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Characterization of mechanical and electric properties of geopolymers synthesized using four locally available fly ashes

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HIGHLIGHTS

- Geopolymers have been synthesized using fly ashes from four different power plants.
- Similar in chemical composition in fly ash doesn't lead to similar strength.
- Early age electric resistance of geopolymers is determined by the reaction rate.
- Late age electric resistance of geopolymers is controlled by the chemical composition.

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ABSTRACT

Geopolymer can be synthesized using industrial wastes such as fly ash, making it a promising material to replace Ordinary Portland Cement (OPC) as a green binder for concrete materials. One major barrier to prevent the wide application of geopolymer is that its properties heavily depend on the source materials. Aiming to evaluate the effect of fly ash on the properties of synthesized geopolymers, this study used four carefully selected fly ash to synthesize geopolymers. Comprehensive experimental programs have been carried out to characterize both the mechanical and electrical properties of these geopolymers. Mechanical testing shows that similar chemical composition in the source materials doesn't guarantee similar compressive strengths of the produced geopolymers, and that high content of calcium doesn't always lead to high compressive strength. Electrical characterization shows that both the magnitude of the electrical impedance and the electrical resistance of the geopolymers at early age measured at frequency of 10 kHz increase with the curing time. It has also been found that at the early age, the electric resistance of the geopolymers is mainly determined by the reaction rate. At the late age, this electric resistance is mainly controlled by the chemical composition. Measurements over one year show that the bulk electric resistances or the diameters of the high frequency arcs of all geopolymers increase with age during the one-year testing period, suggesting that the geopolymerization is a very long process.

1. Introduction

Geopolymers are amorphous three-dimensional alumino-silicate binder materials. They can be synthesized by mixing source material (alumino-silicate reactive materials such as metakaolin, fly-ashes) and an alkaline activator (strong alkaline solutions such as NaOH or KOH) and then curing at room or elevated temperature. Compared with ordinary Portland cement (OPC), geopolymers possess the following advantages: (a) less energy consumption and CO_2 emission during manufacture, (b) higher strength and much less shrinkage, (c) lower permeability (comparable to natural granite), and (d) substantially higher resistance to fire and acid attacks. Geopolymers are considered as "green materials" and strong candidates to replace OPC in selected applications leading to more sustainable infrastructure system.

Although geopolymer possesses many advantages compared with OPC, it suffers from a major drawback: its properties largely depend on its source materials such as fly ashes, which vary from source to source in either physical properties, like particle sizes, amorphous proportion, or chemical compositions. Even from the same source, these properties could change significantly too. The difference in the properties of fly ash is resulted from the difference in coals and the way to burn the coal. Therefore, no standard method exists which can predict the properties of geopolymers based on their mix design. In this study, a compressive experimental program has been carried out to synthesize and characterize geopolymers made from fly ashes from four power plants in and

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near Alabama. Among these four fly ashes, two are low-calcium fly ashes with very similar chemical composition, allowing us to test whether similar chemical composition in fly ash can produce similar properties in the produced geopolymers. Another two are fly ashes with higher calcium content so that the effect of the calcium content, which plays an important role in the properties of the resulted geopolymer, can be examined in this study too.

The effects of these fly ashes on the properties of the produced geopolymers are examined by characterizing their mechanical and electrical properties. Compressive strength is the major mechanical property measured in this study, as shown in many existing studies. The electrical properties of geopolymers, which have received very little attention in the literature, are also measured. According to existing studies on the electrical properties of OPC [1], the electrical conductivity or resistivity of cement paste is mainly caused by the ion transport through the pore solution in OPC-based concrete, which mainly depends on both pore solution conductivity and porosity. Both the pore solution conductivity and porosity evolve with the hydration of cement, which is a dissolution-precipitation process. At the initial stage of the hydration, the conductivity would increase with the release of the ions from the cement. As the hydration develops, the solid structure would hamper the movement of the charge carriers and the free water in pores used to be connected would be separated, which would decrease the conductivity, as observed in experimental studies [2,3]. Therefore, electric properties measurement is an effective method to study cementitious materials at both the micro- and macroscale. Both the fixed frequency and spectral measurements can be used to study the electrical properties of concrete, as summarized by McCarter et al. [6]. It is reasonable to assume that these methods are also applicable to geopolymers. The electrical properties of geopolymers largely depend on free alkali metal ions. However, very few detailed studies have been reported on the electrical properties on geopolymer. Cui et al. [7] investigated the conductivity of geopolymers made with different mix ratios. The measured AC conductivity of the harden geopolymer is 1.5×10^{-4} S m^{-1} at room temperature. Hanjitsuwan et al. [8] measured the electrical conductivity and dielectric constant of fly ash-based geopolymer pastes and found that they are dependent on the frequency range and liquid alkali to ash ratio.

In this study, electric properties of geopolymers made from four different fly ashes were examined using both the fixed frequency and spectral measurements to gain fundamental understanding of the strength development of fly ash based geopolymers.

2. Materials and methods

2.1. Fly ashes

Four locally available fly ashes were chosen to manufacture geopolymer mortar samples. The chemical compositions of these fly ashes are shown in Table 1. These fly ashes were selected to include both the low-calcium fly ashes (Class F, Gaston, Orlando, and Martin Lake in Table 1) and high-calcium fly ash (Class C, Miller in Table 1). Among three Class F fly ashes, fly ash from Martin Lake has a relatively high calcium content than the other two. In this way, we are able to not only examine whether similar chemical composition in fly ash can produce geopolymers with similar properties, but also estimate the effect of the calcium content on the properties of the geopolymers. In addition, fly ashes from Gaston and Orlando have nearly the same amount of Al_2O_3, as shown in Table 1, allowing us to minimize the effect of the content of Al_2O_3 on the produced geopolymers. All fly ashes have Fe_2O_3, whose effect on geopolymers are still under study. Limited study [9] suggests that that high iron content in the source fly ash can cause cracking and subsequent strength losses in geopolymers. All three class-F fly ashes have similar content of Fe_2O_3. Therefore, the possible influence of Fe_2O_3 on geopolymer is not considered in this study. The major physical properties of these fly ashes are shown in Table 2. The particle size distributions are shown in Fig. 1.

Table 2
Physical properties of chosen fly ashes.

Source	Specific gravity	Fineness (% retained on #325 sieve	Mean size (μm)	Mid-size (μm)
Gaston	2.33	17.23	3.43	2.24
Orlando	2.27	14.99	3.41	2.31
Martin Lake	2.48	21.2	3.08	2.05
Miller	2.60	17.98	2.87	2.01

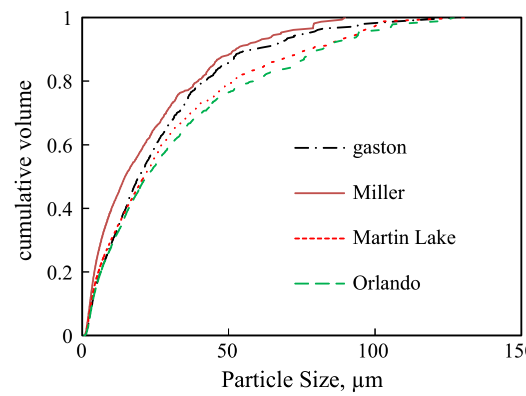

Fig. 1. Particle size distribution of fly ashes.

Not all chemical compositions shown in Table 1 participates in the geopolymerization since only the amorphous phases in fly ashes can be dissolved in the alkaline activator, while the crystalline phases become fillers in the resulting geopolymer. To determine the content of the reactive content in fly ash samples, dissolution testing of fly ash was carried out by dissolving the fly ash in 14 M Sodium Hydroxide (NaOH) solution, which was used to manufacture the geopolymer specimens in this study. Five samples were tested for each fly ash following the below testing procedure:

(1) Weigh specified amount of fly ashes.
(2) Make NaOH aqueous solution with specified concentration (14 M).
(3) Mix well the fly ash and NaOH aqueous solution in a testing bottle.
(4) Vibrate the bottle in oven at 75 °C for 24 h.
(5) Filter the solution for the residue.
(6) Weigh the residue and calculate the dissolution degree of the fly ash.

The dissolution degree is defined as the mass ratio in percentage between the materials dissolved in the NaOH solution and the total mass of the virgin fly ashes. This study uses the dissolution degree as an indicator of the reactivity of the fly ash.

In the initial dissolution testing, about 5 g of fly ash samples were used to mix with 156 g 14 M NaOH solution. This testing shows that the average dissolution degree of four fly ash samples is only 30%, which is much lower than expected. This is because too much fly ash was used in the testing so that NaOH solution was insufficient to dilute the materials leached out of the fly ash. These materials dissolved from the fly ash then formed precipitants, which couldn't be filtered out by the filter papers used in this testing, leading to much lower measured value of the dissolution degree. By reducing the amount of fly ash used in the test, the measured dissolution degree of the fly ash was found to increase accordingly. After

Table 1
Chemical composition of chosen fly ashes.

Source	SiO_2 (%)	Al_2O_3 (%)	Fe_2O_3 (%)	SO_3 (%)	CaO (%)	Moisture (%)	LOI (%)	Na_2O (%)
Gaston	50.38	27.20	9.14	0.30	2.49	0.14	2.95	0.69
Orlando	54.48	27.72	8.14	0.11	1.29	0.15	4.11	0.67
Martin Lake	54.88	19.31	8.46	0.45	8.03	0.10	0.04	0.62
Miller	36.23	19.41	6.45	1.84	23.11	0.11	0.61	1.50