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STANDARD AGGREGATE MATERIALS FOR ALKALI–SILICA REACTION STUDIES

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ABSTRACT

Preliminary studies were carried out to identify candidate materials that may be used in place of Pyrex glass as a standard reactive aggregate in alkali-silica investigations. The various candidate materials were tested for expansion in mortars prepared using either a high-alkali or a low-alkali cement, a nonreactive limestone sand, and some reactive material. The reactive aggregates studied included several commercial glasses, an opal, and a calcined flint. The proportion of limestone replaced by each reactive material was varied so as to bracket the pessimum level of each material. Mortar-bar expansion levels were measured over periods of approximately 6 months to 2 years. Based on these studies, four of the materials are identified as potential candidates as standard reactive materials in mortar-bar expansion studies: Vycor, fused quartz, fused silica, and calcined flint; the calcined flint appears especially promising.

1. INTRODUCTION

It is often necessary to measure expansion due to alkali-silica reaction (ASR) using a standard reactive aggregate. A common standard material is Pyrex glass[1], which is utilized in ASTM C 441-81, Standard Test Method for Effectiveness of Mineral Admixtures in Preventing Excessive Expansion of Concrete Due to the Alkali-Aggregate Reaction. However, the performance of Pyrex glass as a standard reactive material has not always been satisfactory, as discussed below, so studies were carried out at the National Institute of Standards and Technology[2] (NIST) to identify candidate materials to replace Pyrex glass as a standard reactive aggregate in alkali-silica investigations. These studies are the subject of the present report and are covered in greater detail separately [1].

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[1] Certain trade names and company products are identified to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products are necessarily the best available for the purpose.

[2] Formerly the National Bureau of Standards.

To serve as a standard reactive aggregate, certain criteria should be met. The material should produce a high level of expansion in mortar bars, at least of the order of 0.1 percent expansion when used with a cement containing approximately 1.0 percent total alkalies as equivalent sodium oxide (Na_2O). It should produce this expansion rapidly (e.g., within 14 days at 37.8°C) to allow accelerated testing. If the expansion shows a pessimum relationship to the proportion of aggregate that is reactive, the pessimum relationship must be reasonably flat, so that expansion results are not highly sensitive to small changes in the ratio of alkali to reactive aggregate. Expansion must be reduced considerably when measured using low-alkali cement and pozzolanic materials to meet the objectives of ASTM C 441. Finally, the material must be homogeneous within each batch and uniform from batch-to-batch to assure reproducible expansion data.

Pyrex glass has been used as a standard reactive material in measuring expansion due to ASR since 1947. It was specified as a reactive material for mortar-bar expansion tests in the Bureau of Reclamation Specification No. 1904 in 1947 covering the performance of calcined reactive siliceous material for the Davis Dam. Stanton in 1950 [2] discussed the use of Pyrex glass sand and presented expansion results.

Notwithstanding its long history, Pyrex is not satisfactory as a standard reactive material. Stark [3] reported that two samples of Pyrex glass produced variable flow levels in an osmotic cell and variable mortar bar expansion results, and Barneyback [4] noted that Pyrex may produce variable expansion levels, perhaps due to the different cooling rate of the constituents in a lump cullet[3]. The sodium content of Pyrex, approximately 4 percent Na_2O [5], probably also makes it unsuitable. In a recent study of ASR mechanisms, Struble [6] concluded that expansion appears to be a function of i) the pore solution alkalinity, which affects the extent of dissolution of aggregate, and ii) by the proportions of sodium and potassium ions in solution, which affect composition and swelling behavior of the reaction product. Based on this understanding, one would expect considerable differences in expansion behavior between a material releasing appreciable quantities of alkali as it dissolves, such as Pyrex glass, and a material releasing little or no alkali, such as most naturally occurring reactive aggregates. Figg [7] similarly concluded that Pyrex is not appropriate because it will provide additional alkali as it dissolves. He reported that glasses containing certain proportions of alkali and silica can produce expansion even with low-alkali cements. Thus Pyrex does not appear to be satisfactory as a standard reactive material because the expansion it produces is not always reproducible and may be affected by its alkali content.

A few other materials have been proposed as standard reactive materials in ASR studies and were included in the present study. One is Beltane opal, proposed by Diamond and Barneyback in 1976 [8]. Another is fused silica, proposed as a standard reactive material by Figg [7] and Swamy and Al-Asali [9]. More recently, calcined flint was proposed as a standard reactive aggregate by Lumley and Kollek [10].

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[3] Lump cullet is a term that is applied to waste glass from the manufacturing process, either to the residual melt left during a batch process or to broken pieces of finished glass, typically rods and tubes, that are added to the melt.

2. EXPERIMENTAL PROCEDURE

2.1 Materials

The materials used in this study, including their sources, are described in greater detail in the separate report [1]. Two Type I cements were used, one high-alkali and the other low-alkali[4]. The high-alkali cement contained 0.28 percent Na_2O and 1.02 percent K_2O, and the low-alkali cement contained 0.20 percent Na_2O and 0.31 percent K_2O (expressed as percent by mass). The following reactive materials were used in combination with limestone sand: Pyrex glass, a sodium borosilicate glass obtained as a lump cullet; Vycor glass, a high-silica glass manufactured by removing the non-silica constituents of a borosilicate glass by a chemical leaching process, then heat-treating to reduce porosity, obtained as broken tubing with a typical wall thickness of 1 mm; fused quartz glass, produced by melting material from a high-purity quartz deposit, obtained also in the form of broken tubing; fused silica, a high-purity optical glass produced by flame hydrolysis of $SiCl_4$; calcined flint, made by acid-washing a commercial flint, then firing it at 1375°C for 6 hours, obtained in the form of particles 1 mm to 2 mm in size; and Beltane opal, a hydrothermally-altered rhyolite collected by Barneyback from an abandoned mine [4].

2.2 Mortar Bar Expansion Tests

The aggregate materials were crushed as necessary, sieved, and reconstituted to obtain the grading specified in ASTM C 441. In mixes containing reactive material, limestone was replaced by the desired proportion (by mass) of the reactive constituent, and the proportion of limestone replaced was varied so as to bracket the pessimum level of each reactive material. Except for the calcined flint (which had too fine a particle size distribution), the reactive material replaced limestone in each size fraction; for the calcined flint, no replacement could be made in the two coarser fractions, so a correspondingly larger amount of its coarsest fraction (1.18 mm to 0.60 mm) was used.

With the following exceptions, the mortar bars were tested according to ASTM C 227-87, Standard Test Method for Potential Alkali Reactivity of Cement-Aggregate Combinations (Mortar-Bar Method). A single water-to-cement ratio of 0.485 was used. One batch of each mortar was prepared to provide two specimens of each mixture. Length changes were measured periodically (using a length comparator) until expansion appeared to be complete.

3. RESULTS

Results are described in greater detail in the separate report [1]. For all reactive materials, expansion began immediately and increased most rapidly during the initial period. After a few weeks, the length gradually leveled off, such that the expansions at 2 months were usually as high or nearly as high as the final levels, reached after durations of 6 months to 2 years. The final expansion levels with the high-alkali cement varied considerably for the reactive materials studied. The calcined flint and the Beltane opal produced moderate expansion levels, as high as 0.3 percent.

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[4] The designations Type I and low-alkali cement are described in ASTM C 150.

The manufactured glasses (Vycor, fused quartz, and fused silica) produced high expansion levels, nearly 1.0 percent, which were greater than the approximately 0.7 percent expansion produced by Pyrex.

Although these studies did not include enough replacement levels to determine precisely the pessimum proportion for each material, the results provide an indication of the pessimum level. All the manufactured glasses appear to have a high pessimum proportion, in the range 50 percent to 100 percent. The calcined flint shows an intermediate pessimum level, approximately 30 percent. The manufactured glasses and the calcined flint also show a flat pessimum relationship, such that a large variation in the proportion of reactive material (e.g., 10 percent) causes little variation in expansion. Beltane opal, on the other hand, shows a very low pessimum proportion at approximately 2 percent, as was reported previously [6]. The Beltane opal also shows an extremely sharp pessimum relationship, such that even small variations in the proportion of reactive material (e.g., 4 percent) cause much variation in expansion.

Using a low-alkali cement reduced expansion, though not to the same extent for the various reactive materials. With Pyrex, the expansion level using the low-alkali cement was 4 percent of the level using the high-alkali cement. This was not the expected behavior; following the reasoning of Figg [7], we expected that the sodium ions provided by Pyrex would make it relatively insensitive to the alkali level in the cement. Similarly, the expansion level with calcined flint and low-alkali cement was extremely low relative to the level with the high-alkali cement. With Vycor, fused quartz, and fused silica, the effect was not so great; the expansions using the low-alkali cement were approximately 30 percent of the expansions using the high-alkali cement.

In assessing the suitability of each candidate reactive material, we should consider the expansion, the sensitivity of expansion to proportion of aggregate, the extent to which expansion is reduced using a low-alkali cement, and the expected reproducibility. As discussed earlier, Pyrex is not a good candidate for two principal reasons, its lack of reproducibility and its high sodium content. All five candidate replacement materials produced a high final expansion and a high initial rate of expansion. The opal is not a good candidate because its pessimum proportion is very low and its pessimum relationship very sharp. A sharp pessimum relationship is undesirable because the material would have to be tested at many replacement levels to define fully the relationship between expansion and proportion of reactive material for a specific cement. Calcined flint produced high expansion with high-alkali cement and very low expansions with low-alkali cement, whereas the three manufactured glasses, Vycor, fused quartz, and fused silica, produced moderate expansion with low-alkali cement, and thus appear somewhat less suitable than calcined flint as standard reactive materials. Therefore, while additional expansion tests are necessary before a material can be proposed as a standard, these results indicate that calcined flint, Vycor, fused quartz, and fused silica have potential as standard reactive materials for ASR studies.

4. CONCLUSIONS

Because of its reported poor reproducibility and the expected influence of its alkali content, Pyrex glass is not satisfactory as a standard reactive material in investigations of alkali-silica reaction.

Expansion tests using possible alternatives showed the following: with high-alkali cement, calcined flint produced moderate expansions and a rapid early rate of expansion, and with low-alkali cement it produced very little expansion; with high-alkali cement, Vycor, fused quartz, and fused silica produced high expansions and high early rates of expansion, but with low-alkali cement they produced moderate expansions. Based on these results, it is concluded that calcined flint, Vycor, fused quartz, and fused silica have good potential, and that calcined flint offers the best potential as a standard reactive material in alkali-silica reaction investigations. However, additional tests are required to determine reproducibility of the candidate materials before a final recommendation can be made.

5. ACKNOWLEDGEMENTS

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