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Federal Highway Administration  
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Highway Research Center

# Portland Limestone Cement Variability

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Source: FHWA.



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# Acronyms

AASHTO	American Association of State Highway and Transportation Officials	M1	mixture 1
BR	bulk resistivity	M2	mixture 2
CNS1	colloidal nanosilica number 1	NT	Nordic Test
CNS2	colloidal nanosilica number 2	OPC	ordinary portland cement
CS	colloidal nanosilica	PLC	portland limestone cement
cwt	100 lb of cement by weight	QXRD	quantitative X-ray diffraction
$D_{average}$	average particle size diameter	SCM	supplementary cementitious material
$D_{(xx)}$	particle size diameter at xx percentile	SR	surface resistivity
FHWA	Federal Highway Administration	SRA	shrinkage reducing admixture
$H_u$	ultimate heat of hydration	TGA	thermogravimetric analysis
IL(xx)	Type I and limestone blended cement with xx percent limestone content	w/c	water-to-cement ratio
LOI	loss-on-ignition	w/cm	water-to-cementitious materials ratio
LWA	lightweight aggregate	wt. %	weight percentage



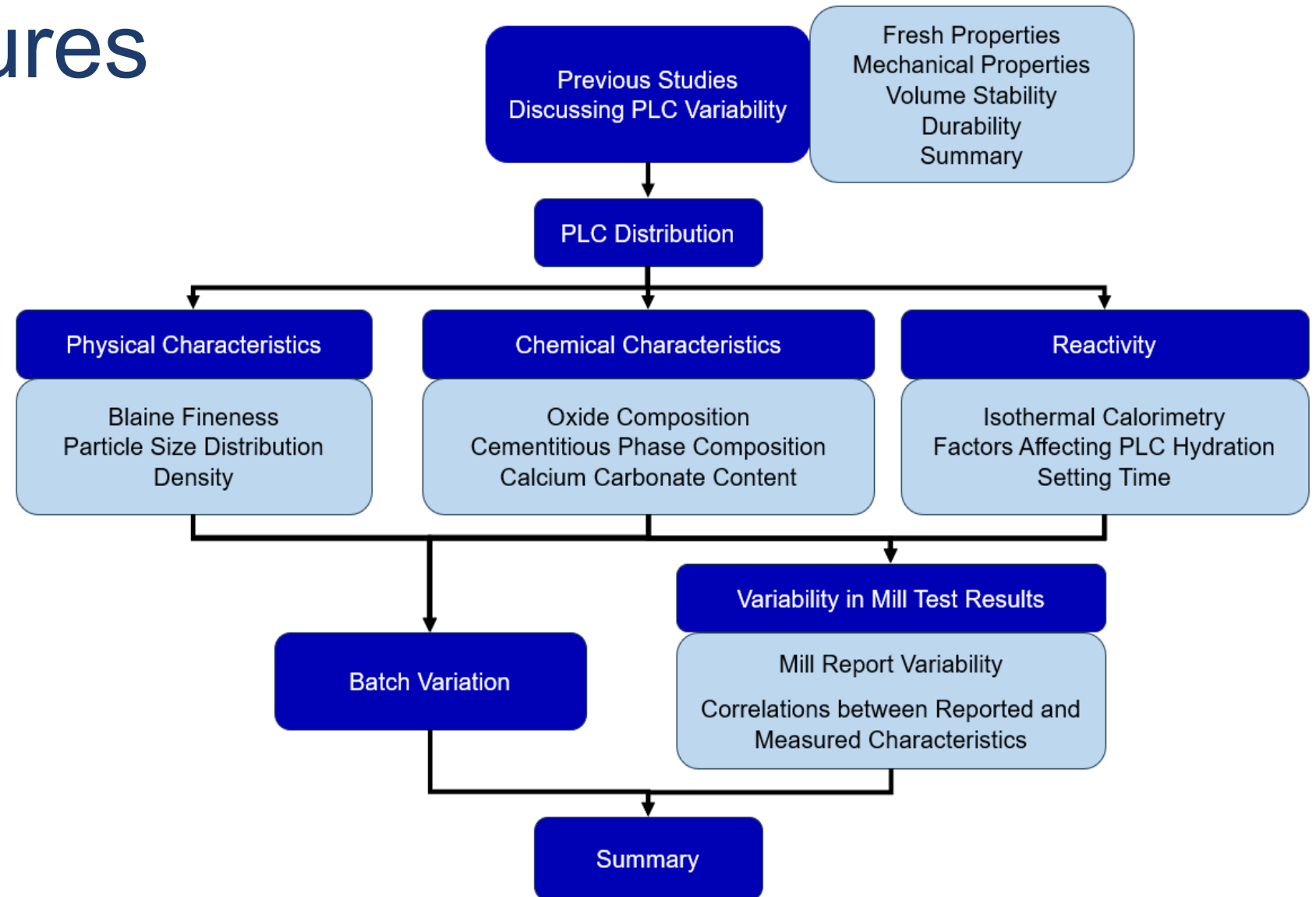
# Chemical Notations

$\text{Al}_2\text{O}_3$	aluminum oxide
$\text{C}_2\text{S}$	dicalcium silicate
$\text{C}_3\text{A}$	tricalcium aluminate
$\text{C}_3\text{S}$	tricalcium silicate
$\text{C}_4\text{AF}$	tetracalcium aluminoferrite
$\text{CaCO}_3$	calcium carbonate
$\text{CaO}$	calcium oxide
$\text{Cr}_2\text{O}_3$	chromium (III) oxide
$\text{Fe}_2\text{O}_3$	iron oxide
$\text{K}_2\text{O}$	potassium oxide

$\text{MgO}$	magnesium oxide
$\text{Mn}_2\text{O}_3$	manganese (III) oxide
$\text{Na}_2\text{O}$	sodium oxide
$\text{P}_2\text{O}_5$	phosphorous pentoxide
$\text{SiO}_2$	silicon dioxide
$\text{SO}_3$	sulfur trioxide
$\text{SrO}$	strontium oxide
$\text{TiO}_2$	titanium dioxide
$\text{ZnO}$	zinc oxide



# Study Features

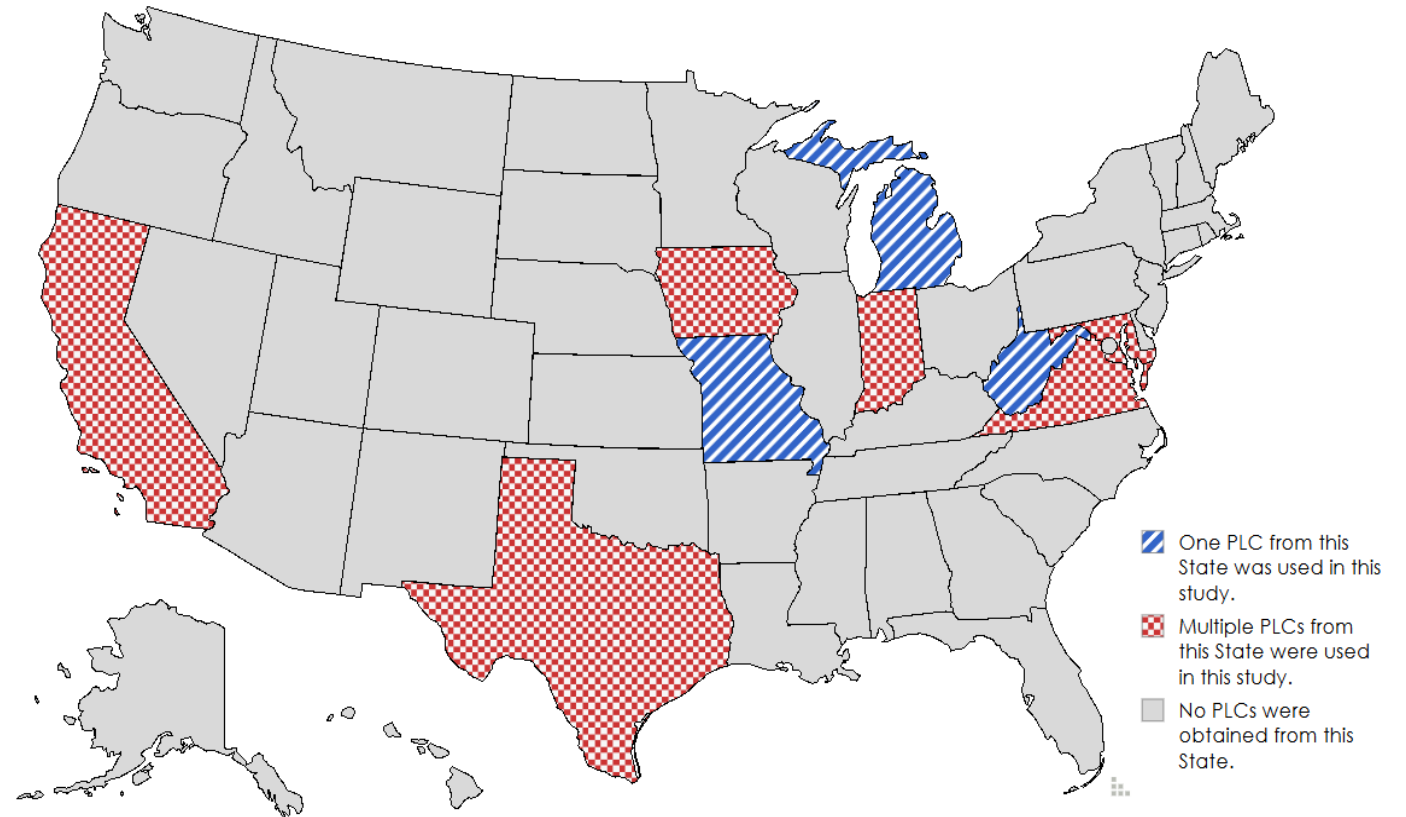


Source: FHWA.



# PLCs

- ▶ Eighteen PLC samples were collected from 13 cement plants in 9 States.
- ▶ PLCs include different production methods, raw material sources, geology, and grinding aids.
- ▶ Four PLC samples were obtained from one cement plant to provide indications of batch-to-batch variability.



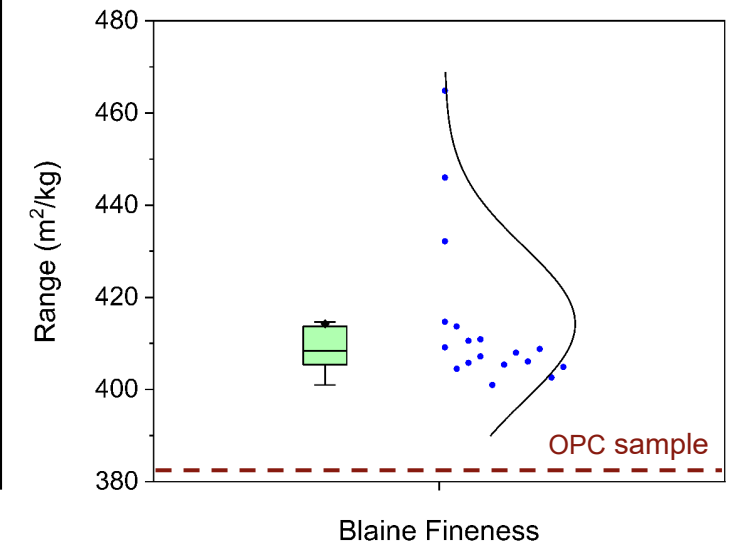
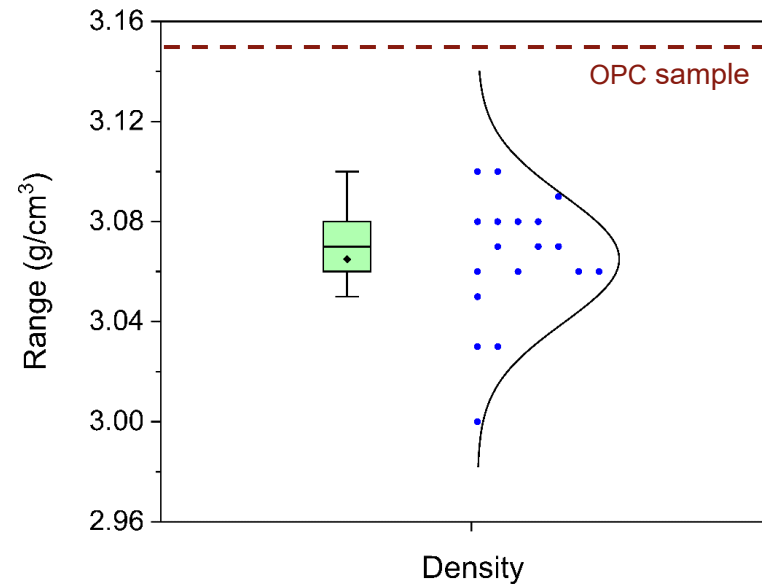
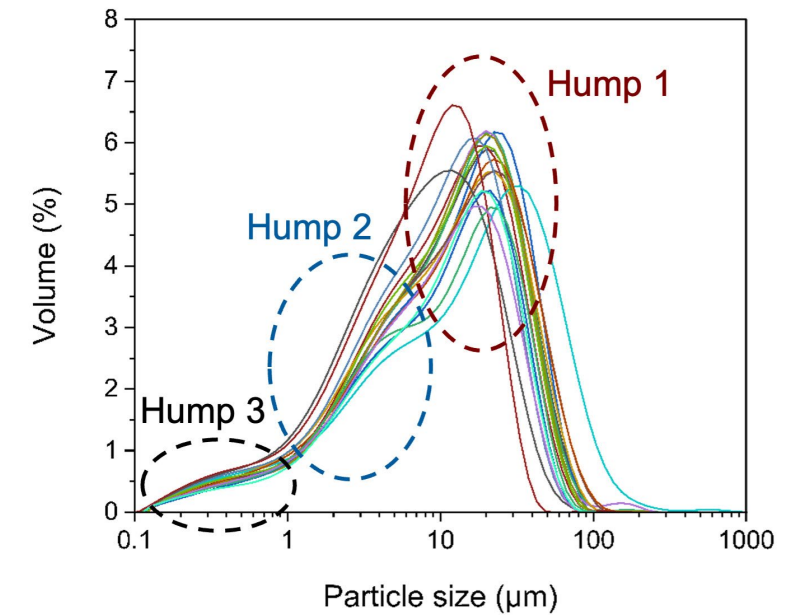
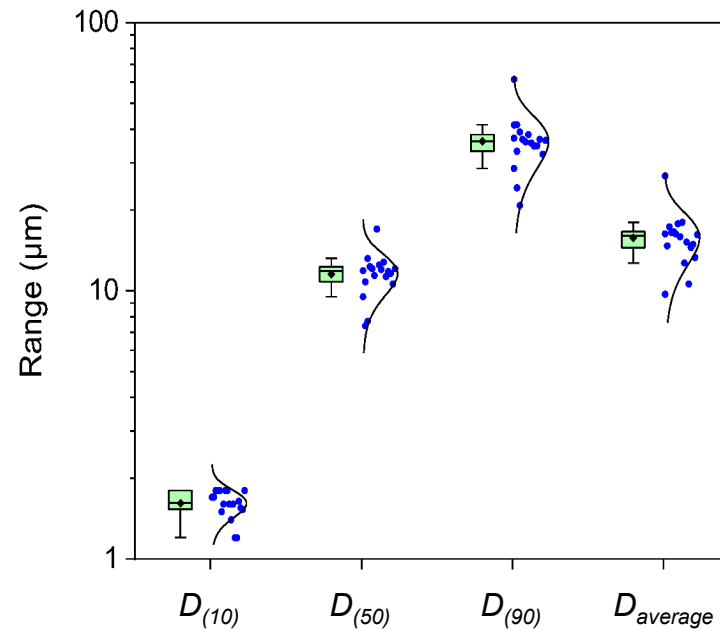
©MapChart.<sup>(1)</sup> Modifications by FHWA.





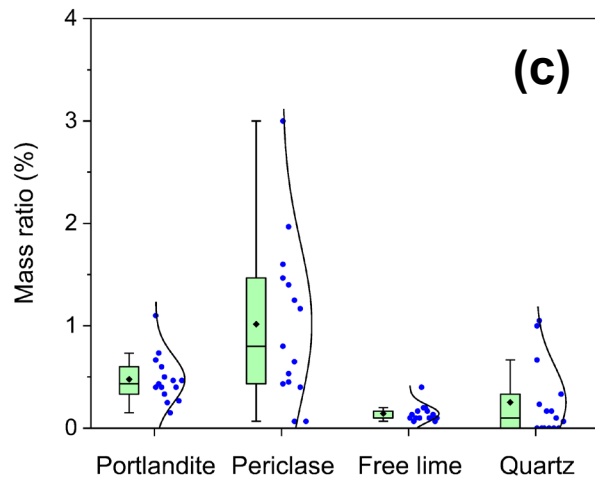
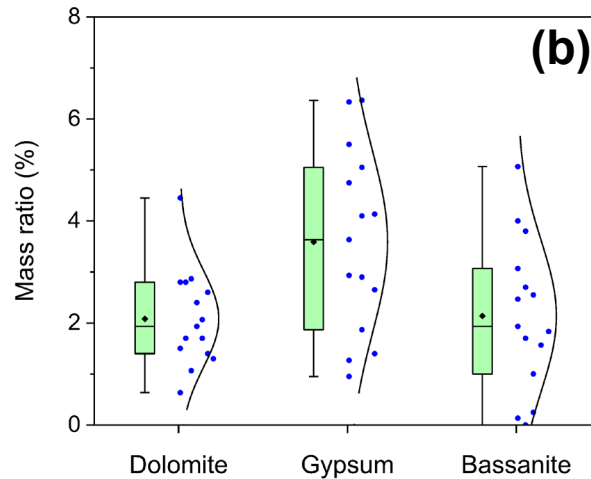
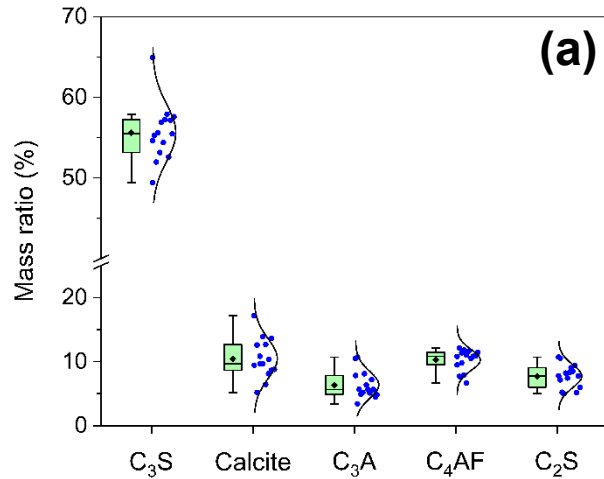
# Physical PLC Variability

- ▶ The finer portions of the PLCs exhibited narrower variability (variability of  $D_{(10)} < D_{(50)} < D_{(90)}$ ).
- ▶ All PLCs featured three distinct particle size groupings.
- ▶ Fifteen out of 18 PLCs exhibited Blaine fineness values ranging from 400 to 415 m<sup>2</sup>/kg.
- ▶ Fifteen out of 18 PLCs exhibited densities ranging from 3.05 to 3.10 g/cm<sup>3</sup>.



All images source: FHWA.

# PLC Phase Composition Variability



Alite (C <sub>3</sub> S)	Calcite (CaCO <sub>3</sub> )	Dolomite (CaMg(CO <sub>3</sub> ) <sub>2</sub> )	Aluminate (C <sub>3</sub> A)	Ferrite (C <sub>4</sub> AF)	Belite (C <sub>2</sub> S)	Portlandite (Ca(OH) <sub>2</sub> )	Gypsum (CaSO <sub>4</sub> ·2H <sub>2</sub> O)	Bassanite (CaSO <sub>4</sub> ·1/2H <sub>2</sub> O)	Total sulfate	Periclase (MgO)	Free lime (f-CaO)	Quartz (SiO <sub>2</sub> )	
* 0.48	-0.2	-0.4	* 0.46	0.2	-0.3	-0.2	-0.3	0.1	-0.3	-0.4	0.2	* 0.56	1-d cumulative heat (J/g PLC)
* 0.49	-0.3	* 0.50	0.1	-0.4	-0.4	0.2	-0.3	0.2	-0.2	-0.4	0.3	* 0.57	3-d cumulative heat (J/g PLC)
0.2	-0.2	* 0.42	0.2	* -0.38	-0.3	0.2	-0.1	-0.1	-0.1	-0.2	0.2	* 0.38	7-d cumulative heat (J/g PLC)
-0.2	0.1	-0.4	0.3	-0.4	-0.1	0.2	0.3	-0.4	0.2	-0.2	-0.1	0.2	H <sub>u</sub> (J/g PLC)

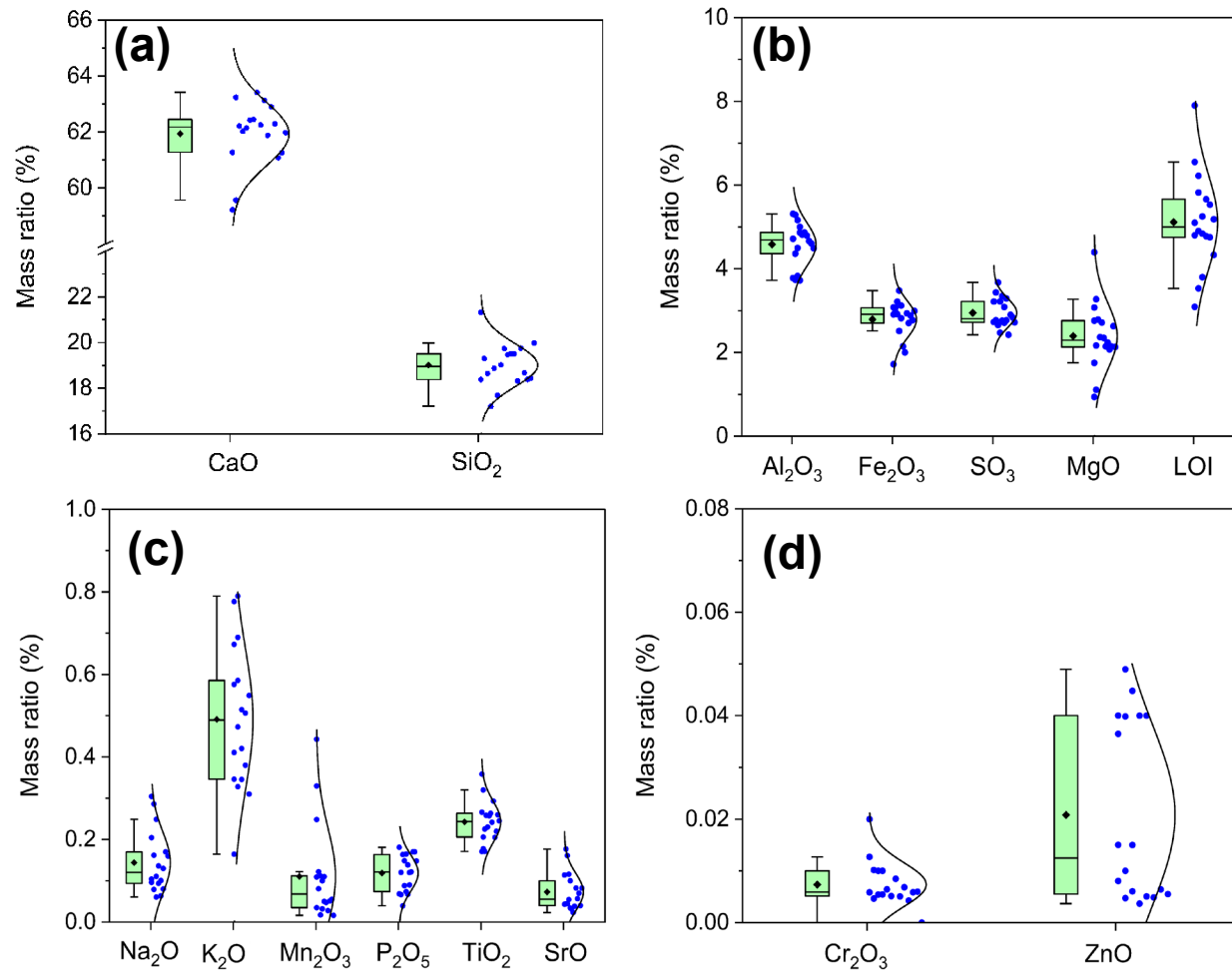
- ▶ Alite and dolomite have high variability and moderate correlation with reactivity.
- ▶ Aluminate had low variability and moderate 1-d reactivity.
- ▶ Gypsum and bassanite have high variability and low correlation with reactivity.

All images source: FHWA.

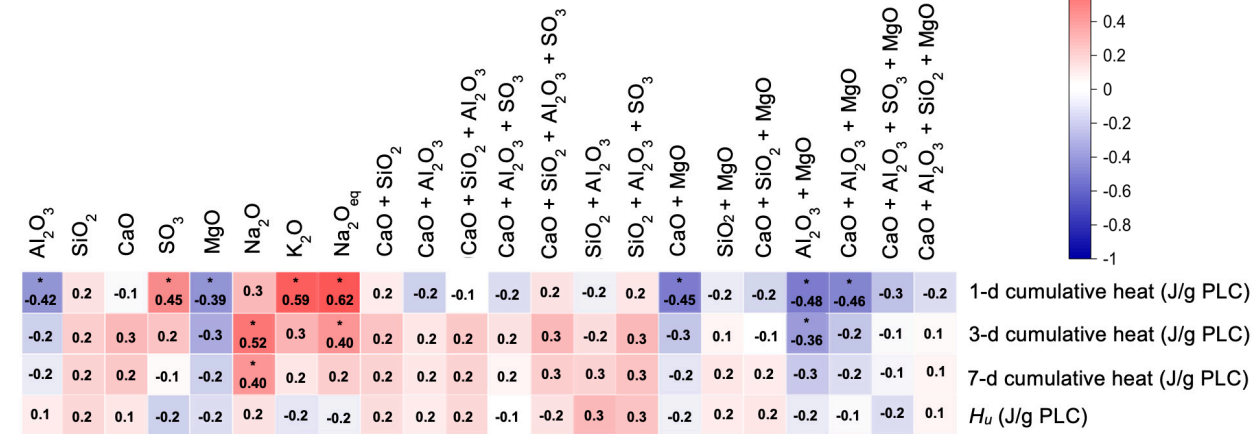




# PLC Oxides Variability



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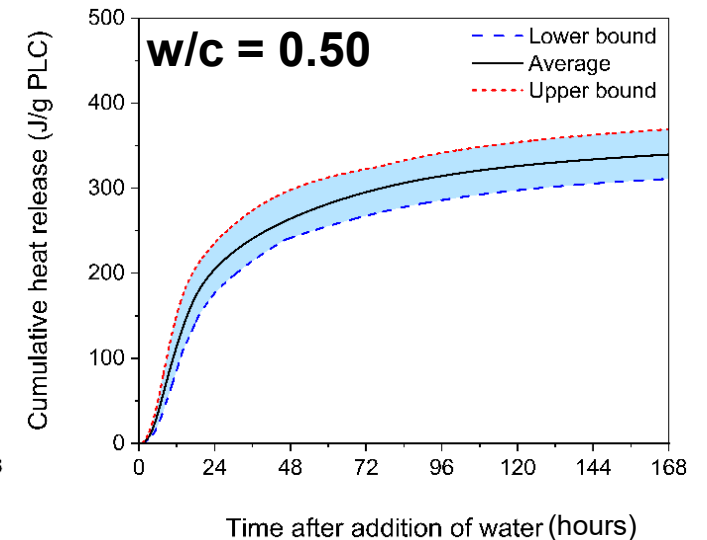
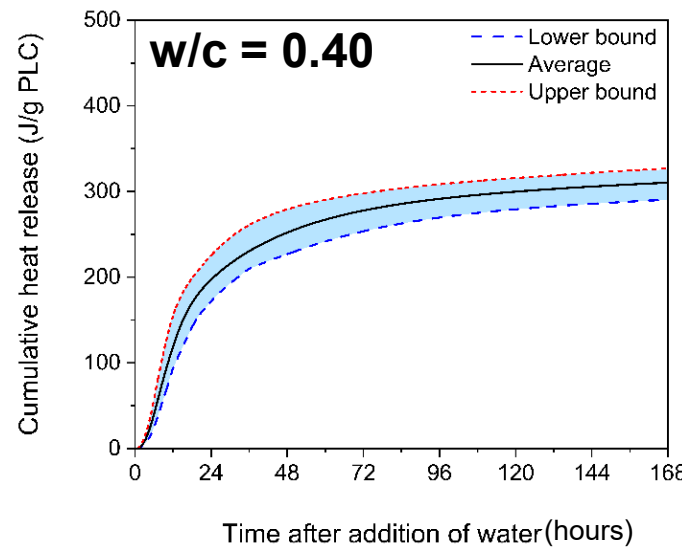
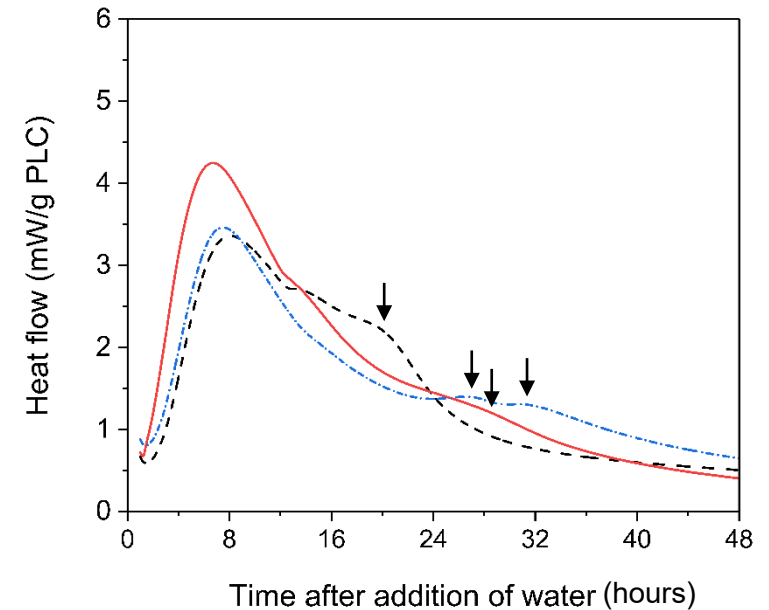


- Moderate variability shown in MgO and moderate correlation with 1-d reactivity.
- SO<sub>3</sub> has tight variability and moderate correlation with 1-d reactivity.
- Alkalis have high variability and high correlation with reactivity.



# PLC Reactivity Variations

- ▶ Renewed aluminate peaks occurred at varying intervals within PLC hydration, ranging from immediately after the alite peak to 32 h after water contact.
- ▶ Cumulative heat release was relatively similar across PLCs when using same w/c.

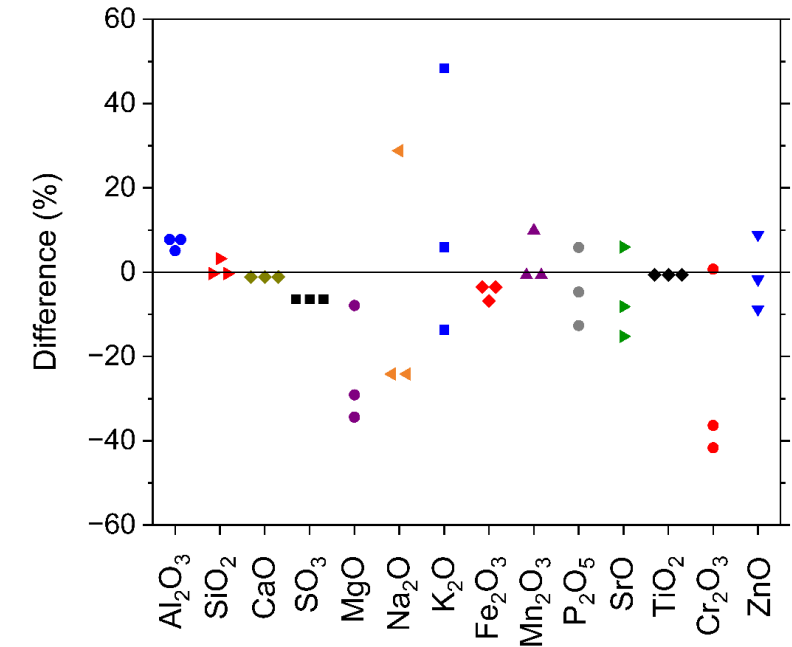
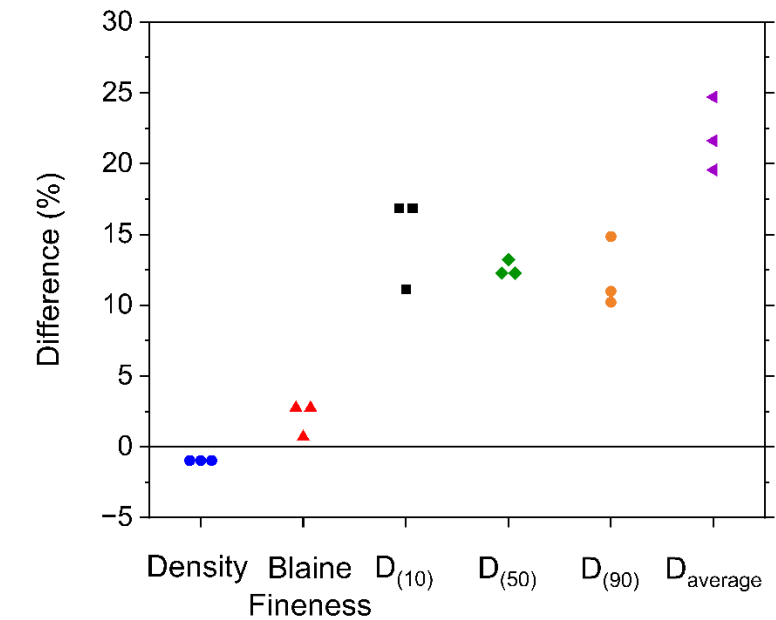
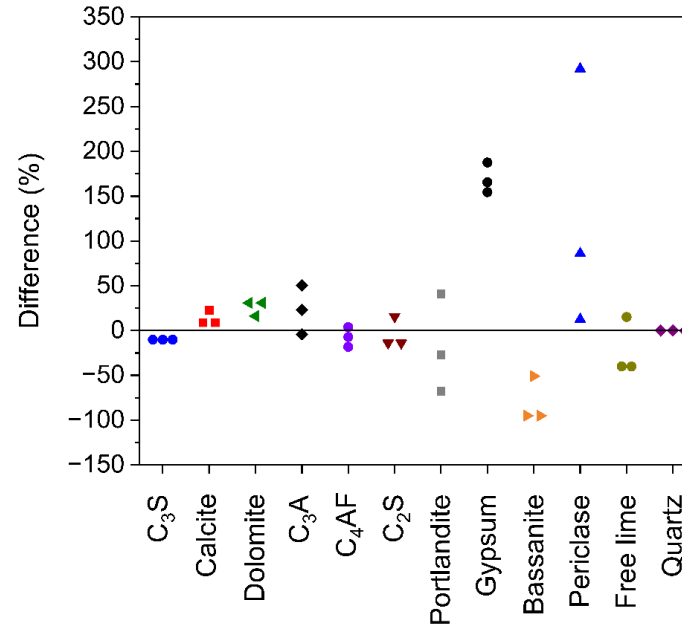


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# Batch-to-Batch Variability

- ▶ The PLCs exhibited minimal variation in early-age reactivity and early-age hydration (up to 7 d) of IL(10).
- ▶ Average particle size showed up to 25 percent variation.
- ▶ Major oxides ( $\text{Al}_2\text{O}_3$ ,  $\text{SiO}_2$ ,  $\text{CaO}$ ,  $\text{SO}_3$ , and  $\text{Fe}_2\text{O}_3$ ) showed up to 8 percent variation.
- ▶ Alite showed up to 10 percent variation.
- ▶ The final setting time exhibited greater variation than the initial setting time.

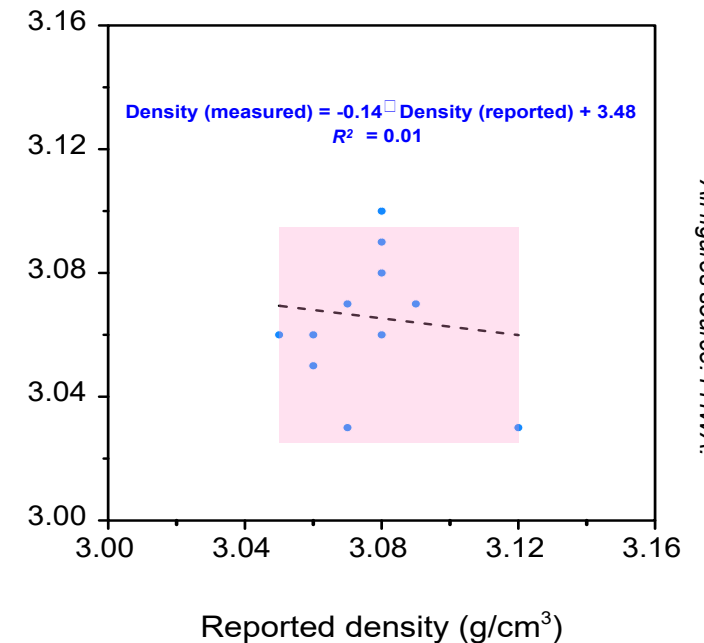
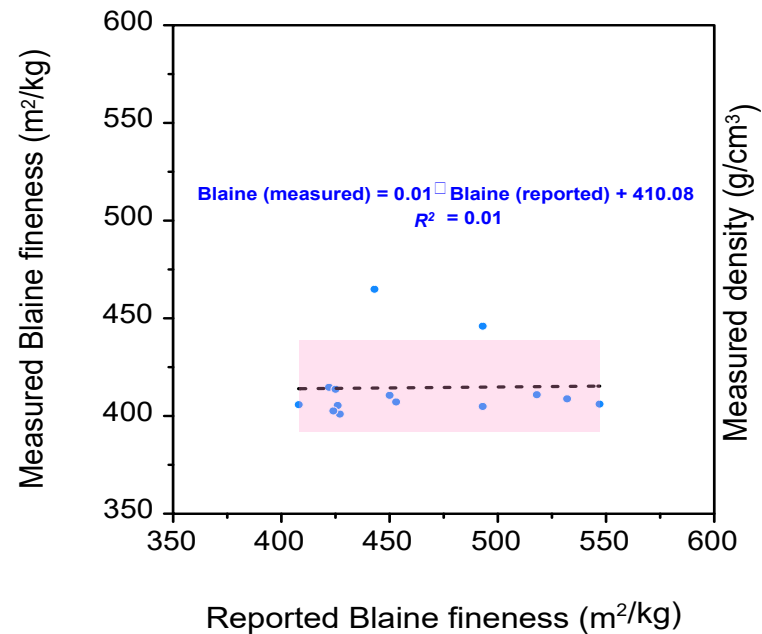
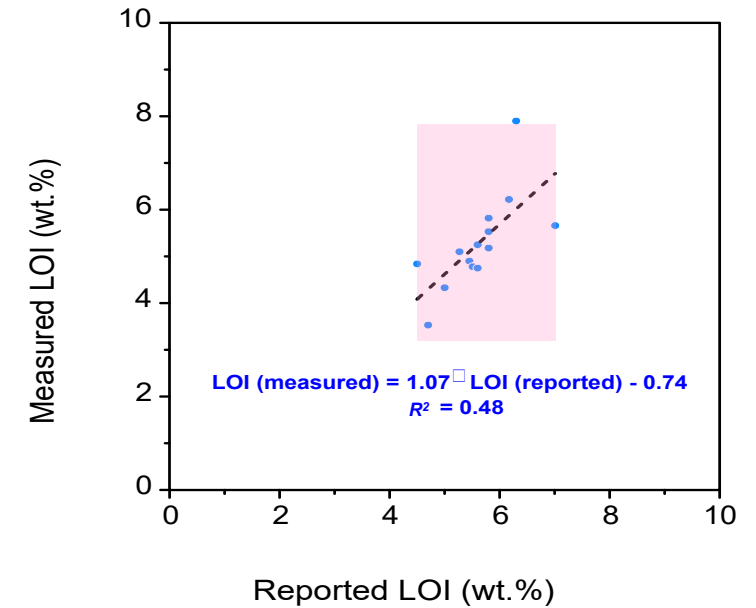
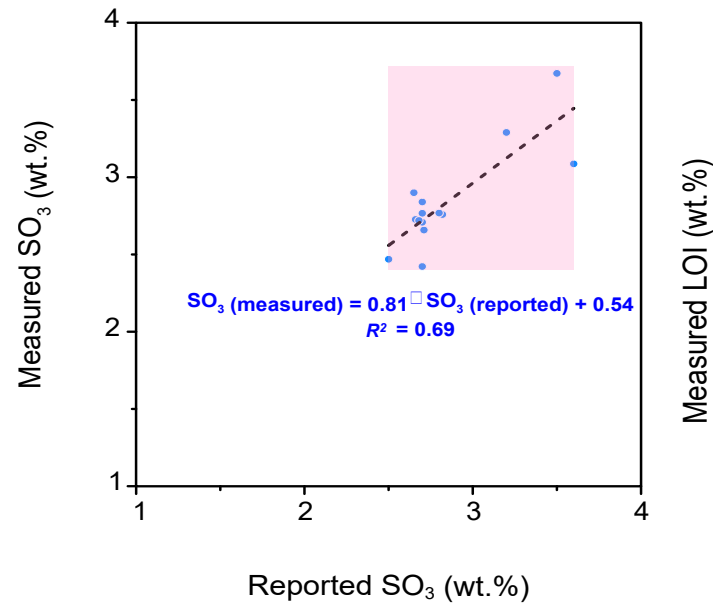


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# Variability in Mill Test Results

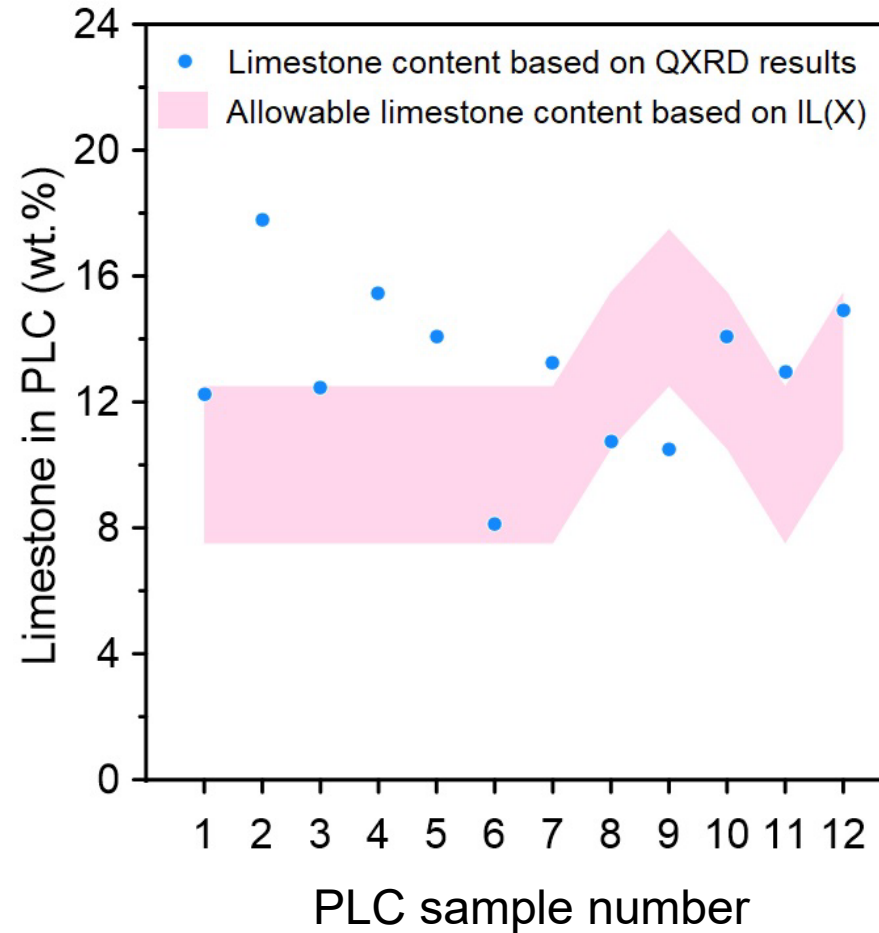
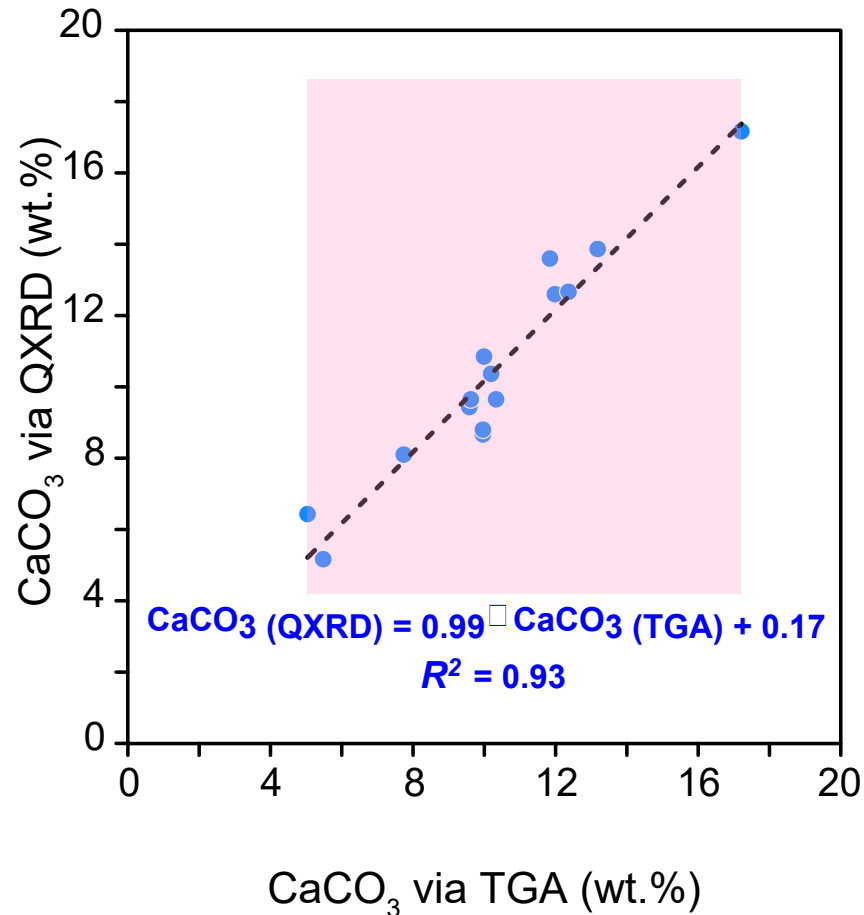
- ▶ There was a positive correlation between measured and reported chemical characteristics of PLCs.
- ▶ There were low correlations between measured and reported physical characteristics.
- ▶ Blaine fineness is a poor metric for PLC.



All figures source: FHWA.



# CaCO<sub>3</sub> and Estimated Limestone Content



Dolomitic limestone is likely used as feedstock for producing the studied PLCs based on negligible quartz content and dolomite presence.

Based on this assumption, the PLC limestone mass content ranges from 7 to 18 percent.

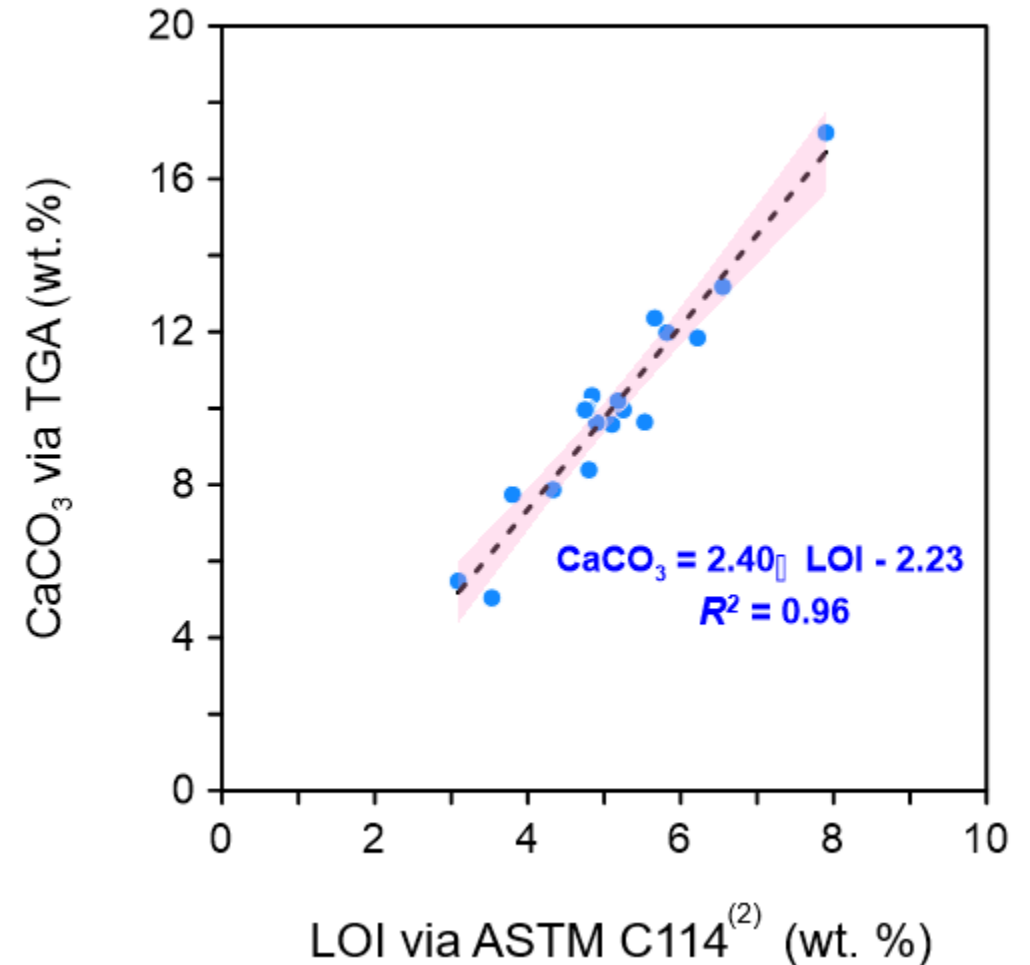
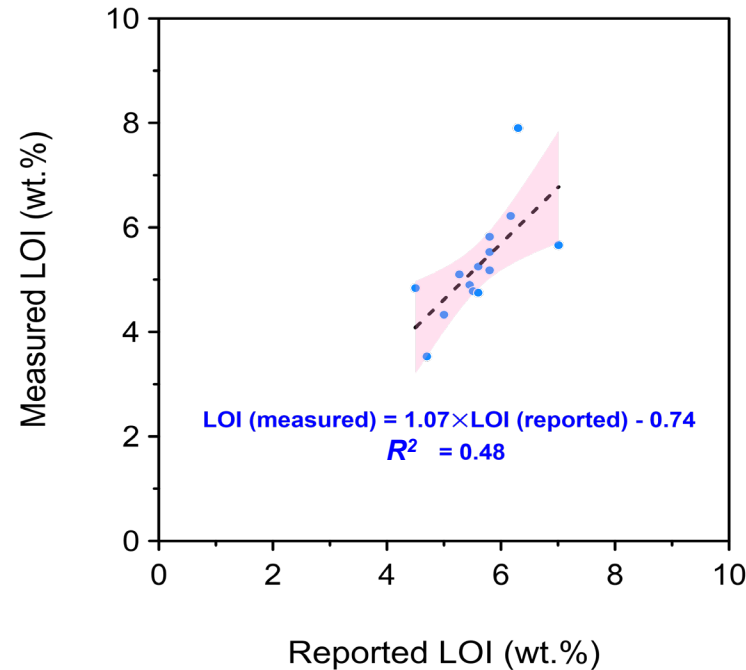
All images source: FHWA.



# Using LOI for $\text{CaCO}_3$ Estimations

Reported and measured LOI were generally similar.

LOI can be used to estimate  $\text{CaCO}_3$ .



All figures source: FHWA.





# Key Findings

- ▶ Reactivity of PLCs correlated well with chemical characteristics and correlated poorly with physical characteristics.
- ▶  $\text{CaCO}_3$  content in PLC can be estimated by measuring LOI using ASTM C114.<sup>(2)</sup>
- ▶ PLC characteristics were similar from batch-to-batch for three batches, with the fourth being more varied.
- ▶ Estimated limestone content and measured  $\text{CaCO}_3$  content varied significantly for PLCs across the country.





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# The Use of Electrical Durability Tests on Concretes with CS

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*September 9, 2025*

Source: FHWA.



# Purpose

- ▶ Investigation 1: Determine if inclusion of commercial CS products in concrete mixtures affects electrical durability test methods differently than nonelectrical test methods.
- ▶ Investigation 2: Evaluate durability of concrete mixtures using raw CS products with different surface areas and amounts.
- ▶ Investigation 3: Measure autogenous shrinkage of various technologies, including CS, LWA, SRA, and latex.







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# Investigation 1

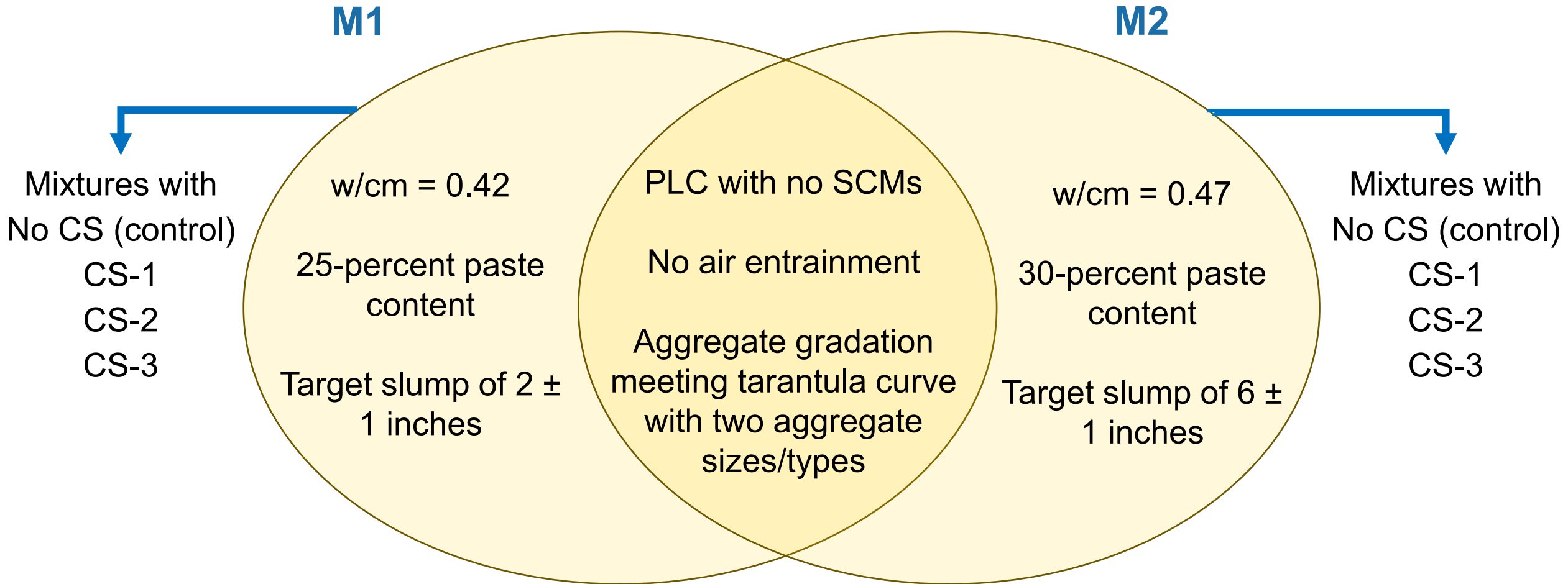
*Electrical Durability Testing of Concretes with Commercial CS Projects*



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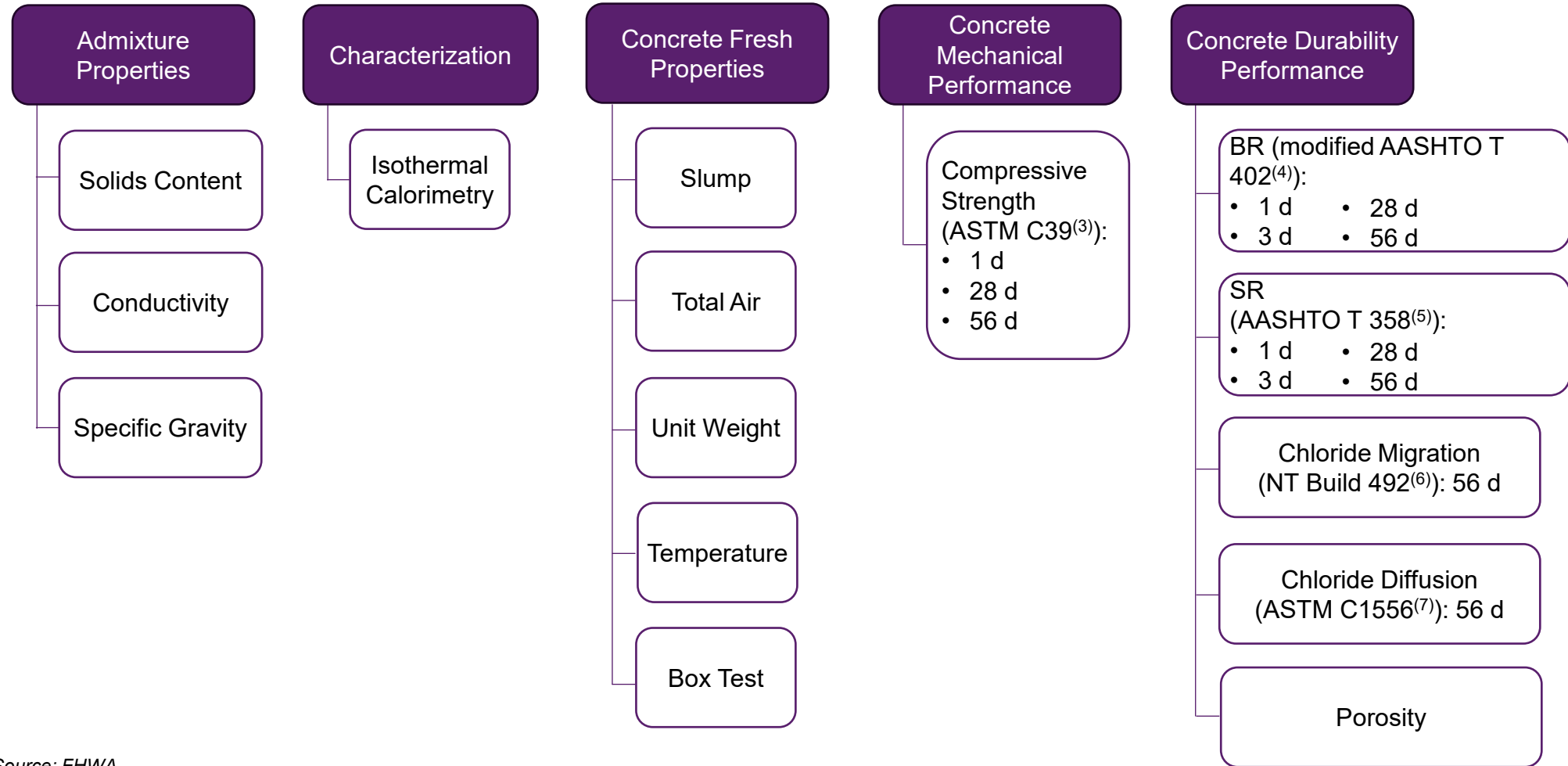
# Mixture Overview



Source: FHWA.



# Test Matrix



Source: FHWA.





# Electrical Versus Physical Tests

## Electrical Tests

Apparent SR<sup>(5)</sup>



BR<sup>(4)</sup>



## Semielectrical Tests



Chloride migration coefficient<sup>(6)</sup>

## Physical Tests

Chloride diffusion coefficient<sup>(7)</sup>



Porosity



All figures source: FHWA.



# Materials Dosage

The **highest** recommended dosages suggested by the manufacturers were used for all of the commercial products.

Materials	Manufacturer Recommended Dosage
CS-1	8– <b>20</b> fl oz per cwt
CS-2	4– <b>8</b> fl oz per cwt
CS-3	0.5– <b>1.5 percent</b> by weight of total cementitious materials content



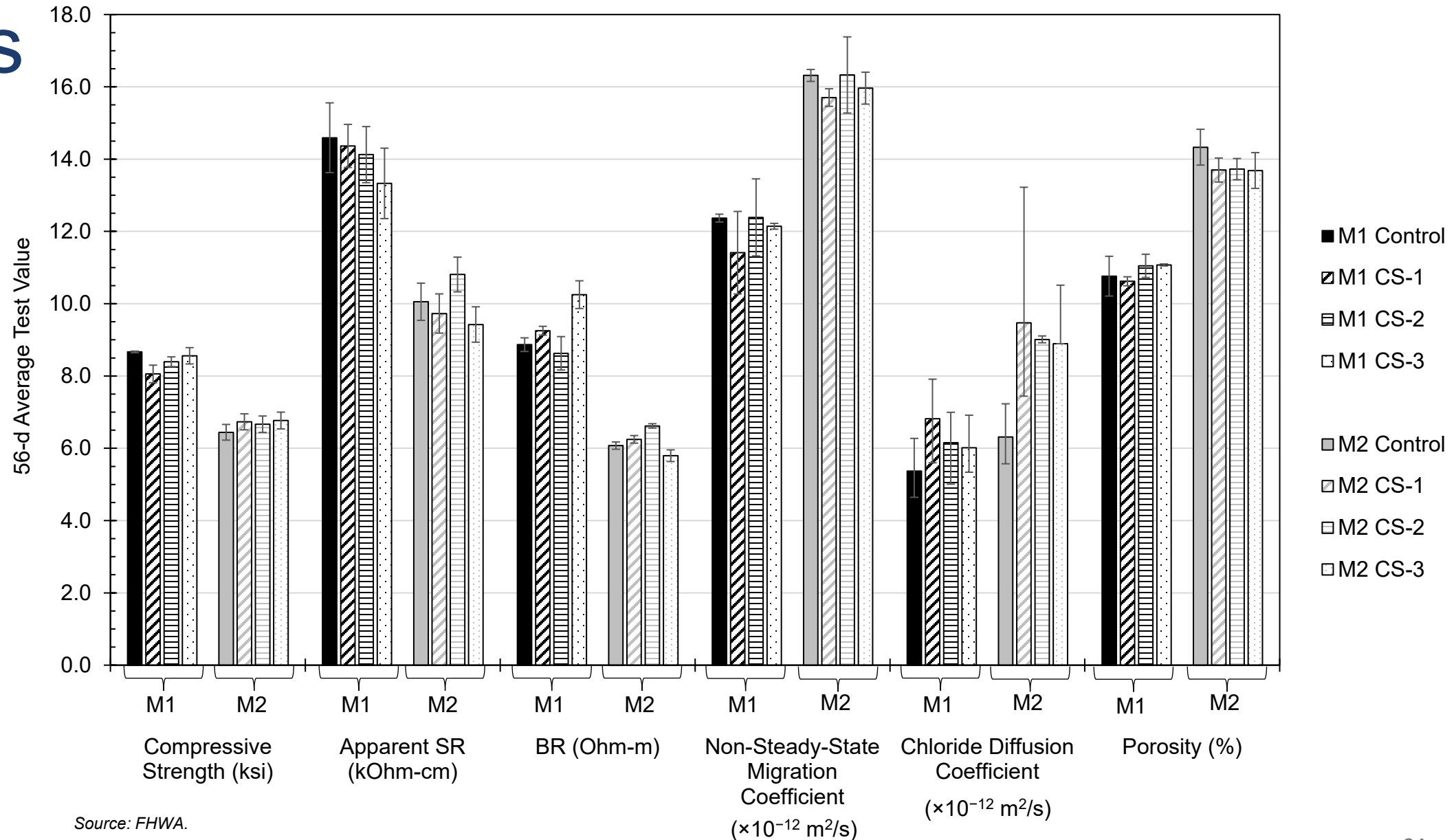
# Admixture Conductivity

The admixtures containing CS were not highly conductive.

Materials	Conductivity ( $\mu\text{S}/\text{cm}$ )
CS-1	3,862 at 22.2 °C
CS-2	2,580 at 22.0 °C
CS-3	7,164 at 21.5 °C
Limewater conditioning solution	12,100 at 23.0 °C
Alkali–concentrated conditioning solution from AASHTO T 402 <sup>(4)</sup>	92,700 at 23.0 °C



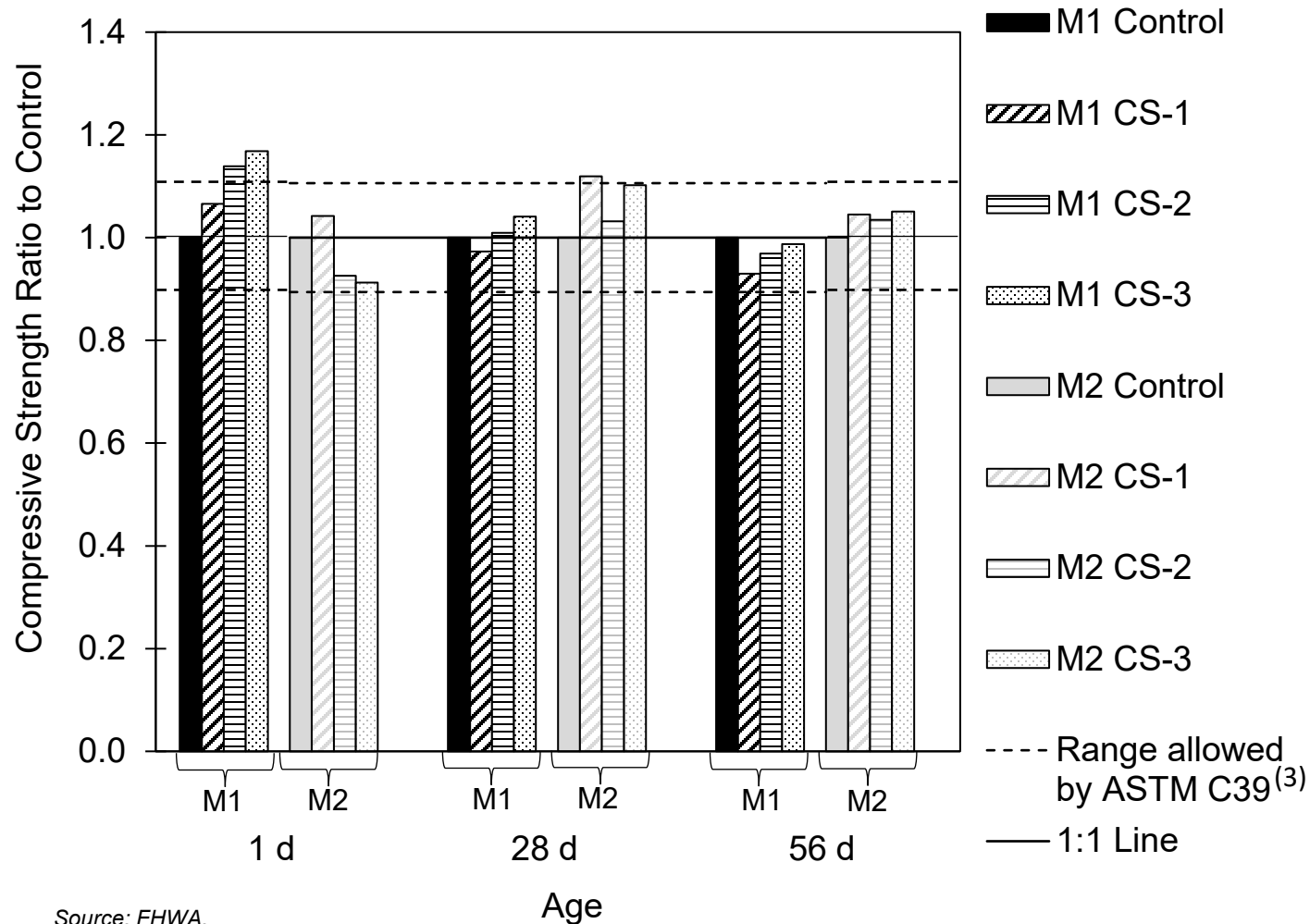
# Results



Source: FHWA.



# Compressive Strength Normalized

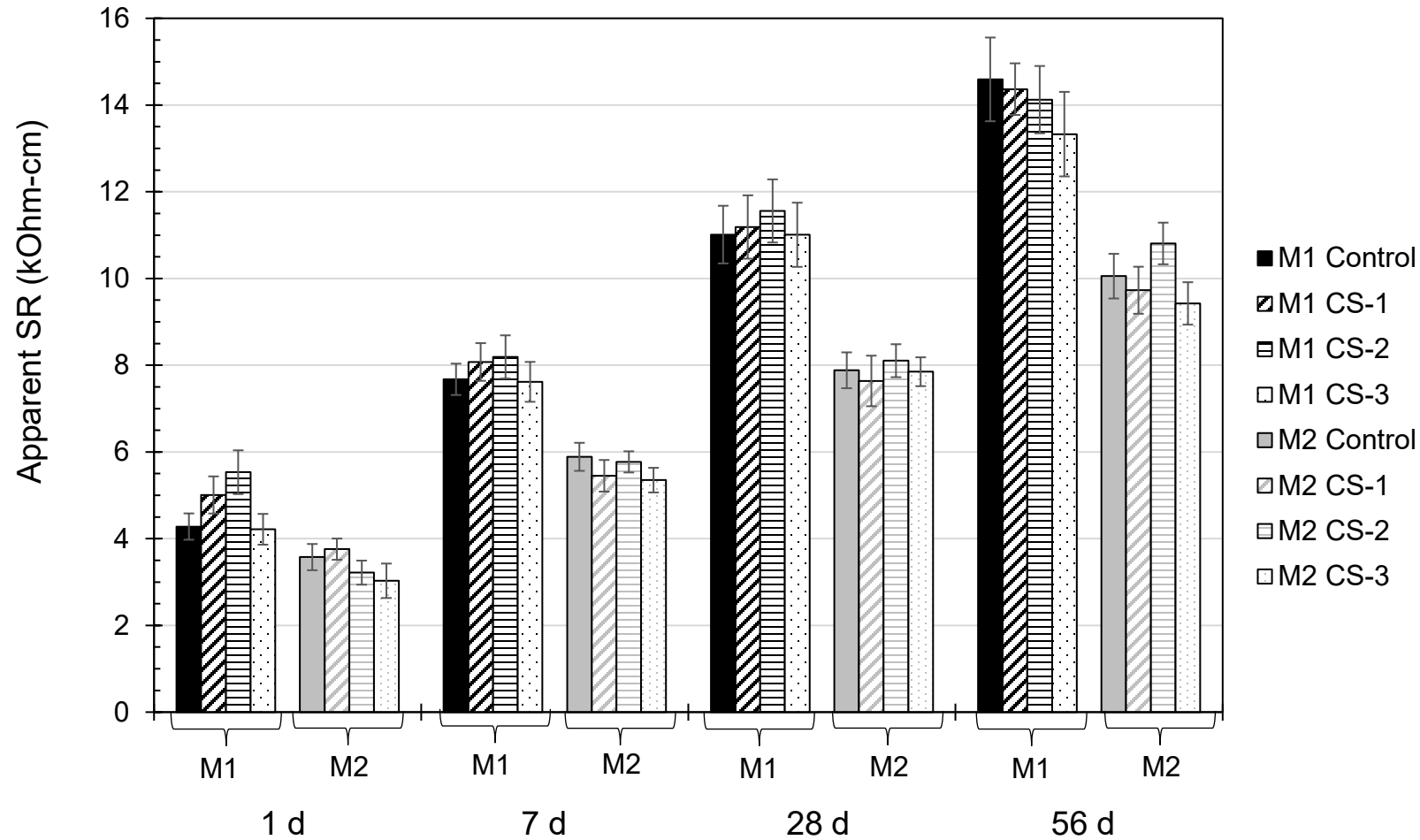


Source: FHWA.

- ▶ The compressive strengths of CS samples are within range of control samples.
- ▶ CS may increase compressive strength at 1 d for M1 with lower w/cm.
- ▶ CS has less influence on strength for M2 with higher w/cm.
- ▶ There is not a significant difference between control samples and samples with CS at 56 d.



# Apparent SR



The apparent SR is not significantly different for the control samples without CS compared to those with CS after very early ages.

The apparent SR is higher for the M1 samples than for the M2 samples.

Source: FHWA.

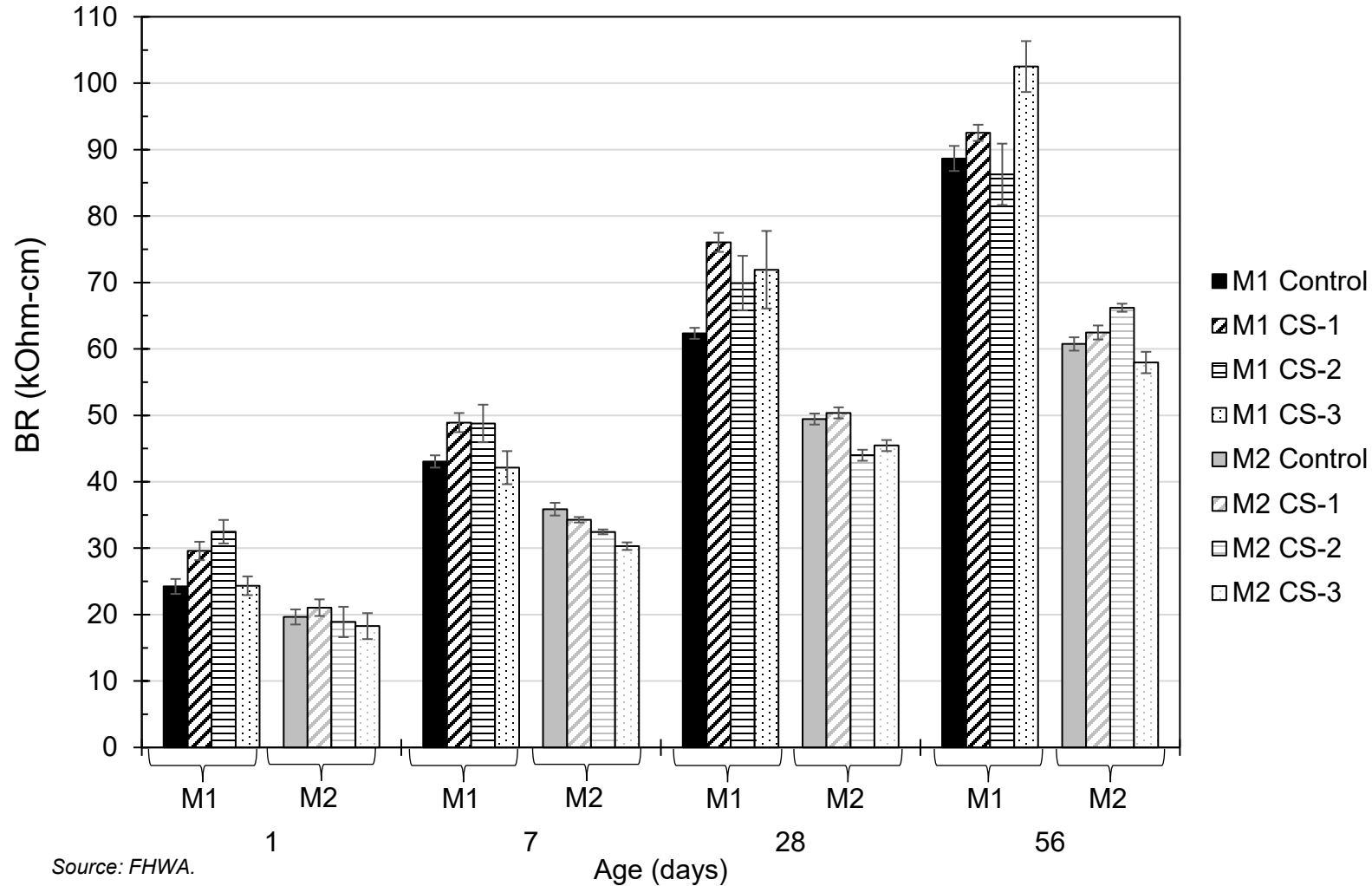


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# BR



Source: FHWA.

- ▶ The BR of concrete with lower w/cm is more affected by the inclusion of CS.
- ▶ The M1 concrete samples have higher resistivity than the M2 concrete samples with or without CS.





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# Investigation 2

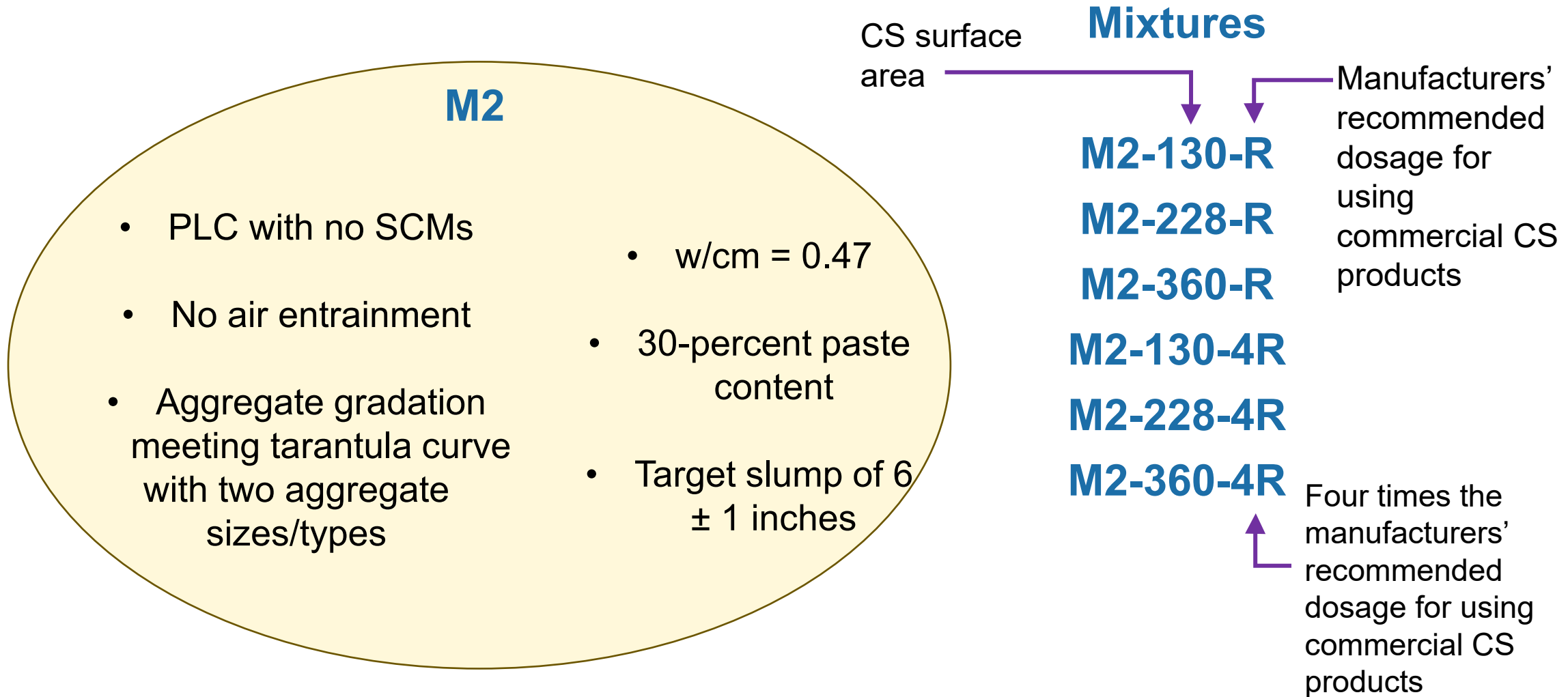
*Durability for Concretes with Raw CS Products*



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# Mixture Identification



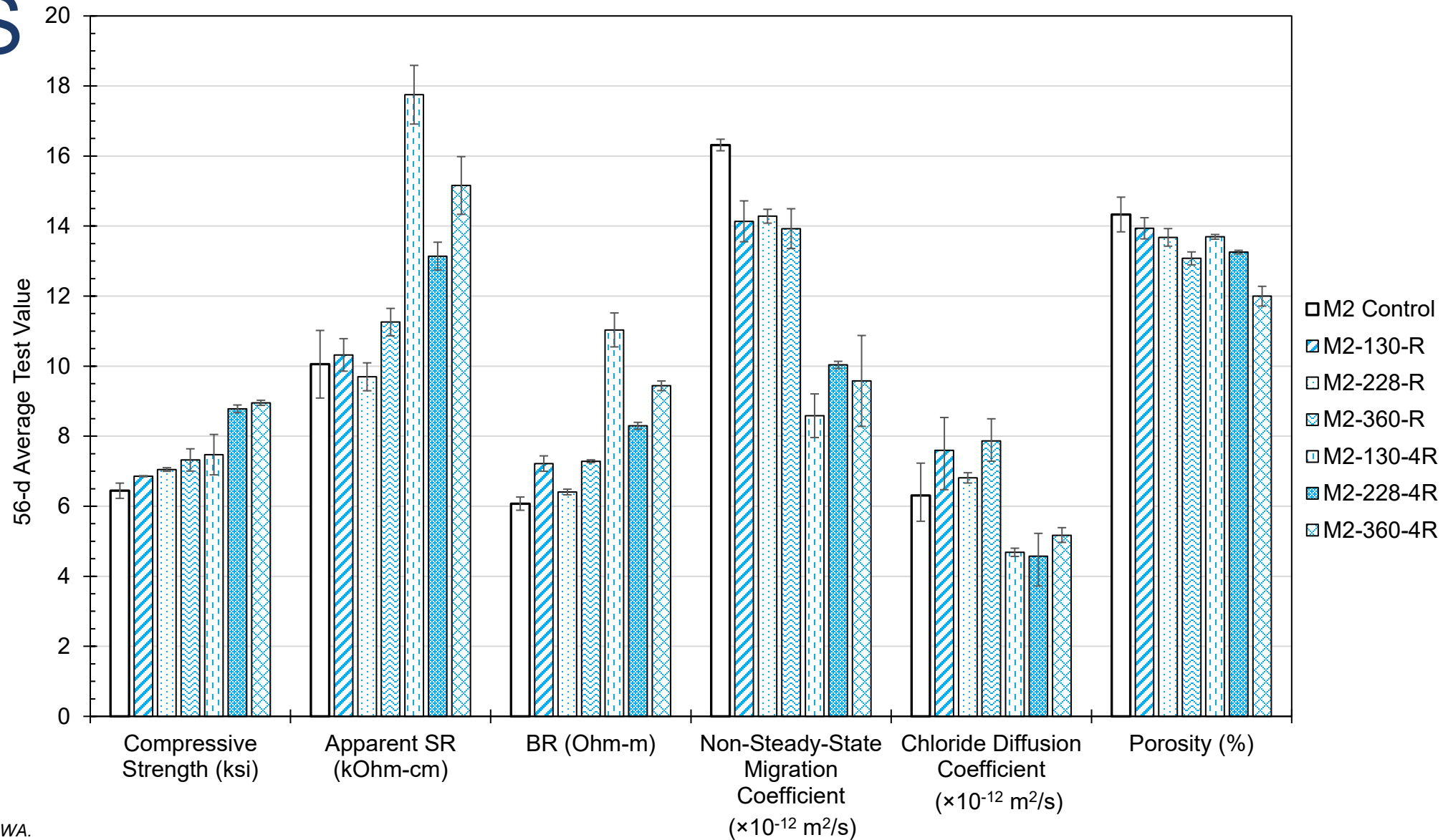
Source: FHWA.



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# Raw CS



Source: FHWA.







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# Investigation 3

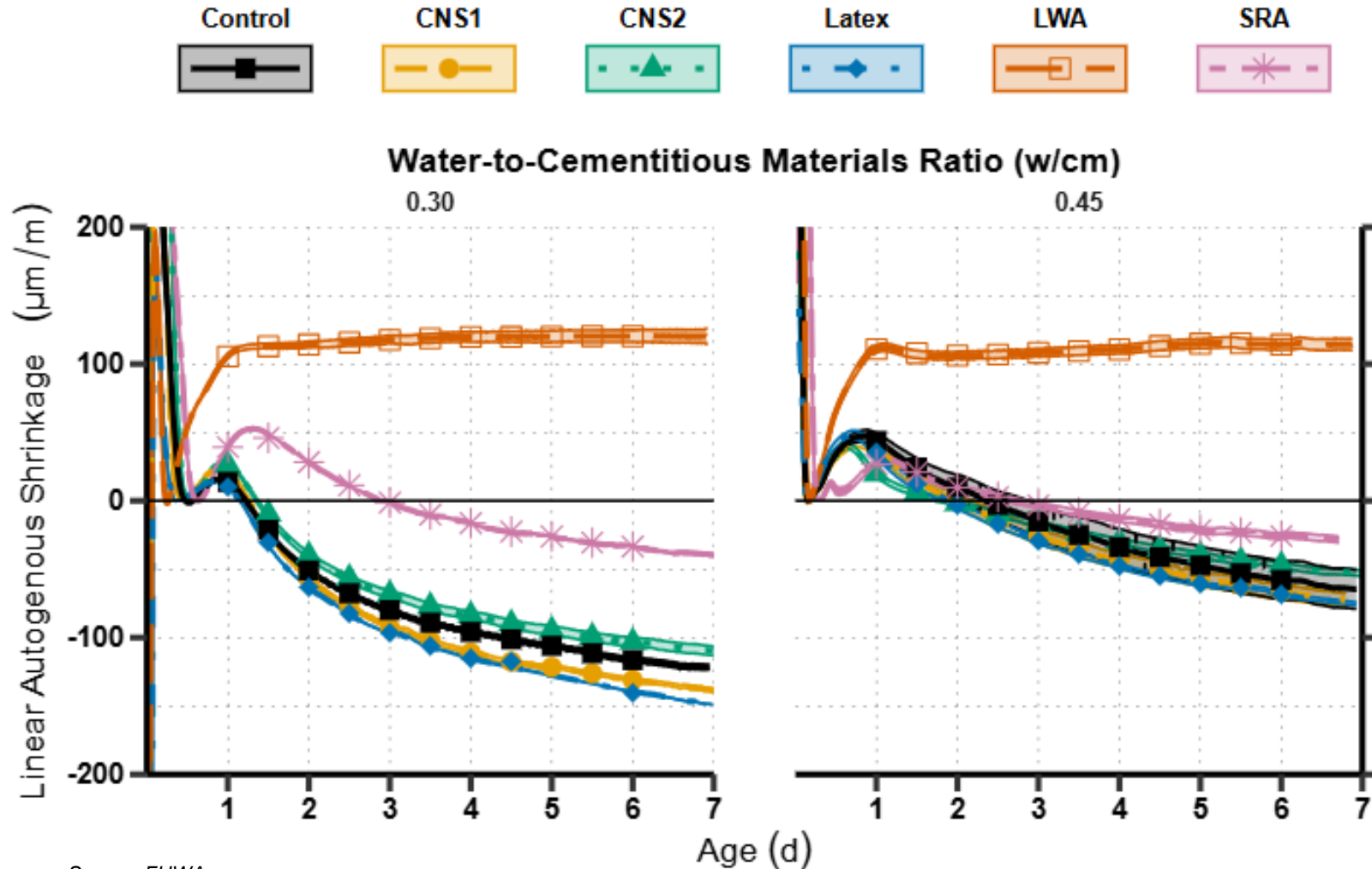
## *Autogenous Shrinkage*



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# Autogenous Shrinkage



Source: FHWA.

- ▶ Internal curing stores water to reduce potential for self-desiccation that leads to autogenous shrinkage.
- ▶ LWA shows no autogenous shrinkage.
- ▶ SRA shows expansion and then shrinkage.
- ▶ CNS, latex, and control show similar autogenous shrinkage.





# Key Takeaways

- ▶ Electrical tests can be used to indicate durability of concretes containing commercial CS products when used within the manufacturer's recommended dosages.
- ▶ If the owner is concerned about using electrical durability tests, bulk chloride diffusion can be performed to indicate transport of ions through the concrete.
- ▶ Commercial CS products do not reduce autogenous shrinkage.
- ▶ Suggest proper curing practices continue to be employed when placing concretes with CS.
- ▶ Suggest concrete durability continue to be improved through mixture design optimization.



# References

1. MapChart. 2025. “MapChart” (website). <https://mapchart.net>, last accessed March 20, 2025.
2. ASTM International. 2022. *Standard Test Methods for Chemical Analysis of Hydraulic Cement*. ASTM C114-18. West Conshohocken, PA: ASTM International.
3. ASTM International. 2021. *Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens*. ASTM C39/C39M-21. West Conshohocken, PA: ASTM International. [https://www.astm.org/c0039\\_c0039m-21.html](https://www.astm.org/c0039_c0039m-21.html), last accessed January 29, 2025.
4. AASHTO. 2023. *Standard Method of Test for Electrical Resistivity of a Concrete Cylinder Tested in a Uniaxial Resistance Test*. AASHTO T 402-23. Washington, DC: American Association of State Highway and Transportation Officials.
5. AASHTO. 2022. *Standard Method of Test for Surface Resistivity Indication of Concrete’s Ability to Resist Chloride Ion Penetration*. AASHTO T 358-22. Washington, DC: American Association of State Highway and Transportation Officials.
6. Nordtest. 1999. *Concrete, Mortar and Cement-based Repair Materials: Chloride Migration Coefficient from Non-Steady-State Migration Experiments*. Report No. NT Build 492. Serravalle Scrivia, Italy: Nordtest.
7. ASTM International. 2016. *Standard Test Method for Determining the Apparent Chloride Diffusion Coefficient of Cementitious Mixtures by Bulk Diffusion*. ASTM C1556-11a. West Conshohocken, PA: ASTM International. <https://www.astm.org/c1556-11a.html>, last accessed January 29, 2025.



# Questions?

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