Developing Specifications for Sustainable Concrete Binder Technologies

R. Douglas Hooton[1], Lawrence Sutter[2] and Mary Christiansen[3]

Abstract

There has been a proliferation of research and development activity focussed on more sustainable binder technologies for concrete construction. Some of these Alternative Cementing Material (ACM) technologies have been used successfully in construction. However, one of the major barriers to adoption of such new materials is the lack of appropriate test methods and specifications, and this precludes their use in building code structural applications. Existing test methods for evaluating portland cement concretes often do not relate well to field performance and therefore are suspect for evaluating new cementing materials, but engineers have historical experience, familiarity and comfort level with using portland cement concrete.

In addition to raising several issues with existing test methods, several alternative approaches to development of specifications for ACMs are presented.

1. University of Toronto, Dept. of Civil Engineering, Toronto, Ontario, Canada M5S1A4 (hooton@civ.utoronto.ca)
2. Michigan Technological University, Materials and Science Engineering, 1400 Townsend Dr. Houghton, MI, 49931-1200 (llsutter@mtu.edu)
3. University of Minnesota-Duluth, 1405 University Drive, Duluth, MN 55812-3007 (muchrist@d.umn.edu)

I. INTRODUCTION

Most concrete construction design codes and the corresponding materials specifications allow only for the use of OPC concrete, with or without the use of supplementary cementitious materials. To embrace the sustainability and performance opportunities presented by ACMs, the concrete industry is faced with the challenge of incorporating ACMs into construction projects. As previously stated, there are a number of low-risk applications where this can and does occur. Likewise, there are numerous niche applications where ACMs offer unique performance benefits and are therefore already embraced. It is the large-scale projects such as roads, dams, and vertical construction where changes in codes and specifications are required.

It is clear that gaps in testing exist, both in terms of the applicability and availability of the necessary tests, but also in terms of the actual results from those tests that are available. Clearly, for architects and engineers to develop a comfort with ACMs, long-term performance results are required. Unfortunately, it takes twenty years to establish that a material will last twenty years; accelerated testing is useful but has its limits. To embrace ACMs in the near term, the concrete construction industry must adopt an overall strategy to i) develop the necessary material specifications and associated tests, ii) use ACMs in noncritical applications (i.e., life safety is not a risk), iii) develop the fundamental concrete performance data required to design structures using ACMs, and iv) amass the necessary data and experience to move ACMs into every appropriate use opportunity. The American Concrete Institute has created and Innovative Task Group, ITG-10, to provide a state of the art report and to recommend guidance for incorporating ACMs into codes and standards. This is being developed by the authors of this paper.

In most cases when a cementitious material is to be used in concrete construction, it needs to comply with architectural or engineering specifications. Material specifications establish limits for key properties and typically define the required testing needed to measure those properties. Current specifications for OPC contain a mixture of prescriptive-compositional and quasi-performance property limits. This approach means that novel materials often cannot meet these requirements due to having a significantly different composition, having different sample preparation or curing needs, or due to limits imposed by the test methods. In many cases, ACMs cannot be adequately evaluated by existing standard test methods.

To circumvent issues caused by prescriptive specifications, another approach is development of purely performance-based specifications that could potentially apply to all cementitious binder materials and combinations, and allow for direct comparisons of different materials. The barriers to this approach include a general lack of agreement regarding the performance properties that need to be tested and the lack of appropriate test methods that would better link a material's properties to performance when used in concrete. This holds true for ACMs and OPC alike.

II. TEST METHODS

The following list covers some of the main issues that need to be demonstrated in the development of standards for new concrete materials where field experience and long-term performance data is not available [1]:

1. The material will do no harm.
2. The material will have required, predictable, and reproducible fresh and hardened properties (e.g., can the material be air-entrained, maintain adequate workability during delivery, placement, and finishing).
3. The material will be sufficiently robust to perform as expected over the range of temperature and humidity encountered in practice.
4. The relationship between different strength properties (e.g., compressive, tensile, shear, flexure, bond) and elastic modulus must be established since the relationships often assumed in building codes may differ from those of commonly used hydraulic cements at different temperatures and humidities.
5. The material has short and long-term volume stability (e.g., thermal, drying shrinkage, creep).
6. The material is durable in different environmental exposures encountered in practice.
7. The material's performance properties when used in concrete demonstrate sufficient uniformity (e.g., uniformity in workability, setting time, strength development).

Assembling the information described above requires a combination of testing the ACM and separately testing the concrete produced using the ACM.

A. Chemical and Mineralogical Composition

Determination of chemical composition is invariably the starting point for testing and specification of cementitious materials. Chemical analysis can be used to identify and quantify the chemical components of a material, both desired and deleterious. Chemical analysis can serve to identify deleterious components (e.g., loss on ignition and MgO content in ASTM C150) but what is deemed deleterious in one material may be desired in a different material. Therefore, prescriptive chemical composition limits in material specifications generally make that specification pertinent to only one narrow class of material. Performance specifications typically do not stipulate chemical requirements and are preferred by those promoting use of ACMs for that reason. However, in the absence of chemical requirements, demonstrating uniformity is a challenge. Most recognized performance tests require significant time to complete (e.g., 7-day strength) and are therefore performed less frequently, making the possibility of variation between tests significant. As tests and specifications evolve for ACMs, likely some type of chemical requirements that relate to uniformity will be required, or rapid performance tests must be developed. As a minimum, required chemical tests should be used to identify and quantify any deleterious components specific to a particular ACM.

In Portland cements, the quantities of the four major minerals impact on their performance, and minor components can impact on volume stability and durability. Similarly, the mineralogy of ACMs need to be known in order to develop a basic understanding of phases that could potentially impact on performance, volume stability and durability.

B. Measuring Strength

Strength is arguably the most critical characteristic to measure if for no other reason than it being the characteristic most specifiers will turn to first when evaluating a cementitious material. Strength (i.e., compressive strength) provides a measure of uniformity that is universally accepted and most importantly, it links directly with concrete performance and the key measures to be examined when developing concrete specifications for use in building codes. Although the actual measurement of strength is easily accomplished, it is a challenge to do so for a wide range of ACMs and in a manner that allows each to be directly compared to each other or OPC strength. The challenge is to develop a mixture design for each type of cementitious material that can be directly compared to each other. This includes providing a curing regime for each ACM mixture that results in comparable mixture design strength. The rate of strength development also varies greatly among ACMs, especially with relation to curing regimes, and so an age at which the strength should be measured and recorded must be agreed upon or perhaps a minimum strength at any age specified.

In standards such as ASTM C109, strength of a cementing material is measured at a fixed *w/cm* or at a given flow. However, for many ACMs, the *w/cm* ratio does not relate to strength, the ACM is not hydraulic, or the measurement of flow may be not applicable. In the case of geopolymers, heat curing is typically required to achieve full strength. ASTM subcommittee C01.13 on Special Cements has recently been working on a modification of the mortar cube strength test for ACMs.

C. Volume Stability

In cementitious binder material specifications including ASTM C150, C595, C618, C989, C1240, C1600 and C1697, volume stability is measured by the C151 autoclave expansion test, the C1038 expansion test and, in some specifications either the C157 (used in C595 and C618) or C596 (used in C1157 and C1600) drying shrinkage test. The purpose of the autoclave test is to protect against deleterious quantities of crystalline MgO (periclase) and CaO (free lime) in binder materials. Paste prisms are cast and in one day exposed to steam for 3h at 215 ºC. After cooling, the length change is measured. In the case of geopolymers, alkali-activated binders, and magnesium silicate binders, this temperature regime would provide an ideal curing environment, and it is unlikely the autoclave test would give an indication of “unsoundness”. Also with ACMs, it is possible that other types of volume stability issues may exist that would be specific to the composition of specific ACMs; potential concerns would likely have to be assessed on a case-by-case basis.

The ASTM C1038 test method may be suitable to assess volume stability of ACMs with respect to excess sulfate content, but the time of test may not capture other potentially expansive reactions that could occur due to other components in certain ACMs; producers would be prudent to run this test for a much longer period than 14 days when initially evaluating their products to check for other potential deleterious expansion mechanisms. These test methods may not be relevant to other types of cementitious systems that may not contain periclase, free lime or calcium sulfates.

However, completely different types of cementitious systems may have other components that result in volume stability issues. If so, the mechanisms need to be understood in order to develop new test methods and specification limits to protect the consumer.

D. Heat of Hydration

The ASTM C1702 isothermal conduction calorimetry heat of hydration test appears to be suitable for measurement of the heat of hydration of ACMs. As has been the case with previous tests discussed, the central challenge is determining a standard mixture for testing. Because of the wide range of heat evolution associated with ACMs (e.g., the high heat release of MOC), it is likely that heat of hydration will evolve to a report only item, at least for ACMs. The recently adopted limits on heat of hydration by C1702 in ASTM C150 may not be applicable to ACMs.

E. Admixture Compatibility

Due to the differing chemical composition and early-age soluble species of different ACMs, it is possible that chemical admixtures formulated for OPC systems will not behave as intended. This problem was acknowledged for alkali-activated slag and fly ash binders, and chemical admixture companies are currently working with manufacturers of ACMs to develop new types of admixtures [2]. Standard Practices such as ASTM C1697 could be used to evaluate hydration kinetics of ACMs combined with different admixtures using isothermal conduction calorimetry. Some issues, such as the ability to air entrain ACM concrete, might be better tested in concrete trials.

III. CONCRETE TESTS

Once test methods and specifications for ACMs have been developed, concretes made with ACMs need to be tested. Basic properties that need to be tested are:

1. Compressive Strength (Including Strength Gain/Degradation with Time)
2. Stiffness (Elastic Modulus)
3. Volumetric Expansion (Poisson's Ratio)
4. Ductility (Toughness)
5. Tensile Strength
6. Temperature Effects (Strength Degradation, Coefficient of Thermal Expansion)
7. Time Dependent Properties (Shrinkage/Creep)
8. Bond
9. Durability
10. Resistance to Freeze-Thaw
11. Resistance to Fluid Transport
12. Resistance to Sulfate Attack
13. Resistance to Acids, Chemical Attack
14. Resistance to Alkali-Aggregate Reaction

A. Compressive Strength

While there is not space to discuss all of the issues listed above, the measurement of compressive strength typifies, in general, the challenge of testing most common properties of ACM-based concrete mixtures. Compressive strength testing of OPC concrete is performed in accordance with ASTM C39, which in turn references ASTM C31 for preparation of field specimens and C192 for preparation of laboratory specimens. ASTM C31 requires specimens to be prepared using a “standard cure” when testing for strength. ASTM C192 imposes conditions for the aggregate used and strict protocols on mixing, consolidation, and curing of specimens, all of which may not be conducive to optimizing properties of an ACM concrete. Given these requirements for the specimens tested, the applicability of C39 is questionable. However, the fundamental test at the heart of C39 (i.e., failure under a compressive load) is valid. The issue is specimen preparation. A generalized approach must be developed for ACM-based concrete with the result being, in this case, a strength test that can be compared to differing types of ACM-based concrete and OPC-concrete.

B. Thermal Properties and Issues

The thermal response of an ACM to different curing regimes, and to both cold and hot weather placing conditions, needs to be determined to demonstrate its robustness when placed in the typical range of field conditions. Some ACMs develop excellent properties when exposed to elevated temperatures, such as when used in thick concrete elements, but thin concrete elements that do not experience these elevated temperatures will not develop the same strengths. Each ACM will have to be tested to determine if its thermal response is significantly different from that of OPC concretes.

The determination of temperature effects, particularly associated with catastrophic events such as fire, will rely heavily on full-scale testing. For structural applications, fire endurance is typically assessed by ASTM E119 Standard Test Methods for Fire Tests of Building Construction and Materials. In the test, a specimen is exposed to a controlled fire designed to achieve specific temperatures over a measured time range. The test is generally applicable to ACM-based concrete as well as OPC concrete. ASTM publication STP-169D [3] provides a summary of how basic material properties affect concrete and some assessment of how high temperature performance can be determined from basic principles. However, from a life-safety perspective, full-scale testing must be conducted.

The coefficient of thermal expansion (CTE) needs to be determined for ACMs. The similarity in CTE between OPC concrete and reinforcing steel is one of the reasons that the composite works well. Large differences in CTE could lead to unexpected problems in service.

C. Time-Dependent Properties

ASTM C157 (linear shrinkage) and C1581 (restrained shrinkage) are intended for use with OPC concrete and predictably, both are very sensitive to the mixture design and curing regime. The curing conditions for these tests would have to be modified to test ACMs.

Although creep is not frequently specified, it is clearly considered in design codes [4],[5] and a fundamental understanding of creep under compressive loading or tensile loading is required for implementation of ACM-based concrete.

D. Bond.

Determination of bond strength is central to the reinforced concrete design process. It is an important piece of information that needs to be determined for ACM concretes and structural reinforcement but a primary challenge for implementing ACM-based concrete into design codes and specifications is that standard procedures for determining bond do not exist. ASTM A944 states, “*The bond strengths obtained using this test method shall not* *be directly applicable to the design of reinforced concrete members*."

E. Resistance to Fluid Ingress

Tests that measure chloride penetration via chloride profiling, such as ASTM C1556 or using chloride-sensitive color indicators, such as Nordtest NT Build 492, are more appropriate than electrical-based measurements such as ASTM C1202 or resistivity tests. Many of the hardened ACMs are characterized by low permeability, which leads to a number of their desired properties. As an example, geopolymers tend to show reduced chloride ingress compared with OPC concrete or even blended cements [6]. However, the interpretation of accelerated test data is complicated by the strong influence of the pore solution composition, which is a function of the nature of both the geopolymer precursor and the activator. This can yield unexpected results when comparing results obtained through tests such as ASTM C1202 with microstructural measurements for the same mixtures [7],[8].

F. Sulfate Resistance

The chemical composition of the binder can vary greatly among ACMs, so predicting resistance to deleterious chemical reactions such as sulphate attack based on chemical composition, as is done in C150, is often inaccurate when it comes to ACMs. For example, sodium sulfate is one of the activators that can be used in alkali-activated systems [9], so immersion tests in sodium sulfate such as ASTM C1012 may not serve the purpose of determining sulfate resistance of ACMs. Many types of geopolymers and alkali-activated binders can show superior performance when compared to sulfate-resisting portland cement, as they often do not contain the monosulfoaluminate phases necessary to form ettringite [10]. There is some evidence to show that calcium-rich geopolymers are not resistant to magnesium sulfate solutions and lose strength [8] Concretes made using Calcium Sulfo-Aluminate cements show excellent sulfate resistance [11].

G. Alkali-Aggregate Reactions

There is concern for alkali-aggregate reaction (AAR) with ACMs. Many of these materials, especially geopolymers, contain high levels of alkaline activators so there is a natural concern about the potential for AAR. However, these alkalis generally become bound in the final product, especially in binders containing high levels of alumina-containing pozzolans or slag.

With respect to testing, the main tests available are ASTM C1260 and ASTM C1293, both of which use elevated temperatures to accelerate AAR reactions, but could also accelerate changes in the properties of ACMs. The fundamental problem is these tests are empirical, which means they are based on experience only and are only valid if done exactly the same way each time. For example, an expansion of less than 0.04% at one year using C1293 has been shown to correlate with field experience that shows those aggregate and cement combinations to be not deleterious. In addition, it is accepted that using ASTM C1260, an expansion of less than 0.10% at 14-days correlates with a C1293 expansion of less than 0.04% at one year. If any aspect of these tests is changed, such as the mixture proportions, solution strengths, exposure time and conditions, or the cement type, the empirical correlation may not apply.

In short, there is no basis to assume these tests can be used with ACM-based concrete and there is no basis to accept the results as demonstrating AAR durability. This is an area where immediate research is needed to begin testing ACM-based materials and simultaneously developing field test sites to obtain long-term exposure data for correlation with standard tests results. In the absence of this correlation, the industry has no guaranteed method of confirming AAR durability with ACM-based concrete.

In general, alkali activated slag concretes are inherently susceptible to alkali-aggregate reaction due to the high alkalinity of their pore solution as a result of the alkali activation [12]-[14].

IV. SPECIFICATIONS

A. Performance Specifications

Development of a purely performance specification, similar to ASTM C1157, *Standard Performance Specification for Hydraulic Cement*, could potentially apply to all types of hydraulic, and possibly non-hydraulic, cementitious binder materials and allow for direct comparisons of performance. Note that C1157 is for hydraulic cements only, and C1157 was developed with the vision of the materials so specified containing some portland cement. Therefore, the applicability to cements that are not hydraulic or contain no portland cement is questionable, as not all performance concerns are addressed in C1157. Overall potential barriers to a performance based approach include, i) a clear list of all potential performance properties is lacking, ii) agreement on a single set of properties to be tested without over testing some materials, or not testing key properties in other materials, and iii) a lack of appropriate test methods that link to performance when these materials are used in concrete. It is more conceivable that a performance specification could be developed for each class of ACM.

Another existing performance specification that could be considered for ACMs is ASTM C1600, *Specification for Rapid Hardening Hydraulic Cement*. However, its adoption across all ACM types could be problematic. Although it is a performance specification, it only has mandatory requirements for setting time, strengths at times ranging from 1.5h up to 28 days of age, drying shrinkage, and autoclave expansion for each of the four strength types listed. Many types of cementing materials can meet the performance requirements of this specification, such as activated fly ash materials. Some calcium aluminate cements could also meet this specification then potentially lose significant strength at later ages in warm, moist environments due to conversion reactions of the initial CAH_{10} hydrate to C_3AH_6 (losing approximately 50% of their original strength). This would not be a problem if the converted strengths were still adequate, but currently the specification is silent on this possible issue. For completely novel materials, additional issues would need to be identified and addressed to provide sufficient confidence in their long-term durability and performance.

B. Using Existing Specifications

The desire of those promoting a given ACM is to include any new material into an existing specification, to facilitate inclusion in building codes where that material specification is referenced. The alternative is that a new material specification must be developed and a modification to the code must occur, both time-consuming activities. Although this latter approach may seem slow, it should be considered that adoption into the building code will be the rate determining step because adoption will necessitate gathering fundamental data on concrete performance, and likely long-term performance data and experience, both of which will take considerable time. Development of a material specification first will facilitate early use in low-risk applications and will accelerate the lengthier step of adoption into a design code.

C.Use of Alternative Cementitious Materials in Non-Critical Applications

To a great extent, ACM producers are already executing this step but additional opportunities need to be identified. The ACI user community can assist by examining and exploiting opportunities to use ACMs in low-risk designs by identifying applications where life safety is not an issue or where environmental conditions pose minor challenges. Alternatively, identify niche applications where unique properties of the ACM provide performance, cost, or sustainability advantages. Development of a material specification for each ACM will facilitate specifying materials in all applications. Moreover, it will provide a means to measure and document uniformity of the ACM from job to job, thereby adding credibility to any performance data gathered.

The following points need to be considered to maximise the beneficial impact of each ACM field trial:

a) To the extent possible, treat each placement as an opportunity to gather data.

b) Document construction and conditions, quantities of materials used, mixture designs, mixture design testing, durability testing, and fresh concrete test results.

c) As opportunities allow, document performance and condition after placement on a regular basis (e.g., annually) using standard approaches such as Long-Term Pavement Performance (LTPP) distress surveys and International Concrete Repair Institute (ICRI) guidelines.

d) Make public all data through publication in journals or other means and use the results to guide larger research projects to develop fundamental performance data.

In addition to characterization of the ACM itself, characterization of the resulting ACM-based concrete is key to adoption of these novel materials in any design code. As an example, many ACI 318 design equations are a mixture of theory and empiricism, where basic engineering relationships form the basis of the equation but data amassed from years of academic and industrial research provide coefficients and corrections that fine tune the equation for a specific application. To provide this data, the required testing is as varied as the breadth of design applications. It will take 10's of years to acquire this data meaning the industry needs to start this testing now.

Unfortunately, unlike with OPC concrete as it was developing, there is no central research agency (e.g., Portland Cement Association) to lead research on the scale that is required, and resources in start-up ACM companies to conduct this work are scarce. Therefore, the industry faces a significant challenge to acquire the necessary data. The ACI can play a role in facilitating research through formation of a technical committee addressing ACMs and in turn, that committee must work with ACI Committee 318 to develop a road map for the data to be determined and a process for conducting that work.

Most challenging is the gathering of long-term performance data. ACM producers should commence immediately with production of test specimens to be placed in various climates around the world, monitor those specimens over time, and document performance. There is no short cut for determining long-term performance. Most of the "rapid" durability tests used for OPC concrete (i.e., tests lasting 16 days to 2 years) historically speaking have been calibrated and corroborated using long-term performance data from various sites (e.g., CANMET Exposure Site in Ottawa, Canada; Treat Island, Maine, USA; PCA Skokie and Sacramento sites, USA; BRE Long-Term Exposure sites, UK). Similar results will be required for ACM-based concrete; some are already underway. Gathering this data is a relatively low cost exercise, but significant time investments are required.

IV. CONCLUSION

Many of the existing test methods for both binder materials and concrete will have to be adapted for evaluation of ACMs. In some cases, the actual test method is completely applicable to both OPCs and ACMS once a broader definition of mixing or curing standards is added, but in other cases a complete overhaul of the standard will be necessary for ACMs to be properly tested. In any case, this will require engagement by standards committees. In addition, ACM producers need to start long-term testing and to engage academic researchers to test relevant structural properties in reinforced concrete.

Gaining acceptance in building codes is challenging. This will require development and documentation of long-term performance data. ACM producers should commence immediately with production of test specimens to be placed in various climates around the world, monitor those specimens over time, and document their performance.

In closing, citing commercial applications of geopolymer concretes, Provis and van Deventer [8] stated, "*it has been necessary to build confidence in geopolymer concrete from scratch in each new market, as consumers wish to see success under local conditions. Small 'low risk' projects, where the cost of replacement is low if performance is not met, must first be completed to build confidence before more complex projects are tackled.*"

REFERENCES

[1] R.D. Hooton. "Current Developments and Future Needs in Standards for Cementitious Materials," *Cem. Conc. Res.*, vol. 78 (Part A), p.165-177, 2015.

[2] J.S.J. van Deventer, Private Communication, Sept. 2015

[3] J.F. Lamond, and J. H. Pielert, "Significance of Tests and Properties of Concrete and Concrete-Making Materials," STP169D. West Conshohocken, PA, ASTM, 2006.

[4] J.R. Libby, *Modern Prestressed Concrete - Design Principles and Construction Methods*. New York, N.Y., Van Norstrand Reinhold.1977.

[5] J.G. MacGregor, *Reinforced Concrete: Mechanics and Design*. Opper Saddle River, NJ, Prentice Hall, 1997.

[6] J. Provis, and J. S. J. van Deventer. "*Alkali Activated Materials: State-of-the-Art Report,"* RILEM TC 224-AAM, Springer, Netherlands**,** 338 pp., 2014.

[7] E.A. Douglas, A. Bilodeau and V.M. Malhotra, "Properties and Durability of Alkali-Activated Slag Concrete," *ACI Matls. J.*, vol. 89, no. 5, p. 509-516, 1992.

[8] J.L. Provis and J. S. J. van Deventer, "Low Carbon Emission Geopolymer Concrete: from Research into Practice," Proc. 27th Biennial National Conf. Concrete Institute of Australia, Melbourne, Australia, 2015.

[9] A.M. Rashad, Y. Bai, P.A.M. Basheer, N.B. Milestone, and N.C. Collier, "Hydration and properties of sodium sulfate activated slag," *Cem. Conc. Comp*.vol. 37, pp. 20-29, 2013.

[10] I. Ismail, S.A. Bernal, J.L. Provis, S. Hamdan, J.S.J. van Deventer, "Microstructural Changes in Alkali Activated Fly Ash/Slag Geopolymers with sulfate Exposure," *Matls. Struct.*, vol. 46, no. 3, pp. 361-373.

[11] K. Quillin, "Performance of belite–sulfoaluminate cements," *Cem. Conc. Res.*, vol. 31, no. 9, pp. 1341-1349, 2001.

[12] P.M. Gifford and J. E. Gillott, "Alkali-silica reaction (ASR) and alkali-carbonate reaction (ACR) in activated blast furnace slag cement (ABFSC) concrete," *Cem. Conc. Res.*, vol. 26, no.1, pp. 21-26, 1996.

[13] T. Bakharev, J. G. Sanjayan and Y. B. Cheng, "Resistance of alkali-activated slag concrete to alkali–aggregate reaction," *Cem. Conc. Res.*, vol. 31, no. 2, pp. 331-334, 2001.

[14] R.R. Lloyd, J. L. Provis and J. S. J. van Deventer, "Pore solution composition and alkali diffusion in inorganic polymer cement," *Cem. Conc. Res.*, vol. 40, no. 9, pp. 1386-1392,2010.