Applications of Chemically Activated Blended Cements with Very High Proportions of Fly Ash

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1. Abstract

The construction of "concrete houses" that use Insulated Concrete Forms (ICF) is known to produce more sustainable structures with lower life-cycle costs compared to traditional light-frame construction. ICF construction can be made competitive in the housing market by developing cost-effective and appropriate blended cements and concrete mix designs. Research has been previously reported [11-16] that relates to the fundamental reaction between chemical activators and pozzolans. This paper extends that research to the field and reports on the development of blended cements and specific grades of concrete for sustainable construction. Results from laboratory testing and full-scale field-trial experiences are presented.

Properties of concrete made with activated blended cements are reported that have a binder mass proportion of 70:30 fly ash:cement and which are used to produce self-compacting concrete with slump flows of 500 – 600 mm, and 3-day and 28-day strengths that are satisfactory for normal ICF residential construction. Test results of various mixtures to examine workability, strength, durability and shrinkage are reported. The paper also summarizes results of two full-scale demonstration projects in 2005/06; in one the ICF concrete has been used to manufacture a residential duplex in Calgary.

2. Introduction

The use of Insulated Concrete Forms in residential construction is one way to assist the construction industry to move towards more sustainable practices, as the emphasis on sustainability accelerates worldwide. In Canada and elsewhere in North America – in both hot and cold climates – ICF construction¹ is gaining popularity. ICF-manufactured concrete homes result in a substantial reduction in life-cycle costs. In one study of 28 sets of ICF vs. wood-frame homes [1] throughout North America, the expected heating energy savings for ICF were 36 to 53% (with 95% confidence). Corresponding cooling-energy savings for houses with air-conditioning in summer were 15 to 48% (95% confidence). Homeowners surveyed liked their ICF homes because (in

¹ An ICF concrete wall consists of two layers of expanded polystyrene panels on the inside and outside of the wall. These act as the forms when the fresh concrete is poured into the cavity between the panels. After the concrete hardens, the "styrofoam " panels remain in place and act as insulation As well, the panels provide an enhanced curing environment for the concrete, prevent rapid drying, and protect the concrete from exposure to the elements during the life of the structure.

order of importance): (1) they were comfortable (e.g. no drafts); (2) they were quiet; (3) they were energy efficient; and (4) they were solid and strong.

On the other hand, there is a major deterrent to the accelerated use of ICF construction for mainstream house construction. The initial capital cost of construction using ICF is higher than that for conventional construction (plywood formed concrete basement and wood-framed superstructure). In one study [2] it was estimated that ICF construction resulted in an increased construction cost – primarily as a result of the cost of ICF forms – of 3.5% of the average selling price of a house.

In ICF construction, the ICF-block comprises about 53% of total materials costs (exterior wall, above grade). Concrete accounts for 28% of the cost, rebar 8% and miscellaneous 11%. The study reported here concentrated upon reducing the cost of construction by reducing the cost of the concrete. This was done by developing an appropriate activated blended cement in which the binder consists of 70% fly ash and 30% Portland cement. This was coupled with the design of a self-compacting concrete in which the total Portland cement content per m³ of concrete was 111 kg. This paper reports on some properties of this concrete and a full-scale field application; one component of a larger project is described which has the general objectives:

- 1. to bring the initial capital cost of ICF concrete construction down by reducing the cost of materials and construction methods.
- 2. to produce more energy-efficient and sustainable solutions for residential and light-commercial construction.
- 3. to improve the quality of construction and the resultant product and thus increase the service-life of residential structures.
- 3. Chemical Activation of High Fly-Ash Blended Cements

It is well established that if concretes containing high proportions of fly ash are cured well and for long periods the strength of these concretes is greater than that of a corresponding control mix that contains only Portland cement. When environmental exposure of concrete is minimum or nil, the main deterrent to the use of very-high fly ash contents is the slow early gain in strength of such mixes; early strengths (1-7 days) are not high enough to allow regular construction practices to proceed (e.g. form removal, backfilling). The slower concrete-strength gain is due to the lower reactivity of most pozzolans relative to Portland cement.

Low early strength can be overcome by the use of ternary blends with higherreactivity pozzolans, such as silica fume, but this defeats the objective of lowering the cost of materials. Alternatively, many researchers have reported [3-10, for example], that pozzolans of various types (natural, blast furnace slag, fly ash) can be activated by various chemicals, such as sodium hydroxide, sodium chloride, sodium sulphate, and sodium and potassium silicates. Pozzolanic reaction can also be enhanced by grinding pozzolans and by elevated curing temperatures [11] – both of which require more energy than chemical activation for the manufacture of concrete.

Analysis of the literature and test results clearly show that the efficiency of a particular chemical activator is largely dependent upon the type and chemistry of the pozzolan being activated, and upon curing conditions – especially curing temperatures [12]. The activator must be chosen to suit the particular pozzolan that is intended for use.

The current practical application reported below uses a low-calcium fly ash derived from subbituminous coal. Studies of the reaction of this type of fly ash (common in Alberta), other fly ashes, slags and natural pozzolans with various chemical activators have been reported [12-16]. These fundamental test results show that the use of sodium sulphate (per mass of binder) as a component in the activation process significantly accelerates pozzolanic reactivity and this results in improved 1- and 3-day strengths as well as higher later age (180 day) strengths. Other activators such as calcium chloride and sodium hydroxide, when used with this type of ash, have lesser influence.

Fundamental examination [16,17] indicates that sodium sulphate improves strength in this system by two processes: (1) increase in the alkalinity of pore solution, thus permitting more rapid dissolution from the surface of fly ash particles; (2) early formation of ettringite which persists for extended periods if the total sulphate content of the system remains high. Tests show [14] that if 2% or less sodium sulphate by mass of binder is used, then Afm phase, rather than Aft phase exists after 7 days of hydration. However, if sodium sulphate is greater than 2%, Aft persists throughout the 180 day observation period.

These observations imply that sodium sulphate can be an effective base for an activator that boosts early-age strengths of high fly-ash concretes -- and may also help to improve later-age strengths. For concrete in exposed environments the use of sulphate-based activators requires caution and detailed testing. Depending upon the environment and the porosity of the concrete, leaching could result in depletion of sulphate in pore solutions and subsequent decomposition of ettringite, loss of strength and increase in susceptibility to time-dependent volumetric expansion.

4. Materials and Mix Proportions.

This paper reports on progress to implement the use of a sodium-sulphatebased activator for practical concretes. The application centres on the use of a very-high fly ash content concrete for the core of ICF walls. To minimize costs of concrete placement, the concrete was designed to be selfcompacting, with a target slump-flow centering around 600 mm and a target 28 day strength between 15 and 20 MPa. Residential concrete is often required by code to have 15MPa strength at 28 days age.

Because the core of ICF walls is protected against freeze-thaw and significant moisture ingress-egress, air-entrainment was not used. After the design and testing of several different mixes [18,19] the optimum mix design for ICF walls (designated by the name "EcoA") was determined. The properties of the Canadian Standards Association (CSA) Type 10 Portland cement and Alberta fly ash used to manufacture EcoA are given in Table 1. The fine and coarse aggregates are quartzite-based with some limestone and trace amounts of sandstone/arkose, siltstone, concretions and chert. Aggregate gradations conformed to CSA limits. The aggregates used are standard for concretes that are cast in Calgary.

	Portland Cement	Fly Ash	
Calcium oxide (CaO)	61.8	11	
Silicon dioxide (SiO ₂)	20.8	54.3	
Aluminium oxide (Al ₂ O ₃)	4.5	23.7	
Ferric oxide (Fe ₂ O ₃)	3.3	3.6	
Magnesium oxide (MgO)	3.44		
Sulphur trioxide (SO ₃)	2.69	0.15	
Equivalent alkali	0.51		
Loss on ignition	1.68	0.34	
Specific gravity (g/cm ³)	3.15	2.03	
Fineness (retained on No. 325 sieve)	4.57	15.4	
Soundness (Autoclave expansion)	0.09	0.1	
The fly ash has a strength-activity index with cement (ASTM C595) of 89.1 at 7d and 109 at 28d. Water requirement of the ash is 94.5% of control.			

Table 1. Chemical and Physical Properties of Cement and Fly Ash

Mix proportions of EcoA are compared to a typical or traditional (TRAD) residential "wall mix" in Table 2. The TRAD mix was manufactured using the same materials as EcoA. Further details of materials, pilot test program, and mix-design testing can be found in [18-20].

	EcoA	TRAD
Cement (kg)	111	168
Fly Ash	259	42
Coarse Aggregate	813	1099
Fine Aggregate	809	747
Water	207	168
Activator	Yes	No

Table 2. Mix Proportions /m³ of EcoA and Traditional (TRAD) Wall Mix

EcoA is a self-compacting concrete, whereas the traditional mix is not. Thus, EcoA contains substantially more fine material in order to obtain a cohesive mix with high fluidity. This is realized through the use of more fly ash and a higher mass of binder. The proportion of the fly ash in the binder of EcoA is 70%.

5. Comparison of Strengths

The objective of using an activator in a self-compacting ICF concrete was to boost early-age strengths in order to continue to permit regular construction practices, such as early backfilling. The aim was to achieve the same, or greater, strengths as that of the traditional mix. Accordingly, standard laboratory compressive-strength tests were performed by casting, moist-curing and testing 100 mm diameter concrete cylinders, in accordance with Canadian Standards Association, A23.1 requirements. Figure 1 gives a comparison of strengths determined at ages from 3 to 380 days.

Figure 1. Compressive Strength of EcoA vs. Traditional Concrete (log scale)



The EcoA and TRAD concretes are similar in strength until an age in the range 30-50 days. After this time the TRAD concrete, that contains significantly more Portland cement, continues to gain strength.

6. Evaluation of Alkali-Aggregate Reactivity and Sulphate Resistance

Even though EcoA is intended for a non-exposed environment, there may be situations in which EcoA is used in ICF basement walls. At the lower level of such walls, just above the footings, EcoA may be exposed to sulphatebearing groundwaters and lower-sections of wall might be subjected to wetting-drying conditions (tests are ongoing to see if this is so). Accordingly, standard Mortar-Bar expansion tests (ASTM C1012) were performed on the EcoA binder, and the CSA A23.2-14A concrete-prism test was performed using the EcoA binder and the coarse- and fine-aggregate described above. These aggregates are known to be moderately prone to alkali-aggregate reactivity. Table 3 shows the expansion of concrete prisms made with the EcoA binder and the TRAD binder, combined with local coarse and fine aggregate.

Age	TRAD	EcoA	
125 days	0.013 (0.006)	-0.003 (0.004)	
320 days	0.022 (0.005)	0.009 (0.002)	

Table 3. Expansion of Concrete Prisms (%) average of 3 bars and (standard deviation)

Expansions of both concretes are small, yet the expansion of the EcoA concrete is significantly less than that of the TRAD concrete. These results, albeit limited in scope, suggest that the combination of the EcoA binder with local aggregates gives satisfactory performance with respect to alkali-aggregate expansion.

ASTM C1012 tests for sulphate resistance were started during the development stages of EcoA, and thus the specific EcoA binder/activator combination was not evaluated. However, 5 different binders that envelop the EcoA binder were tested. These five binders and the subsequent expansions (average of 5 or 6 bars), are given in Figure 2.



Figure 2. Expansion of Mortar Bars in ASTM C1012 Test

Expansions even after 720 days for all bars are much less than the 0.050% maximum allowed by CSA at 180 days for a sulphate-resistant cement. The benefits of using high proportions of fly ash is clearly shown. The addition of the activator appears to produce a slight increase in expansions, but expansions are still well below the allowable.

7. Shrinkage

Concrete prisms manufactured with EcoA and TRAD were water cured for 56 days and then subjected to ASTM C157 exposure to measure the relative shrinkage of the two mixes. Test results shown in Figure 3 are the averages of 3 prisms. Due to the lower cement content and lower water/binder ration of EcoA compared to TRAD, shrinkage of EcoA is less than TRAD at all ages



Figure 3. Shrinkage of Concrete Prisms, ASTM C157

8. Implementation of EcoA Concrete 1: Full-height Wall Cast

The suitability of EcoA for field application was first examined by casting a full-height (3.4 m), 1.2 m wide wall in the structural testing laboratory at the University of Calgary (Figure 4). The concrete was mixed in a pan-mixer in the concrete materials laboratory and then lifted by crane in a hopper to an in-line pump-truck parked at the loading doors of the structural laboratory. The concrete was then pumped through approximately 60 metres of 50mm hose to the top of the wall. Due to limitations in concrete-mixer capacity, the wall was filled by 3 lifts of 1-1.5 m each (note, however, that previous site-tests using SCC concrete have shown that the "Superform" type of form used here can withstand single lifts of 3.4 m before blowout of the forms at the bottom occurs).



The wall was instrumented to measure shrinkage in both the vertical and horizontal directions for 30 days after casting. At an age of 80 days, a total of 16 4inch cores were extracted from the wall: four cores were taken at 4 different levels above ground (Table 4). The compressive strength of 3 of the 4 cores was measured at an age of 84 days, while the 4th core at each height was tested for density, absorption and volume of permeable voids (VPV) as per ASTM Test Method C642.

During the casting procedure there were no visual observations that EcoA was prone to segregation. The mix appeared sufficiently cohesive while it was in the pump hopper and anecdotal

evidence from the pump operator indicated that the mix was typical of other pumpable concretes. The properties of the concrete as determined from cores tested at 84 days are given in Table 4.

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Height	Density	Compressive	Immersion	Volume of
Above		Strength @ 84	4 Absorption	Permeable
Ground	(kg/m3)	days	(%)	Voids
(m)		(MPa)		(%)
2.4	2256	17.2	7.5	16.2
1.8	2270	18.6	7.2	15.7
1.2	2284	20.2	7.0	15.1
0.6	2291	24.3	6.7	14.5
Average	2275	20.1	7.1	15.4

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Effectively no data have been published on typical values of absorption and VPV of residential class concretes. However, a recent unpublished study by the author tested 80 cores from a variety of 2-year old traditionallymanufactured foundations with satisfactory strengths. The average immersion-absorption was 6.1% (standard deviation 0.9%) and the average VPV was 15.3% (standard deviation 1.6%). Although absorptions given in Table 4 are higher than average, the overall results suggest that the porosity of EcoA is not atypical of normal foundation concrete. Table 4 shows that there is a gradient in all measured parameters from the bottom to the top of the wall. Compressive strength, in particular shows a reduction of 7 MPa from the 0.6 m to the 2.4 m level. To put this finding in perspective, another unpublished study by the author analyzed cores from 27 traditionally-cast concrete walls; cores were taken from the bottom, middle and top of 2.7 m walls and analyzed for strength, immersion absorption and density. The core strengths of these walls varied from 14.5 MPa to 45 MPa. It was found that on average strength from the bottom to the top dropped by 10.2 MPa, absorption increased by 1.0%, and density dropped by 63 kg/m³. These changes are significantly greater than those observed in Table 4 and suggest that if segregation is an issue it is an issue in all medium- to high-w/cm concrete walls.

Shrinkage in the test wall was measured by transducer in the horizontal (200 mm gauge length) and vertical (500 mm gauge length) for the first 30 days. Shrinkage of the concrete in both directions was small. It is interesting, however, to note that horizontal shrinkage at 30 days of 0.0002% was much smaller than the shrinkage in the vertical direction of 0.012%.

9. Implementation of EcoA Concrete 2: Duplex Construction



Figure 5. Duplex Cast using EcoA

In order to assess the suitability of EcoA for commercial production, a two-storey ICF duplex in cast in mid-Calgary was September of 2005 (Figure 5). All the walls (including the basement wall) were formed using 300 mm wide ICF units with a clear cavity of 175 mm. The walls were constructed in three steps, (a) footing to the first-floor level – 3m lift; (b) first floor to second floor - 3m ft. lift; (c) second floor to roof joists – 2.7m lift. Walls in each of these levels were cast in a single lift. Three different concrete mixes were used, one for each lift: a commercial SCC was used for the

first lift (foundation to first-floor); EcoA was used for lift 2, and a modification of EcoA -- denoted EcoAx – with $\frac{1}{2}$ of the activator dosage was used for the third lift (2nd floor to roof). In addition, the walls were instrumented to monitor the concrete temperature variations due to heat of hydration and effect of the ambient temperature on the concrete (insulated by the ICF). These results

are the subject of another report. The description below is a summary; a full report can be found elsewhere [20].

A commercial ready-mix, dry-batching plant, pumping equipment and procedures were used. Complete batching was done at the ready-mix plant and the concrete was transported to site over a distance of approximately 30 km. Slump flow of concrete from each truck was measured at site and varied from 425 mm to 505 mm. 100 mm x 200 mm cylinders were cast on site for compressive strength testing. No transportation or placement issues (pumping or compaction) were observed during the cast; in general, the self-compacting EcoA behaved in a similar manner to any other commercially available SCC mixes when used in a similar application. One exception was the presence of concrete clumps (2-7 cm diameter) in the concrete mix that was deposited into the pump hopper. Formation of the clumps was attributed to the nature of the dry mix batching plant used and the relatively high fluidity of the concrete which hinders the break-up of clumps in the concrete truck. Extended mixing in the concrete truck or withholding the water until the concrete has partially mixed at the batching plant had a positive effect to reduce clumping. A later investigation using X-ray diffraction analysis (XRD) revealed that the clumps consisted almost entirely of fly ash - suggesting the possibility of fly ash storage problems at the ready-mix plant.

Test cylinders were taken from site and either cured on-site in prefabricated styrofoam forms (to simulate the concrete in the ICF forms) or transported to the laboratory and moist-cured according to standard methods. Cylinder strengths are reported elsewhere [19, 20]. To obtain a more accurate estimate of the strengths in the structure, two concrete cores were taken from walls of each of the lifts and tested for compressive strength at the ages of 46





days (commercial mix) and 28 days (EcoA and EcoAx); the average results are given in Figure 6. Strengths obtained from individual cores are shown by white circles on the graph

The 28-day compressive strengths of EcoA cores taken from the walls (Figure 6) were satisfactory and, were, on average, greater than 46-dav strengths of the commercial mix. However, the strength of the two cores from the commercial-mix were 15.6 and 23.7 MPa, and rebar was observed in the lower-strength core. The true strength of the core from

the commercial mix is likely to be closer to 23.7 MPa. The 28-day strength of the EcoA mix is satisfactory for purpose despite the significantly lower cement content; this can be attributed to the superior curing conditions (insulation and moisture retention) within the ICF forms which benefits high fly-ash content concretes greatly. The superior curing environment is shown through temperature and humidity instrumentation results reported elsewhere [20]. Reduction in the activator dosage (EcoAx) results in lower strengths at all ages.

The standard-cylinder tests of the site-cast concrete [19,20] showed a reverse trend. The average 28-day compressive strengths of the site-cast lab cured EcoA cylinders was 16.5 MPa, while the 28-day cylinder strength of the commercial mix was 19.5 MPa.

10. Summary

Fundamental research indicates that the slow initial reactivity of some pozzolans can be enhanced through the use of chemical activators. However, care must be taken to match particular pozzolans with particular activators in order to achieve the desired results. Results are reported from laboratory and field tests where a sodium-sulphate based activator is paired with an Alberta sub-bituminous fly ash to make a very-high ash content self-compacting concretes -- EcoA. This concrete is intended for use in ICF wall construction. Laboratory and field results show satisfactory engineering performance of EcoA for the purpose intended. With respect to strength, durability, shrinkage and field-application EcoA often outperforms equivalent traditional wallconcrete.

11. Acknowledgements

The research reported here was performed with financial support from the Government of Canada Action Plan 2000 on Climate Change, and the Natural Sciences and Engineering Research Council of Canada. Substantial support during field research has been provided by Kanas Corporation. The researchers are grateful to Foothills Ready-Mix for allowing the EcoA concrete to be prepared at its plant.

12. References

[1] VanderWerf, P, Energy Comparisons of Concrete Homes Versus Wood Frame Homes, Portland Cement Association, Report RP119, 1997

[2] U.S. Department of Housing and Urban Development, Concrete Homes Versus Wood Frame Homes – Installed Cost, Acoustic, and Thermal Performance, Portland Cement Association, RP123, 1998.

[3] Arjunan, P., Silsbee, M.R., Roy, D.M., Chemical Activation of Low Calcium Fly Ash, Part 1: Identification of Suitable Activators and their Dosage, 2001 International Ash Utilization Symposium, Center for Applied Energy Research, University of Kentucky, Paper #105.

[4] Bakharev, T., Sanjayan, J.G., Chen, Yi-B., Alkali Activation of Australian Slag Cements, Cem. Concr. Res., 29, 1999, 113-120

[5] Dongxu, L., Zhongzi, X., Zhimin, L., Zhihua, P., Lin, C., The Activation and Hydration of Glassy Cementitious Materials, Cem. Concr. Res., 32, 2002, 1145-1152.

[6] Fraay, A.L.A., Bijen, J.M., de Haan, Y.M., The Reaction of Fly Ash in Concrete: a Critical Examination, Cem. Concr. Res., V 19, 1989, pp 235-246.

[7] Jiang, W., Alkali Activated Cementitious Materials: Mechanisms, Microstructure and Properties, PhD thesis, The Pennsylvania State University, December 1997, pp 231.

[8] Palomo, A., Grutzeck, M.W., Blanco, M.T., Alkali-activated fly ashes: a Cement for the Future, Cem. Concr. Res., V 29, 1999, 1323-1329

[9] Poon, C.S., Kou, S.C., Lam, L., Lin, Z.S., Activation of fly ash/cement systems using calcium sulfate anhydrite (CaSO₄), Cem. Concr. Res., V 31, 2001, 873-881

[10] Williams, P.J., Biernacki, J.J., Walker, L.R., Meyer, H.M., Rawn, C.J., Bai, J., Microanalysis of alkali-activated fly ash-CH pastes, Cem. Concr. Res., V 32, 2002, 963-972

[11] Shi C., Day, R.L., Comparison of different methods for enhancing reactivity of pozzolans Cem. Concr. Res., V. 31, No. 5, May 2001, 813-818

[12] Shi, C. and Day, R.L., Selectivity of Alkaline Activators for the Activation of Slags, Cem. Concr. and Aggregates, American Society for Testing and Materials, Summer 1996, pp 8-14

[13] Shi, C., Day, R.L., Chemical Activation of Blended Cements Made with Lime and Natural Pozzolans, Cem. Concr. Res., V 23, 1993, 1389-1396

[14] Shi, C., Day, R.L., Acceleration of the Reactivity of Fly Ash by Chemical Activation, Cem. Concr. Res., V. 25, 1995, 15-21.

[15] Shi, C., Day, R.L., Pozzolanic Reaction in the Presence of Chemical Activators, Part I. Reaction Kinetics, Cem. Concr. Res., V30, 2000, 51-58.

[16] Shi, C., Day, R.L., Pozzolanic Reaction in the Presence of Chemical Activators, Part II. Reaction Products and Mechanism, Cem. Concr. Res., V 30, 2000, 607-613

[17] Shi, C., Activation of Natural Pozzolans, Fly Ashes and Blast Furnace Slag, PhD Thesis, University of Calgary, December, 1992, 253pp.

[18] Nazir, M., Day, R., Moore, L., Development of Energy Efficient and Sustainable Concretes for Insulated Concrete Form Construction, Canadian Society for Civil Engineering, Annual Conference, Toronto, 2005, pp GC-172-1 to GC-172-10

[19] Day, R.L., Nazir, M., Moore, L., Laboratory and Case Studies of the Use of High-Volume Fly-Ash Concrete in Insulated-Concrete-Form Construction, Canadian Society for Civil Engineering, 1st International Construction Specialty Conference, Calgary, May 24-26, 2006, paper CT-101

[20] Moore, L.M., Sustainable Concrete for Insulated Concrete Form (ICF) Construction, MSc thesis, University of Calgary, June, 2006.