Construction and Building Materials 67 (2014) 20-28

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Effect of initial water content and curing moisture conditions on the development of fly ash-based geopolymers in heat and ambient temperature

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HIGHLIGHTS

• Alkali activated fly ash geopolymers are synthesized at room temperature.

• Curing moisture conditions after hardening are controlled in various methods.

- Low initial water content plus low moisture condition during curing are beneficial.
- Samples got similar strength levels to the heated ones in a relatively short time.

ARTICLE INFO

Article history: Available online 2 December 2013

Keywords: Fly ash Geopolymer Water Curing X-ray diffraction

ABSTRACT

It has been claimed that water has no role to play in the formation of hardened geopolymer paste apart from providing the workability and necessary reaction medium. It has further been generally accepted that heat treatment is necessary for producing fly ash geopolymer concrete. This is considered a drawback affecting its manufacture and feasibility. The research presented in this paper was conducted in the aim of improving the ambient-cured (20 °C) geopolymer. This has been achieved by controlling the moisture condition of the specimens after hardening. This paper also examines whether there are mechanical effects as a result of variation in the initial water content. It further explores whether a combination of such variation together with controlling the curing regime may lead to desired improvement in the mechanical properties. The role of water was studied by monitoring the microstructural and mineralogical developments and relating them to the gain in strength. It has been found that reducing water content in the initial state and controlling the moisture condition during a curing period after hardening are beneficial and lead to extra strength gain in the ambient-cured fly ash-based geopolymer. This helps the ambient-cured geopolymer achieve strength levels comparable to those of heat treatment within a relatively short curing period.

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1. Introduction

'Geopolymer' is a given name to describe a new class of synthetic materials. According to the newest definition by the Geopolymer Institute, geopolymer refers to the ambient X-ray amorphous materials that are composed of mineral molecule chains or 3-D networks linked with covalent bonds [1]. In this paper, the aluminosilicate-based type of geopolymer that is dominantly composed of SiO₄ and AlO₄ tetrahedra was investigated. The empirical formula given for this type of geopolymer is $M_n(-(SiO_2)_z-AlO_2)_n$, wH₂O. In this formula, *n* is the degree of polycondensation; M is the monovalent cation like K⁺ or Na⁺; *z* is the ratio of Si/Al and can be 1, 2 or 3. This geopolymer can be synthesized from fly ash and alkali activating solution. Davidovits summarised the basic principles of the synthetic reactions and hardening process by two series of equations as follows [2]:

Series 1:

$$(Si_2O_5, Al_2O_2)_n + 3nH_2O \xrightarrow{\text{NaOH/KOH}} n(OH)_3 - Si - O - Al^-(OH)_3$$
(1)

$$n(OH)_{3}-Si-O-AI^{-}(OH)_{3} \xrightarrow{NaOH/KOH} (-SiO-O-AI^{-}O-O_{n})_{n}$$

+ 3nH₂O (2)







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^{0950-0618/\$ -} see front matter © 2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.conbuildmat.2013.10.047

Series 2:

$$(Si_2O_5, Al_2O_2)_n + 2nSiO_2 + 4nH_2O$$

$$\xrightarrow{\text{NaOH/KOH}} n(OH)_3Si-O-Al^-(OH)_2-O-Si(OH)_3$$
(3)

$$\begin{array}{c} n(OH)_{3}Si{-}O{-}Al^{-}(OH)_{2}{-}O{-}Si(OH)_{3} \\ \xrightarrow{NaOH/KOH} (-SiO{-}O{-}Al^{-}O{-}O{-}SiO{-}O{-})_{n} + 4nH_{2}O \end{array} \tag{4}$$

Under the strong alkali attack, the silicon and aluminum oxides from the fly ash are dissolved and form into the aluminosilicate ions shown in the first equation of each series above. At this moment, the mixture appears to be a gel or dough-like phase. The aluminosilicate ions accumulate and form into polycondensed products and water as shown in the second equation of each series. These polycondensed products could precipitate from the original gel or dough-like phase and turn into hardened 3-D molecule networks, which provides the required strength and durability for geopolymer products [3].

The aluminosilicate-based geopolymer has been recommended as a new construction material performing in a similar way to cement in concrete. More advanced properties derived from geopolymers, such as higher early strength, better thermal and chemical resistance, much less CO₂ emission and more efficient consumption of fly ash wastes, provide a possibility to replace the traditional Portland cement in future sustainable concrete production [3–6]. However, this aluminosilicate geopolymer which is derived solely from fly ash as the solid base has been seen to always require heat treatment at an early stage so as to achieve the desired strength and form. This might be caused by the fact that fly ash needs higher activation energy, and that heat also helps the hardening of the geopolymer as a type of thermosetting material [3,7,8]. Such heat treatment could be considered a drawback affecting ease of manufacture, economy and consequently the feasibility of this new construction material. Investigations have been carried out to find a way to avoid heat treatment. For example, mechanical activation by high energy milling has been used to reduce the fly ash particle's size for achieving improved reactivity [9]. Reactive solid precursors like slag were also tried as additives in order to produce geopolymers at room temperature [10]. But these methods also bring in new financial burdens or more complex manufacturing processes.

The research presented in this paper describes a new direction in the production of fly ash-based geopolymers. This is achieved by introducing a moisture controlled curing condition. The water effect was initially related to the workability of the fresh mix [7,11]. Recent researches have been more concerned about the water's effect on the final hardened products. It was found that water could exist inside the final products as free or bound water, and thus had an influence on the microstructure [12]. However, there is scarcely any investigation of the water effect during the developing period from the time of the geopolymer being in a freshly mixed state until hardening is achieved. As the previous Davidovits' reaction model assumed [2], water acts as the medium and also participates in several intermediate reactions during the geopolymer formation such as the dissolution, polycondensation, etc. For these intermediate reactions, the variation of water amount could affect the reaction equilibrium. This has been supported by the measurement of heat evolution of metakaolin-based geopolymers, which showed that abundant water was beneficial for the dissolution and hydrolysis part but harmful to the polycondensation part [13]. Curing under a low relative humidity environment has also been found to have positive effect with the mild heated metakaolin-based geopolymers [14]. These observed effects imply that water may be engineered to affect the progress of geopolymer reactions in ways that can possibly be as important as those attributed to the activators.

In this paper, the effect of water was researched to see if it could be utilized to improve the growth of the ambient-cured fly ashbased geopolymers. The geopolymer synthesis was performed such that a specified water amount was added as part of the mixture and a certain specified curing regime was applied for a certain period after hardening. The evaluation of the water's role was done by observing the relative characteristics of strength, microstructural and mineralogical development under different curing conditions. These characteristics were also compared with the heat-cured specimens to find out whether a certain water amount and a specific curing regime could result in similar accelerating effect as heat does.

2. Experimental details

2.1. Materials

Geopolymer pastes made with fly ash and alkali solutions were investigated here. The fly ash used was obtained from Eraring thermal power station located 30 km southwest of the Newcastle, New South Wales of Australia, and purchased from Boral Corporation. The chemical composition of the fly ash is listed in Table 1. The low content of calcium oxide indicates that this fly ash belongs to Class F category. For the liquid base, NaOH flakes (>98% purity), Grade D Na₂SiO₃ solution with the weight ratio of SiO₂/Na₂O = 2 (29.4% SiO₂, 14.7% Na₂O), and deionized water were used to prepare the alkali solutions. The specific gravity values of all the used ingredients are listed in Table 2.

2.2. Mix designs

To control the diversity of water content of each design as required by this research, the parameter 'Water-to-Geopolymer Solids Mass Ratio' (W/G) proposed by Rangan is also introduced. The 'Water' refers to the mass of H_2O brought from the activator. The 'Geopolymer Solids' term refers to the total mass of the materials in solid phase and the solid part in the liquids (the solutes), that is: the mass of fly ash, Na₂O and SiO₂ [7]. In this research, the mass ratio of fly ash to activator solutes in the 'Geopolymer Solids' was made constant for all the mixtures. Thus, the variation in the *y* values would cause variation in the (W/G) values, leading to different water contents. With known (W/G) values, the relevant 'Fly Ash-to-Activator Mass Ratio' (F/A) that commonly applied to geopolymer practical mixes would be obtained [7]. The calculated results of the three mix designs are listed in Table 3. For every 100 kg fly ash, 4.144 kg NaOH flakes and 33.434 kg silicate solution were always added, but the water amounts were varied.

2.3. Specimen preparation

Initially, the activator was prepared through a two-step method. At first, the NaOH solution with specified concentration was prepared by mixing the specified mass of NaOH flakes and deionized water displayed in Table 3. The mixing was done manually, using the appropriate necessary protective measures. The concentration of the prepared NaOH solution was 14.3 M, 10.8 M and 8.7 M, when *y* (explained in the previous section)=11, 12 and 13, respectively. The mixed solution was placed without disturbance for at least one day to cool down and become uniformly homogeneous. The NaOH solution was sealed to prevent the reaction with CO₂ from the atmosphere. The final phase of the activator preparation was done by adding Na₂SiO₃ solution into the prepared NaOH solution just before the geopolymer synthesis. The activator was then stirred manually for 3–5 min and kept still for further 5 min to become uniform.

After the materials preparation, the activator was poured onto the dry fly ash and mixed using a Hobart Mixer for 5 min. The mixture displayed a uniform phase with certain viscosity which resembled that of a slurry or plasticine depending on the different water contents. Then the mixture was cast into 50*50*50 mm plastic Download English Version:

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