

Performance Evaluation of Metakaolin Based Geopolymer Containing Parawood Ash and Oil Palm Ash Blends

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Experimental tests were used to identify the optimum mix of metakaolin (MK) geopolymer mortars needed to achieve high early compressive strength. These tests investigated compressive strength, drying shrinkage, and sodium sulfate expansion of binary and ternary MK-based geopolymer mortar samples containing Parawood ash (PWA) and oil palm ash (OPA) in different replacement levels. The following amounts of PWA and OPA were used: 10 %, 20 %, 30 % and 5 %, 10 %, 15 % by weight of MK, respectively. Sodium hydroxide, sodium silicate and a curing temperature of 80 °C for 2 h were used as parameters to activate the geopolymerization of mortars. Thirteen geopolymer mortar formulations containing PWA and OPA were prepared, and the compressive strengths at 2 h, 6 h, 24 h, 7 days and 28 days were determined. The highest compressive strength of 62 MPa, after 2 h at 80 °C and 2 h at ambient temperature, was obtained with 5 % OPA.

Keywords: compressive strength, geopolymer, parawood ash, oil palm ash.

1. INTRODUCTION

The term “geopolymer” appears in the fields of materials science and materials engineering. The geopolymer process is a chemical reaction between aluminosilicate materials and alkaline solutions under elevated curing temperatures. Geopolymers are binders that exhibit good physical and chemical properties, and have a wide range of potential applications. Geopolymers were developed for use as construction materials [1]. Currently though, mainly cements are still used for construction [2–5] even in such specific applications as shielding barriers [6]. Products from geopolymers have certain advantages, including high early strength, high strength, low shrinkage, and resistance to chemical agents [7]. The important raw materials of geopolymers include fly ash, blast furnace slag, and metakaolin (MK) or calcined kaolin clay containing SiO₂ and Al₂O₃, which are the main chemical constituents. Recently, geopolymers have replaced conventional raw materials such as rice husk and bark ashes [8, 9].

Oil palm ash (OPA) is a by-product of the use of palm kernels, palm fibers, palm shells, and empty fruit bunches, when used as biomass fuel to replace petroleum in electricity generation. OPA has been reported to have main chemical component SiO₂. However, there have been few studies on the use of OPA to develop geopolymer binder in combination with MK and fly ash. Yusuf et al. [10] studied the binary replacement of MK with ultrafine palm oil fuel ash in geopolymer concrete with ground blast furnace slag to achieve the compressive strength of 69 MPa.

Parawood ash (PWA) is a by-product of burning Parawood lumber or waste, for example from furniture industry. Parawood is often used as raw material by biomass power plants. Currently, PWA is disposed of in

landfills, which has the potential to cause environmental problems for the industry and health risks for the public. It seems that there is no prior publication about the use of PWA as a material in geopolymer.

Drying shrinkage and sulfate attack are the most important problems concerning the durability of concrete structures. Under sulphate attack and high temperature conditions, binder paste undergoes deterioration resulting from expansion and shrinkage, cracking. Some researchers [11, 12] report that fly ash based geopolymers have low drying shrinkage characteristics. Chindaprasirt et al. [13] showed that high-calcium fly ash based geopolymer mortars present excellent volume stability with very little shrinkage when exposed to a 50 % RH environment.

Research by Sata et al. [14] shows that sodium sulfate expansion of fly ash geopolymer is lower than that of ordinary cement. The fly ash geopolymer mortars gave excellent resistance to sodium sulfate attack.

In the present study, geopolymers were prepared, with PWA and OPA ratios of 0 %–30 % and 0 %–15 %, respectively. They were prepared as hot mixtures, using sodium silicate and sodium hydroxide as activators, and heat-cured in an oven at 80 °C for 2 h. The study aimed to analyze the effects on compressive strength, drying shrinkage, and sulphate expansion, and on characteristics observed with SEM and XRD techniques.

2. EXPERIMENTAL PROGRAMS

2.1. Materials

The MK was derived from kaolin by calcination at 750 °C for 2 h. The raw kaolin came from a kaolin mine in Narathiwat province of southern Thailand. The diffractograms of the natural kaolin and calcined sample are compared in Fig. 1. The crystalline phases of MK consisted of quartz, illite, and microcline. The crystal in Fig. 1 show kaolinite and illite in untreated kaolin; and absence of

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kaolinite but presence of illite, microcline, and quartz in MK. According to Zibouche [15] and Wang, [16] quartz stabilizes the geopolymerization reaction. In PWA the crystalline phases detected are calcite, quartz, and arcanite, while OPA contains quartz and sylvite. PWA from the combustion of Parawood was collected from a biomass thermal power plant. OPA was collected from a palm oil mill in the Krabi province of southern Thailand. Both PWA and OPA were ground in a ball mill for approximately 4 h. In this study, distributions of the particle size were measured from wet dispersions using Malvern Hydro 2000 MU volume sample dispersion units available for the Mastersizer 2000 granulometer, are shown in Fig. 2. The major chemical compositions analyzed by X-ray fluorescence (XRF) of the cementitious materials are given in Table 1.

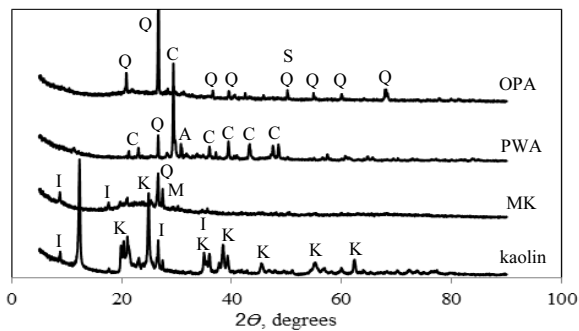


Fig. 1. XRD patterns of natural kaolin, MK, PWA and OPA. The peaks are indicated with Q – quartz, M – microcline, K – kaolinite, I – illite, A – arcanite, C – calcite, S – sylvite

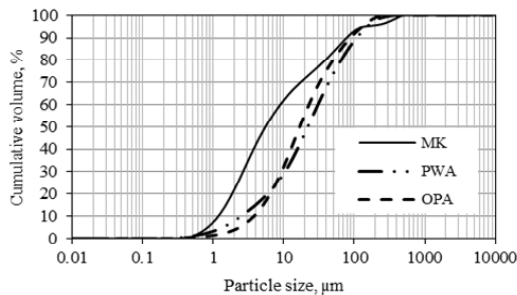


Fig. 2. Particle size distributions of raw materials

Table 1. Chemical compositions (wt.%) of materials used

Oxides	MK	PWA	OPA
SiO ₂	50.30	2.57	38.37
Al ₂ O ₃	41.02	0.53	1.48
Fe ₂ O ₃	1.05	0.56	3.01
CaO	0.33	41.19	13.84
TiO ₂	1.50	–	0.21
MgO	–	4.52	3.00
K ₂ O	4.08	16.11	14.09
Other	–	10.78	5.57

An alkaline solution was formed by mixing into water sodium hydroxide in flake form (NaOH with 98 % purity) and sodium silicate. The sodium silicate solution had a composition by weight of 14.14 % Na₂O, 27.67 % SiO₂ and 56.28 % H₂O. The composition was analyzed using inductively coupled plasma-optical emission spectrometer (ICP-OES) for Na₂O and SiO₂, and by drying at 103 °C–105 °C for H₂O. In this study, river sand was used as the fine aggregate of the geopolymer mortars. The

specific gravity and maximum granule size of the river sand were 2.51 mm and 4.75 mm, respectively.

2.2. Mixture proportions

Geopolymer mortar samples were mixed from the raw materials and river sand as inert filler. Then, sodium hydroxide was mixed into sodium silicate and added to water, and the mixture temperature became 74 °C ± 2 °C from reaction heat. When the powder raw materials and river sand were added, the mixture temperature became 48 °C ± 2 °C. The texture of the samples was quite sticky and fast-setting, and to cast them in an acrylic mold required effort.

The factors in the experimental design led to 13 different formulations (Table 2). This is a full factorial for PWA (0, 10, 20 or 30 % by wt. of MK) and OPA (0, 5, 10, or 15 % by wt. of MK), plus the case with 30 % PWA. The mass ratio of sand/raw materials (metakaolin, PWA and OPA)/activator/water was 3/1/0.83/0.45. The activator used was a mixture of sodium silicate to sodium hydroxide in a weight ratio of 2.5 : 1.

Table 2. Mixture proportions of geopolymer mortars (%)

Code of mixture	MK	PWA	OPA
Control	100	0	0
P10	90	10	0
P20	80	20	0
P30	70	30	0
O5	95	0	5
O10	90	0	10
O15	85	0	15
P10O5	85	10	5
P10O10	80	10	10
P10O15	75	10	15
P20O5	75	20	5
P20O10	70	20	10
P20O15	65	20	15

2.3. Test procedure

After geopolymer casting, the molds were wrapped with a polyvinyl sheet to prevent moisture loss, cured in an oven at 80 °C for 2 h, and aged with the wrapping removed at 30 °C ± 2 °C ambient temperature with 70 % ± 5 % relative humidity. The compressive strength was tested at 2 h, 6 h, 24 h, 7 days and 28 days of aging, in accordance with ASTM C109/C109M-97. The geopolymer mortar specimens were cubes of size (50 × 50 × 50) mm.

The drying shrinkage test was performed using a length comparator in accordance with ASTM C490-96. The geopolymer mortars were prepared using 2 h curing at 80 °C. After demolding, they were measured for initial size. Shrinkage was measured during a period of 1 to 30 weeks.

Sulfate expansion of the geopolymer mortars was determined according to ASTM C1012-96 using 5 % sodium. Both drying shrinkage and sulfate expansion tests were done with prismatic specimens of (25 × 25 × 285) mm, that were kept at ambient temperature of 30 °C ± 2 °C with 70 % ± 5 % relative humidity throughout this study. Sulfate expansion tests were conducted during a period of 1 to 30 weeks.

The chemical compositions of MK, PWA, and OPA were determined by X-ray fluorescence with a spectrometer (PW2400, PHILIPS) for the oxides.

A JMS-5800 LV model scanning electron microscope (JEOL, Japan) was used to identify the microstructure of the geopolymer mortars. Small scraps of the samples were tested using scanning electron microscopy.

Powder XRD analyses were conducted using an X'Pert MPD X-ray diffractometer (PHILIPS) at angles from 5° to 90° (2θ) using the clay and rock 0.4 program. The kaolin, MK, PWA, OPA, and geopolymer paste were characterized directly. XRD was conducted to identify the dominant crystalline phases and to detect the positions of the peaks.

3. RESULTS AND DISCUSSION

3.1. Compressive strength

The effects of PWA-OPA content on the compressive strength of metakaolin based-geopolymer mortar are shown in Fig. 3. The early compressive strengths at 2 h are in general good, partly because the geopolymer mortar was prepared as a hot mixture before oven curing it. The compressive strengths of mortars containing PWA are graphed in Fig. 3, a. The inclusion of PWA decreased the compressive strength relative to control. This is due to the reduction of Si and Al when MK is replaced by PWA, affecting the main geopolymerization reaction. Winnefeld [17] found that high calcium caused low strength, in part by its poor reactivity with alkaline activators, and fly ash based geopolymers have elevated calcium content. Test results confirmed that adding CaO into raw materials decreased compressive strength. These results are similar to those of Temujin et al. [18], who reported a curing temperature of 70 °C. However, PWA has larger particle size than MK. This can reduce the compressive strength due to poor accessibility for reaction. Compressive strength values of geopolymer mortars containing OPA are in Fig. 3, b. The initial compressive strength at 2 h is lowered by OPA content of geopolymer mortars. The later strength development of 5 % OPA mixtures is better than the control, and this content appears near optimal. The compressive strengths kept improving over 28 days.

The compressive strength differences between 10 % contents of PWA and OPA may be due to different amorphous phase contents (see Fig. 1). Also the average particles sizes differ (see Fig. 2), and finer particles with greater surface area tend to be more reactive [19]. Finer particles may also fill cavities between large particles, increasing packing density and leading to stronger samples.

The compressive strengths of ternary PWA-OPA geopolymer mortars are shown in Figs. 3, c and d. The specimens P10 and P10O5 were duplicated, confirming that 5 % OPA improved the strength, 10 % PWA degraded it, and the combination in P10O5 was an improvement over control. However, replacement of raw materials with PWA containing a high amount of CaO may give unstable geopolymer binders [20].

3.2. Drying shrinkage

The drying shrinkages are presented in Fig. 4. Comparison with the control shows that PWA decreased drying shrinkage. The shrinkage was rapid during the first three weeks, then a lower rate of shrinkage was observed

until 30 weeks. Especially the geopolymer mortar with 30 % PWA had very low drying shrinkage. Similar trends in drying shrinkage were reported by Chareera [21]. Geopolymers with fine-sized calcined kaolin particles have high shrinkage [22]. This is due to fine particles having a larger geopolymerization reaction surface area, and if they are packed inadequately in the slurry they will produce high shrinkage.

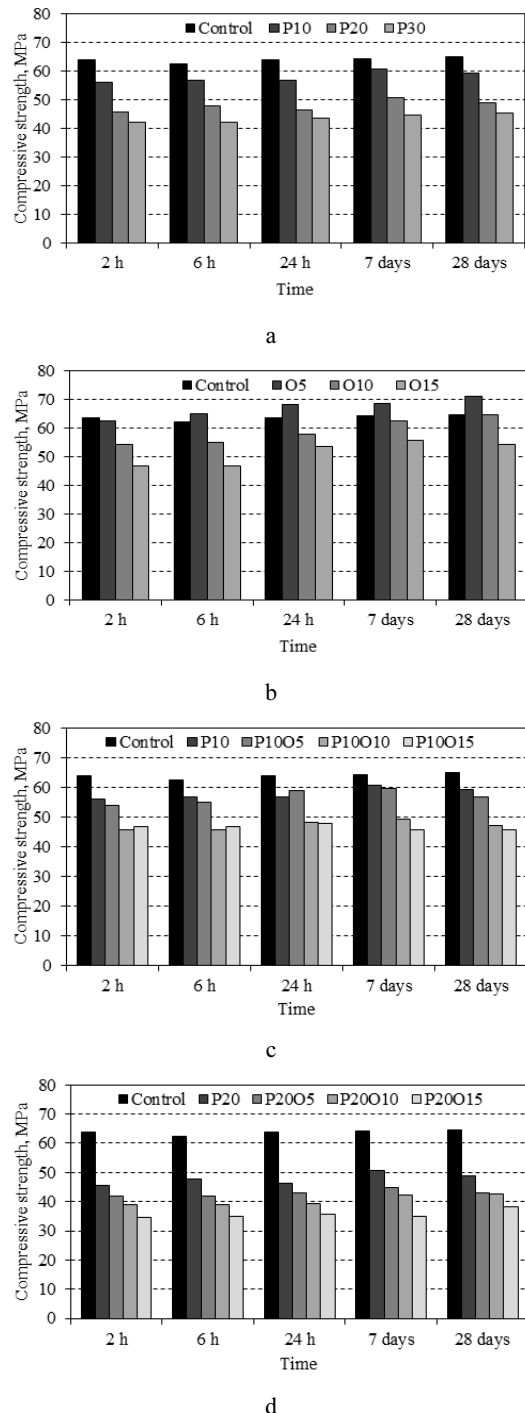
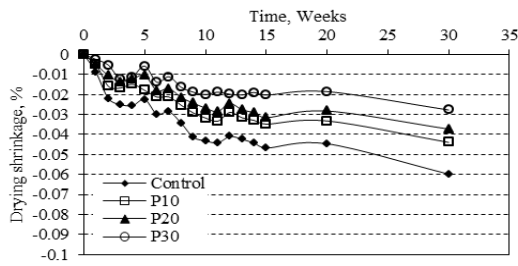
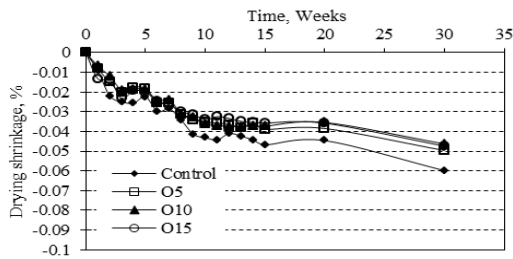


Fig. 3. Compressive strengths of geopolymer mortars: a – PWA; b – OPA; c – 10 % WPA/OPA; d – 20 % WPA/OPA

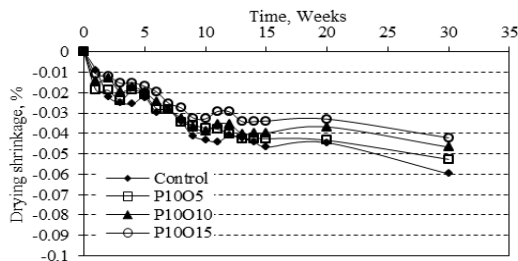
The drying shrinkages of geopolymer mortars containing OPA are shown in Fig. 4, b. OPA tends to decrease drying shrinkage. Shrinkage was again rapid for the first 3 weeks, and the rate then decreased gradually.



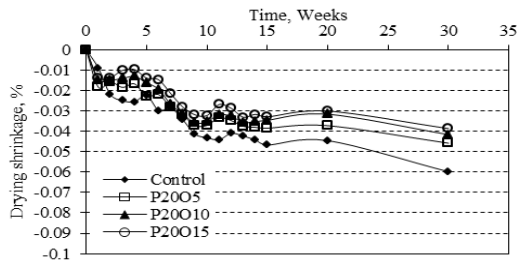
a



b



c



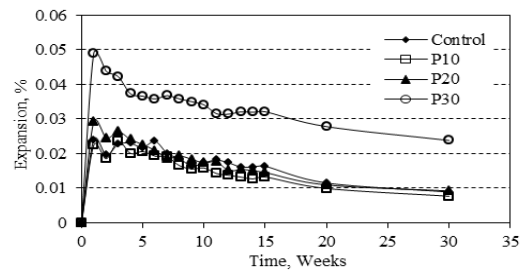
d

Fig. 4. Drying shrinkages of geopolymer mortars: a – PWA; b – OPA; c – 10 % WPA/OPA; d – 20 % WPA/OPA

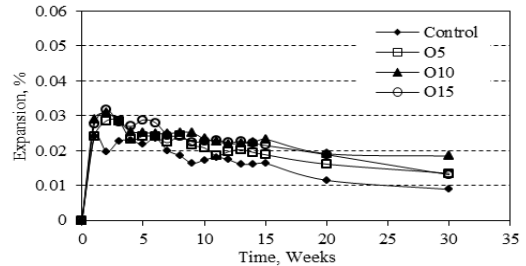
The drying shrinkages of ternary PWA-OPA geopolymer mortars are shown in Figs. 4, c and d. Again, substitution of MK by PWA-OPA lowered the shrinkage relative to control, especially at long times where the results are clear and stable. At early times, the shrinkages of some blends appear higher than of control. Portland cement mortars had drying shrinkages 0.0484 %, 0.0765 %, 0.0993 % and 0.1004 % measured at 7 days, 28 days, 2 months and 3 months, respectively [23]. In comparison our geopolymers shrunk less.

3.3. Sulfate expansion

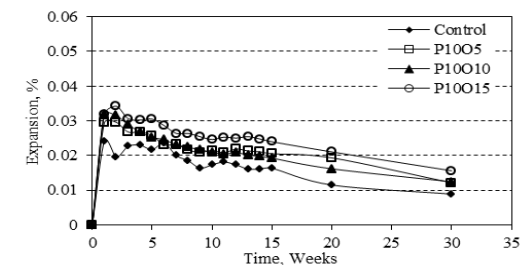
The expansions caused by sodium sulfate solutions are shown in Fig. 5. The early expansion in the first week of submersion, in sodium sulfate solutions, was rapid. After a maximum peak, the expansion decreased for the rest of the 30 weeks. Expansion of mortars containing only PWA is shown in Fig. 5, a. These mortars had a high resistance to the sodium sulfate attack. After submersion in the solution



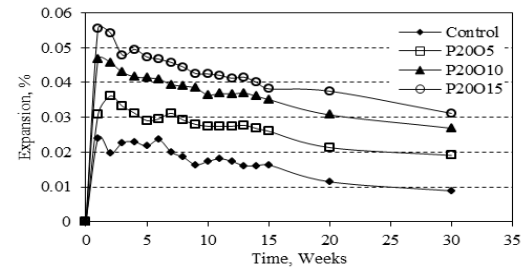
a



b



c



d

Fig. 5. Sodium sulfate expansion of geopolymer mortars: a – PWA; b – OPA; c – 10 % PWA/OPA; d – 20 % PWA/OPA

for 4 and 30 weeks, the expansions varied in the ranges from 0.020 % to 0.037 % and 0.007 % to 0.023 %, respectively. The expansion of mortars containing 10 % and 20 % PWA, submerged in sodium sulfate solutions, was similar to control immersed in Na_2SO_4 , see Figure 5, a. However, mortar containing 30 % PWA had the highest expansion, possibly due to the average particles size (d_{50} 6.308 μm for MK against 25.128 μm for PWA), as larger particle size tends to increase sulfate expansion [24].

The expansion of geopolymer mortars containing the OPA submerged in sodium sulfate solutions is shown in Fig. 5, b. The expansion ranges of the geopolymer mortars were 0.023 % to 0.027 % and 0.009 % to 0.018 %, for aged 4 and 30 weeks, respectively. Later expansions of these mortars containing OPA were much higher than control. Expansion time profiles of ternary PWA-OPA geopolymer mortars are shown in Figs. 5, c and d. The expansions were in the range from 0.023 % to 0.049 % at 4 weeks of immersion, and from 0.009 % to 0.031 % at 30

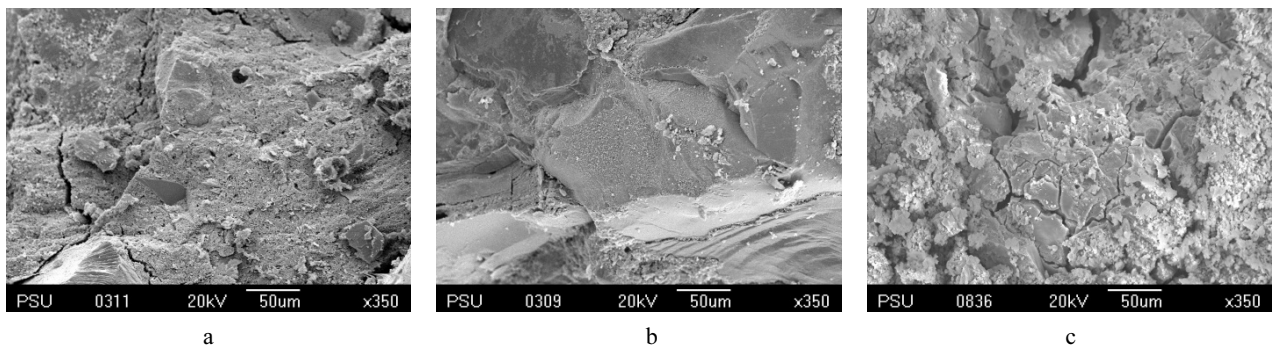


Fig. 6. Microstructure of the surface of hardened geopolymer mortar: a – control; b – O5; c – P20

weeks. Notably, the ternary geopolymer mortars containing PWA-OPA expanded more than the control mortars.

It was observed that high strength geopolymer samples expanded less than lower strength samples, when submerged in Na_2SO_4 .

Chareerat [21] reported that the particle size of the raw materials was essential to the expansion of the submerged geopolymer in sulfate solution. The raw materials with a small particle size expanded less than with large particles. For ordinary portland cement the addition of nano-silica to the concrete mixture substantially improves sulfate resistance [25]. Moreover, high CaO content of raw materials also caused large porosity [26]. So, increasing the particle size and CaO content produced greater porosity, in agreement with the images in Fig. 6. The increased porosity allows penetration of the sulfate solutions into the mortar, and this may contribute to the expansion. This matches our observation that the samples P30, P20O10 and P20O15, with 30 % or higher partial replacement of MK, had the highest expansions. However, hardened geopolymer became a strong structure that could not be cracked [22].

In general, the hydration products were provided by ordinary Portland cement. These products were highly sensitive to sulfate attack compared with the geopolymerization products. The main geopolymerization product is from sodium hydroxide, sodium silicate and other raw materials (Si and Al are essential chemical components). Geopolymers submerged in sodium and magnesium sulfate solutions have a comparatively stable cross-linked aluminosilicate polymer structure [27].

3.4. Microstructure

SEM results shown in Fig. 6 represent typical microstructures of geopolymerization products obtained in this study. Some unreacted raw materials were covered with flakes in the Control and P20 samples (see Figs. 6, a and c) and the samples appeared nonhomogeneous. In comparison the O5 sample had less unreacted raw materials (see Fig. 6, b), and appeared more homogeneous. Also the geopolymer mortar microstructure appeared finer in O5. This may be related to the high compressive strengths of the O5 samples shown in Fig. 3.

3.5. X-ray diffraction

The XRD patterns of Control, O5 and P20 samples are shown in Fig. 7, at 28 days of aging. The geopolymers were aluminosilicates with crystallised compounds, such as quartz (SiO_2), microcline (KAlSi_3O_8), and calcite (CaCO_3). The Control showed broad peaks from quartz at 21, 26, 36,

42 and $50^\circ 2\theta$, and from microcline at $27^\circ 2\theta$. The CaO from PWA in P20 samples had calcite mineral causing a broad peak at $29^\circ 2\theta$, and these samples had low strength. Geopolymer containing OPA (O5) had XRD pattern indicating amorphous structure. In all three geopolymers, amorphous materials are an important fraction, with an evident broad peak in the $25^\circ\text{--}35^\circ 2\theta$ range. The amorphous fraction in metakaolin remains in the geopolymers after the polymerization reactions.

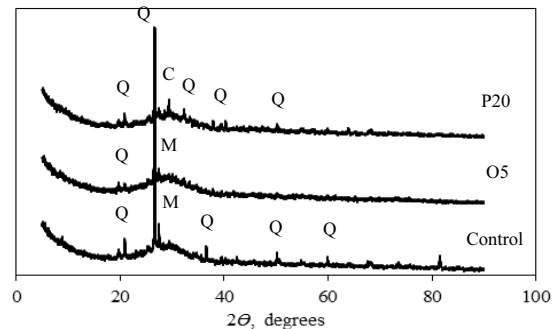


Fig. 7. X-ray diffraction patterns of geopolymer: Q – the peaks of quartz; M – the peaks of microcline; C – the peaks of calcite

4. CONCLUSIONS

The influence of PWA and OPA contents on binary and ternary MK-based geopolymer mortars was experimentally investigated.

The compressive strength improved by adding 5 % OPA relative to MK by weight. Both PWA and OPA significantly reduced drying shrinkage.

The CaO content was in general harmful to the geopolymers, and with 30 % PWA the expansion in sodium sulfate solutions was excessive.

Only minor effects on compressive strength were observed with long aging times, and these raw materials appear suitable for applications requiring the high strength,

low shrinkage, and superior durability properties of rapid-set geopolymer mortars demonstrated.

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