Microstructural and thermodynamic analyses elucidate the effects of expansive agent and shrinkage reducing admixture on performance of fiber-reinforced mortar

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Outline

- Investigation method
- Experimental methods
- Experimental results
- Mechanical and shrinkage properties
- Microstructural analysis
- Thermodynamic analysis
- Conclusions

Investigation method

- **Mechanical and shrinkage properties**
  - Compressive strength
  - Fiber pull-out
  - Shrinkage

  Measuring effect of EA and SRA on FRM macro mechanical properties

- **Microstructure analysis**
  - XRD
  - TGA
  - MIP
  - SEM EDS
  - Micro indentation

  Measuring effect of EA and SRA on FRM microstructure

- **Thermodynamic analysis**
  - Phase assemblage
  - Compressive strength
  - Chemical shrinkage

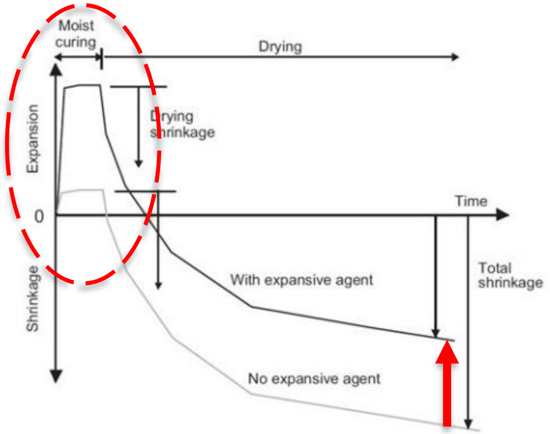
  EA and SRA effects on FRM based on thermodynamic principles

Materials

- **Expansive agent (EA)**

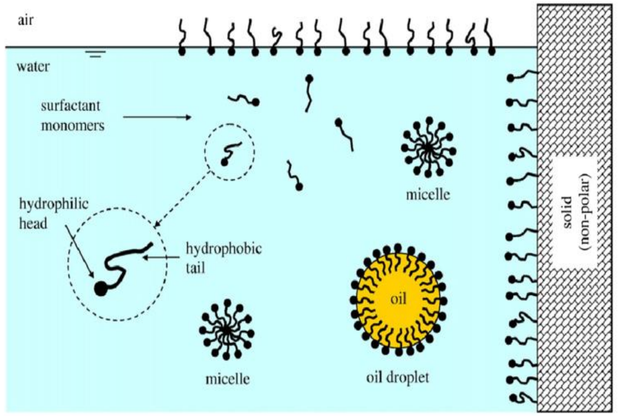
Initial expansion compensates for shrinkage

CaO-based (Portlandite) $CaO + H_2O \rightarrow Ca(OH)_2$



- **Shrinkage reducing admixture (SRA)**

Non-ionic surfactant reduces the surface tension of pore solution.



Experimental methods

Mixture matrix

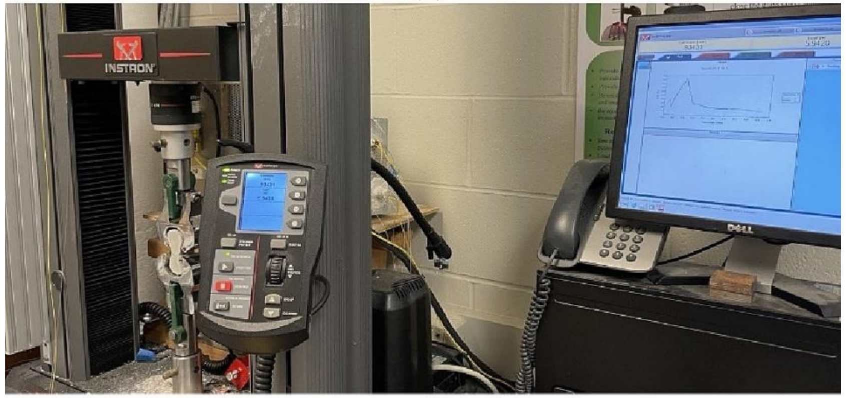
Mixture	Replacement ratio (%)	
	EA	SRA
REF	0	0
10EA	10	0
2SRA	0	2
10EA2SRA	10	2

w/cm = 0.4

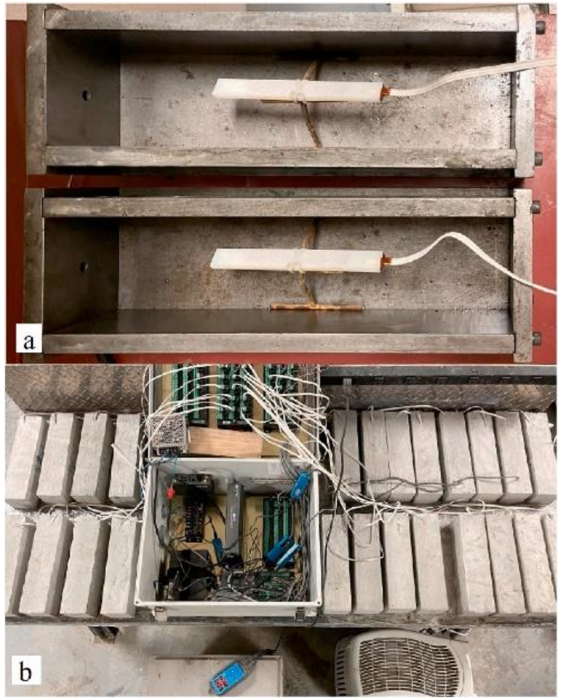
Steel fiber = 0.5%

Aspect ratio = 65

(a)

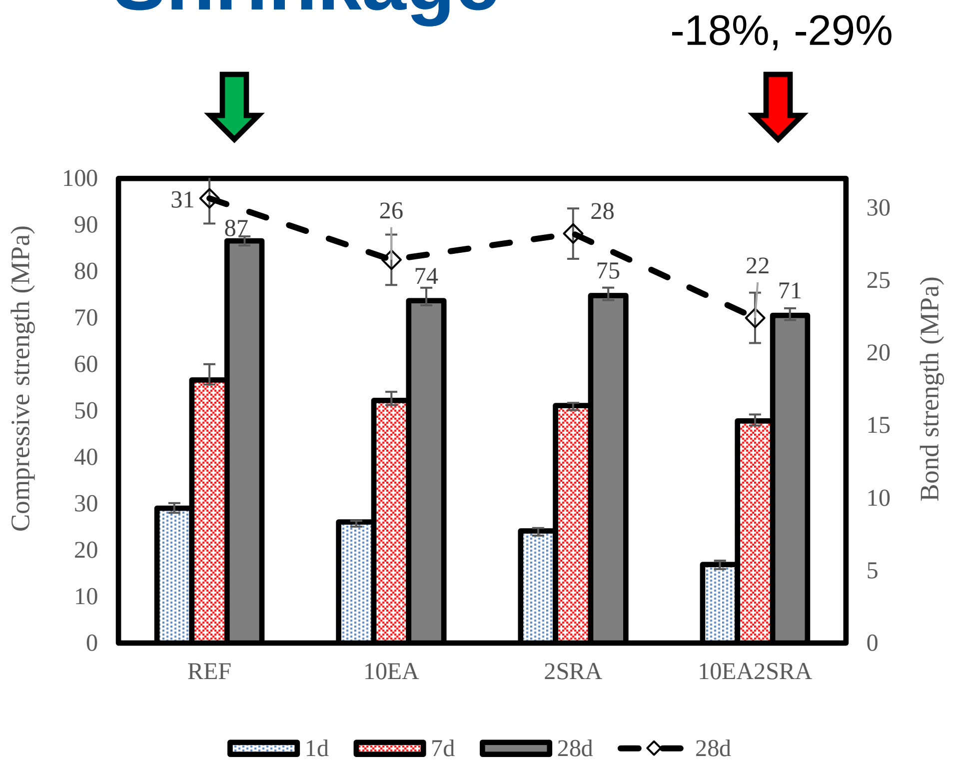


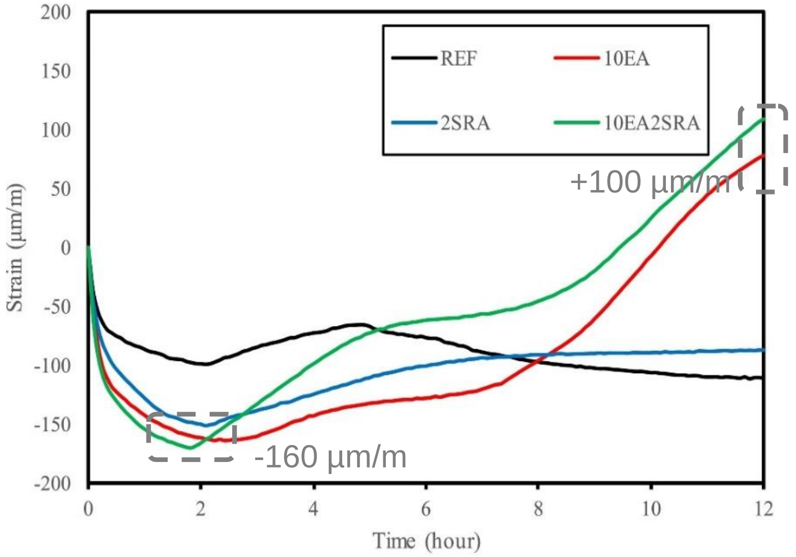
(b)



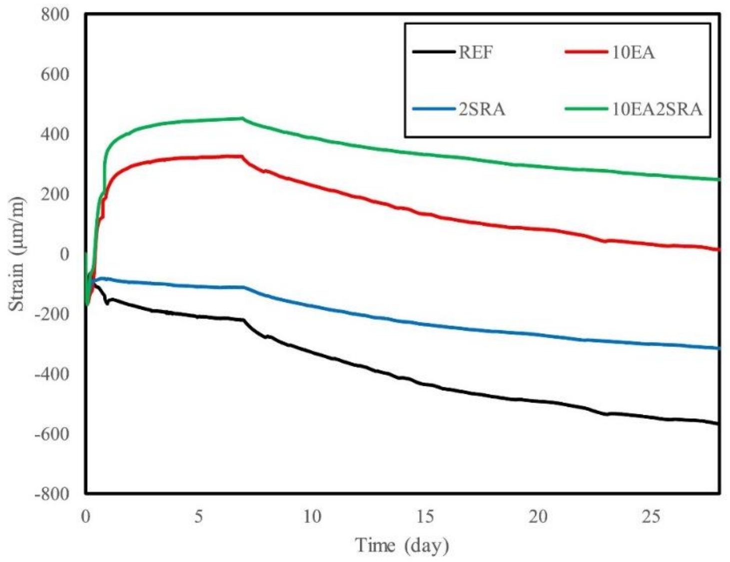
Experimental results: Mechanical and shrinkage properties

Compressive strength
Fiber pull-out
Shrinkage





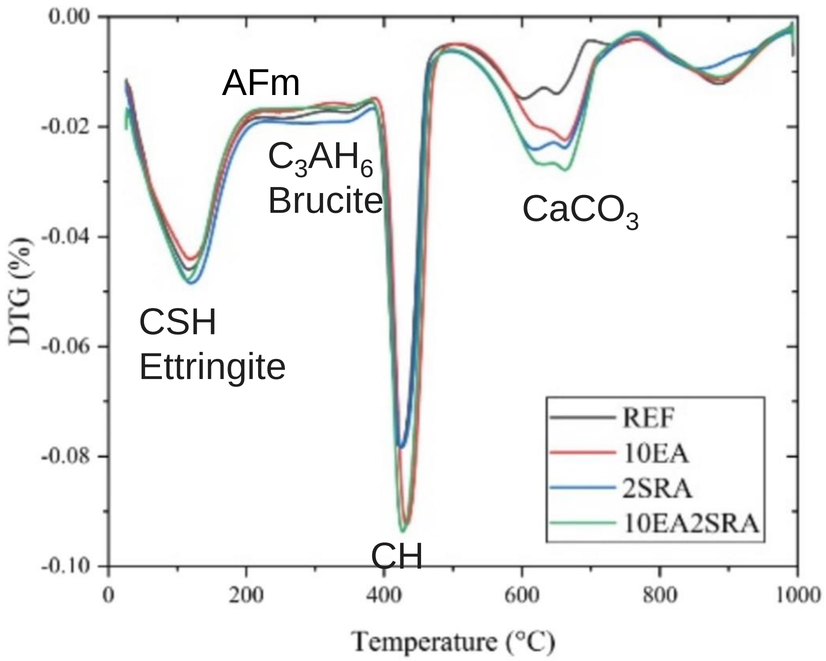
Effect of EA and SRA on initial (12h) shrinkage



Effect of EA and SRA on 28d shrinkage

Experimental results: Microstructural analysis

TGA
XRD
MIP



TGA

Mixture	Median pore size (nm)	Total porosity (%)	Percentage of pore type on total pore volume (%)		
			Gel pores (d < 30 nm)	Capillary pores	Macro pores
REF	38	19.4	21.4	78.3	0.3
10EA	71	26.3	17.6	41.1	41.3
2SRA	31	20.7	19.5	80.4	0.1
10EA2SRA	53	25.9	18.0	73.1	8.9

MIP

Mixture	Ettringite (wt%)	Portlandite (wt%)	C_3S/C_2S (wt%)
REF	3.5	13.2	6.0
10EA	2.8	19.3	5.5
2SRA	3.4	13.1	6.3
10EA2SRA	3.5	14.4	5.3

XRD

Experimental results: Microstructural analysis

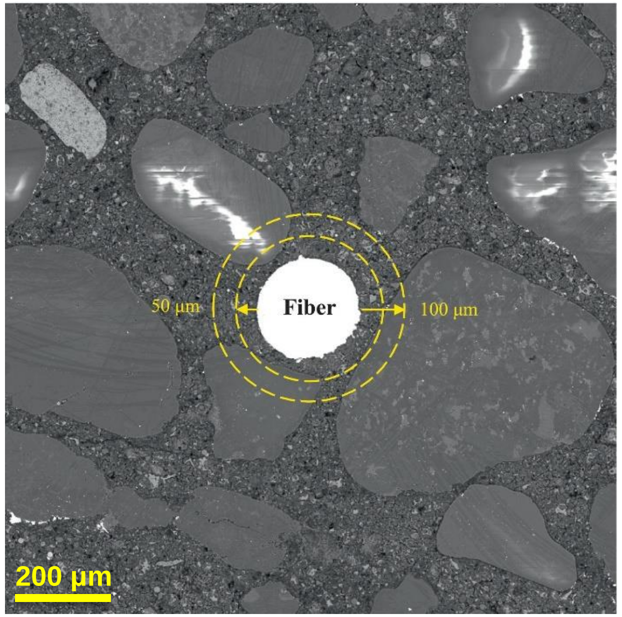
SEM EDS point scanning

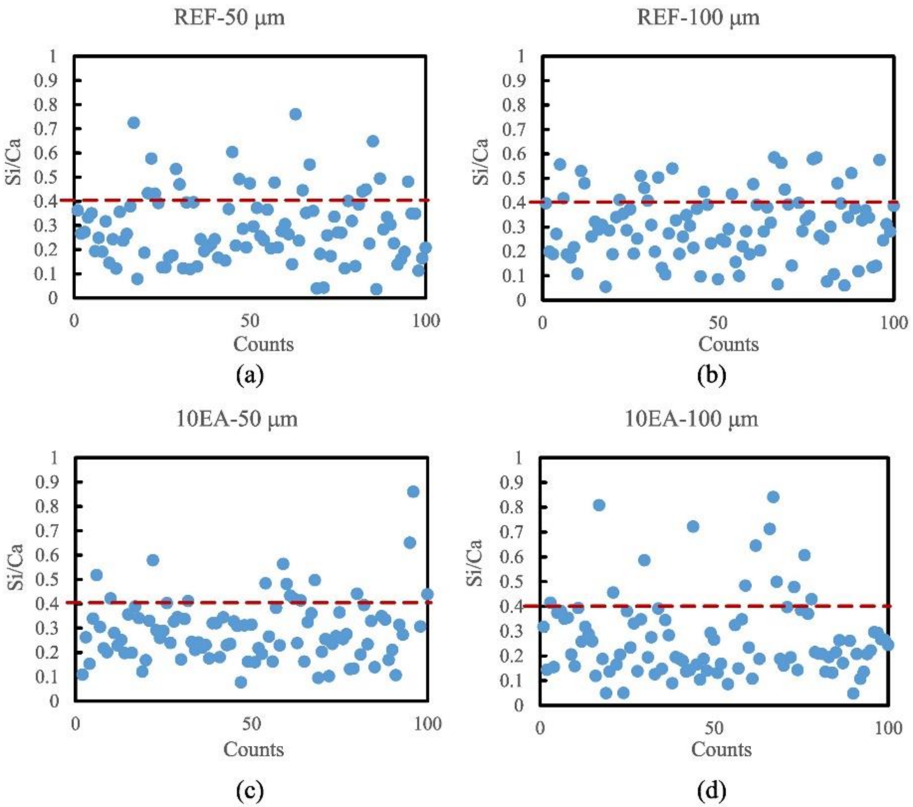
Si/Ca=0.4-1.25 → CSH

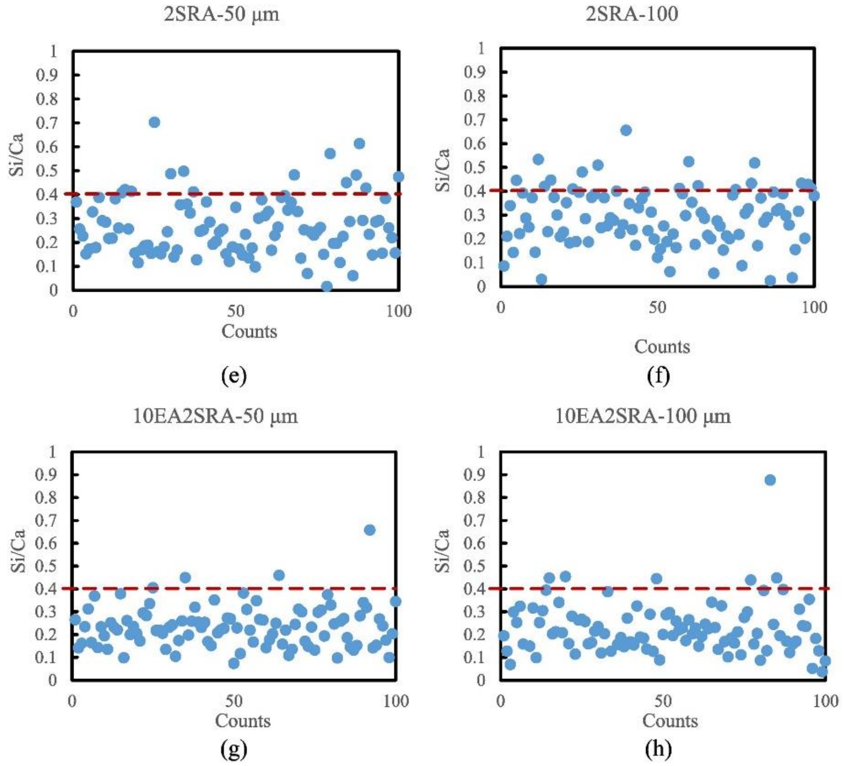
Higher Si/Ca → longer silicate chain

enhanced hardness and elastic modulus

Higher Ca → CH, AFt, and AFm

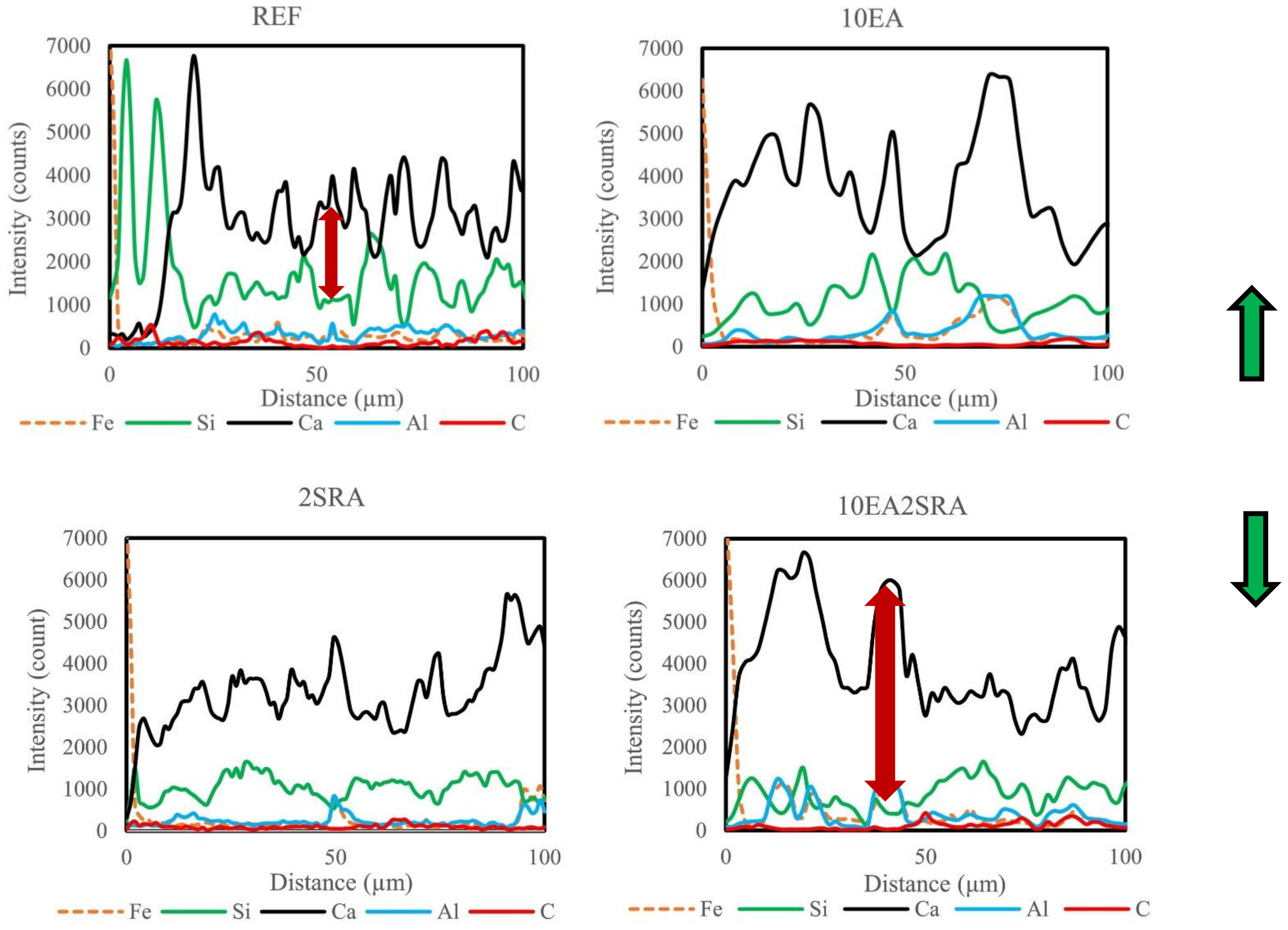






Experimental results: Microstructural analysis

SEM EDS line scanning



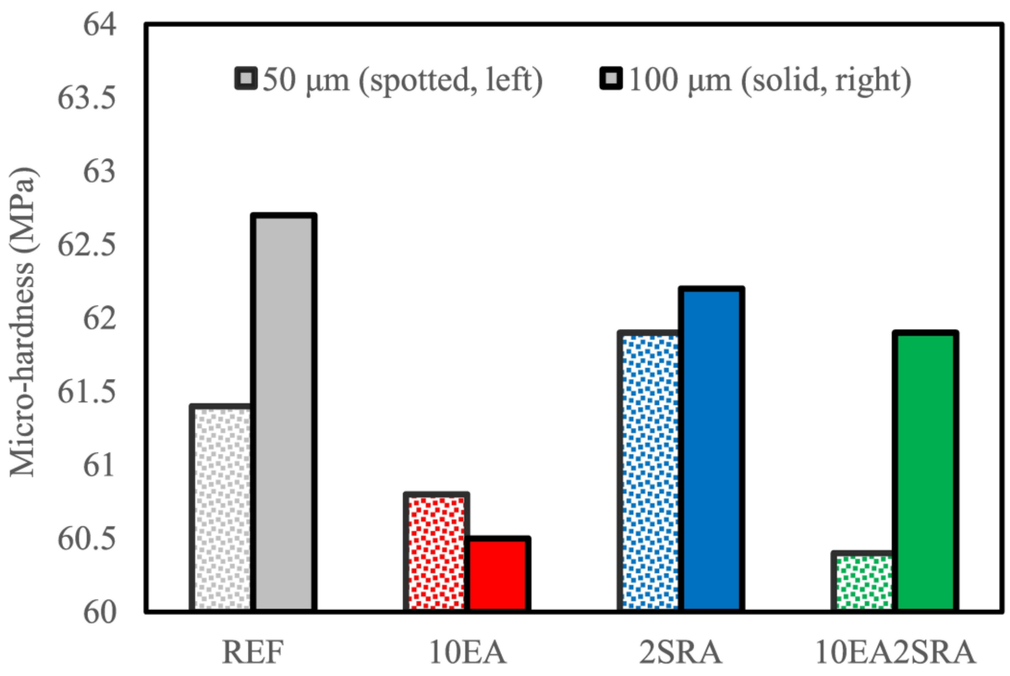
Si/Ca > 0.4

Gap between Si and Ca

Narrow for REF

Wide for 10EA2SRA

Micro-hardness



10EA
10EA2SRA → Lower strength

10EA
Strength reduction between 50 and 100 µm

GEMS (Gibbs Energy Minimization)

- Minimizing Gibbs energy of a complex chemical system at given T and P.
- Mass balances are computed based on equilibrium phase assemblages and speciation in the aqueous phase, from total bulk composition.

$\Delta G = \Delta H - T\,\Delta S$

H: enthalpy (total energy)
T: temperature
S: entropy (degree of disorder)

Cementitious materials

GEMS SELEKTOR

Calcite, Portlandite, Gypsum, CSH, AFt, AFm, etc.

Ca^{2+} $CaOH^{-}$ $HSiO^{3-}$ SO_4^{2-}

Solid, Aqueous, & Gaseous Phases

Thermodynamic analysis

Input

Mixture proportioning & chemical compositions of binder materials

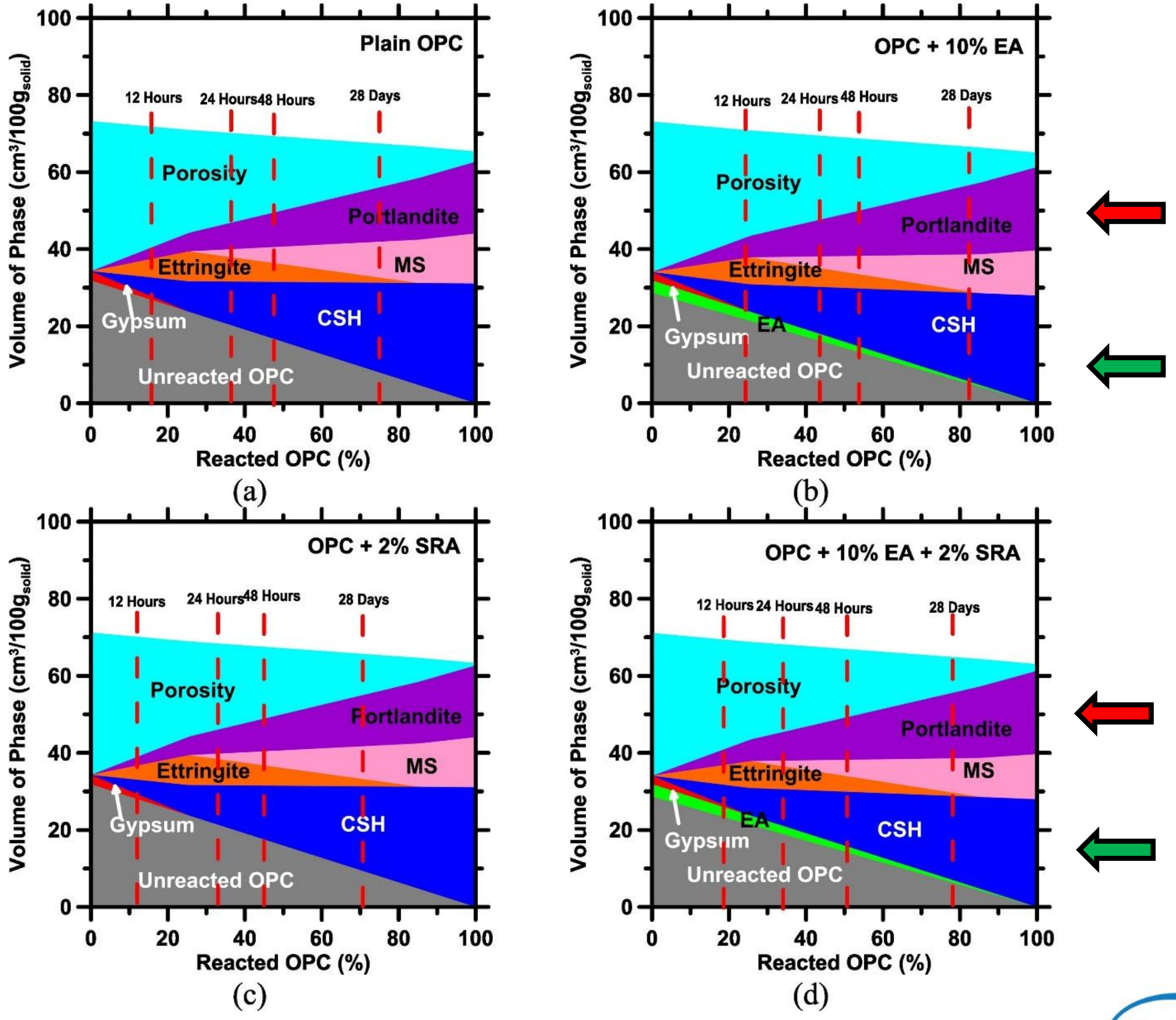
Output

- Hydrates (phase assemblages)
- Degree of reaction of binder (cumulative heat release)
- Porosity
- Chemical shrinkage
- pH

Applicable to OPC and blended systems

Thermodynamic analysis

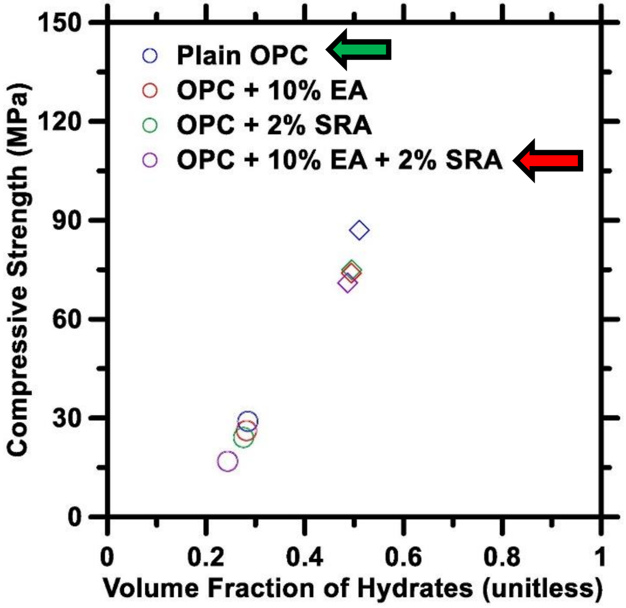
Phase assemblage

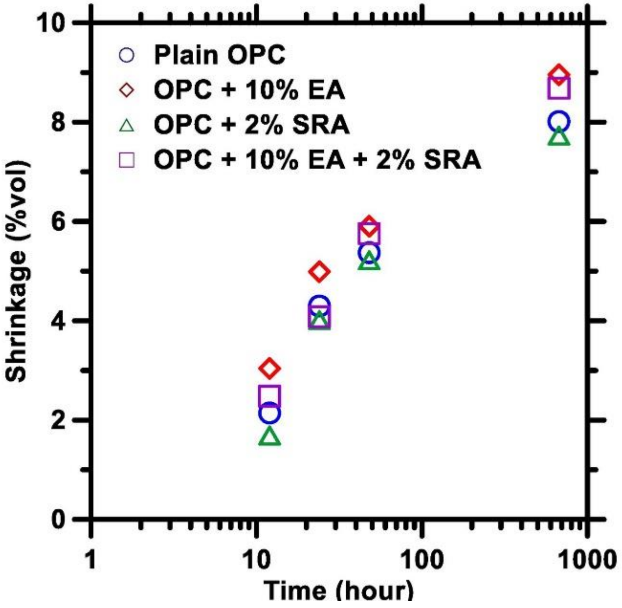


10EA
10EA2SRA

Less CSH
More CH
More porosity

Compressive strength
Chemical shrinkage





Conclusions

1. EA and EA+SRA eliminated shrinkage after 10h and remained in expansion after 28 days → high CH.
2. High content EA/SRA reduced mechanical properties (18% f'_c, 29% fiber pull-out).
3. EDS LS indicated strong fiber-matrix ITZ for REF → higher fiber pull-out strength. Large gap between Si and Ca with the use of EA/SRA indicated lower density and quality of microstructure.
4. EDS PS showed that Si/Ca reduced with EA/SRA. Higher Si/Ca over 0.4 was observed in the strip between 50 and 100 μm (confirmed by Micro-hardness).
5. MIP results indicated higher porosity using high contents of EA/SRA, especially 10EA (higher macro pores), while lowest for REF mixture.
6. Thermodynamic simulations → phase assemblages of mixtures. 10EA formed more CH but less CSH. 10EA2SRA while effective in shrinkage elimination exhibited lowest mechanical properties.

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References

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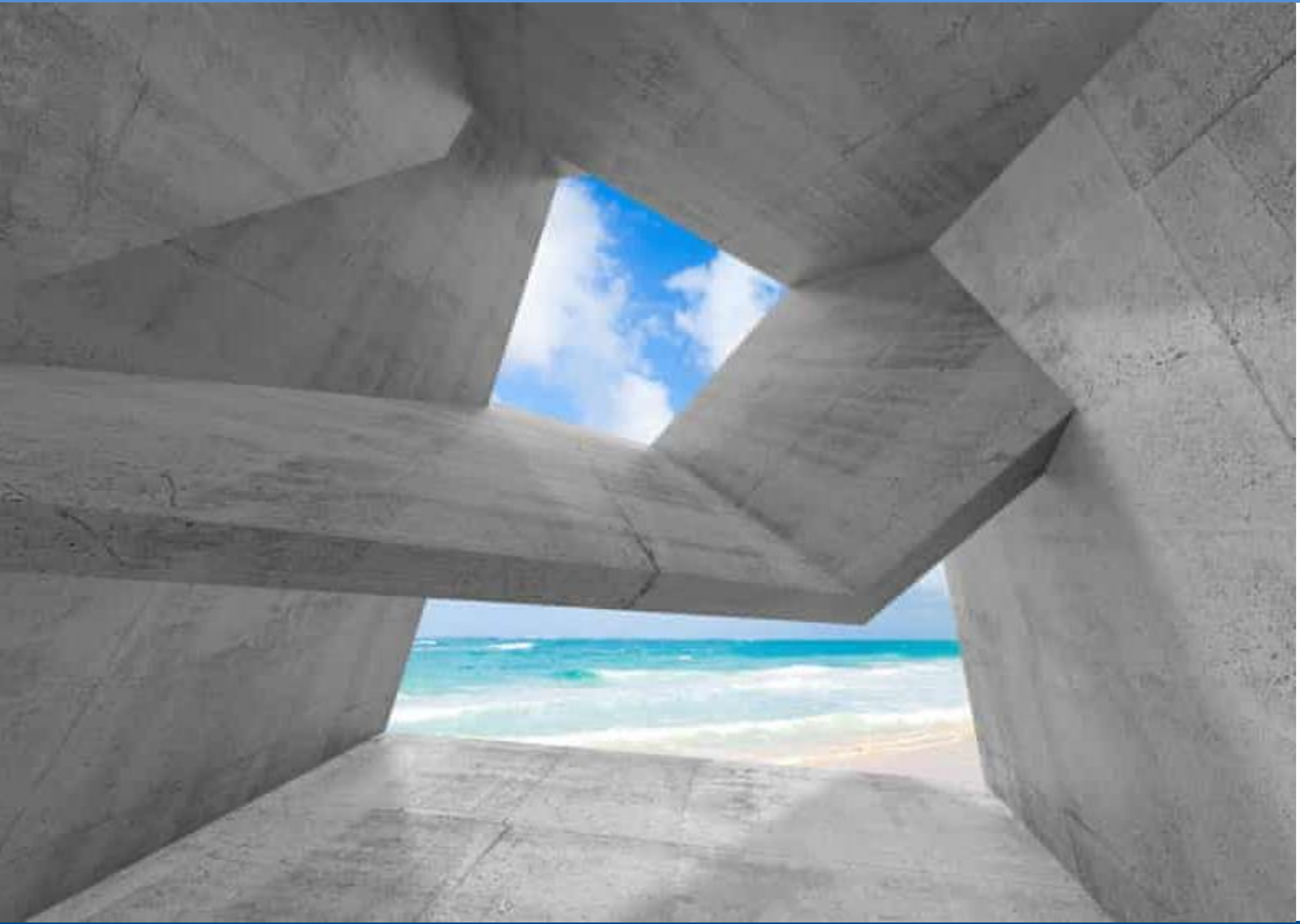

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Any Questions?