Investigation of Autoclave Methods for Determining Alkali-Silica Reactivity of Concrete Aggregates

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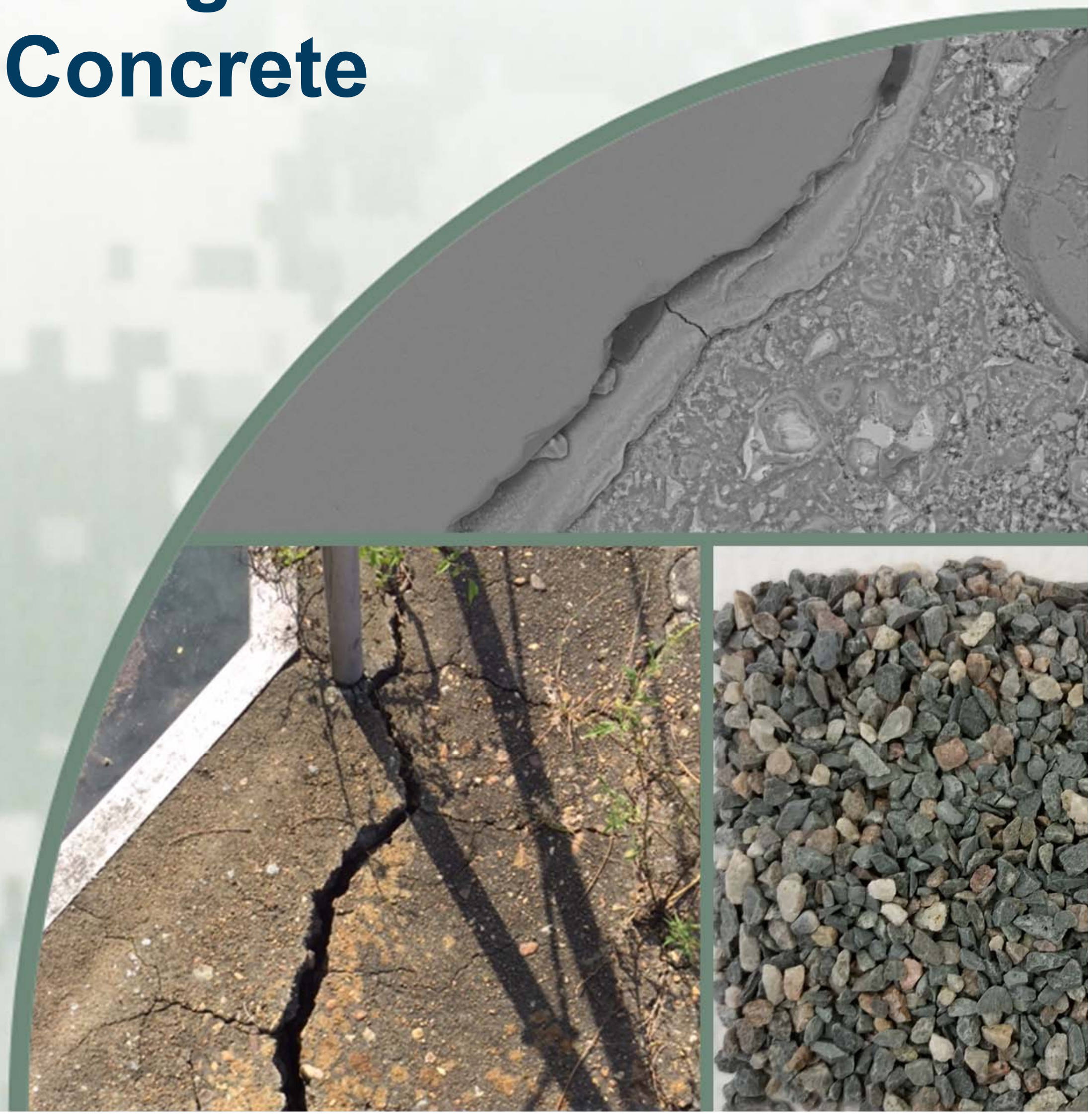

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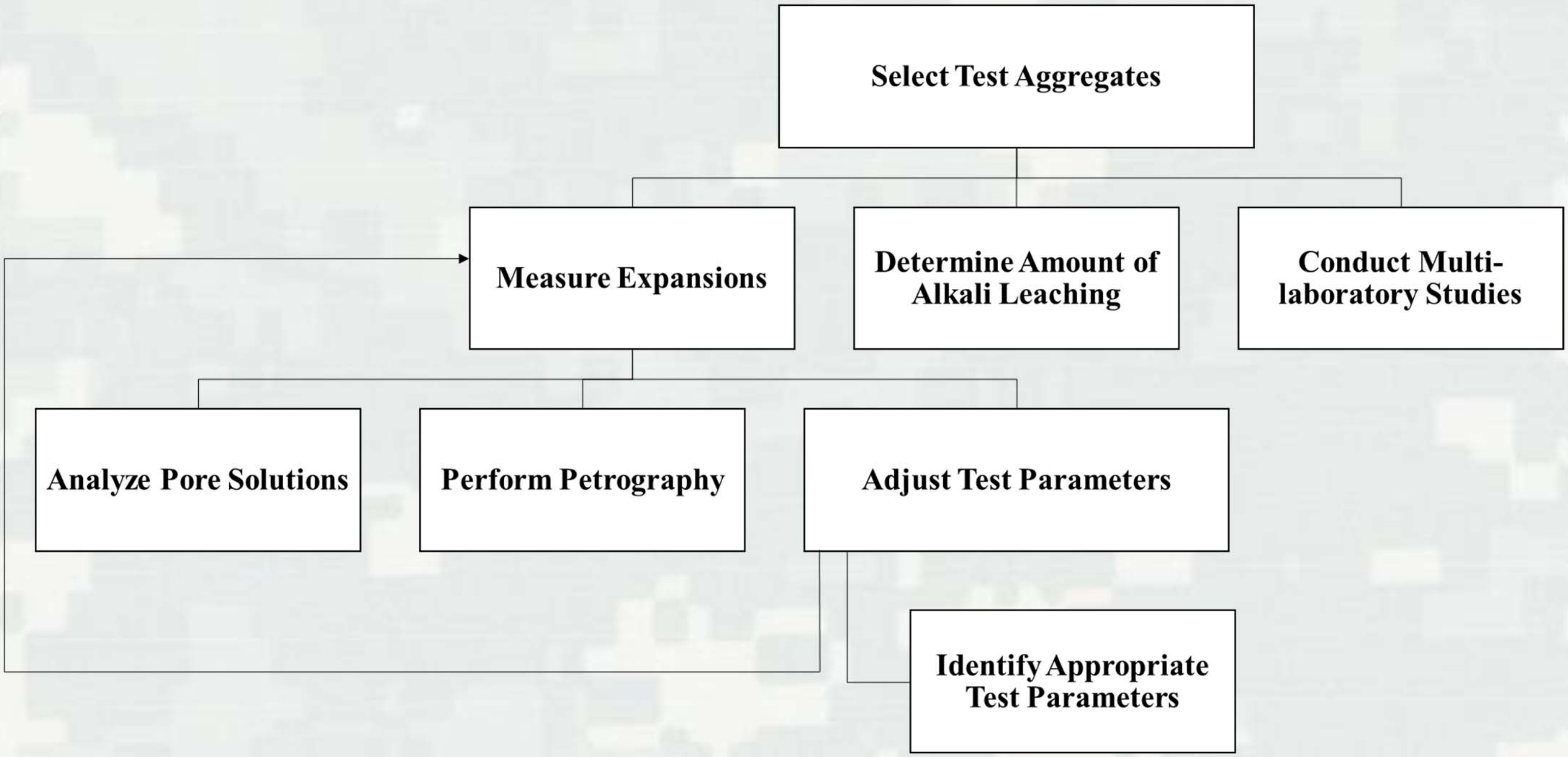
Autoclave Test Methods

Test Parameter	Chinese Autoclave Method (1983)	GBRC (1987)	Nishibayashi et al. (1987)	Laval/ CANMET (1991)	Nishibayashi et al. (1996)	ACPT (2013)
Duration (from mixing)	3 days	3 days	2 days	3 days	unknown	4 days
Duration of Conditioning	6 hours	2 hours	4 to 5 hours	5 hours	4 hours	24 hours
Specimen Type	Mortar	Mortar	Mortar	Mortar	Concrete	Concrete
Specimen Size, mm	10 x 10 x 40	40 x 40 x 160	40 x 40 x 160	25 x 25 x 285	75 x 75 x 400	75 x 75 x 285
w/cm	0.30	unknown	0.45	0.50	0.54	0.42
Na_2O_{eq}, by mass of cement	1.5%	2.5%	1.5%	3.5%	3.0%	3.0%
Temperature	150 °C	111 °C	128 °C	130 °C	133 °C	133 °C
Conditioning	In 10% KOH solution inside autoclave	In boiling water inside pressure vessel	Inside autoclave	Inside autoclave	Inside autoclave	Inside autoclave
Proposed Expansion Limit	-	-	-	0.15%	-	0.08%

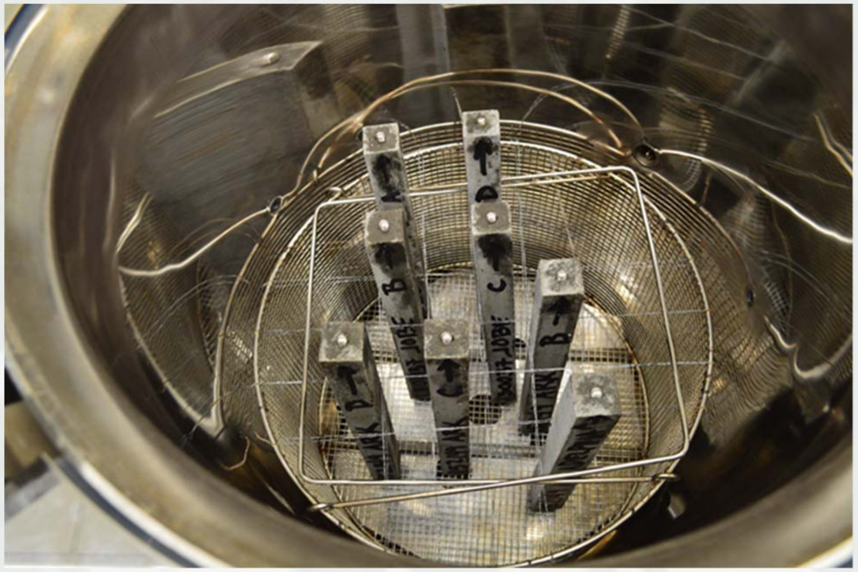
Research Questions

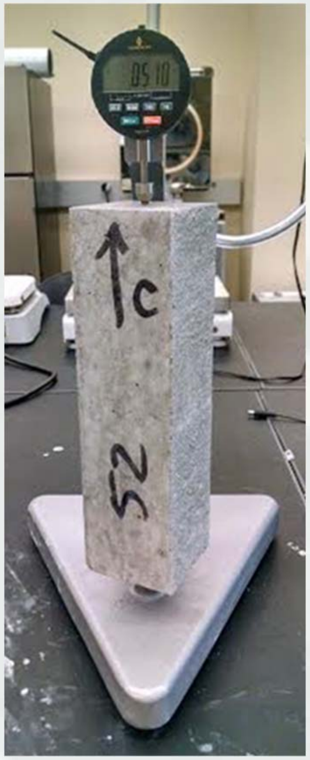
- Are autoclave test methods repeatable?
- How effectively do autoclave methods discriminate between non-reactive and reactive aggregates compared to other standardized test methods?
- Is ASR the cause of expansion in these specimens or is expansion due to some other mechanism?
- Is there a place for autoclave testing in the overall protocol for determining aggregate reactivity?

Overview of Research



Autoclaving





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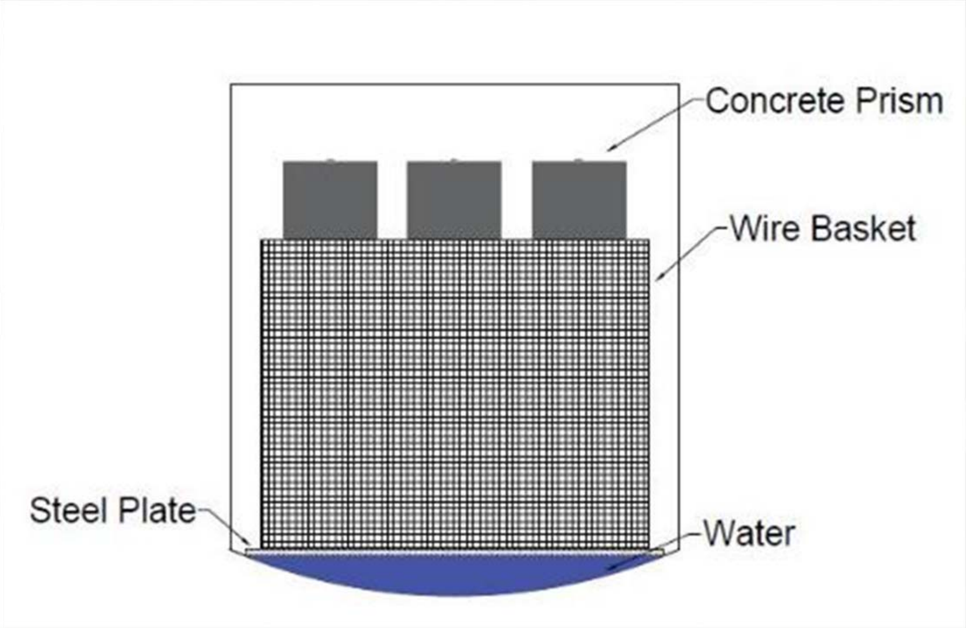
Alkali Leaching

Causes reduced or halted specimen expansion.

Influenced by:
- Temperature
- Specimen size
- Specimen porosity



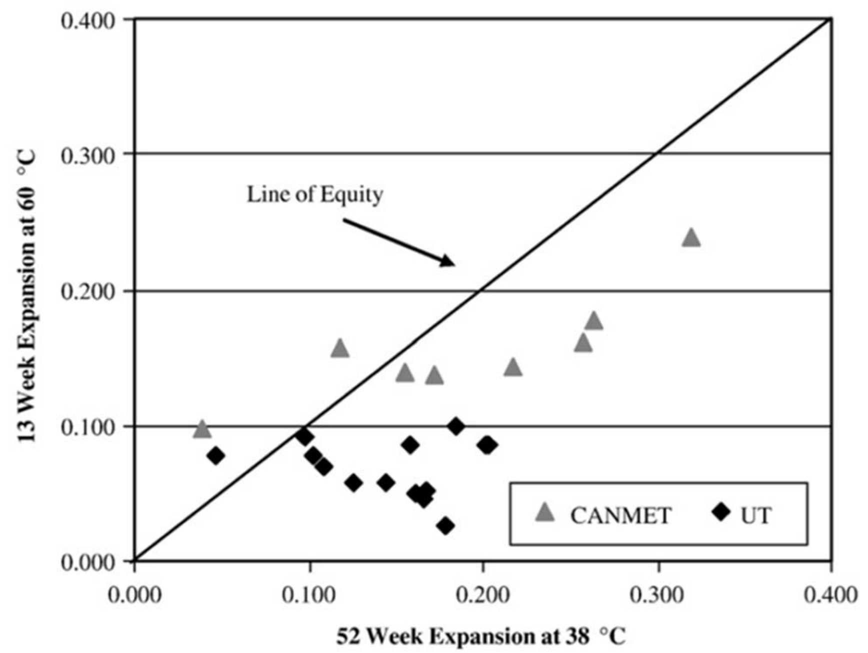


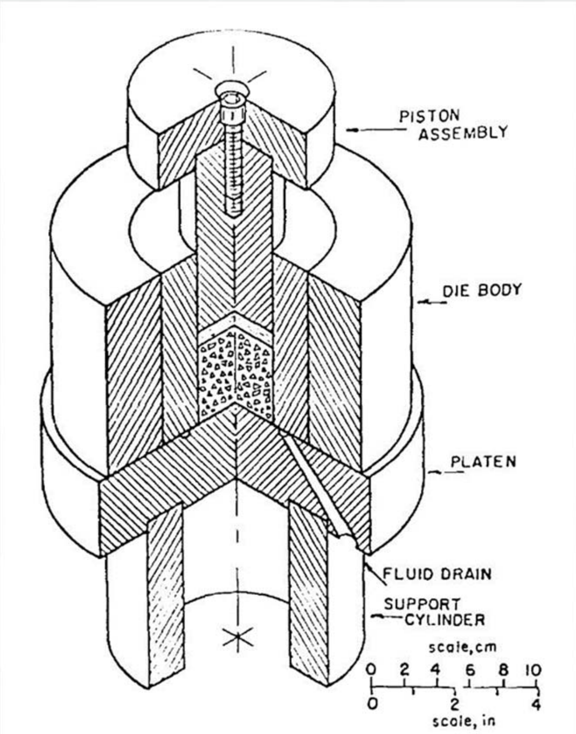
Fig. 1. Comparison of expansion at 52 weeks in the CPT at 38 °C versus expansion at 13 weeks in the CPT at 60 °C: CANMET and UT laboratory results.

Ideker et al. (2010)

Pore Solution

Expression

- ~ 700 MPa
- 200 g sample yields > 6 mL pore solution (> 35% evaporable water)



(Barneyback & Diamond, 1981)

Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)

- Na^{+}
- K^{+}
- Si^{4+}
- Ca^{2+}
- Al^{3+}
- Fe^{2+}

Titration to determine pH.

$[Na^{+}+K^{+}] = [OH^{-}]$ in unboosted concrete and in CPT prisms

Ion Chromatography (IC) to measure $[SO_4^{2-}]$

MULTI-LABORATORY STUDY OF FIVE-HOUR AUTOCLAVE TEST (LAVAL/CANMET METHOD)

Wood, S.G.; Giannini, E.R.; Bentivegna, A.F.; Rashidian, H.D.; Rangaraju P.R.; Drimalas, T.; Ramsey, M.A.; Johnson, T.R.; Moser, R.D. “Five-Hour Autoclave Test for Determining Potential Alkali-Silica Reactivity of Concrete Aggregates: A Multi-Laboratory Study,” *Advances in Civil Engineering Materials*, 2017.

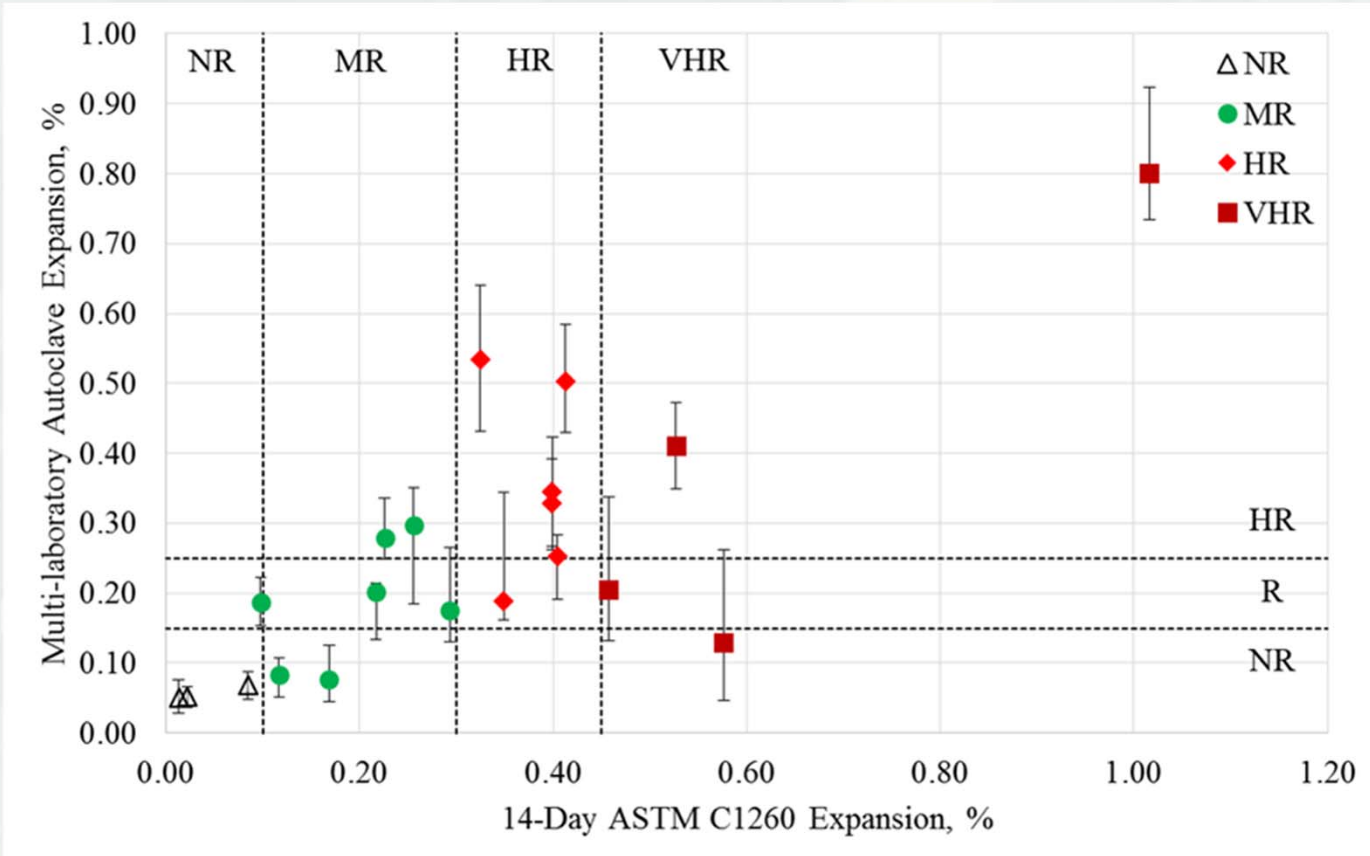
Objectives

- Determine if the autoclave method is suitable for identification of alkali-silica reactive aggregates used in rapid construction of short-life structures.
- Determine the amount of alkali leaching involved in the test.
- Evaluate variability and repeatability of the test method.

Overview of Study

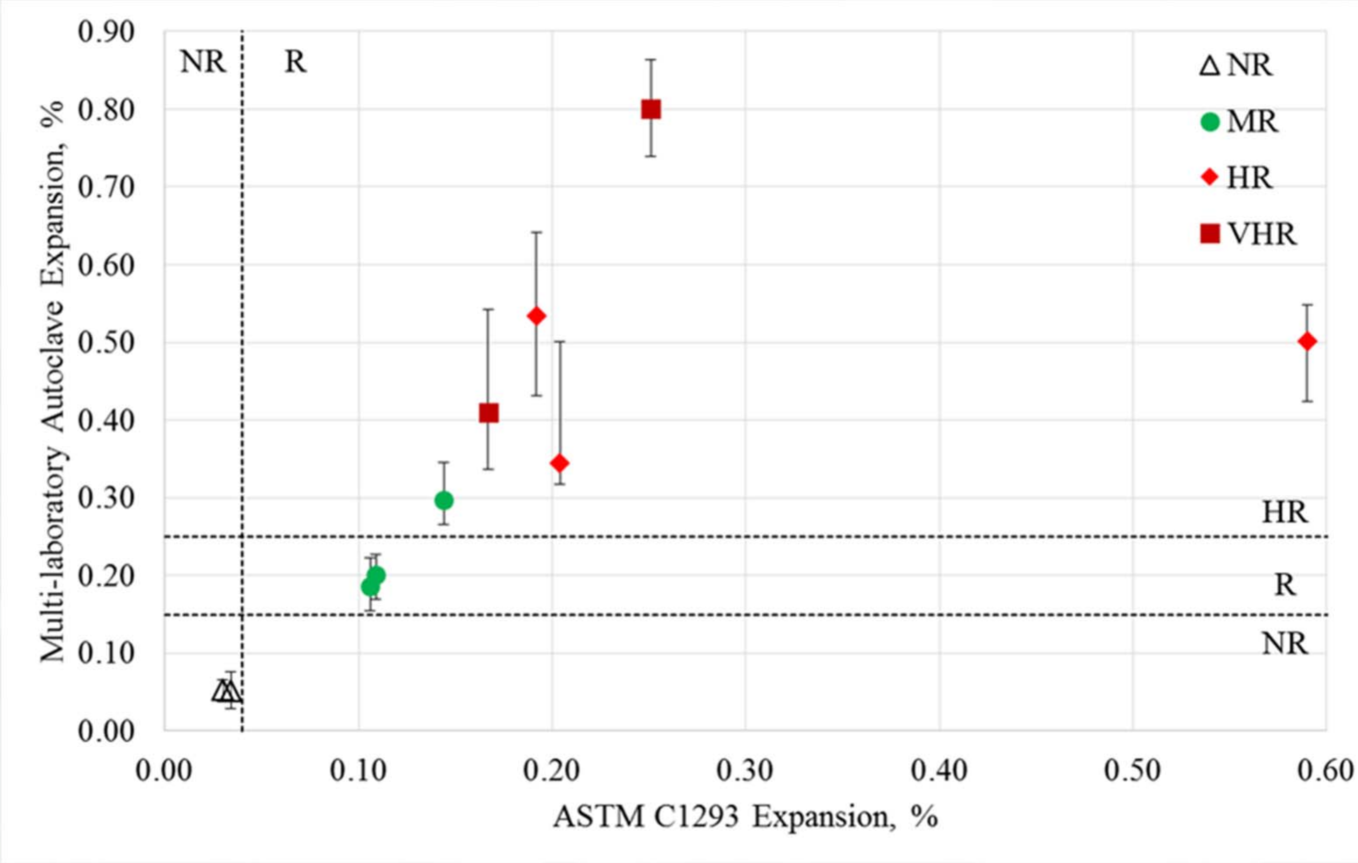
- 20 aggregates
- 1 Type I/II cement – 0.52% Na_2O_{eq}
- ASTM C1260 mortar bars but with NaOH to boost Na_2O_{eq} to 3.5%
- Autoclaved for 5 hours at 130 °C (0.17 MPa gauge pressure)
- 5 laboratories
- Reactivity Classifications:
  - NR: Non-reactive
  - MR: Moderately Reactive
  - HR: Highly Reactive
  - VHR: Very Highly Reactive

Autoclave vs. ASTM C1260



85% agreement for 20 aggregates

Autoclave vs. ASTM C1293



100% agreement for 10 aggregates

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Variability

Test Method	Within-laboratory CV	Multi-laboratory CV
Autoclave	5.9%	20.0%
ASTM C1260 (expansion > 0.10% at 14 days)	2.94%	15.4%
ASTM C1293 (expansion > 0.014%)	12%	23%

Alkali Leaching

Na_2O_{eq} = (Na x 1.35) + (1.20 x K x 0.658)

Aggregate	Number of Mortar Bars	Autoclave Water Concentration, mg/L			Leached Alkalis, %
		[Na^+]	[K^+]	[Na_2O_{eq}]	
MR1	4	125	32	193	8.7
MR3	3	94	20	142	8.6
MR4, MR7	7	151	33	230	5.9
HR3	3	83	21	129	7.8
NR1, HR2, HR5	10	291	65	444	8.4

Autoclave Alkali Leaching: 6 - 9% per mortar bar

Conclusions

- Autoclave agreement with ASTM C1260 = 85% (20 aggregates)
- Autoclave agreement with ASTM C1293 = 100% (10 aggregates)
- Autoclave within-laboratory CV = 5.9%
- Autoclave multi-laboratory CV = 20.0%
- Alkali leaching 6 to 9%
  - ASTM C1293: 12 to 25%

EVALUATION OF THE AUTOCLAVED CONCRETE PRISM TEST (ACPT)

Objectives

- Determine influence of cement alkalinity on expansion
- Evaluate the ACPT in its effectiveness to characterize aggregate reactivity
- Trace migration of alkalis
- Determine mechanism of prism expansion

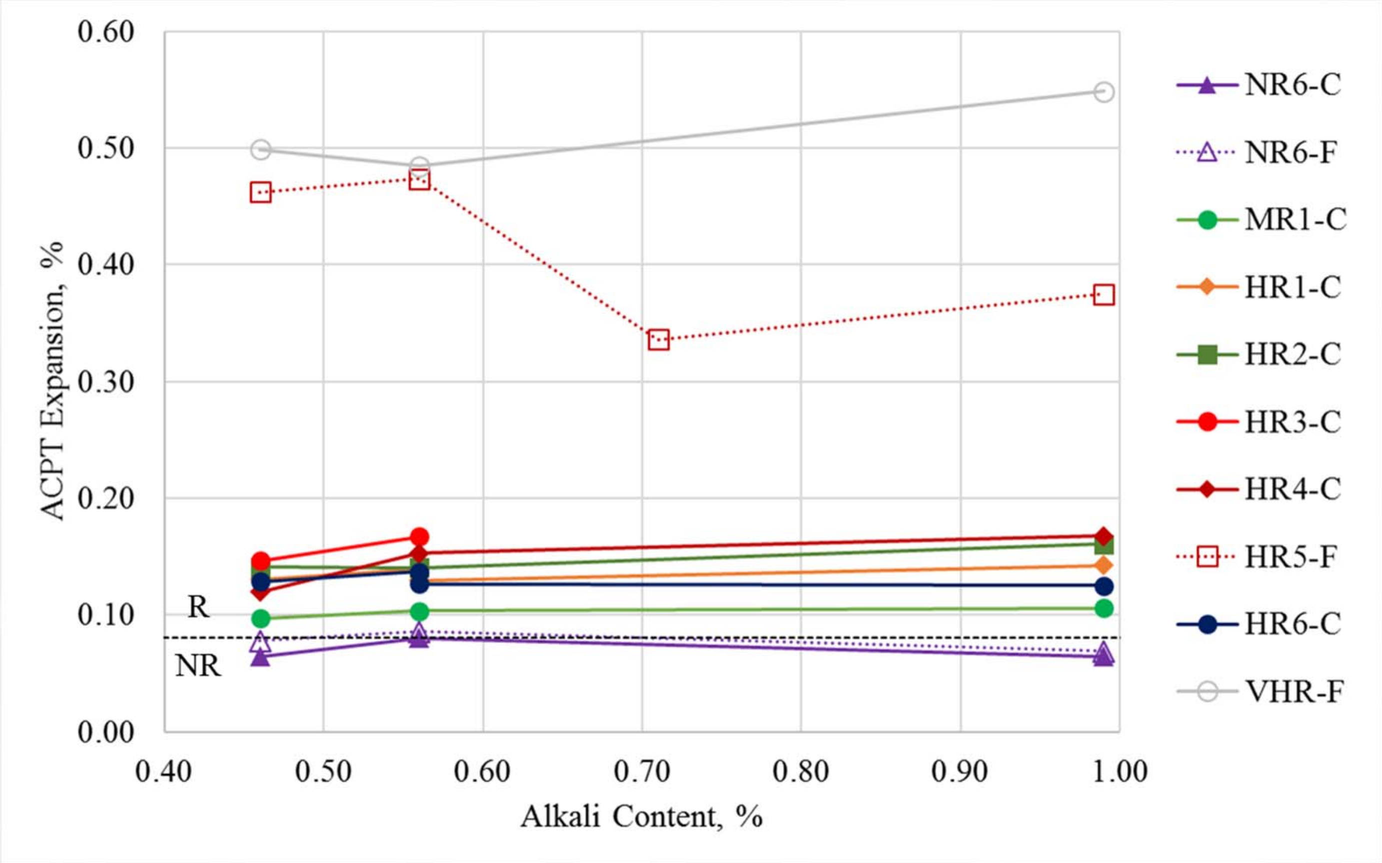
Overview of Study

- 22 aggregates
- 4 Type I/II cements (Na_2O_{eq} from 0.46 to 0.99%)
- 55 total tests using sets of 3 prisms
- Concrete prisms like ASTM C1293 but with NaOH to boost Na_2O_{eq} to 3.0%
- ICP-OES to determine alkali concentrations in autoclave water and pore solution
- IC to determine sulfate concentrations in pore solution
- Petrography to identify reaction product

Mixture Designations:
NR1-C-A

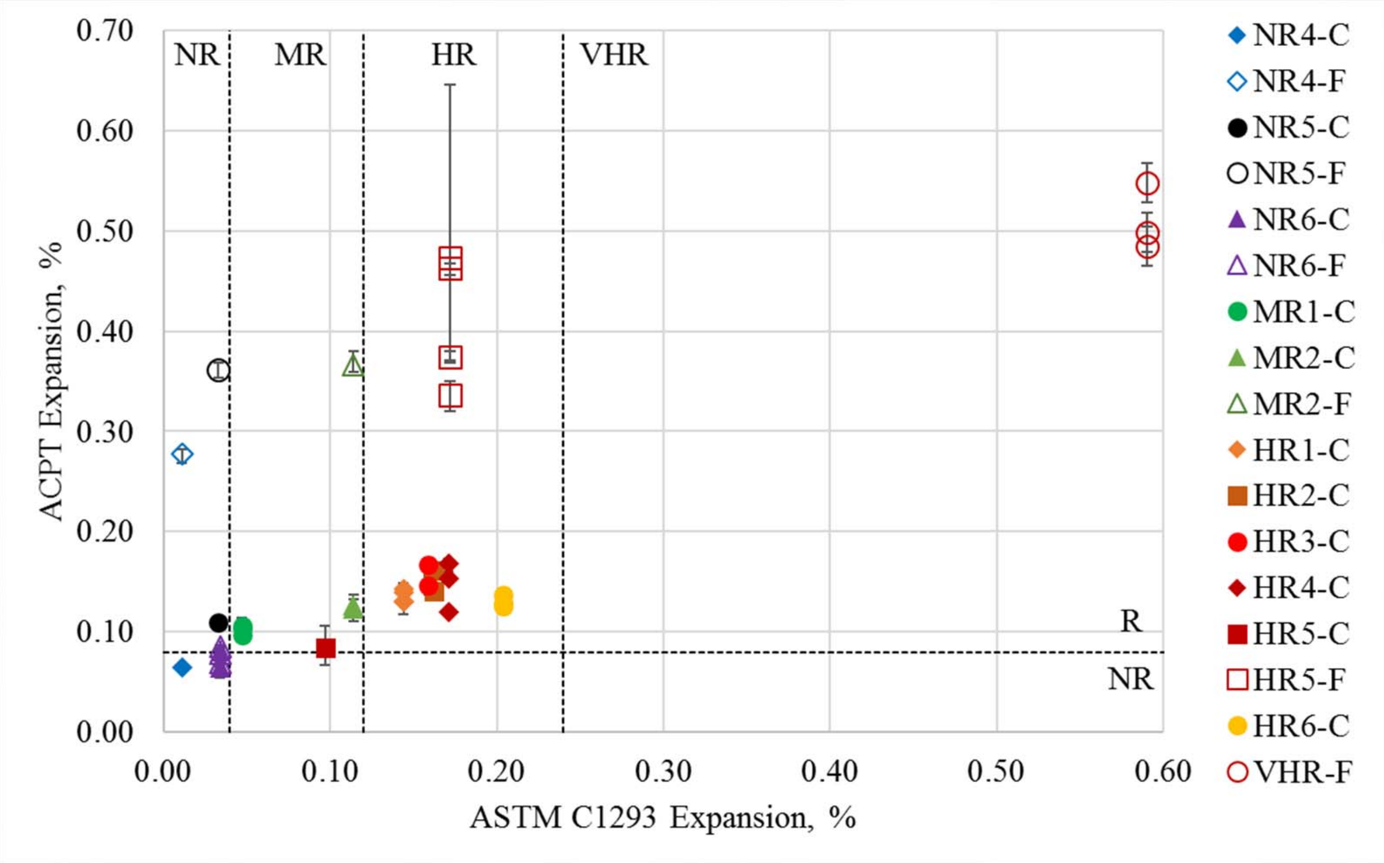
Aggregate Abbreviation
Coarse/Fine
Cement

Influence of Cement Alkalinity



ACPT vs. ASTM C1293

Overall Agreement: 85%



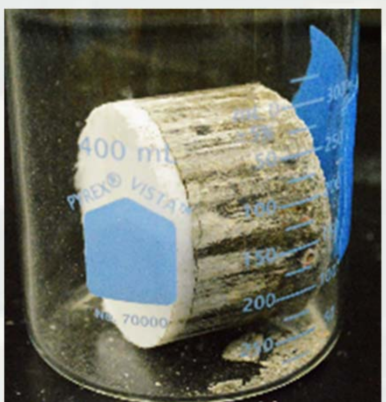
Coarse Aggregates: 93% Fine Aggregates: 69%

Alkali Leaching

Mixture	Autoclave Water Concentration, mg/L			Leached Alkalis, %
	K^+	Na^+	Na_2O_{eq}	
NR1-C-A	75.3	308.5	476.0	5.3
NR3-F-A	76.5	305.9	473.4	5.3
NR2-F-D	206.8	403.3	707.7	7.9
NR6-C-B	72.3	280.5	435.7	4.9
HR1-C-B	68.2	262.7	408.4	4.6
HR3-C-B	65.0	299.7	455.9	5.1
HR6-C-B	73.7	259.7	408.8	4.6
MR3-C-A	146.0	636.4	974.4	10.9
MR3-C-B	136.7	518.1	807.3	9.0
MR3-C-D	165.7	328.9	574.9	6.4
VHR-F-D	107.1	238.1	405.9	4.5

Alkali Leaching in ACPT = 4 to 11%

Pore Solution Analysis



Mixture	$[Na^{+}+K^{+}]$	$[OH^{-}]$	$[SO_4^{2-}]$	$[OH^{-}+2SO_4^{2-}]$	Average pH
NR1-C-A	1345	468	-	-	13.67
NR3-F-A	1191	423	-	-	13.63
NR2-F-D	1208	423	-	-	13.63
NR6-C-B	1014	427	-	-	13.63
HR1-C-B	1056	420[a]	289	998	13.62[a]
HR3-C-B	851	360[a]	258	876	13.56[a]
HR6-C-B	1404	460[a]	687	1834	13.66[a]
MR3-C-A	1162	405[b]			13.61[b]
MR3-C-B	1338	430			13.63
MR3-C-D	1392	470	-	-	13.67
VHR-F-D	802	387	-	-	13.59

[a] Titrated once.
[b] Titrated twice.

$[Na^{+}+K^{+}] \neq [OH^{-}]$

ICP-OES Titration IC Titration

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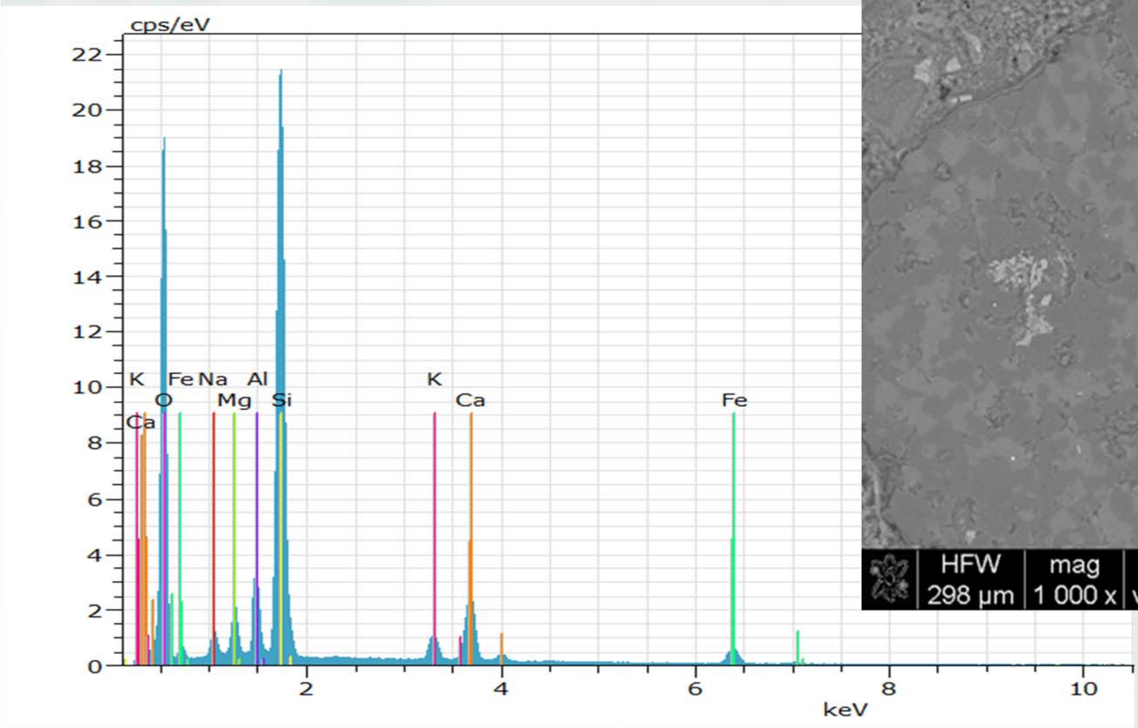
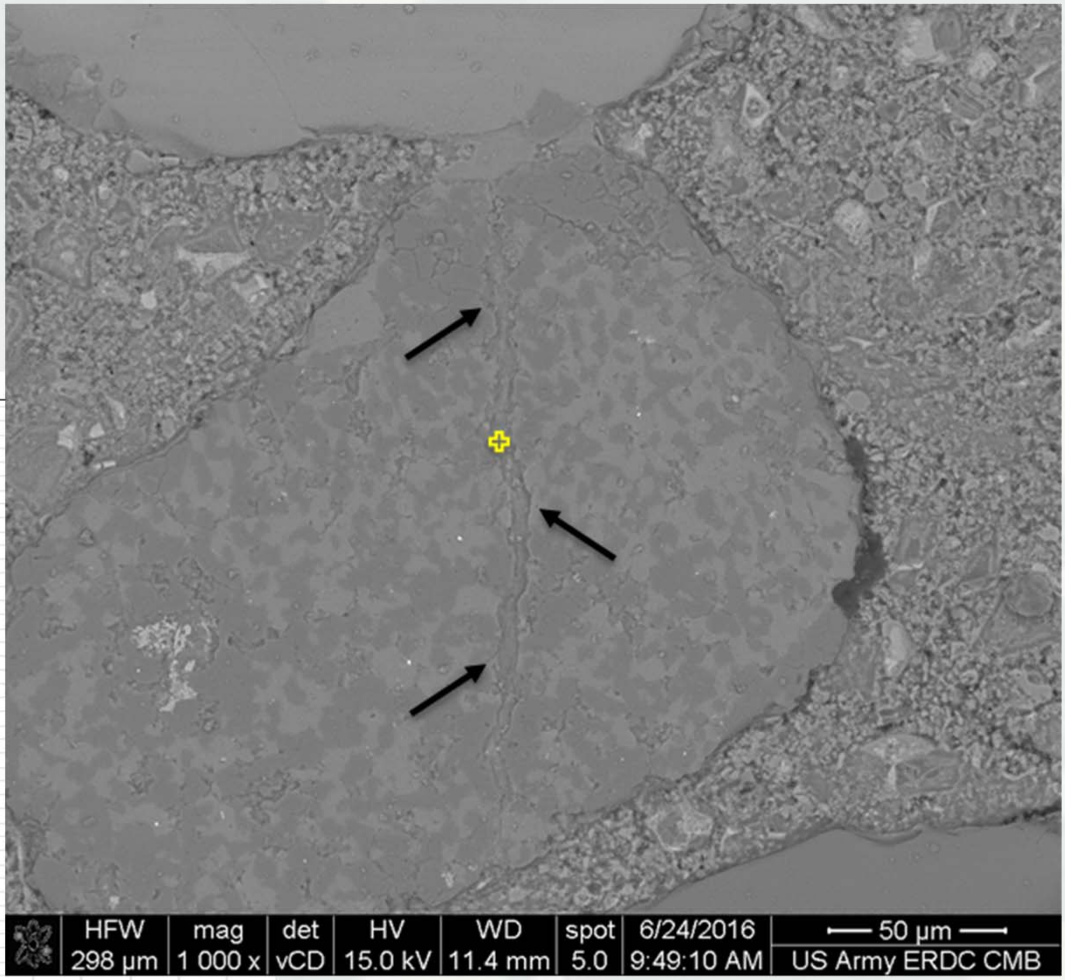
ASR Reaction Product

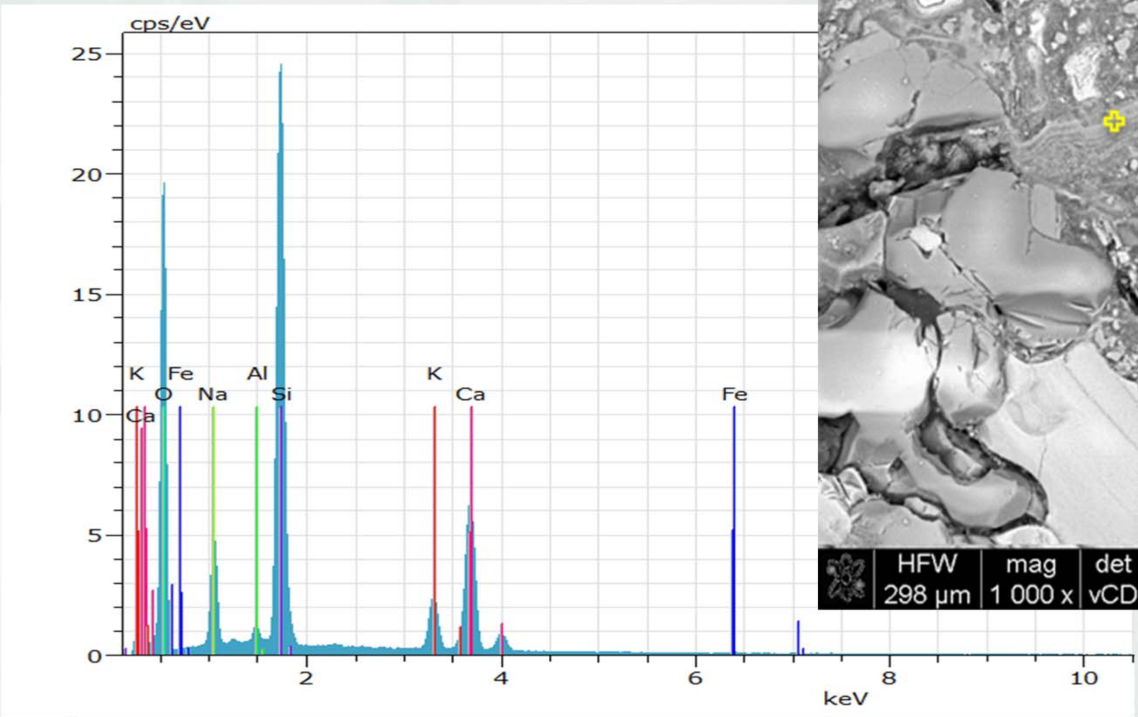
Jobe Aggregate
(VHR-F)

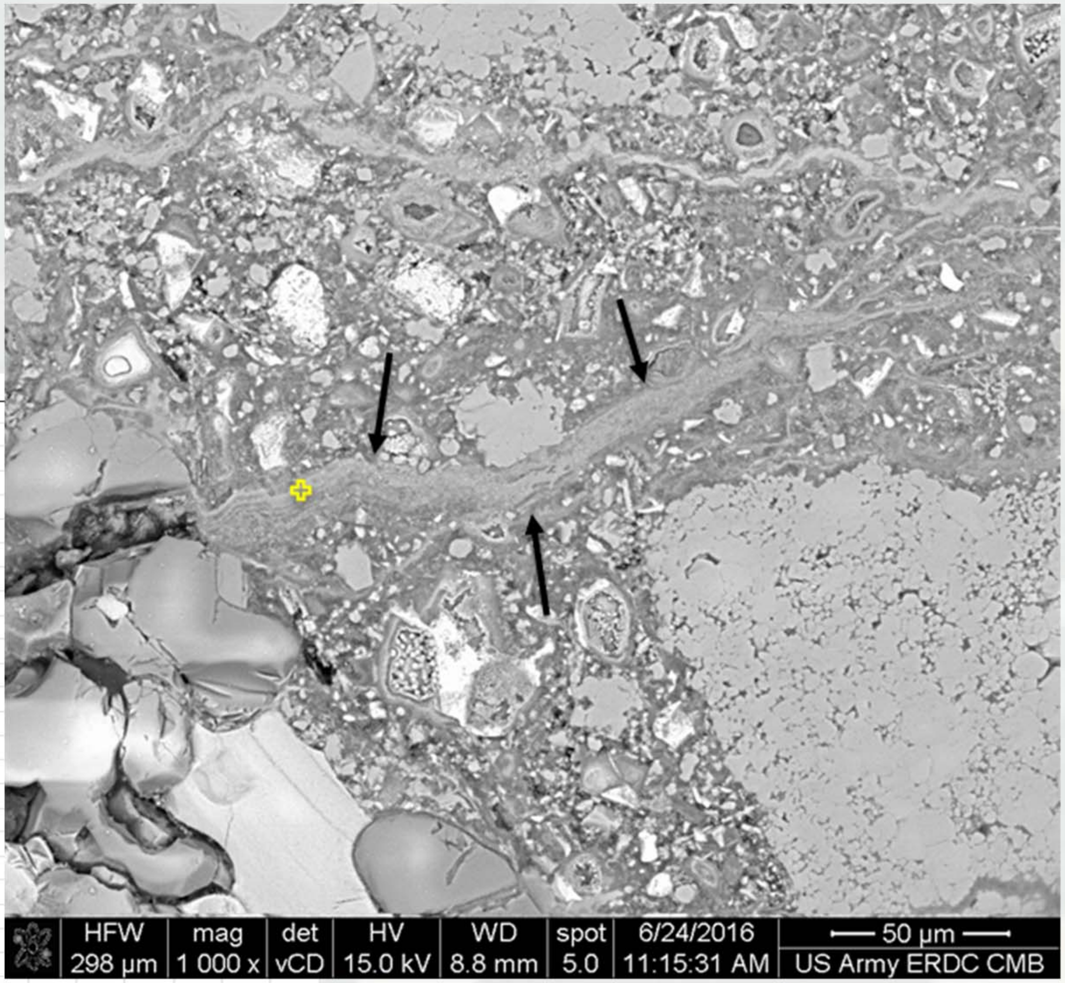




ASR Reaction Product

Las Placitas Aggregate
(HR3-C)





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Conclusions

- Cement alkalinity had essentially no influence on prism expansion.
- ACPT expansions were, on average, more than twice as large for fine aggregate fractions than for coarse aggregate fractions of the same aggregate
- There was a better correlation between ACPT expansions and ASTM C1293 expansions for coarse aggregates (93%) than for fine aggregates (69%).
- Alkali leaching in the ACPT (4 to 11%) was substantially lower than reported alkali leaching in ASTM C1293 (12 to 25%).
- Measuring [SO_4^{2-}] using IC largely resolve the charge imbalance for two of three samples
- SEM/EDX confirmed presence of ASR reaction product in two reactive prisms.

Suggested Applications of Autoclave Testing for ASR

ASTM C1778 – Standard Guide for Reducing the Risk of Deleterious Alkali-Aggregate Reaction in Concrete

TABLE 1 Classification of Aggregate Reactivity

Aggregate-Reactivity Class	Description of Aggregate Reactivity	1-Year Expansion in Test Method C1293, %	14-Day Expansion in Test Method C1260, %
R0	Non-reactive	<0.04	<0.10
R1	Moderately reactive	≥0.04, <0.12	≥0.10, <0.30
R2	Highly reactive	≥0.12, <0.24	≥0.30, <0.45
R3	Very highly reactive	≥0.24	≥0.45

Reactivity Class	Description	ACPT (Coarse Aggregates)	Laval/CANMET (Fine Aggregates)
R0	Non-reactive	< 0.09	< 0.15
R1	Moderately Reactive	≥ 0.09, < 0.12	≥ 0.15, < 0.25
R2	Highly Reactive	≥ 0.12, < 0.15	≥ 0.25
R3	Very Highly Reactive	≥ 0.15	≥ 0.40

TABLE 2 Determining the Level of ASR Risk

Size and Exposure Conditions	Aggregate-Reactivity Class			
	R0	R1	R2	R3
Non-massive[A] concrete in a dry[B] environment	Level 1	Level 1	Level 2	Level 3
Massive[A] elements in a dry[B] environment	Level 1	Level 2	Level 3	Level 4
All concrete exposed to humid air, buried or immersed	Level 1	Level 3	Level 4	Level 5
All concrete exposed to alkalies in service[C]	Level 1	Level 4	Level 5	Level 6

[A] A massive element has a least dimension of greater than 0.9 m [3 ft].
[B] A dry environment corresponds to an average ambient relative humidity lower than 60 %, normally only found in the interior of buildings.
[C] Examples of structures exposed to alkalies in service include marine structures exposed to seawater and highway structures exposed to deicing salts (for example, NaCl) or anti-icing salts (for example, potassium acetate, sodium formate, and so forth).

Suggested Applications of Autoclave Testing for ASR

TABLE 3 Structures Classified on Basis of the Severity of Consequences Should ASR[A] Occur (Modified for Highway Structures from RILEM TC 191-ARP)

Class	Consequence of ASR	Acceptability of ASR	Examples[B]
Class SC1	Safety, economic, or environmental consequences small or negligible	Some deterioration from ASR may be tolerated	Non-load-bearing elements inside buildings Concrete elements not exposed to moisture Temporary structures (service life < 5 years)
Class SC2	Some safety, economic, or environmental consequences if major deterioration	Moderate risk of ASR is acceptable	Sidewalks, curbs, and gutters Elements with service life < 40 years
Class SC3	Significant safety, economic, or environmental consequences if minor damage	Minor risk of ASR may be acceptable	Pavements Foundations elements Retaining walls Culverts Highway barriers Rural, low-volume roads Precast elements in which economic costs of replacement are severe Service life normally 40 to 74 years
Class SC4	Serious safety, economic, or environmental consequences if minor damage	ASR cannot be tolerated	Major bridges Power plants Dams Nuclear facilities Water treatment facilities Waste water treatment facilities Tunnels Critical elements that are very difficult to inspect or repair Service life normally ≥75 years

[A] This table does not consider the consequences of damage as a result of ACR. This protocol does not permit the use of alkali-carbonate aggregates.
[B] The types of structures listed under each class are meant to serve as examples. Some owners may decide to use their own classification system. For example, sidewalks, curbs, and gutters may be placed in the SC3 class in some jurisdictions.

Questions?

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