

# Fabrication and Testing of CSAB Cements in Mortar and Concrete that Utilize Circulating Fluidized Bed Combustion Byproducts

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## INTRODUCTION

The utilization of circulating fluidized bed combustion (CFBC) ash to make cement products that provide added value and offset CO<sub>2</sub> production is the objective of this research. Research by Bernardo et al. (2004) has confirmed that high quality CSAB clinker can be produced using CFBC spent bed material as its principal feedstock, at kiln temperatures of 1200 to 1350°C.<sup>3</sup> CFBC burns coal in the presence of a bed of slaked limestone, which effectively absorbs sulfur dioxide (SO<sub>2</sub>) to form anhydrite (CaSO<sub>4</sub>). CFBC produces two kinds of spent bed materials, coarse bottom ash and a much finer fly ash. Both of these products are very high in calcium. When properly conditioned these materials are capable of acting as hydraulic cements, forming both calcium aluminosulfate minerals, most importantly ettringite, as well as calcium-alumina-silica gels, like that formed from Portland cement.

The research to generate CSAB cement was centered on two components. The first was the production of a cement that maximizes the proportion of Kentucky coal combustion byproducts while achieving acceptable strength development. The second research component was the evaluation of the durability of mortars and concrete prepared from the CSAB cement. Issues to be addressed include strength optimization, dimensional stability (e.g. shrinkage and expansion), and durability (e.g. freeze-thaw, chlorination and carbonation resistance). The CSAB cements produced from the CFBC byproducts are comparable to both a Type 1 ordinary Portland cement and a commercially produced CSAB cement from China. Approximately 1 million tons per year are manufactured in China which has special standards for the cements.<sup>12,13</sup>

The 300 MW Gilbert circulating fluidized bed combustion electric generation unit operating at East Kentucky Power Cooperative's Spurlock Power Plant in Maysville, Kentucky, is currently the cleanest in the state. It is also one of the most economical. The circulating fluidized bed combustor (CFBC) burns coal in the presence of a bed of slaked limestone, which effectively absorbs sulfur dioxide (SO<sub>2</sub>) to form anhydrite (CaSO<sub>4</sub>). Its low temperature operation produces much less thermal NO<sub>x</sub> than

pulverized coal combustion (PCC). However because it uses a higher Ca/S ratio than a scrubbed PCC system, it consumes more limestone, produces more solid waste and CO<sub>2</sub> than conventional coal plants. On a per megawatt basis, CFBC produces four to six times the solid waste as a conventional un-scrubbed PCC plant and about two times as much as a scrubbed PCC plant.<sup>1,2</sup> The Gilbert plant will produce approximately 400,000 tons of spent bed material per year and, along with two additional planned CFBC units, will add about 6% to Kentucky's generating capacity but increase the quantity of coal combustion byproducts (CCBs) by almost 14%.<sup>1,2</sup> This large influx of spent bed material is the reason for this research because such a large influx of CCBs will need to be addressed. Past research has shown the potential of using CFBC material as a raw material for the production of a low-energy, rapid-hardening cement.<sup>3</sup>

## CSAB CEMENT FORMULATIONS

Mixtures of FBC spent bed material, PCC fly ash, bauxite and limestone were interground for clinkering. The clinker tests were conducted from 1000°C to 1250°C and included compositions in the stability fields of Klein's compound and belite. Mineral composition of the products was determined by X-ray diffraction. The first cement formulation was calculated using Bogue equations that were modified for phases in CSAB clinker; this formulation is termed "CSAB#1". The phases assumed to be present were Klein's compound, belite, ferrite (C<sub>4</sub>AF), calcium sulfate, and a minor amount of lime (<0.5%). However, it was found that the normative equations could not be used to optimize the CSAB compositions, probably because of the formation of minor amounts of other phases such as gehlenite, and the simplistic assumption that the aluminum:iron ratio in the ferrite phase = 1. Therefore, adjustments were made to the formulations to meet several objectives: 1) minimize the proportion of limestone used and thus the free lime formed (CaO), 2) maximize the proportions of byproducts (i.e. CFBC and PCC ash), and 3) produce a cement that will approach the performance of the commercial CSAB cement. The adjustments were made by analyzing each clinker using XRD until the desired composition was achieved resulting in the synthesis of formulations termed "CSAB#2" and "CSAB#4". Figure 1 shows the XRD profiles of the laboratory synthesized clinkers compared to three commercial CSAB cements: China CSAB, Commercial CSAB #1, and Commercial CSAB #2. The chemical mineral composition of these cements, in addition to Portland cement, is provided in Table 1. The Gilbert FBC material is a potential source of CaO and SO<sub>3</sub> and thus was used as a partial substitute for gypsum and limestone in the laboratory CSAB raw materials.

The effects of firing temperature were examined by XRD using the CSAB#1 clinker formulation. The firing programs consisted of heating the raw mix at 1175°C, 1200°C, 1225°C, and lastly 1250°C for one hour each. The resulting clinkers were slowly cooled within the furnace. Visually there was a progressively darker and notable volume loss with increasing firing temperature as seen in Figure 2. The darkening color reflects the increased level of sintering. Each clinker was milled in a ball mill along with gypsum, which is used to "activate" the Klein's compound to form additional ettringite during hydration. Class F fly ash was also milled with the clinker to serve as a filler for certain

formulations. The milling of gypsum and/or fly ash with the clinker to make the final cement product is known as “process addition”.

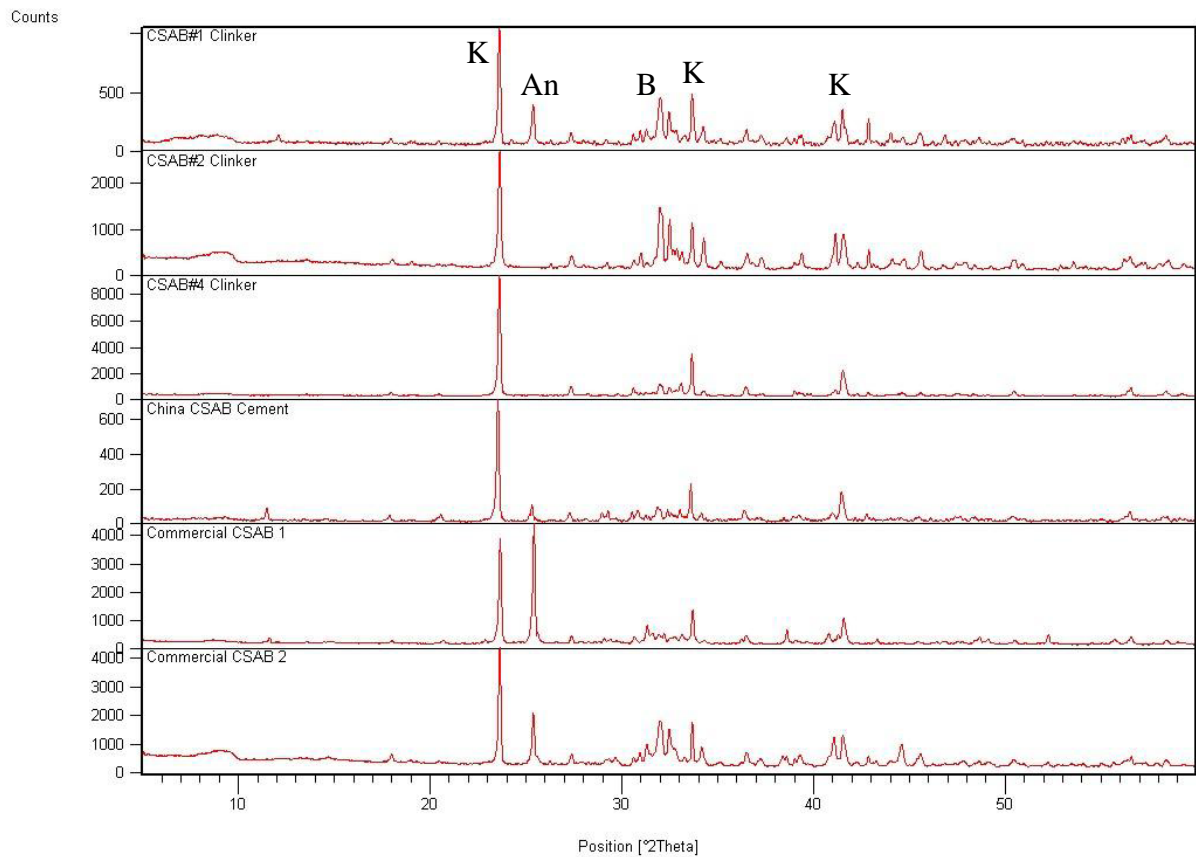


Figure 1. XRD profiles of the three laboratory synthesized clinkers compared to the commercially available CSAB cements. K = Klein's Compound; An = anhydrite; B = belite ( $C_2S$ )

Cement	Cement Composition (%)							
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O	SO <sub>3</sub>
OPC	20.5	5.4	2.6	63.9	2.1	0.61	0.21	3.0
China CSAB	11.12	26.94	1.76	44.99	3.18	0.04	0.19	12.23
Commercial CSAB 1	5.89	20.48	2.53	42.29	0.78	0.1	0.15	25.71
Commercial CSAB 2	14.92	16.12	1.32	48.91	1.63	0.24	0.49	15.46
CSAB#2	16.90	16.95	2.39	47.10	2.25	0.15	0.68	13.52
CSAB#4	8.21	24.30	2.59	40.02	1.32	0.14	0.62	22.30

Table 1. CSAB cement composition from XRF analysis.

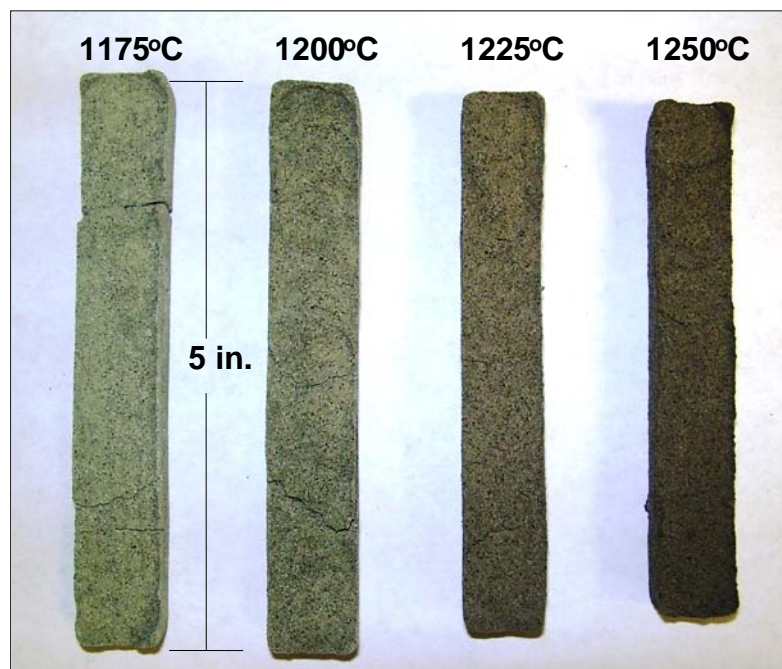


Figure 2. CSAB clinker demonstrating color variation and mass loss based on oven-firing temperature

The optimum firing temperature for the FBC material based CSAB cement was chosen to be 1250°C. At this temperature the maximum amount of Klein's compound and belite was formed with minimal quantities of silicosulfate, an unreactive phase.<sup>9</sup> Table 2 provides a list of phases present in the cement formulations.

Phase	Composition	OPC	China CSAB	CSAB#2	CSAB#4
$C_4A_3S'$	$Ca_4Al_6O_{12}SO_4$	—	√	√	√
$C_2S$	$Ca_2SiO_4$	√	√	√	√
$C_4AF$	$Ca_2(Al,Fe^{+3})_2O_5$	√	—	√	—
$CS'$	$CaSO_4$	√	√	—	—
$C$	$CaO$	—	—	—	—
$C_2AS$	$Ca_2Al_{2.22}Si_{.78}O_{6.79}(OH)_{.22}$	—	—	—	—
√ = Major phase present • = Minor phase — = Not detected or trace					

Table 2. Comparison of clinker phases.

## FABRICATION OF BULK CSAB CLINKER CEMENT

The material formulation discussed in the previous section was used to create larger samples of FBC-derived CSAB cement for mortar testing. Based on the calculated mix proportions determined from the modified Bogue equations, the raw materials were proportioned and ground in a ball mill to reach an approximate particle size of 16 microns. The ground mix was then placed into zirconia crucibles and fired in an electric furnace at 1250°C for one hour and then air-cooled. The resulting clinker was soft and required little effort to grind to cement fineness (Figure 3). The ground clinker was analyzed to check that the expected phases were present. Calcium sulfate ( $CaSO_4$ ) is necessary in CSAB cement to promote strength development through the formation of ettringite.<sup>4,7</sup> The calcium sulfate can be added by proportioning the CSAB clinker to contain excess  $CaSO_4$  as anhydrite, or by intergrinding gypsum or anhydrite with the CSAB clinker; the cement mixes within this project were fabricated by intergrinding FGD gypsum (Taylor, 2001).



Figure 3. CSAB#2 cement clinker.

## STRENGTH TESTING OF CSAB MORTAR AND CONCRETE

To establish benchmarks for strength performance, the commercial CSAB cements described earlier were tested. Mortar cubes were prepared for the cement formulations following ASTM C 305 and C 109 protocols. The mortar mix proportions are provided in Table 3. The data for the commercially available CSAB cements are shown in Figure 4. As expected for rapid-setting, high-strength cements, the 1-day strength significantly exceeded that of OPC. The rapid strength gain, accompanied by rapid heat evolution, is characteristic of CSA cements.<sup>4,7</sup> This is primarily due to the presence of a large amount of Klein's compound, which readily forms ettringite upon hydration (Figure 5 and Figure 6). At 28-days of curing the cement from China matched the strength of the OPC but was then surpassed by the latter. The leveling-off of strength gain for the CSAB mortars was a result of the consumption of the available ettringite-forming compounds. The extremely-high strength of the Commercial 1 cement is attributed to the large amount of Klein's compound and anhydrite available in the cement. However, the Commercial 2 cement has a large amount of Klein's compound but half of the available anhydrite, based on peak intensity. The lesser amount of anhydrite in the cement explains the slower strength gain after 1-day of curing compared to Commercial 1.

	OPC	China CSAB	CSAB#1	CSAB#2	CSAB#4 HS	CSAB#4 MS	CSAB#4 LS
Cement (g)	500	500	500	450	500	500	500
Sand (g)	1375	1375	1375	1237.5	1375	1375	1375
Water (g)	242	242	238.8	217.8	242	215	201
Flow (%)	112	112	81	117	120	109	109
Time of Set (minutes)	189	116	-	88	67	78	110

Table 3. Mortar mix proportions following ASTM C 305 and C 109 protocols.

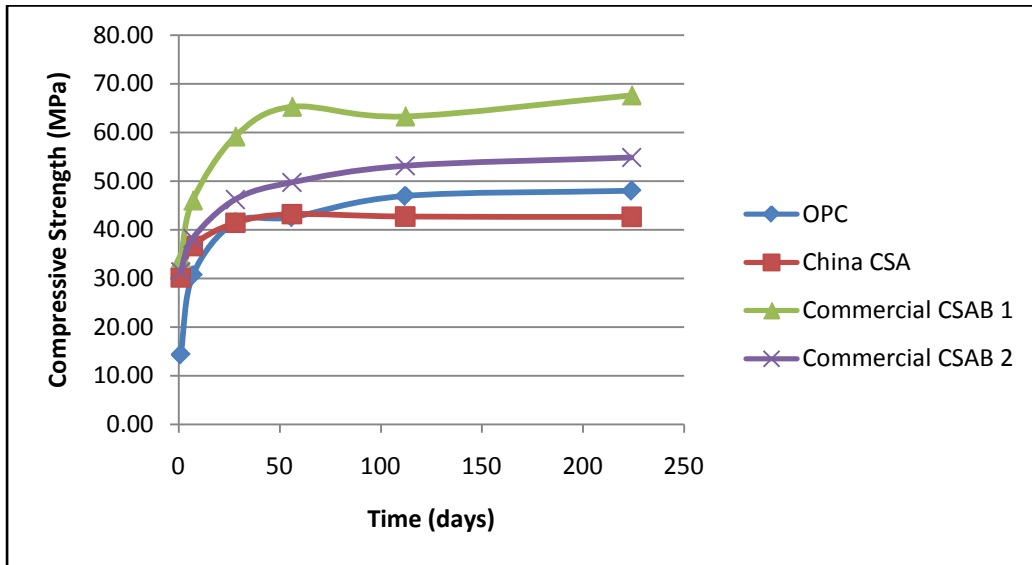


Figure 4. Compressive strength of mortar cubes made with commercially available CSAB cement.

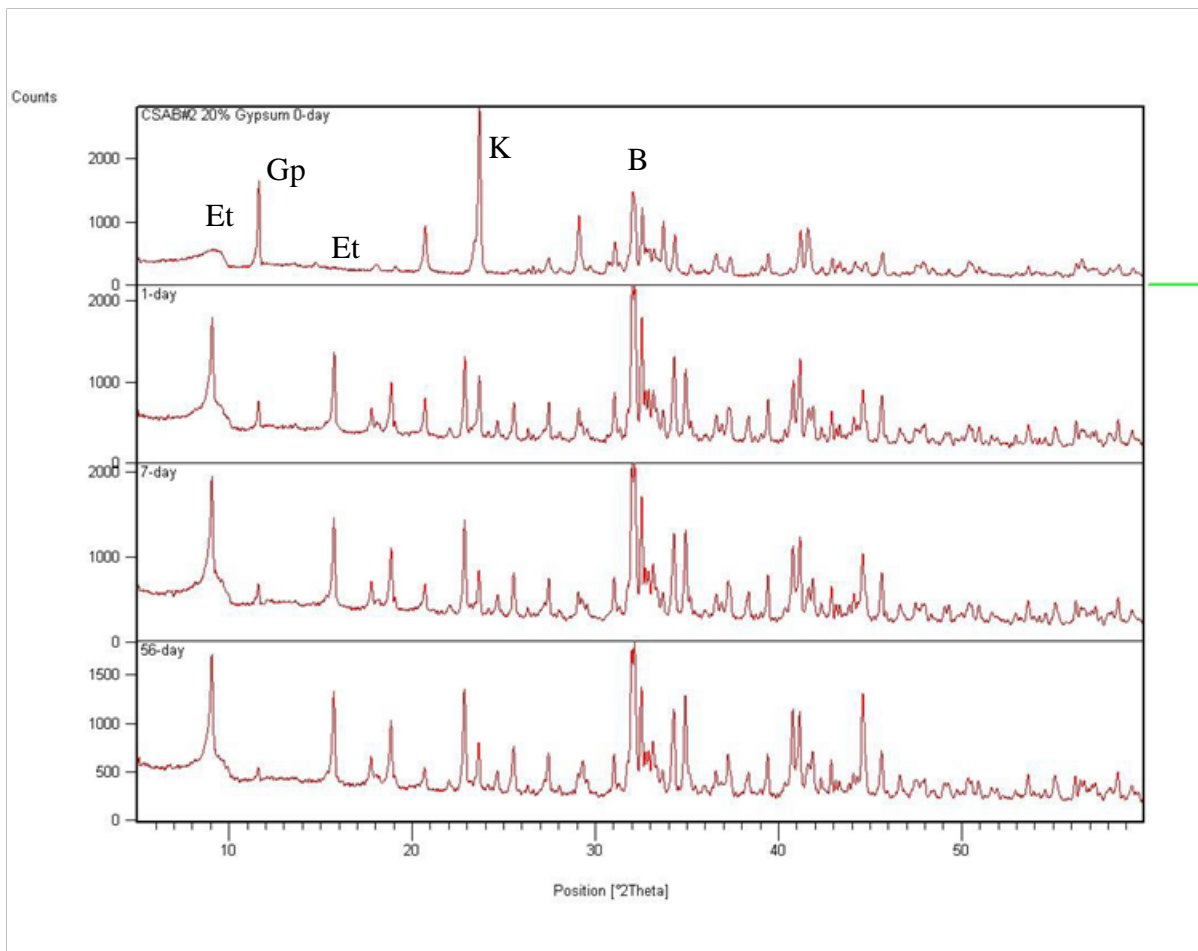


Figure 5. XRD profiles for the hydration of the CSAB#2 20% gypsum cement. Gp = gypsum; K = Klein's Compound; B = belite; Et = ettringite



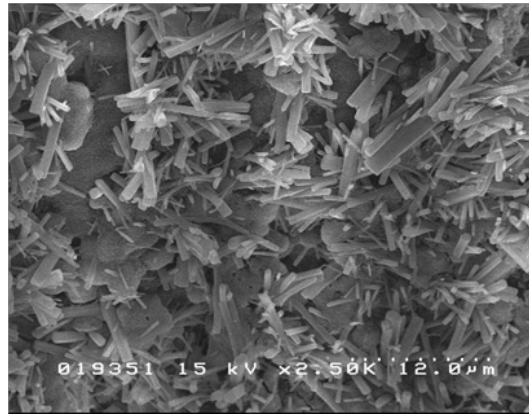


Figure 6. SEM image, under 2500x magnification, showing the formation of well-defined ettringite crystals in the CSAB#2 20% gypsum cement after 7-days of curing. The scale bar represents a length of 12 microns.

After compressive strength testing of the commercial CSA cement mortars, the laboratory CSAB#2 clinker was interground 20% by mass FGD gypsum and mortars prepared according to ASTM C 109. The mortar prepared with this cement did not experience expansive cracking but exhibited considerably lower strength than the commercial cements (Figure 7), probably because it contained substantially more belite. However, its strength gain was good and it would likely qualify as a general rapid hardening (GRH) or medium rapid hardening (MRH) cement under ASTM C 1600 requirements. Of particular interest is that the performance was achieved with cement that comprises approximately 40% coal combustion byproduct.

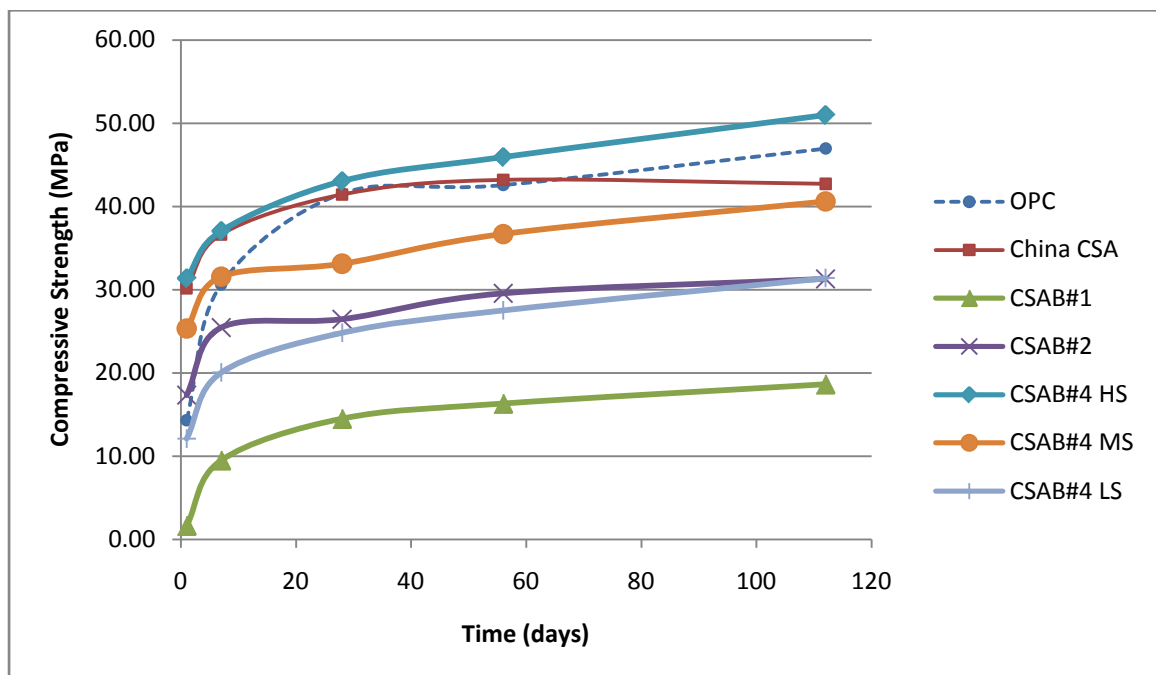


Figure 7. Compressive strength of mortar cubes using the CSAB#4 cement.



The compressive strength development of CSAB#4 mortar was comparable to the China CSAB cement (Figure 7), which is not surprising since both contained a large amount of Klein's compound (Figure 1). CSAB#4 HS mortar cubes matched the performance of the China CSAB cubes from 1 to 7-days, and then from 28- to 112-days they greatly exceeded the China CSAB cubes. The continued strength gain exhibited by the CSAB#4 HS cubes can likely be attributed to the presence of the active belite phase in the clinker. Based on ASTM C 1600, CSAB#4 HS would qualify as a very rapid hardening (VRH) cement. CSAB#4 MS produced a mortar that achieved approximately 26 MPa in 1 day, 32 MPa in 7 days, and 35 MPa in 28 days, which meets the criteria for a VRH cement. It is interesting that this cement gains strength more rapidly than the CSAB #2 formulation despite the higher percentage of byproduct in CSAB#4 MS. The CSAB#4 LS mortar data showed a substantially lower strength development curve than the CSAB#4 HS or MS mortars. The LS cubes had similar 1-day strength data when compared to OPC but failed to gain strength as quickly as the OPC or CSAB#4 HS and MS mortars. This was primarily due to the increased percentage of coal byproduct additions used in the cement. Although this lowered the strength, it helped to produce a more dimensionally stable mortar (discussed later).

Figures 8, 9, and 10 depict the growth of ettringite crystals in the CSAB#4 HS mortar. Figure 8 is an image of ettringite crystals with an average length of 10 to 20 microns. The predominantly visible crystals formed in the space between the sand grains and cement paste. However in the bottom-left corner of the image the outline of several ettringite crystals can be seen within the cement paste. Figure 9 shows ettringite crystals with an average length of 5 to 20 microns that have formed within the cement paste and created a dense network of interlocking crystals and paste. The rapid-strength gain of CSA cements can be attributed to this geometry. Figure 10 shows an SEM image on the right, under 70x magnification, of the CSAB#4 mortar with grains of sand clearly embedded in the cement paste. By increasing the magnification to 3000x and focusing on the surface of one sand grain ettringite crystals, approximately 10 microns in length, have melded to the surface of the sand grain, thereby bridging the interfacial zone between the grain and the surrounding paste.

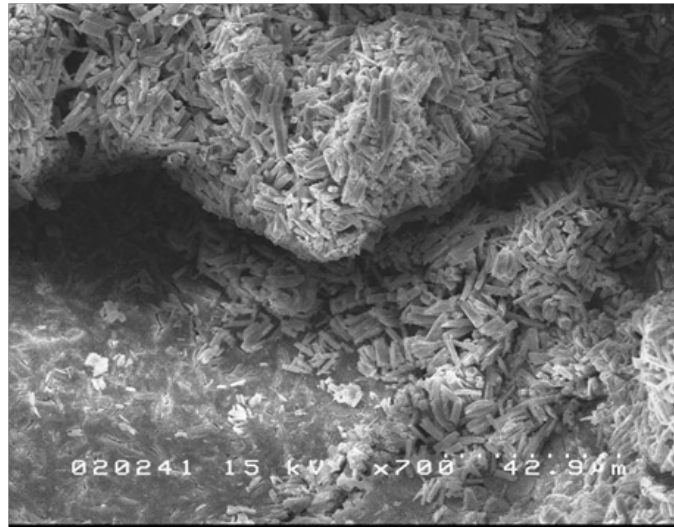


Figure 8. SEM image, under 700x magnification, of ettringite crystals in the CSAB#4 cement mortar.

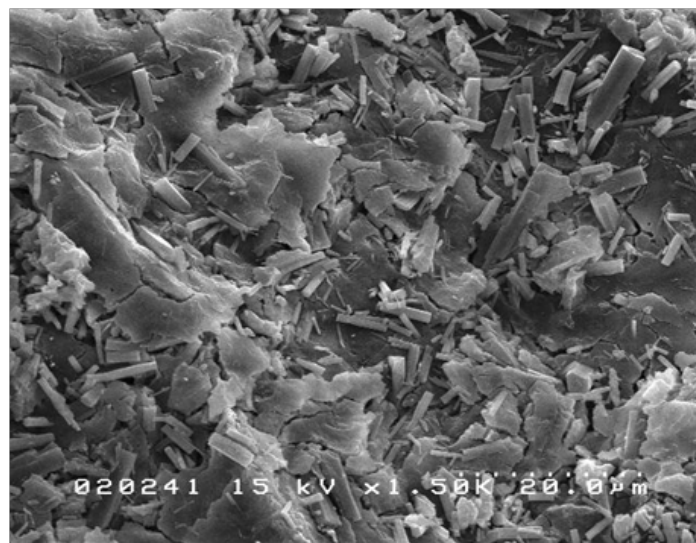


Figure 9. SEM image, under 1500x magnification, showing ettringite crystals within the CSAB#4 cement paste.

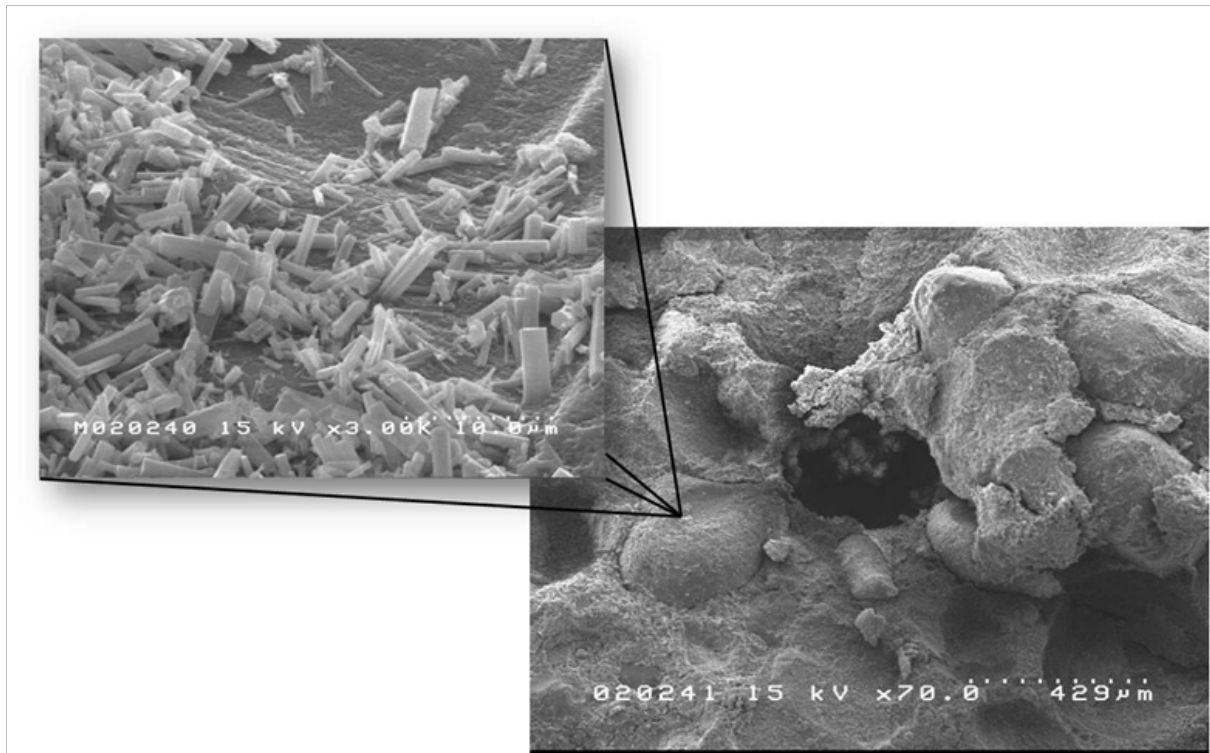


Figure 10. SEM image of the CSAB#4 mortar with sand grains clearly embedded in cement paste, with a zoomed view of the sand grain surface.

The CSAB cements were evaluated for strength performance in concrete. Concrete mixtures were proportioned and mixed following ASTM methods. 4 in. x 8 in. cylindrical specimens were made following ASTM C 192. The first set consisted of Type-1 OPC concrete, the second set contained China CSAB cement, and the third set was prepared using the laboratory CSAB#2 cement. The cements were prepared in a concrete based on a nominal 28-day compressive strength of 41 MPa (6000 PSI). For this concrete the  $W:CM = 0.45$ . Polycarboxylate high-range water reducer (BASF Glenium 3030) was used to increase slump. The fresh concrete properties of slump and unit weight were determined following ASTM C 143 and ASTM C 138, respectively. Air content was measured using a pressure Type B meter. Table 4 contains the material properties and concrete mix design for the test specimens. The time of set was determined for the OPC, China CSAB, and CSAB#2 concrete mixes, following ASTM C 403 protocols (Table 4). Figure 11 provides the concrete compressive strength data. As expected, the China CSAB concrete attained a higher strength after 1-day of curing than the OPC specimens. Whereas the CSAB#2 concrete had approximately the same strength as the concrete made with OPC. However after 28-days of curing the OPC specimens continued to strength, whereas the strength gain for the CSAB concrete specimens slowed considerably.

Mix Design (kg/m <sup>3</sup> )	41 MPa (6000 psi)		
	OPC	China CSAB	CSAB#2
<i>Cement</i>	335.0	335.0	335.0
<i>Coarse Aggregate</i>	624.7	614.5	655.5
<i>Intermediate Aggregate</i>	371.0	364.9	389.3
<i>Fine Aggregate</i>	825.7	812.2	866.5
<i>Water</i>	174.8	147.8	147.8
<i>Water Retarder (ml)</i>	18.4	18.7	28.4
<i>Slump (in.)</i>	4.25	4.25	2.5
<i>Unit Weight</i>	2375.7	2365.7	2394.3
<i>Air Content (%)</i>	*	*	3.2
<i>Water/Cement</i>	0.441	0.441	0.450
<i>Time of Set (minutes)</i>	325	164	88
* no data available due to equipment failure			

Table 4. Mix design data for the molded concrete specimens.

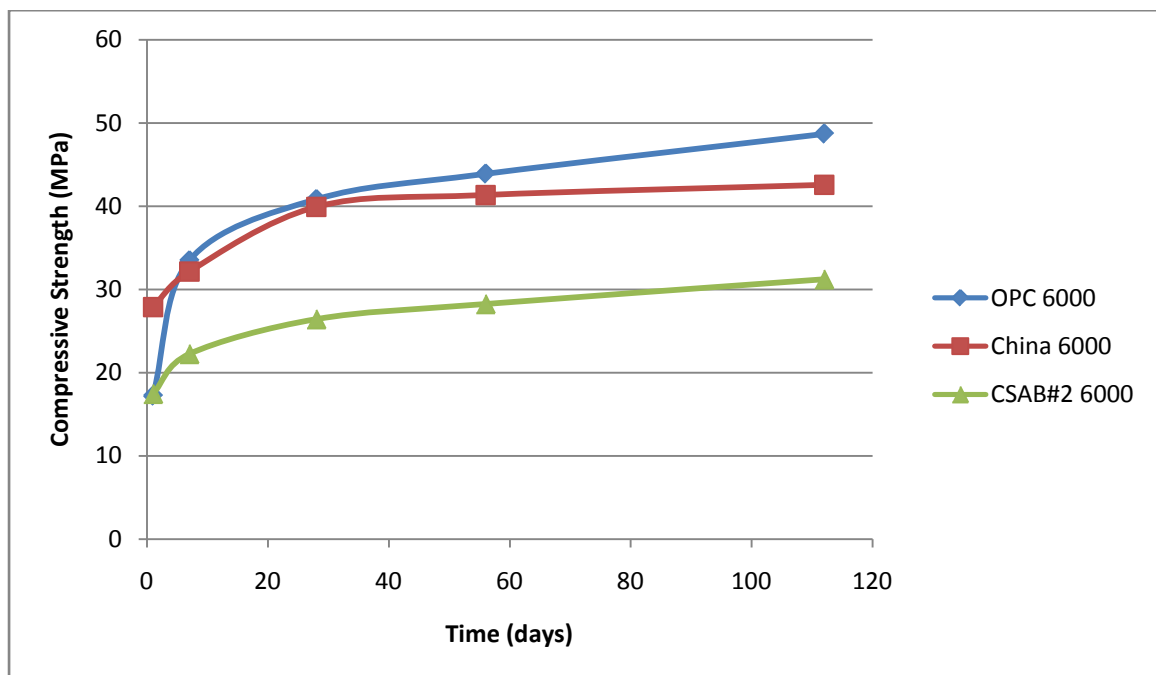


Figure 11. CSAB cement concrete compressive strength performance compared to OPC.

## CARBONATION TESTING

Corrosion of steel in concrete is the greatest threat to the durability and integrity of concrete structures in many countries.<sup>5</sup> Carbonation could exacerbate the corrosion potential by lowering the pH, and CSA concretes have been found to carbonate at a faster rate than Portland cement concrete.<sup>8</sup> However, the rate depends on the proportions of the different phases present.<sup>2,8,12</sup> Furthermore, the data on the effects of carbonation on concrete strength have been inconsistent.

Atmospheric carbon dioxide, when dissolved in the pore water in concrete or mortar, reacts rapidly with the calcium hydroxide produced during cement hydration to form calcium carbonate. If all calcium hydroxide is reacted then the pH is lowered from  $>12$  to about 8.5.<sup>5,10</sup> The carbonation reaction occurs first at the surface of the mortar and progresses inwards via diffusion through the carbonated layer.<sup>5</sup> Carbonation itself is not necessarily damaging, but in reinforced or prestressed concrete, carbonation may reach the steel reinforcement and the reduced alkalinity renders the passive layer around the reinforcement ineffective and makes the steel susceptible to corrosion.<sup>5,10</sup> Following a testing procedure devised by the University of Dundee's Concrete Technology Unit in Scotland, an accelerated carbonation test was carried out in an enriched atmosphere of  $2\% \pm 0.1\% \text{ CO}_2$ ,  $22^\circ\text{C} \pm 0.5^\circ\text{C}$ , and  $56\% \pm 2\%$  relative humidity (Figure 12).<sup>11</sup> Mortar cubes were prepared using OPC, China CSAB, and laboratory CSAB cement following ASTM C 305 and C 109 protocols, then placed into the accelerated carbonation chamber.



Figure 12. a) Carbonation chamber with attached heat exchanger; in the foreground is the gas control valve and temperature control module. b) A view inside the chamber; with the dehumidifier at the bottom, and the relative humidity logger on the left.

To determine the depth of carbonation, a pH indicator was used. A 2% solution of phenolphthalein in ethanol was sprayed onto the mortar surface, which highlighted areas with a pH greater than 10.5 pink, while leaving the carbonated mortar (with pH less than 10) colorless. A typical area after testing is shown in Figure 13. The depth to the pink/clear boundary was then measured and recorded. Table 5 shows the results of the carbonation test.



Figure 13. Mortar cubes that have been tested for compressive strength then sprayed with phenolphthalein. The rim around the pink area is the carbonation layer. These cubes have been cured for 112 days.

Carbonation Depth (mm)	28 – Day		56 – Day		112 – Day		Percent Carbonated
	Avg.	Max.	Avg.	Max.	Avg.	Max.	
<i>Control (OPC)</i>	-	-	5.50	7.75	6.90	8.76	27.2%
<i>China CSAB</i>	3.85	6.32	10.16	13.21	11.47	13.86	45.2%
<i>CSAB#2</i>	-	-	9.74	12.70	12.67	15.37	49.9%
<i>CSAB#4</i>	5.71	7.15	11.42	13.28	21.91	22.24	86.3%

Table 5. Results of the carbonation test showing depths of carbonation.

The data in Table 5 shows that the OPC mortar cubes underwent the least amount of carbonation averaging 5.50 mm in 56-days and increased to only 6.90 mm after 112-days. The China CSAB mortar cubes experienced rapid carbonation within 56 days but thereafter the carbonation rate decreased, reaching only 11.47 mm after 112-days. The carbonation depth in the CSAB#2 mortar cubes reached an average depth of 9.74 mm after 56-days but increased to 12.67 mm after 112-days. The CSAB#4 LS cement, which was produced using greater than 70% CCBs, experienced the highest carbonation depth of 11.42 mm after 56-days, and doubled that after 112-days to 21.91 mm. Of interest is the depth of carbonation experienced by each cement relative to a



maximum carbonation depth of 25.4 mm (i.e. the center of a 50.8 mm or 2 in. cube), the OPC cubes reached only 27.2% of total carbonation. The China CSAB cubes reached 45.2%, the CSAB#2 cubes 49.9%, and the CSAB#4 LS cubes reached nearly a complete carbonation of 86.3%.

The results show that, after 56 days and 112 days of exposure, all CSAB mortars carbonated to a greater extent than the OPC specimens. Table 6 shows the unconfined compressive strength (UCS) data from the carbonation experiments. The first column for each specimen shows the strength data of cubes cured under normal conditions. The second column shows the strength data for the mortar cubes exposed to the enriched CO<sub>2</sub> environment. The data indicated that, at each curing period, carbonation increased the compressive strength of the OPC, China CSAB and CSAB#2 cubes; the CSAB#4 LS mortars cured in 2% CO<sub>2</sub> were stronger at 28 days, but weaker at 56 and 112 days. The strength increase can be attributed to the formation of calcium carbonate which occupies a greater volume than the calcium hydroxide, and so the porosity of the carbonated zone is reduced, increasing the surface hardness and strength, and reducing the surface permeability.<sup>1,5,6</sup> However, the CO<sub>2</sub> enriched cubes exhibited a regression in strength after 28 days curing, possibly due to deterioration of ettringite.

Unconfined Compressive Strength (MPa)	Normal Curing			CO <sub>2</sub> Enriched Curing		
	<i>28-day</i>	<i>56-day</i>	<i>112-day</i>	<i>28-day</i>	<i>56-day</i>	<i>112-day</i>
<i>Control (OPC)</i>	41.5	42.6	46.9	n/a	56.3	54.1
<i>China CSAB</i>	34.4	31.9	37.2	51.2	44.8	41.4
<i>CSAB#2</i>	28.2	29.4	n/a	n/a	40.7	37.7
<i>CSAB#4</i>	45.1	40.1	46.7	48.6	39.7	32.8

Table 6. Comparison of the unconfined compressive strength data for the mortar cubes cured under normal conditions compared to those cured in a CO<sub>2</sub> enriched environment.

## CONCLUSIONS

The Gilbert fluidized bed combustion material has potential for use in the production of calcium sulfoaluminate belite cements. The utilization of the Gilbert CFBC spent bed material in CSAB cement shows potential as a large-volume use for the material. Heating FBC bottom ash, PCC fly ash, limestone, and bauxite at 1250°C, produced a large quantity of Klein's compound and belite. The Gilbert FBC ash provides needed calcium sulfate and, particularly, calcium oxide. The calcium oxide within the ash is an effective substitute for limestone, which is required as a raw material for CSAB cement clinker. In fact, if changes in the Gilbert FBC combustion process were to result in substantially less lime in the spent bed material, its' value as a CSAB clinker raw



material would be limited since FGD gypsum would provide a more concentrated and refined source of calcium sulfate.

The synthesized CSAB clinkers were soft and readily milled to cement fineness. Milling the clinker with FGD gypsum was effective in provide the additional calcium and sulfate required to “activate” the clinker to form ettringite. The compressive strength of the commercial and laboratory CSAB cements produced high-early strengths that exceeded those of ordinary Portland cement. Additional long-term strength was possibly provided by hydration of dicalcium silicate ( $C_2S$ ) within the clinker. The durability of the laboratory CSAB cements was similar to that of commercially available CSAB cements. The tests in which the CSAB cements performed well were resistance to deicer chemicals, drying shrinkage and resistance to freezing and thawing. However, all of the CSAB cements carbonated more rapidly than OPC and tended to undergo strength regression as carbonation became more extensive. Thus, the CSAB cements would likely provide little protection for reinforcing steel within concrete.

Milling the laboratory CSAB clinker with Class F fly ash, in addition to FGD gypsum, appeared to improve the dimensional stability of CSAB mortar. In every cement that contained fly ash addition, destructive expansion did not occur and drying shrinkage improved. However, fly ash addition generally decreased the compressive strength, although the water reduction achieved with the fly ash helped to offset this (see Table 3). Future work will focus on optimizing the quantity of fly ash addition to provide maximum water reduction benefits and minimize the strength loss. A major issue regarding the production of CSAB cement is one of cost. Because CSAB clinker production requires substantial quantities of bauxite, the cost of these cements is high. In order to minimize or eliminate bauxite, alternatives to this raw material need to be pursued. One approach is to formulate high-ferroaluminate CSAB cements using feed materials that contain high percentages of iron. High-iron calcium aluminate cements are currently produced where rapid strength gain and fire resistance is desired. The replacement of some bauxite with high-iron raw materials will have the net effect of replacing some of the aluminum with iron, which is considerably less expensive. The feed materials could include coal fly ash and/or red mud, which is a byproduct of bauxite processing. The production of high-ferroaluminate CSAB cements will thus be pursued in future research.

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