

## CLASS C FLY ASH ACTIVATED BY LOW ALKALINITY ACTIVATOR WITH CONTROLLED SETTING

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The American Society for Testing and Materials (ASTM) standard C618 recognizes two major classes of fly ashes, Class C and Class F. The lower limit of ( $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) for Class F fly ash (FFA) is 70 wt.% and for Class C fly ash (CFA) 50 wt.%. Typically, CFA has a higher calcium oxide content (20 to 30 wt.%) than FFA. CFA usually displays self-cementing behavior by reacting with water to produce calcium silicate and calcium aluminate hydrates. Hydration of CFA exhibits rapid stiffening and fast setting, yielding lower compressive strength. Recently, there has been an increased interest in alkali activated high-Ca fly ashes. Alkali silicate activated high-Ca fly ash-based geopolymers exhibit good strength and durability. The time of setting of a geopolymer paste decreases exponentially with increasing CaO content in the fly ash. Above 10 wt.% CaO, the time of setting is typically less than 30 min.

In this paper, we report on Class C fly ash activated by low alkalinity activator with controlled setting. The fly ash contained about 83.0% glass, 6.7% tricalcium aluminate ( $\text{C}_3\text{A}$ ), 5.3% quartz, 3.3% periclase, and 1.7% lime. w/CFA ratio was fixed at 0.32. Tests were conducted with the following compositions (by weight of CFA): 1. Water only; 2. 2% NaOH; 3. 2% NaOH and 2%  $\text{Na}_3\text{PO}_4$ ; 4. 2% NaOH and 4%  $\text{Na}_3\text{PO}_4$ ; 5. 2% NaOH and 6%  $\text{Na}_3\text{PO}_4$ ; and 6. 2%  $\text{Na}_3\text{PO}_4$  only. 2% NaOH (~1.6 M) was employed as the alkali activator and  $\text{Na}_3\text{PO}_4$  as the sole set retarder. Mortar and paste samples were characterized for set times by AutoVicat, heat of hydration by isothermal calorimetry (IC), compressive strength, and reaction products by XRD and SEM/EDS. In addition, the set retarding mechanism was explored by a set of leaching experiments (compositions #1, #2 and #3) with w/CFA at 40.

Both water and 2% NaOH activated CFAs exhibited flash setting (<14 min). Initial set time increased with increasing  $\text{Na}_3\text{PO}_4$  while NaOH was fixed at 2%. The initial set was as long as 177 min while  $\text{Na}_3\text{PO}_4$  increased to 6% (Composition #5). With 2%  $\text{Na}_3\text{PO}_4$  alone, the initial set was 322 min (Composition #6). Our studies reveal strong set retarding effect of  $\text{Na}_3\text{PO}_4$  is related to instant removal of calcium cations by precipitating apatite during early stages of hydration or alkali activation.

XRD and SEM/EDS results show stratlingite is the primary secondary phase and it occurs in clusters embedded in the (C,N)ASH gel in the 2% NaOH activated CFAs.

IC results for hydration of CFA in water show an initial heat flow peak related to rapid hydration of  $\text{C}_3\text{A}$  present in CFA at ~55 min with acceleration starting from ~10 min and a main heat flow peak related to stratlingite formation occurring at ~300 min. The initial peak shifted to 10 min in the 2% NaOH activated CFA while the main peak was missing. When  $\text{Na}_3\text{PO}_4$  was introduced in the 2% NaOH activated CFA compositions (#3, #4, and #5), heat flows for both the initial and main peaks were greatly reduced. Apparently, a greater extension of set time by  $\text{Na}_3\text{PO}_4$  is related to a greater reduction of the rate of katoite.

We have observed a strong synergistic effect of NaOH and  $\text{Na}_3\text{PO}_4$  on compressive strength development. The 2% NaOH activated CFA exhibited flashing setting with a low compressive strength of 13.9 MPa (56 days), whereas compressive strength was as high as 65 MPa (56 days) with a set time extended to 177 min while  $\text{Na}_3\text{PO}_4$  was amended. This synergistic effect can be explained by precipitation of apatite to removed calcium cations by  $\text{Na}_3\text{PO}_4$  during the earliest curing stages, resulting in a great extension of set time and an increased concentration of NaOH in the pore solution that lead to accelerated alkali activation of CFA during later curing times.

The carbon dioxide emission was estimated to be ~45kg  $\text{CO}_2\text{e/kg}$  binder as compared to 820kg  $\text{CO}_2\text{e/kg}$  for Portland cement. Low alkalinity activated CFA represents a low cost, ultralow carbon, high strength alkali activated fly ash material.