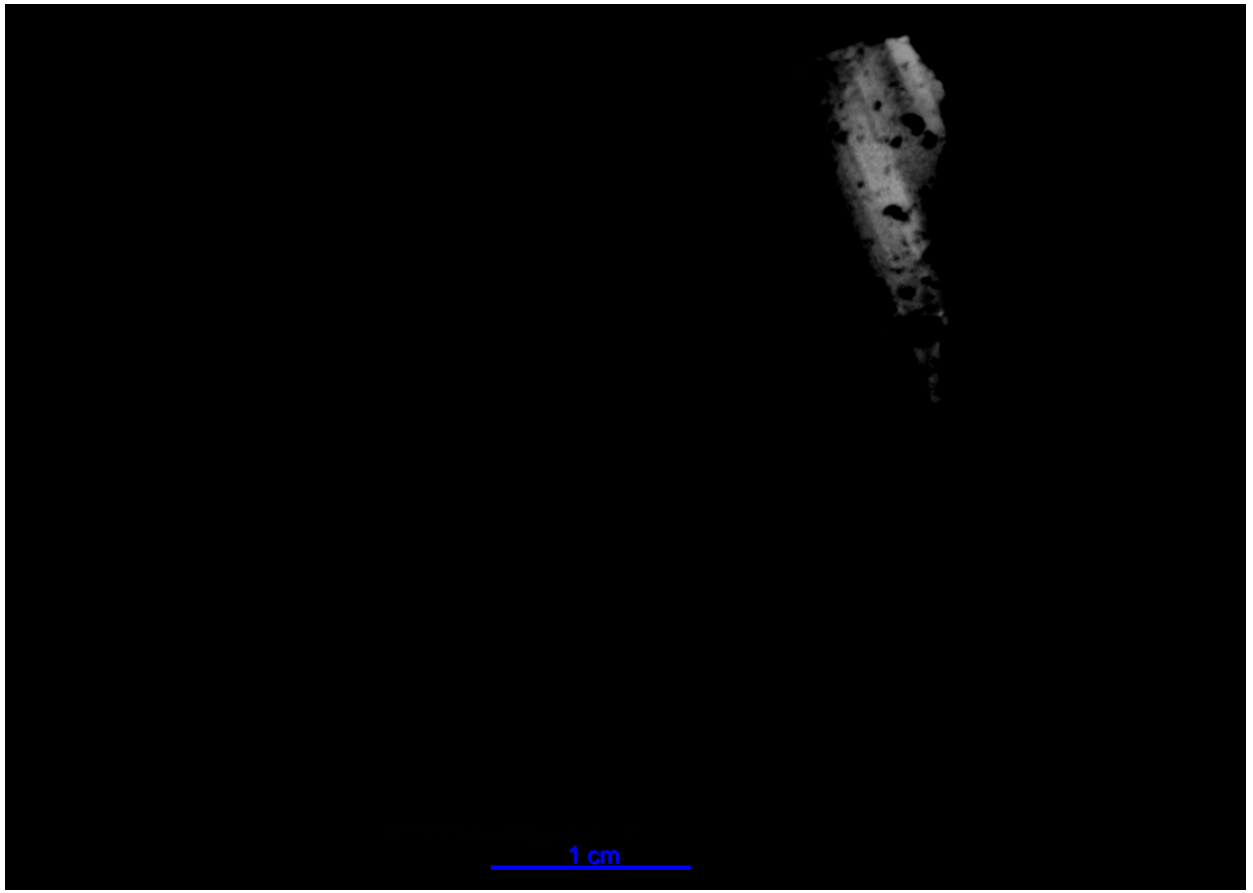
Sample Volume: 53,462 mm³

Porosity: 3.4 %

Micro-CT for Concrete w/c=0.42

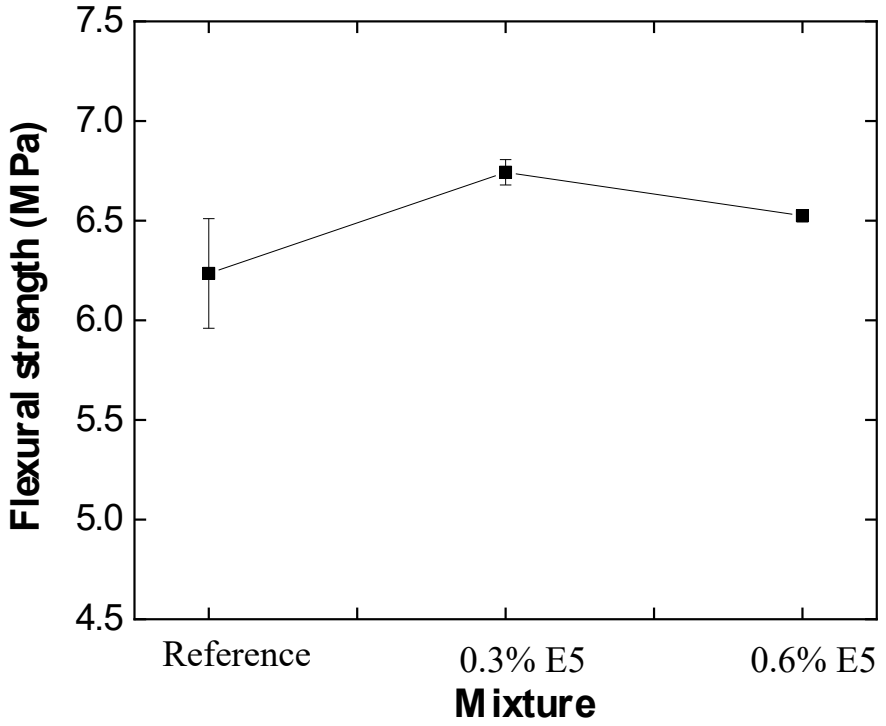
Reference sample





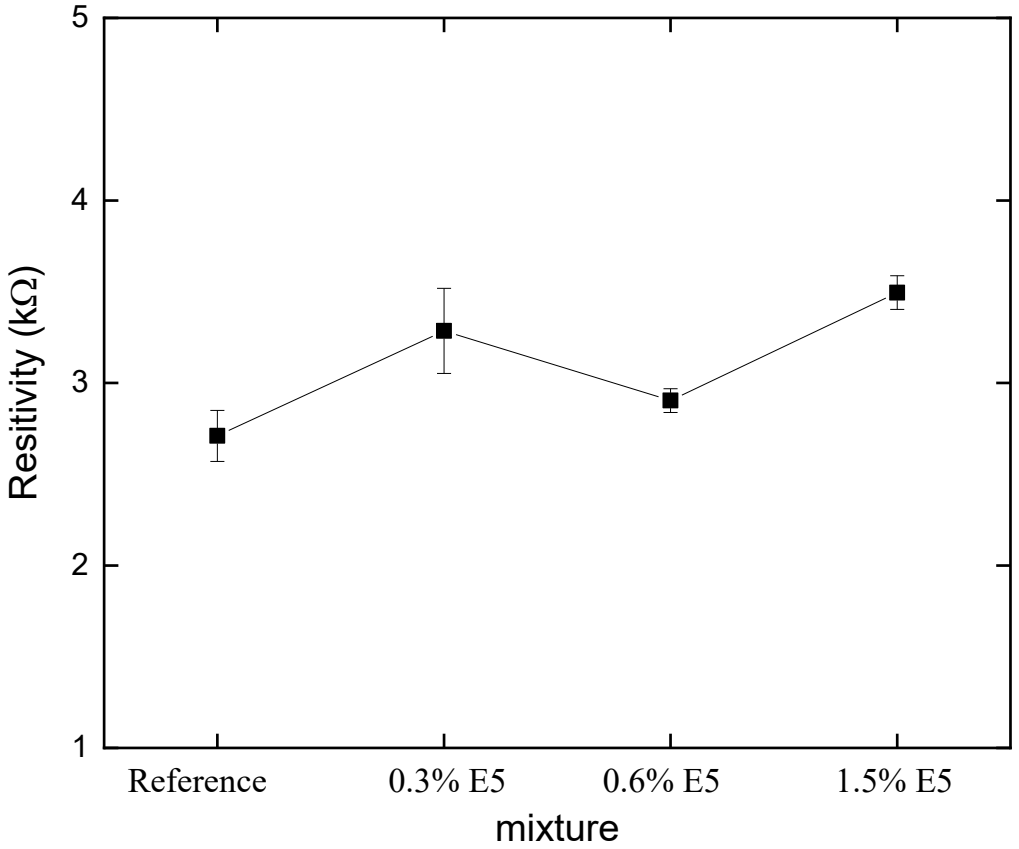
7 days flexural strength

INDOT 564 - w/c=0.42



The addition of the E5 clearly improved the flexural strength of the beam (4 × 4 × 15) sample.

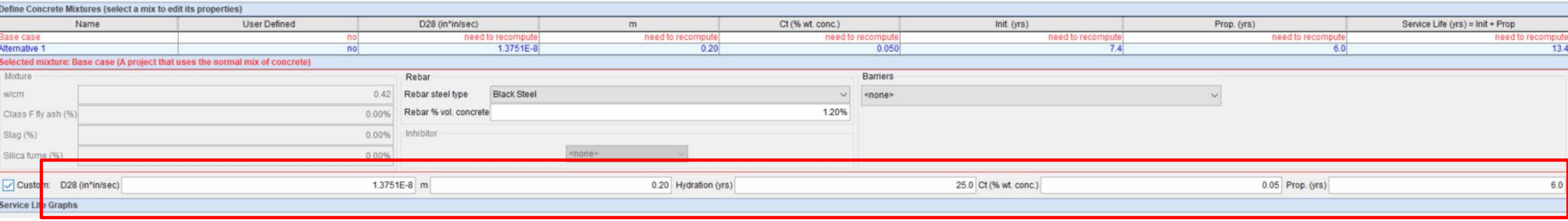
Electrical Resistivity



- the resistivity value can be directly related to the chloride diffusion coefficient of concrete.

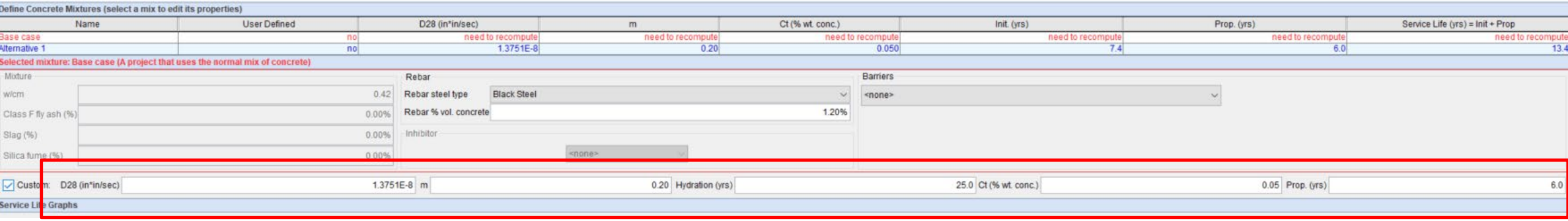
Lu, Xinying. "Application of the Nernst-Einstein equation to concrete." *Cement and Concrete Research* 27.2 (1997): 293-302.

Life-365™ service life prediction



- D28 (in*in/sec), (chloride) diffusion coefficient (ASTM C1556)
- m, diffusion decay index (based on the composition of the concrete)
- hydration (yrs), hydration years (default 25 years)
- ct (%wt.conc), chloride concentration necessary to initiate corrosion (based on the type of reinforcing steel)
- Prop. (yrs), propagation period (based on the type of reinforcing steel used)

Life-365™ service life prediction



- m, diffusion decay index (based on the SCMs of the concrete

$$D(t) = D_{ref} \cdot \left(\frac{t_{ref}}{t} \right)^m,$$

where $D(t)$ = diffusion coefficient at time t,

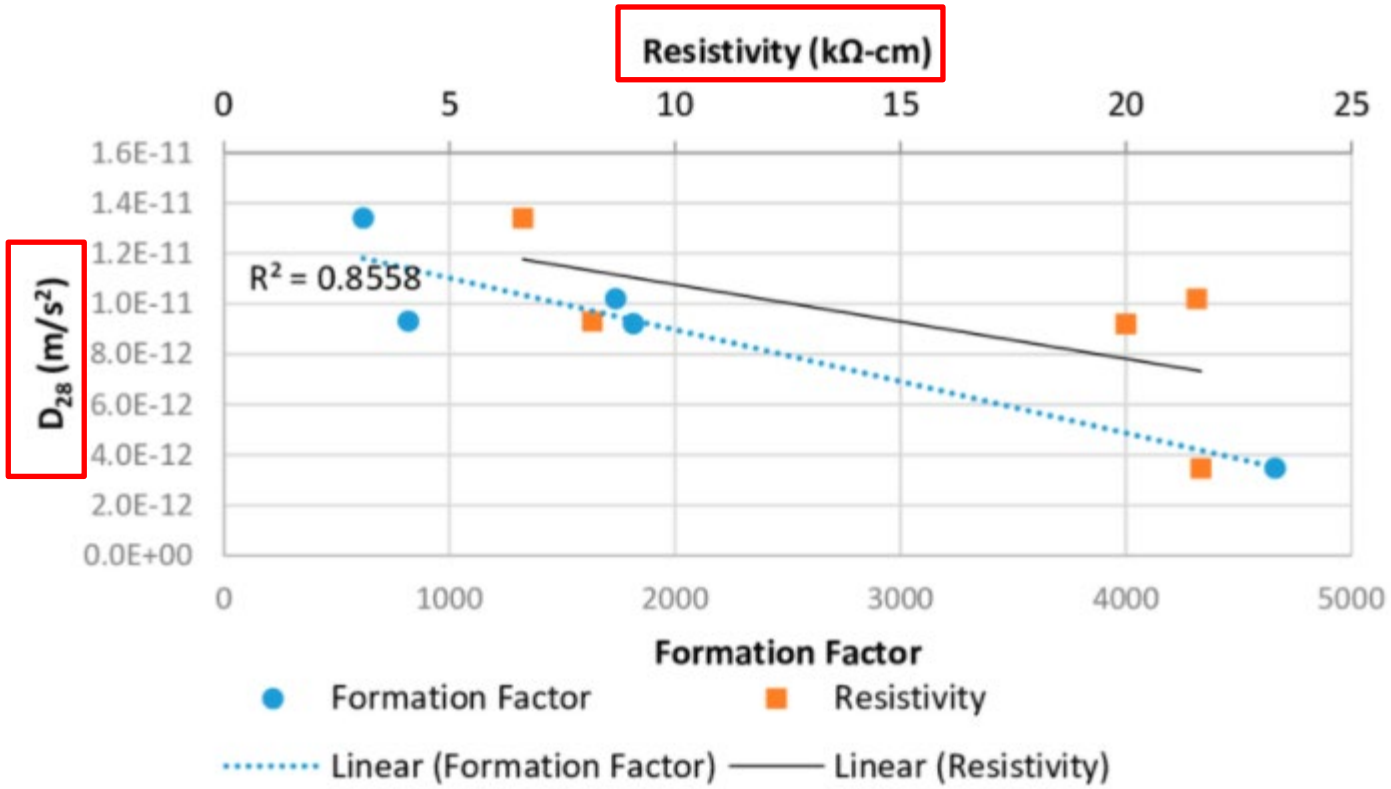
D_{ref} = diffusion coefficient at some reference time t_{ref}, and

m = constant (depending on mixture proportions).

Concrete Mixture	*m*
PC Concrete	0.264
Fly Ash Concrete	0.700
Slag cement Concrete	0.620

- The relationship between diffusivity and time is best described by a power law

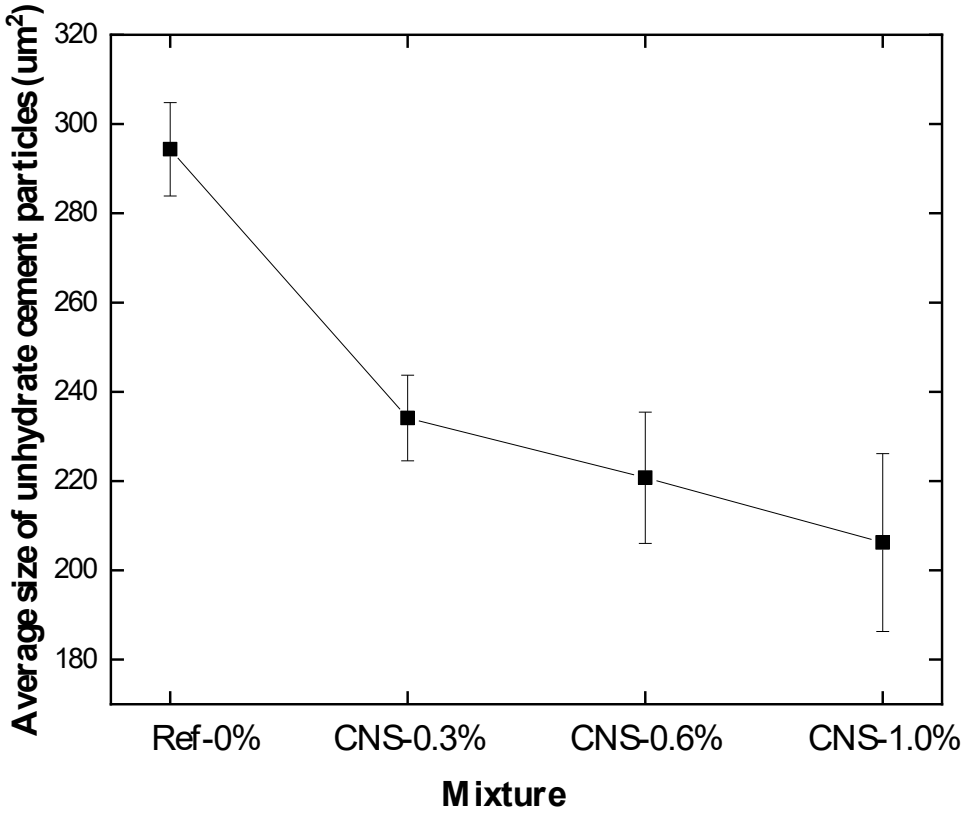
Electrical Resistivity



- The resistivity value can be directly related to the chloride diffusion coefficient of concrete.

Abd El Fattah, Ahmed, et al. "Field validation of concrete transport property measurement methods." *Materials* 13.5 (2020): 1166.

Unhydrated cement particles size analysis



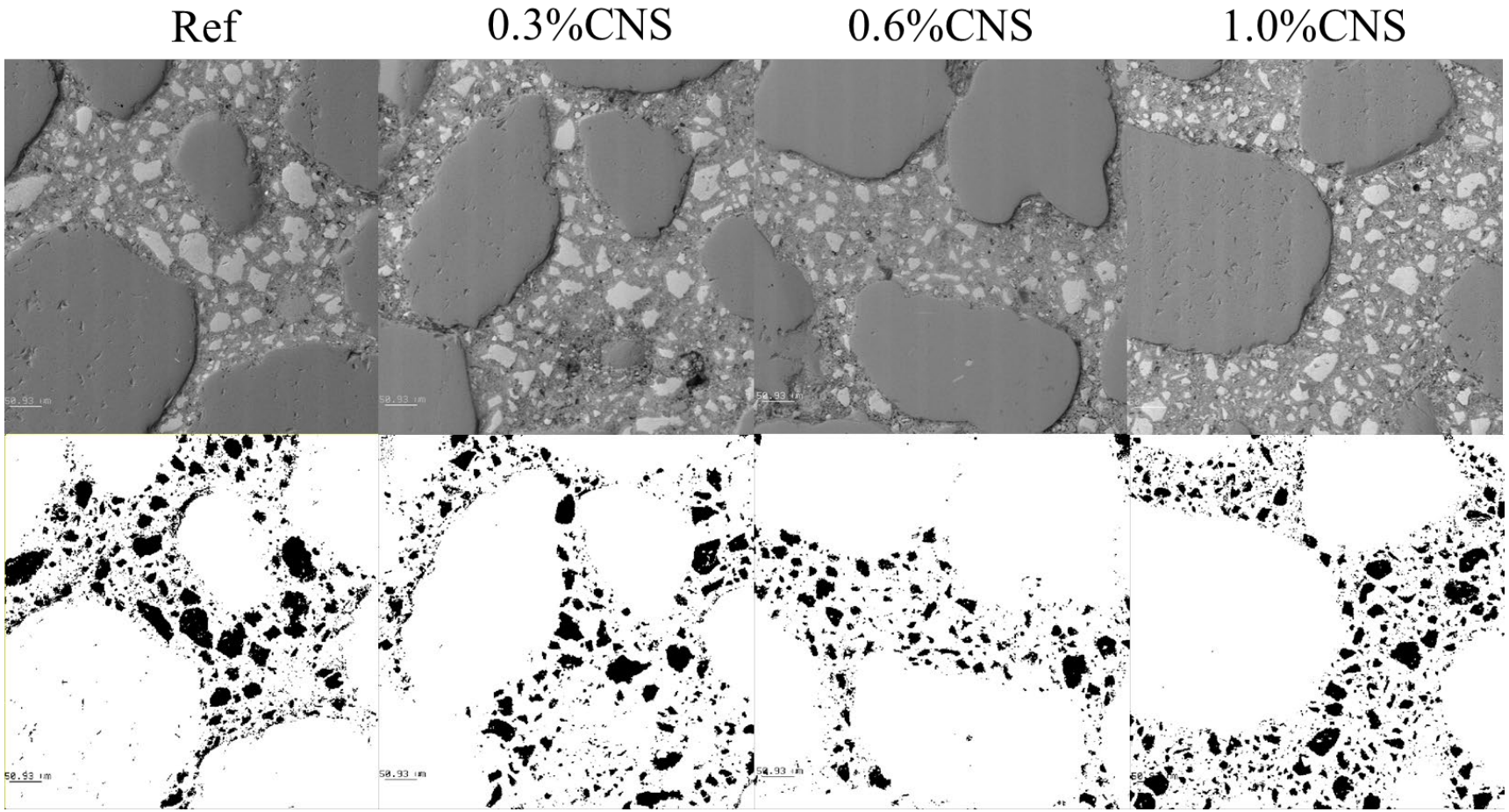
Mortar-w/c=0.47

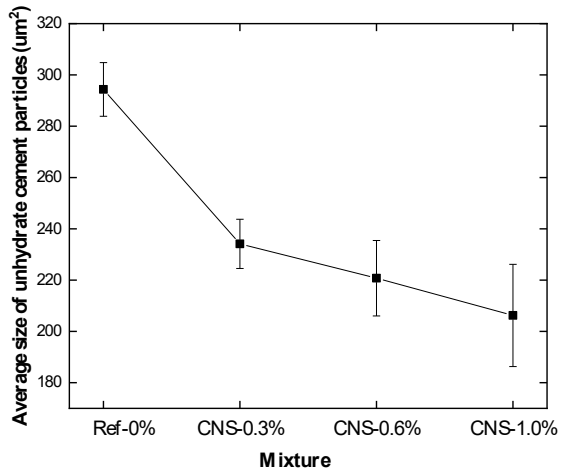
	Reference	0.3%CNS	0.6%CNS	1.0%CNS
Ave (μm^2)	294.346	234.146	220.749	206.235
SD	10.461	9.600	14.708	19.937

* CNS: colloidal nanosilica (E5)

- A more thorough analysis of the size of the unhydrated cement particles is conducted. Each data point represents the average result of 5 images.
- The result indicates that the incorporation of the CNS (E5) reduced the size of the unhydrated cement particles, which is the outcome of the improved hydration.

SEM images comparison

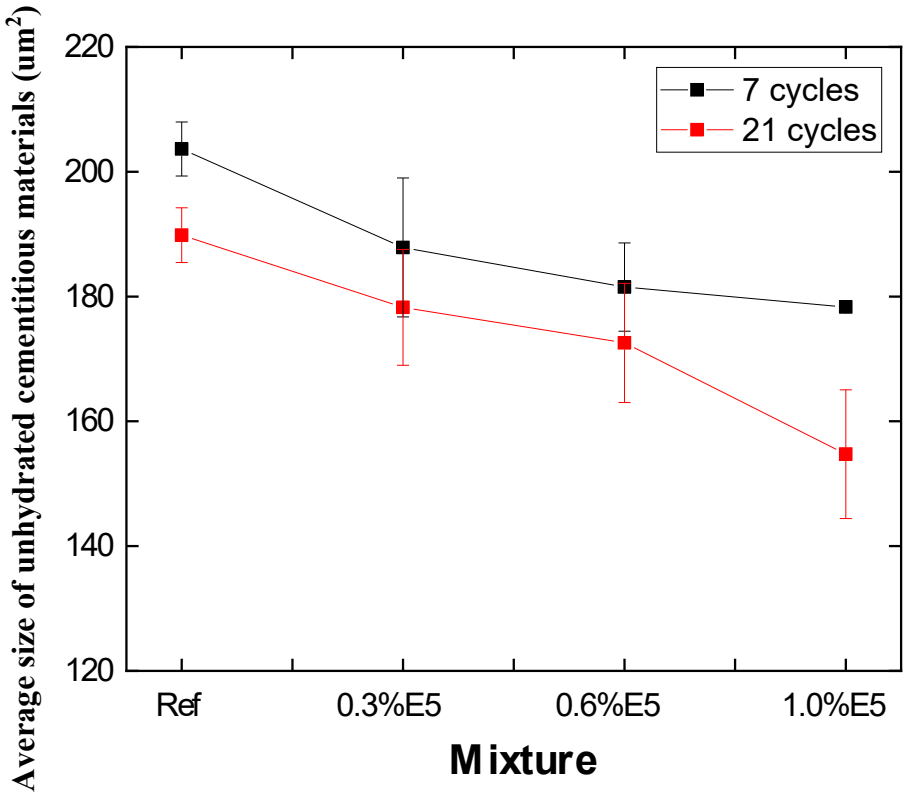




28d mortar - w/c=0.47

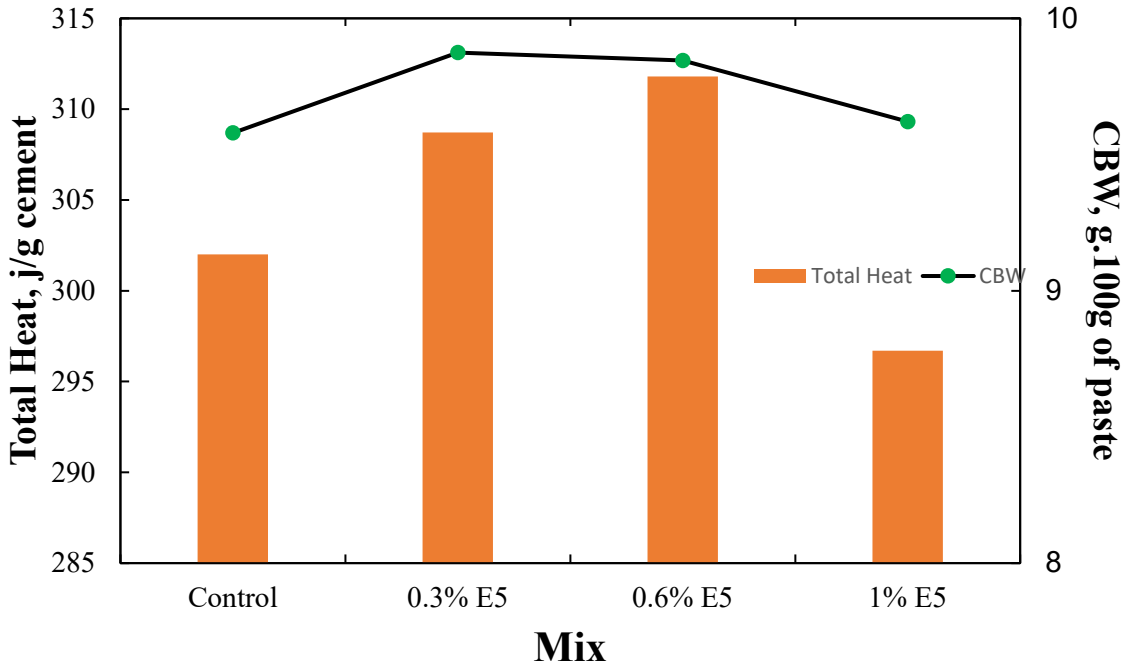
- Representative images of each mixture were compared and the reduction of the size of the unhydrated cement particles can be observed.

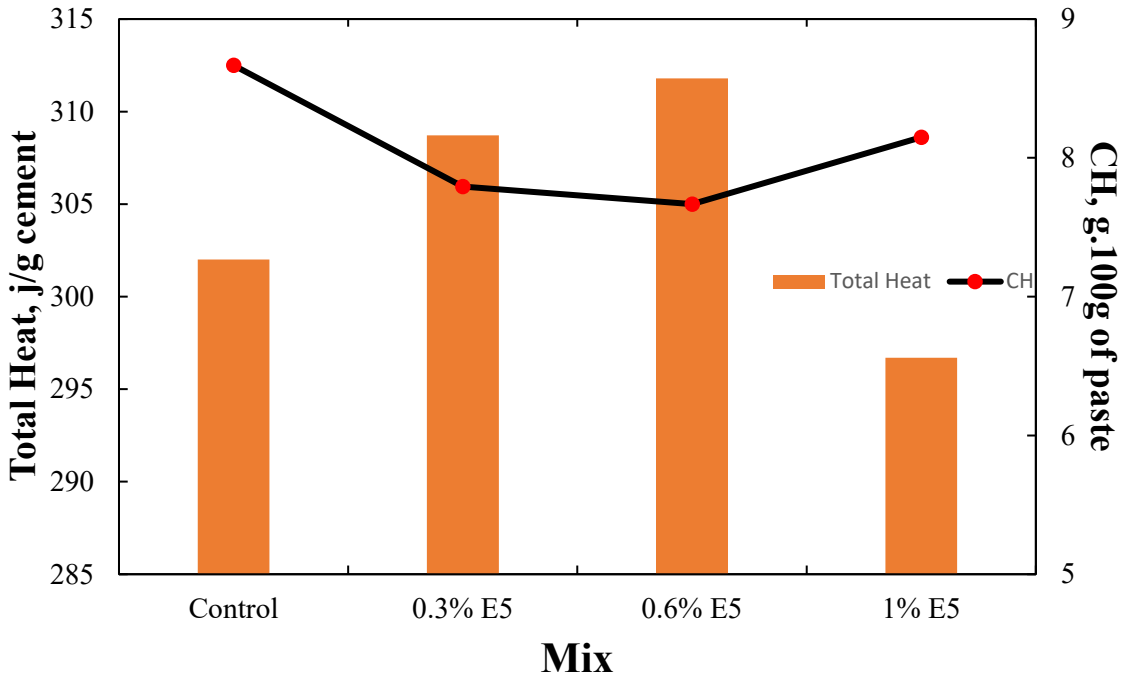
SEM for SHCC-E5 and image analysis



- As the curing age extended, the size of the unhydrated particles reduced.
- The addition of the E5 led to a decrease of the size of the unhydated particles, which implies an improvement of the hydration.

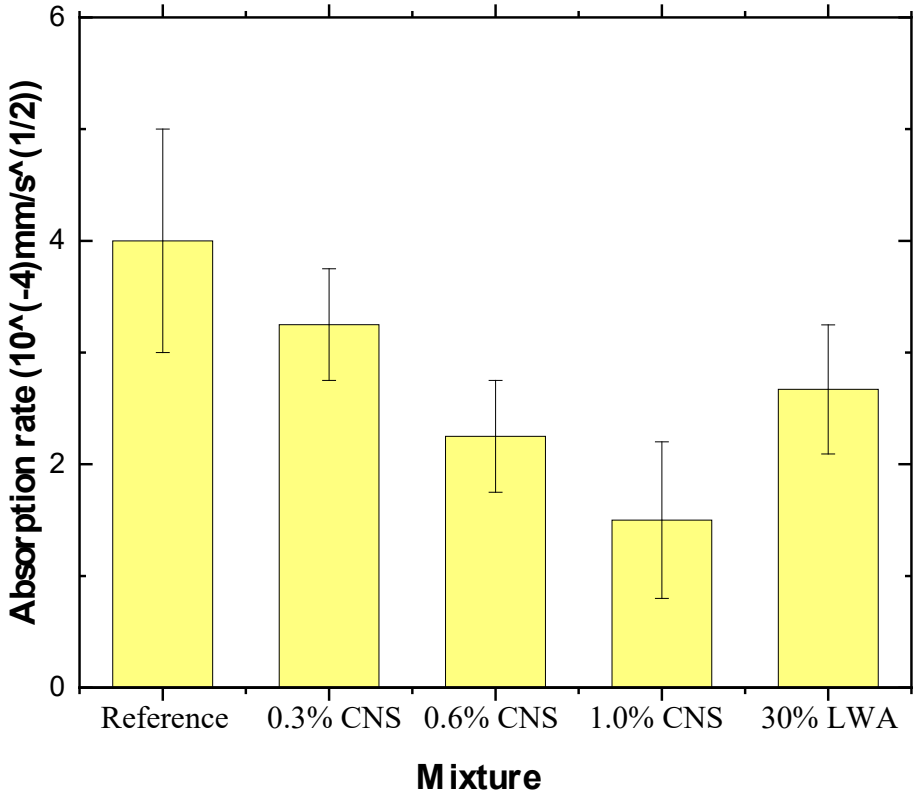
Analysis of the IC and TGA





- The result of the IC well correlated with the result of the TGA (CH/CBW).
  - ❑ With the addition of the E5-CNS, the total heat release during hydration was firstly increased and then decreased, while the TGA shows a same trend for the content of the chemistry bound water.
- The CH content show an inverse correlation with the IC.
  - ❑ There was pozzolanic reaction due to the incorporation of the E5-CNS, which consumed the CH;
  - ❑ The pozzolanic reaction improved the overall hydration performance of the cement paste.

Sorptivity test (w/c = 0.47)

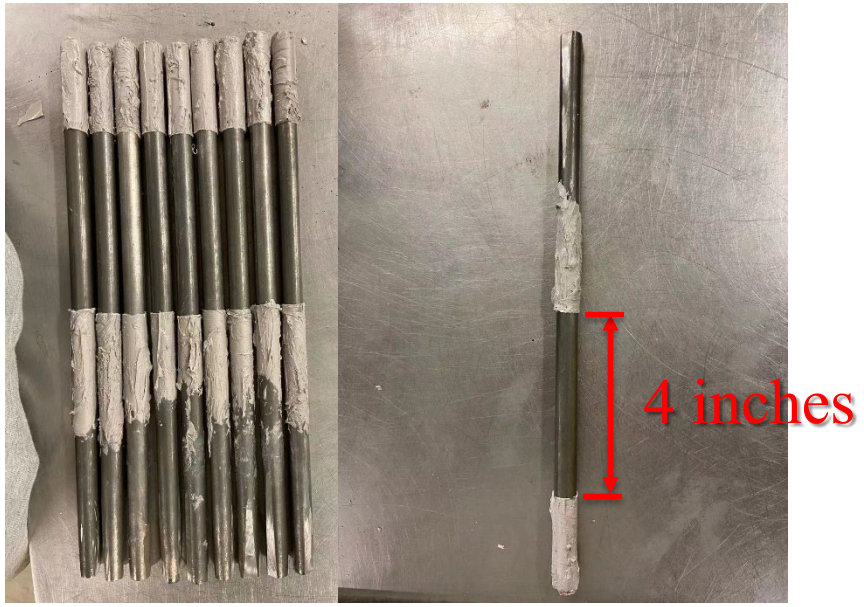


- It can be observed that the incorporation of the internal curing agents effectively reduced the average absorption rate of the sample by over **18.15%.** For E5-CNS, as the dosage increased, the absorption rate was further reduced from $4.0 \times 10\text{-}4 mm/s^{0.5}$ to less than $1.5 \times 10\text{-}4 mm/s^{0.5}$.
- Since the reduction of the absorption rate could improve the pore structure and extend the service life of the concrete, the use of the E5-CNS **favors the durability (lower ion/liquid permeability),** of the concrete materials.

Prepare for rebar pull-off test

Setup for rebar pull-off

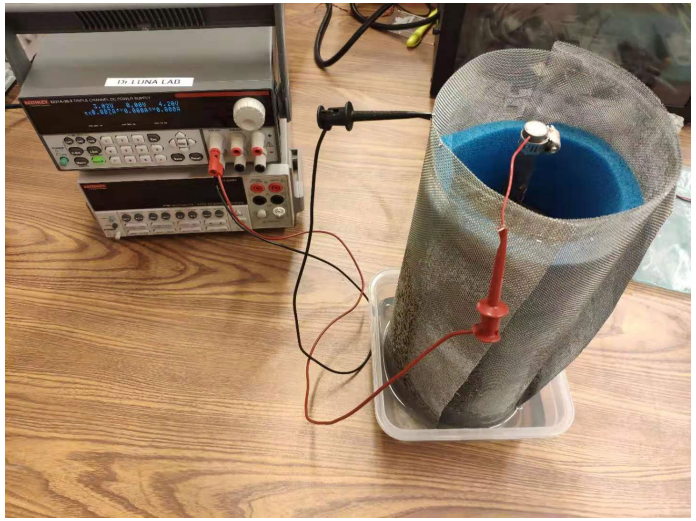
Rebar washed by the sulfuric acid
(get rid of rust and coating oil)



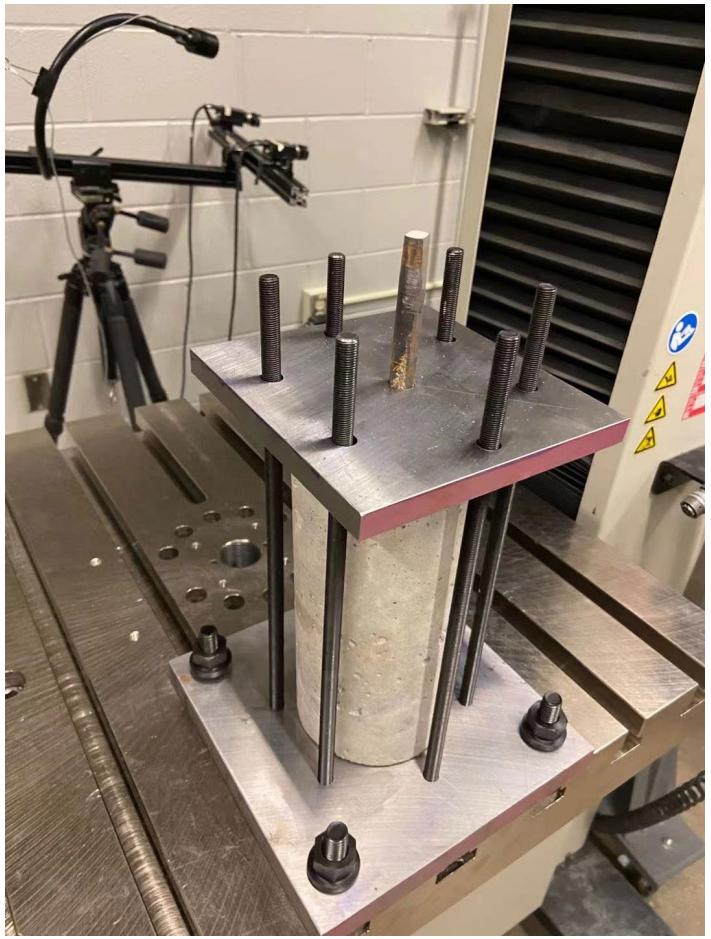
Rebar coated with the epoxy
(to limit the area that expose for the corrosion)

Prepare for rebar pull-off test

Sample preparation

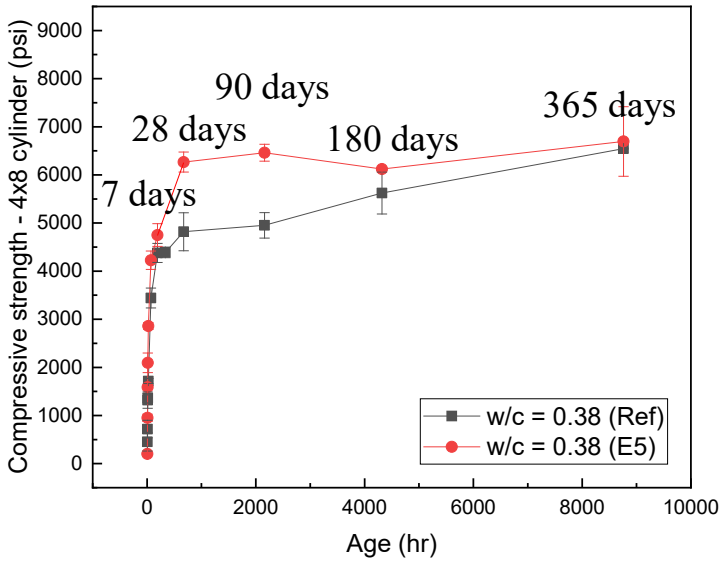


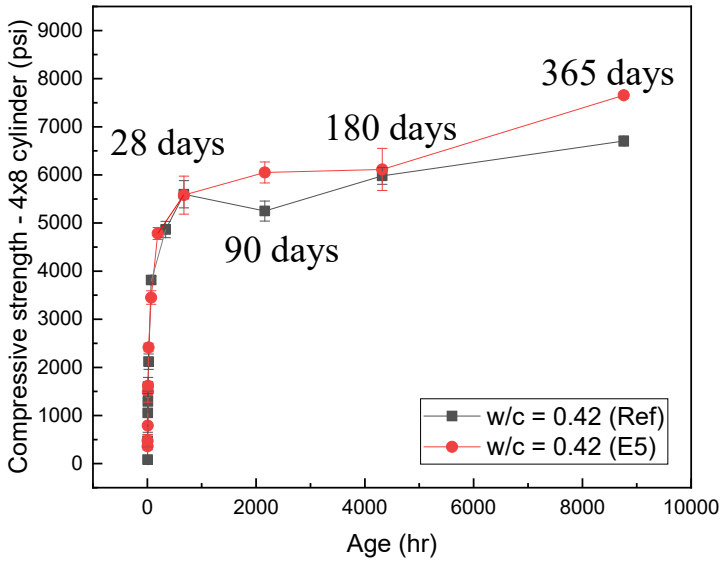
Accelerated corrosion

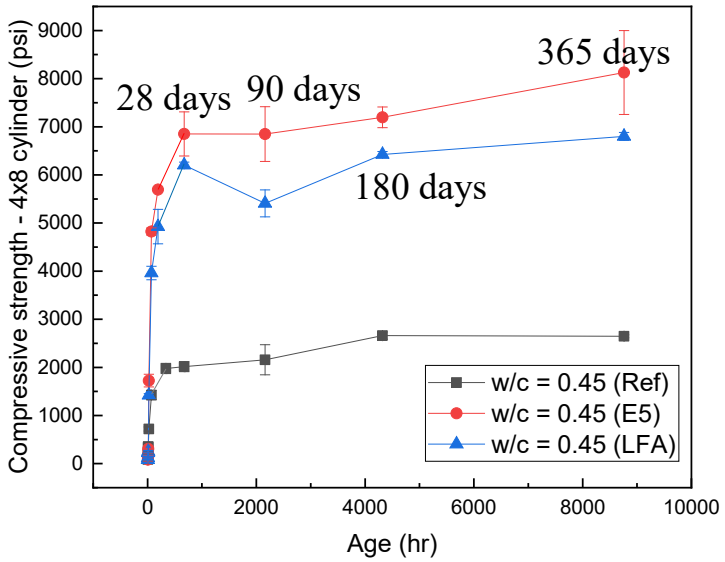


Rebar pull-off

Large slab testing results – 4x8 mold

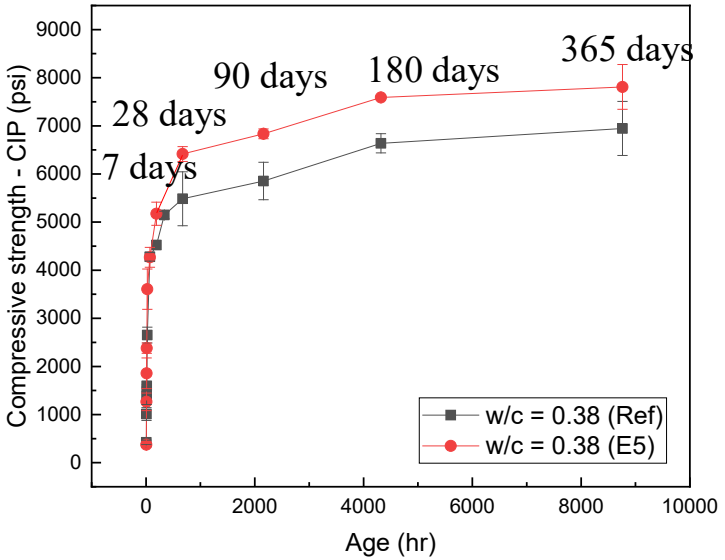


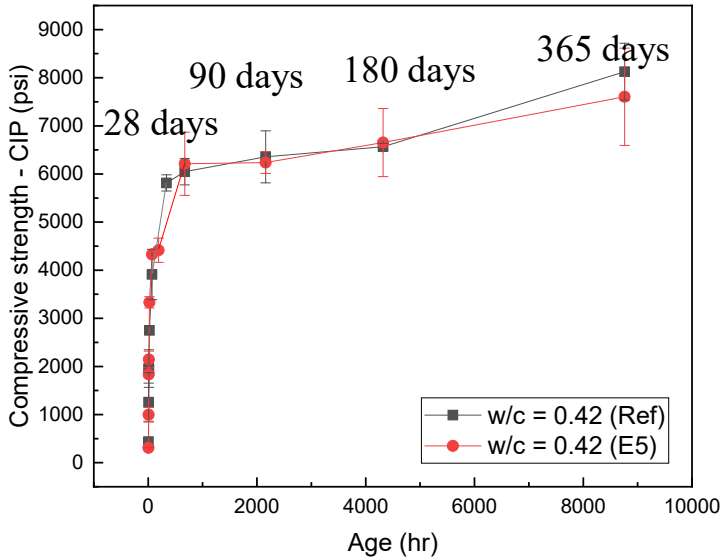


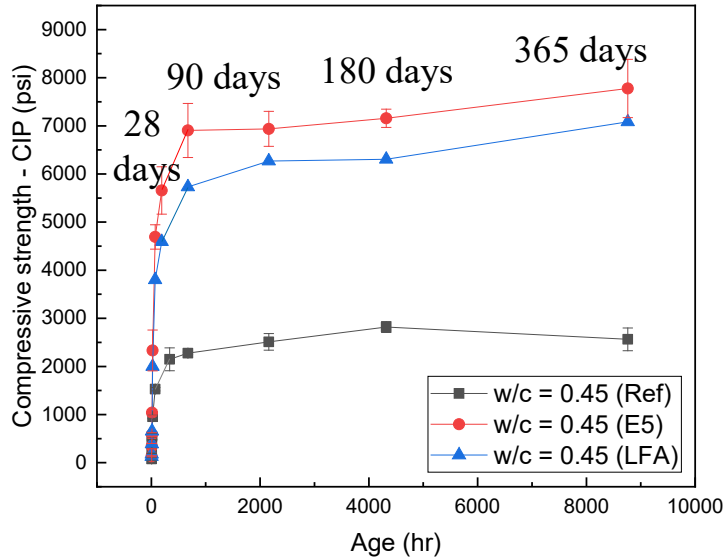


- It has observed the sample with E5 still increase (400-1000 psi) in compressive strength for 90 days

Large slab testing results – CIP mold



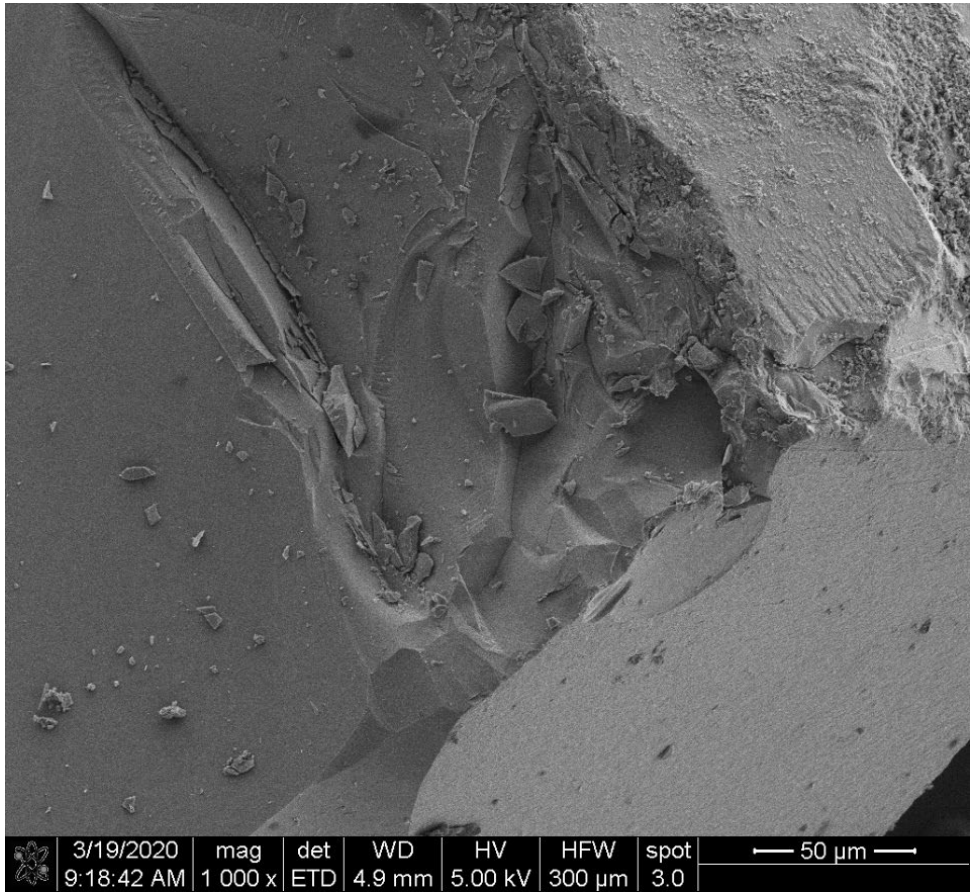


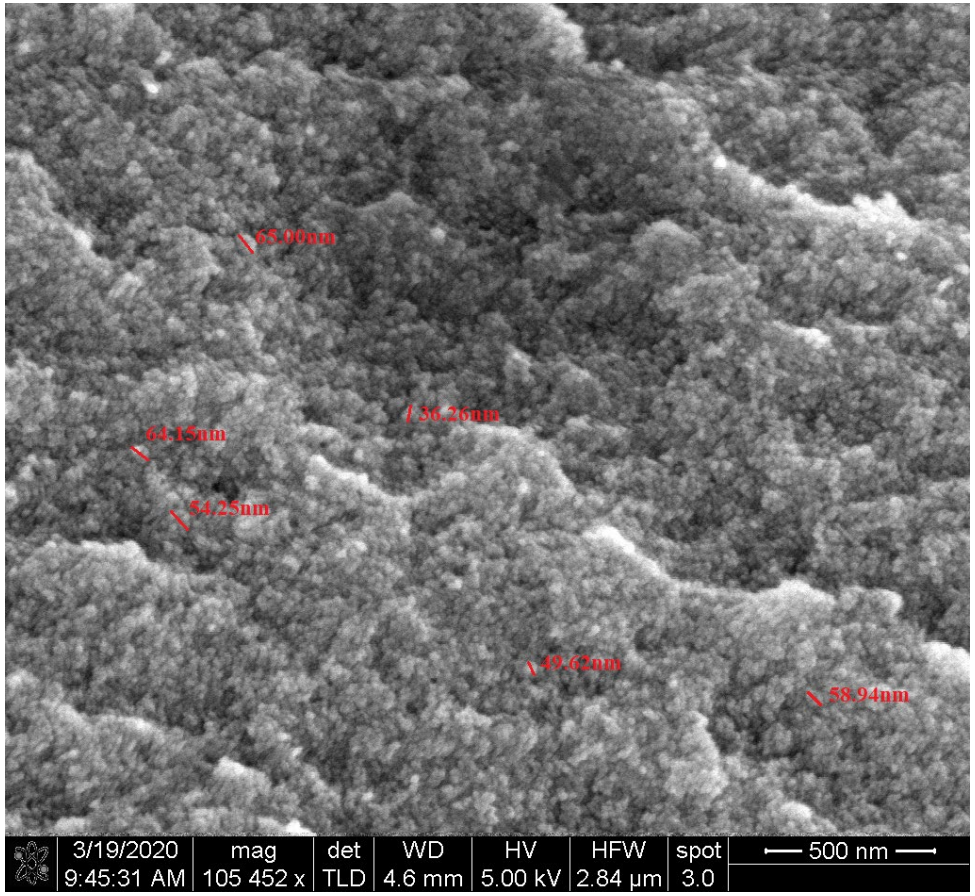


- It has observed the sample with E5 and LFA continuously gaining strength for 365 days

Properties of E5

Scanning Electron Microscope (SEM)

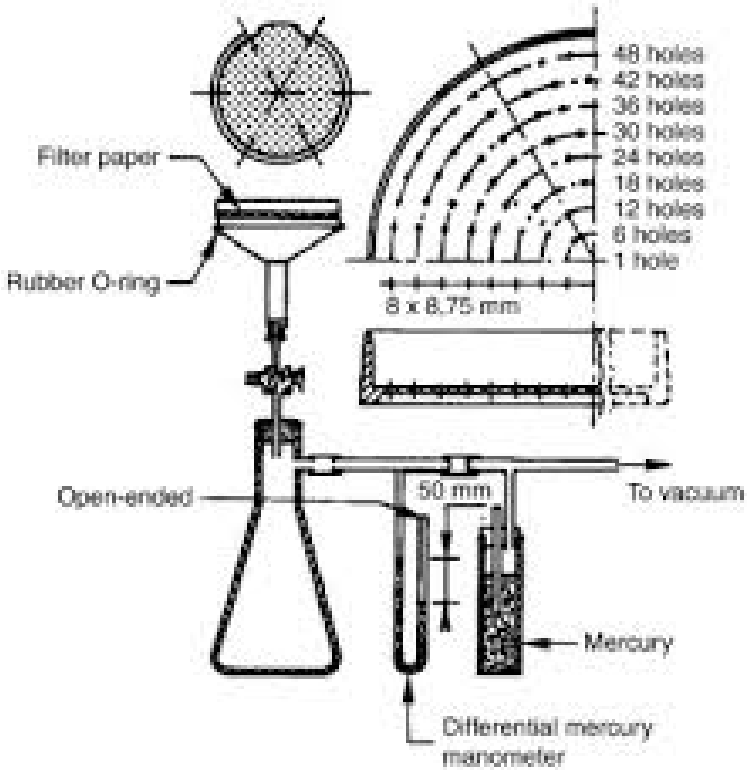




Average size: ~54nm

- If we use the immersion mode of SEM (higher resolution), we can see the nano-sized particles on the surface.

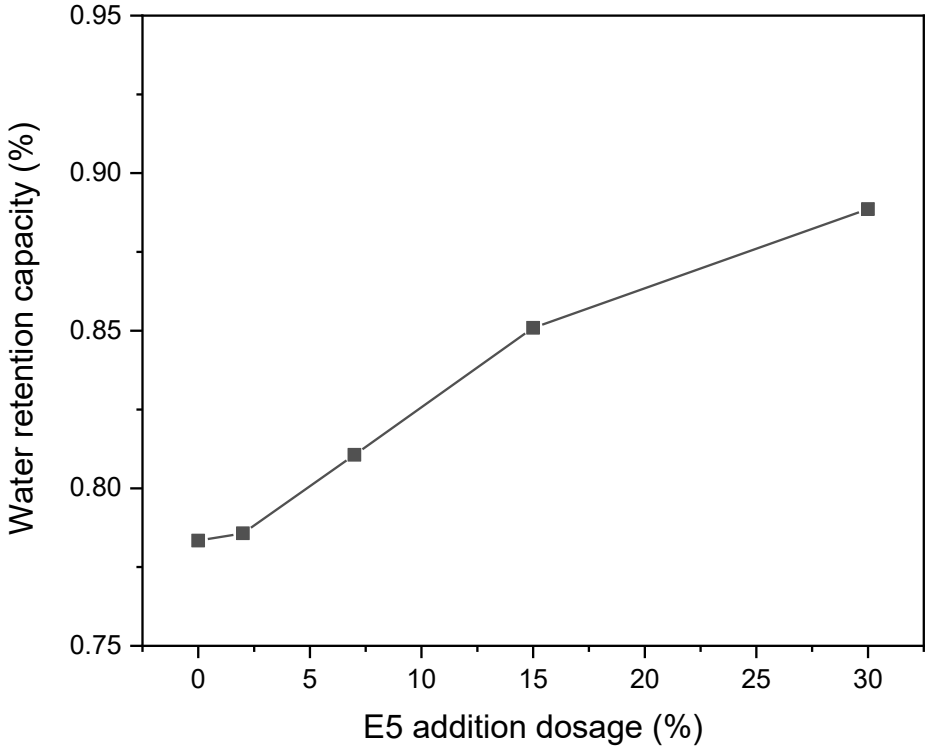
Water Retention Capacity



ASTM C91 standard

- Vacuum pumping method is a standard method to measure the water retention capacity of fresh cement paste;
- Higher water retention capacity usually means that the formed flocs in fresh cement paste has higher capacity to hold water inside its structure;

Water Retention Capacity



- E5 is promoted as internal curing agent, indicating that it can resist the excessive water loss during curing.
- According to the results, we can see that the water retention capacity was greatly enhanced by E5, even at cement's fresh state.

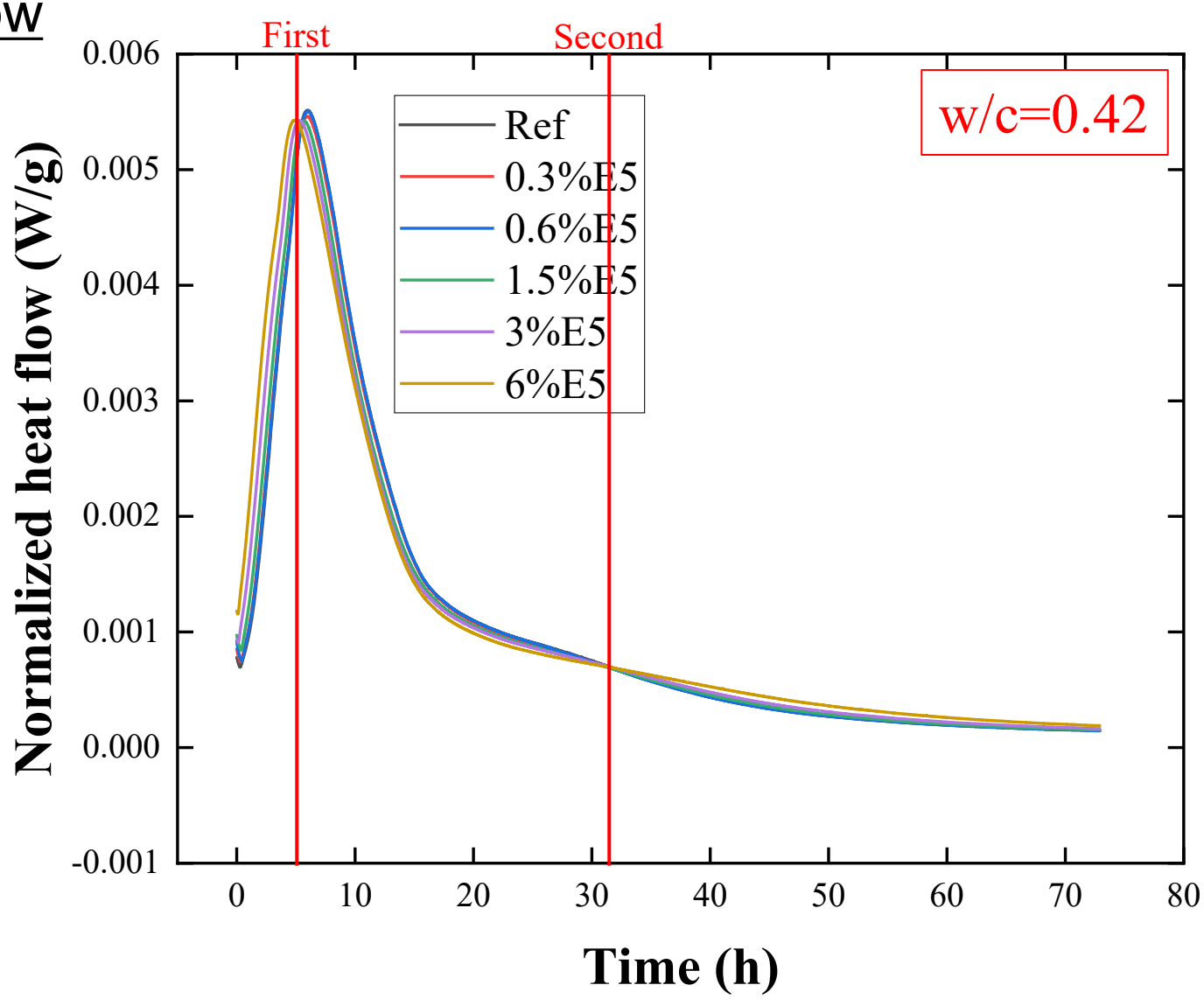
Effect of E5 on concrete performance

Isothermal Calorimetry (IC) Test for E5 Sample

- E5-Internal cure dosage: 0, 0.3%, 0.6%, 1.5%, 3%, 6%; w/c=0.42
- Experimental data: 3 days heat flow and cumulative heat curve.

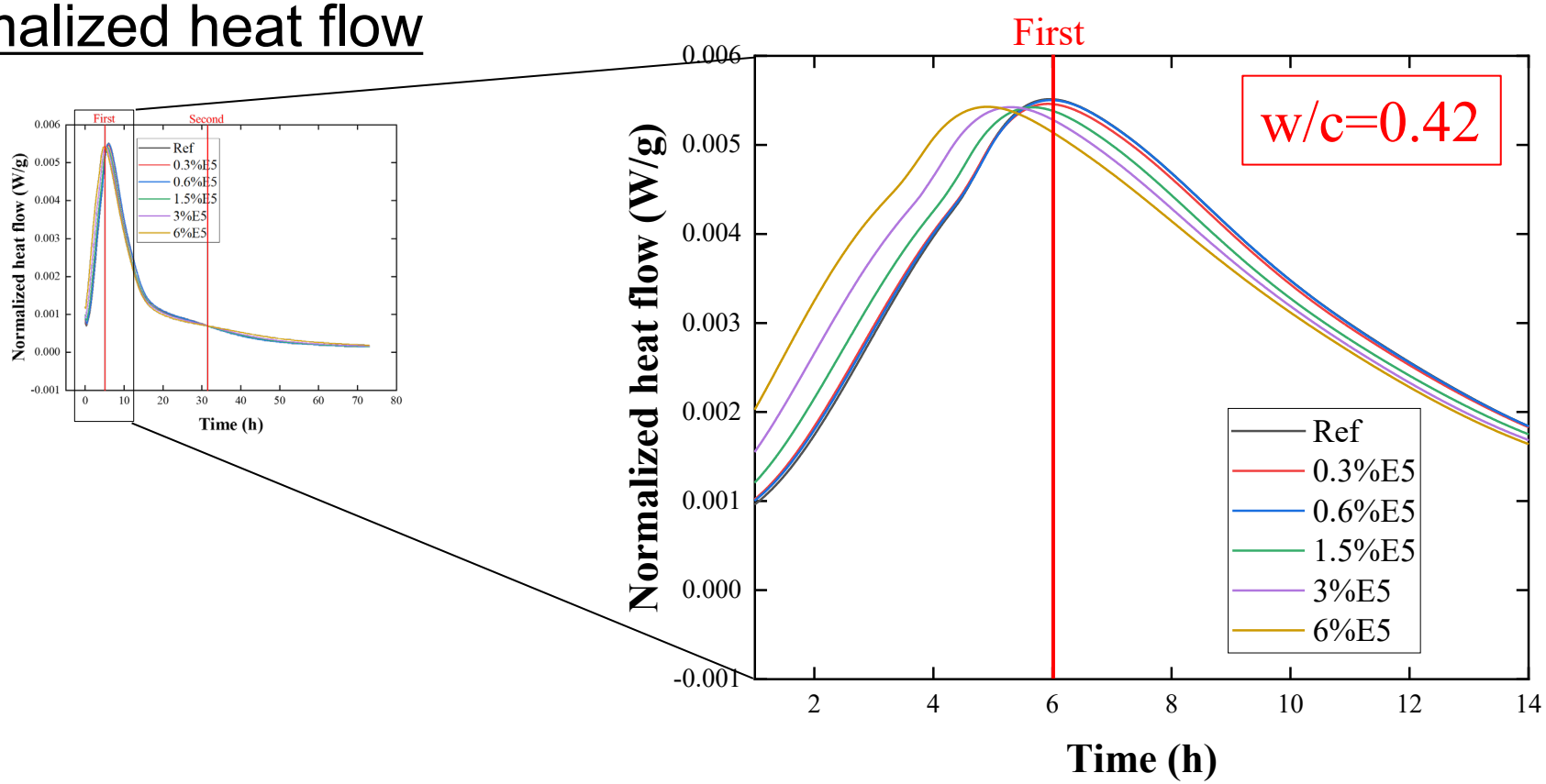
Isothermal Calorimetry (IC) Testing-result

Normalized heat flow



IC Testing

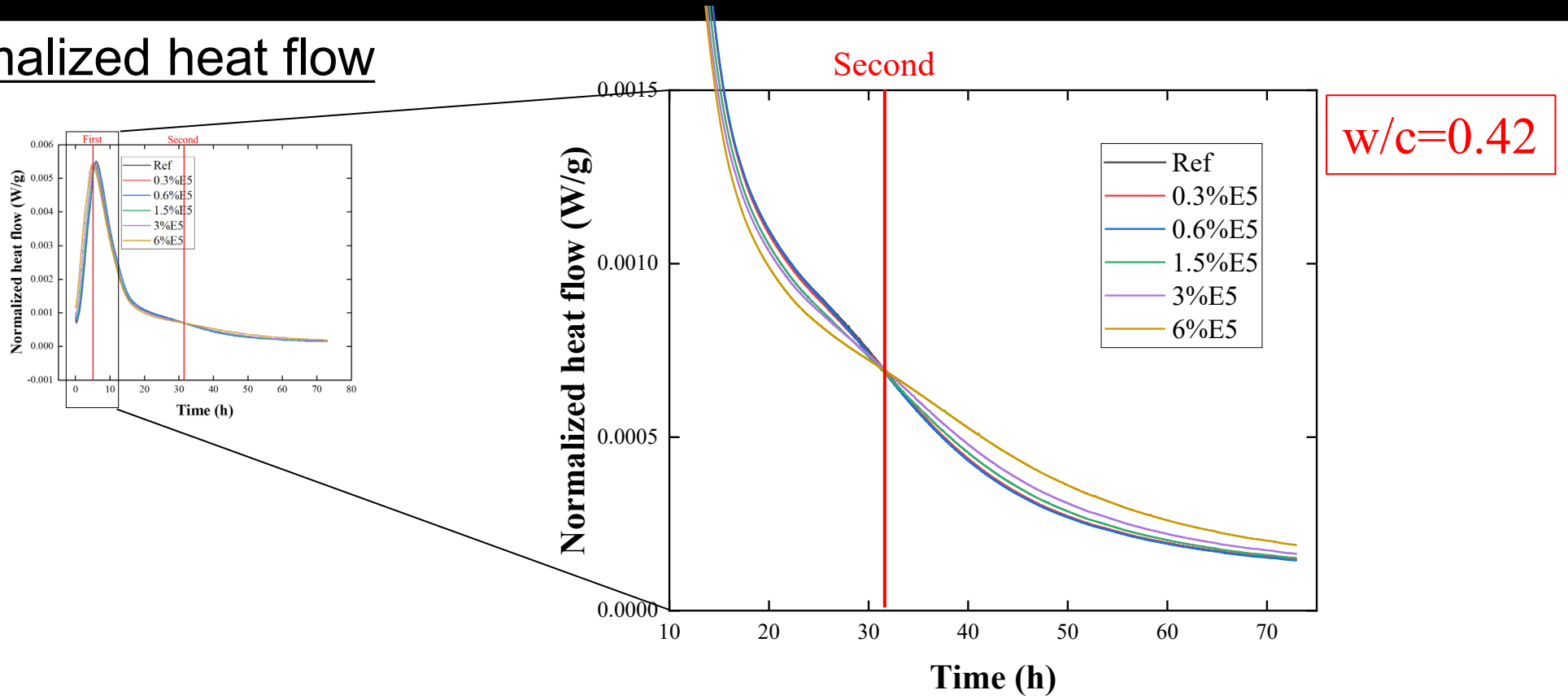
Normalized heat flow



- It can be seen from the figure that the peak of the heat flow curve shift to left as the E5 dosage increased, which represent a faster setting of the mixture. It can be due to the seeding effect of the nano particles increase the average surface area.

IC Testing

Normalized heat flow

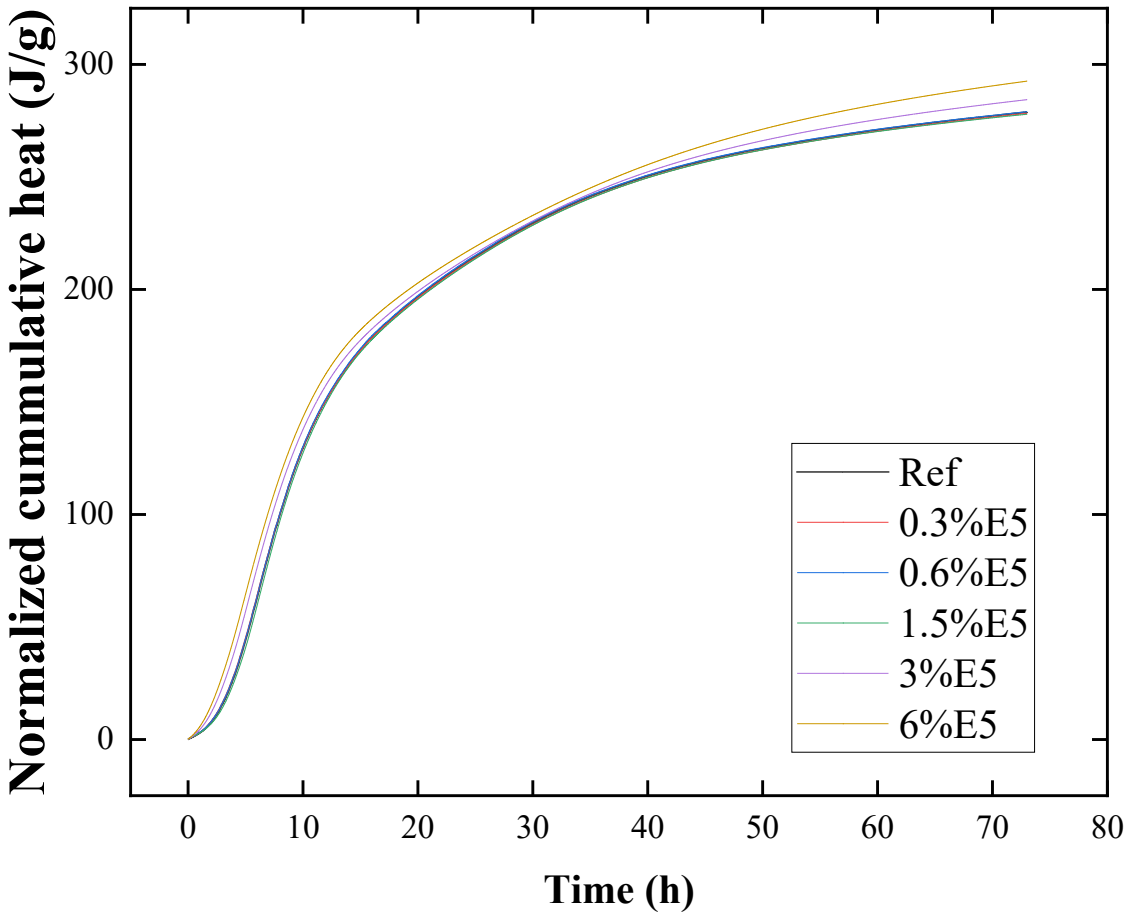


w/c=0.42

- After around 32 hours, after a short converge of the curves, sample with higher dosage of the E5 shows a higher heat flow. This may be due to the water that was holding by the E5 been released and improved the hydration.

IC Testing

Normalized cumulative heat



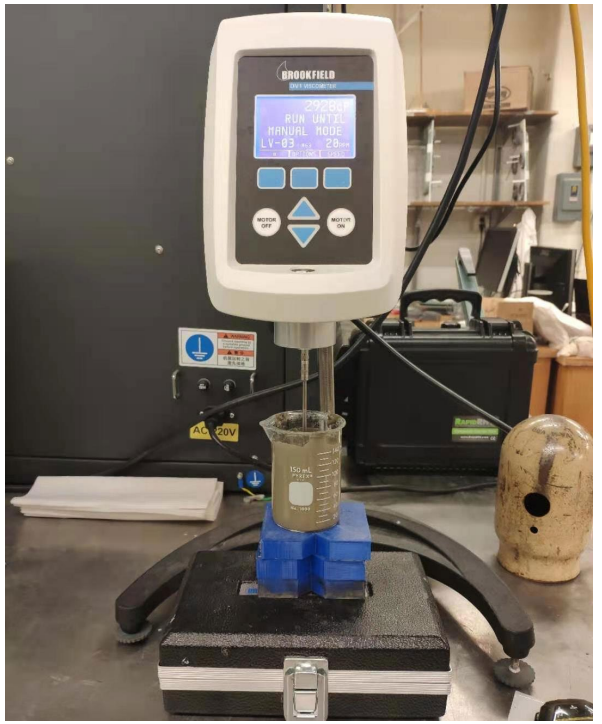
w/c=0.42

- Overall, the result indicates that the E5™ increase the early hydration rate.

Viscosity Evaluation with E5

Experimental design

- Different dosages of the E5-Internal cure were tested with cement (w/c=0.42);
- Procedure: Cement plus water, mix for 2mins, add E5 and mix for 1 more minute, start testing at 4min, first data record at 5min.
- According to SP company, no water adjustment is needed when E5 product is used. Therefore, the viscosity of the mixtures are designed without water adjustment.

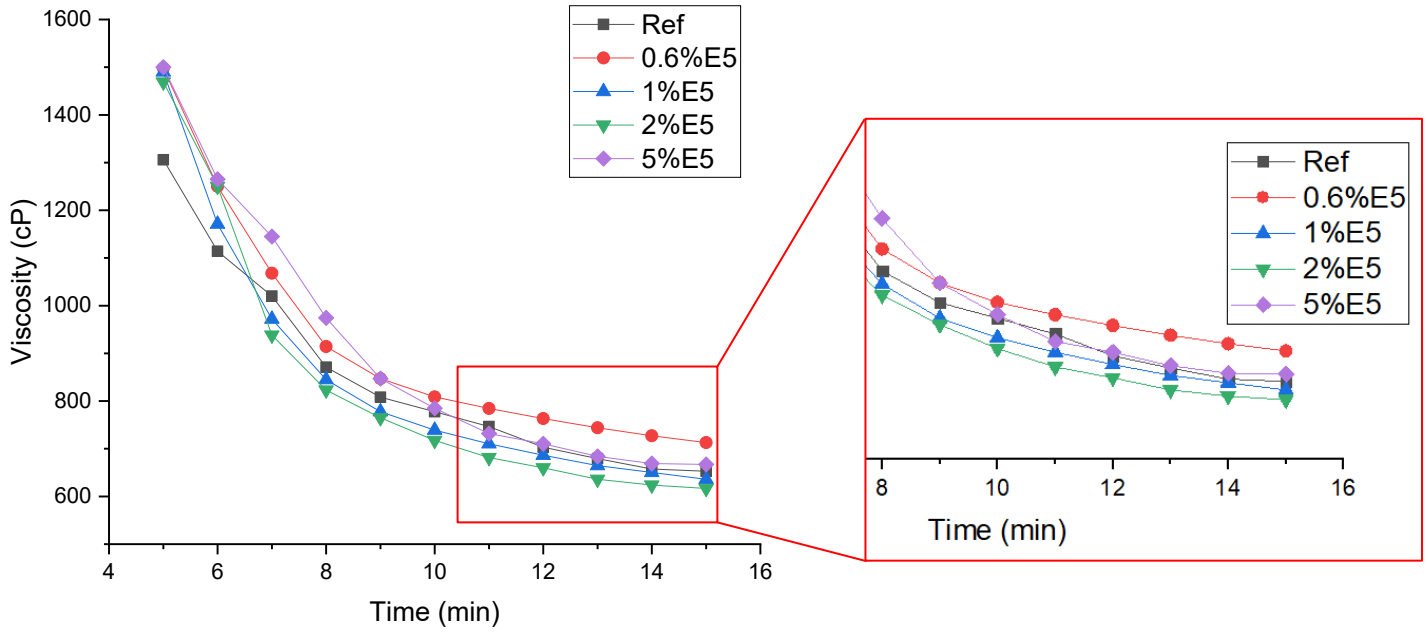


Viscosity Evaluation with E5

Cement paste (mixing water no adjusted)

w/c=0.42

Mixing water was NOT adjusted



- The result indicates that with no water adjustment, the viscosity was only slightly affected by the E5.
- When 1% and 2% of the E5 was used, the viscosity of the paste was decreased. For higher dosage, the viscosity was increased when 5% of the E5 was used.

Concrete Performance Evaluation

Concrete design:

Cement (Type I) (lb/cyd)	w/c	FA (lb/cyd)	CA (lb/cyd)	CA/FA ratio	FA %	CA %
564	0.42	1344	1800	1.34	0.43	0.57

*FA: fine aggregate, CA: coarse aggregate

Purpose:

- Very early age (8~12 hrs.) EMI testing;
- 1, 3, 7, 28 and 90 days mechanical test;
- Air content and density evaluation

Experimental plan:

- E5 product dosage: 0, 0.3%, 0.6%, 1.5%, 3.0% (by weight of cement);
- Sample preparation: Cylinder.

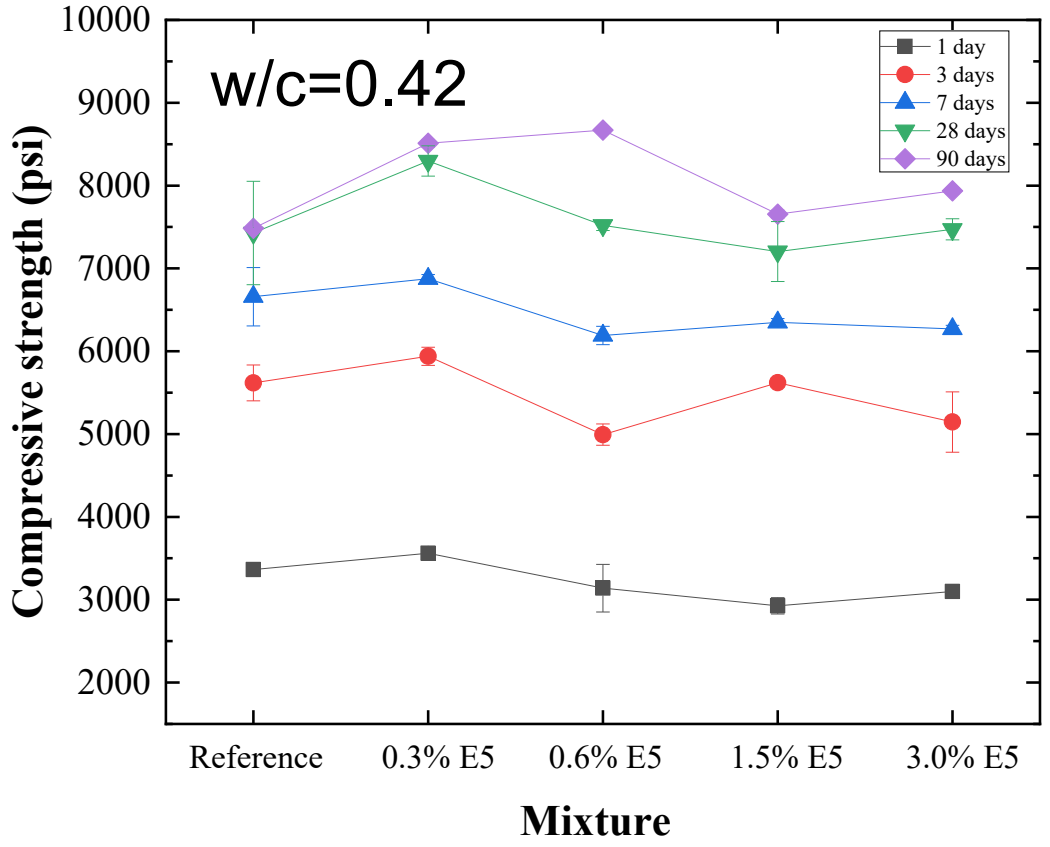
Air Content & Density

Air content and fresh concrete density (very early age) w/c=0.42

Mixture	Ref-0%E5	0.3%E5	0.6%E5	1.5%E5	3.0%E5
Air content (%)	2.7	2.4	2.1	3.0	2.2
Density (kg/m^3)	2469.2	2485.0	2462.1	2440.7	2468.6

- The density and air content of the fresh concrete were tested right after the mixing;
- The use of the E5^{TM} did not significantly affect the slump, air content and the density of the fresh concrete;

E5™-Mechanical result (90 days)

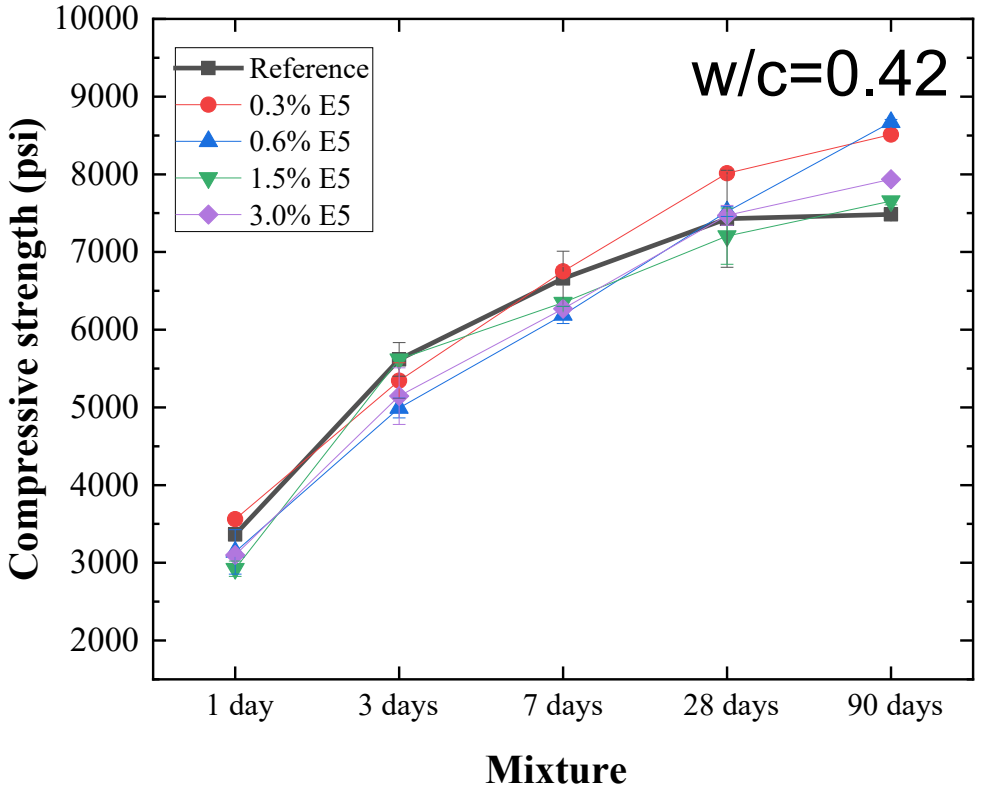


Each data point represents the average value of three tested samples.

- All the data in this figure came from mixtures using oven-dry aggregate.
- Overall, it can be observed that the addition of the E5-internal cure slightly increased the mechanical strength of the concrete under wet (moisture) curing.

*Wet curing: moist room at a temperature of 23℃ with relative humidity of not less than 95% - ASTM C192

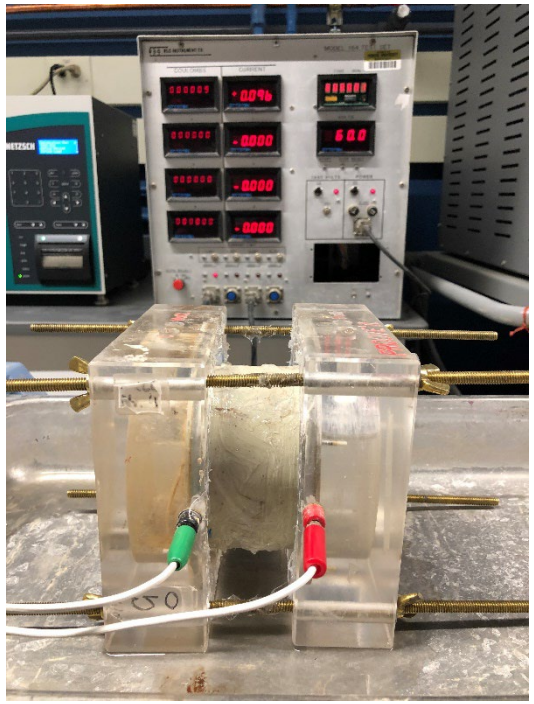
E5™-Mechanical result (90 days)



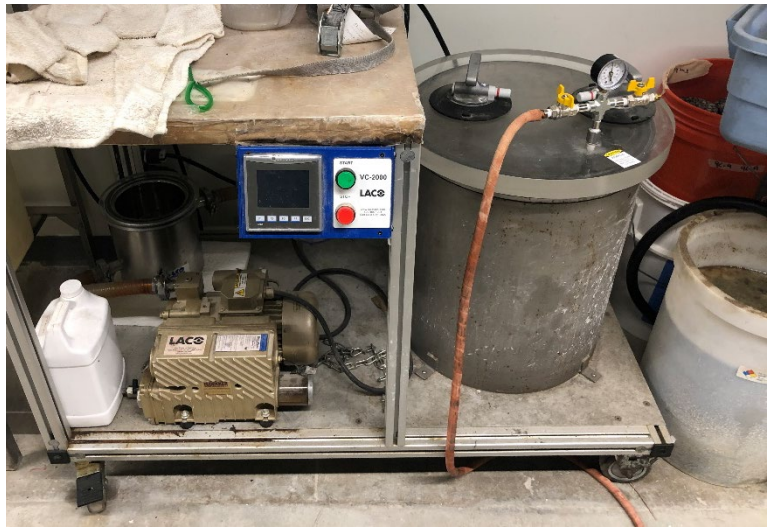
- It can be seen that sample without E5 had a slower strength increasing rate (**black dots**), compared with E5 samples.
- E5-internal cure may further improve the performance of the concrete at later age.
  - ❑ Pozzolanic reaction (Proved by TGA)
  - ❑ Better microstructure (proved by SEM)

Rapid Chloride Permeability Test (RCPT)

- Objective: evaluate the long-term (120 days) durability performance of concrete with E5 (0.3% - 3%)
- According to ASTM C1202, “Standard Test Method for Electrical Indication of Concrete’s Ability to Resist Chloride Ion Penetration”



RCPT setup

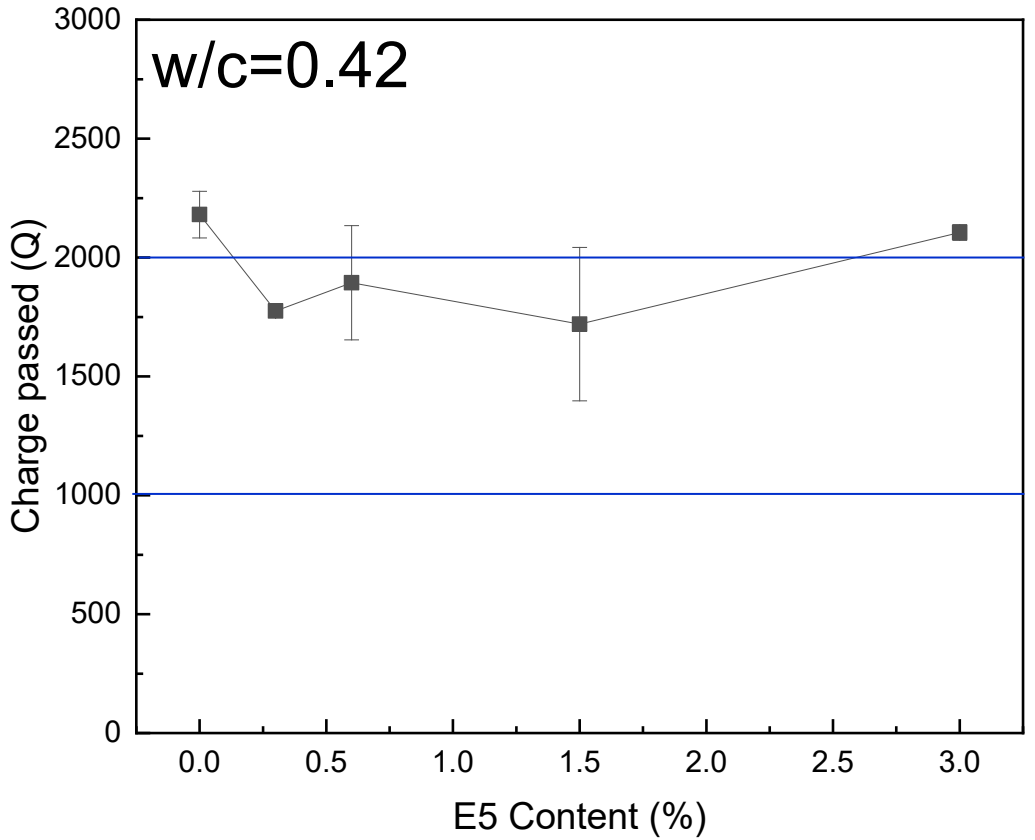


Concrete sample saturation system

RCPT results for samples with different E5%

Charge Passed (Coulombs)	Chloride Permeability	Typical of
>4,000	High	High W/C ratio (>0.60) conventional PCC
2,000–4,000	Moderate	Moderate W/C ratio (0.40–0.50) conventional PCC
1,000–2,000	Low	Low W/C ratio (<0.40) conventional PCC
100–1,000	Very Low	Latex-modified concrete or internally-sealed concrete
<100	Negligible	Polymer-impregnated concrete, Polymer concrete

ASTM-C1202 Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration



- The results indicated that the E5 content from 0.3%-1.5% can improve the long-term protection of concrete from chloride penetration.

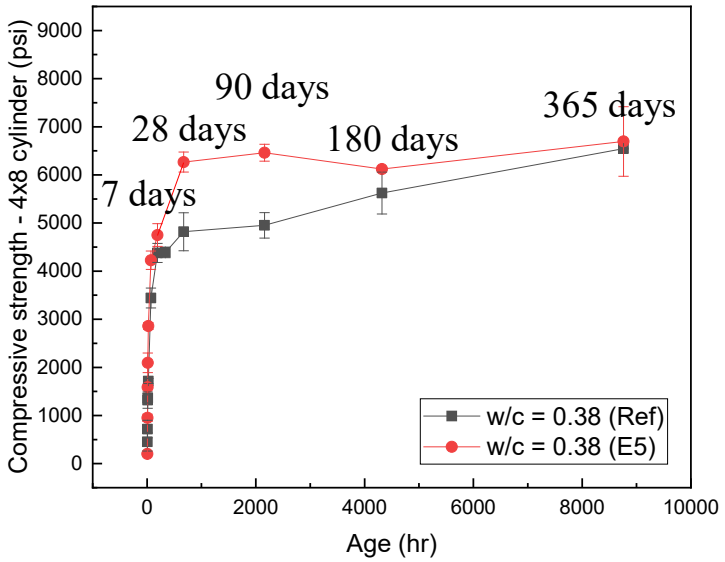
Large slab pouring and testing

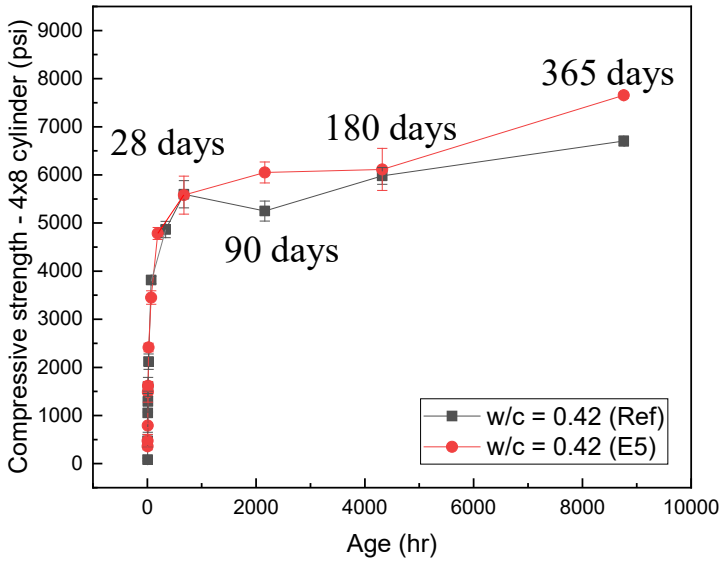
Slab testing - Mix design

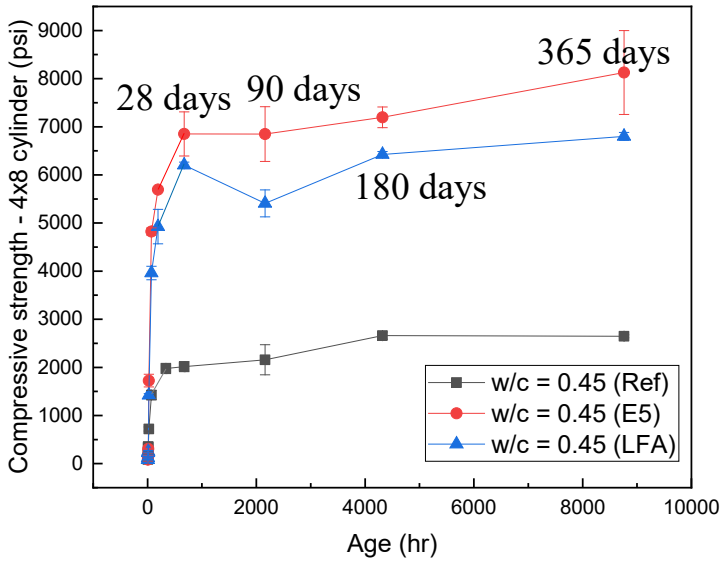
Mix design (lbs/3CY)

Slab	Cement	#8 Gravel AP	Natural Sand	Water reducer	Water (gal)	E5 (oz)
0.38_E5	1690	5780	4520	116	49.94	67.68
0.42_E5	1690	5780	4520	116	55.94	67.68
0.45_E5	1685	5680	4540	68	63.63	67.68
0.45_LFA	1405	5700	4580	56	46.82	112 (LFA)
0.38_Ref	1705	5760	4520	34	50.42	0
0.42_Ref	1745	5640	4480	67	54.98	0
0.45_Ref	1675	5680	4480	68	66.03	0

Large slab testing results – 4x8 mold

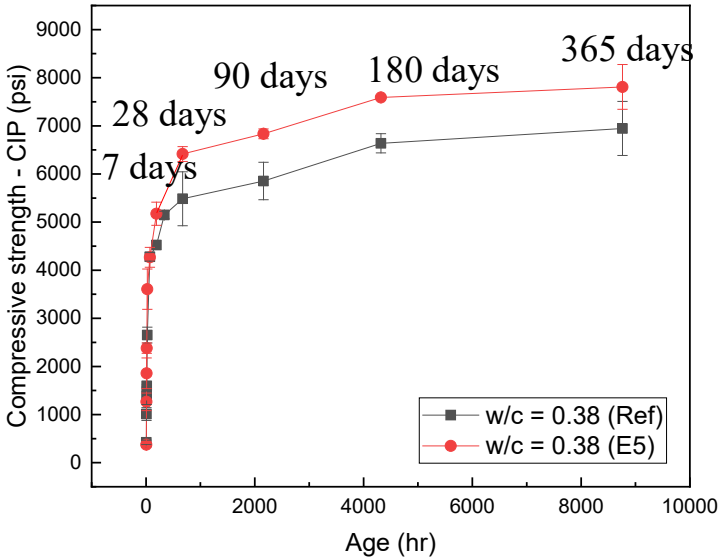


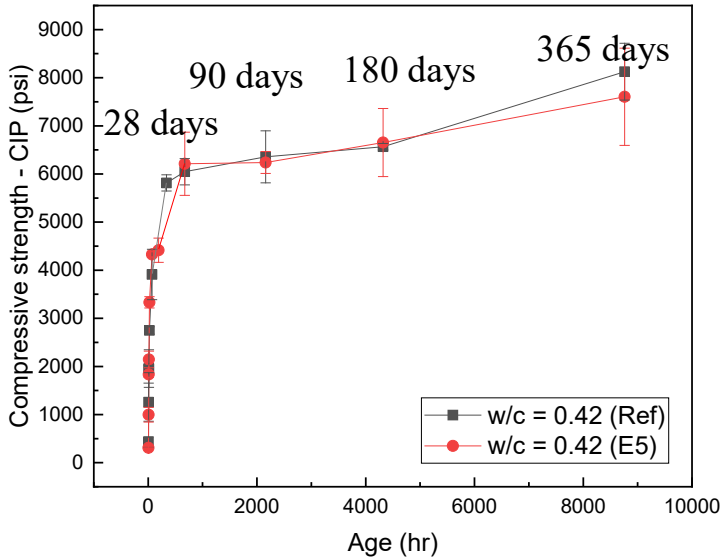


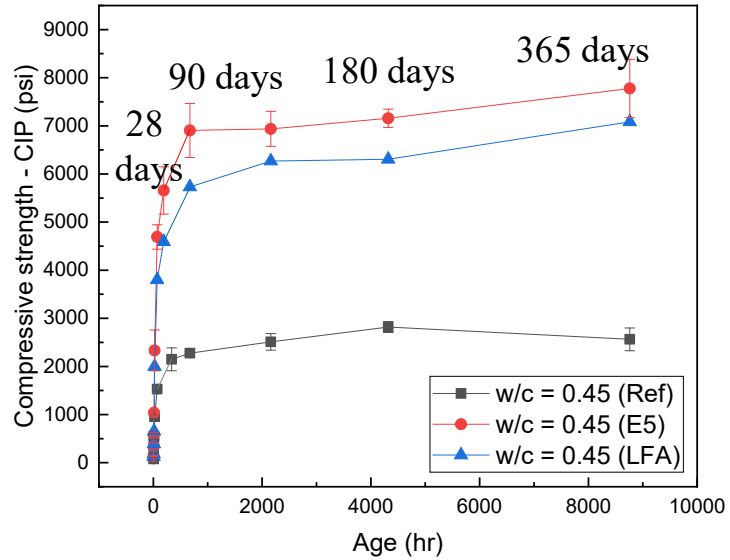


- It has observed the sample with E5 still increase (400-1000 psi) in compressive strength for 90 days

Large slab testing results – CIP mold

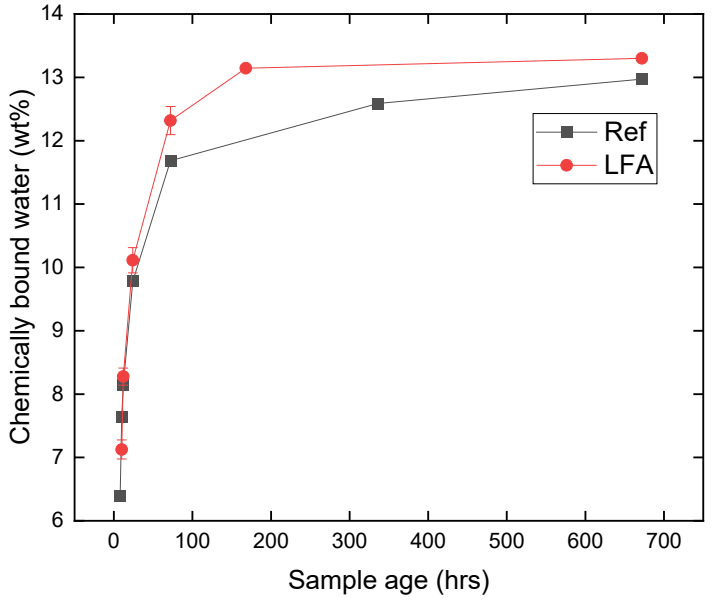


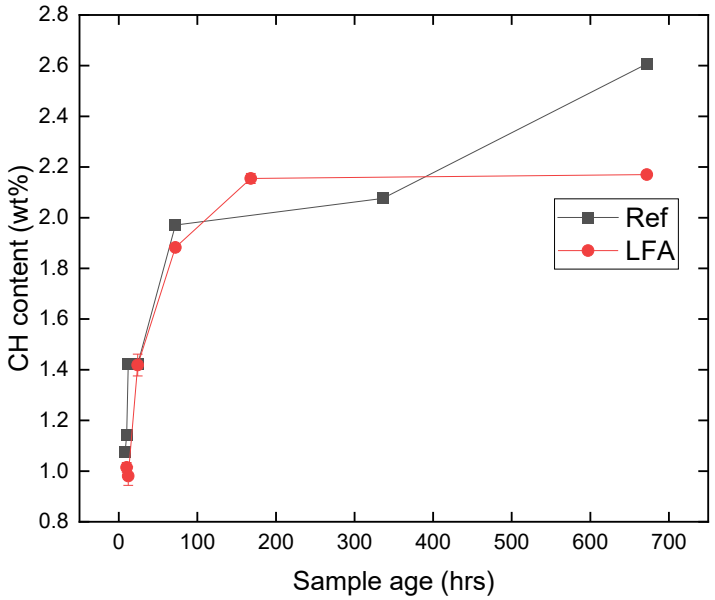


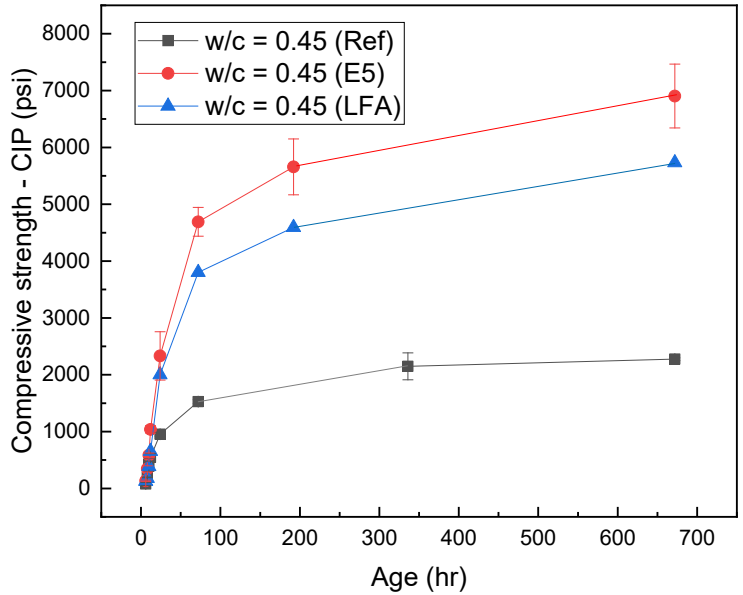


- It has observed the sample with E5 and LFA continuously gaining strength for 365 days

TGA Result







- The result of the chemically bound water indicates a higher hydration degree was achieved when LFA was used.
- The result of the CH content indicates a lower amount of CH in the sample with LFA, which may be due to the pozzolanic reaction.
- Compared with the reference slab, sample with LFA exhibited a higher chemically bound water content (indicates a higher degree of hydration), which is coincide with the mechanical test result.

Liquid Fly Ash (LFA)

Shrinkage testing-LFA

- Evaluate different dosages of the LFA and water to cement ratio on the **drying shrinkage** and **compressive strength** of the sample.

Mixture design

	Cement	Sand	w/c	LFA
Reference-0.47	1	3	0.47	0
0.3%LFA-0.47	1	3	0.47	0.003
0.6%LFA-0.47	1	3	0.47	0.006
1%LFA-0.47	1	3	0.47	0.015
Reference-0.50	1	3	0.50	0
0.3%LFA-0.50	1	3	0.50	0.003
0.6%LFA-0.50	1	3	0.50	0.006
1%LFA-0.50	1	3	0.50	0.015
Reference-0.45	1	3	0.45	0
0.3%LFA-0.45	1	3	0.45	0.003
0.6%LFA-0.45	1	3	0.45	0.006
1%LFA-0.45	1	3	0.45	0.015

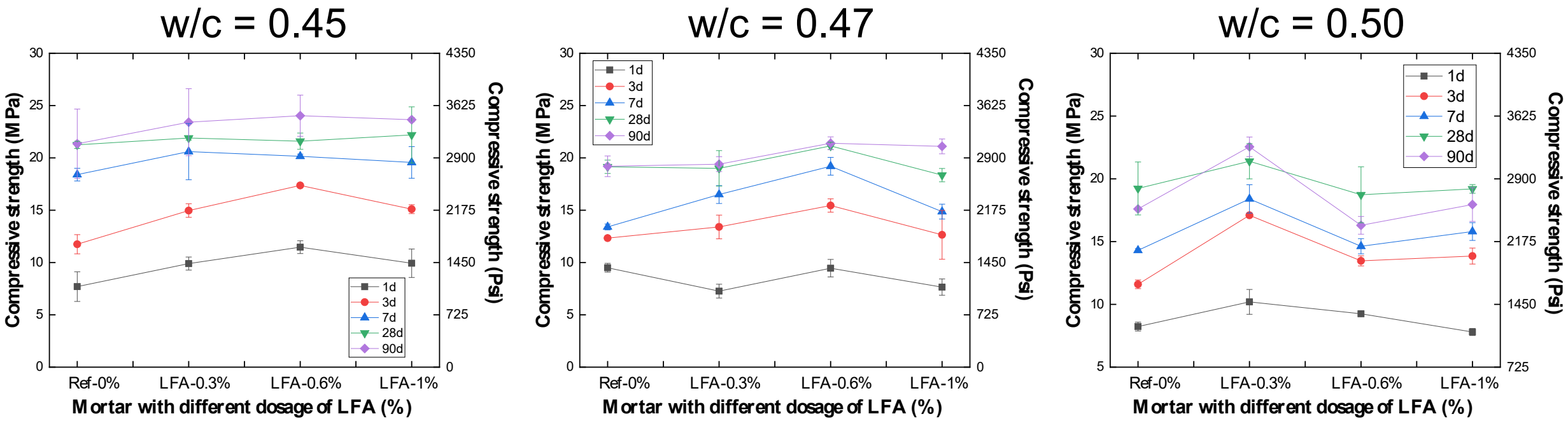
Shrinkage testing-LFA

- Evaluate different dosages of the LFA and water to cement ratio on the **drying shrinkage** and **compressive strength** of the sample.

Samples and setup for drying shrinkage measurement

Compressive strength

Mortar with LFA



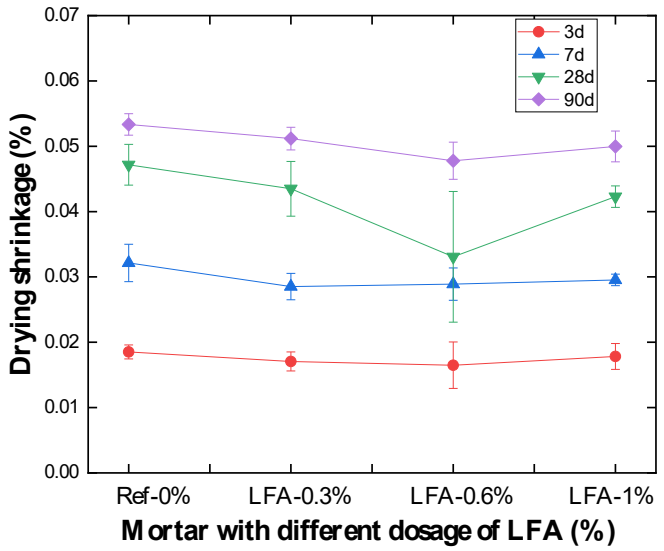
1. The incorporation of 0.3% and 0.6% LFA improved the compressive strength especially at the age of 3 and 7 days;

2. Mixtures without LFA didn't show significant strength development from 28 days to 90 days.

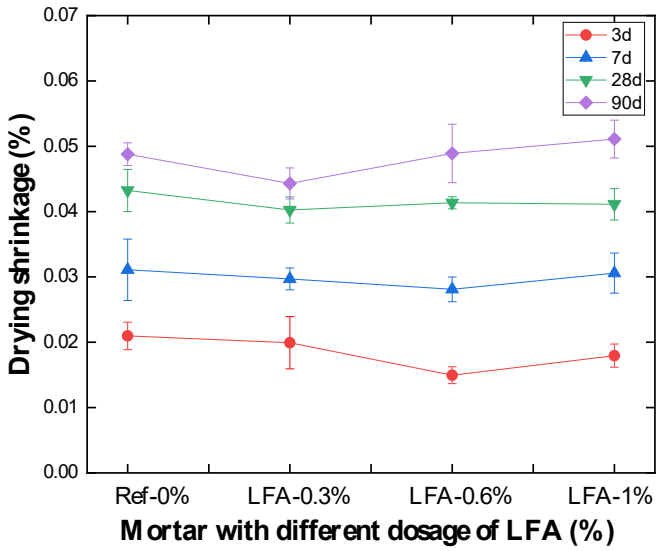
Drying shrinkage

Mortar with LFA

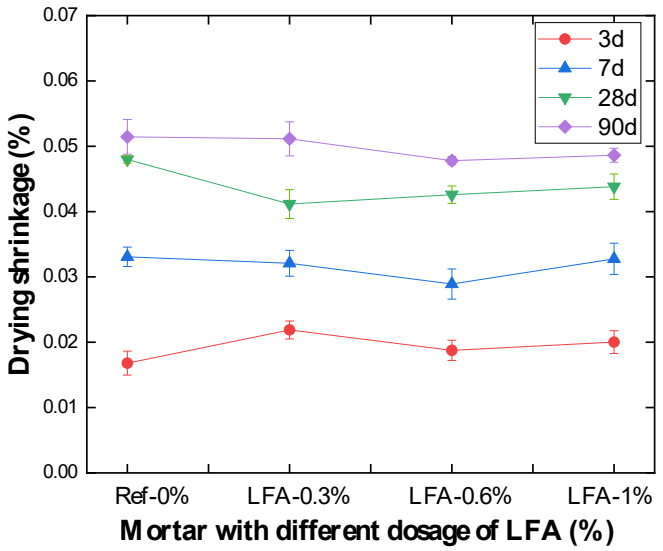
w/c = 0.45



w/c = 0.47



w/c = 0.50



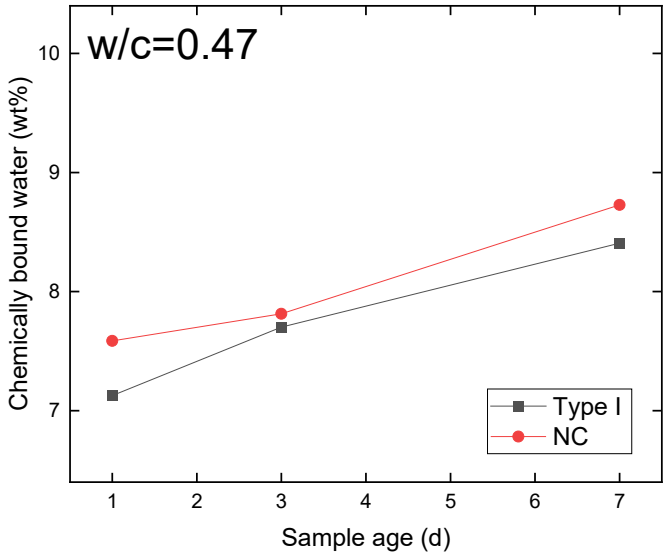
- At w/c of 0.45 and 0.47, the use of the LFA slightly decreased the drying shrinkage of the mortar (dosage of 0.3 and 0.6%).
- At w/c of 0.50, the LFA did not significantly affect the drying shrinkage.

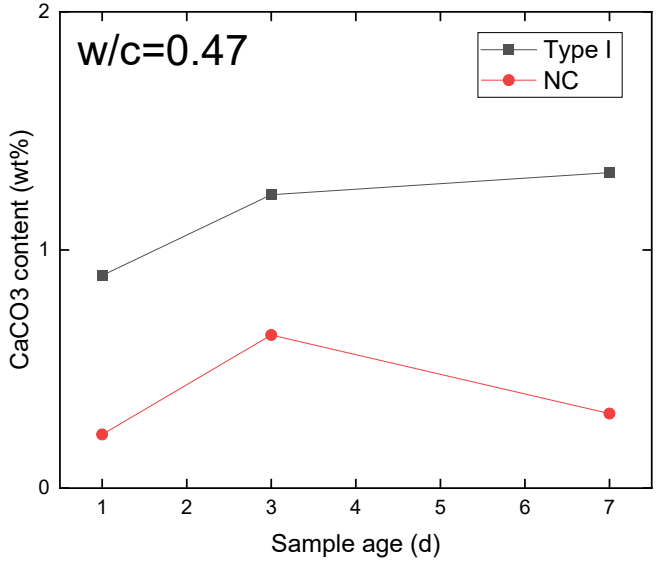
Cement paste evaluation-LFA

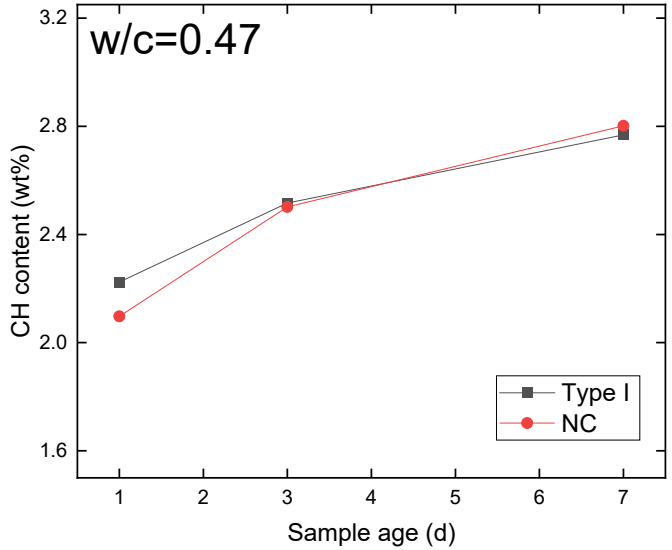
- Cement paste with the incorporation of LFA was evaluated by TGA
- Cement type: Type I and new cement (cement provided by SP company)
- LFA dosages: 0, 0.3%, 0.6% and 1%
- w/c: 0.47

Type I CEM vs New cement (NC)

❑ TGA result for pure cement paste with w/c of 0.47 (Type I vs NC)



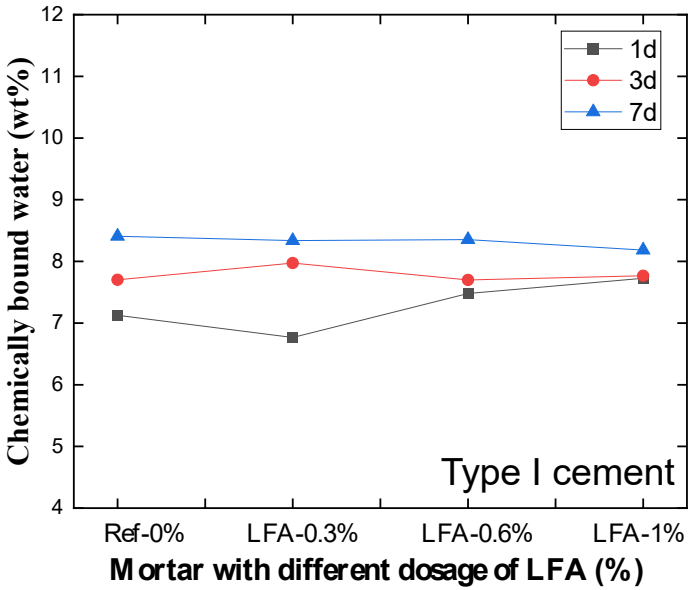


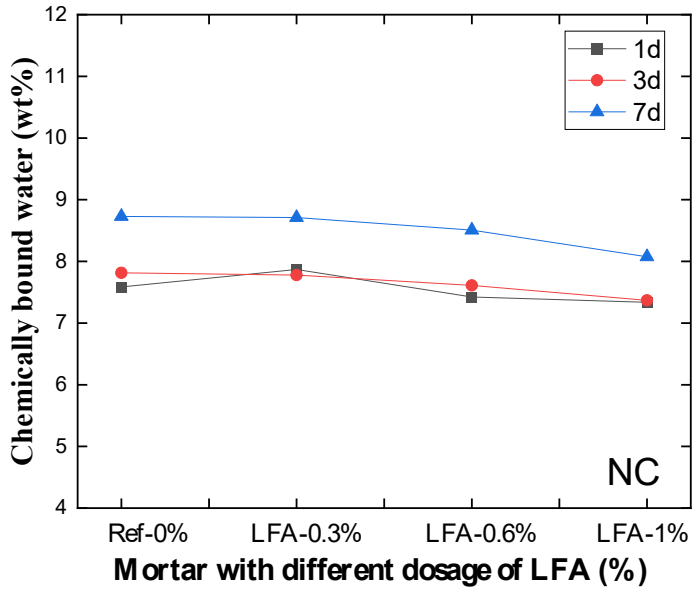


- NC has a higher reactivity;
- The use of the NC can result in a less amount of carbonation.

* The new cement is the cement that provided by the SP comp

TGA result

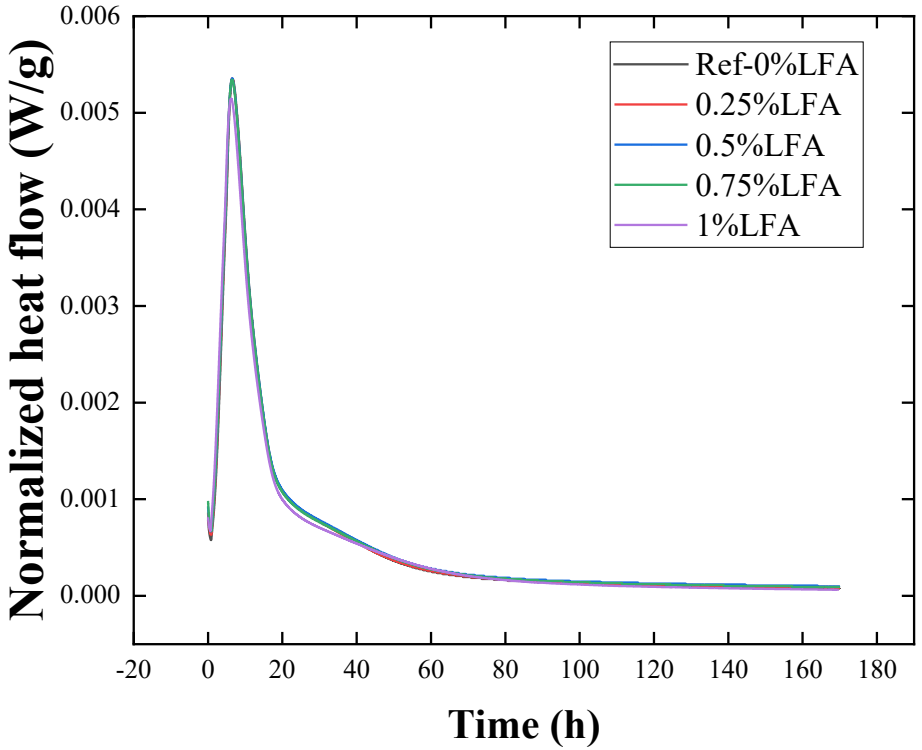


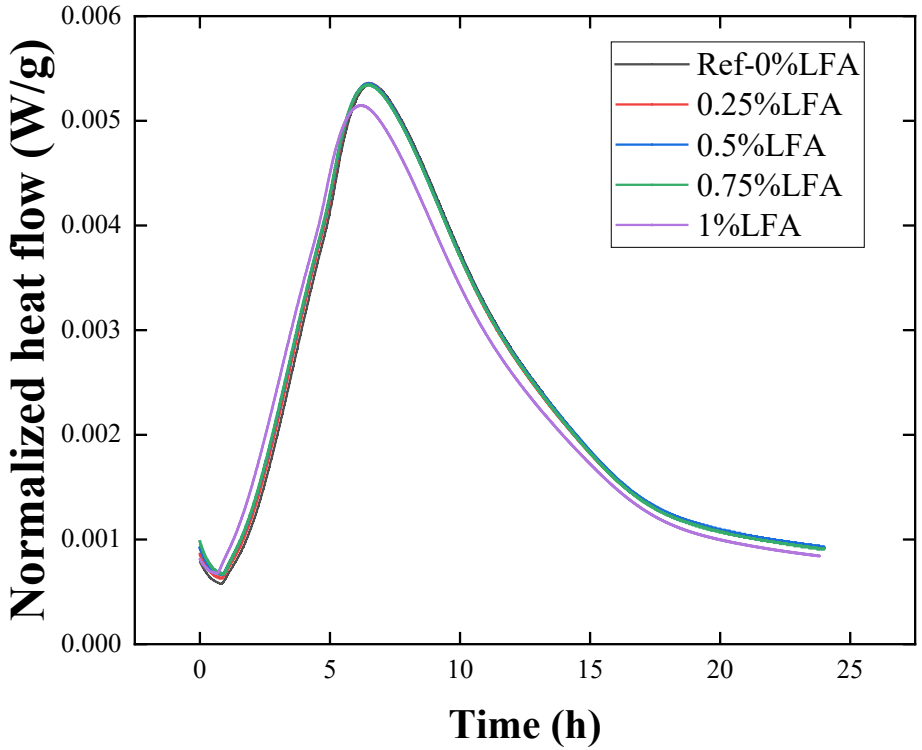


- The addition of the LFA did not significantly affect the hydration degree of the cement;
- The use of the NC resulted in a higher content of chemically bound water.

* The new cement is the cement that provided by the SP company

IC Result for Cement Paste with LFA

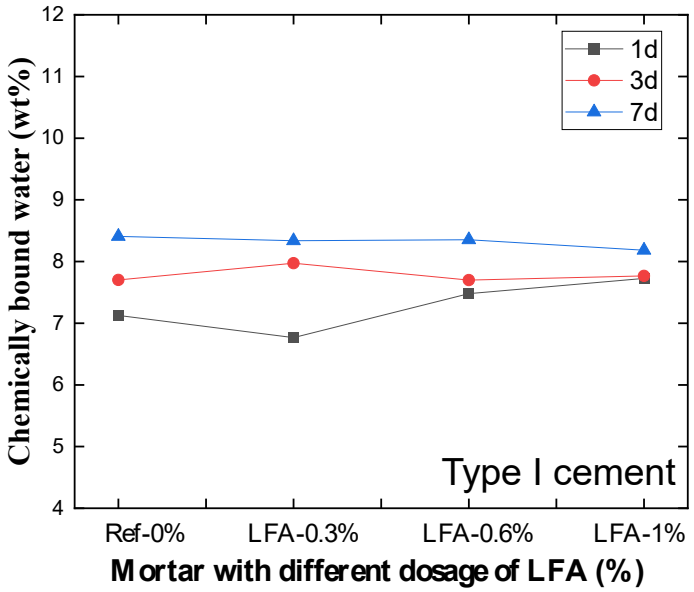


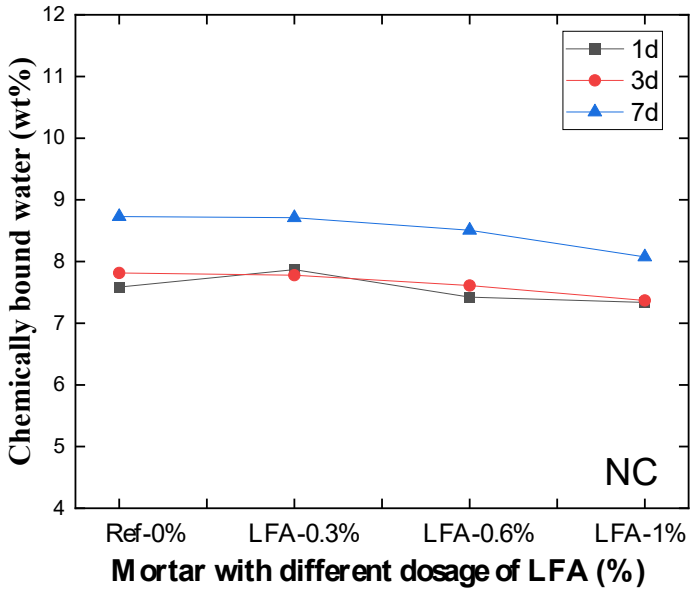


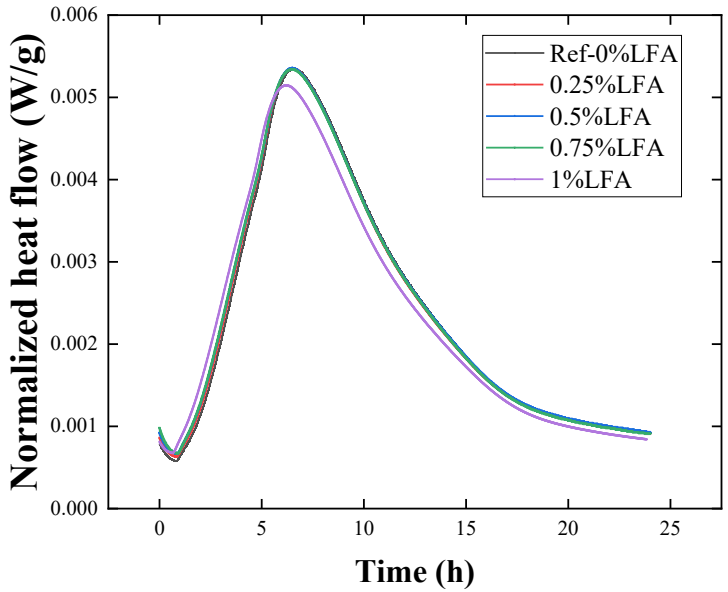
- The IC testing result indicates that the addition of LFA may increase the very early age (0~6 hrs) hydration rate.

TGA result

Discuss with IC result



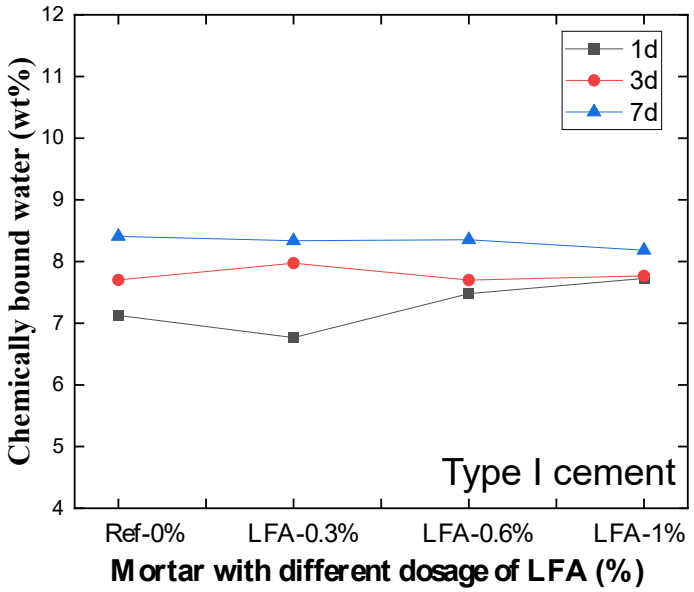


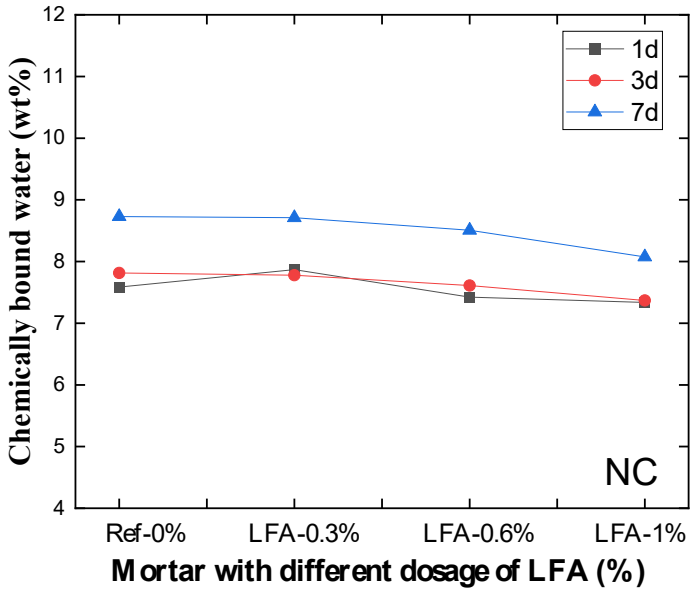


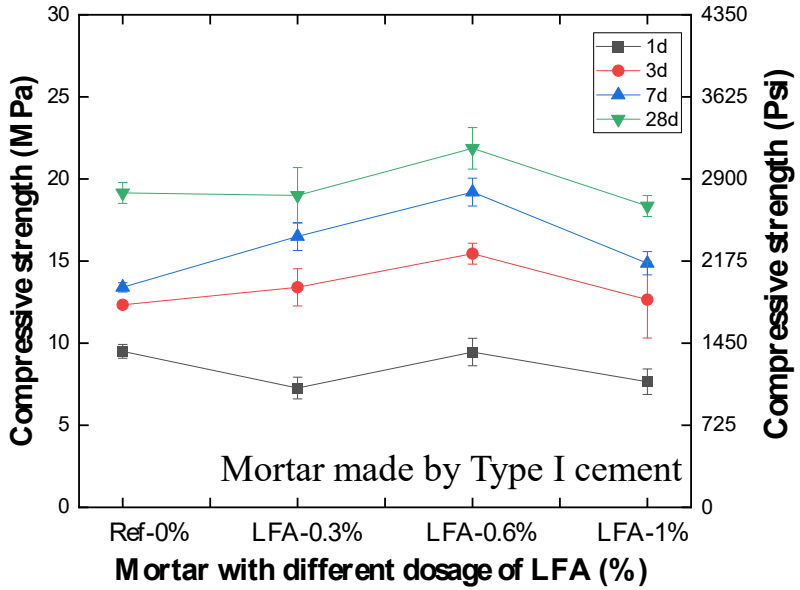
- From the result of the IC testing, it can be notice that the heat flow for mixture with LFA almost overlapped with the Reference sample. This confirms the TGA result that the incorporation of the LFA would not significantly change the hydration of the cement.

TGA result

Discuss with mechanical test result





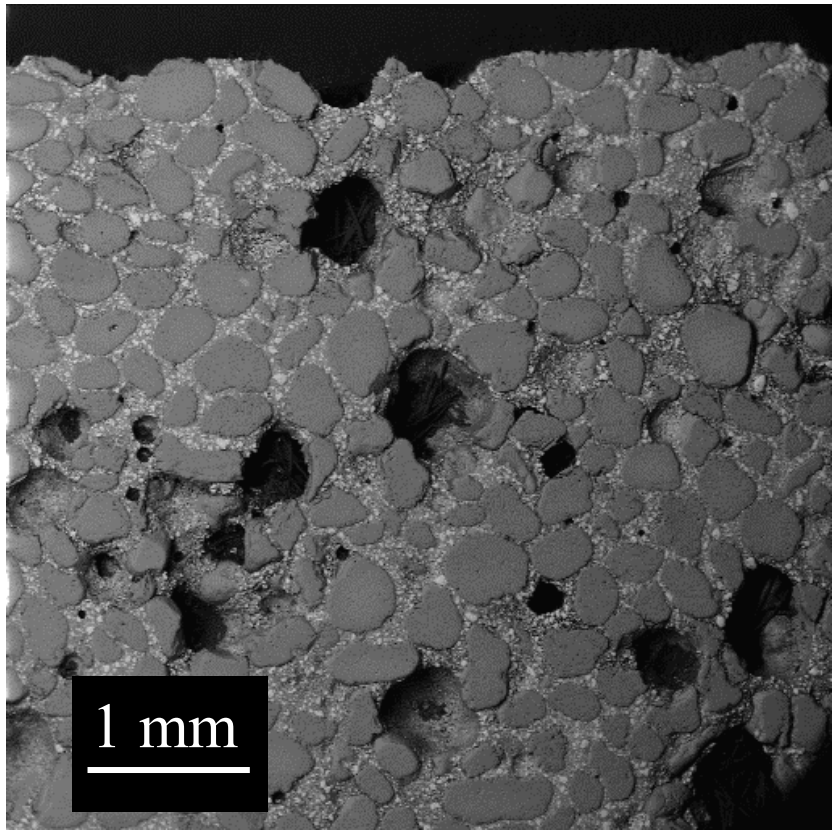


- In the mechanical test result, we noticed the incorporation of the LFA improved the strength of the mortar. Since the IC and TGA tests suggest the hydration degree of cement didn't change, the increase of the strength may be due to the improvement of the pore structure and ITZ.

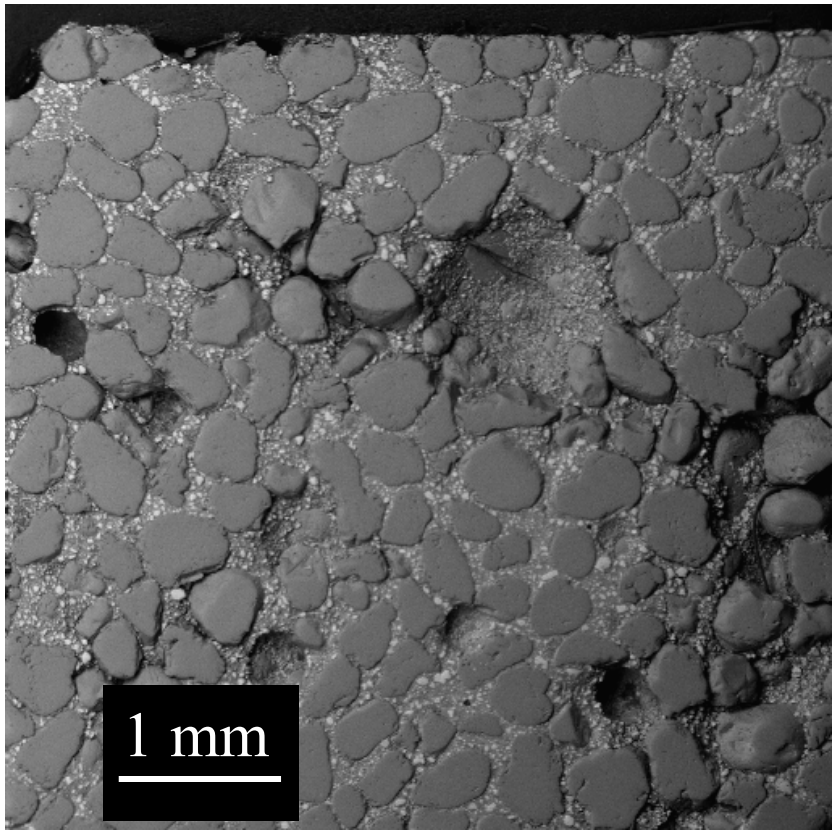
- SEM analysis can be conducted to further analyze the mechanism.

SEM of the mortar sample

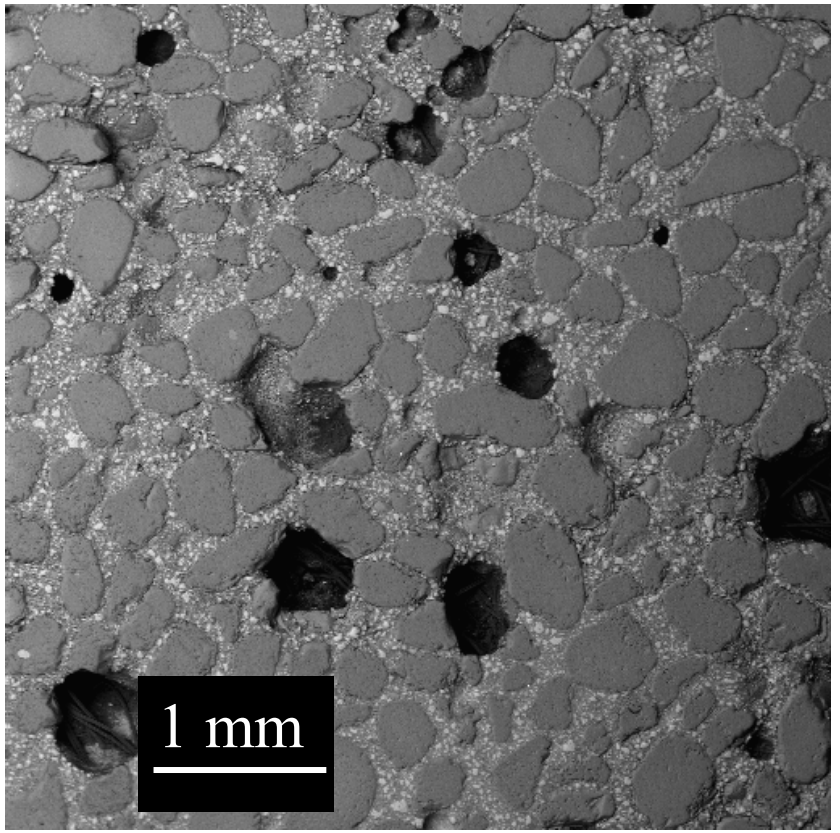
Reference



0.3% LFA



1% LFA

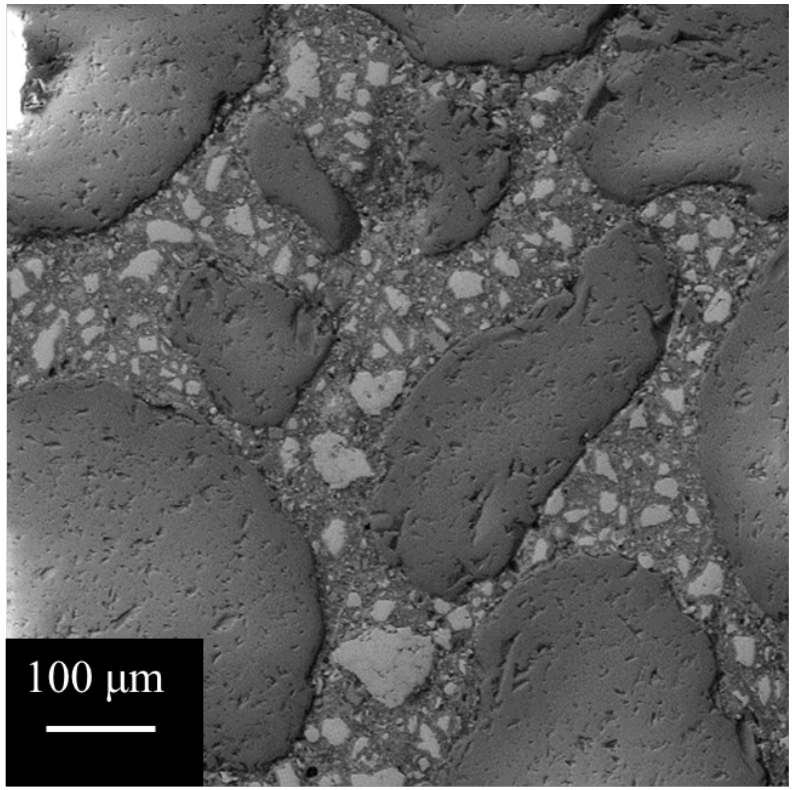


w/cem = 0.47, at 7 days

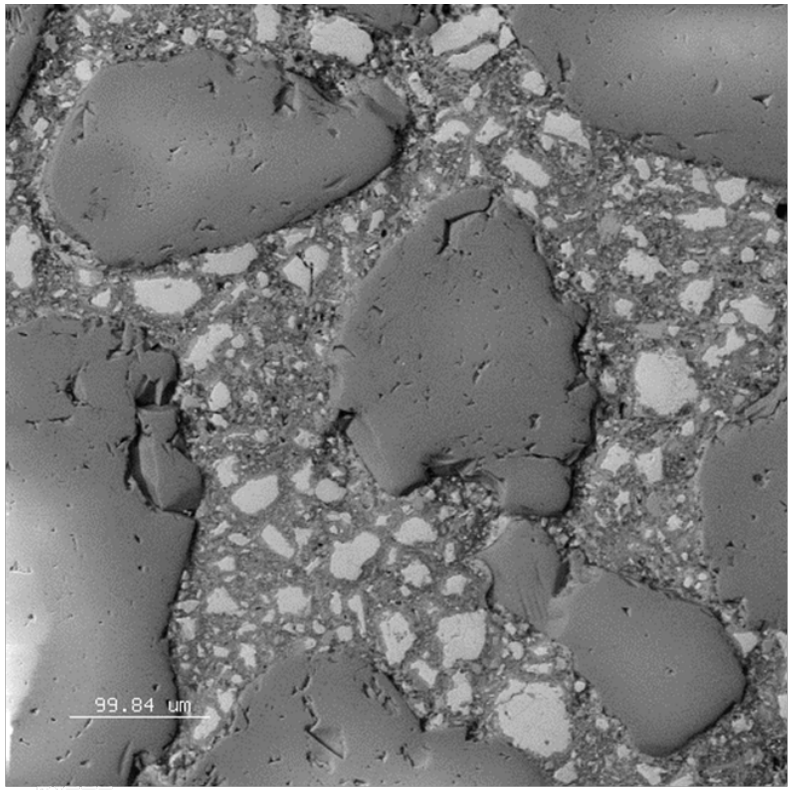
- General view of the matrices of the samples at low magnification.
- The observation showed that the matrix of LFA 1% sample seems to be much denser.

SEM of the mortar sample

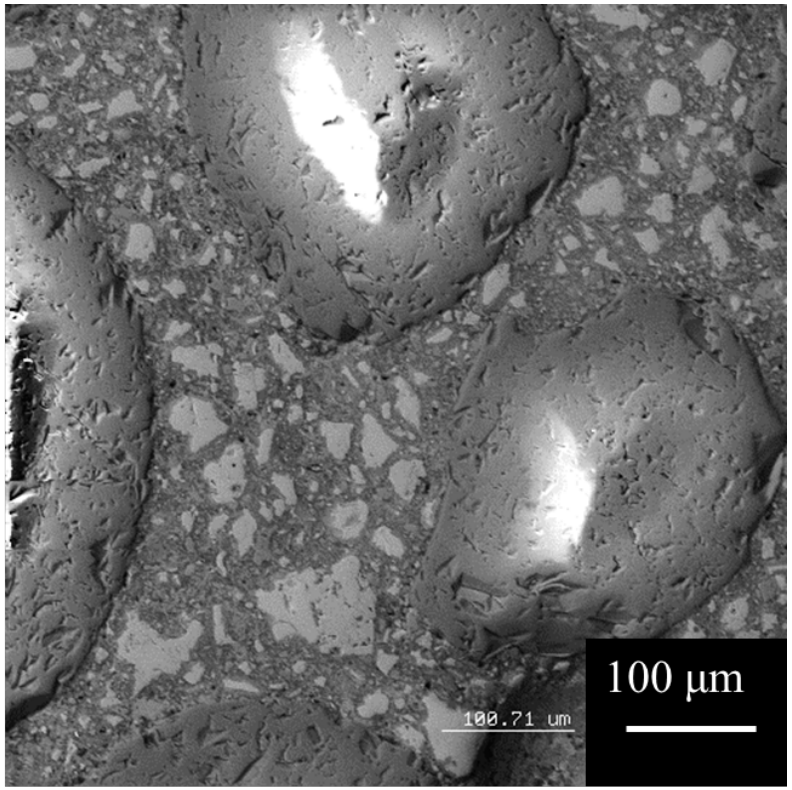
Reference



0.3% LFA



1% LFA

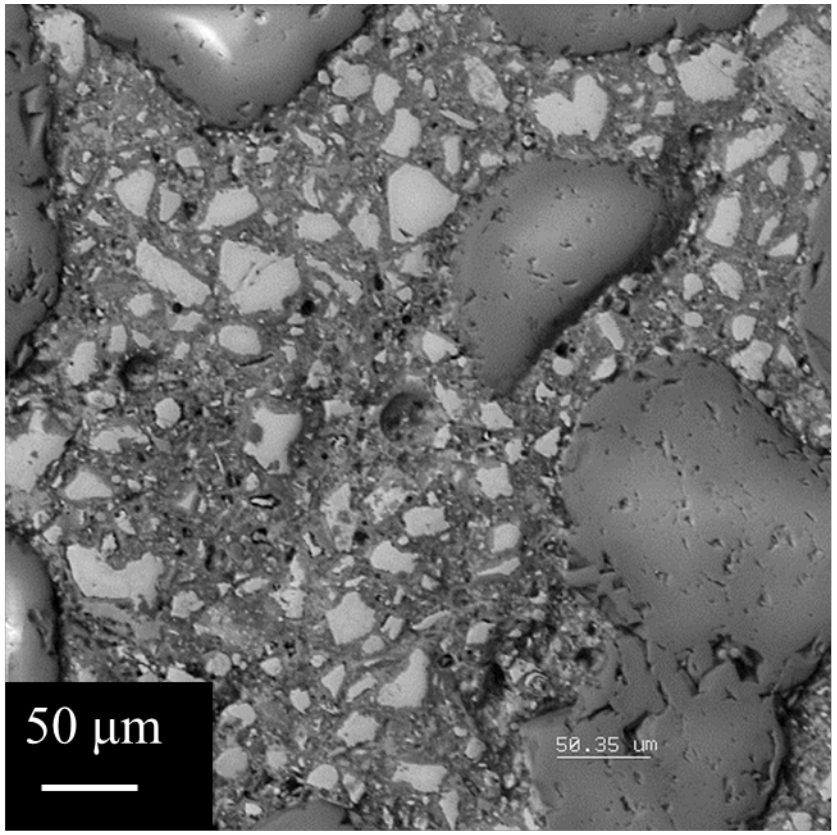


- At 7-day age, all three matrices contain considerable amount of un-hydrated cement grains.
- Also, all three matrices at this age seem to be free of any cracking.
- No de-bonding of the sand particles was observed.

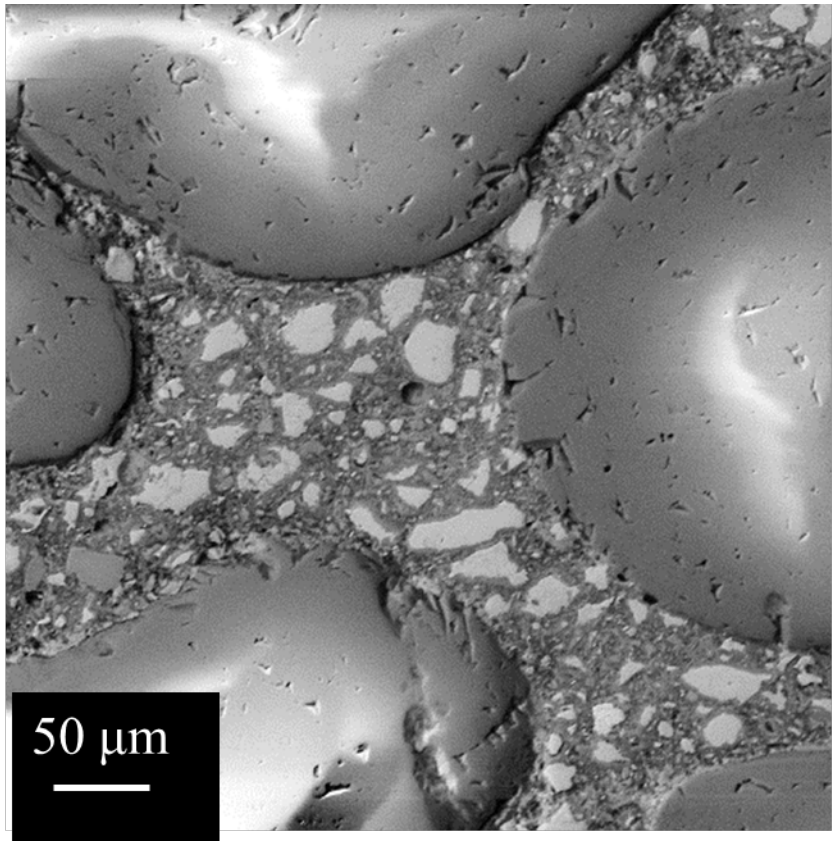
w/cem = 0.47, at 7 days

SEM of the mortar sample

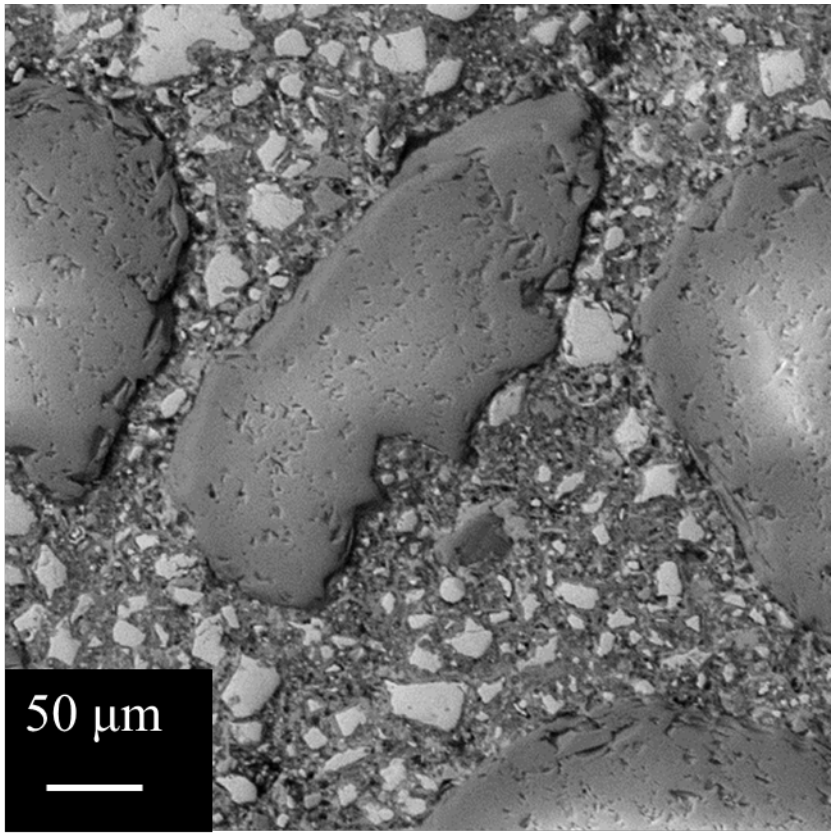
Reference



0.3% LFA



1% LFA



- It seems that in case of LFA 1% mix, the matrix contains mostly large un-reacted cement grains.
- Whereas the control matrix seems to contain the variety of the un-reacted cement grain sizes.

w/cem = 0.47, at 7 days

INDOT bridge mixture with E5

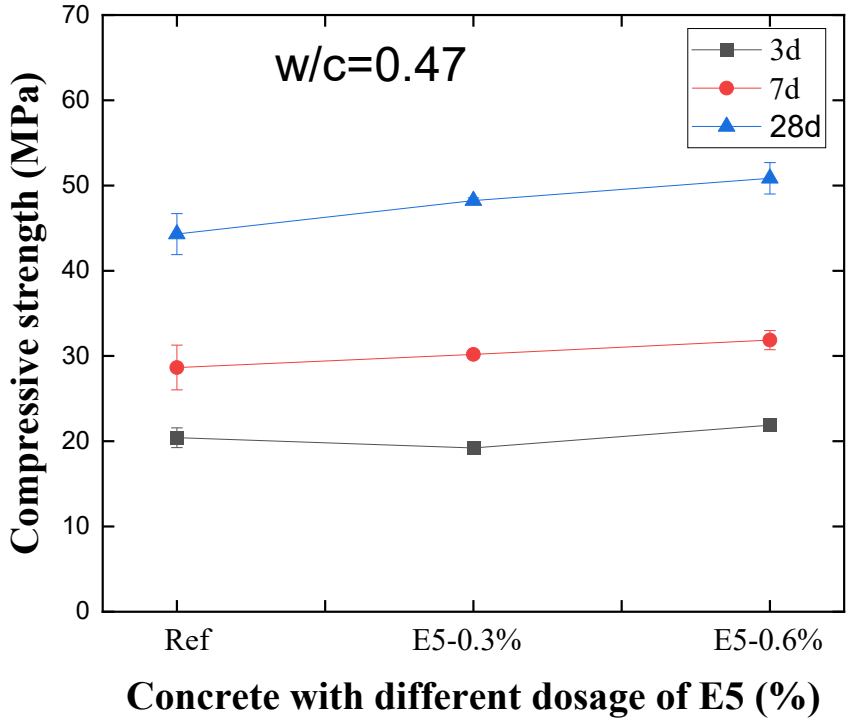
INDOT Bridge –Mixture design

	Cement (Type I)	Slag	w/c	Sand	Stone	CA/FA ratio	E5™
lb/cyd	461	197	0.47	1170	1689	1.44	0.0%/0.3%/0.6%

- Test property: compressive strength and water absorption
- Sample: 3 × 6” cylinders and 4 × 6” cylinders
- Testing ages: 3, 7 and 28 days
- E5 dosage: 0%, 0.3% and 0.6%
- Air entraining agent (AEA) and superplasticizer (SP) were used.

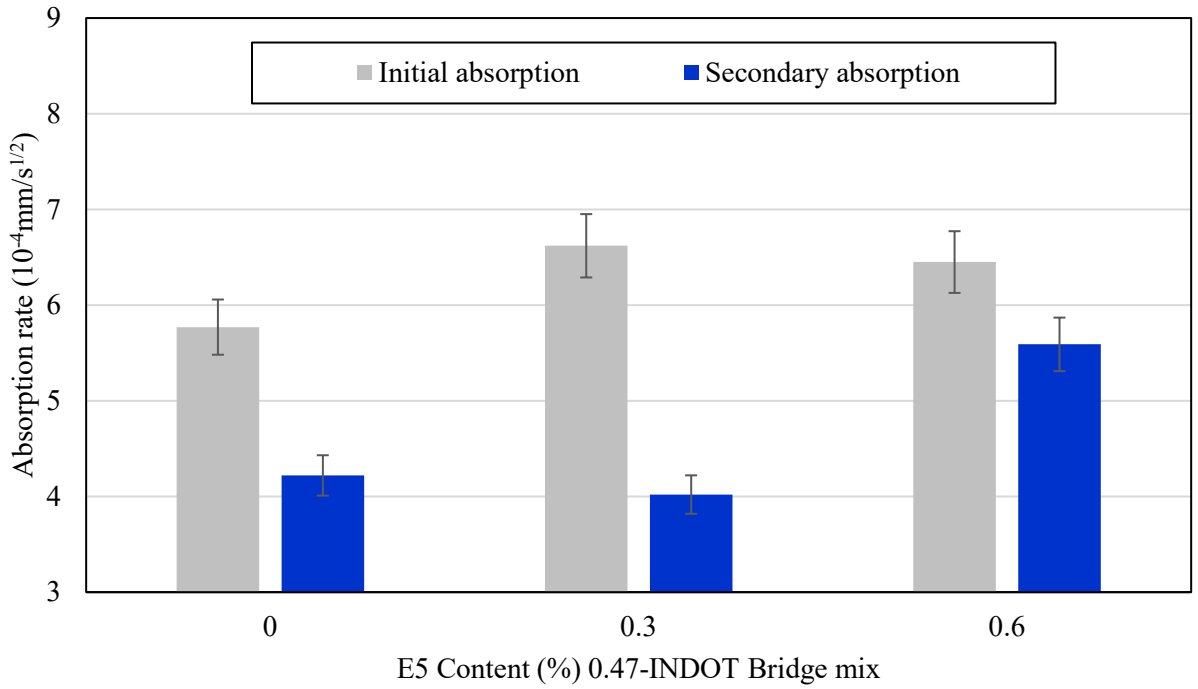
30% slag replacement

INDOT Bridge –Compressive strength



- The incorporation of the E5 into the INDOT bridge mixture can increase the strength of the concrete.

Water sorptivity test



- For absorption rate, concrete with E5-nanosilica shown higher absorption rate than the reference sample.
- Possible reason:
  - The sample with E5 might have more connective capillarity pores for water transmission

Evaluate the internal curing effect of E5 (w/c =0.47)

Experimental design- Internal humidity with E5

No.	Mixture	CEM (lb/cyd)	Sand (lb/cyd)	Stone (lb/cyd)	w/c	E5 (lb/cyd)	Lightweight aggregate (lb/cyd)
1	Ref 0.47-w/o IC	564	1400	1913	0.47	0	0
2	Ref-LWA-30%	564	980	1913	0.47	0	420
3	0.47-E5-0.3%	564	1400	1913	0.47	1.69	0
4	0.47-E5-0.6%	564	1400	1913	0.47	3.38	0
5	0.47-E5-1.0%	564	1400	1913	0.47	5.65	0
6	Ref 0.42-w/o IC	564	1400	1913	0.42	0	0
7	0.42-E5-0.3%	564	1400	1913	0.42	1.69	0

Objective

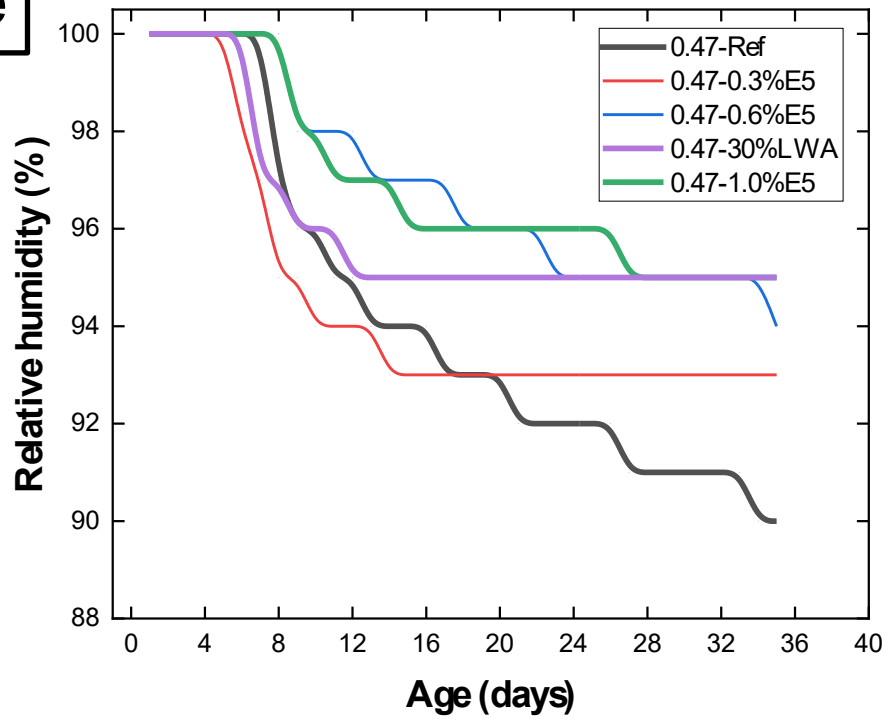
- Internal curing effect of the E5 compare with LWA;
- Influence of the E5 dosage on its internal curing effect;
- Influence of the w/c on the internal curing effect of E5.

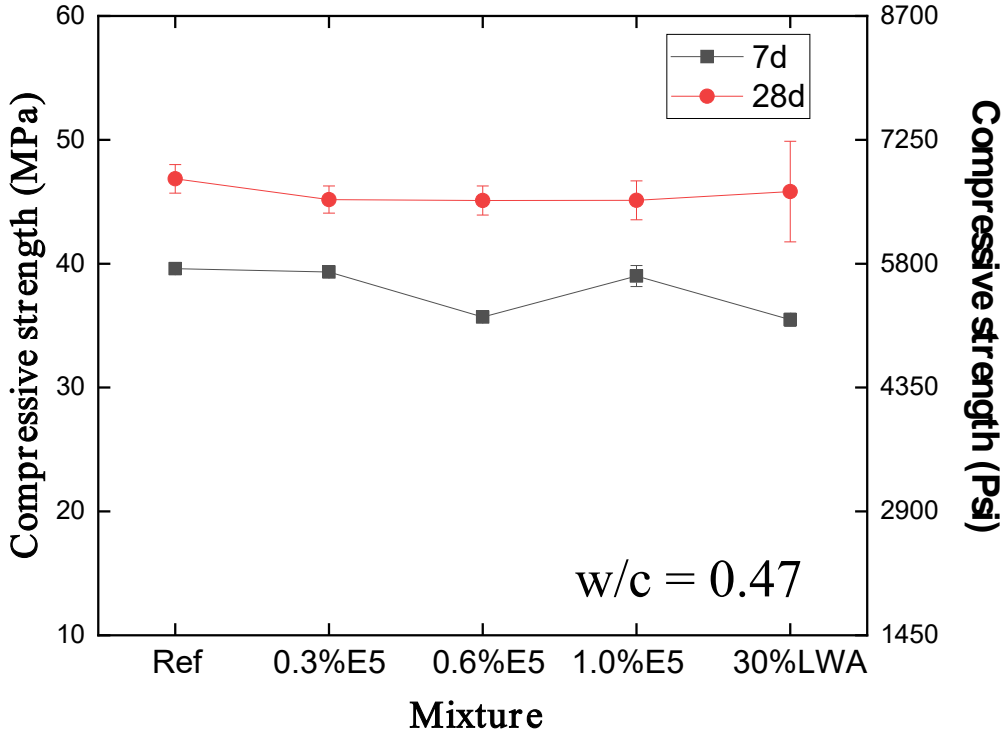
Sample preparation

- 4 × 8” cylinders;
- 3 × 6” cylinders.

Internal humidity with E5

Concrete

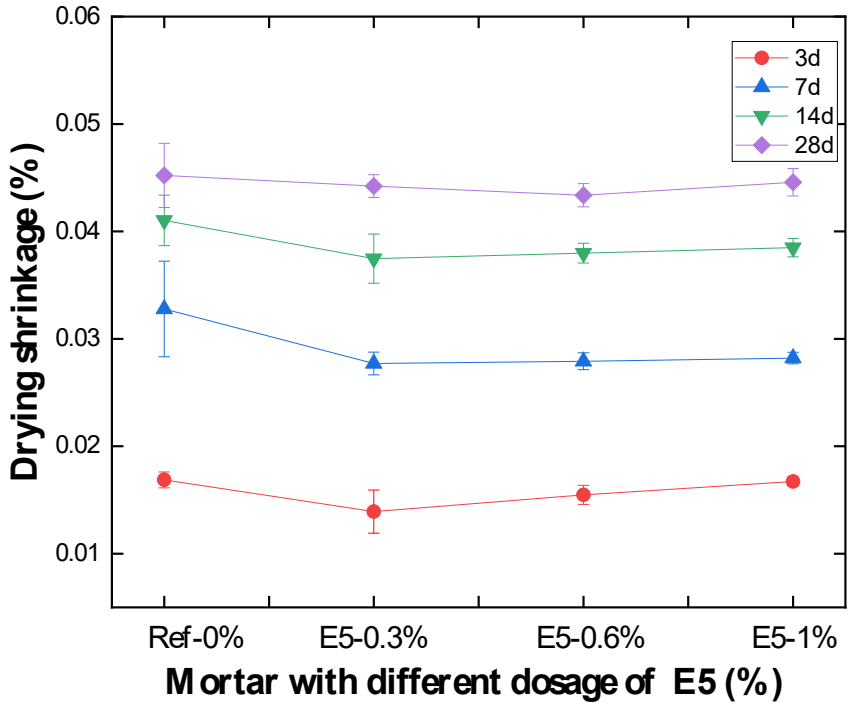




- Since INDOT curing method was used, all the mixtures can maintain at a high internal humidity level;
- E5 can slow down the water evaporation rate due to the floc network formed by colloidal nano silica;
- LWA can supply additional water to maintain the humidity (internal curing);
  - ❑ Both of the E5 and LWA exhibited internal curing effect by maintaining high internal humidity, but based on different mechanisms.

Drying shrinkage (w/c = 0.47)

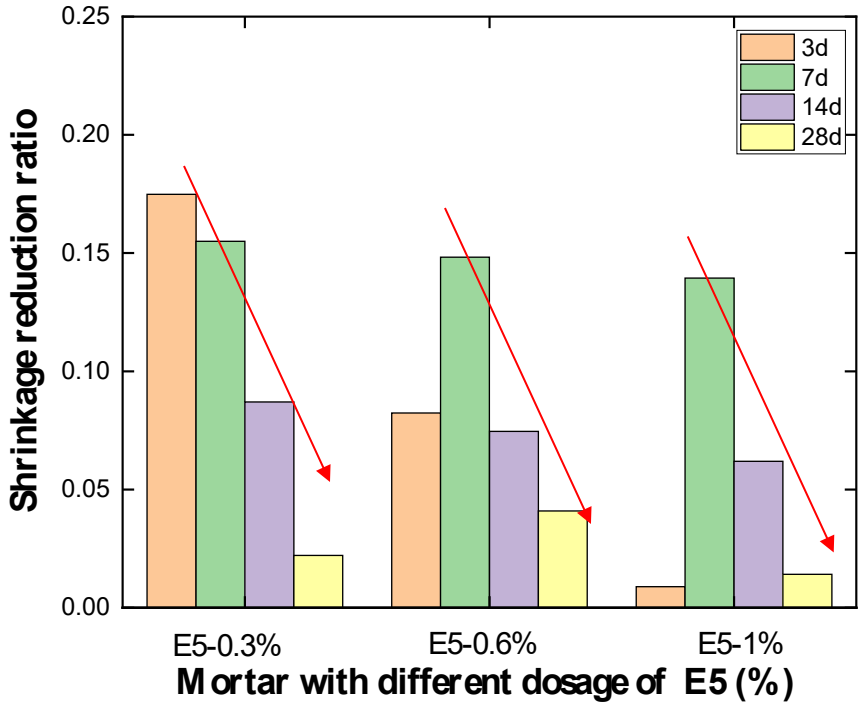
Mortar: w/c = 0.47



- To further prove the internal curing effect of the E5, drying shrinkage of the mortar sample was tested.
- The drying shrinkage comes from the losing of the water (evaporation, hydration and other reaction…)
- The incorporation of E5 reduce the drying shrinkage, while it is more effective in early ages.

Drying shrinkage (w/c = 0.47)

Mortar: w/c = 0.47

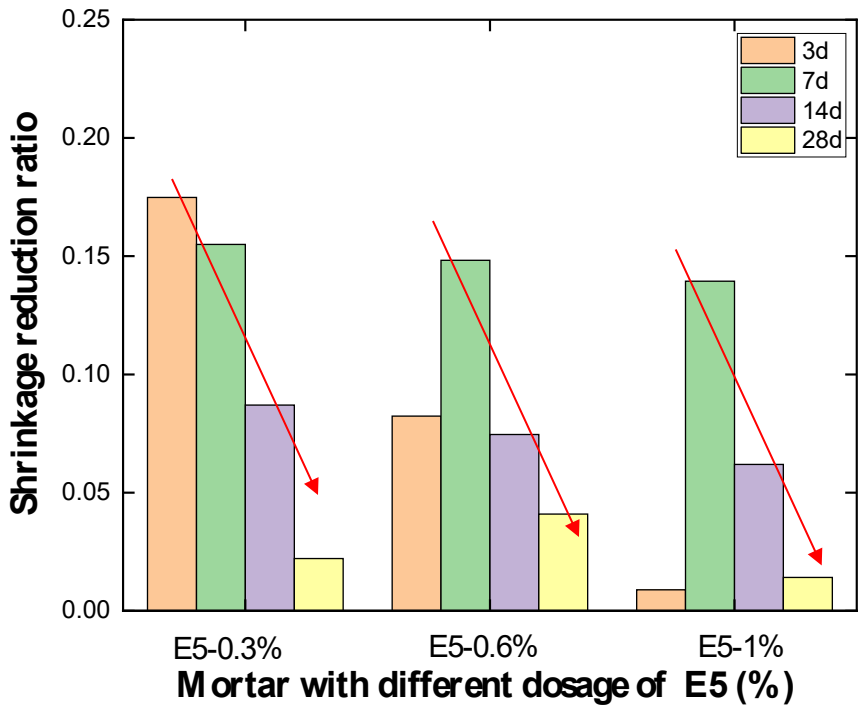


* *Reduction ratio = 1 – (D_{ref} / D_{E5})*

- Shrinkage reduction ratio is calculated, where D_{ref} represents the drying shrinkage of the reference sample, D_{E5} is the shrinkage of the sample with E5.
- At the age of 3 and 7 days, the shrinkage reduction ratios were higher. When it comes to later age, the reduction ratio decreased.

Drying shrinkage (w/c = 0.47)

Mortar: w/c = 0.47



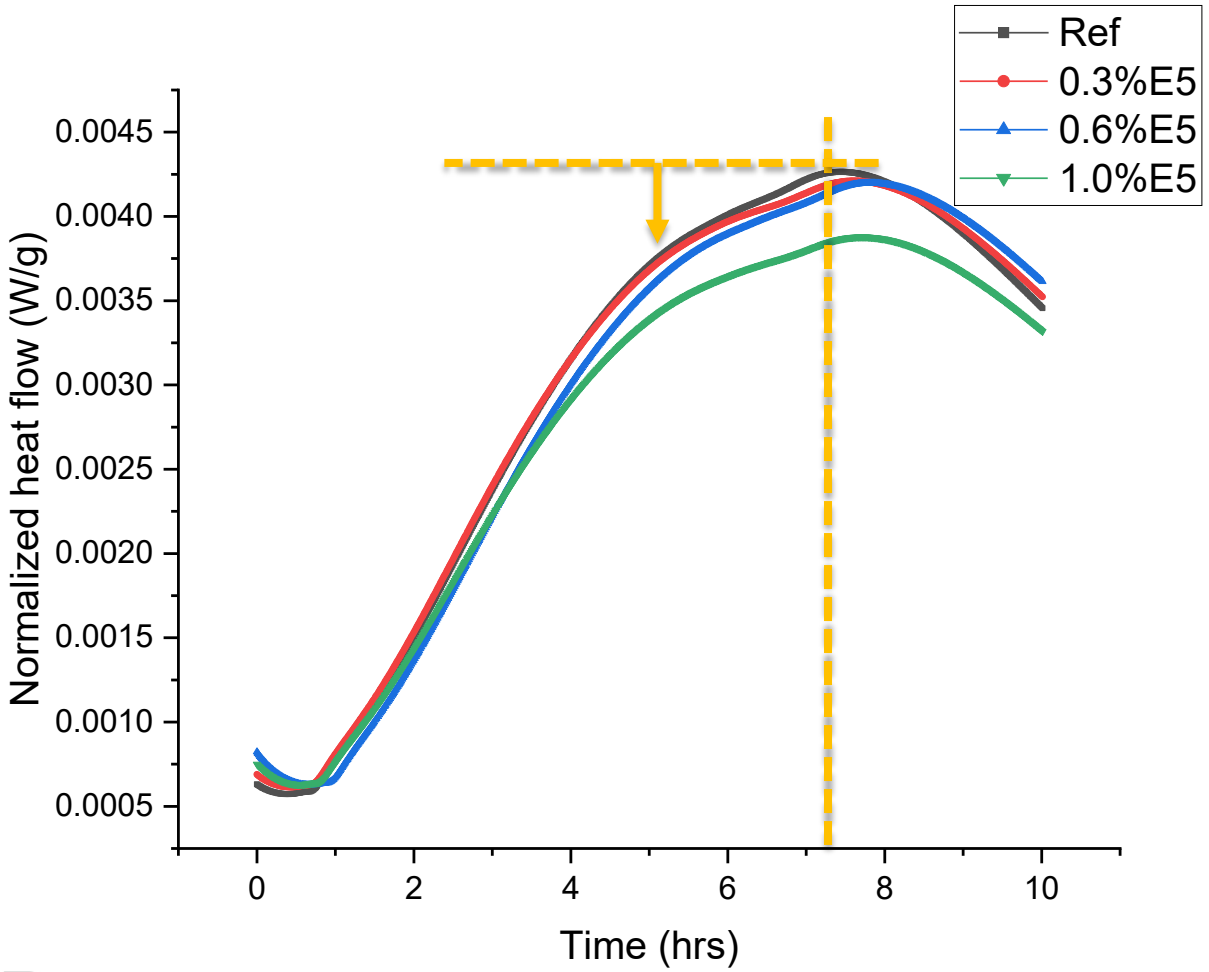
* *Reduction ratio = 1 – (D_{ref} / D_{E5})*

- The E5 reduced the evaporation of the water by holding the water inside the floc network it formed. At early age, this process prevent the intense drying of the mixture (reduce shrinkage). As the hydration process continue, the water held by the E5 started to participate into the hydration, while the reference sample may have already lost all the water. Therefore, a lower shrinkage reduction ratio was observed at later age.

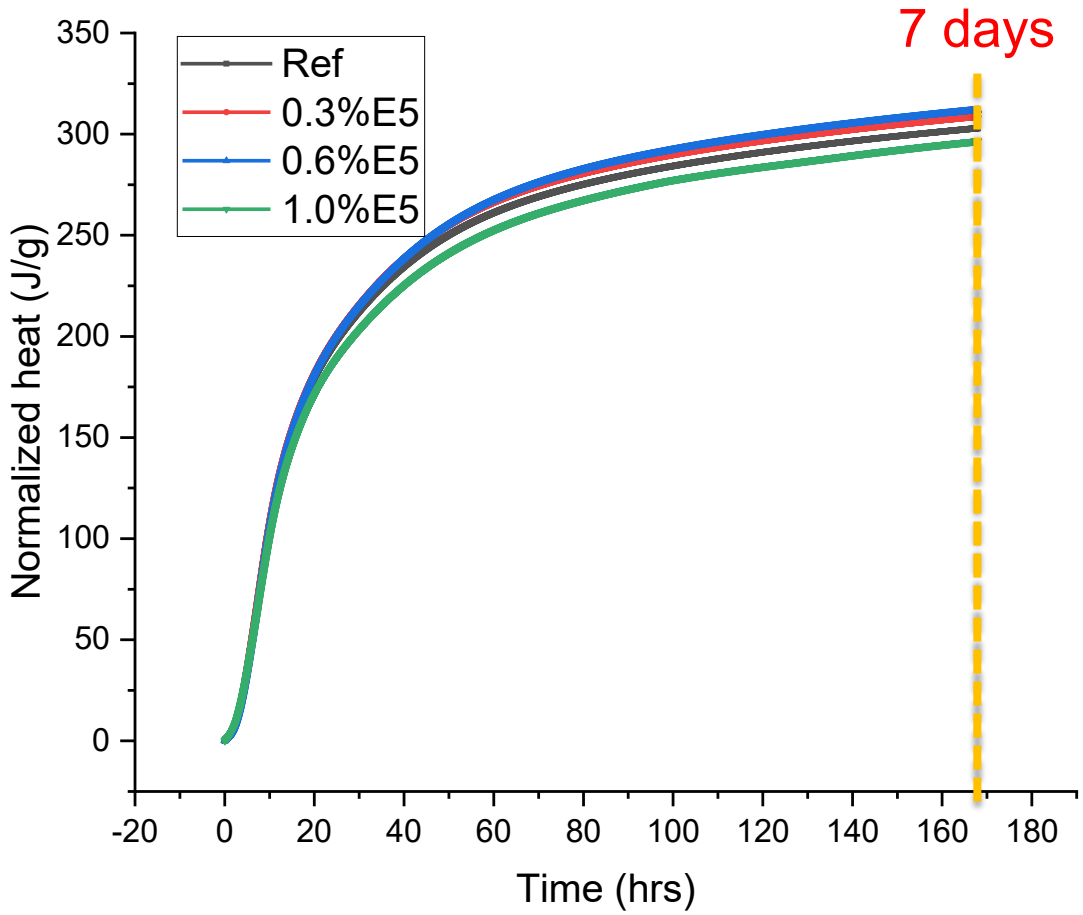
IC Result

Cement paste: w/c = 0.47

Ref>0.6%>0.3%>1.0%



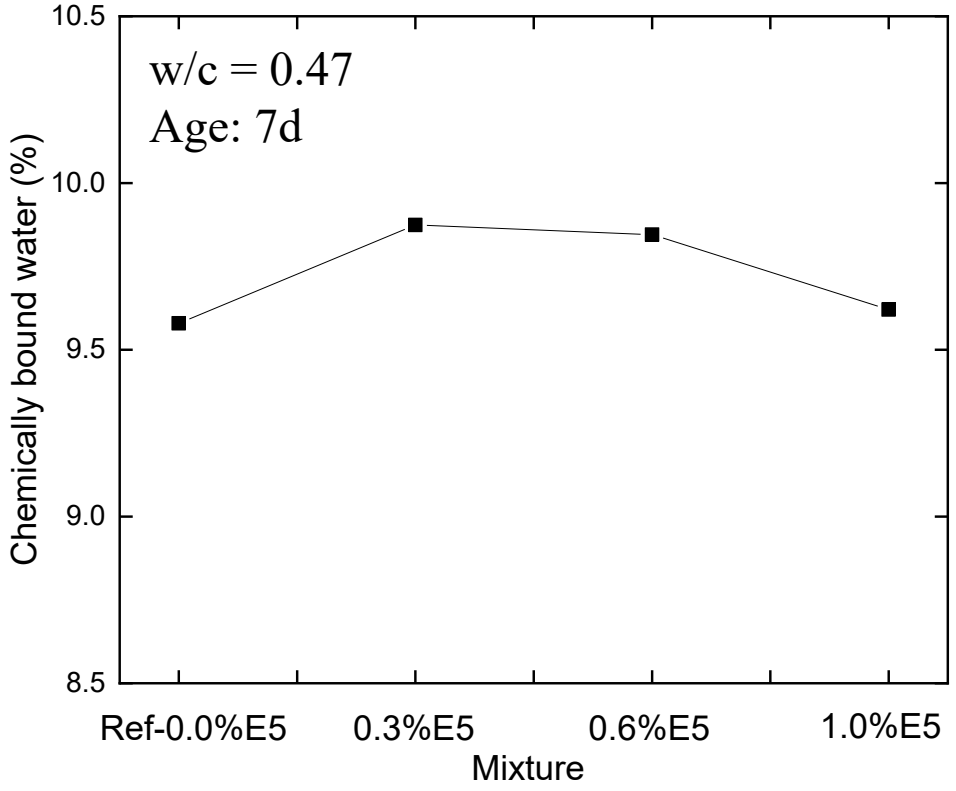
0.6%>0.3%>Ref>1.0%

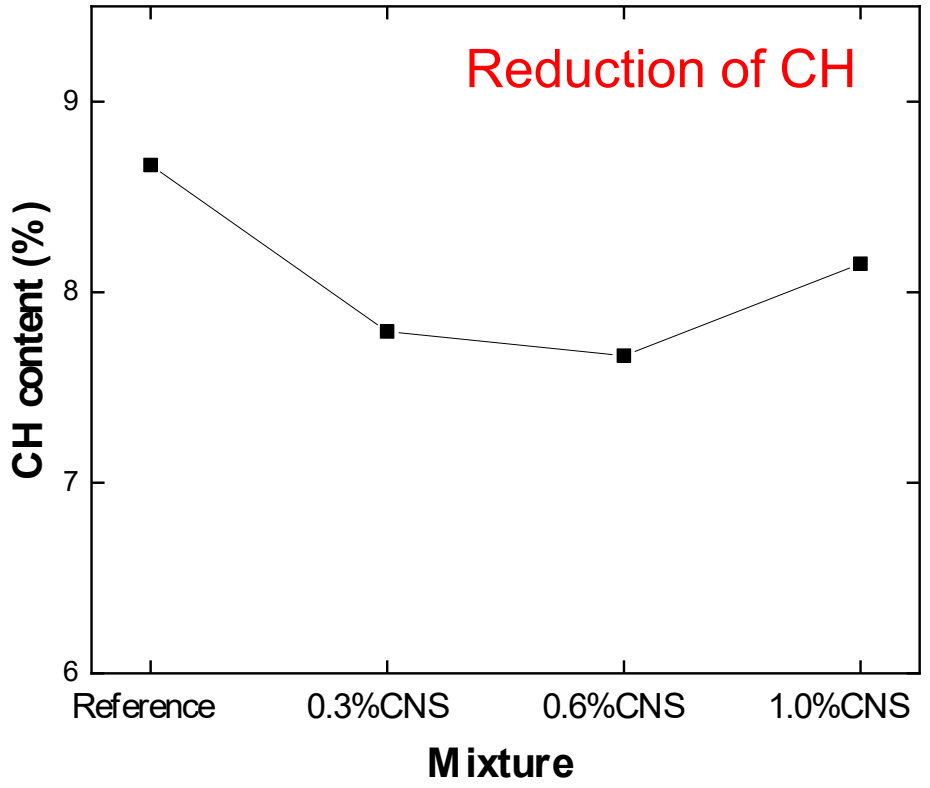


IC-TGA Result

Cement paste

w/c = 0.47
Age: 7d



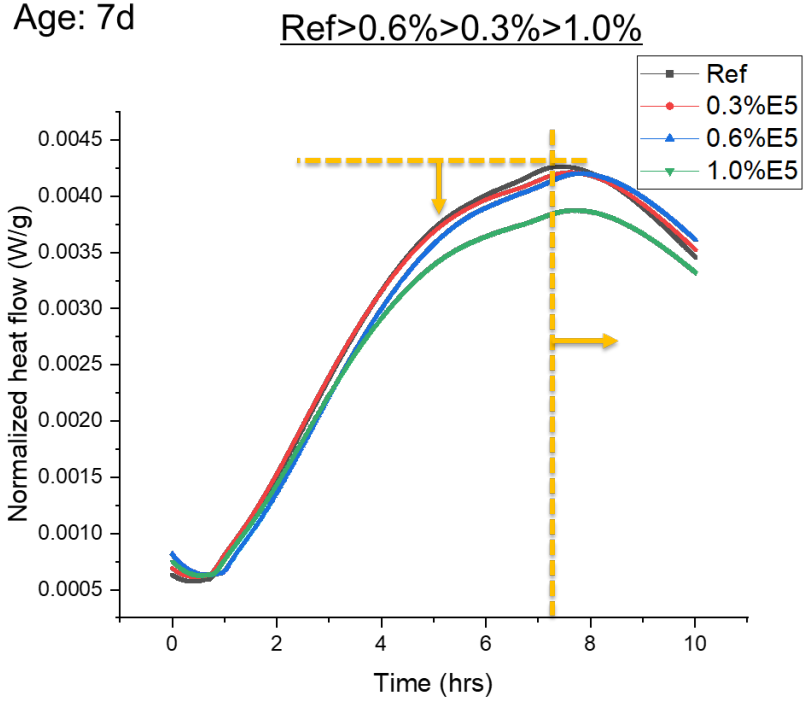


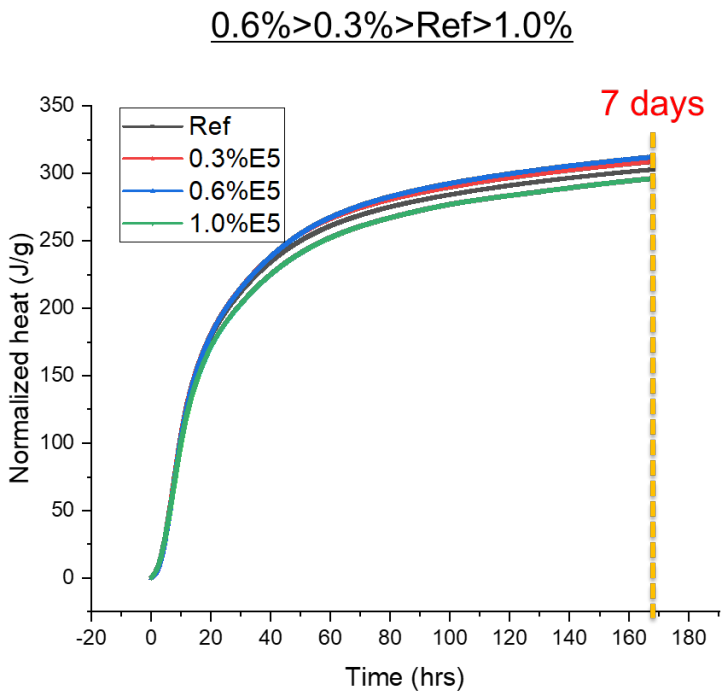
Paste from IC test was used for TGA.

IC Result

Summary

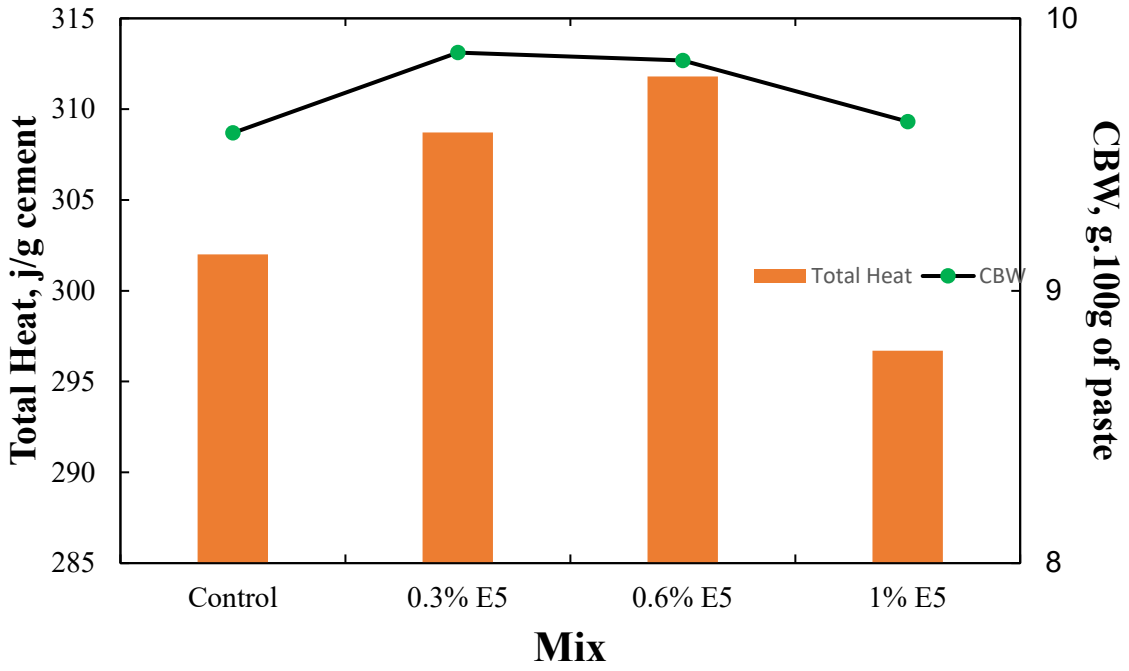
w/c = 0.47
Age: 7d

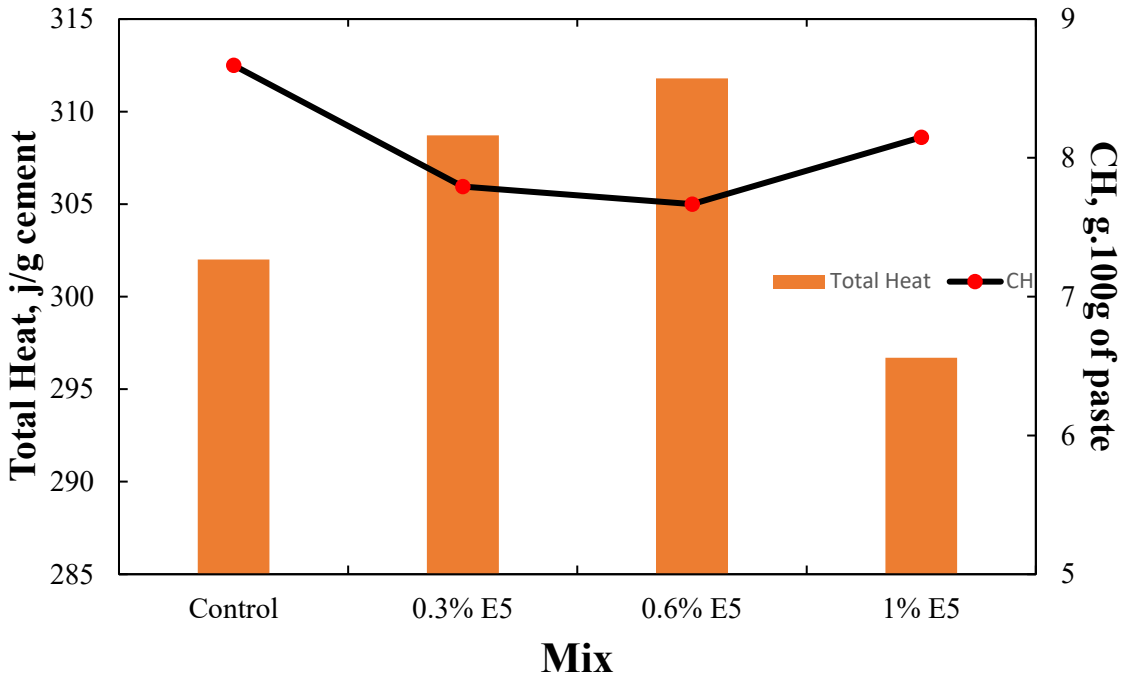




- The addition of E5 allows the mixture to hydrate more 'smoothly'.
  - Lower peak of heat flow, but higher cumulative heat. The hydration of the cement was relatively slower but was able to continuously react at a high reaction rate. Therefore, an overall higher hydration degree can be achieved. And a lower drying shrinkage can be expected, especially at early age.

Analysis of the IC and TGA

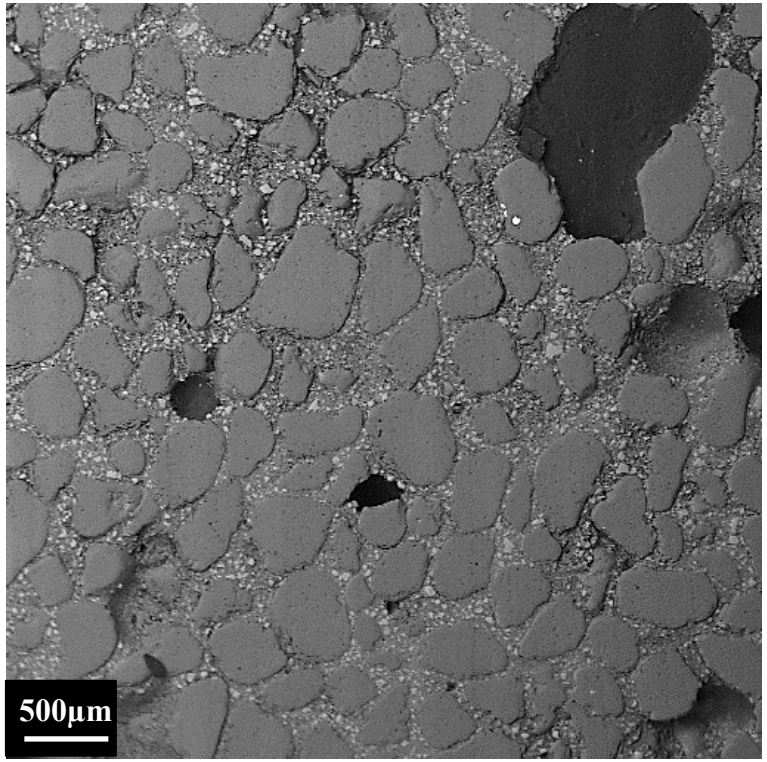




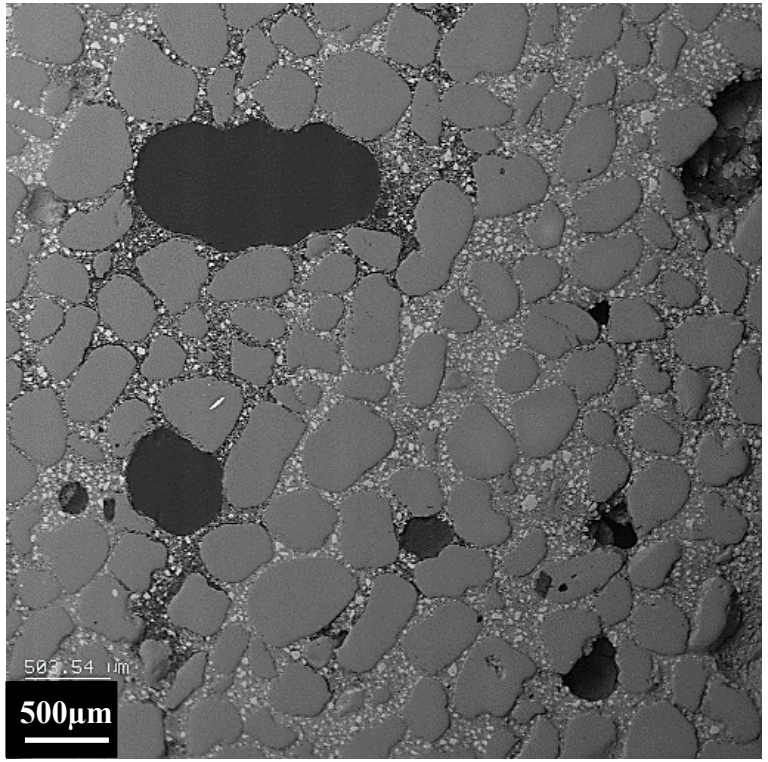
- The result of the IC well correlated with the result of the TGA (CH/CBW).
  - ❑ With the addition of the E5-CNS, the total heat release during hydration was firstly increased and then decreased, while the TGA shows a same trend for the content of the chemistry bound water.
- The CH content show an inverse correlation with the IC.
  - ❑ There was pozzolanic reaction due to the incorporation of the E5-CNS, which consumed the CH;
  - ❑ The pozzolanic reaction improved the overall hydration performance of the cement paste.

SEM of the mortar (w/c=0.47)

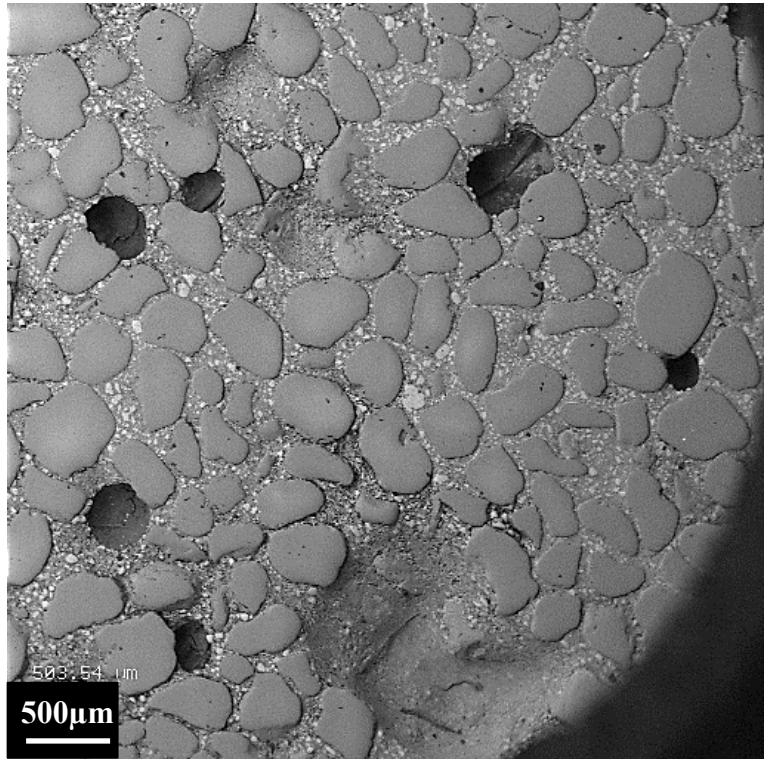
Ref



0.3%E5



0.6%E5

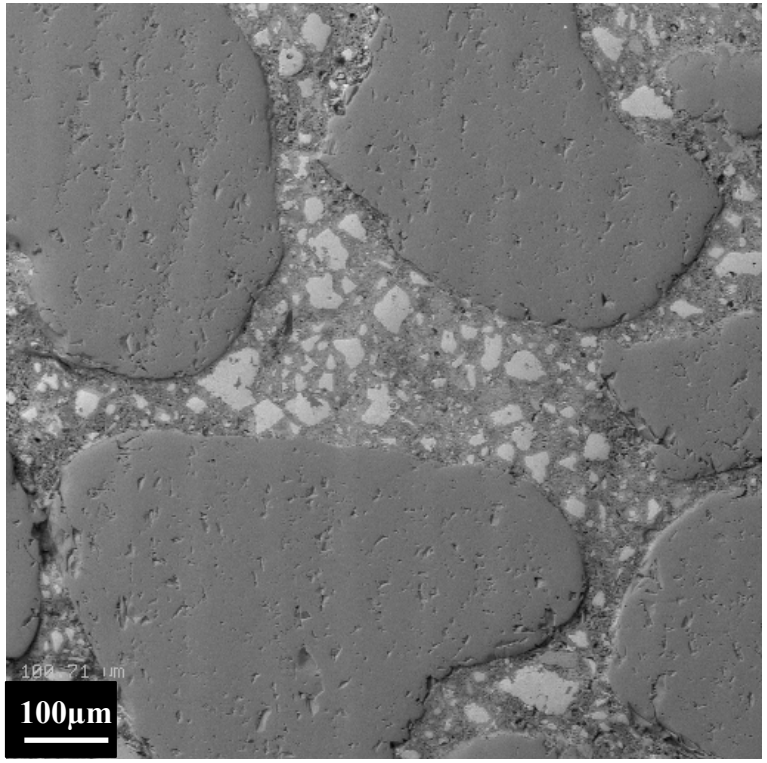


28 days sample

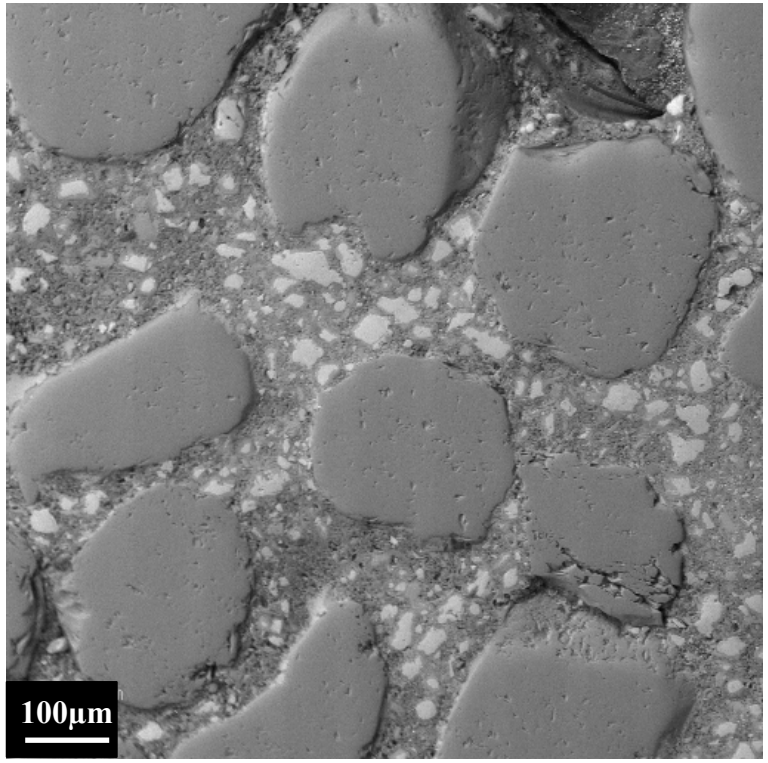
- Overall, the samples were dense and compact.

SEM of the mortar (w/c=0.47)

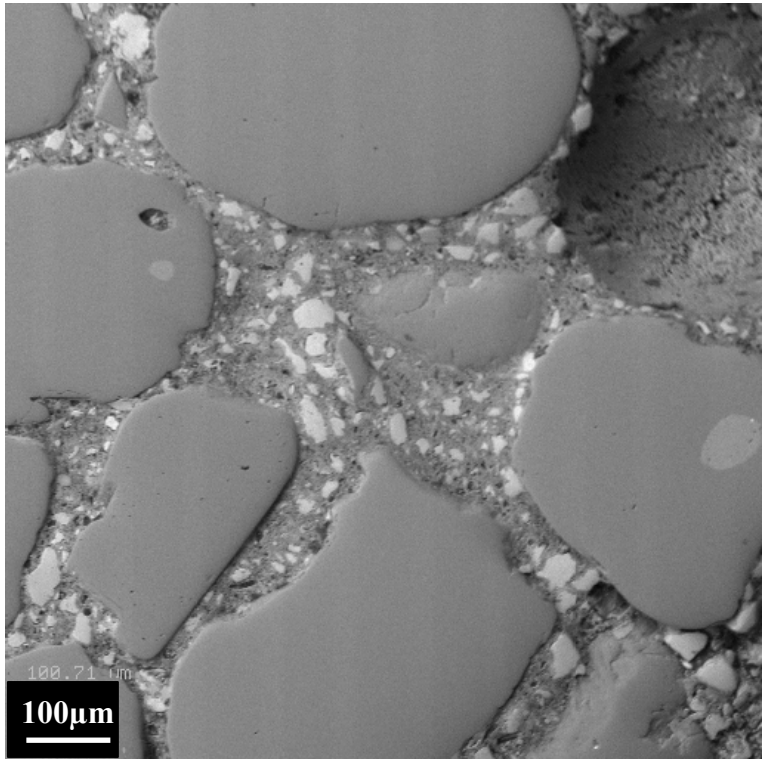
Ref



0.3%E5



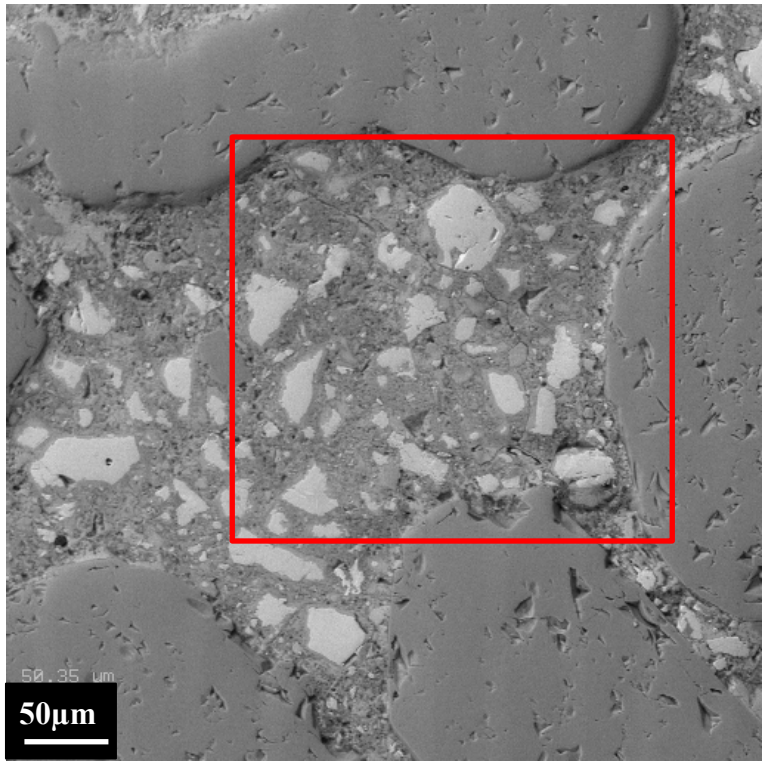
0.6%E5



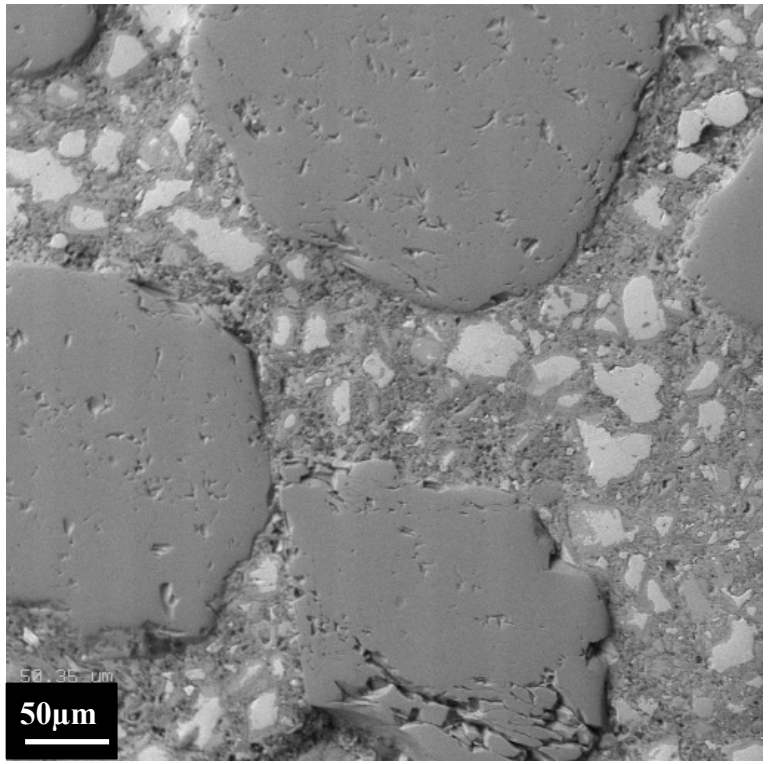
- It was observed that Ref has larger size of un-hydrate cement particles. (whitish region)

SEM of the mortar (w/c=0.47)

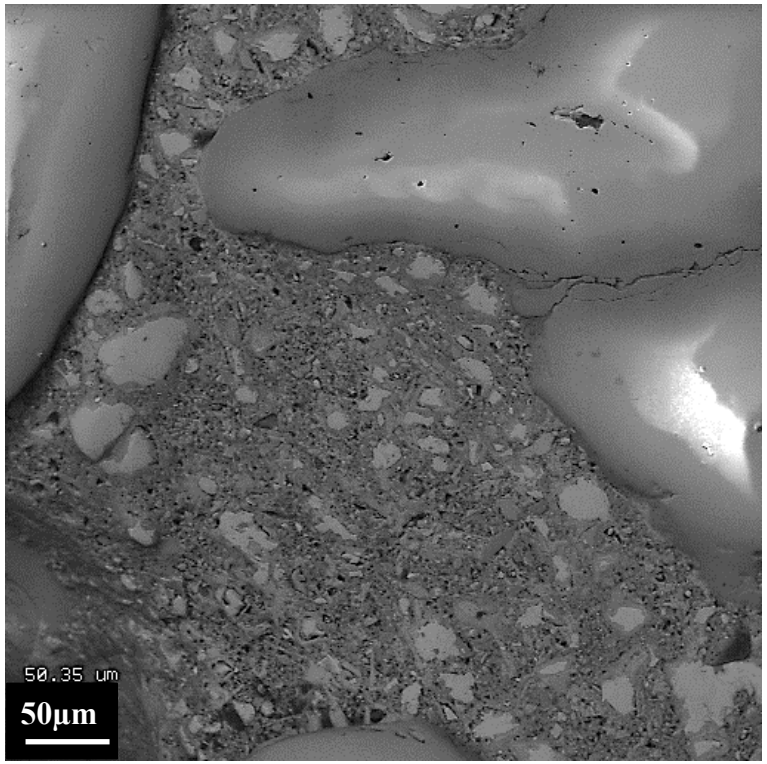
Ref



0.3%E5



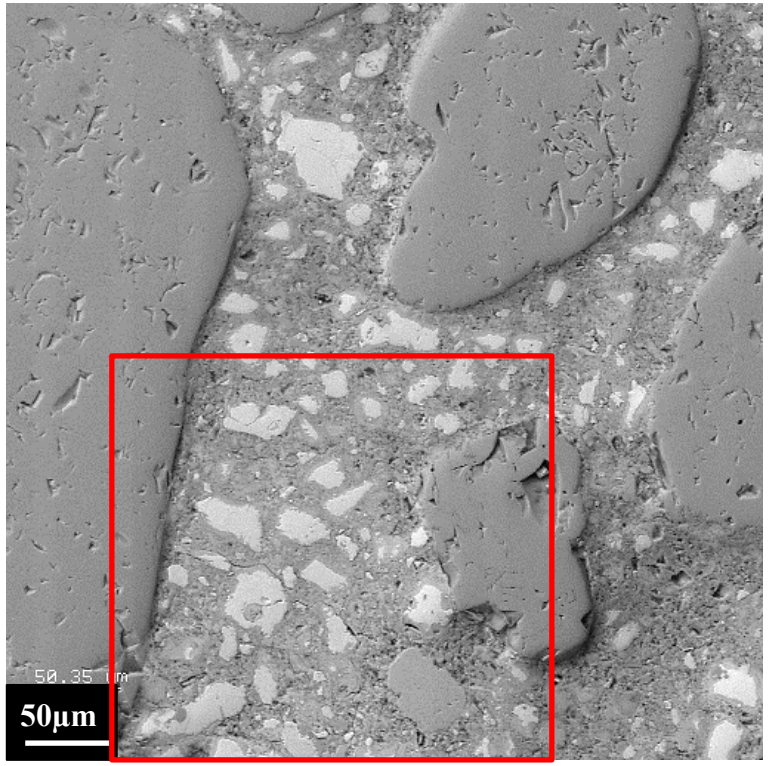
0.6%E5



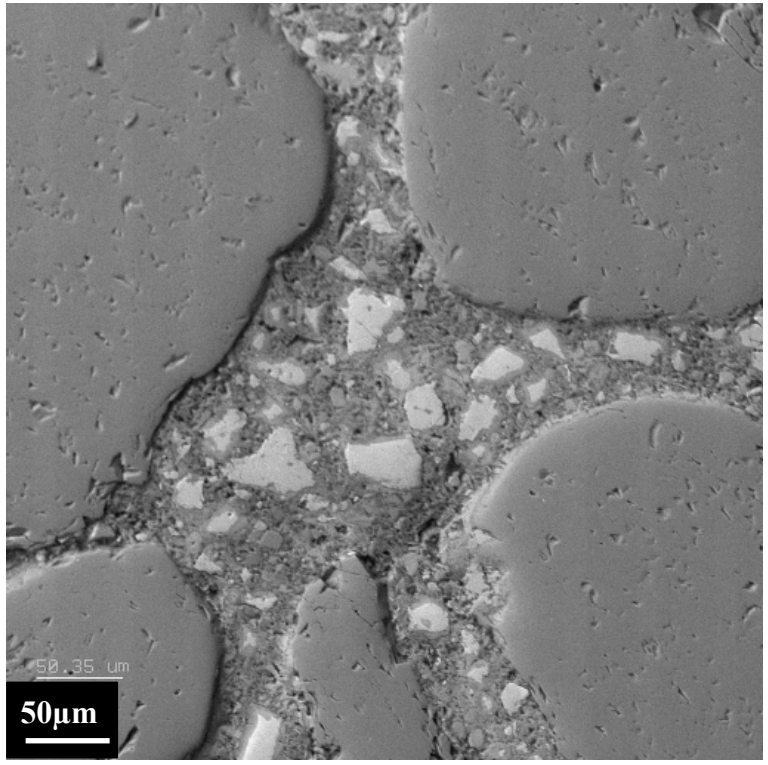
- Cracks were found in the Ref sample, while sample with E5 had better quality of the matrix.

SEM of the mortar (w/c=0.47)

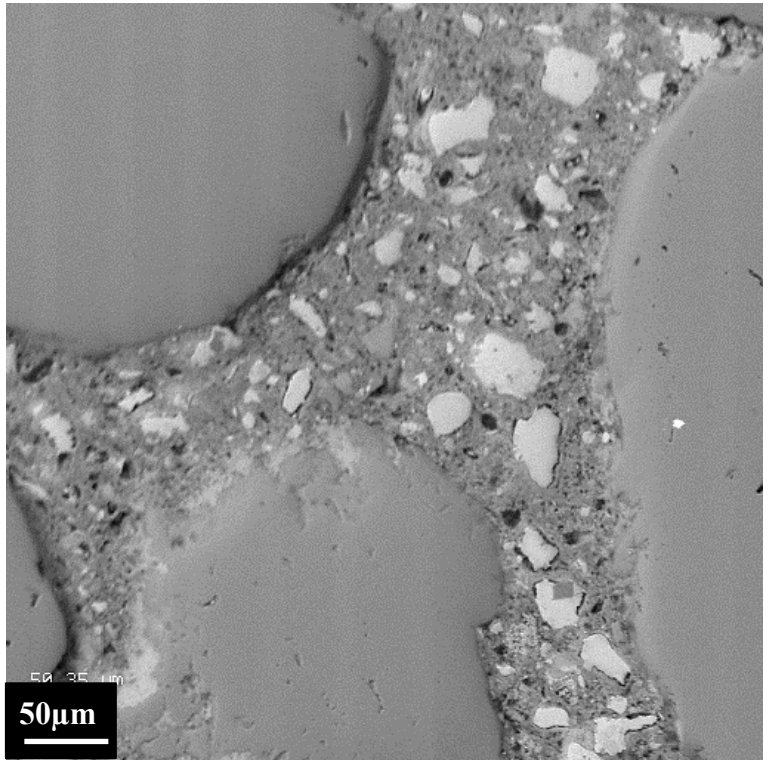
Ref



0.3%E5

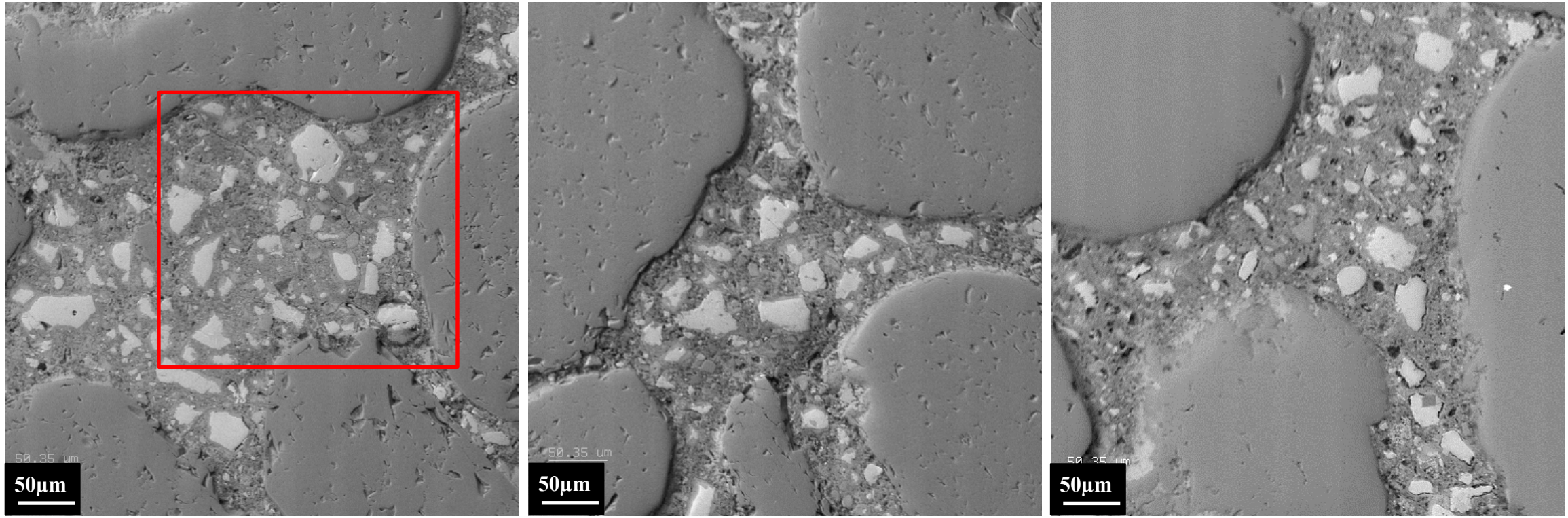


0.6%E5



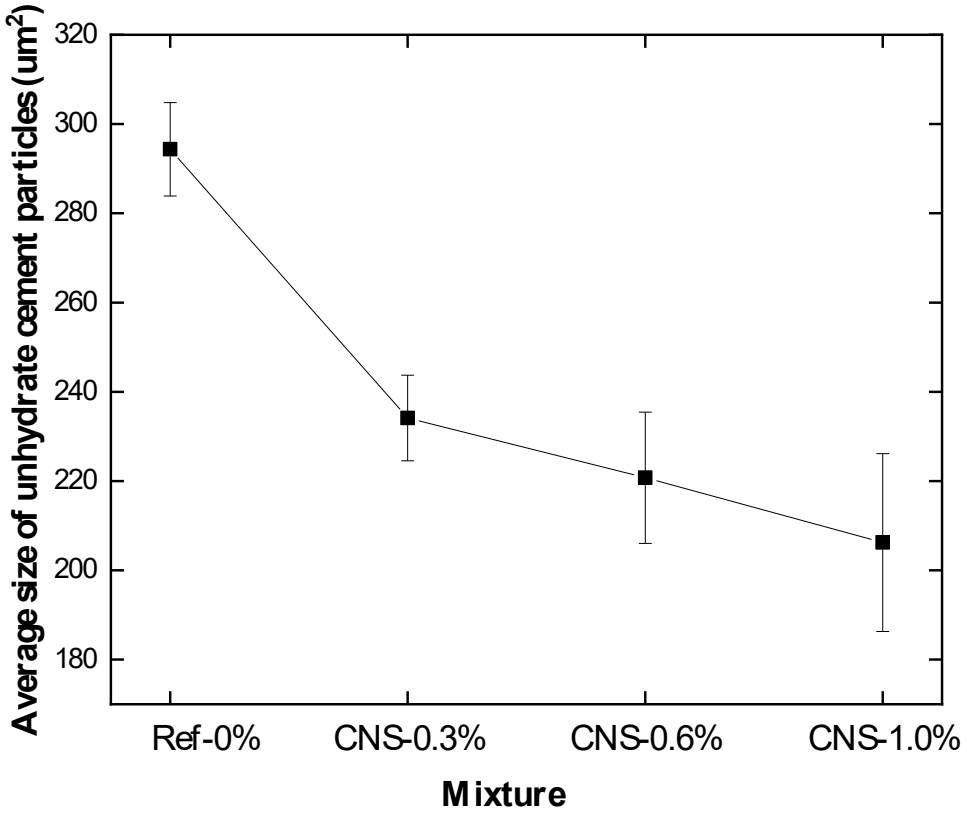
- Cracks were found in the Ref sample, while sample with E5 had better quality of the matrix.

SEM of the mortar (w/c=0.47)



- Cracks were found in the Ref sample, while sample with E5 had better quality of the matrix.

Unhydrated cement particles size analysis



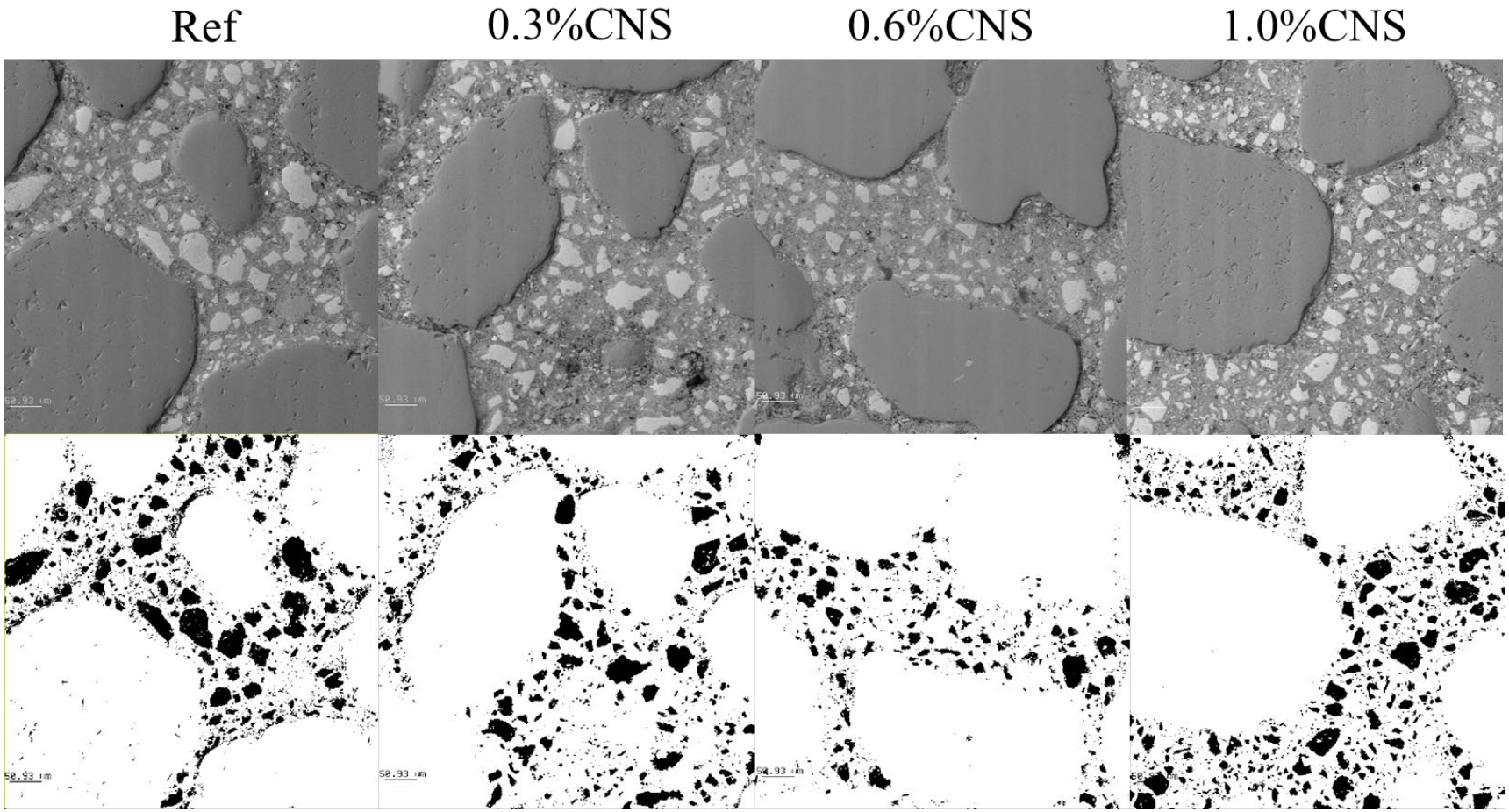
Mortar-w/c=0.47

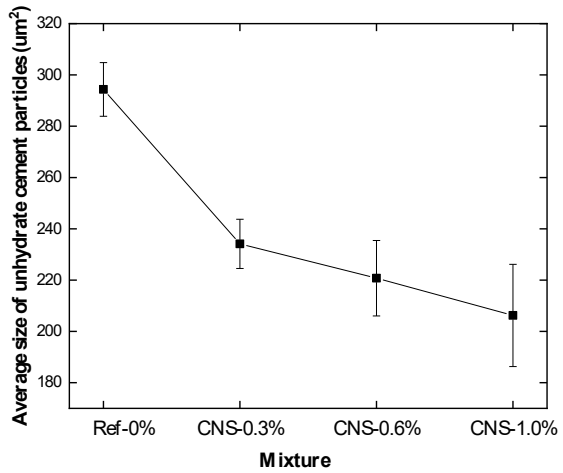
	Reference	0.3%CNS	0.6%CNS	1.0%CNS
Ave (μm^2)	294.346	234.146	220.749	206.235
SD	10.461	9.600	14.708	19.937

* CNS: colloidal nanosilica (E5)

- A more thorough analysis of the size of the unhydrated cement particles is conducted. Each data point represents the average result of 5 images.
- The result indicates that the incorporation of the CNS (E5) reduced the size of the unhydrated cement particles, which is the outcome of the improved hydration.

SEM images comparison





28d mortar - w/c=0.47

- Representative images of each mixture were compared and the reduction of the size of the unhydrated cement particles can be observed.

SHCC-E5 design and sample testing

Experimental design for LFA/E5-SHCC

- **Investigate the E5 as internal curing agent to improve the self-healing performance of SHCC**

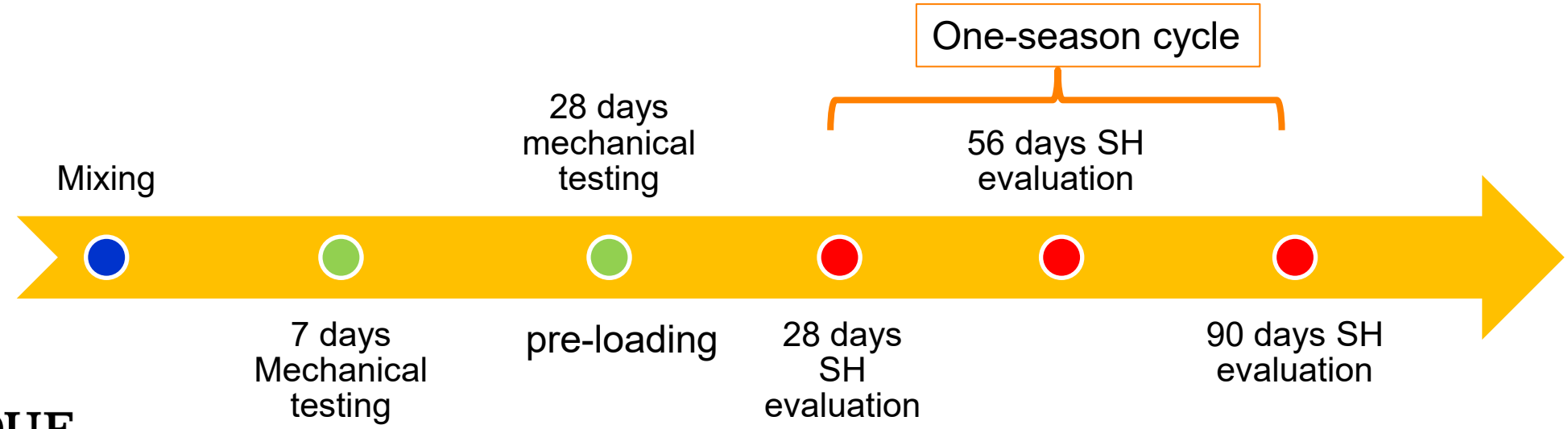
Mixture list

No.	w/c	E5/LFA Volume
1	0.35	0 % (Ref)
2	0.35	0.3% E5
3	0.35	0.6% E5
4	0.35	1.0% E5

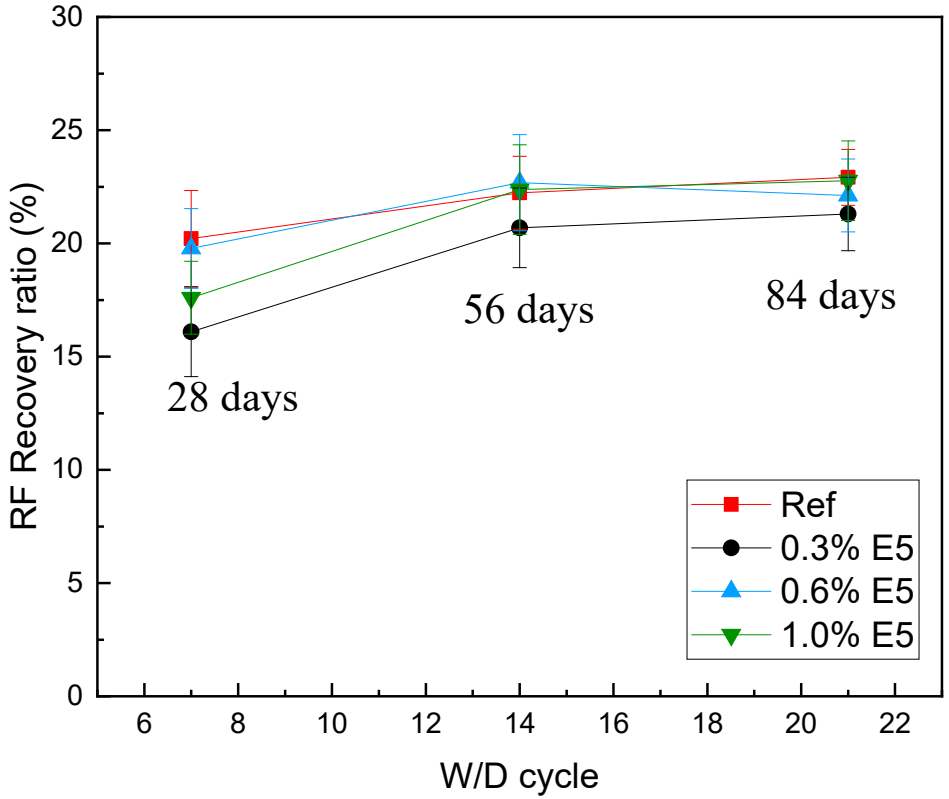
Dog-bone specimen under tensile loading

Experimental design for LFA/E5-SHCC

- **Sample age**: 7, 28 day (more 28-day sample for pre-load)
- **Environmental conditions**: (28/56/90-day evaluation)
  - Wet-dry cycle
  - Dry curing (50%RH)
- **Mechanical Testing**:
  - Tensile testing (pre-load 0.5% for self-healing)
  - Compressive testing
  - Flexural testing
- **Evaluate self healing**:
  - RF testing
  - Water permeability
  - Microscope for observe cracking on the sample before and after curing
  - Post self healing tensile testing
- **Self healing product investigation**
  - SEM for the sample after self healing
  - EDS to determine the precipitation after self healing

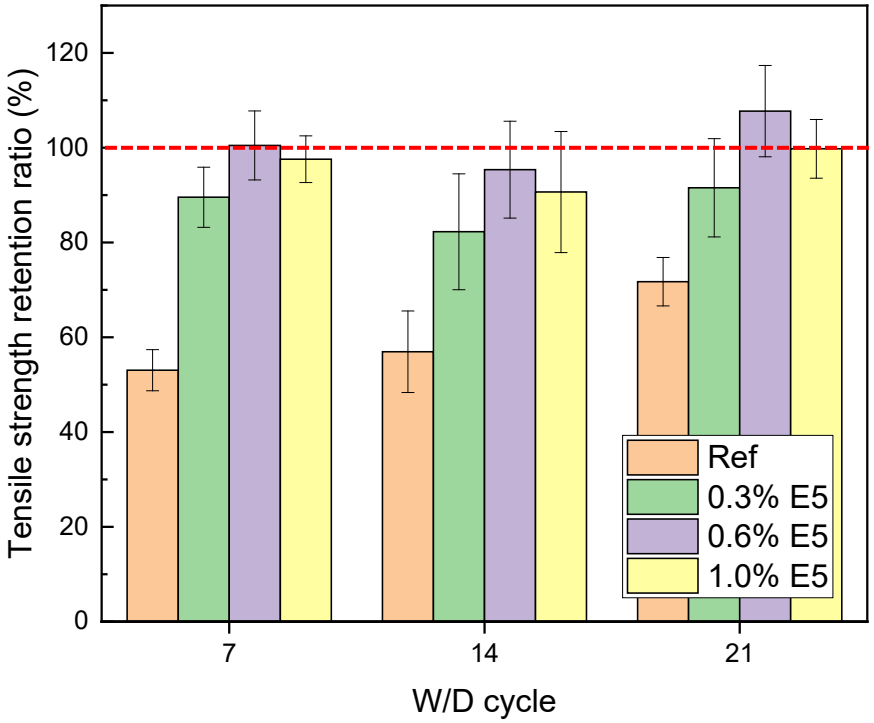


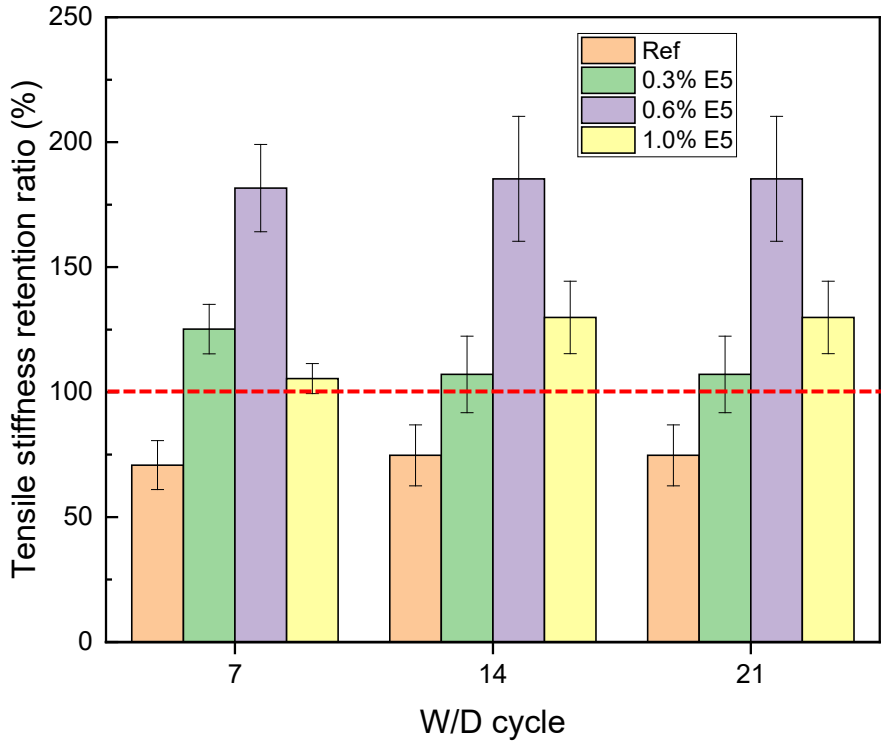
RF recovery results



- The RF recovery results of nano silica based SHCC increased over time
- The recovery tend to slow down after 14 cycles (56 days)

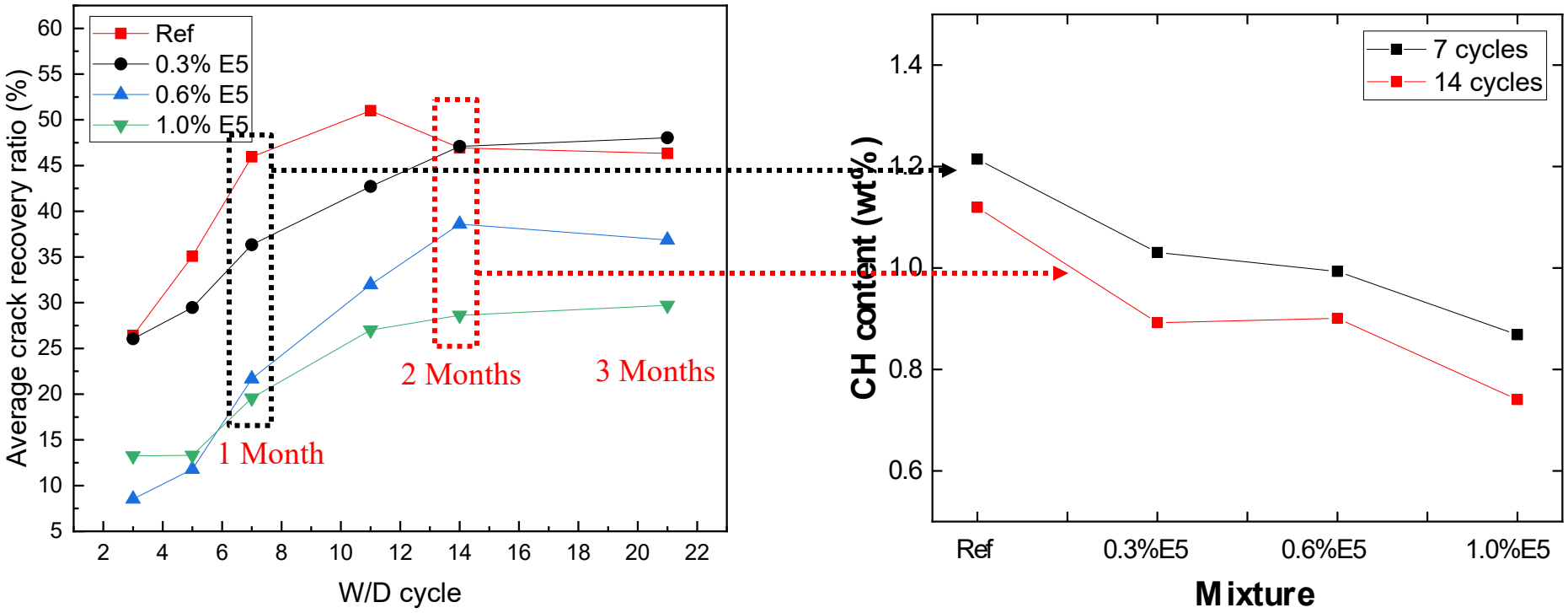
Tensile Strength Recovery results





- The tensile strength retention ratio of nano silica based SHCC (0.6% and 1%E5) reached 100% or above.
- 0.6% E5 SHCC presents highest tensile stiffness retention ratio

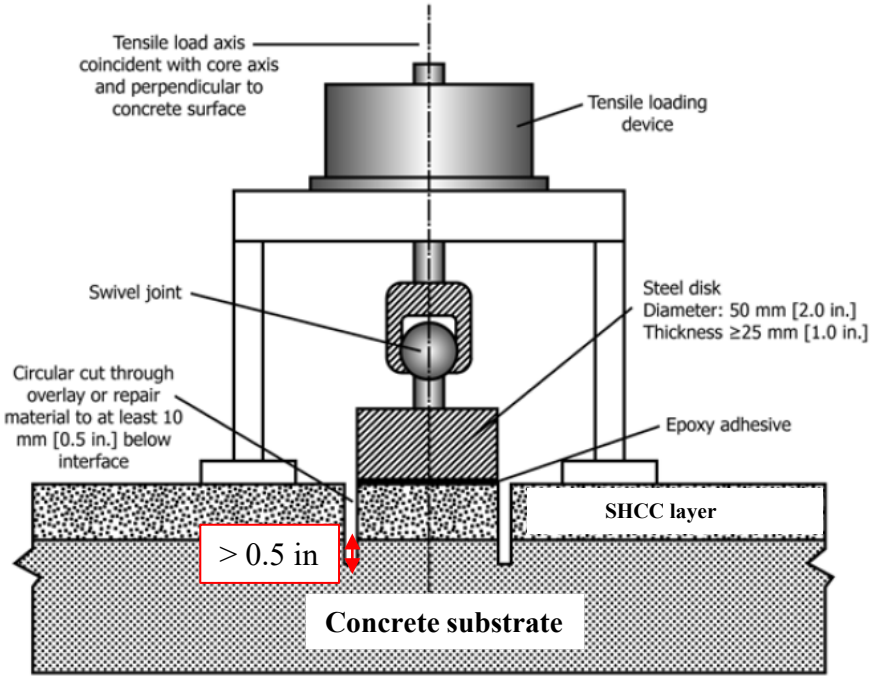
Crack analysis (After SH)



- TGA data align with the results of crack recovery ratio
- The recovery of SHCC tend to slow down after 14 cycles (56 days SH), might be due to run out of un-hydrated cementitious materials for further hydration or pozzolanic reaction

Pull off test - ASTM C1583

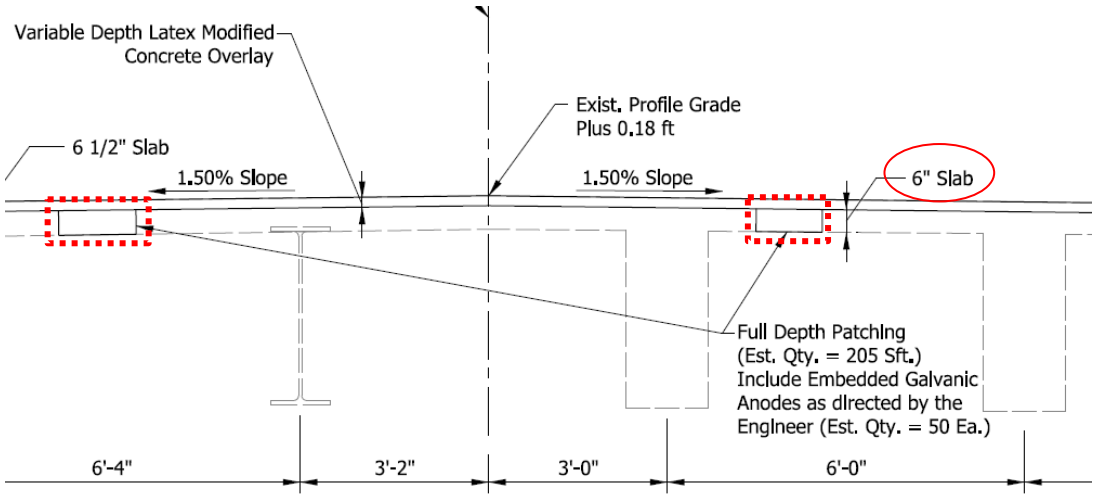
- Standard Test Method for Tensile Strength of Concrete Surfaces and the Bond Strength or Tensile Strength of Concrete Repair and Overlay Materials by Direct Tension (Pull-off Method)



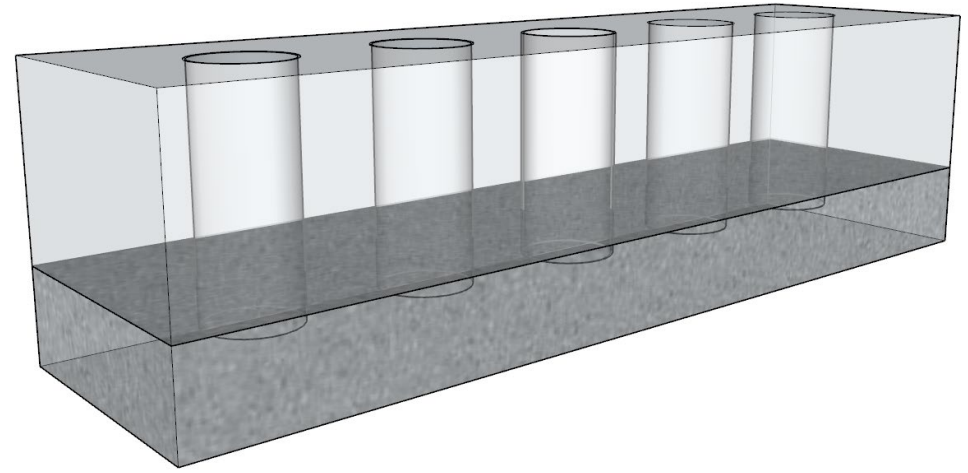
Proceq DY-225
Pull-off testers

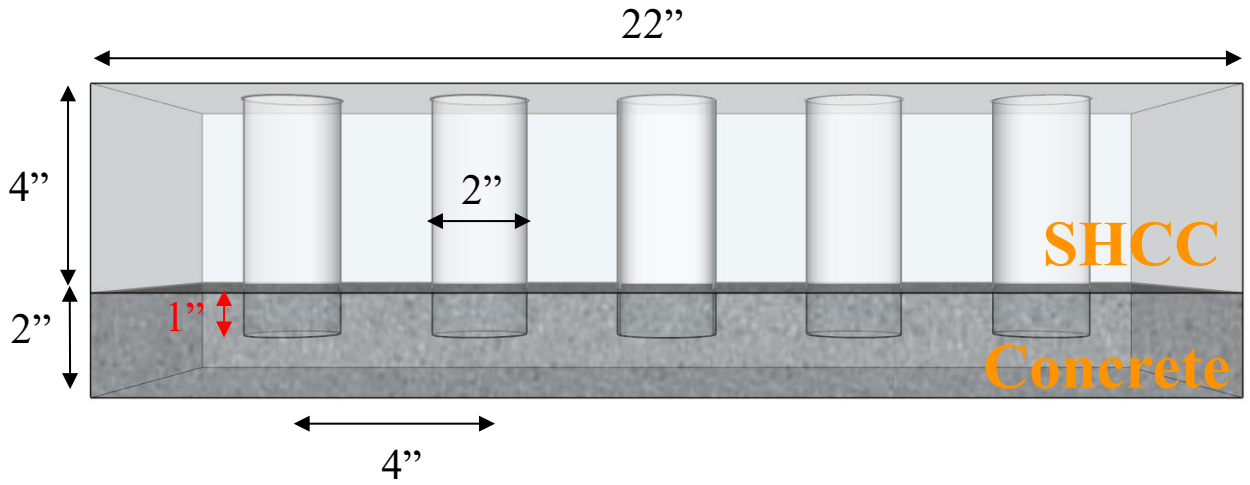
Working Range:185 to 1847 psi
Maximum Pulling Speed: 0.086 in/min

Sample schematic



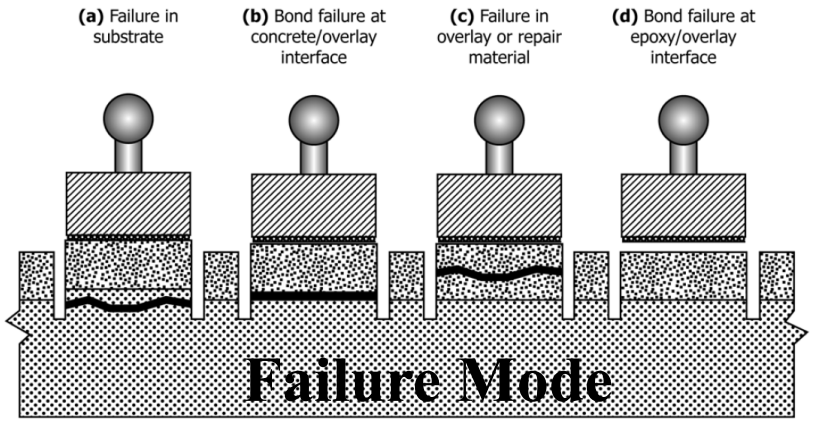
INDOT Bridge rehabilitations plans
Route SR 68 AT: PR 9+68
Project No. 1296794





Testing procedure and sampling

1. Casting the concrete substrate and cure for 56 days
2. Casting the SHCC overlay on the top of concrete substrate
3. Drilling the 2 inches-diameter core at the age of interest, and a least 0.5 in. depth below the interface
4. Clean the SHCC surface
5. Bonding the steel disk on the 2 inches core using epoxy, and wait for the drying of epoxy
6. Conduct pull-off test and record the failure mold (Three individual test results with similar failure mold per sample – ASTM C1583)



Cast Concrete Substrate

Cast SHCC

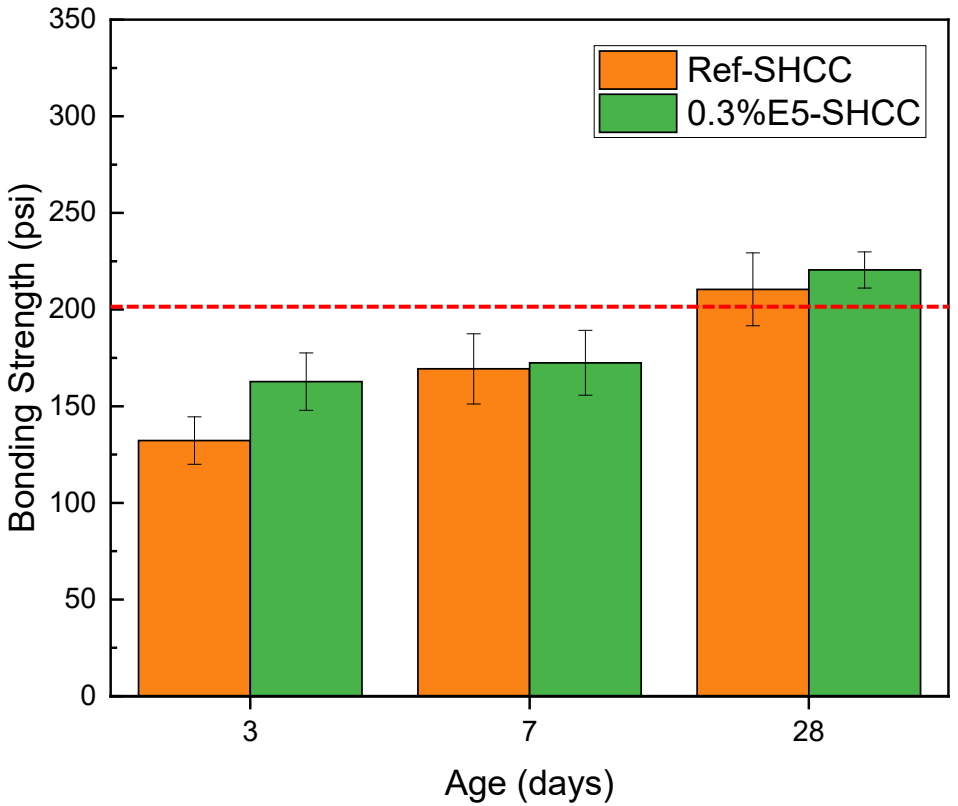
Core drilling

Pull off testing

SHCC Pull off results

Bond strength test results may be qualified as follows:
(Over 28 days)
≥ 300 psi (2.1 MPa), excellent
250 to 299 psi (1.7 to 2.1 MPa), very good
200 to 249 psi (1.4 to 1.7), good
100 to 199 psi (0.7 to 1.4 MPa), fair
0 to 99 psi (0 to 0.7 MPa), poor.

(Sprinkel et al, 2000, VDOT Report)

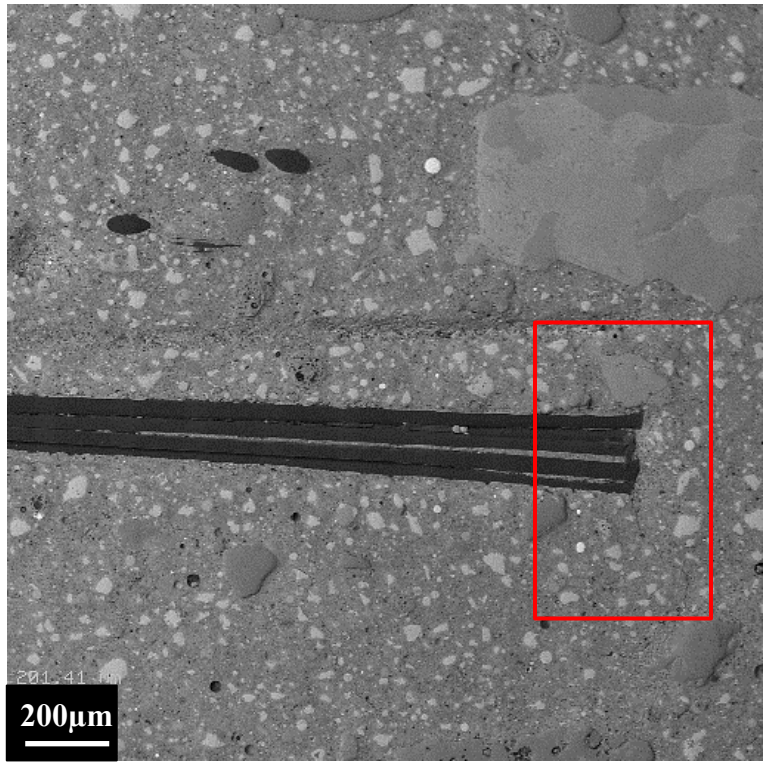


Pulled samples

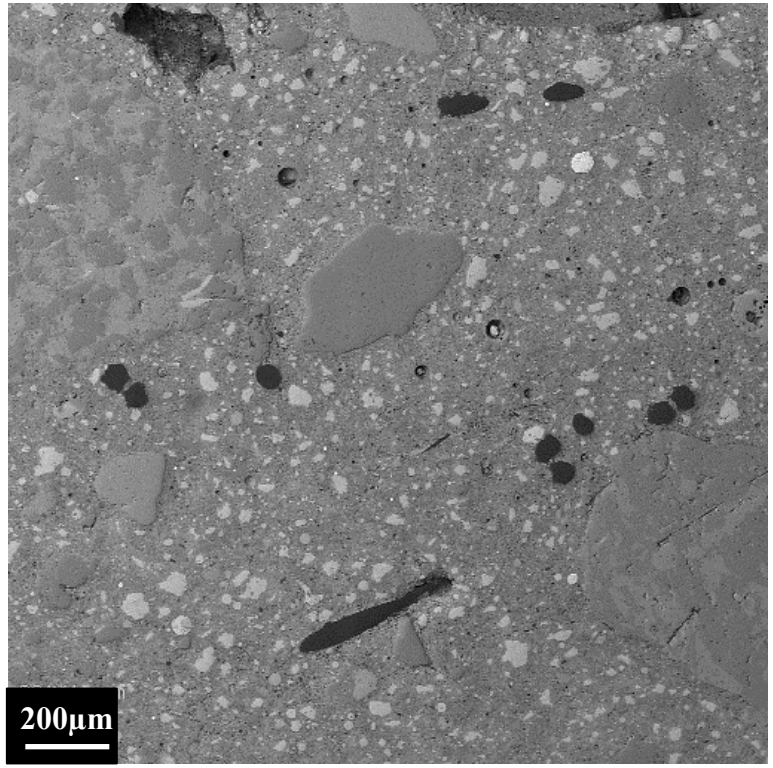
- For the early age bonding strength of SHCC are regarded as “Fair” based on VDOT report (not standard).
- For early age results, 0.3 E5 SHCC shown higher bonding strength than Reference SHCC due to the accelerated reaction of nanosilica which improve the interfacial adhesion between concrete and SHCC.
- 0.3% E5 SHCC shown slightly higher bonding strength than Reference SHCC might be due to nanosilica refined interfacial zoon between concrete and SHCC to improve the interfacial adhesion.

SEM – (28 days self-healed sample)

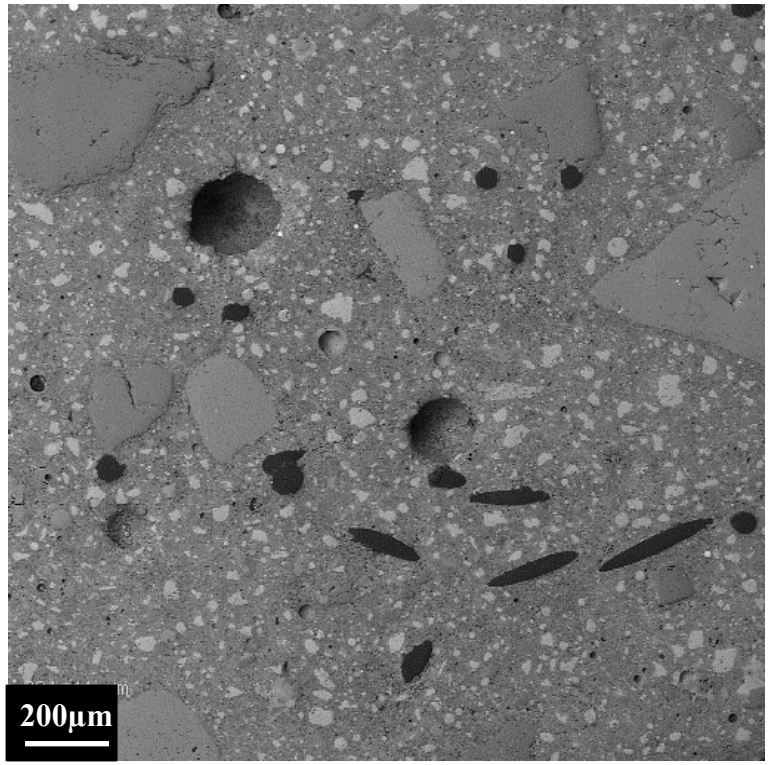
Ref



0.3%E5



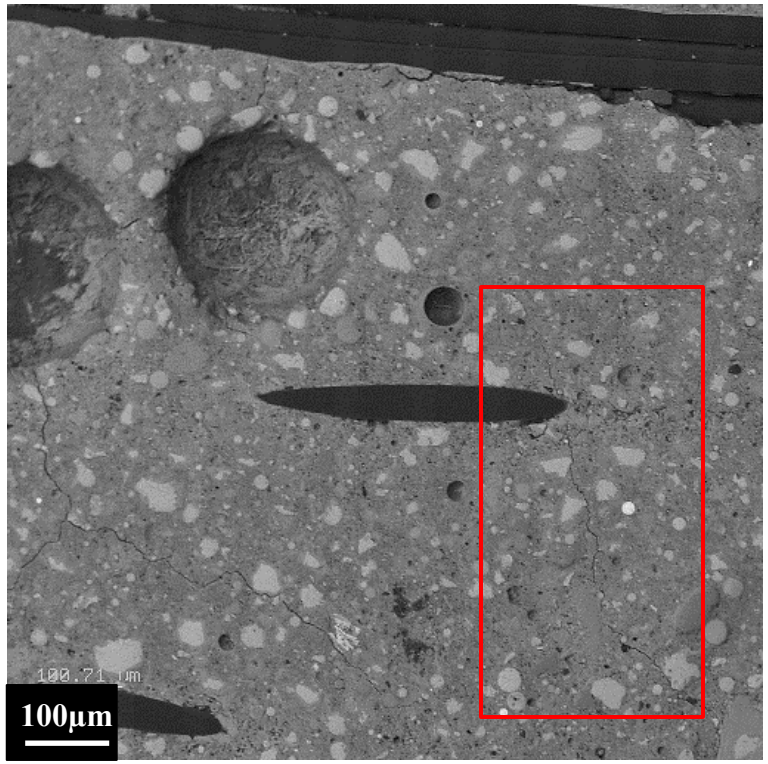
0.6%E5



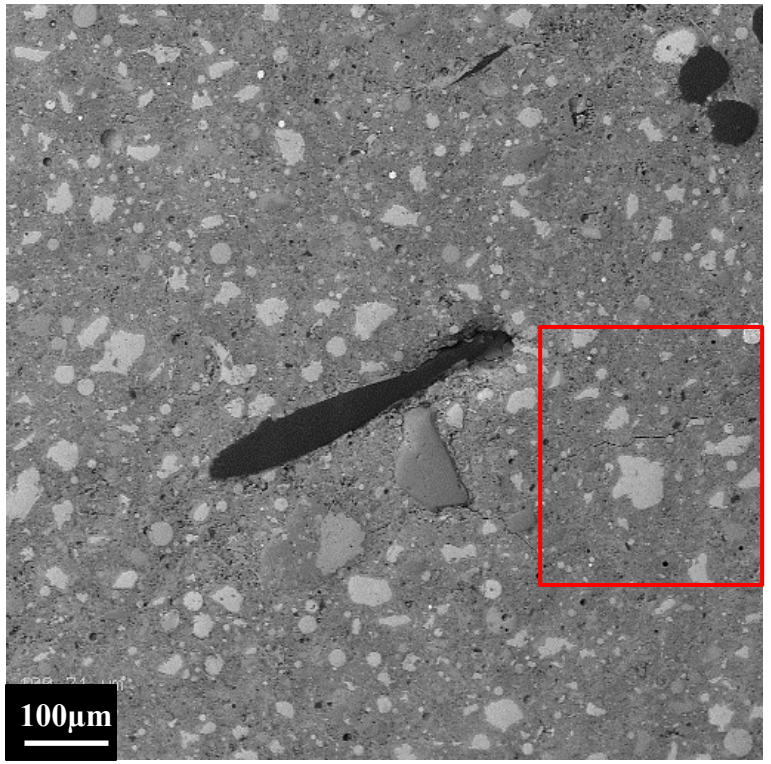
- Samples after 28 days self-healing were used for SEM.
- Cracks were found in the Ref sample especially at the tip of the fibers, which may reflect the quality of the interfacial transition zone (ITZ).

SEM – (28 days self-healed sample)

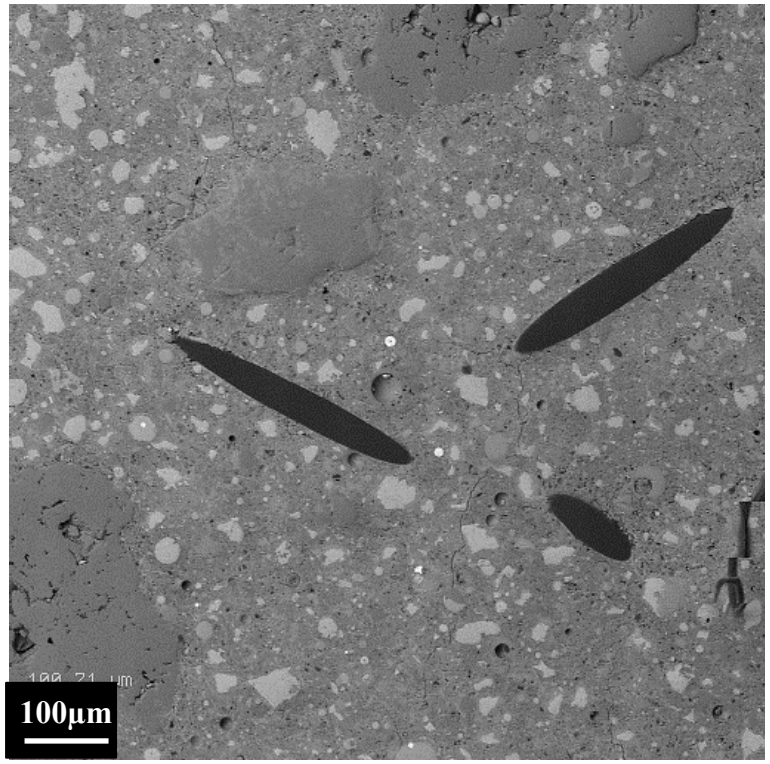
Ref



0.3%E5



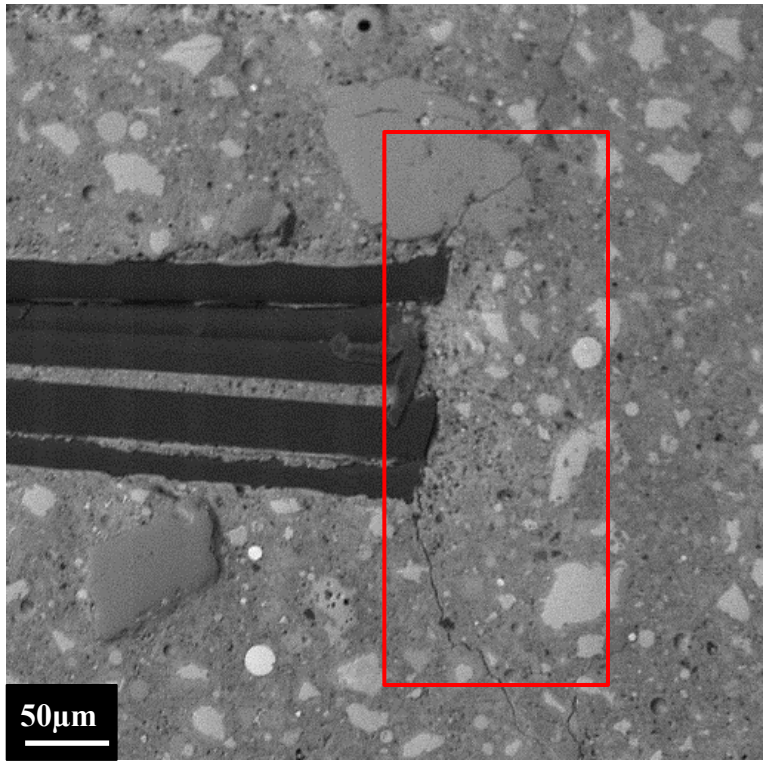
0.6%E5



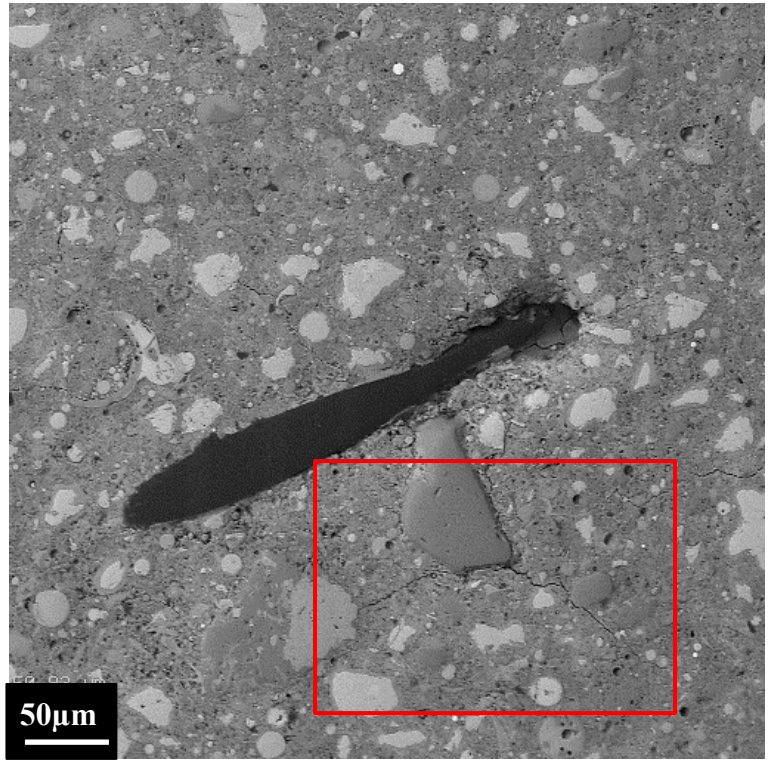
- Cracks were also found in the sample with E5, but mostly in the matrix. (the sample used for SEM were pre-damaged sample after 28 days healing, therefore cracks were expected)

SEM – (28 days self-healed sample)

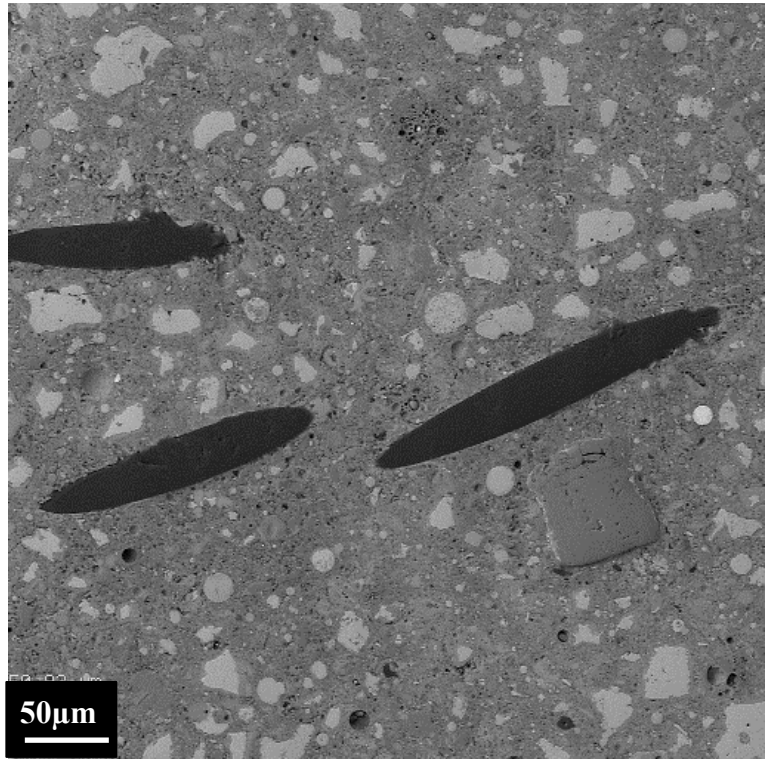
Ref



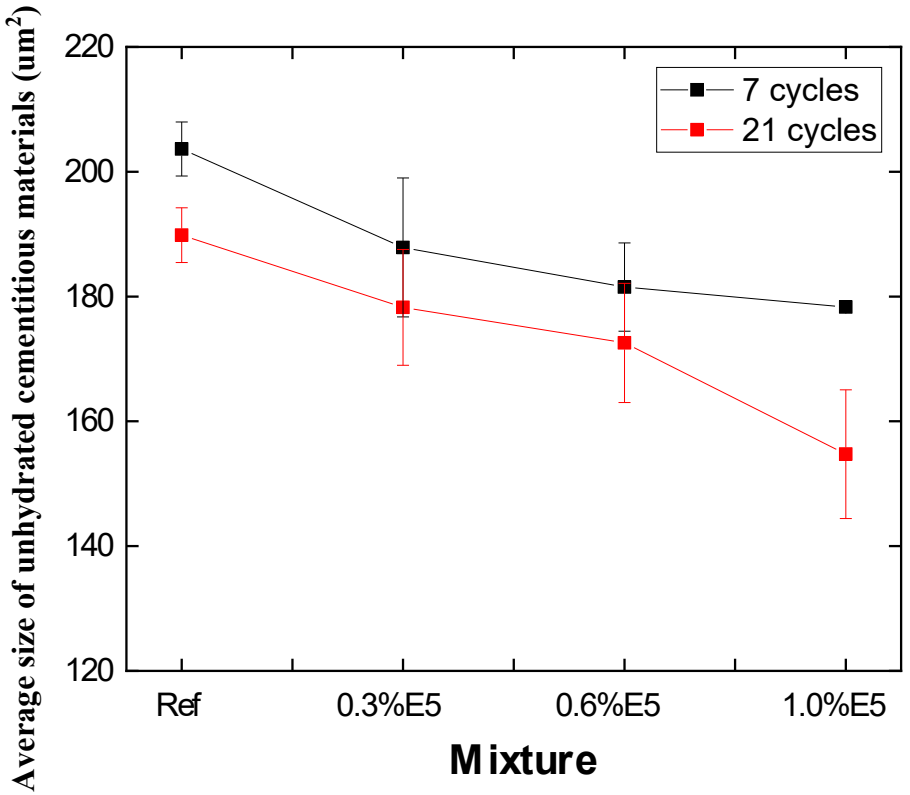
0.3%E5



0.6%E5



SEM for SHCC-E5 and image analysis



- As the curing age extended, the size of the unhydrated particles reduced.
- The addition of the E5 led to a decrease of the size of the unhydated particles, which implies an improvement of the hydration.

Summary

Study of E5

1. The incorporation of E5-Internal cure improved the water retention capacity of the cement paste, which indicates its internal curing effect;
2. The viscosity of the cement paste was only slightly affected by the E5-Internal cure.
3. Under the moisture curing, the compressive test for the lab-prepared sample indicates the addition of *E5-Internal cure* slightly increased the compressive strength;
4. Large slab testing results indicate higher strength of E5 samples compared to references.
5. The incorporation of the E5 not only increased the strength of the concrete, but also improved the long-term protection of concrete from chloride penetration.
6. The internal curing effect of the E5 is been proven by the internal humidity measurement and drying shrinkage test.
7. From SEM images, sample with E5 exhibited denser matrix with higher quality and the smaller un-hydrated cement particles suggest an improved hydration process.
8. The investigation of the SHCC-E5 indicates that the E5 can improve the ductility and self-healing ability of the design mortar.

Summary

Study of LFA

1. Large slab testing results indicate higher strength of LFA samples compared to references.
2. The investigation of the LFA suggests that the early age compressive strength was improved by the incorporation of the LFA
3. The drying shrinkage of the sample could be slightly reduced with the addition of the LFA.
4. A denser matrix was observed from the sample with LFA.

Future works…

- Corrosion test for concrete with E5
- Micro-CT for concrete with E5
- Continue SEM analysis for sample at different ages
- Further investigate the floc network formed by the E5

Appendix

Workability (w/o WRA)

Mixture	Sand Condition	Slump (in)
0.42 Ref	Dried sand	0.75
0.42 E5-0.3%	Dried sand	0.50
0.42 E5-0.6%	Dried sand	1.50
0.42 E5-1.5%	Dried sand	0.50
0.42 E5-3.0%	Dried sand	1.00

0.42 Ref
Dry sand

0.42 E5-0.3%
Dried sand

0.42 E5-0.6%
Dried sand

0.42 E5-1.5%
Dried sand

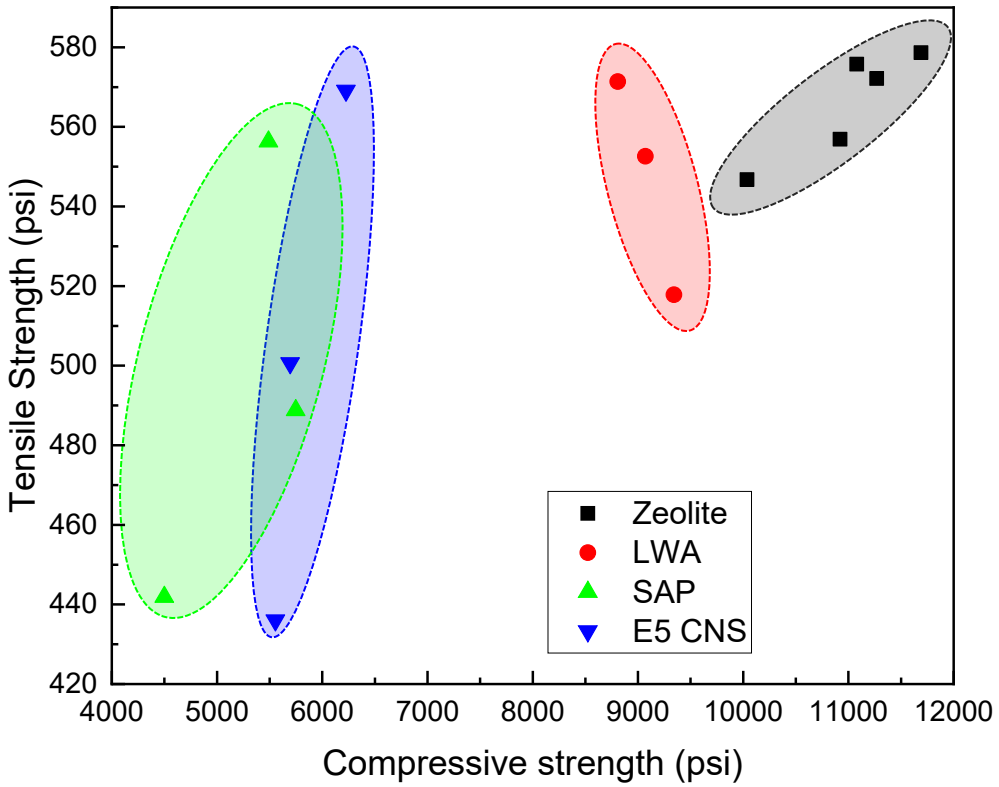
0.42 E5-3.0%
Dried sand

Recommendation Table

Mix	Compressive Strength	Tensile Strength	Ductility	Self-Healing Performance	Workability	Note
Zeolite SHCC	★★★	★★★	★★☆	★★☆	Good	Need to prewet zeolite, Expensive
LWA SHCC	★★☆	★★☆	★★☆	★☆☆	Good	Need to prewet LWA
SAP SHCC	★☆☆	★☆☆	★★☆	Self-Sealing	Poor	Leave large pores
E5 SHCC (w/ VMA)	★★☆	★★☆	★★★	★★★	Good	-

- Zeolite based SHCC presented good mechanical strength and workability, however, the zeolite is expensive, and it is not convenience to access in the Indiana.
- Other the other hand, E5 based SHCC performed good mechanical strength and excellent self-healing performance and ductility is more applicable in the practical engineering.

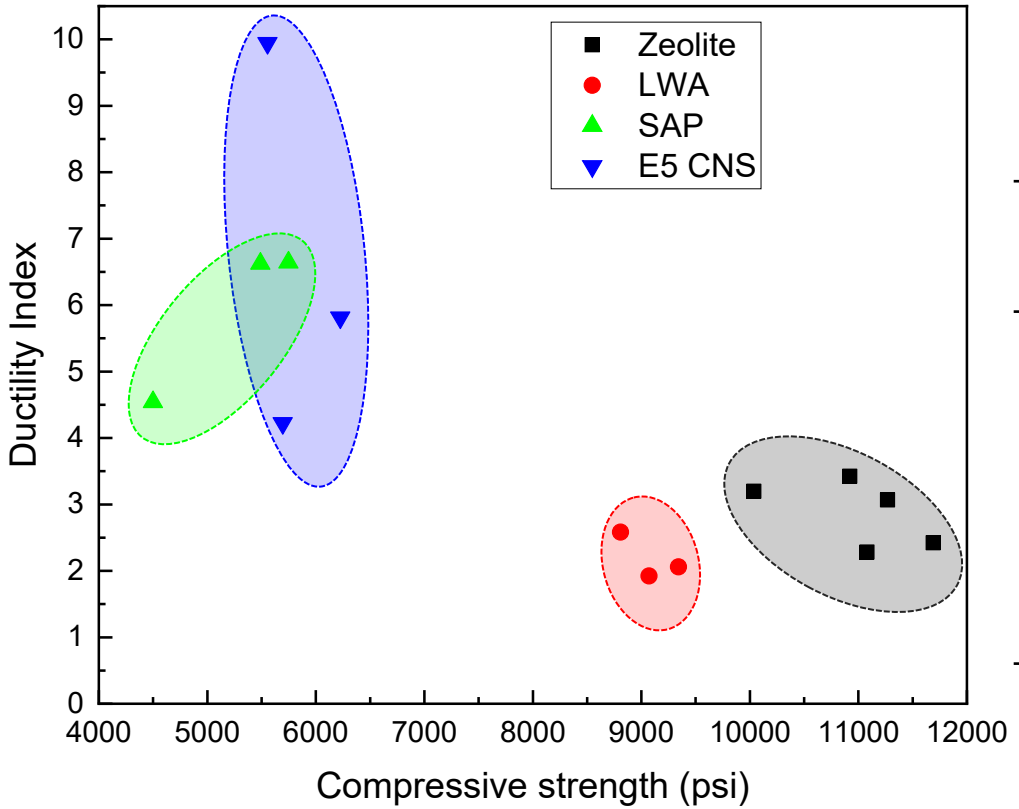
Mechanical performance of different mixtures



Mix	W/C	Fly Ash (% cement wt.)	Slag (% cement wt.)	PVA Fiber (volume)
Zeolite SHCC	0.35	0.15	0.15	2%
LWA SHCC	0.35	0.15	0.15	2%
SAP SHCC	0.35	0.15	0.15	2%
E5 CNS SHCC (w/ VMA)	0.35	0.50	0	1%

- In general, most of the SHCC mix can reach 5000 psi for compressive strength and 400 psi for tensile strength
- Zeolite based SHCC shown highest compressive and tensile strength because the higher water absorption rate of zeolite for internal curing compare with others internal curing agents.
- CNS SHCC mix has higher fly ash content which results in lower mechanical strength.

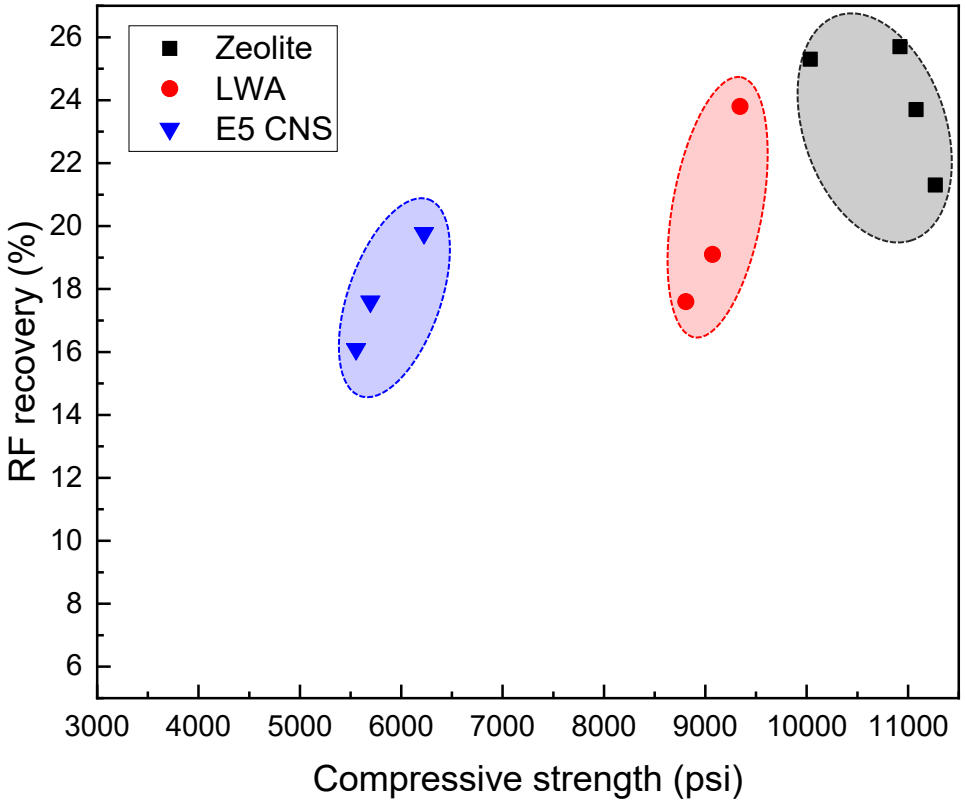
Ductility Performance



Mix	W/C	Fly Ash (% cement wt.)	Slag (% cement wt.)	PVA Fiber (volume)
Zeolite SHCC	0.35	0.15	0.15	2%
LWA SHCC	0.35	0.15	0.15	2%
SAP SHCC	0.35	0.15	0.15	2%
E5 CNS SHCC (w/ VMA)	0.35	0.50	0	1%

- All the SHCC mix presented strain-hardening behavior with the ductility index greater than 1.
- E5 CNS SHCC shown highest ductility due to the incorporation of VMA and nano silica which optimized interfacial properties between fiber and matrix even the fiber volume is lower than other sets.

Self healing Efficiency



Mix	W/C	Fly Ash (% cement wt.)	Slag (% cement wt.)	PVA Fiber (volume)
Zeolite SHCC	0.35	0.15	0.15	2%
LWA SHCC	0.35	0.15	0.15	2%
E5 CNS SHCC (w/ VMA)	0.35	0.50	0	1%

- All the SHCC sample shown satisfied self-healing performance after 28 days wet-dry cycles (7 cycles).
- Due to the lower pre-damage level (0.5% tensile strain) of zeolite SHCC and LWA SHCC, these two samples present slightly higher RF recovery ratio than nano-silica based SHCC (pre-damage 0.8% tensile strain)