

## Research Article

# Development and Performance Evaluation of Very High Early Strength Geopolymer for Rapid Road Repair

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High early strength is the most important property of pavement repair materials to allow quick reopening to traffic. With this in mind, we have experimentally investigated geopolymers using low cost raw materials available in Thailand. The geopolymer mortar was metakaolin (MK), mixed with parawood ash (PWA, rubberwood ash) or oil palm ash (OPA) as binder agent. Rubberwood is often used as raw material for biomass power plants in Thailand, especially at latex glove factories and seafood factories, and burning rubberwood generates PWA. Both PWA and OPA are therefore low cost residual waste, locally available in mass quantities. The geopolymer samples were characterized for compressive strength, drying shrinkage, and bond strength to Portland cement mortar with slant shear test. The experimental design varied the contents of PWA and OPA and the heat curing time (1, 2 and 4 h) after hot mixture process. The hot mixture process resulted in very high early strength. In addition, we achieved high compressive strengths, low drying shrinkage, and very significant bond strength enhancement by use of the ashes.

## 1. Introduction

Many concrete infrastructures, such as concrete pavement, bridge decks, parking structures, highways, and airport runways, have high repair costs when damaged. This is partly because special very-rapid-setting materials are used to shorten the repair time in order to quickly get back to normal use. The materials for rapid road repair must have high early compressive strength, and include polyurethane polymer, epoxy polymer, thermosetting vinyl polymer, and magnesium polyphosphate.

Rapid concrete repair methods are of economic significance and therefore have been widely studied. Relatively recent extensive studies have taken place at the Center for Transportation Research of the University of Texas at Austin [1] and the Texas Transportation Institute at the Texas A&M University [2]. Modified Portland cement has been investigated [3, 4] along with magnesia-phosphate [5, 6]. These studies have emphasized rapid setting. The current study used materials locally available in Thailand, including waste

streams, in the production of geopolymers for these applications.

Studied in materials science and engineering, the geopolymer process takes place by chemical reactions between aluminosilicate materials and alkaline solutions, under elevated curing temperatures. Common raw materials for geopolymer are fly ash and metakaolin containing  $\text{SiO}_2$  and  $\text{Al}_2\text{O}_3$ . The geopolymer benefits include high strength and high early strength [7]. However, some studies [8–10] have also assessed other mechanical properties such as the compressive strength at 1, 7, and 28 days. These other properties are not of primary importance in concrete pavement repair, with the priority on early opening to traffic.

The current study focuses on metakaolin (MK), with parawood ash (PWA, i.e., ash from rubberwood) or oil palm ash (OPA).

Little research is available on the utilization of PWA or OPA waste. In addition, the mechanical properties and behavior of MK-based geopolymers with PWA or OPA are still not well established or understood. Thus, further research is

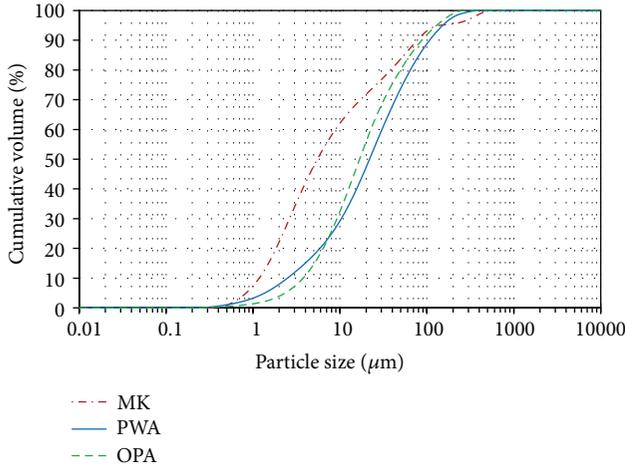


FIGURE 1: Particle size distributions of raw materials.

needed to evaluate the effects of PWA or OPA in geopolymer composite materials. The current study on MK-based geopolymers containing PWA or OPA addresses these gaps in knowledge about new environmentally friendly materials particularly for concrete pavement repair.

We have created and tested MK-based geopolymers containing PWA or OPA as partial replacements for MK. The amount of PWA ranged up to 30% and of OPA up to 15% by weight of MK. We also tested different heat curing times, so that all of the main factors influencing geopolymer properties were varied in the experimental design. Various key characteristics of concrete repair materials were determined for the geopolymer.

## 2. Experimental Program

**2.1. Materials.** The kaolin used in this study was calcined at 750°C for 2 h and used as Si-Al cementitious material. The chemical composition of the MK was analyzed using X-ray fluorescence (XRF); it had the main components 50.30% SiO<sub>2</sub> and 41.02% Al<sub>2</sub>O<sub>3</sub>. Grinding the MK in a ball mill produced particles with mass-average size  $d_{50} = 6.31 \mu\text{m}$  (Figure 1). Both chemical composition and physical properties of this MK are listed in Table 1.

PWA generated during the combustion of parawood for electricity production was collected from a biomass thermal power plant. The OPA used in this study was collected from a palm oil mill. Both the PWA and the OPA were ground in a ball mill for approximately 4 h. The particle size distributions determined by laser scattering are shown in Figure 1: the  $d_{50}$  values were 25.13  $\mu\text{m}$  for PWA and 19.31  $\mu\text{m}$  for OPA. The chemical compositions were analyzed by XRF. OPA had the main chemical components 13.84% K<sub>2</sub>O, 38.37% SiO<sub>2</sub>, and 14.09% CaO by weight, while PWA had similarly 16.11% K<sub>2</sub>O and 41.19% CaO. The chemical compositions and physical properties of PWA and OPA are listed in Table 1.

The alkaline activator used was a mixture of sodium hydroxide (NaOH) in flakes of 98% purity, sodium silicate

TABLE 1: Chemical compositions (wt%) and physical properties of cementitious materials used.

Chemical compositions	MK	PWA	OPA
SiO <sub>2</sub>	50.30	2.57	38.37
Al <sub>2</sub> O <sub>3</sub>	41.02	0.53	1.48
Fe <sub>2</sub> O <sub>3</sub>	1.05	0.56	3.01
CaO	0.33	41.19	13.84
TiO <sub>2</sub>	1.50	—	0.21
MgO	—	4.52	3.00
K <sub>2</sub> O	4.08	16.11	14.09
P <sub>2</sub> O <sub>5</sub>	—	3.06	4.15
SO <sub>3</sub>	—	5.54	1.42
Cl	—	0.82	—
MaO <sub>2</sub>	—	1.36	—
Loss on ignition (LOI)	1.72	23.74	20.43
Specific surface area (m <sup>2</sup> /g) BET	13.61	8.71	13.06
$d_{10}$ ( $\mu\text{m}$ )	1.352	2.860	4.321
$d_{50}$ ( $\mu\text{m}$ )	6.308	25.128	19.305
$d_{90}$ ( $\mu\text{m}$ )	88.803	123.450	100.109

(Na<sub>2</sub>SiO<sub>3</sub>), and water. The sodium silicate solution contained 14.14% Na<sub>2</sub>O, 27.67% SiO<sub>2</sub>, and 56.28% H<sub>2</sub>O by weight.

River sand was used as the fine aggregate inert component of the geopolymer mortars. The specific gravity of the river sand was 2.51 and the maximum size was 4.75 mm. The water absorption of the river sand was 1.48%, tested in accordance with ASTM C128 [11].

**2.2. Mixed Proportions.** Geopolymer mortars were prepared by mixing the raw materials (MK with PWA or OPA), river sand, and alkaline activators (sodium silicate, sodium hydroxide, and water). The alkaline activator was mixed first at 74 ± 2°C from reaction heat. The final mixing of alkaline activator, reactive raw material, and river sand took place at 48 ± 2°C. These elevated temperatures were essential to fast setting. Details of the ingredient proportions and molar ratios are shown in Tables 2 and 3, respectively. The samples were mixed manually, not with a motorized mixer. The slurries were poured into acrylic 50 × 50 × 50 mm molds to set and form samples for compressive strength testing. The samples were compacted in accordance with ASTM 109/C109M [12]. Samples were also formed in acrylic 25 × 25 × 285 mm molds, compacted in accordance with ASTM C596 [13], for use in tests of drying shrinkage. The samples in acrylic molds were wrapped with polyvinyl sheets to prevent loss of moisture. All of the samples were heated in an electric oven at 80°C after casting. After curing for 1, 2, or 4 h, they were removed from the oven. After unwrapping and demolding, the samples were stored at ambient temperature of 30 ± 2°C with a relative humidity 70 ± 5% until they were tested. The bond strength test samples were prepared in 50 × 50 × 50 mm acrylic molds slanted to give an interface at 45° (Figure 2) against Portland cement mortar, which was cured at ambient temperature for 7 days. Afterwards, the geopolymer was added to these molds to form a cube with the set Portland cement. The molds

TABLE 2: Mixed proportions of geopolymer mortars (by weight).

Mix	MK (%)	PWA (%)	OPA (%)	SS/N ratio	A/R ratio	W/R ratio	RS/R ratio	Heat curing (h)
Control-1	100	—	—					
P10-1	90	10	—					
P20-1	80	20	—					
P30-1	70	30	—	2.5	0.83	0.45	3	1
O5-1	95	—	5					
O10-1	90	—	10					
O15-1	85	—	15					
Control-2	100	—	—					
P10-2	90	10	—					
P20-2	80	20	—					
P30-2	70	30	—	2.5	0.83	0.45	3	2
O5-2	95	—	5					
O10-2	90	—	10					
O15-2	85	—	15					
Control-4	100	—	—					
P10-4	90	10	—					
P20-4	80	20	—					
P30-4	70	30	—	2.5	0.83	0.45	3	4
O5-4	95	—	5					
O10-4	90	—	10					
O15-4	85	—	15					

Note: SS: sodium silicate; N: sodium hydroxide; A: (SS + N);  
R: raw materials (MK, PWA, and OPA); W: water; RS: river sand.

TABLE 3: Mixture formulations.

Sample	SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> (molar ratio)	CaO/SiO <sub>2</sub> (molar ratio)	Na <sub>2</sub> O/SiO <sub>2</sub> (molar ratio)	H <sub>2</sub> O/Na <sub>2</sub> O (molar ratio)
Control	2.77	0.01	0.21	11.43
P10	2.85	0.08	0.23	11.43
P20	2.95	0.16	0.25	11.43
P30	3.09	0.26	0.27	11.43
O5	2.88	0.02	0.22	11.43
O10	3.01	0.03	0.22	11.43
O15	3.15	0.04	0.22	11.43

were wrapped with polyvinyl sheet and heated to 80°C. After curing for 1, 2, or 4 h, the samples were unwrapped and demolded. They were stored at ambient temperature for 24 h and then tested.

**2.3. Test Procedure.** Geopolymer mortars were wrapped with polyvinyl sheets to prevent moisture loss, cured in an oven at 80°C for 1, 2, or 4 h, and cooled at ambient temperature of 30 ± 2°C with 70 ± 5% relative humidity. The compressive strengths of geopolymer mortars were determined according to ASTM 109/C109M [12], using a universal testing machine at a loading rate of 5.00 mm/min. For each mixture, three cast samples were tested at each age of 2, 6, 24 h, and 7 days.

The drying shrinkage was determined with a length comparator in accordance with ASTM C490 [14]. The geopolymer



FIGURE 2: Typical specimens with geopolymer mortar bonded to Portland cement mortar.

mortars were prepared using 1, 2, or 4 h of curing at 80°C. Measurements were taken up to 30 weeks of age after curing.

The bond strength between Portland cement mortar and geopolymer mortar was determined by a shear test, from 50 × 50 × 50 mm slant shear samples with interface line at 45° (see Figure 2). The samples were tested under compression using the standard procedure for testing compressive strength. The bond strength was tested 24 h after heat curing, in ambient temperature. Bond failure of a slant shear sample is shown in Figure 3. The slant shear test has become the most widely accepted test for the bonding of repair materials to concrete [15]. However, there is no general standard available for this test [15, 16].



FIGURE 3: Slant shear sample after failure in compression test.

### 3. Results and Discussion

**3.1. Compressive Strength.** The effects of PWA or OPA content on the compressive strength of metakaolin-based geopolymer mortar are shown in Figures 4 to 8. The early 2 h compressive strength is high, partly because the geopolymer mortars were prepared as a hot mixture before curing in an oven.

The compressive strengths for different mixtures at the observed ages are presented in Figure 4. The early 2 h compressive strength ranged from 15 MPa to 71 MPa, while the final observation at 7 days ranged from 42 MPa to 70 MPa. The different curing times had no effect on the final compressive strength of these geopolymers.

The compressive strength of geopolymer mortars with PWA, heat-cured at 80°C for 1 h, is shown in Figure 5. The compressive strength clearly decreased with PWA content, especially at 2 h observation time. This may partly be due to particle size effects ( $d_{50}$  6.31  $\mu\text{m}$  of MK against 25.13  $\mu\text{m}$  of PWA); the greater surface area of finer particles makes them more active in polymerization [17]. Another contributing factor may be the reduction of Si and Al when MK is replaced with PWA. The CaO in PWA did not participate in geopolymerization and may have hydrated slowly. Winnefeld et al. [18] found that the lower strength incurred by high calcium content may in part be caused by poor reactivity with alkaline activators in fly-ash-based geopolymers. Test results confirmed that adding CaO into raw materials decreased compressive strength. Promising results have also been reported [19] for a curing temperature of 70°C.

Compressive strengths with OPA, heat-cured at 80°C for 1 h, are shown in Figure 6. The compressive strengths again decrease with OPA content, but at 5% OPA the final 7-day strength remains unchanged. The compressive strength values remain acceptable also at OPA contents of 10% and 15%. The main chemical component in OPA was SiO<sub>2</sub>, while PWA mainly had CaO, partly causing the different effects on compressive strength. Also, the average particles size differs, and the smaller-sized OPA may be more reactive due to this [17].

The effects on compressive strength might also be explained with molar ratios (SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub>). Figure 4 shows the

variation in compressive strengths of the samples measured after heat curing. Assuming this ratio was the determining factor, the most favorable SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratios were in control and O5, these being SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> = 2.77 and 2.88, respectively. Chindaprasirt et al. [20] have reported similar trends, with SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> up to 2.87, for high calcium fly-ash-based geopolymer systems. However, adding OPA beyond the optimal amount decreases the compressive strength because it also contains CaO. In addition, the compressive strength decreased with the amount of PWA (high CaO). This is consistent with previous studies [19] on fly-ash-based geopolymers. Previous studies [20–22] have also suggested that the ratio SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> influences the properties of the geopolymer binders. Generally, the geopolymer binders have been prepared using fly ash and metakaolin, with the ratio SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> within the range from 2 to 4 [20, 21].

For all mixtures, long heat curing times increased the compressive strength observed at 2 h in ambient temperature. Heat curing may accelerate the degree of geopolymerization by encouraging the formation of mineral phases. For the samples cured for 1, 2, and 4 h, with 10% PWA or OPA, the compressive strengths for up to 7 days of age are shown in Figures 7 and 8. The samples cured for 2 and 4 h did not develop dramatically higher long-term compressive strengths; instead, the prominent effects are in the early strength development. Furthermore, with 10% OPA, the curing time only had a marginal effect; see Figure 8.

**3.2. Drying Shrinkage.** All geopolymer mortars were exposed to 30 ± 2°C and 70 ± 5% relative humidity for a prolonged time, up to 30 weeks. The drying shrinkages of all samples are presented in Figures 9 to 12. The drying shrinkage of control samples is lower than with 10%, 20%, and 30% PWA (Figure 9). The average particle size of PWA is larger than of MK and correlates negatively with the specific surface area. The geopolymer reaction was incomplete (see Figures 5 and 6) after heat curing at 80°C for 1 h, but the excess water could not evaporate because the samples were wrapped with polyvinyl sheets. After carving off the polyvinyl sheet, the evaporation of pore water could take place; this is easier with large pores corresponding to coarser particles. This is how PWA content may contribute to the drying shrinkage and increase it relative to control samples. Also, OPA tends to increase drying shrinkage (Figure 10). In the control and with 5%, 10%, or 15% OPA, drying shrinkage was rapid in the 1 to 8 h interval. Our MK-based geopolymer containing PWA or OPA had lower drying shrinkages than those reported for slag mortar with sodium silicate, sodium hydroxide, and sodium carbonate activators [23].

Figures 11 and 12 illustrate the drying shrinkage of geopolymer mortars containing 10% PWA and 10% OPA, respectively, heat-cured at 80°C for 1, 2, or 4 h. The samples exhibited similar behavior, in terms of longer curing decreasing the drying shrinkage. The shrinkage of the samples containing 10% PWA is presented in Figure 11. With 4 h curing, there was less shrinkage than with shorter curing. The use of water in the geopolymerization reaction increases with curing time. The drying shrinkage of samples containing 10% OPA is presented in Figure 12. The samples cured for 2 and 4 h had

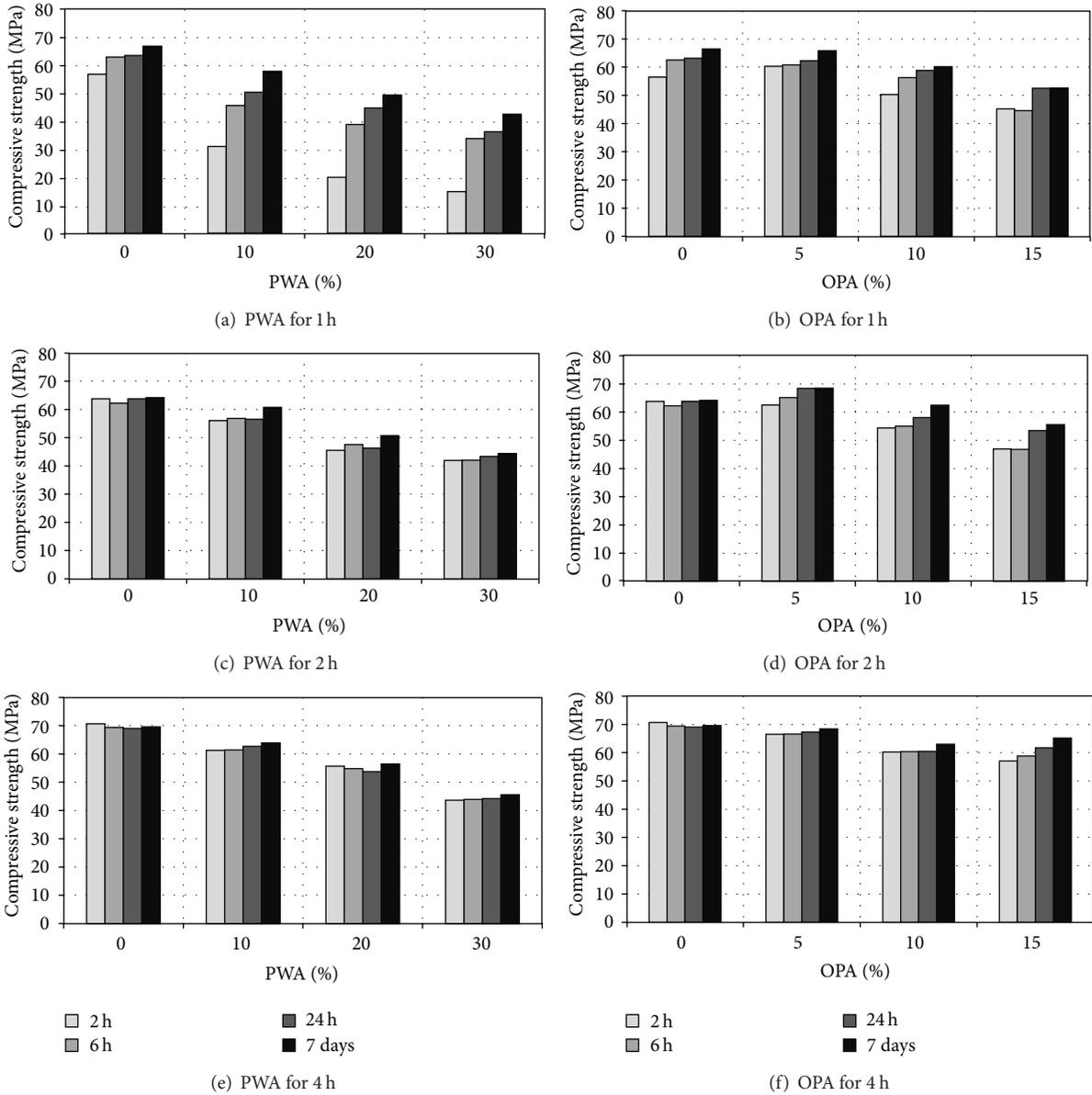


FIGURE 4: Compressive strengths for different mixtures and curing times.

similar drying shrinkage values at all ages, with the shrinkage continuing throughout the observed 30 weeks. For specimens cured for 1h, the drying shrinkage was much higher at all ages than with the longer cure times. Again, longer cure times left less water for evaporation, due to higher extent of geopolymerization reactions that used up water.

**3.3. Drying Shrinkage and Weight Loss.** Figures 13 and 14 show the relationship between drying shrinkage and weight loss, expressed as percentage of initial weight, for geopolymer mortars containing 10% PWA or 10% OPA. At the longer curing times, the weight loss is smaller and so is the shrinkage. This may be due to the higher extent of geopolymerization reaction, in comparison with shorter curing time. Likely, the weight loss is mostly from evaporation of water.

**3.4. Drying Shrinkage and Compressive Strength.** The relationship of drying shrinkage with compressive strength at 7 days is shown in Figure 15. The high compressive strength samples have a stable aluminosilicate polymer structure [24] from geopolymerization, resisting shrinkage. Also, this mechanism may contribute to the effects of curing time on shrinkage.

**3.5. Bond Strength.** The results on bond strength to Portland cement mortar are shown in Figure 16. The bond strength improved with heat curing time for all formulations, and the samples containing PWA or OPA had dramatically higher slant shear strengths than the control sample with only MK, for all heat curing times tested. Particle size effects may contribute to this, as the larger particles (samples with

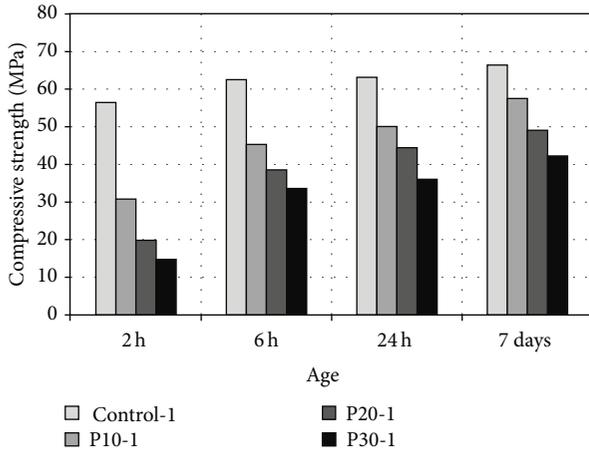


FIGURE 5: Compressive strengths of geopolymer mortars containing PWA, heat-cured at 80°C for 1h.

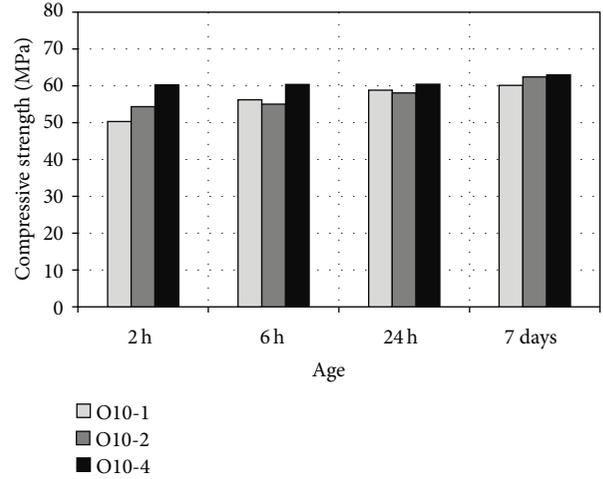


FIGURE 8: Compressive strength of geopolymer mortar containing 10% OPA.

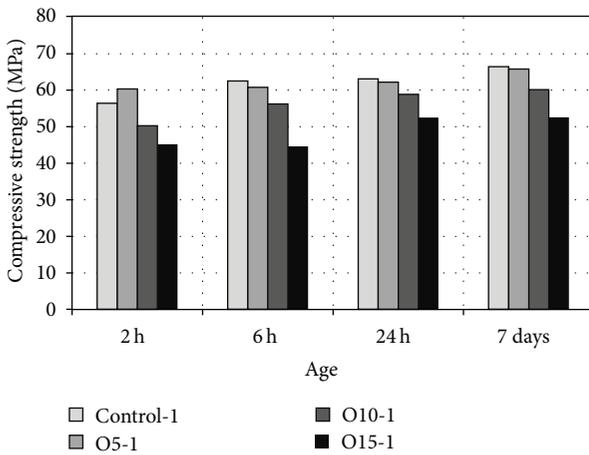


FIGURE 6: Compressive strengths of geopolymer mortars containing OPA, heat-cured at 80°C for 1h.

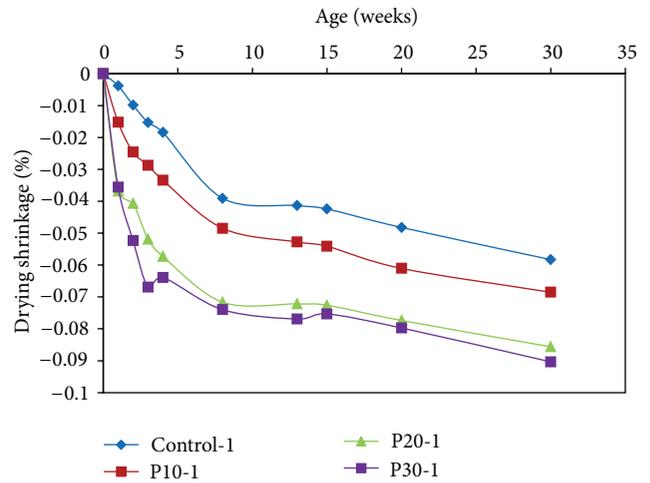


FIGURE 9: Drying shrinkages of geopolymer mortars containing PWA, heat-cured at 80°C for 1h.

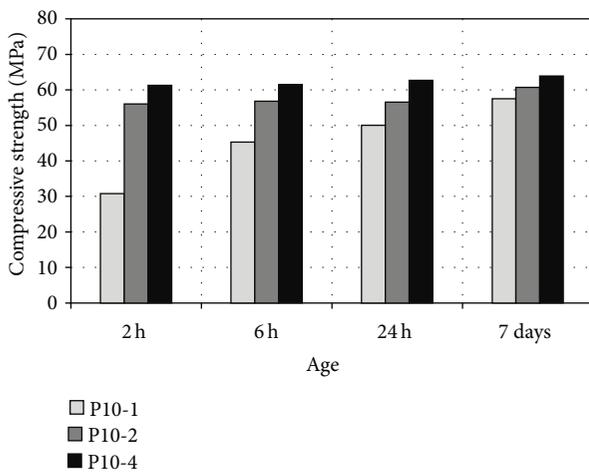


FIGURE 7: Compressive strength of geopolymer mortar containing 10% PWA.

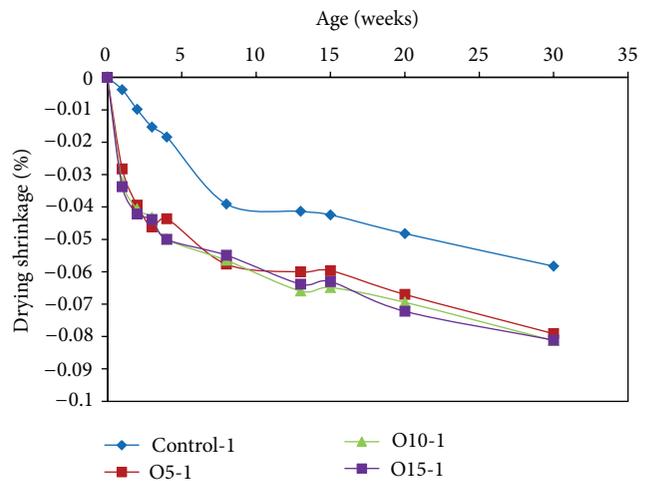


FIGURE 10: Drying shrinkages of geopolymer mortars containing OPA, heat-cured at 80°C for 1h.

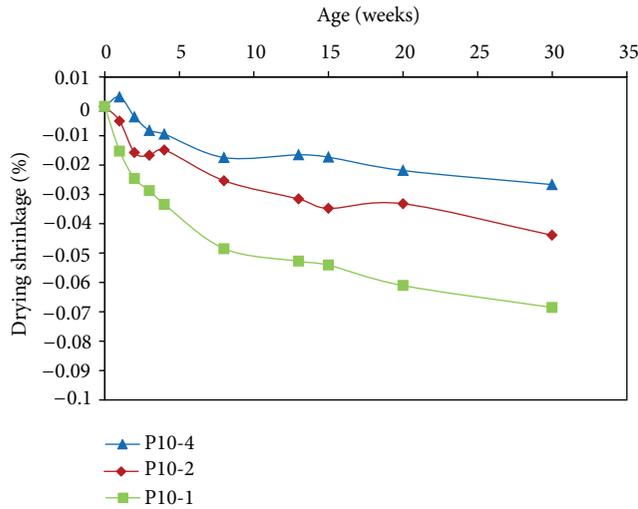


FIGURE 11: Drying shrinkage of geopolymer mortars containing 10% PWA.

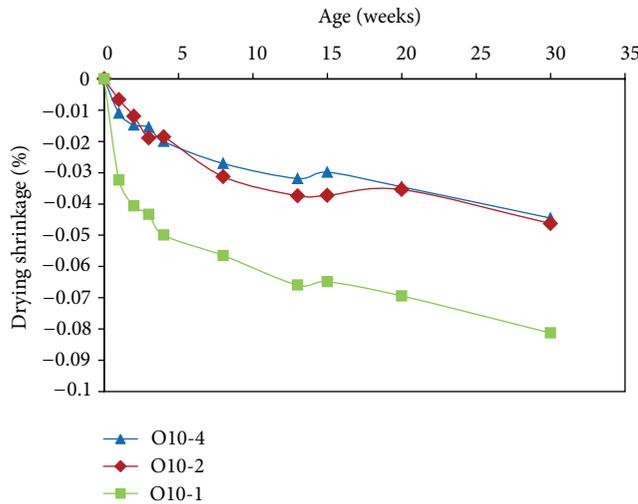


FIGURE 12: Drying shrinkage of geopolymer mortars containing 10% OPA.

PWA and OPA) have a comparatively low specific surface area. Then, the slurries containing PWA and OPA may flow better, improving the surface contact with the OPC samples. Another potential mechanism is that the fast setting control samples might harden before fully developing surface bonds with OPC. Finally, there is the possibility of actual chemical reaction effects.

#### 4. Conclusions

Geopolymer mortars from metakaolin with PWA or OPA were synthesized and studied, with the following main conclusions.

- (i) OPA appears more advantageous than PWA, as a supplementary raw material in these geopolymers, due to overall better strength characteristics.

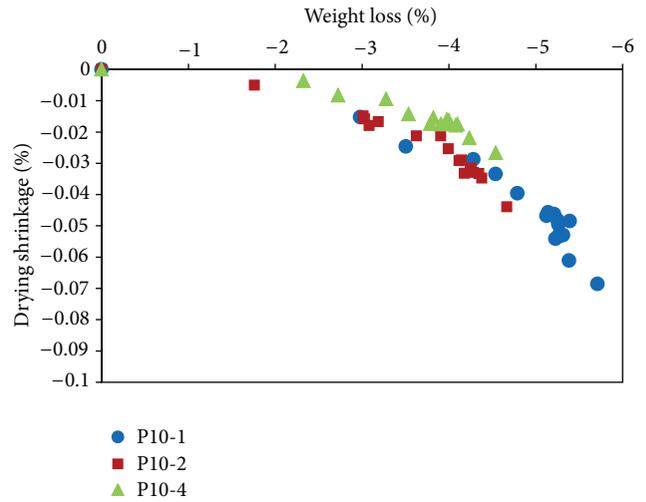


FIGURE 13: Relationship of weight loss on drying shrinkage with 10% PWA.

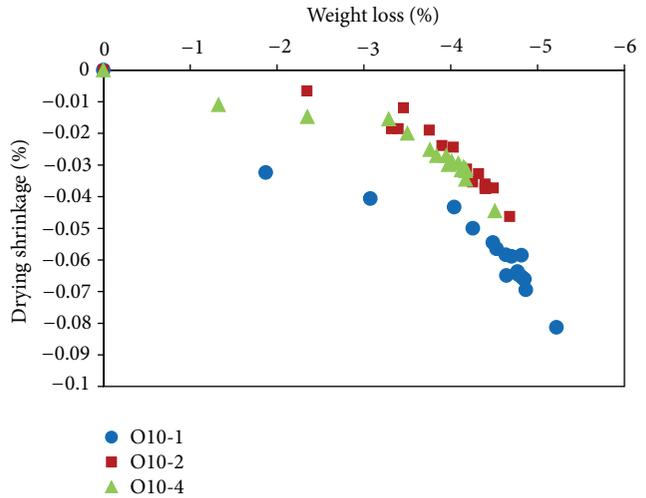


FIGURE 14: Relationship of weight loss on drying shrinkage with 10% OPA.

- (ii) Both OPA and PWA dramatically (see Figure 16) increased the bond strength to Portland cement mortar and may be necessary components in the geopolymers for use in concrete repair.
- (iii) Compressive strength of geopolymers can be improved by increased heat curing, here tested up to 4 h. However, with only 10% of OPA, heat curing at 80°C for 1 h already showed maximal strength, while with 10% PWA, longer cure times affect especially early strength development.
- (iv) Long heat curing times also decreased drying shrinkage, potentially due to the well-developed strength.
- (v) The longer heat curing times also used more water in the geopolymerization process, reducing the amount left to evaporate and detectable as weight loss.

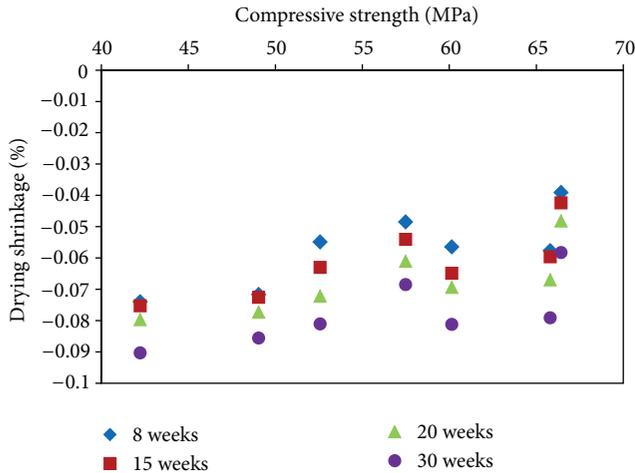


FIGURE 15: Relation between compressive strengths and drying shrinkage for samples heat-cured at 80°C for 1h.

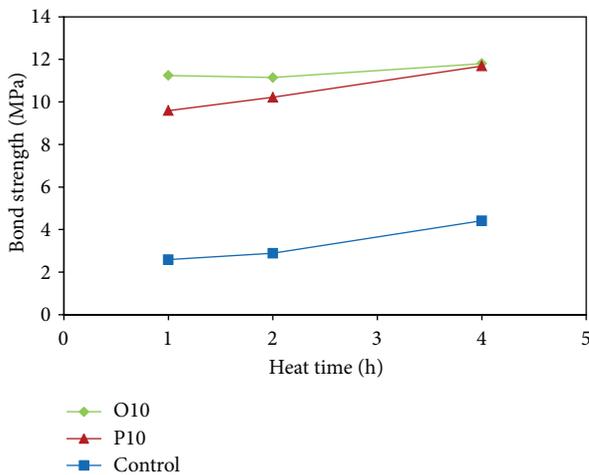


FIGURE 16: Bond strength to Portland cement mortar of select geopolymer samples, measured by compression test of slant shear.

The postcuring weight loss had a tight relationship to drying shrinkage.

Currently, geopolymer binders have not yet reached field applications. However, portable steam boilers may enable their practical use. Results from an ongoing field study of these geopolymers with high early strength will be the subject of a future publication, complementing the current laboratory study.

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