Evaluating Expansion Potential of CCBs

David J. Hassett¹

¹ University of North Dakota, Energy & Environmental Research Center, 15 North 23rd Street, Grand Forks, ND 58203

KEYWORDS: expansion, swell, ettringite formation, CCBs

ABSTRACT

An evaluation of American Society for Testing and Materials (ASTM) methods used to detect swelling and expansion of materials was conducted by the Energy & Environmental Research Center (EERC). It was concluded that these test methods are generally based on similar protocols for a wide variety of materials. Further, no ASTM methods were specific for coal combustion by-products (CCBs), nor do the existing and modified methods provide quality-assured data on swell potential for CCBs. Several expansion mechanisms have been proposed for reactive or high-calcium CCBs. Reactive CCBs include moderate- to high-calcium fly ash, fly ash–dry flue gas desulfurization mixtures, and fluidized-bed fly ash. The proposed expansion mechanisms associated with soils, so the application of expansion tests designed for soils has the potential to provide inaccurate information.

The EERC has initiated development of a test to predict expansion potential for CCBs. The test under development is based on material density. Densities of material suspected of having swell potential are determined using ASTM C188-95, Standard Test Method for Density of Hydraulic Cement. The sample of interest is tested for density using C188, and then the same material is hydrated, dried at 50°C, and retested. Reduction in density can be related to swell potential based on proposed expansion mechanisms.

INTRODUCTION

The determination of potential for expansion in coal combustion by-products (CCBs) can be problematic using laboratory expansion tests. Experience has shown that the expansive hydration reactions often seen in alkaline ash samples, such as the formation of ettringite, do not always show dimensional changes in laboratory test specimens prepared for expansion tests. This is despite the fact that expansive reactions are occurring. The reason for this is that crystals of ettringite which form in the test specimens do not always press directly on neighboring particles. If the ettringite crystals grow into the void spaces, expansion will not be evident in the laboratory specimens. The hypothesis for the test development is that if mineralogical and compositional changes in a material such as fly ash result in expansion, the mass must also experience a respective negative change in bulk density. The determination of bulk

density of a mass of ash before and after hydration should then afford a rather simple and reliable means of determining potential for expansion.

EXPERIMENTAL

Eighteen different fly ash samples ranging in composition and pH were selected for inclusion into our study on expansion. Densities of samples were determined using American Society for Testing and Materials (ASTM) C188-95, Standard Test Method for Density of Hydraulic Cement, also commonly used for fly ash. This test utilizes LeChatelier's principle of displacement of a liquid to determine bulk density of a finely powdered material. In this test, kerosene was the liquid used, and commercially available LeChatelier bottles were used. Densities were determined on dry samples and following hydration. All determinations were done in duplicate.

Hydration was carried out by placing 150 grams of dry ash into a bottle with approximately 200 mL of distilled deionized water. The bottles were mixed using endover-end rotation at 30 rpm. Rotation was continuous for a week and then intermittent for the remainder of the hydration time. For intermittent rotation, we used a microprocessor-based on-off timer that turned the rotator on for 15 minutes every 2 hours. We assumed that the likelihood for the material to form a monolith had passed after 1 week of rotation and that intermittent rotation was adequate to prevent the solids from caking into the bottom of the bottles. For most of the samples, a 30-day hydration period was used. At the end of the hydration period, the ash was filtered through course filter paper in a Buchner funnel and washed with distilled deionized water. Air was drawn through the ash to remove excess water. The wet ash was placed in an evaporating dish and placed into an oven at 50°C. Drying at 50°C allows for the free water to be driven off of the hydrated ash without affecting the crystalline structure of any ettringite that may have formed. Heating was continued until a constant weight was achieved. At that time, the bulk density of the hydrated ash was determined using ASTM C188.

RESULTS AND DISCUSSION

ASTM reports that using this test, the standard deviation for portland cements using C188 has been found to be 0.012. Therefore, the results of two properly conducted tests by the same operator should not differ by more than 0.03.¹ It can be seen later in Table 1 that a difference of 0.03 results in a calculated swell of approximately 1%, depending on the density of the unhydrated material. It was for this reason it was decided that results at or below 2% had little meaning.

The density results of the 18 dry and hydrated ash samples are shown in Table 1. Results of the change in density along with pH values are also shown. These samples have been sorted by the difference, or percent change, in density. A negative change in density indicates a potential for swell. Samples with percent changes in density of less than 2% after 30 days of hydration were considered negligible. Using this assumption, only 7 of the 18 samples tested would be considered to have a swell potential.

Table 1. Owen't otential after 50 Days of Hydration and Additional Oample mormation								
Ash		Dry	Hydrated					
Sample	Ash Type	Density ¹	Density	Difference	% Change	рН		
01-008	Subbituminous	2.74	2.27	-0.47	-17.2	11.89		
00-048	Lignite + FGD ²	2.46	2.11	-0.35	-14.2	12.18		
00-052	Lignite	2.55	2.29	-0.26	-10.2	12.35		
99-189	PRB ³	2.41	2.17	-0.24	-10.0	11.02		
99-188	PRB + FGD	2.39	2.19	-0.20	-8.4	12.34		
00-050	PRB	2.56	2.35	-0.21	-8.0	11.98		
99-456	Subbituminous	2.44	2.36	-0.08	-3.3	12.55		
99-187	Bituminous	2.44	2.41	-0.03	-1.2	12.21		
99-185	Bituminous	2.28	2.26	-0.02	-0.9	12.65		
02-042	Lignite	2.47	2.45	-0.02	-0.8	11.94		
01-001	Bituminous	2.05	2.04	-0.01	-0.5	12.63		
01-003	Bituminous	2.21	2.21	0.00	0.0	10.95		
99-726	Bituminous	2.25	2.25	0.00	0.0	8.38		
00-046	Lignite	2.52	2.525	0.00	0.2	11.74		
02-012	E. Bituminous	2.19	2.2	0.01	0.5	5.00		
99-191	Bituminous	2.46	2.48	0.02	0.8	10.08		
01-002	E. Bituminous	2.19	2.23	0.04	1.8	4.03		
99-693	Bituminous	2.11	2.17	0.06	2.8	11.54		

Table 1 Swell Potential after 30 Days of Hydration and Additional Sample Information

¹ Densities in g/cm³.
² Flue gas desulfurization.
³ Powder River Basin.

Since it was not known if 30 days was a reasonable equilibration time for ash hydration reactions to occur, a timed hydration series was undertaken using an ash sample known to have a high swell potential because of a relatively large change in density at 30 days. The results of these tests carried out to 13 weeks on Sample 00-052 are shown in Table 2 and also graphically in Figure 1.

Table 2. Results of the Timed Hydration Series

Hydration Time	Sample	Dry Density ¹	Hydrated Density	Difference	% Change					
2 weeks	00-052	2.55	2.31	-0.24	-9.4					
4 weeks	00-052	2.55	2.25	-0.30	-11.8					
8 weeks	00-052	2.55	2.21	-0.34	-13.3					
13 weeks	00-052	2.55	2.10	-0.45	-17.6					

¹ Densities in g/cm^3 .



Figure 1. Timed expansion series.

As can be seen in Figure 1, 13 weeks appears to be an insufficient time for this sample to completely hydrate. A linear least squares fit line is shown through the four experimental points. The equation of the line, as well as the R² value, is shown in the figure. Although it is impossible to be certain, the hydration and associated changes in density appear to be progressing linearly up to 14 weeks. Additional experiments are planned with this sample and others to determine what an adequate hydration time may be. This is certain to be different for various CCBs but grossly underestimating swell potential can result in damage and associated high costs. If more hydration time had been used for this series of samples, it is likely that more than seven of the samples would have shown significant swell potential.

CONCLUSIONS

It appears that changes in density can be used to predict potential for expansion in dry powdered materials such as CCBs. The determination of density in hydrated and unhydrated samples requires only the use of inexpensive LeChatelier bottles, kerosene, a temperature-controlled laboratory oven, and an adequate analytical balance. Although inter- and intralaboratory testing will be required to validate this as a method and considerably more work is required, the groundwork has been laid for this to become a standard test in the near future.

ACKNOWLEDGMENTS

This paper was prepared with the support of U.S. Department of Energy (DOE) under Cooperative Agreement No. DE-FC26-98FT40321. However, any opinions, findings,

conclusions, or recommendations expressed herein are those of the author(s) and do not necessarily reflect the views of DOE.

The authors also wish to acknowledge support provided by the Coal Ash Resources Research Consortium[™], with funding from Alliant Energy; Ash Resources; Great River Energy; Ish Inc.; LaFarge N.A.; Otter Tail Power Company; Tennessee Valley Authority; and Xcel Energy.

Robert Ducioame, Linnea Schluessler, and Amy Gieske, University of North Dakota students, are also gratefully acknowledged for performing much of the laboratory work presented here.

REFERENCES

[1] ASTM C 188-95, Standard Test Method for Density of Hydraulic Cement, Section 4, Volume 04.01, 2002, pp. 181-182.