Properties of high-calcium and low-calcium fly ash combination geopolymer mortar containing recycled aggregate

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ABSTRACT

This study presents the properties of a recycled aggregate geopolymer mortar made with a blend of high-calcium fly ash (HCF) and low-calcium fly ash (LCF). An experimental study was divided into two series. In series I, an effort was made to produce a more durable HCF geopolymer by partially replacing a portion of the HCF with LCF. A mortar with a 50:50 weight blend of HCF and LCF provided a high early strength and showed excellent potential in an acidic environment. In series II, recycled aggregate was used in the LCF-blended HCF geopolymer mortar. The results showed that the compressive strength of the geopolymer mortar decreased with an increase in the recycled aggregate content. The results also indicated that application of the mortar made with recycled aggregate under aggressive conditions should be avoided. However, a mixture with 25% recycled aggregate showed a compressive strength similar to that of the control mixture containing 100% natural aggregate.

1. Introduction

The use of recycled aggregate from old concrete in the production of new concrete and mortar is a well-known method of reducing the massive volume of natural resources being used, as well as the quantity of dumped construction and demolition wastes (CDW). Recycled fine aggregate (RA), which has a particle size of less than approximately 4.5 mm, is a manufactured material that is produced by crushing waste concrete. It comprises approximately 50% of the total amount of the crushed CDW in coarse recycled aggregate production (Ulsen et al., 2013). Unfortunately, the properties of concrete made with RA are unacceptable for structural concrete (Cuenca-Moyano et al., 2014). This is because RA has a porous structure, which causes the strength and durability of concrete manufactured with it to be inferior to that prepared with natural aggregate (Evangelista and de Brito, 2010). However, the use of recycled sand may provide a good opportunity for mortar pavement applications, such as hallway and sidewalk pavement. These non-structural composites are not necessarily designed to be high-quality materials, e.g., those with high strength or low permeability. Thus, it is of interest to note here that waste concrete has not been extensively recycled as RA, especially for geopolymer concrete. Recently, numerous researchers have attempted to use waste concrete to make recycled aggregate geopolymer materials (Kathirvel and Kaliyaperumal, 2016; Nuaklong et al., 2018a, 2018b; Shi et al., 2012), but none of these have yet been adopted for fine aggregate applications. In other words, recycled sand has not had an extensive influence on the properties of geopolymer mortar and concrete.

Although geopolymers based on sodium hydroxide and sodium silicate still have a large environmental footprint, if well designed, they can be low-CO₂ materials (Provis et al., 2015). One geopolymer without sodium silicate was shown to produce a reduced carbon footprint (Ouellet-Plamondon and Habert, 2014; Luukkonen et al., 2018). Geopolymers based on waste activators also have reduced environmental footprints (Passuello et al., 2017; Moraes et al., 2018). Although there has also been some concern about the cost (Abdalqader et al., 2016), this is offset by the relatively good properties obtained with sodium-based alkali

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activators. A binding agent is formed by the reaction of alkaline activators with materials that are rich in SiO$_2$ and Al$_2$O$_3$. These are considered to be the 3rd generation of cement composites. They are similar to lime and ordinary Portland cement (OPC) in their ability to bind the aggregate for mortar and concrete production (Li et al., 2010; Sumesh et al., 2017). Geopolymers are superior to OPC in terms of their low environmental impact and application of industrial waste materials (blast furnace slag, fly ash, etc.) (Duan et al., 2015). In addition, this alternative binder suffers extraordinarily limited adverse effects from chemical attacks and shows a high early strength (Xie and Kayali, 2016).

Fly ash is an aluminosilicate material that is widely used for the preparation of geopolymer composites, especially low-calcium fly ash (LCF) (Nath and Sarker, 2017). In fact, one of the most important benefits of using fly ash for geopolymer synthesis is its easy availability compared to other source materials (Mehta and Siddique, 2017). In Thailand, most fly ash is obtained from burning lignite coal, which contains a large amount of calcium, or high-calcium fly ash (HCF). The content of the calcium compound is one of the significant factors affecting the behavior of the fresh and hardened geopolymer material. Chindaprasirt et al. (2012) reported that the rapid hardening process is associated with the rapid formation of a calcium silicate hydrate (CSH) phase. In addition, high deterioration was found when samples were exposed to aggressive solutions (Mehta and Siddique, 2017). Based on these previously discussed investigations, it is possible to conclude that the reduced calcium content of the source material is responsible for setting time delay and improvements against chemical attack. For this reason, the replacement of HCF with LCF is seen as a good choice in preparing a workable and durable geopolymer binder.

Thus, this research attempted to prepare an LCF–HCF combination geopolymer mortar that incorporated RA and to study its strength behavior, transport properties, and sulfuric acid resistance.

2. Experimental

2.1. Raw materials

The following alkaline activators were used to prepare the geopolymer composites: (1) a sodium hydroxide solution (NaOH, NH) with a 10 M (M) concentration and (2) a commercial sodium silicate solution (Na$_2$SiO$_3$, NS), which mainly contained Na$_2$O, SiO$_2$, and water at 12.3%, 30.3%, and 57.3%, respectively. HCF and LCF were utilized as source materials to manufacture the geopolymer binder. The specific gravity for the HCF was determined to be 2.43, while that for the LCF was 2.50. Portland cement type I with a specific gravity of 3.13 and tap water were used to produce OPC-based mortar. The chemical composition, loss in ignition, and fineness values of the HCF, LCF, and OPC are listed in Table 1. The mixtures made with LCF were expected to have lower flow values as a result of the finer particles (Table 1) and more shape angularity than those made with the HCF, as can be seen in Fig. 1. A superplasticizer (SP) was used in several mixtures to improve the workability and produce a mortar flow of 110±5%. Although the 0RA mortar showed a flow value of 119%, no segregation pattern was found in this mixture. The OPC mortar was prepared in accordance with ASTM C305 (2014a). After mixing, the OPC samples were cured in molds for 1 d. They were then demolded and cured in saturated lime water until the testing day.

In the case of the geopolymer mortar, the preparation of the samples was divided into three steps. Each step was carried out for 5 min. The HCF and LCF were first dry mixed separately to ensure homogeneity before the addition of the NH solution. Next, the prepared dry mixture of natural sand/RA in a saturated surface dry state was mixed with the cement slurry. Last, an NS solution with SP (1–2% fly ash by weight) was added. After mixing, the geopolymer samples were left at ambient temperature for 1 h before heat curing at 60 °C for 2 d. After that, the samples were demolded, sealed with plastic film, and kept at 25 °C in a controlled room (50% RH) until the testing day.

2.2. Test methods

Table 4 summarizes the details of the experimental program. For series I, this study focused on the determining the porosity, water absorption, compressive strength, and bulk dry density values at 7, 28, and 90 d in order to investigate the time-dependent behaviors of geopolymer/Portland cementing materials. In addition, the setting time was tested to determine the reactivity of the HCF and LCF during alkaline

| Type   | SiO$_2$ | Al$_2$O$_3$ | CaO | Fe$_2$O$_3$ | MgO | K$_2$O | SO$_3$ | LOI (%) | % retained on 45 μm mesh |
|--------|---------|-------------|-----|-------------|-----|--------|--------|--------|-------------------------|
| HCF    | 36.2    | 19.9        | 14.2| 11.9        | 1.9 | 2.4    | 3.6    | 0.4    | 34.8                    |
| LCF    | 60.6    | 25.8        | 4.2 | 2.9         | 0.51| 1.8    | 0.42   | 2.3    | 28.9                    |
| OPC    | 17.2    | 4.02        | 63.1| 3.11        | 0.94| 0.64   | 3.9    | 0.9    | 5.0                     |

The proportions of all the mortar mixtures are given in Table 3. To discover the influences of the LCF and RA on the properties of the geopolymer mortar, the mixtures were divided into two series. In series I, there were four geopolymer mortar mixtures designed to investigate the effect of the LCF content on the setting time, strength development, and durability of the geopolymer mortar. A binder-to-natural sand ratio of 1:2.75 by weight was used in all the mixtures. The levels of the LCF substitution for HCF were 0%, 25%, 50%, and 100% by weight (denoted by 0LCF, 25LCF, 50LCF, and 100LCF, respectively). The total alkaline activator/binder ratio and NS/HA ratio were kept constant at 0.55 and 1.0 by weight, respectively. In addition, to compare the results obtained using the conventional Portland cement-based binders, an OPC mortar mixture with a water-to-cement ratio of 0.50 was prepared.

In series II, the effect of the RA replacement on the properties of the hybrid geopolymer mortar was investigated. Five mixtures of geopolymer mortar were manufactured with a 50:50 HCF-to-LCF ratio. Natural sand was replaced by 0%, 25%, 50%, 75%, and 100% recycled concrete aggregate by volume (denoted by 0RA, 25RA, 50RA, 75RA, and 100RA, respectively). Because of the angular shape of the RA, which led to a decrease in the workability of the mixtures, it was necessary to decrease the NS solution (Evangelista and de Brito, 2010; Nath and Sarker, 2015). The NS/HA ratio was adjusted from 1.0 to 0.6 for all the mixtures, leading to an increase in the flowability. The results were compared to those of the control mixture made with natural aggregate.
activation. In series II, only the 7 d physical and transport properties were determined.

The sulfuric acid resistance was observed for both series of mortars. At the age of 28 d, the mortar samples were immersed in a 3% sulfuric acid solution (H₂SO₄). The weight losses of the mortar specimens were determined at 7, 14, 28, 56, 84, and 120 d, following ASTM C267 (2012).

3. Results and discussion

3.1. Series I: influence of LCF on properties of HCF geopolymer mortar

3.1.1. Setting time

The setting time results are presented in Fig. 3. As expected, the initial and final setting times of mixtures prepared with higher contents of LCF showed a low rate of hardening. Increasing the LCF content in the mixture led to a decrease in the amount of CaO. This CaO caused accelerated setting with the rapid formation of the CSH phase (Chindaprasirt et al., 2012). For instance, the initial setting times for the geopolymer mortars made with 0%, 25%, and 50% LCF were 23, 28, and 107 min, respectively. The results also illustrated that both the initial and final setting times of the mixture prepared using LCF alone were approximately three times longer than those of the conventional mortar (OPC mixture).

3.1.2. Porosity and water absorption

Table 5 lists the molar ratios of the geopolymer mortars. The water absorption and porosity values of the geopolymer mortars at 7, 28, and 90 d are shown in Figs. 4 and 5, respectively. The geopolymer mortars with LCF contents of 0%, 25%, and 50% had porosity values at 7 d of 17.9%, 15.9%, and 14.5%, respectively. Increasing the SiO₂/Al₂O₃ mole ratio of the samples with incorporated LCF resulted in a more compact geopolymer microstructure (Chindaprasirt et al., 2012; Xie and Ozbak-kaloug, 2015). However, a small amount of CaO in the LCF led to the formation of a small quantity of a CASH-like phase, which was more homogenous and had a denser microstructure than the NASH phase, especially in the mixture prepared using only LCF (Chindaprasirt et al., 2012; Ismail et al., 2013). This may explain the reason for the higher porosity observed in 100LCF compared with the 25LCF and 50LCF mixtures.
Table 4
Experimental details.

| Tests                  | Standards | Specimens | Testing days | Series I | Series II |
|------------------------|-----------|-----------|--------------|----------|-----------|
| Setting time           | ASTM C807 | -         | Mixing date  | -        | -         |
| Fresh property         | ASTM C1437| -         | Mixing date  | Mixing date |
| Porosity               | ASTM C642 | 50 mm cube | 7, 28, and 90 d | 7 d      |            |
| Water absorption       | ASTM C642 | 50 mm cube | 7, 28, and 90 d | 7 d      |            |
| Bulk dry density       | ASTM C642 | 50 mm cube | 7, 28, and 90 d | 7 d      |            |
| Compression            | ASTM C109 | 50 mm cube | 7, 28, and 90 d | 7 d      |            |
| Acid resistance        | ASTM C267 | 50 mm cube | 7, 14, 28, 56, 84, and 120 d |          |            |

The development of the pore structure of the mortar over time will be discussed in detail in this section. The results indicated that the presence of CaO in the source materials could enhance the improvement in the microstructure of the geopolymer matrix. In other words, the gel microstructure development of the geopolymer mortar after 7 d decreased with an increase in the LCF content. For example, there were 5.1%, 2.8%, and 1.5% decreases in the volume of the permeable voids from 7 d to 90 d for the 0LCF, 25LCF, and 100LCF mixtures, respectively. Even though the pore volume in the geopolymer matrix continuously decreased with time, this decrease was slower in comparison to that of the OPC-based mortar. Ma et al. (2013) reported that the cavities in unreacted fly ash particles (with hollow interiors) still existed even at a later age, which suggested that the geopolymer gel could not fill this space. In addition, they (Ma et al., 2013) commented that the alkali activation process for the fly ash was almost completed in the first 7 d.

The water absorption capacity is related to the pore volume of the material, with a lower pore space resulting in a lower absorption after immersion. The water transport process was blocked by the reaction products, especially in the later stages. The growth of reaction products formed in both the geopolymer and OPC mortars was effective in reducing the water absorption. For example, the water absorption values of the 100LCF mixture at 7 and 90 d were 8.5% and 7.2%, while those of the OPC mortar at the same ages were 8.0 and 7.3%, respectively.

3.1.3. Compressive strength and bulk dry density

The compressive strength data provided in Fig. 6 indicate that the highest compressive strengths for the geopolymer mortar at 7 and 28 d would be observed when 50% LCF was incorporated. However, the compressive strength at the later age was lower than those of the 0LCF and 25LCF mixtures. Geopolymer mortars made with 0% and 25% LCF had compressive strengths at 90 d that were 133% and 117% greater than those at 7 d, respectively, while the gain for the sample with 50% LCF was approximately 4%. This indicated that the incorporated LCF made a significant improvement only in the early-age compressive strength.

The results showed that the strength development in the geopolymer system over time was slower when the HCF was replaced by LCF. For example, the compressive strengths of the 25LCF and 50LCF mixtures at 90 d were increased by 10% and 2%, respectively, compared to their compressive strengths at 28 d. Increasing the LCF content led to an increase in both SiO2 and Al2O3, whereas the CaO content was significantly decreased. In addition to the formation of a CSH or CASH-like phase, the presence of CaO hindered the formation of poorly crystalline zeolite phases, which had a detrimental effect on the development of the geopolymer strength when excess alumina was present in the system, possibly reducing the gain in compressive strength at later ages (Chindaprasirt et al., 2012; Winnefeld et al., 2010).

The bulk dry density values of the mortar mixtures are also given in Fig. 6. The density of the geopolymer mixture varied between 1.96 and 2.07 g/cm³. Because the specific gravities of the binders (HCF and LCF) showed very little difference, the bulk dry density of the geopolymer mortar did not vary with LCF content. In relation to the development of the density of the geopolymer mortar with time, the results indicated that the age of the sample had no significant effect on the density. Similar trends were obtained for the OPC mixture.

3.1.4. Sulfuric acid resistance

As shown in Fig. 7, the mortar mixtures blended with LCF showed higher acid resistances, especially when the LCF percentage was increased. For instance, at 84 d, the mortars with 0%, 25%, 50%, and 100% LCF showed weight losses of 6.1%, 3.3%, 2.2%, and 1.4%, respectively. The improvement in acid resistance was due to a reduction in the CaO/SiO2 mole ratio of the starting mixture when LCF was introduced in the mix. Sata et al. (2012) confirmed that a low calcium content in a cement-based material resulted in lower deterioration by a sulfuric acid solution. Chindaprasirt et al. (2013) explained that the presence of CaO leads to the formation of CSH and Ca(OH)2 in a geopolymer composite. These calcium-based hydrated products can be easily decomposed by an aggressive solution. In relation to the silica content, Chindaprasirt et al. (2012) reported that the availability of more silicate species for polycondensation when extra silica oxide is present in the system leads to the production of denser microstructures and a good acid-resistant material. Thus, it is possible that the CaO/SiO2 mole ratio may indicate the performance of a fly ash geopolymer mortar made with natural sand in the presence of an H2SO4 solution. It can be clearly seen from Fig. 8 that the samples prepared using 50% and 100% LCF had excellent resistances to acid attack, with no signs of surface deterioration, even though they faced the harshest conditions found in a wastewater system (3% H2SO4, pH of 0.3) (Chindaprasirt and Rattanasek, 2016). In contrast, the visual appearance of the OPC-based mortar after immersion in H2SO4 indicated a serious problem due to the high CaO content in the composite. This result
Fig. 4. Porosities of geopolymer mortars with various low-calcium fly ash (LCF) contents.

Fig. 5. Water absorption values of geopolymer mortars with various low-calcium fly ash (LCF) contents.

Fig. 6. Compressive strengths and bulk dry densities of mortars.
confirmed those previously reported by Sata et al. (2012) and Bakharev (2005) that OPC materials had lower acid resistances than geopolymer composites.

3.2. Series II: influence of RA on properties of LCF-blended geopolymer mortar

The previously mentioned results (series I) showed that the LCF effectively improved the early age strength and acid resistance of the HCF geopolymer composite. The maximum 7 d compressive strength was obtained when 50% of the HCF was replaced with LCF. This advantage would make the sample beneficial for use in precast applications. In addition, LCF can be used to delay the setting rate of the geopolymer mortar, which would make its placement and finishing easier. The initial and final setting times of the 50LCF mixture were close to those of the OPC mortar. For these reasons, the 50% LCF-incorporated mortar was found to be the most suitable for a recycled aggregate geopolymer mortar.

3.2.1. Porosity and water absorption

Figs. 9 and 10 show the porosity and water absorption values of the hybrid geopolymer mortars made with different RA contents. The results show that the higher inclusion of RA resulted in a looser structure. The porosity was increased from 18.6% in 0RA to 24.9% in 100RA. Zhao et al. (2015) found that the excess water that moved from the recycled aggregate to the matrix caused a large interfacial transition zone (ITZ) in mixtures made with recycled aggregate. This led to an increase in the porosity of the mortar.

The results showed a clear increasing trend for the volume of water absorbed by specimens as the RA content increased. The mixture made with 25%, 50%, 75%, and 100% RA showed water absorption values of 11.1%, 11.3%, 12.6%, and 13.4%, which were 2.1%, 2.3%, 3.6%, and 4.4% higher than that of the 0RA mixture, respectively. This agreed with the results reported by Evangelista and de Brito (2010), who...
incorporated fine recycled concrete aggregate in Portland cement concrete. The porous old mortar adhering to the surface of the recycled aggregate contributed to the increase in porosity and water absorption of the mixtures (Zhao et al., 2015).

3.2.2. Compressive strength and bulk dry density

With the inclusion of RA, the compressive strength and bulk dry density of the geopolymer mortar decreased significantly, as listed in Table 6. Both the compressive strength and density of the mortar tended to decrease with an increase in the RA replacement level. For specimens prepared with 0%, 25%, 50%, 75%, and 100% RA, the compressive strengths were found to be 46.9, 46.6, 44.2, 43.6, and 40.6 MPa, respectively. The compressive strength of the mortar was related to its porosity, as can be seen in Fig. 11. This indicated that a higher pore volume led to a reduction in the compressive strength of the mortar. The fact that the dry-rodded density of the RA was 29% lower than that of the NA resulted in a reduction in the bulk dry density in the range of 2.5–8.0% for the geopolymer mortar made with RA, in comparison to the control mixture (0RA). The lowest density of 1.84 g/cm³ was obtained for the 100RA mixture. These results were supported by those of Ledesma et al. (2014).

3.2.3. Sulfuric acid resistance

Fig. 12 shows the influence of the RA on the acid resistance of the geopolymer materials. Serious surface deterioration was found in the geopolymer mortars containing RA, as shown in Fig. 13. This is in agreement with the study by Nuaklong et al. (2016) on geopolymer concrete made with RA. This was caused by both the porous structure and Ca-rich hydration product (CSH, Ca (OH)₂) of the old mortar on the surface of the RA, which could lead to high degradation (Nuaklong et al., 2016; Zhao et al., 2015). In addition, the rate of deterioration increased with the RA content. For example, at 84 d of exposure, the weight loss was the highest for the 100RA mixture (77.0%), followed by those of the 75RA (74.3%), 50RA (67.3%), 25RA (38.0%), and 0RA mixtures (2.0%).

Based on the experimental results, although the use of recycled concrete as a replacement for natural sand in the geopolymer mortar decreased its performance, a mixture made with 25% RA achieved a strength and impermeability similar to those of the control mixture (0RA). This indicated that the best RA mortar in this series was the 25RA mixture, which had a compressive strength of 46.6 MPa and water absorption of 11.1%. However, the weakness of this composite was its low acid resistance.

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**Table 6**

Compressive strength and bulk dry densities.

| Properties           | 0RA  | 25RA | 50RA | 75RA | 100RA |
|----------------------|------|------|------|------|-------|
| Compressive strength | 46.9 | 46.6 | 44.2 | 43.6 | 40.6  |
| (MPa)                |      |      |      |      |       |
| Bulk dry density     | 2.00 | 1.95 | 1.92 | 1.88 | 1.84  |
| (g/cm³)              |      |      |      |      |       |

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**Fig. 10.** Water absorption values of low-calcium fly ash (LCF) blended geopolymer mortars at the age of 7 days.

**Fig. 11.** Correlation between compressive strength and porosity of geopolymer mortar.

**Fig. 12.** Weight changes in geopolymer mortars immersed in H₂SO₄ solution.
4. Conclusions

In the first experimental series, the effect of LCF on the properties of HCF geopolymer mortar was investigated. A workable geopolymer mortar was achieved with the incorporation of LCF. The mixture made with LCF required a long time to set. The inclusion of LCF for up to 50% of the total binder helped to decrease the microstructural porosity and in turn increased the strength by the formation of the NASH phase with a high SiO$_2$/Al$_2$O$_3$ mole ratio, especially at an early age. Within the CaO/SiO$_2$ ratio range of 0.07–0.17, the mixtures could effectively protect a geopolymer material from acid attack. However, the highest compressive strength at the age of 90 d was observed to be 56.9 MPa for the mixture prepared with HCF alone.

Series II was used to examine the compressive strength and durability properties of hybrid geopolymer mortars made with RA. The pore volume of the geopolymer mortar increased with the RA content. This resulted in a greater absorption capacity. In addition, a reduction in the compressive strength of the specimens was observed, which led to a reduction in durability, especially when 100% RA was used. The incorporation of RA significantly reduced the acid resistance of the geopolymer. An acceptable weight loss was found for a 25% RA mixture. Mixtures with more than 50% RA had high weight losses after 120 d of immersion in an acid solution. However, the compressive strength of the mixture prepared using 25% RA was found to be comparable to that of the control mixture made with only natural aggregate. It was particularly interesting to note that the normal geopolymer mortar made with natural river sand (ORA mixture, CaO/SiO$_2$ = 0.18) did not show any significant weight loss after 120 d of immersion in the 3% H$_2$SO$_4$ solution.

Declarations

Author contribution statement

Peem Nuaklong: Performed the experiments; Wrote the paper.
Ampol Wongsa: Performed the experiments; Analyzed and interpreted the data.
Vanchai Sata: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.
Kornkanok Boonserm: Analyzed and interpreted the data.
Jay Sanjayan & Prinya Chindaprasirt: Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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