

Production and Performance of Laboratory Produced High-Volume Fly Ash Blended Cements in Concrete

by

N. Bouzoubaâ* and V. M. Malhotra**

ABSTRACT

This paper reports the development at CANMET of high-volume fly ash blended cements, and their performance in concrete. The blended cements are made by intergrinding approximately 55% of fly ash and 45% of ASTM Type I or Type III cement clinker together with small amounts of gypsum and a dry superplasticizer. The concrete made with the HVFA blended cements has adequate early-age and excellent later-age mechanical properties, and demonstrates satisfactory performance in durability aspects such as resistance to freezing and thawing cycling, and chloride-ion penetration.

Keywords: bleeding, blended cement, compressive strength, durability, fly ash, grinding, modulus of elasticity, scaling, setting, shrinkage, superplasticizer, tensile strength.

* Research Scientist and ** Scientist Emeritus, International Centre for Sustainable Development of Cement and Concrete (ICON), CANMET, Natural Resources Canada, Ottawa, Canada.

INTRODUCTION

The production of portland cement contributes significantly to the CO₂ emissions into the atmosphere. For every tonne of portland cement produced, approximately one tonne of CO₂ is released into the atmosphere. It is imperative, therefore, that technologies be developed to reduce the production of portland cement clinker in rotary kilns while maintaining the target production of cement to meet the demand of the construction industry.

In the early 1980s, CANMET developed high-volume fly ash (HVFA) concrete, in which 55-60% of the portland cement is replaced by a low calcium fly ash, a by-product of thermal power plants. This type of concrete has demonstrated excellent mechanical properties and long-term durability, and is slowly getting acceptance worldwide [1-8]. However, at present fly ash has to be added at a ready-mixed concrete batch plant to produce high-volume fly ash concrete; this necessitates additional storage silos and quality control at ready-mixed concrete plants. In order to resolve these issues, CANMET in collaboration with Environment Canada, and later in partnership with Electric Power Research Institute, U.S.A., undertook major projects in the mid 1990s to develop a blended cement incorporating high volumes of ASTM Class F fly ash. The blended cement is made by intergrinding approximately 55% of a low-calcium fly ash, and 45% of ASTM Type I or Type III cement clinker together with small amounts of gypsum and a dry superplasticizer [9-16].

This paper reports the development of the HVFA blended cements at CANMET, and discusses their performance in concrete.

MATERIALS

Most investigations on the development of HVFA blended cements at CANMET were carried out using ASTM Type I and Type III portland cement clinkers. Several ASTM Class F fly ashes from various sources in Canada and the U.S.A., having different chemical compositions and physical properties, were used. A sulphonated, naphthalene formaldehyde condensate superplasticizer in a dry powder form was incorporated in some of the blended cements.

The physical properties and chemical analysis of the clinkers and some of the fly ashes used are shown in Table 1.

PRODUCTION OF THE HVFA BLENDED CEMENTS AT CANMET

To produce HVFA blended cements that meet the requirements of ASTM C 1157M, two procedures were used at CANMET. The first procedure involved two stages; these consisted of producing a cement with or without a superplasticizer to a predetermined Blaine fineness, and then intergrinding 45% of the above cement with 55% of a fly ash for a predetermined time. This procedure has the advantage of starting with a fixed Blaine fineness of the cement thus ensuring that mortars or concrete made with the blended cements have a certain minimum compressive strength.

The intergrinding time of the fly ash with the cement can range from 20 to 80 minutes depending

on the fineness and the type of the fly ash used. However, there is an optimum duration of intergrinding the fly ashes with the cements. The results show that, the intergrinding up to the optimum time decreased the water requirement, and increased the strength activity indices of the blended cements [16]. Beyond the optimum grinding time, the water requirement increased, and the strength activity indices either decreased or did not increase significantly.

The incorporation of a superplasticizer in the HVFA blended cement helped the mortars made with the HVFA blended cement to achieve the compressive strength required by ASTM C 1157M standard as a result of the reduction in the water-to-blended cements ratio. However, the use of a superplasticizer in HVFA blended cements did retard their setting times.

The second procedure consisted of producing a blended cement by grinding together the cement clinker, gypsum, and fly ash for a predetermined time. This procedure was adopted because for some fly ashes, the use of the first procedure did not result in the production of blended cement that meets the requirements of ASTM C 1157M.

A ceramic grinding mill, 420 mm in length and 500 mm in diameter with a grinding capacity of approximately 10 kg material, was used for the production of the cements. The weight ratio of the materials to be ground to the grinding media was 1:7. Before the clinker was fed to the grinding mill, it was crushed so that all the particles were less than 0.6 mm.

MIXTURE PROPORTIONS

The mixture proportions of the concrete made with HVFA blended cements were the same as the typical mixture proportions of the HVFA concrete developed at CANMET [17]. These mixture proportions were optimized to produce a high-performance, air-entrained concrete both for mass and structural concrete applications. The typical mixture proportions are shown in Table 2; for example, 370 kg of blended cement per 1 m³ of concrete is used with a water-to-blended cement ratio of 0.32. As the HVFA blended cements meet the requirements of ASTM C 1157M, the blended cements could be used without any restrictions on the mixture proportions of concrete.

PROPERTIES OF FRESH CONCRETE MADE WITH THE HVFA BLENDED CEMENTS

Slump, air content and dosage of admixtures

The dosage of the superplasticizer used for concrete made with the HVFA blended cements to reach a certain slump varies considerably with the type of the fly ash used in the blended cement, and whether the superplasticizer is incorporated in the blended cement or not. In general, the concrete made with blended cements incorporating a superplasticizer requires higher dosage of a superplasticizer than that made without a superplasticizer. For example, the concrete made with the HVFA blended cements may require a dosage of superplasticizer of 3 to 6 L/m³ of concrete to achieve a slump of 100 mm.

The entrained air content of the concrete made with the HVFA blended cements is usually kept between 5 and 7%. It has been found that this percentage of entrained air usually results in

satisfactory bubble-spacing factor in the hardened concrete. The dosage of the AEA required for obtaining an air content in the range mentioned above is also strongly influenced by the type of the fly ash used in the blended cement, and whether the blended cement is incorporating the superplasticizer or not. The concrete made with the blended cements incorporating a superplasticizer usually requires a lower dosage of the AEA than that made with the blended cements without the superplasticizer.

Bleeding, setting time and autogenous temperature rise

The bleeding of the concrete made with the HVFA blended cements ranges from being very low to negligible due to the very low water content in the concrete.

The setting time of the concrete made with this type of cement is generally longer than that of concrete made with portland cement only. This is expected considering the high fly ash content in the blended cement, and the large amount of the superplasticizer used. However, in general, the concrete made with the HVFA blended cements does not show unacceptable retardation in setting time, and demonstrates enough strength development to result in adequate strength at one day (Table 3).

The maximum autogenous temperature rise in concrete made with the HVFA blended cements is much lower than that of the concrete made with a portland cement only, due to the low cement content in the HVFA blended cements. In a 150 X 300 mm cylinder, the maximum autogenous temperature rise in concrete made with the HVFA blended cements was 16.7 °C, and the corresponding temperature rise in concrete made with portland cement only was 29.9 °C. This demonstrates that the HVFA blended cement is ideally suited for mass concrete.

MECHANICAL PROPERTIES OF CONCRETE MADE WITH THE HVFA BLENDED CEMENTS

The mechanical properties of the concrete made with the HVFA blended cements depend primarily on the type of fly ash used in the blended cements. In general, these properties are excellent due to the dense microstructure, and the low water content of the concrete made with this type of cement. Typical mechanical properties of concrete made with the HVFA blended cements are given in Table 3.

Compressive strength

Compressive strengths of 10, 27, 40, and 53 MPa at 1, 7, 28, and 91 days, respectively were obtained with concrete made with the HVFA blended cements without the use of the superplasticizer. The concrete made with the HVFA blended cements incorporating the superplasticizer developed lower compressive strength at early ages (5 MPa at 1 day), probably due to the high dosage of the superplasticizer used for the concrete made with such blended cements. However, by increasing the intergrinding time of the fly ash with the cement, a compressive strength in the order of 16 MPa was obtained for concrete made with some HVFA blended cements incorporating the superplasticizer. In general, the compressive strength at 1, 7, 28, and 91 days of the concrete made with the HVFA blended cements represents 45%, 85%, 100%, and 125% of the compressive strength of the concrete made with normal portland cement, respectively.

Flexural- and splitting-tensile strengths

Flexural strength in the order of 4.8 and 5.7 MPa can be obtained at 14 and 28 days, respectively, for the concrete made with the HVFA blended cements. The 28-day splitting-tensile strength of the concrete made with the blended cements is in the order of 3.5 MPa.

Young's modulus of elasticity

The Young's modulus of elasticity of concrete made with the HVFA blended cements is in the order of 36 and 40 GPa at 28 and 91 days, respectively. The high modulus achieved is probably due to the fact that a considerable portion of the unreacted fly ash, consisting of glassy spherical particles, acts as a fine aggregate, and there is a strong interfacial bond between the paste and the aggregates.

Drying shrinkage

The drying shrinkage strains of the concrete made with the HVFA blended cement are comparable to, or lower than that of the concrete made with portland cement only, with measured values in the order of 500×10^{-6} after 32 weeks of exposure to dry air.

DURABILITY OF CONCRETE MADE WITH THE HVFA BLENDED CEMENTS

Several laboratory investigations involving fly ashes from various sources in Canada and the U.S.A. have demonstrated excellent durability of concrete made with the HVFA blended cements. Some typical durability test results are given in Table 4.

Resistance to chloride-ion penetration

Concrete made with the HVFA blended cements showed higher resistance to the chloride-ion penetration than the conventional portland cement concrete of similar strength in the tests performed according to ASTM C 1202. For example, the 28-day value of the total charge passed in coulombs, a measure of the resistance of concrete to the chloride-ion penetration, ranges from 300 to 2000 for the concrete made with the HVFA blended cements, versus 2500 to 6000 coulombs for the control portland cement concrete. A value of less than 1000 coulombs is indicative of concrete with very low permeability.

Resistance to freezing and thawing cycling

Concrete made with the HVFA blended cements shows durability factors of >100 after 300 cycles of freezing and thawing performed according to ASTM C 666 Procedure A. As in conventional concrete, the freezing and thawing resistance of the concrete made with the HVFA blended cements is a function of its air-void parameters. For these concretes, the spacing factor ranged from 0.200 to 0.250 mm, and is considered satisfactory for the resistance of the concrete to freezing and thawing cycling [9].

De-icing salt-scaling resistance

The concrete made with the HVFA blended cements performs poorly in de-icing salt scaling laboratory test in accordance with ASTM C 672. Regardless of the type of fly ash used in the production of the blended cements the concrete exhibits severe scaling with a visual rating of 5 according to ASTM C 672 [9, 10, 15]. However, the experimental HVFA concretes sidewalks in

eastern Canada have shown satisfactory performance after four winters during which period the sidewalks have gone through more than 400 cycles of freezing and thawing combined with numerous applications of de-icing salts (unpublished data). It is believed that ASTM C 672 is a very severe test, and is not satisfactory for determining the de-icing salt scaling resistance of HVFA concrete in actual field applications. Further research is needed in this area.

ECONOMIC CONSIDERATION

The cost of the blended cements discussed above will depend on the cost of the cement clinker, fly ash and the energy required for grinding. No detailed data have been developed on this issue but it is believed that the cost of the blended cements should be lower than the cost of normal portland cement.

CONCLUDING REMARKS

The high-volume fly ash blended cements can be produced with cement clinkers and fly ashes having a wide range of chemical compositions and physical properties. Most of the blended cements so produced can meet the ASTM requirement for blended cements if appropriate grinding procedure is used. The concrete made with the HVFA blended cements has adequate early-age and excellent later-age mechanical properties, and demonstrates remarkable performance in durability aspects such as resistance to freezing and thawing cycling, and chloride-ion penetration. Because of the low cement content, the temperature rise in concrete made with the HVFA blended cements is rather low. Thus, high-volume fly ash blended cements are ideal for concrete structures where high heat of hydration is a concern.

REFERENCES

1. Langley, W.S., Carette, G.G. and Malhotra, V.M. "Structural Concrete Incorporating High Volumes of ASTM Class F Fly Ash" *ACI Materials Journal*, Vol. 86, No. 5, 1989, pp. 507-514.
2. Sivasundaram, V., Carette, G.G. and Malhotra, V.M. "Properties of Concrete Incorporating Low Quantity of Cement and High Volumes of Low Calcium Fly Ash" *ACI Special Publication SP-114*, Vol. 1, 1989, pp. 45-71 (ed.: V.M. Malhotra).
3. Langley, W.S., Carette, G.G. and Malhotra, V.M. "Strength Development and Temperature Rise in Large Concrete Blocks Containing High Volumes of Low-Calcium (ASTM Class F) Fly Ash" *MSL Division Report MSL 90-24 (OP&J)*, Energy, Mines and Resources Canada, Ottawa, Canada, 1990, 24 pp.
4. Sivasundaram, V., Carette, G.G. and Malhotra, V.M. "Mechanical Properties, Creep, and Resistance to Diffusion of Chloride Ions of Concretes Incorporating High Volumes of ASTM Class F Fly Ashes from Seven Different Sources" *ACI Materials Journal*, Vol. 88, No. 4, 1991, pp. 407-416.
5. Bisailon, A., Rivest, M. and Malhotra, V.M. "Performance of High Volume Fly Ash Concrete

in Large Experimental Monoliths" MSL Division Report MSL 92-24 (OP&J), Energy, Mines and Resources Canada, Ottawa, 1992, 37 pp.

6. Bilodeau, A. and Malhotra, V.M. "Concrete Incorporating High Volumes of ASTM Class F Fly Ashes: Mechanical Properties and Resistance to De-Icing Salt Scaling and to Chloride-Ion Penetration" ACI Special Publication SP-132, Vol. 1, 1992, pp. 319-349 (ed.: V.M. Malhotra).
7. Malhotra, V.M. "CANMET Investigations Dealing with High-Volume Fly Ash Concrete" Advances in Concrete Technology, Second Edition, CANMET, Natural Resources Canada Publication, Ottawa, 1994.
8. Bilodeau, A., Sivasundaram, V., Painter, K.E., and Malhotra, V.M. "Durability of Concrete Incorporating High Volumes of Fly Ash from Sources in the U.S." ACI Materials Journal, Vol. 91, No. 1, January-February 1994, pp. 3-12.
9. Bouzoubaâ, N., Zhang, M.H., and Malhotra, V.M. "High-Volume Fly Ash Blended Cement: Status Report", EPRI Report TE-114025, Electric Power Research Institute, Palo Alto, California, USA, October 1999.
10. Bouzoubaâ, N., Zhang, M.H., and Malhotra, V.M. "Mechanical Properties and Durability of Concrete made with High-Volume Fly Ash Blended Cement Using a Coarse Fly Ash", CANMET Report MTL 99-19 (J), Natural Resources Canada, Ottawa, July 1999, 25 pp. (submitted to Cement and Concrete Research).
11. Bouzoubaâ, N., Zhang, M.H., and Malhotra, V.M. "Laboratory Produced High-Volume Fly Ash Blended Cements: Compressive Strength and Resistance to The Chloride-Ion Penetration of Concrete", CANMET Report MTL 99-16 (J), Natural Resources Canada, Ottawa, May 1999, 19 pp. (submitted to Cement and Concrete Research).
12. Bouzoubaâ, N., Zhang, M.H., and Malhotra, V.M. "Optimization of the Grinding Process in the Production of High-Volume Fly Ash Blended cements", CANMET Report MTL 99-10 (TR), Natural Resources Canada, Ottawa, May 1999, 34 pp.
13. Bouzoubaâ, N., Zhang, M.H., Bilodeau, A., and Malhotra, V.M. "Laboratory-Produced High-Volume Fly Ash Cements: Physical Properties and Compressive Strength of Mortars", Cement and Concrete Research, Vol. 28, No. 11, Nov. 1998, pp. 1555-1569.
14. Bouzoubaâ, N., Zhang, M.H., and Malhotra, V.M. "Effect of Grinding on Physical & Mechanical Properties of High-Volume Fly Ash Blended Cements", World Cement, Vol. 29, No. 11, Nov. 1998, pp. 96-101.
15. Bouzoubaâ, N., Zhang, M.H., Bilodeau, A., and Malhotra, V.M. "Mechanical Properties and Durability of Concrete Made with High-Volume Fly Ash Blended

Cements", ACI Special Publication SP-178, June 1998, pp. 575-603.

16. Bouzoubaâ, N., Zhang, M. H., Bilodeau, A., and Malhotra, V. M., "Effect of the Grinding on the Physical Properties of Fly Ashes," *Cement and Concrete Research*, Vol. 27, No. 12, Dec. 1997, pp. 1861-1874.
17. Bilodeau, A. and Malhotra, V.M. "High-Performance Concrete Incorporating Large Volumes of ASTM Class F Fly Ash" ACI Special Publication SP - 149, Nov. 1994, pp. 177-193 (ed.: V.M. Malhotra).

Table 1 - Physical properties and chemical analysis of the clinker and fly ashes used in HVFA blended cements

	Clinker		Fly ash							
	ASTM Type I	ASTM Type III	Lingan	Sundance	Forestburg	Point Tupper	Genesee	Belews Creek	Coal Creek	Monco
<u>Physical Tests</u>										
Specific gravity	3.23	3.11	2.82	2.08	1.97	2.58	1.95	2.28	2.44	2.53
Fineness										
-passing 45 μ m, %	-	-	87.4	83.6	78.6	75.2	63.5	79.8	76.1	-
-specific surface, Blaine, cm ² /g	-	-	2730	3060	2230	2270	1960	2270	2050	1880
-median particle size (μ m)	-	-	14	12.4	17.8	14.0	21.2	17.6	13.0	16.9
Water requirement, %	-	-	97.9	99.2	93.5	95.6	95.9	94.7	94.7	97.9
Pozzolanic Activity Index, %										
-7-day	-	-	84.1	94.5	82.4	79.6	81.7	77.5	85.8	85.4
-28-day	-	-	88.6	106.9	91.0	89.2	84.5	86.8	88.9	88.1
<u>Chemical Analyses, %</u>										
Silicon dioxide (SiO ₂)	22.3	21.7	36.9	52.4	56.8	42.7	62.6	56.2	50.5	45.2
Aluminum oxide (Al ₂ O ₃)	4.5	5.2	18.4	23.4	21.5	20.3	20.9	30.0	15.4	24.0
Ferric oxide (Fe ₂ O ₃)	3.4	3.0	35.1	4.7	4.9	23.7	4.5	5.1	8.8	16.2
Calcium oxide (CaO)	65.5	64.9	3.7	13.4	8.8	4.2	5.8	1.1	15.6	6.8
Magnesium oxide (MgO)	2.9	3.2	1.0	1.3	1.6	1.2	1.5	0.8	4.0	1.1
Sodium oxide (Na ₂ O)	0.4	0.3	0.8	3.6	3.9	0.9	2.5	0.3	2.5	1.2
Potassium oxide (K ₂ O)	0.8	0.8	1.6	0.6	1.1	2.6	1.7	2.2	2.0	2.1
Equivalent alkali (Na ₂ O+0.658K ₂ O)	0.9	0.8	1.9	4.0	4.6	2.6	3.6	1.8	3.8	2.6
Phosphorous oxide (P ₂ O ₅)	0.2	0.1	0.3	0.2	0.4	0.7	0.3	0.2	0.1	0.5
Titanium oxide (TiO ₂)	0.2	0.3	0.9	0.8	0.5	0.9	0.1	1.7	0.7	1.2
Sulphur trioxide (SO ₃)	<0.01	0.4	1.8	0.2	0.1	1.6	0.7	0.4	1.5	1.0
Loss on ignition	0.01	0.3	2.4	0.3	0.3	2.4	0.1	2.1	0.3	1.4

Table 2 - Typical mixture proportions for HVFA concrete

Typical mixture proportion	
HVFA blended cement (45% portland cement clinker including gypsum + 55% fly ash)	370 kg/m ³ (625 lb/yd ³)
Water	120 kg/m ³ (202 lb/yd ³)
Coarse aggregate	1195 kg/m ³ (2014 lb/yd ³)
Fine aggregate	645 kg/m ³ (1087 lb/yd ³)
Air-entraining admixture	200 mL/m ³ (153 mL/yd ³)
Superplasticizer	4.5 L/m ³ (9.2 lb/yd ³)

Table 3 - Typical mechanical properties of concrete made with the high-volume fly ash blended cements

	Concrete made with the HVFA blended cement	Conventional concrete with similar 28-d compressive strength (ASTM Type I cement)
Compressive Strength		
1 day	10 ± 2 MPa	21 MPa
7 days	27 ± 4 MPa	32 MPa
28 days	40 ± 4 MPa	38 MPa
91 days	55 ± 5 MPa	43 MPa
Flexural strength		
14 days	4.8 ± 0.8 MPa	4.9 MPa
28 days	5.7 ± 0.6 MPa	5.8 MPa
Splitting-tensile strength		
28 days	3.5 ± 0.3 MPa	3.3 MPa
Young's modulus of elasticity		
28 days	36 ± 2 GPa	34 GPa
91 days	40 ± 2 GPa	34 GPa
Drying shrinkage strain at 224 days	500 ± 70 x 10 ⁻⁶	480 x 10 ⁻⁶

1 MPa = 145 psi

Table 4 - Typical durability characteristics of concrete made with the high-volume fly ash blended cements

	Concrete made with the HVFA blended cement	Conventional concrete with similar 28-d compressive strength (ASTM Type I cement)
Freezing and thawing cycling (ASTM C 666, Procedure A) Durability factors after 300 cycles	> 100	98
Resistance to chloride-ion penetration (ASTM C 1202) 28 days 91 days 365 days	300 - 2000 coulombs 250 - 350 coulombs 250 coulombs	6000 coulombs 3500 coulombs 2000 coulombs
Deicing salt scaling resistance (ASTM C 672) Visual rating	5	1