Effect of Metakaolin and Silica fume on the Strength of Fly ash Based Geopolymer mortars

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Abstract—The impacts of metakaolin and silica fume on the mechanical properties of fly ash based geopolymer mortars were discussed in this study. Fluidity, 3-d and 28-d flexural strength and compressive strength were tested, microstructures were also observed by Scanning Electron Microscope (SEM). studies concluded that the metakaolin contained geopolymer mortars has better mechanical properties and denser microstructures, but silica fume contained geopolymer mortars have an adverse situation, the addition of silica fume should be under 5% and the best addition need to be further studied.

1. Introduction

Concrete is the most commonly used construction material. As the major raw material in concrete, the production of cement has a huge carbon dioxide emission, research shows that about 0.8ton carbon dioxide is given off 1ton cement clinker production,\textsuperscript{1} in addition, cement production is increasing around the world by about 5% per annum, and this will be responsible for about 12% of total CO\textsubscript{2} given off by 2020.\textsuperscript{2} Therefore, it is extremely important to develop a new applicable binding material to substitute the traditional cement in construction industries against environmental pollution by minimizing the emission of CO\textsubscript{2}.\textsuperscript{3} Geopolymer is a new aluminosilicate material with three-dimensional reticular structure, which was composed by the base materials containing affluent aluminium and silicon that was activated by adopting alkaline solution to serve as a binder.\textsuperscript{4} It is reported that the energy consumption and CO\textsubscript{2} emission of 1ton geopolymer production are 41% and 20% of ordinary Portland cement (OPC),\textsuperscript{5} in addition, geopolymer has excellent mechanical properties and durability. Metakaolin is usually used to prepare high performance geopolymer, but because of its relatively high cost and limited storage, fly ash has gradually become a new mainstream geopolymer raw material because of its low cost and environmental protection advantages.

This research describes an attempt of using metakaolin and silica fume as additive to strengthen the mechanical properties of fly ash based geopolymer, sodium hydroxide and water glass was used as alkaline activator, fly ash and metakaolin was used as raw material, the standard sand was used as aggregate, the geopolymer mortar was made by mixing raw material, alkaline activator and aggregate. The compressive strength and flexural strength were tested and the microstructure was observed.
2. Experimental Materials and Procedure

2.1. Experimental Materials
Fly ash was supplied freely by a company from Sichuan Province (China), metakaolin, silica fume, sodium hydroxide and water glass were purchased from a Chinese chemical company from Gongyi and Chengdu. The peculiar properties of those raw materials are illustrated in Table 1, Table 2 and Table 3. The Used activating solution consisted of waterglass (Na$_2$SiO$_3$) and sodium hydroxide (NaOH) and the molarity in this study was 10 M, the sodium hydroxide is chemical grade pellets with more than 98% purity, both sodium hydroxide and waterglass were commercially obtained. The standard sand was produced by Xiamen AISIO Standard Sand Co., Ltd.

Table 1. Chemical composition of fly ash and metakaolin.

|            | SiO$_2$ | Al$_2$O$_3$ | FeO$_3$ | K$_2$O | MgO | CaO |
|------------|---------|-------------|---------|--------|-----|-----|
| Fly ash    | 56.004  | 30.267      | 4.361   | 1.71   | 0.237 | 2.36 |
| Metakaolin | 45.234  | 44.92       | 4.665   | 0.165  | 0    | 0.542 |

Table 2. The basic properties of metakaolin.

|            | heating loss (%) | fineness | Al$_2$O$_3$+SiO$_2$/(%) content | Na$_2$O+K$_2$O/(%) content |
|------------|------------------|----------|--------------------------------|--------------------------|
| standard   | ≤1.0             | ≥80.0    | ≤1.0                           | ≥90                      | ≤1.0                     |
| metakaolin | 0.37             | 81       | 0.36                           | 90.8                     | 0.57                     |

Table 3. The basic properties of silica fume.

|            | activation index (7d/%) | specific surface/(m$^2$/kg) | ratio of water demand/(%) | chloridion content/(%) | SiO$_2$ content/(%) | total alkalinity/(CaO) |
|------------|-------------------------|----------------------------|--------------------------|-----------------------|--------------------|------------------------|
| standard   | ≥105                    | ≥15000                     | ≤125                     | ≤0.10                 | ≥85                | /                      |
| silica     | 110                     | 23700                      | 124                      | 0.006                 | 96.52              | 0.25                   |

2.2. Experimental Procedure
The 10 M NAOH solution was prepared by sodium hydroxide grade pellets, then mixed the solution with waterglass and cool down to room temperature for one day. The alkali-activator and raw material were poured into the vibrating bowl with 30 seconds mixing, then the standard sand was mixed with geopolymer paste for 2 minutes, the resulting geopolymer mortar was poured in 40×40×160mm steel moulds, and cure at 60°C for 8h, then demoulded, after 28d of curing at room temperature, the resulting cured specimens were taken physical properties test and microstructures were also observed by Scanning Electron Microscope (SEM). The experimental procedure and the mix proportions and fluidity of geopolymer mortars are shown in Fig 1 and Table 4.
Table 4. The mix proportions and fluidity of geopolymer mortars.

|                  | F1   | F2   | F3   | M1   | M2   | M3   |
|------------------|------|------|------|------|------|------|
| FA (kg/m³)       | 225  | 112.5| /    | 225  | 112.5| /    |
| MK (kg/m³)       | /    | 112.5| 225  | /    | 112.5| 225  |
| Si (kg/m³)       | /    | /    | /    | 11.25| 11.25| 11.25|
| Alkali-activator | 235  | 250  | 310  | 235  | 250  | 310  |
| Standard sand    | 1350 | 1350 | 1350 | 1350 | 1350 | 1350 |
| Fluidity (mm)    | 18   | 19   | 18   | 22   | 23   | 21   |

3. **Results and discussion**

3.1. **Fluidity and Mechanical properties of geopolymer mortars**

As seen from the results in Table 4, every sample exhibited an improvement in minimum alkali-activator requirement with the addition of metakaolin. It can be observed from Fig 3 that the sample with silica fume addition shows higher fluidity concerning the plain samples, this is relate to the fact that silica fume has a small particles size and hence are responsible for less porosity.

As shown in Fig 5 and Fig 6, the flexural strength and compressive strength can be observed to increases with aging time. The replacement of fly ash with metakaolin was found to be beneficial in improving the investigated compressive strength properties, and the sample with the addition of metakaolin can promote the rapid development of compressive strength in geopolymers, the compressive strength of sample F3 (all metakaolin) with 3d curing reached 90.8% that of 28d curing, and the rate of F1(all fly ash) and F2 (50%fly ash plus 50% metakaolin) is 76.5% and 84.2%. Sample
M1, M2, M3 (with silica fume addition) have nearly the same compressive strength development trend concerning the plain samples (with no silica fume addition), but the compressive strength can be observed to decreases with silica fume addition, moreover, after silica fume addition, the loss of compressive strength of sample which fly ash was replaced by metakaolin is smaller, the loss is within 3Mpa, is much less than sample with fly ash which is more than 7Mpa.

Fig 5. The flexural strength of GM

Fig 6. The compressive strength of GM

The SEM images of geopolymer mortars are presented in Fig 7. Geopolymer mortars with metakaolin addition appear to have relatively more compact and denser microstructures than those geopolymer mortars with no metakaolin addition. Fig 7a show that fly ash in the presence of alkaline medium undergoes an incomplete geopolymerization with the appearance of unreacted fly ash sphere. Moreover, the matrix phase distribution is not uniform, and the surface produces less condensed colloids, which can clearly see that the void between fly ash spheres and more large pores left by the spalling of spherical structures leave the worst density of the specimen structure. Fig 7b show that the sample undergoes more fully geopolymerization, fly ash sphere have been almost dissolved. Fig 7d and e and f appear to have similar microstructures to those no silica fume addition.

Fig 7. SEM micrographs of geopolymer mortars
4. Conclusion

Based on the results and discussions presented above, the conclusions are obtained as below:

(1) The geopolymer mortars with the addition of metakaolin indeed have a better performance in terms of compression strength and flexural strength, but the worse fluidity and the more alkaline activator will be taken place with the addition of metakaolin.

(2) The addition of silica fume will reduce the mechanical properties of the geopolymer to some extent, however, the better fluidity and the less alkaline activator will be taken place.

(3) The microstructure analysis by SEM shows that the geopolymerization of the geopolymer mortars can be in progress with the development of metakaolin addition.

(4) The addition of silica fume should be under 5% and the best addition needs to be further studied.

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