Alkali Activated Autoclaved Aerated Concrete made with Fly Ash Derived Cenospheres: Effect of Fly Ash and Precuring Temperature

Michael W. Grutzeck, Materials Research Institute, MRL Building, The Pennsylvania State University, University Park, PA 16802 (gur@psu.edu)
Stephen Kwan, Materials Research Institute, MRL Building, The Pennsylvania State University, University Park, PA 16802 (sxk13@psu.edu)
Maria DiCola, Materials Research Institute, MRL Building, The Pennsylvania State University, University Park, PA 16802 (mld7@psu.edu)

ABSTRACT

It is possible to make reduced density autoclaved aerated concrete (AAC) - like materials without foaming by adding fly ash derived cenospheres to the formulation. The cenospheres are robust enough to allow all types of mixing including shear mixing associated with macro defect free processing (MDF). Work has demonstrated that fly ash derived AAC can be produced with and without alkali activation. Sodium hydroxide activated samples produced various zeolites within the matrix whereas conventional lime based AAC produced tobermorite and katoite. Strengths of the samples are related to the nature of the precuring conditions, which ultimately affect microstructure and strength development during final autoclave processing.

KEY WORDS: autoclaved aerated concrete, AAC, alkali activated, fly ash, cenospheres

INTRODUCTION

Autoclaved aerated concrete (AAC) is a popular building material in use all over the world. It has a 50-year history of successful use in all environments for all types of buildings (Wittmann, 1983, 1992). Two types of AAC are commonly being manufactured. Both use combinations of lime and Portland cement mixed with either ground quartz sand or Class F fly ash. AAC offers a wide array of positive attributes vis à vis concrete block and insulated wood
frame construction for residents of North America. However, the final decision to use AAC instead of more conventional building materials will ultimately hinge on the cost of making, transporting and assembling AAC at its point of use. To date, even after intensive lobbying by the AAC industry, the North American consumer has not made the switch. In fact two of the original German manufacturers have pulled up stakes and gone home.

Environmentally speaking, AAC is less material and energy consumptive than many construction materials. It is estimated that the total amount of energy consumed during production (including energy invested in making Portland cement, lime and aluminum powder) is on the order of 1000 MJ/m$^3$ (Aroni et al., 1993). It achieves this status because some AAC manufacturers already use fly ash and other industrial waste in their formulations coupled with the traditional low energy processing in an autoclave at ~12 bars and 180°C. The introduction of a cellular structure during initial formation of the AAC allows one to produce 3-4 m$^3$ of AAC from 1 m$^3$ starting materials (Aroni et al., 1993). When making lime-silica AAC, ground quartz flour is mixed with lime (CaO), Portland cement and water to make a thin slurry. At the last moment, aluminum powder is added to the mixture. The mixture is then transferred to a massive steel mold wherein the slurry begins to foam. The foaming causes it to rise in the mold much like a cake leavened with baking powder will rise in an oven. After 45 minutes or so, the AAC “cake” occupies approximately 3-4 times as much space as it did when it was first poured. It has also developed enough green strength to be demolded, cut with wire saws to a variety of shapes (blocks and/or panels), and placed in a steam-heated autoclave. After treating at 180°C for 10-18 hours the block or panel is removed and ready for use. Densities are on the order of 400-600 kg/m$^3$ and compressive strengths range from 2 to 5 MPa (Aroni et al., 1993). AAC can also be made from Class F fly ash. The use of fly ash in this manner saves the AAC manufacturer the
time and cost of ball milling quartz sand and also keeps the fly ash out of the World’s landfills. However due to the $\text{Al}_2\text{O}_3$ content of the fly ash, using it instead of quartz does introduce a second phase in the final product, in addition to tobermorite fly ash based AAC contains aluminum substituted hydrogarnet (katoite).

In light of the above, the promotion of fly ash-based AAC as an environmentally friendly “green material” is reasonable. In addition to fly ash, AAC formulations can also include other by-product materials such as silica rich mine tailings and recycled glass cullet as well as alternate sources of lime and Portland cement including ground granulated blast furnace slag and cement kiln dust. In fact, it is possible to produce AAC entirely from these by-products. The latter substitutions are environmentally desirable in as much as the manufacture of Portland cement and lime require a great deal of energy and produce a great deal of carbon dioxide off gas.

Given the many advantages of AAC, the issue at hand at this point in time in North America is customer appeal, which is ultimately linked to cost. The outward appearance and livability of an AAC and conventional house are nearly identical, but the similarity ends there. An AAC house is self-insulating, fireproof, and insect and mildew resistant, while not containing known allergens such as VOCs or fiberglass. The current building industry is well established and much of the cost of a wood framed house is labor. In contrast, AAC walls (exterior and interior) can be assembled with thin-set mortar in a week or less. Window and door openings as well as channels for interior wiring and plumbing can be cut after the fact using carbide tipped woodworking equipment: hand saws, routers and the like. Outer wall finishes can be stucco or brick. Interior finishes can be conventional dry wall or plaster. A motivated “do-it-your selfer” could assemble an AAC house from a kit, much like a Sears house kit of the 1930s. AAC could make affordable housing a reality.
The overall objective of the currently funded work is to find a means of toughening AAC in order to make it less prone to breakage and edge damage that often takes place during handling and placing. The damage problem is costly, but even more important is the fact that the builder/customer is left with a negative impression that is hard to overcome. Adoption of a new building material in North America will take time as first impressions are replaced by more favorable ones. If the proposed work is successful, it will provide a deeper understanding of the underlying chemistry of AAC and the feasibility of the proposed toughening process. If implemented by the industry, the proposed technology could provide builders with a more robust AAC resulting in greater user satisfaction and possibly new market penetration, e.g. using AAC to adsorb energy under extraordinary circumstances (runaway trucks and/or blast protection).

The work described below examines the feasibility of using alkali activation of fly ash based AAC to bring about a phase change, and alternate methods of introducing cellular structure to the material. It is known that micrometer-sized wollastonite and xonotlite particles can be used to toughen cement pastes (Low and Beaudoin, 1993a and b). Thus it was hypnotized that in situ growth of well dispersed micrometer sized zeolites in place of larger katoite crystals normally present in the matrix could serve a similar purpose. Sodium hydroxide (NaOH) was used to supply the needed alkali. At first all formulations contained only Class F fly ash and small amounts of either a concentrated NaOH solution or a molar equivalent Ca(OH)$_2$ slurry. These samples were precured and then autoclaved. The data were used to contrast behavior of materials resulting from a conventional pozzolanic reaction compared with those resulting from an alkali activated reaction. Due to the difficulty of foaming alkali containing mixtures with aluminum powder, cenospheres derived from Class F fly ash were used in a second series of samples to lower the density of the final product. The cenospheres imparted voids as well as a
degree of robustness to the mixtures, which made it possible to use shear mixing to prepare a reduced density material. Finally Portland cement was added to the alkali containing mixtures. It was anticipated that phase behavior would parallel those in a traditionally foamed AAC and that findings would be directly applicable to conventional AAC manufacturing. It was also anticipated that the new processing techniques employed to make reduced density AAC-like materials might possibly lead to the development of a new technology and new markets.

Experimental Method and Results

100% Fly Ash and Cenosphere Samples

100% Class F fly ash (Ft. Martin-Allegheny Power-Table 1) was mixed with just enough 12 M NaOH containing a few drops of superplasticizer (Mighty RD-1) to make a thick (modeling clay-like consistency) paste. The paste was manually kneaded, much like bread dough, on a counter top until smooth and then it was compacted into a 2-inch mineral oil coated

Table 1. Composition and Characteristics of Starting Materials (wt %)

<table>
<thead>
<tr>
<th>Component</th>
<th>Ft. Martin Fly Ash</th>
<th>Bruce Mansfield Cenospheres</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO&lt;sub&gt;2&lt;/sub&gt;</td>
<td>52.74</td>
<td>55.</td>
</tr>
<tr>
<td>TiO&lt;sub&gt;2&lt;/sub&gt;</td>
<td>-nd-</td>
<td>1.27</td>
</tr>
<tr>
<td>Al&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>24.31</td>
<td>34.</td>
</tr>
<tr>
<td>Fe&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>11.47</td>
<td>2.75</td>
</tr>
<tr>
<td>Cr&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>-nd-</td>
<td>0.04</td>
</tr>
<tr>
<td>CaO</td>
<td>3.36</td>
<td>0.09</td>
</tr>
<tr>
<td>MgO</td>
<td>0.81</td>
<td>0.34</td>
</tr>
<tr>
<td>MnO</td>
<td>-nd-</td>
<td>0.06</td>
</tr>
<tr>
<td>ZnO</td>
<td>-nd-</td>
<td>0.02</td>
</tr>
<tr>
<td>Alkalis</td>
<td>1.87</td>
<td>3.37</td>
</tr>
<tr>
<td>SO&lt;sub&gt;3&lt;/sub&gt;</td>
<td>0.70</td>
<td>-nd-</td>
</tr>
<tr>
<td>105°C H&lt;sub&gt;2&lt;/sub&gt;O</td>
<td>0.20</td>
<td>-nd-</td>
</tr>
<tr>
<td>LOI</td>
<td>1.92</td>
<td>-nd-</td>
</tr>
<tr>
<td>#325 Sieve</td>
<td>24.90</td>
<td>-nd-</td>
</tr>
<tr>
<td>Density (g/cc)</td>
<td>2.37</td>
<td>-nd-</td>
</tr>
<tr>
<td>Strength at 28 days (%)</td>
<td>79.75</td>
<td>-nd-</td>
</tr>
</tbody>
</table>

-nd- not determined.
brass cube mode. The sample was troweled off and then covered with a glass plate. The mold was placed in a 38°C walk-in curing chamber overnight. After precuring, it was demolded and then autoclaved at 180°C for an additional 16 hours. The compressive strength of the sample was 59 MPa, which is similar to the strength reported by Palomo et al. (1999) for a similar material. The X-ray diffraction pattern of the alkali activated fly ash sample (see Figure 1a) indicates that it contains residual glass (amorphous hump centered at ≈22°2θ), residual quartz and mullite.

Figure 1. X-ray diffraction patterns of samples made from fly ash and cenospheres cured as a function of temperature. (M = Mullite, Q = Quartz, NaP1 = Na-P1, HS = Hydroxysodalite, G = Gmelinite, C=Na-Chabazite, A=Analcime.)
crystalline zeolites (Na-P1, hydroxysodalite and Na-chabazite), and X-ray amorphous
tectosilicates (amorphous hump centered at \( \approx 29^\circ 2^\theta \)). The accompanying microstructure for this
sample is given in Figure 2. The matrix is dense and exhibits very little evidence of crystal
growth at both levels of magnification (Photos 2A and 2B). The matrix seems more “gel-like”
than crystalline, a characteristic in keeping with its strength and X-ray diffraction pattern.

Figure 2. Microstructure of autoclaved 100% Class F fly ash sample made with 12M NaOH.
Original magnifications: (A) 2000X and (B) 10000X.

Due to the high specific gravity of the sample (~1700 kg/m\(^3\)), a second sample was made
using 100% cenospheres derived from a Class F fly ash. These are commercially available from
First Energy’s Bruce Mansfield plant available through US Natural Resources, Inc. (Table 1).
The cenospheres were mixed with the same 12M NaOH (containing Mighty RD-1) used
previously and again kneaded by hand with the caveat that a few extra grams of NaOH solution
were necessary to attain a similar “play dough” consistency. The mix, which now occupied
about twice as much volume, was packed into two 2-inch mineral oil coated brass molds rather
than one, and again precured at 38°C overnight. After precuring, the two cubes were demolded.
One was autoclaved for an additional 16 hours at 180°C and the other was placed on a platform in a desiccator containing water and cured at 90°C over night (~16 hours). Interestingly, the 90°C sample was twice as strong (2 MPa) as the 180°C autoclaved sample (1 MPa). X-ray diffraction data for these two samples are given in Figures 1b and 1c. The 90°C sample (Figure 1b) is generally devoid of secondary crystalline zeolites, save a trace of hydroxysodalite. Instead the pattern is dominated by a large X-ray amorphous hump with superimposed peaks representing residual quartz and mullite found in the Class F fly ash starting material. More salient is the shift of the maximum of the amorphous hump from ≈22°2θ to one centered near ≈29°2θ. This shift is a result of the development of a sodium-modified hydroxylated tectosilicate network structure (zeolite-like) in the sample during the curing process. These precursors represent nanoscale structures that will ultimately crystallize at higher temperatures. The X-ray diffraction pattern for the 180°C sample (Figure 1c) contains significantly more crystalline material than its 90°C counterpart. Autoclaving at 180°C caused most of the glass in the cenospheres to react (dissolve) and nucleate and grow into crystalline zeolites: Na-P1, hydroxysodalite, gmelinite, Na-chabazite and analcime. The phase development is similar to that observed for the 100% fly ash sample (Figure 1a), but it is better developed and the sample as a whole contains less residual glass. It is worth noting however that the sample cured at 90°C was stronger than the autoclaved sample. These differences are reflected in their microstructures (see Figure 3). Photos A and B represent a sample precured at 38°C followed by curing at 90°C overnight. The cenospheres show varying amounts of dissolution due to the high alkaline mixing solution, but significant amounts of residual glass still remain (note cross section of cenospheres in Photo A). The cenospheres are covered with a continuous “gel-like” coating. It is suggested that this coating is holding the cenospheres together. Photos C and D are
micrographs of a 100% cenosphere sample precured at 38°C followed by autoclaving at 180°C overnight. The dissolution and subsequent reaction of the cenospheres is much more evident in this sample. The shells now consist entirely of zeolite crystals. Massive crystals are also evident in Photo D. The formation of zeolite crystals seems to have a negative impact on the compressive strength of the autoclaved sample.

Figure 3. Micrographs of a 100% cenosphere sample cured at different temperatures. (samples A and B) were precured at 38°C and cured at 90°C, and (samples C and D) were precured at 38°C and autoclaved at 180°. Original magnifications: (A,C) 250X and (B,D) 2000X.

Apparently the glassy cenospheres are better held together by the precursor gel that formed at 90°C, the lower reactivity at 90°C provides more continuous coverage and more strength than the nearly completely zeolitized sample. The 180°C cured samples are almost
totally crystalline and have a very porous, almost lacy structure that seems to impart weakness rather than strength. The specific gravity of the 90°C cured sample was 618 kg/m³; a conventional AAC sample would have been much stronger. As a result of the very low compressive strength, it was decided to examine a 50:50 blend of fly ash and cenospheres as a mean of providing strength and a more moderate density.

50:50 Cenosphere:Fly Ash Samples

A 50:50 by weight fly ash/cenosphere sample was again mixed and kneaded by hand as described above. In this instance, overnight precuring took place in the brass mold at 90°C instead of 38°C. Two samples were prepared. One was tested after curing at 90°C overnight, whereas the other sample was autoclaved at 180°C for an additional 16 hours. Unlike the earlier samples, analysis of these X-ray diffraction patterns indicated that curing temperature had made a significant difference in the phase development of the two samples. See Figure 4. The 90°C sample contains residual glass and residual quartz and mullite. The pattern is dominated by a large X-ray amorphous hump centered at $\approx 27\text{-}28^\circ 2\theta$. Only trace amounts of hydroxysodalite can be detected. See Figure 4a. The 90°C plus 180°C autoclaving sample was radically different. Although it still contains a trace of mullite and hydroxysodalite, the quartz peak and most if not all of the amorphous background are gone. The pattern is more crystalline. See Figure 4b. The sample consists of nearly pure Na-P1 zeolite with small amounts hydroxysodalite and residual quartz and mullite.

Micrographs for the two 50:50 samples are given in Figure 5. The difference in density of the samples is quite obvious from the micrographs. The appearance of the samples at 250x magnification closely resembles that of AAC. At higher magnifications (2000x and 1000x)
however, the not so obvious differences become quite apparent. The 90°C sample (Figure 5A and 5B) contains a significant amount of residual glass and open porosity. The 90°C/180°C cured sample (Figure 5C and 5D) is considerably more crystalline. Crystals are part of the matrix and fill once open pores. Using a blend of fly ash and cenospheres could provide an alternate method of developing a cellular structure on the microscale.

![X-ray diffraction patterns of 50:50 samples of fly ash and cenospheres cured under different conditions.](image)

Figure 4. X-ray diffraction patterns of 50:50 samples of fly ash and cenospheres cured under different conditions. (M = Mullite, Q = Quartz, NaP1 = Na-P1, HS = Hydroxysodalite).
The interesting outcome, which may be due to a phase change, is the radical difference in compressive strength of these samples. Although specific gravities were nearly the same (924 kg/m$^3$ (90°C) versus 976 kg/m$^3$ (90°C/180°C), the strength fell from 21 MPa for the 90°C sample to 9 MPa when the 90°C sample was subsequently autoclaved at 180°C. It is suggested that the 90°C cure caused the sample to develop initial strength and X-ray amorphous precursor phases.

![Figure 5. Comparison of microstructure of a 50:50 fly ash:cenosphere sample cured at various temperatures.](image)

that subsequently crystallized at 180°C. The crystallization process disrupted the continuity of the structure resulting in the lower strength of the 180°C sample. Although the 9 MPa value is still in keeping with the expected values for a 900 kg/m$^3$ sample (Aroni et al., 1993), the
posibility of manufacturing a stronger material at 90°C seems attractive because an autoclave would not be needed to cure the material.


In conventional lime based AAC, tobermorite is the phase responsible for strength. Due to the complete lack of crystalline tobermorite in the above mixtures, it was decided to add Portland cement to the mixture in order to add increase the calcium oxide content enough to produce tobermorite along with the zeolite. The recipe chosen contained the following materials:

- 40g Portland cement (Type III) Southdown Plant, Wampum, PA.
- 40g First Energy - Bruce Mansfield cenospheres (US Natural Resources)
- 100g Ft. Martin fly ash (Allegheny Power)

The dry ingredients were pre-blended and then mixed with 20g NaOH mixed in 40g H₂O containing a few drops of Mighty RD-1 (~12 M NaOH). A few extra drops of the ~12 M NaOH solution was added during the mixing and kneading process to obtain the same workability as above. For the sake of comparison, a duplicate sample was made in which the NaOH was replaced by a molar equivalent of CaO. In this instance 14g CaO was added to 40 g of DI water (containing a few drops of Mighty RD-1) in order to slake the lime prior to mixing. The dry ingredients were then mixed with the liquid and the resulting paste kneaded as before. A few grams of extra water were added to make a workable clay-like paste. Both mixtures were placed in glass covered 2-inch brass cube molds and precured overnight at 90°C. Both samples were already extremely hard on de-molding. Both 90°C cured samples were then autoclaved at 180°C overnight (approximately 16 hours). The specific gravity before and after autoclaving did not change significantly: Final values were approximately the same as their initial ones, i.e. 1526 kg/m³ (NaOH sample) vs. 1335 kg/m³ (CaO sample), suggesting that water vapor from the
autoclave did not contribute water to the hydration reaction to any great extent. The X-ray diffraction patterns for the NaOH autoclaved sample are dominated by analcime while the CaO autoclaved sample consists primarily of tobermorite and partially silica substituted hydrogarnet (katoite). Both samples contain residual mullite and quartz. See Figure 6.

![X-ray diffraction patterns](image)

**Figure 6.** X-ray diffraction patterns of Portland cement containing mixtures (40:40:100) cured at various temperatures. (T=Tobermorite, K = Katoite, A = Analcime, Q = Quartz)

Microstructure of the samples are given in Figure 7. At low magnifications the samples look alike (A and C), but at higher magnifications the NaOH sample (B) appears massive and tends to contain zeolites growing inside of cenosphere cavities. The CaO sample (D) ends to
consist of a lower porosity matrix that contains less cenosphere casts and little if any zeolites. Instead it is dominated by abundant tobermorite crystals filling space and leaving little porosity. Strength behavior was as follows: the NaOH-containing sample maxed out at 14 MPa. The companion lime-containing sample reached 32 MPa. If correct, the lower strength of the 90°C precured NaOH sample might be attributed to the development of precursor phases at 90°C that subsequently changed to analcime on curing to 180°C. The phase change could cause matrix weakness if there is a volume change associated with it. Conversely, the CaO containing sample developed tobermorite at 90°C and continued to improve its crystallinity and strength at 180°C, no phase change occurred. In this instance, the tobermorite sample is about twice as strong as one might expect for an AAC sample having a similar density (Aroni et al., 1993).

Figure 7. Comparison of microstructure of 40-40-100 samples made with NaOH and Ca(OH)$_2$. (A,C) 200X and (B,D) 2000X.
In order to test this hypothesis, the same two samples were prepared again, but this time they were precured overnight at 38°C instead of 90°C prior to autoclaving. X-ray diffraction traces are given in Figure 8. Phase development for the lime-containing sample (Figure 8b) is identical to that for the 90°C precured sample depicted in Figure 6b. Both traces contain tobermorite and katoite. The NaOH sample (Figure 8a) is also nearly identical, containing mostly analcime but now also a trace of hydroxysodalite. In both cases the change in precuring
temperatures did not affect phase development to any great extent, but it radically affected the development of strength. The NaOH sample maxed out at 28 MPa and the lime sample attained 16 MPa, a complete reversal in behavior compared to the 90°C precured samples. The microstructures of the samples are given in Figure 9. They are significantly different from their counterparts given in Figure 7. Cenospheres and most of the fly ash particles in the NaOH containing sample (Figure 8A and 8B) have reacted and been replaced by a large quantity of irregular crystalline masses. The lime-containing sample consists of less well defined tobermorite crystals (Figure 8C and 8D) compared to its 90°C precured counterpart presented earlier. The NaOH cured sample is rather interesting. All traces of cenospheres are gone and only a few fly ash spheres remain. The matrix is dominated by small irregular crystalline masses.
that must interlock on the microscale and provide the increased strength noted for this sample. It is suggested that precuring at 38°C allowed significant dissolution and gel formation and that the gel subsequently nucleated and crystallized when heated to 180°C. Crystallization normally does not occur at 38°C, but, when the sample was precured at 90°C rather than 38°C, the nucleation and growth process occurred directly without the formation of a large amount of gel phase. This apparently reduced the degree of reactivity and the total amount of zeolite that formed. The sample still contains glassy material and an AAC like structure, which is not as strong. The CaO sample appears much like its Figure 7 counterpart except it was harder to find distinct tobermorite crystals. In addition, once they were identified, they did not exhibit the same degree of crystallinity as the sample precured at 90°C (compare Figures 7D and 9D).

Unexpectedly, the 38°C cured CaO-cured sample was half as strong as its 90°C counterpart. It is suggested that a lime rich sorosilicate C-S-H formed initially at 38°C and then subsequently crystallized to tobermorite when it was heated to 180°C (Sato and Grutzeck, 1992). C-S-H is the normal precursor phase for tobermorite, but in this instance the sample had already developed enough strength that the conversion process disrupted the matrix. Normally AAC is autoclaved within a few hours of demolding while it is still relatively soft. This sample had cured overnight at 38°C and was already quite hard prior to autoclaving. The specific gravity of the 38°C sample (1500 kg/m³) was slightly lower than the sample precured at 90°C (1526 kg/m³). This is not really significant, and a priori one would not anticipate the drop in compressive strength from 32 MPa to 16 MPa unless some other mechanism were operative. It is suggested that the 38°C precure had locked in some other phase, which changed on further heating; it is suggested that C-S-H was this phase. The increase in strength of the NaOH sample seems justified, in light of our
previously discussed hypothesis. Analcime formed directly from a well-developed gel having little strength, therefore the matrix was not disrupted.

CONCLUSIONS

Autoclaving shear mixed samples containing fly ash and/or cenospheres mixed with concentrated sodium hydroxide solution provides a means of producing a range of materials having varying densities and strengths that are based upon the development of zeolites as the binder phase. Although the objective of the work was to produce both tobermorite and zeolites at the same time, this has not yet been achieved. Further experiments are in progress. However, it was shown that shear mixing a paste formulated with Class F cenospheres, Class F fly ash, Portland cement and either concentrated lime water or sodium hydroxide solution allows one to make an extrudable AAC-like material without a foaming step. The material contains voids similar to those found in AAC that result from the added cenospheres, but the voids occur at the micrometer scale. Density is reduced even though MDF mixing technology is employed.

Although precuring conditions did not affect phase development, it did affect microstructure and strength. When NaOH is used, precuring at 38°C prior to autoclaving allows for maximum aluminosilicate gel formation prior to crystallization. Nearly all of the cenospheres and fly ash particles are consumed during this process. If the sample is activated with CaO, an initial precure at 90°C seems to provide greater strength.

The following explanation for the observed behavior of the CaO system is based upon a new model for the hydration process (Grutzeck, 1999). When a lime-rich sample is precured at 38°C, the lime will react with the fly ash and form a predominantly dimeric sorosilicate C-S-H on the surfaces of the fly ash. At 90°C, the situation is similar, but because of the lowered
solubility of Ca(OH)$_2$ at elevated temperatures and the fact that sorosilicate C-S-H is not stable at temperatures above 80°C, dreierkette chains found in tobermorite form directly without the sorosilicate intermediary that forms at 38°C. Upon autoclaving, the 90°C precured sample will continue to build crystallinity of the already existing phase (tobermorite). The situation at 38°C is slightly different in as much as autoclaving necessitates the change of the original sorosilicate C-S-H into one dominated by chains (tobermorite).

In the case of the NaOH sample, precuring tends to allow the solution phase to dissolve significant quantities of alumina and silica from the fly ash and at the same time depress the Ca$^{2+}$ ion solubility. Once in solution, the solvated sodium aluminosilicates ions tend to combine and the form insoluble gels. When heated the gel tends to crystallize as zeolites. Both samples formed precursor gels, but in the case of the 38°C sample, the process was nearly complete. The gel occupied formally open spaces and, once it crystallized, became a rather continuous fine-grained matrix. The sample precured at 90°C crystallized almost immediately without significant gel formation. This caused much of the sodium to enter the zeolite structure at early times while much of the glassy phase still remained. The zeolites tend to form coatings on the surfaces of cenospheres and fly ash grains rather than consume them.

It is concluded that the precuring step plays a significant role in determining early microstructural development of these materials, which in turn ultimately impacts the performance of the final product. In a lime-based AAC, it is suggested that a 90°C precure could in fact increase the strength of the final product by a factor of two. Likewise in an alkali activated zeolite-containing system a 38°C precure leads to similar two-fold increase in strength versus a precure at 90°C.
REFERENCES


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