Emerging Rapid Aggregate and Concrete Test Methods for Formulating ASR-Resistant Concrete

K.W. Liu, A.K. Mukhopadhyay

Texas A&M Transportation Institute, Texas A&M University, 3135 TAMU, College Station, TX 77843, USA

ABSTRACT

The main objective of this study was to develop rapid aggregate and concrete test methods and a combined innovative approach for formulating performance based ASR resistant concrete mixes. An innovative step by step approach has been developed to formulate ASR resistant concrete mixes based on four recommended steps. In step 1, determination of aggregate ASR composite activation energy (CAE) and threshold alkalinity (THA) by using a rapid aggregate chemical test called volumetric change measuring device (VCMD) is performed. The lower the CAE the higher the reactivity is. Based on the measured CAE and THA, mix design formulation is conducted in step 2 by applying mix design controls and special protection measures. In step 3, verification and adjustment of the mix developed in step 2 is performed based on THA and pore solution alkalinity (PSA) relationship - PSA needs to be below THA in order to prevent/minimize ASR. Mix design validation by using a newly developed accelerated concrete cylinder test (ACCT) is a part of step 4. Job concrete mixes made of aggregates with different levels of ASR reactivity were tested using the above approach with the four steps. The CAE-based method shows better correlation with ASTM C1293 than ASTM C1260 and was found to be effective to consistently identify the aggregates belong to false positive (i.e., failed by C1260 but passed by C1293) and negative (passed by C1260 but failed by C1293) categories. The proposed approach has the ability to rapidly assess the ASR potential of each aggregate at various alkali loadings and tailoring mix design depending on the level of protection needed.

Keywords: Alkali-Silica Reaction, ASR-resistant concrete mix, Concrete durability

INTRODUCTION

Alkali-silica reaction (ASR) in concrete is a chemical reaction between alkali hydroxides in pore solution and the reactive form of silica in aggregates and produce ASR gel. The product of this reaction is a gel known as ASR gel. In the presence of sufficient moisture (> 80% RH), the gel absorbs moisture and swells. Swelling leads to tensile stresses in concrete. When these stresses exceed the tensile strength of concrete, crack formation initiates (Chatterji 1989, Chatterji et al. 1986, Diamond 1976, Garcia-Diaz et al. 2006, Hanson 1944, Ichikawa and Miura 2007, McGowan and Vivian 1952, Powers and Steinour 1955, Rodrigues et al. 1999). The current approach of predicting aggregate reactivity and preventing damaging ASR in fresh concrete heavily depends on accelerated mortar-bar tests (AMBTs) [e.g., ASTM C1260/1567] and concrete prism test (CPT) [e.g., ASTM C1293]. AASHTO R80-17 (AASHTO 2017b) provides a procedure for evaluating aggregate reactivity by ASTM C1260 and/or ASTM C1293 and determining measures to prevent ASR on the basis of performance testing or prescriptive selection from a list of different options. Although these approaches have resulted in significant advances in the avoidance of ASR damage in concrete structures, there were limitations and drawbacks (Bauer et al. 2006, Marks 1996, Swamy 1992). ASTM C1260 is a rapid test (14 days testing period) but reliability is questionable because of severity of test conditions and some limitations in the sample preparation. ASTM C1293 is accepted mostly as a reliable test method but long testing time (1-2 year) is a major drawback. A comparative assessment between ASTM C1260 and C1293 leads to generation of aggregates belong to false positive and negative categories followed by creating exclusion list (i.e., recommending not to use those aggregates) by many agencies (TxDOT 2004). New cases of ASR are continuously being reported despite the advancement of the last decades. Therefore, the demand for developing rapid and reliable ASR test methods is high.

The current practice is to assign a common relatively lower level of alkali loading (e.g., 2.1-2.4 kg/m^3) for all concrete mixes irrespective of type of applications such as an example of one size fits for all. However, observation like "aggregates produce expansive gel even at low alkali loadings" questions on the applicability of this kind of approach. Fundamentally, the effective approach of designing ASR resistant mix relies on determining aggregate threshold alkali (THA) of ASR. The current ASR tests are not

capable to determine aggregate THA and test the effects of cement alkalis on ASR. It would be beneficial to accurately, fairly, and rapidly assess the ASR potential of each aggregate at various alkali loadings and develop performance based ASR resistant concrete mixes. The recent acceptance of AASHTO PP84-17 (AASHTO 2017a), a provisional standard on performance engineered materials, is an indication of increasing awareness and importance of using performance based mixes for construction applications. A reliable test along with performance based mix design approach will enable to allow locally available materials (e.g., marginal materials, aggregates in the exclusion list, recycled materials etc.) and promote sustainability in one hand and ensuring durable long lasting structures on the other hand. An effective way of tailoring mix design depending on the level of protection needed is warranted. This will ensure valuable resource conservation and avoid paying for premium ASR protection when only minor protection is needed. More specifically, Industry is looking for a test method or approach to test effectiveness of ASR resistance of field concrete mixtures before placement. No test(s) is currently available to test field concrete mixtures.

To address the above issues, the authors attempted to develop rapid and reliable ASR test methods (i.e., an aggregate chemical test and a concrete cylinder test) and a combined innovative performance based approach to formulate ASR resistance concrete mixes. The scientific theories (e.g., Arrhenius rate theory) and material science aspects of reaction and expansion mechanisms were applied for data interpretation and determining composite activation energy (CAE) as a reactivity parameter for the aggregate chemical test. Developing a procedure to determine aggregate threshold alkalinity (THA) was also an important part in the aggregate solution test. The relationship between pore solution alkalinity (PSA) and THA was the main basis to formulate ASR resistant mixes. A combined approach based on scientific theories and rapid and reliable test methods has the potential to address the above issues and facilities formulating ASR resistance mixes, which ensure long lasting durable concrete.

MATERIALS AND METHODS

Materials

Aggregates (both fine and coarse) with varying levels of reactivity were tested. The reactivity data measured by ASTM C1260, C1293, and AASHTO T364-17 along with the type of reactive siliceous constituents determined by ASTM C295 for the tested aggregates are presented in Table 1.

Table 1. List of aggregates with relevant material data

Aggregate	ASTM C1260 (14-day Exp., %)	ASTM C1293 (1-year Exp., %)	ASTM C295 (reactive constitute)	AASHTO T364-17 (KJ/mole)
FA1 (HR)	0.381	0.391	Acid volcanic, Chert	26.9
FA2 (NR)	0.003	-	Few siliceous inclusions in LS	62.5
FA3 (R)	0.182	0.100	Low strained QTZ, Chert	40.6
FA4 (R)	0.317	0.058	Low strained QTZ, Chalcedony, Chert	32.6
CA1 (NR)	0.012	0.027	Few siliceous inclusions in LS	61.7
CA2 (HR)	1.300	-	Acid volcanic	17.6
CA3 (R)	0.227	0.071	Chalcedony, Chert	41.8

FA: fine aggregate, CA: coarse aggregate, QTZ: quartz, LS: limestone, HR: highly reactive, NR: none reactive, R: reactive

Methods

A combined approach has been developed to formulate ASR resistant mixes, which can be broken down into four steps as follows:

Step 1. Aggregate reactivity and THA determination

Step 2. Concrete mix design formulation by applying conventional mix design controls and special mix design controls (in case of critical situation)

Step 3. Concrete mix design adjustment and verification

Step 4. Concrete mix design validation

Aggregate Reactivity and THA Determination

A rapid (within 5 days) aggregate-solution (chemical) method based on volumetric change measuring device (VCMD) was developed to determine aggregate reactivity in terms of measuring CAE of ASR (AASHTO T364-17) (AASHTO 2017c). The VCMD based chemical method was applied to test all the selected aggregates in Table 1 and determine their alkali silica reactivity in terms of measuring CAE. In this test, as-received aggregate is immersed in 0.5N NaOH (NH) + $Ca(OH)_2$ (CH) solution (similar to concrete pore solution) and solution volume change is measured through float-LVDT-data acquisition system over time at three temperatures (e.g., 60, 70, and 80°C inside an oven) followed by calculating rate constants at the three tested temperatures (T) and determining CAE based on Arrhenius rate theory. The lower the CAE the higher the reactivity is i.e., a highly reactive aggregate needs to overcome a lower energy barrier to initiate ASR compared to a slowly reactive aggregate. Based on a comparative assessment between VCMD method and ASTM C1260/C1293, the CAE-based reactivity prediction showed a better correlation with ASTM C1293 than ASTM C1260. Consistent identification of aggregates belong to false positive (i.e., failed by C1260 but passed by

C1293) and negative (passed by C1260 but failed by C1293) categories (Mukhopadhyay and Liu 2014) was found to be main benefit of the VCMD method. The VCMD method was also used to conduct aggregate testing at multiple alkali levels (e.g., 1N NH + CH, 0.5N NH+CH, 0.25N NH + CH) in order to develop CAE vs. alkalinity relationship and determine aggregate THA of ASR from that relationship. In general, the higher the aggregate reactivity (i.e., the lower the CAE) the lower the THA is. The THA is a very useful parameter to determine permissible concrete alkali loading for different aggregate sources. PSA of different concrete mixtures covering low to high alkali loadings was determined by using the pore solution extraction techniques (Barneyback and Diamond 1981) and a X-ray fluorescence (XRF). A linear relationship between PSA (normality) and alkali loadings (kg/m^3) was then developed. This linear equation was used to covert the measured THA (normality) to threshold alkali loading (TAL, in kg/m^3). A reactive aggregate can practically behave as non-reactive or very slow reactive if concrete alkali loading remains below TAL. The VCMD method has the merit to be used as an alternative to the ASTM C1260. However, the user has the option to select any suitable rapid and reliable method to determine aggregate reactivity and THA.

Concrete mix design formulation by applying conventional mix design controls and special mix design controls

Safe level of concrete alkali loading changes depending on the measured TAL of individual aggregate. The current practice is to assign a common alkali loading (i.e., 2.1-2.4 kg/m^3) for all concrete mixes irrespective of aggregate threshold alkali loadings and type of applications (case of one size fits for all). For example, if the aggregate reactivity is high and TAL is low and the concrete needs to be placed under a high severity exposure conditions (e.g., high rainfall, high T, exposed to external chemicals such as de-icing and chloride etc.), the use of suitable special protection measures (e.g., use of lithium compounds or other suitable chemicals) in addition to the conventional mix design controls (e.g., 35% of class F fly ash or use of a combination of SCMs, etc.) is needed. On the other hand, if the reactivity is low and TAL is high, the use of conventional mix design controls (e.g., 20-25% FAR) without using any special protection measures is adequate. Depending on the reactivity and TAL of an aggregate, fly ash of varying CaO% can be judiciously used to make concrete. For example, a fly ash with a high CaO% (e.g., blended ash or class C ash), can be used with an aggregate of high TAL and low reactivity. However, the use of a good quality class F ash is must for an aggregate with low TAL and high reactivity.

Concrete Mix Design Adjustment and Verification (optional but recommended)

This step provides an additional control, i.e., PSA needs to be below THA in order to prevent or minimize ASR. The mix design formulation by applying conventional mix design controls and special protection measures (wherever needed) should satisfy this requirement. However, some SCMs (especially some fly ashes) may contribute alkalis in pore solution and alkali loading assignment based on cement alkali alone may not provide adequate control. Therefore, it is recommended to determine PSA of the concrete mix by extracting pore solution from equivalent cement paste samples after suitable hydration age followed by chemical analysis by an XRF or any suitable device. If the PSA is not lower than THA, fine tuning of the concrete mix by applying additional controls (i.e., suitable combination of mix design ± special protection measures depending on the requirements) needs to be performed. If pore solution extraction method is not available, introduction of test to measure soluble alkalis of fly ash thought to be effective to avoid using fly ash with high soluble alkalis.

Concrete Mix Design Validation

Verification and validation of the mixes by using rapid but reliable concrete test(s) is the best way to ensure safe and durable mixes. However, the current practice of using ASTM C1293 to validate mixes takes 2-year time. An accelerated concrete cylinder test (ACCT) is recommended to use to validate the mixes. An ACCT was developed earlier (Liu and Mukhopadhyay 2015, Mukhopadhyay and Liu 2014) to determine the length change of concrete cylinder (76.2 × 152.4 mm) using LVDT-data acquisition system due to ASR at a temperature of 60°C. In this test, concrete cylinder is immersed in a soak solution with chemistry equal to concrete PSA, which ensures no leaching test condition. Because the data collection in the ACCT is automatic through LVDT (no human error) under constant temperature inside an oven (no error due to temperature difference) and leak-proof condition, the reliability of the ACCT is high. ACCT with relatively low alkali loading (i.e., 2.7 kg/m^3 without any alkali boosting) was effectively used to determine aggregate reactivity in a relatively short time (e.g., 28 days for very highly and highly reactive aggregates and 42 days for a moderately reactive aggregate) and showed a favorable comparison with ASTM C1293. It is to be noted that ASTM C1293 uses high alkali loadings (i.e., 4.0-5.3 kg/m^3) through alkali boosting. It is anticipated that the adjusted mixes (job concrete mixes after step 2 or 3) will not show any expansion or expansion will remain below the limit (i.e., 0.04%) by the ACCT at the specified testing period (56-70 days) (Mukhopadhyay and Liu 2014) and these mixes can safely be used for the intended applications. However, the mixes that don't pass by the concrete validation testing can't be used for the intended applications without further adjustment. Although ACCT is strongly recommended in this step, the user has the option to

select any suitable rapid and reliable concrete test method, which has the ability to test job concrete mixtures.

RESULTS AND DISCUSSION

Formulation of ASR resistance concrete mixes using aggregates with varying levels of ASR reactivity and two class F fly ash with varying replacement levels were conducted using the proposed 4 steps. Table 2 as part of step 1 shows CAE-based ASR aggregate classification system. The results of steps 1 to 3 are presented in Table 3. A brief discussion for the results obtained by applying the recommended steps are provided below:

Table 2. CAE-based ASR aggregate classification

Aggregate reactivity class	Description of aggregate reactivity	CAE ranges** (KJ/mole)
R0	Nonreactive	≥60
R1	Moderate reactive	45-62
R2	Highly reactive	30-45
R3	Very highly reactive	≤30

**: Based on AASHTO T364-17

Table 3. Results of the proposed steps 1 to 3

Mix	Step 1: aggregate reactivity class	Step 1: THA (N)/TAL (kg/m^3)*	Step 2: mix design controls (kg/m^3)	Step 3: mix design adjustment and verification (PSA, N)	Step 4: ACCT Exp. prediction based on THA vs. PSA
1	R3 (FA1) R0 (CA1)	0.35/1.8 (FA1)	1.7	0.33 < THA	< 0.04%
			1.9	0.35 = THA	≥ 0.04%
			2.0	0.43 > THA	> 0.04%
			2.1	0.44 > THA	> 0.04%
			2.7	0.66 > THA	> 0.04%
2	R3 (CA2) R0 (FA2)	0.29/1.7 (CA2)	1.9	0.35 > THA	> 0.04%
			2.1	0.44 > THA	> 0.04%
			2.7	0.66 > THA	> 0.04%
3	R2 (FA3) R2 (CA3)	0.45/2.2 (FA3)	1.9	0.35 < THA	< 0.04%
		0.46/2.2 (CA3)	2.1	0.44 < THA	< 0.04%
			2.7	0.66 > THA	> 0.04%
4	R2 (FA4) R0 (CA1)	0.52/2.4 (FA4)	1.8	0.38 < THA	< 0.04%
			2.7	0.66 > THA	> 0.04%
			5.3	1.01 > THA	> 0.04%

N: normality, Exp.: expansion, *: Based on a linear relationship between PSA (N) and alkali loadings (kg/m^3)

Aggregate Reactivity and THA Determination
Aggregates were tested by AASHTO T364-17 as well as the conventional methods (i.e., ASTM C1260 and C1293). The reactivity classification listed in Table 3 (2nd column) based on AASHTO T364-17 is in good agreement with the prediction based on ASTM C1260 and C1293 tests (Table 1). The THA (normality) / TAL (kg/m^3) values for each aggregate

were determined and presented in the 3rd column of Table 3 as a part of step 1.

Concrete mix design formulation by applying conventional mix design controls and special mix design controls
All four mixes listed in Table 3 are standard mixes with different levels of alkali loadings (1.7 to 5.3 kg/m^3). The use of a high-alkali (Na_2O_{eq} = 0.82%) Type I/II Portland cement and adding extra NaOH pallets were adequate to achieve the alkali loading of 5.3 kg/m^3. The water-to-cement ratio (0.45) and coarse aggregate factor (0.76) remain constant for all the mixes. The mixes with high alkali loadings (i.e., alkali boosted mix with 5.3 kg/m^3) are similar to standard mixes specified for ASTM C 1293. As a part of conventional mix design practice to control ASR, two class F fly ashes were used with varying replacement levels. The reduction of alkali loading with increasing fly ash replacement (FAR) levels is manifested in 4th column for step 2. For example, the alkali loading for Mix 1 without any class F fly ash is 2.7 kg/m^3. The alkali loading reduced to 2.1, 2.0, 1.9, and 1.7 kg/m^3 with corresponding fly ash replacement levels 20, 25, 30 and 35% respectively.

The Table 3 indicates that there are some aggregates (FA1 and CA2) for which concrete alkali loading should be below 2.1 kg/m^3 and the current conventional mix design controls (i.e., 2.1-2.4 kg/m^3) will not be adequate to control ASR for those aggregates.

Concrete Mix Design Adjustment and Verification (optional but recommended)
Pore solution were extracted from the representative paste specimens (50.8 x 101.6 mm) for each mix and chemical composition is determined by XRF to know the PSA. The measured PSA values for all the studied mixtures are listed in 5th column for step 3 in Table 3. A comparative assessment between THA values in 3rd column (step 1) and PSA values in 5th column (step 3) allows predicting the ACCT expansion behavior (6th column - step 4) of the different mixes. For example, no measurable ASR expansion or expansion will be below the limit (0.04%) is predicted for the mixes with PSA < THA [e.g., Mix 1 with 1.7 kg/m^3 (35% FAR), Mix 3 with 1.9 kg/m^3 (30% FAR) and Mix 4 with 1.8 kg/m^3] and these are safe mixes. Similarly, when the THA is very close to PSA (e.g., boarder line cases - Mix 1 with 1.9 kg/m^3 (30% FAR) and Mix 3 with 2.1 kg/m^3 (20% FAR), the expansion can be equal to or slightly greater or slightly lower than the expansion limit (0.04%) depending on the aggregate reactivity. It is recommended to increase the fly ash content (> 30% for Mix 1 and > 20% for Mix 3) in order to make these mixes safe. However, when the PSA is greater than THA, the expansion will be higher than the limit at a specified testing duration and those mixes (e.g., Mix 1 with 0-25% FAR, Mix 2 with 0-30% FAR, Mix 3 with 0% FAR, and Mix 4 with alkali loading 2.7-5.3 kg/m^3) are not safe.

Concrete Mix Design Validation

The predicted ACCT expansion (6th column in Table 3) is validated by testing those mixes by the ACCT methods. The results (expansion vs. time curves) for the studied mixtures are shown in Figures 1 to 4.
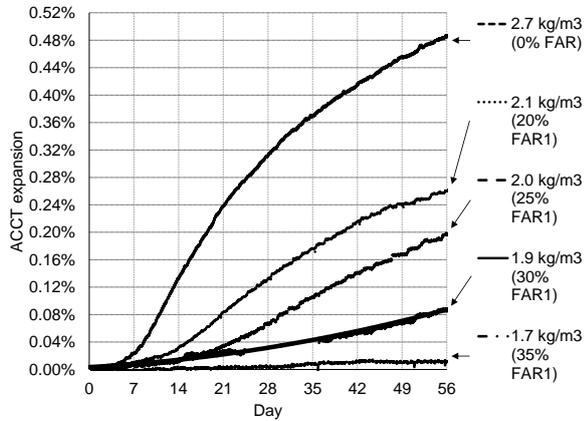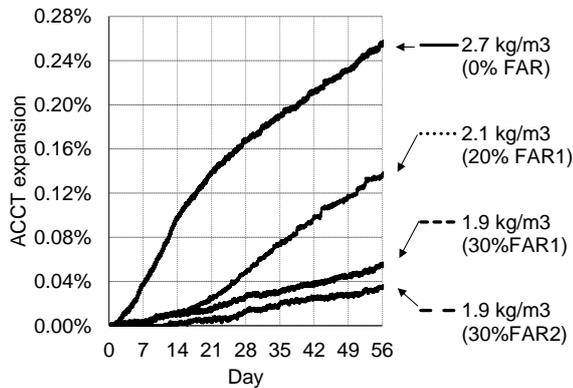

Fig. 1. Expansion over time for Mix 1

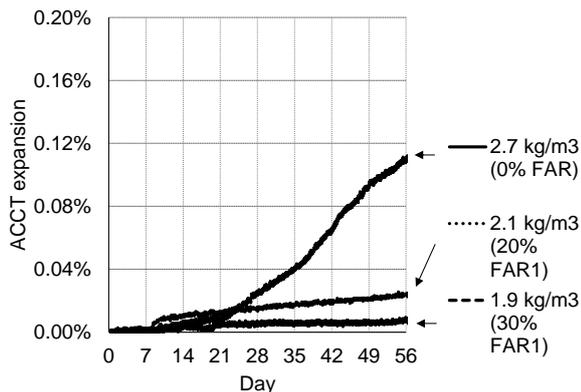**Fig. 2.** Expansion over time for Mix 2
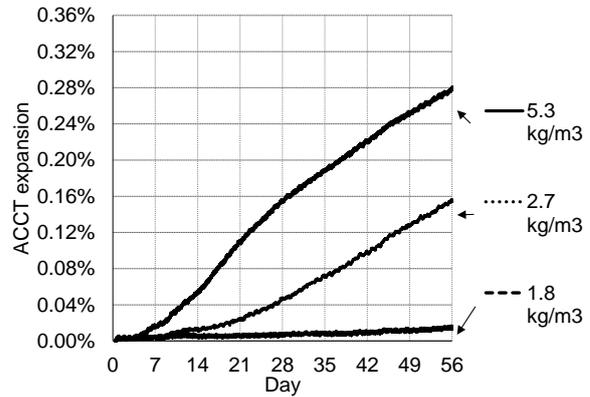

Fig. 3. Expansion over time for Mix 3

Fig. 4. Expansion over time for Mix 4

A perusal of Figures 1-4 clearly indicates that the predicted expansion based on PSA and THA relationship (6th column in Table 3) is validated by the ACCT expansion data in general. Some DOTs allow only 25% fly ash in their mixes (a standard replacement level irrespective of fly ash type or quality) and the results in Table 3 indicate that for some mixes 25% FAR (2.0 kg/m^3) (Mixes 1 and 2) are not adequate to control ASR. For the aggregate used for Mix 1, 35% FAR (1.7 kg/m^3) is adequate. For the aggregate used in Mix 2, 35-40% FAR may be adequate. For Mix 2, use of lithium compound or other chemicals along with conventional fly ash level (if high fly ash content is not allowed) is recommended to make this as ASR resistant safe mix. For Mix 3, 30% FAR is adequate but 25% FAR may work. Therefore, this approach has great potential to optimize fly ash and other SCMs replacement level depending on aggregate reactivity and PSA, which is new and very innovative. Generating more data might be needed in order to safely (high reliability) apply this approach to approve concrete mixes in ASR prone area and ensure long lasting durable concrete.

CONCLUSIONS AND RECOMMENDATIONS

All the four mix design steps 1 to 4 are recommended in order to develop ASR resistant case specific mixes (performance based) with high reliability. After generating a large volume of data using all the four steps, if a strong agreement between mixes developed through steps 1-3 and validation testing in step 4 is observed then requirement of concrete validation testing (step 4) can be considered as optional. However, the user needs to take this decision based on experience and proper judgement. For the aggregates with which the reactivity prediction based on the current rapid (e.g., ASTM C1260) test methods is satisfactory, CAE measurement through aggregate-solution test may not be needed and the user can develop mix based on guidelines in step 2 and mix design verification in step 3. However, mix design validation through the direct concrete validation testing (step 4)

will be very useful and highly recommended. Table 4 provides different options on using different steps along with test duration and reliability for formulating ASR resistant concrete mixes.

Table 4. Different options for formation of ASR resistant mixes

Option	Step	Test duration	Reliability	Outcomes
I	1 to 4	~ 3 months	High	Formulation of ASR resistant mixes (Job concrete mixes) with validation by concrete testing
II	1 to 3	≤ 20 days	Medium	Formulation of ASR resistant mixes (Job concrete mixes) without concrete validation testing
III	4	28-42 days	Medium-High	Determination of aggregate reactivity followed by mix design formulation using current practices
IV	4	56-70 days	High	Validation of ASR resistant mixes based on option III or preventive measures provided in AASHTO R80-17

The proposed approach can be easily merged with the flow chart recommended by AASHTO R80-17 to formulate ASR resistant mixes. For example, VCMD (step 1) and ACCT (step 4) can be used wherever R80-17 recommends using test methods (i.e., AMBT or CPT) to determine aggregate reactivity class (R0 to R3) in Table 1 of R80-17. Based on the aggregate reactivity class determined in the proposed step 1, the level of ASR risk, level of prevention, and preventive measures can be selected according to Tables 2, 3, 5 to 8 in R80-17 to formulate ASR resistant mixes. Verification and validation of the ASR resistant mixes by using the proposed step 4 (i.e., ACCT) is an effective way to test job concrete mixes and ensure placement of safe and durable concrete mixes.

Acknowledgement

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