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EFFECT OF CURING REGIMES ON STRENGTH OF GEOPOLYMER MORTARS BASED ON FLY ASH AND METAKAOLIN

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ABSTRACT

Geopolymer materials have garnered significant attention as sustainable alternatives to traditional Portland cement-based materials. This study explores the effect of various curing regimes on the compressive strength (at 7 and 28 days) and flexural strength (at 28 days) of geopolymer mortars composed of fly ash (FA) and metakaolin (MK) in varying proportions. Four mixes were used: 100% FA; 60% FA+ 40% MK; 50% FA+10% OPC+ 40% MK; and 100% OPC. The binders were activated using 15% sodium silicate (solid content) with an alkali modulus (Ms = SiO_2/Na_2O) equal to 1. The curing conditions included ambient curing and elevated temperature curing at 80 °C under different setups. The results demonstrate that the curing regime significantly influences the mechanical properties of alkali-activated mortars (AAMs). Where, the results showed that heat curing regime enhanced the flexural, early-age compressive strength and the microstructure, in comparison with other regimes. The findings provide valuable insights into optimizing curing practices for geopolymer mortars to achieve desired mechanical properties. Replacing 40% of fly ash with metakaolin improved the microstructure of the matrix and decreased micro-cracks.

Keywords: Fly ash, Metakaolin, Heat curing, Alkali-activated, Geopolymer.

INTRODUCTION

Cement is one of the most extensively used building materials in the world, since the infrastructure and economic growth are directly related to cement uses. Moreover, concrete is coming in the second place of consumed materials, just after water (Qizi & Barotaliyevna, 2023). However, cement industry is one of the main sources of CO_2 emissions as a result of emission sources due to calcination and the emissions of fuels in cement kiln (Gale et al., 2003; Worrell et al., 2001). Where, it contributes with 5-8 % of all anthropogenic CO_2 emissions worldwide (Hendriks et al., 2003). However, in recent years, the demand has increased for sustainable construction materials that led to the exploration of alternatives to ordinary Portland cement (OPC). Alkali-activated materials (AAMs), particularly those based on fly ash (FA) and metakaolin (MK), are among the most promising options due to their low carbon footprint and ability to exploit industrial by-products, as well as improving durability and chemical resistance if it produced in appropriate way

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(Amer et al., 2021; Bashar et al., 2016; Humad, Kothari, et al., 2019; J.Davidovits, 1991; Provis & Deventer, 2009). AAMs are produced through the activation of aluminosilicate precursors with alkaline solutions (activators), resulting in a three-dimensional aluminosilicate network that imparts high mechanical strength and durability (Davidovits, 1994; Juenger et al., 2011; Provis & Jannie S.J. van Deventer, 2014; Rattanasak & Chindaprasirt, 2009; Shi et al., 2011).

The effects of curing temperature, curing duration, activator/ fly ash ratio and the type of activator, of AAMs mechanical characteristics were explored by other researchers (El-Wafa & Fukuzawa, 2018),(Abd Razak et al., 2022), where, steam curing gave the best early-age strengths. Moreover, (Palomo et al., 1999) found that, increasing curing time enhanced the mechanical strength, and temperature is an important factor especially for the first 2 and 5 hr. of curing. Furthermore, the type and the dose of the activator effected essentially the development of reactions.

In general, curing procedure and heat treatment influenced significantly the microstructure and mechanical properties of alkali-activated materials system through accelerating the dissolution and precipitation reactions, (Jaarsveld et al., 2002),(Hardjito et al., 2008). Moreover, humid curing recorded decreasing in drying shrinkage, (Yuxin Cai 1 et al., 2019) while dry curing caused a severe micro-cracking in the binder matrix, which reduced the overall strength.

(Humad, Provis, et al., 2019) demonstrate that, heat curing increased the compressive strengths of alkali-activated slag concrete activated with 10% sodium silicate (Ms1) and sodium carbonate. All heat-treated samples reduced drying shrinkage by 50% after one month of sealed curing and the samples showed the lowest micro cracking and most consistent microstructure in comparison with lab-cured samples. However, (Yaprak et al., 2019) reported that alkali-activated mortars based on slag, fly ash, and glass powder treated under steam-cured recorded higher early strength values in comparison with samples cured with water. While, (Masi et al., 2021) found that alkali-activated mortars based on pre-treated fly ash and cured at ambient temperature had equivalent mechanical performance to specimens cured at 70 ◦C.

On the other hand, low curing temperatures and slow hydration, allowed unfavorable pores to form and caused a reduction in compressive strength (Wei et al., 2021). Since, producing of geopolymer materials affected strongly by curing temperature, therefore, increasing temperature caused developing alkaline activation and lowered ion mobility by providing reactant particle energy (Alonso & Palomo, 2001). Moreover, curing conditions influence the kinetic reaction, microstructure development, and the performance of the final matrix products. However, due to the varies type and chemical composition of precursors, more researches are needed to study the effect of curing procedures on the properties and microstructure of AAMs.

Since geopolymer material still a new construction alternative material to cement and it affected strongly by many factors such as binder and alkali-activator type, curing regimes and activator dosage, therefore, more researches are needed. This study aims to add more investigations about the effect of different curing procedures on strength development and microstructures of alkali-activated mortars based on FA and MK.

The aim of this study is to investigate the impact of different curing regimes on the mechanical strength development of geopolymer mortars synthesized from fly ash and metakaolin.

MATERIALS

The materials used in this study were Iraqi Portland Cement brand Karasta that used for the reference mix. Fly ash (FA) Class F obtained from China (Hebei Weiran Building Materials Technology Co., Ltd) and metakaolin (MK) sourced from calcined kaolinite clay obtained from Dewekhla, Al-Ramadi desert /Iraq. The liquid sodium silicate (SS) produced in the United Arab Emirates, had an alkali modulus (Ms = SiO2/Na2O) of 2.44, with a silicon dioxide (SiO2) content of 32-33 wt.%, a sodium oxide (Na2O) content of 13.1-13.7 wt.%, and a solids content of 54.1 wt.%. To achieve an alkali modulus equal to (1), sodium hydroxide (SH) flakes, with a purity of 97%, were added to sodium silicate to adjust the alkali modules (Ms). Figure 1 shows the FA, MK, OPC materials and activator. The proportion of alkaline activator was 10 % as weight percent of binder. The specific gravity and chemical composition of the cement, FA and MK are shown in Table 1, Cement was tested according to Iraqi specifications (IQS No.5, 1984), while FA and MK were tested following ASTM C618 (C618, 2015). The water to binder ratios (w/b) that used in this study were (0.4), plasticizer (lignosulphonate/ Zhejiang Nader Iot Technology Co Ltd) obtained from China was used in dosage of 1% as weight percent of binder, moreover, the binder to sand ratio was 1:2.

MIX PROPORTIONS

Mix proportions and curing procedures of Alkali-activated mortars are illustrated in Table 2.

SPECIMEN PREPARATION AND CURING PROCEDURE

The solution of alkali activator sodium silicate (SS) with alkali modulus (Ms) equal to (1) and mixing water were prepared and mixed one day before casting. To produce the alkali-activated mortars, the dry ingredients were mixed for 2 minutes at low speed in an 8-liter capacity mixer according to the mix proportion that is presented in Table 2. Plasticizer (lignosulphonate) (PL) in powder form was used in 1% of binder weight, was added at the initial stage with the dry ingredients. Then the alkali activator solution and mixing water were added. Then all ingredients were mixed for 3 minutes to have homogenous mix. After that, fresh properties were tested. For hardened properties, the molds were cleaned and lubricated with oil, then mortars were cast into molds and manual compacting was used following the ASTM C109 (C109/109M-16a, 2016) for compressive tests, C348 (C348, 1998) for flexural tests.

Then, five different curing regimes were employed in this study as shown below, see Figure 2 & Figure 3 and Table 2.

- **Room then Water Curing (RW):** Specimens were cast at lab. then demolded after 24 hr. and cured in water up to 28 days.
- **Heat (Oven) then Water Curing (OW):** Specimens were cast at lab. then cured in oven (80°C) for the first 24 hours, then demolded and immersed in water up to 28 days at room temperature (25±3) °C and (50±10) % relative humidity.

- **Heat or Oven Curing (OC):** Specimens were cast at lab. then cured in oven (80°C) for the first 24 hours, then demolded and kept in plastic films at room temperature (25±3) °C and (50±10) % relative humidity up to 28 days.
- **Room Curing (RC):** Specimens were cast at lab. then demolded after 24 hr. then kept in plastic films at room temperature (25±3) °C and (50±10) % relative humidity up to 28 days.
- **Room then Oven curing (ROC):** Specimens were cast at lab. then demolded after 24 hr. then kept in plastic films and cured in oven (80°C) for 24 hr. After that, they kept in plastic films at room temperature (25±3) °C and (50±10) % relative humidity up to 28 days.

TESTS

Fresh properties

The flow test was used to measure workability and uniformity of alkali-activated mortars. This test determines mortar spread and adhesion following the ASTM C1437 standards (C1437, 2013). The diameter of the spread mortar was measured in two perpendicular directions as shown in Figure 4, and the average diameter was calculated.
The Vicat Apparatus was used to determine the initial and final setting times of alkali-activated paste, as shown in Figure 5, following the ASTM C191 standard (C191-13, 2005).

Hardened Properties

The determined mechanical properties of the alkali-activated mortars were compressive and flexural strengths. The compressive strength was measured on cube specimens having dimensions of 50×50×50 mm. Three mortar cubes were tested for each mix after 7- and 28-days age following the ASTM C109 standard (C109/109M-16a, 2016), as shown in Figure 6. The loading rate was kept constant at 900 N/sec.

The flexural strength was determined on mortar specimen beams having dimensions of 40×40×160 mm at 28-day age. The three point-bending method was used following ASTM C348 (C348, 1998) standard, as shown in Figure 7.

Microstructural characteristics

The microstructural features of alkali-activated mortars were analyzed using Scanning Electron Microscopy (SEM) on 28-day specimens to assess morphology, matrix compactness, and the presence of microcracks or unreacted materials.

RESULTS AND DISCUSSION

Flow test results

The flow test results are presented in Figure 8. No bleeding was detected in any mix of the alkali-activated mortars, but the mixes exhibited a relatively fast slump loss.

The results of slump flow test presented different values for each mix based on the

proportions of the binder components, where the mix with 100% fly ash showed the highest slump flow value and decreasing the fly ash proportion % led to decrease the flow test value, as shown in fig. 8. The conventional cement mortar (Ref) demonstrated lower flow values compared to the F100 geopolymer mixture, that related to the spherical glassy particles shape of fly ash, where this spherical shaped of particles act as mini-ball bearings within the mortar mix, which provide a lubricant influence and also reduce the frictional losses and give flat work finish ability in mixture (Babor et al., 2009; Duggal, 2019; Jiao et al., 2018; Ulusoy, 2023), (Chen & Tang, 2024; de Hita & Criado, 2022; Fang et al., 2018; Hameed et al., 2022; UPADHYAY et al., 2007; Vyas et al., 2021). Moreover, MK particles have high specific surface area and high electrostatic charge density which increased the water demand and caused a reduction in flow test results (Alonso & Palomo, 2001).

Setting time test results

The results of initial and final setting time of geopolymer paste mixes are presented in Figure 9. The results demonstrated that the binder components proportion significantly affect the setting time of geopolymer mortars, where the geopolymer mortars mix displayed longer initial and final setting time in comparison with Ref. mix. Moreover, replacing fly ash with 40% metakaolin (Mixes F6M4 and F5P1M4) caused decreasing in initial and final setting time in comparison with mix containing 100% fly ash (Mix F100). That related to the lower initial reactivity of fly ash at room temperature, moreover, fly ash needs for a pH ≥13 to react at an expressive rate (Bernal et al., 2013; Nath & Sarker, 2014; Sakai et al., 2005). Similar results gained by others (Humad, Kothari, et al., 2019; Lee & Lee, 2013).

Compressive strength test results

The compressive strength test results of experimental mortars are presented in Figure 10. The data indicated that the curing regimes significantly affected the strength development of geopolymer mortars based on fly ash and metakaolin. Utilization of heat curing led to a rapid gain in strength, mainly at early ages, where heat curing showed the highest strength values at 28 days.

The compressive strength test results of reference mortars and geopolymer that cured in different regimes revealed different behavior depending on the binder components. In general, the compressive strength results of geopolymer mortars recorded lower values in comparison with reference mixes for all curing regimes, as shown in Figure 10. Where, the compressive strength values of Geopolymer mortars ranged between 13 to 22 MPa at 7-day age and between 20 to 32 MPa at 28-day age. While the compressive strength test results of reference mixes cured in different regimes ranged between 26 to 35 MPa at age 7-day and 41 to 51 MPa at age 28-day. The reference mixes specimens that cured in oven for 24hr. then in water (Ref-OW) exhibited the highest compressive strengths at 7 and 28 days, as shown in Figure 10-A.

For Geopolymer mortars specimens that are based on 100% fly ash, the best curing regime was the oven curing for 24 hr. then kept in plastic film (Mix F100-OC) as shown in figure 10-B. In general, in other studies using fly ash as binder in producing alkali-activated material, caused delaying in setting times, reduced the Poisson's ratio, modulus of elasticity and compressive

strength, conversely increasing ductility and toughness, (Ali Naqi et al., 2022; Criado et al., 2016; Kaya & Köksal, 2021; Lee & Lee, 2013; Matsuda et al., 2019; Mohamed, 2023). That could be related to the experiential further coarser pore size distribution in comparison with alkali-activated mortars based on blast furnace slag, (Al-Majidi et al., 2016; Azimi & Toufigh, 2023; Ebrahim et al., 2024; Ferna´ndez-Jime´nez et al., 2003). Moreover, using the heat curing (Mix F100-OC) enhances the initial rate of hydration by accelerating the dissolution of aluminosilicate precursors and promoting fast formation of C-A-S-H and N-A-S-H (Bhina et al., 2023; Kong et al., 2021; Nongnuang et al., 2022; Rabie et al., 2022).

Replacing the fly ash with 40% metakaolin (Mix F6M4) or with 10% Ordinary Portland cement + 40% metakaolin (Mix F5P1M4) displayed higher compressive strength values in comparison with 100% fly ash (mix F100). However, both mixes (F6M4) and (F5P1M4) presented approximately similar compressive strength values and comparable for all curing regimes, as shown in figure 10-C and figure 10-D. Moreover, the oven curing (OC) was the best curing regime for all Geopolymer mortars, agreed with others (Al-shathr & Al-attar, 2016; Jaarsveld et al., 2002; ÖMÜR et al., 2023; Rangan & Hardjito, 2005).

Flexural strength test results

The flexural strength test results that are shown in Figure 11, presented a similar pattern to the compressive strength test results. The flexural strength values of geopolymer mortars recorded lower values in comparison with reference mixes for all curing regimes. The flexural strength values of geopolymer mortars ranged between 4 to 8 MPa at 28-day age. While the flexural strength test results of reference mixes cured in different regimes ranged between 8 to 11 MPa at 28-day age. The reference mixes specimens that are cured in oven 24hr. Then in water (Ref-OW) exhibited the highest flexural strengths, as shown in Figure 11-A.

For Geopolymer mortars specimens that are based on 100% fly ash, the best curing regime was the oven curing (Mix F100-OC) as shown in Figure 11-B. Replacing the fly ash with 40% metakaolin (Mix F6M4) or with 10% Ordinary Portland cement + 40% metakaolin (Mix F5P1M4) displayed little higher flexural strength values in comparison with 100% fly ash (mix F100). However, both mixes (F6M4) and (F5P1M4) presented comparable flexural strength values for all curing regimes, as shown in Figure 11-C and Figure 11-D. Moreover, the oven curing for 24 hr. and kept the specimens in plastic film (OC) was the best curing regime for all geopolymer mortars because oven curing enhances the dissolution of Si and Al from fly ash, speeding up polycondensation into C-A-S-H and N-A-S-H gels.

Microstructural characteristics

The Scanning Electron Microscope (SEM) analysis of the oven curing for 24 hr. and kept in plastic film (OC) specimens at 28 days revealed a microstructure with fewer unreacted fly ash particles. The results revealed that the oven cured specimen contains 60% fly ash and 40% metakaolin (Mix F6M4) exhibits denser and more homogeneous matrix with no micro-cracks in comparison with mix that contains 100% fly ash (mix F100) as shown in Figure 12-a and Figure 12-b. That could be related to the formation of crystalline phases such as zeolite and hydrosodalite, which are indicative of a higher degree of geopolymerization in thermally cured samples that contain

metakaolin (Marsh, 2019; Mishra et al., 2022; Nguyen et al., 2017; Randers et al., 2024; Yılmaz et al., 2023; Yu & Jia, 2022).

The heat curing accelerated the strength gaining in samples and that could be related to enhancing the kinetics reaction and more full dissolution of the FA and MK particles, which led to more extensive and interconnected geopolymer network, (Muhammad & Baharom, 2019; Nongnuang et al., 2022; Rabie et al., 2022; Bilondi et al., 2025).

CONCLUSION

- The curing method significantly influences the strength and microstructural development of geopolymer mortars made from fly ash and metakaolin.
- Oven curing at elevated temperatures enhances compressive and flexural strengths, especially at early ages, due to improved reaction kinetics and microstructural development.
- Water curing and ambient curing result in lower strength compared to oven curing, which may not be adequate for applications requiring high early strength.
- Optimizing curing conditions is crucial to achieving desired performance in geopolymer materials, as curing temperature and methods play a key role in hydration and strength development.
- Replacing 40% of fly ash with metakaolin improves the matrix microstructure under heat-cured regimes and reduces microcracks.

TABLES

Table 1. The specific gravity and chemical composition of cement, fly ash and metakaolin

Chemical composition, %	Cement	Fly ash	Metakaolin
Silica (SiO_2)	20.13	83.10	81.84
Alumina (Al_2O_3)	5.67	6.18	12.01
Iron Oxide (Fe_2O_3)	3.96	2.08	0.68
SiO_2+ Al_2O_3+ Fe_2O_3	-	91.36	94.53
Sulfate (SO_3)	1.63	0.27	0.20
Lime (CaO)	59.00	2.67	0.71
Magnesia (MgO)	2.11	0.33	0.19
Loss on ignition (L.O.I)	3.25	3.10	3.75
Specific gravity	3.15	2.40	2.61

Table 2. Mix proportion and curing procedure of Alkali-Activated mortars.

Mix I D	OPC%	F A %	MK %	SS %	Binder: Sand	w/ b	Plastci z er	Curing

F 100- RWC	0	100	0	15	1:2	0.4	1%	Sealed molds after casting in lab., Demolding after 24hr. at ambient temperature then water cured (RW)
F 6M4- RWC	0	60	40	15	1:2	0.4	1%	
F 5P1M4- RWC	10	50	40	15	1:2	0.4	1%	
Ref- RWC	100	0	0	15	1:2	0.4	1%	
F 100- OWC	0	100	0	15	1:2	0.4	1%	Sealed molds after casting in lab., Oven cured for 24hr. at 80° C, then Demolding and water cured (OW)
F 6M4- OWC	0	60	40	15	1:2	0.4	1%	
F 5P1M4- OWC	10	50	40	15	1:2	0.4	1%	
Ref - OWC	100	0	0	15	1:2	0.4	1%	
F 100- RC	0	100	0	15	1:2	0.4	1%	Sealed molds after casting in lab., Demolding after 24hr then kept samples in plastic film at ambient temperature (RC)
F 6M4- RC	0	60	40	15	1:2	0.4	1%	
F 5P1M4- RC	10	50	40	15	1:2	0.4	1%	
Ref - RC	100	0	0	15	1:2	0.4	1%	
F 100- OC	0	100	0	15	1:2	0.4	1%	Sealed molds after casting in lab., Oven cured for 24hr. at 80° C, Demolding then kept samples in plastic film at ambient temperature (OC)
F 6M4- OC	0	60	40	15	1:2	0.4	1%	
F 5P1M4- OC	10	50	40	15	1:2	0.4	1%	
Ref - OC	100	0	0	15	1:2	0.4	1%	
F 100- ROC	0	100	0	15	1:2	0.4	1%	Sealed molds after casting in lab., Demolding after 24hr then oven cured for 24hr. at 80° C then kept samples in plastic film at ambient temperature (ROC)
F 6M4- ROC	0	60	40	15	1:2	0.4	1%	
F 5P1M4- ROC	10	50	40	15	1:2	0.4	1%	
Ref - ROC	100	0	0	15	1:2	0.4	1%	

Where symbols denote: **F 100:** 100% fly ash, **RWC:** room then water curing, **F 6M4:** 60% fly ash +40% metakaolin, **OWC:** Oven then water curing, **P1:** 10% Ordinary Portland cement, **OC:** oven curing, **RC:** room curing, **ROC:** room then oven curing.

FIGURS

Fig. 1. The materials that used in this study a) Fly ash, b) Ordinary Portland cement, c) metakaolin, d) SS activator.

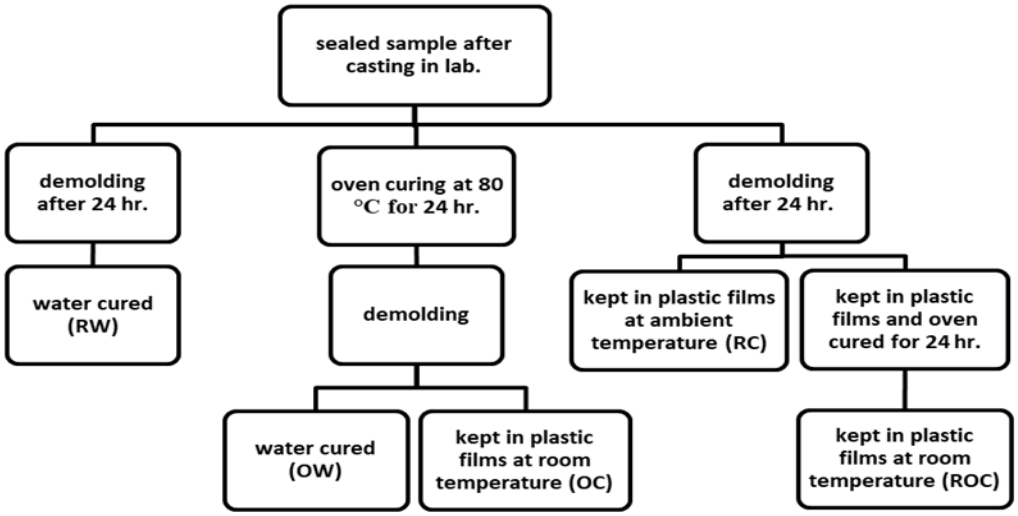

Fig. 2. Curing regimes that were used in this study.

Fig. 3. Curing Regimes, a) Sealed molds at ambient temperature, b) Sealed molds in oven at 80°C, c) Demolded sample, d) Sealed cured sample at ambient temperature after demolding, e) Cured sample in water after demolding.

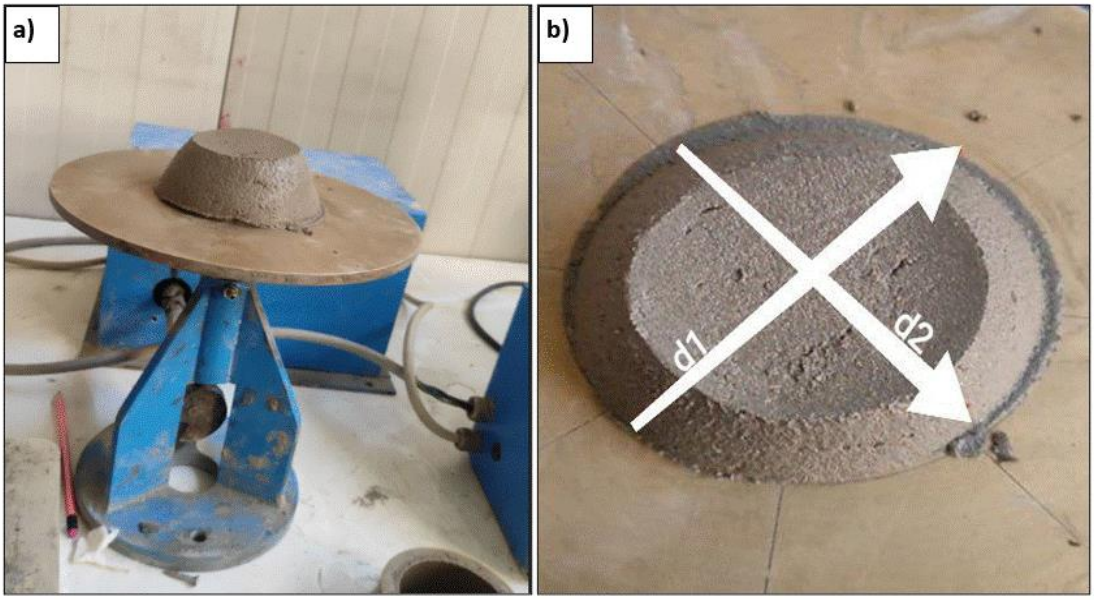

Fig. 4. Flow test of mortars a) Apparatus of flow test, b) The examined sample.

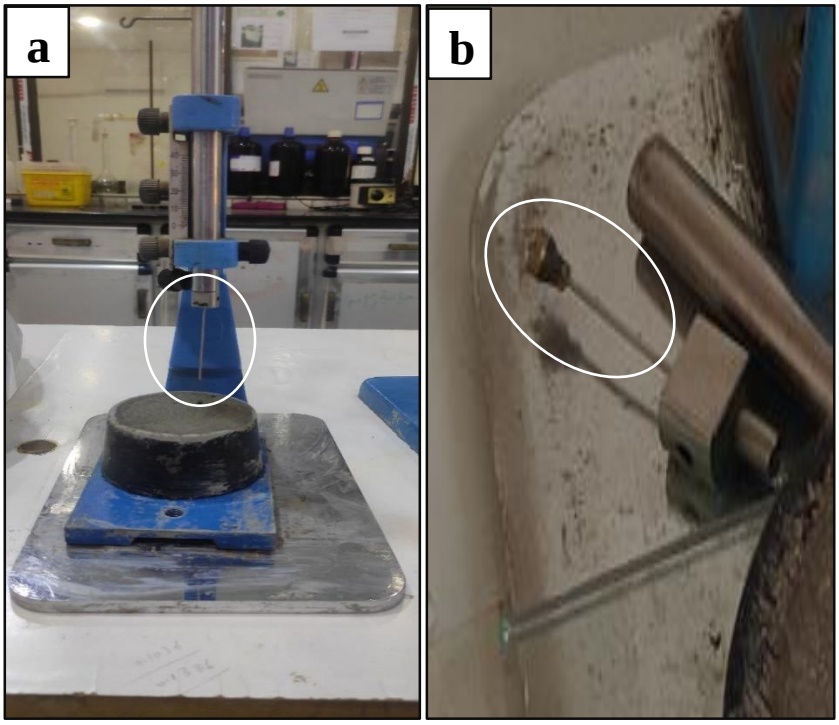

Fig. 5. Setting time test, a) initial, b) final.

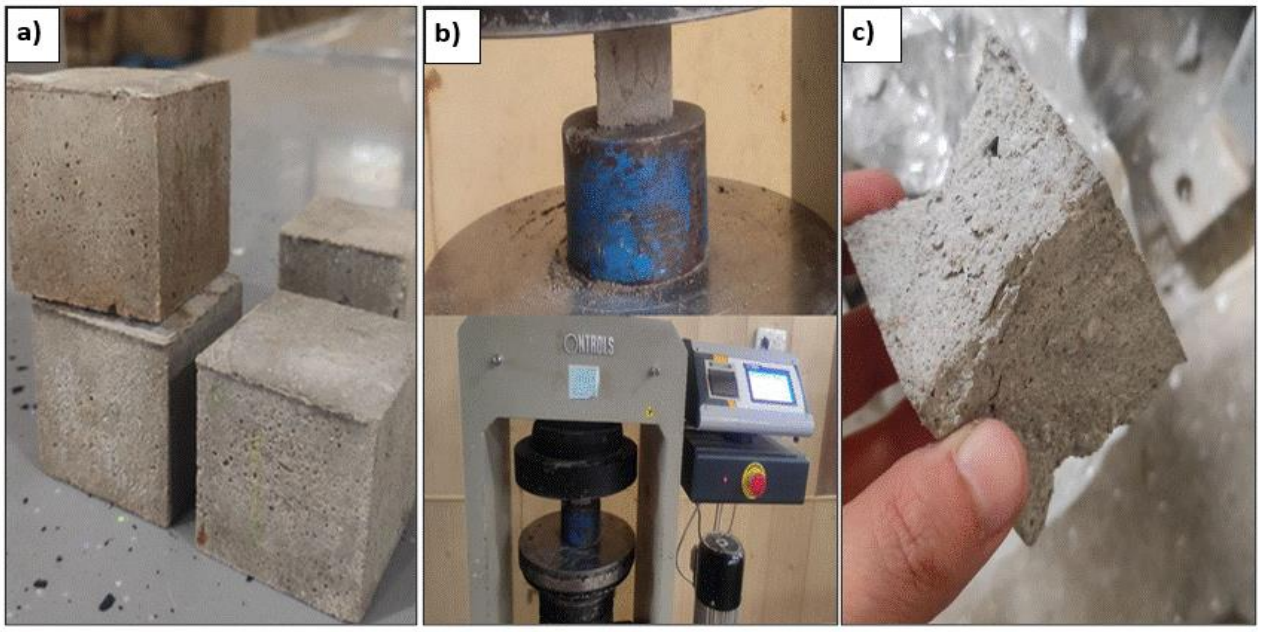

Fig. 6. Compressive Strength test, a) The test sample, b) Compressive strength test machine, c) Tested sample.

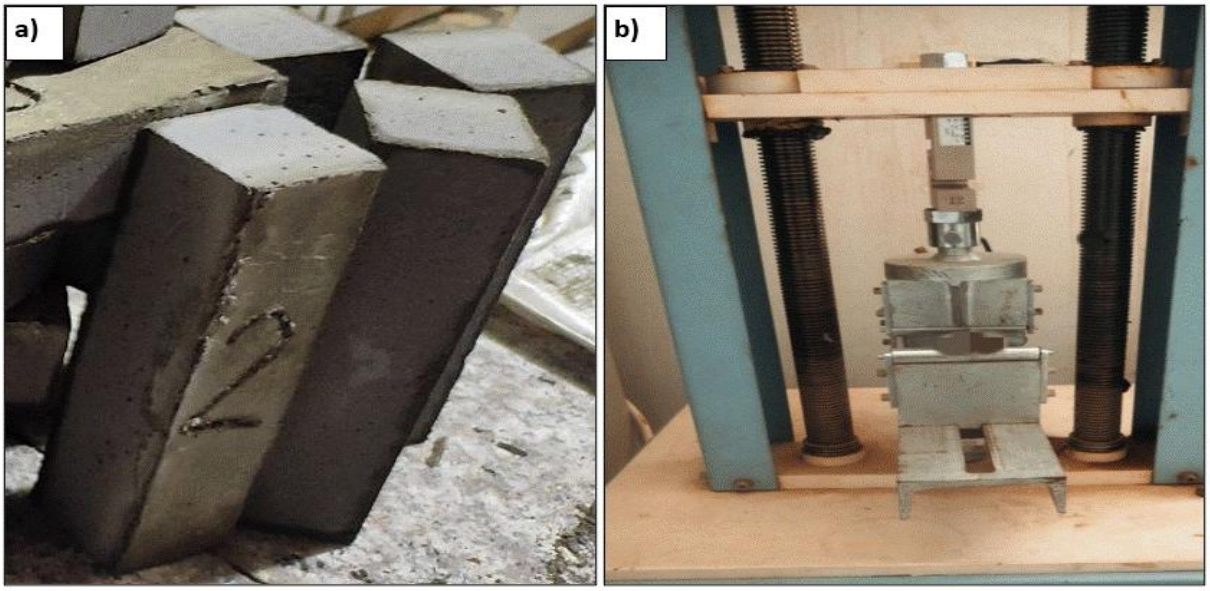

Fig. 7. Flexural strength test. a) The test sample, b) Flexural strength test machine.

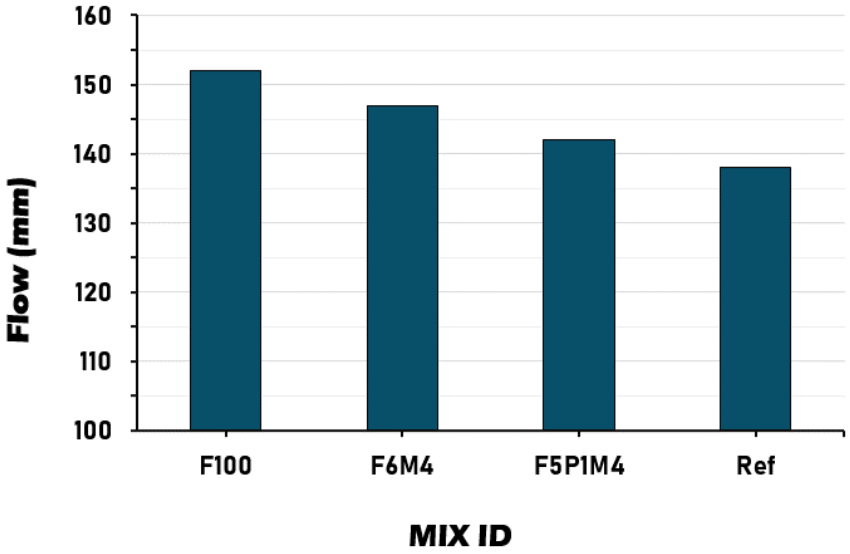

Fig. 8. Flow test results of mortar.

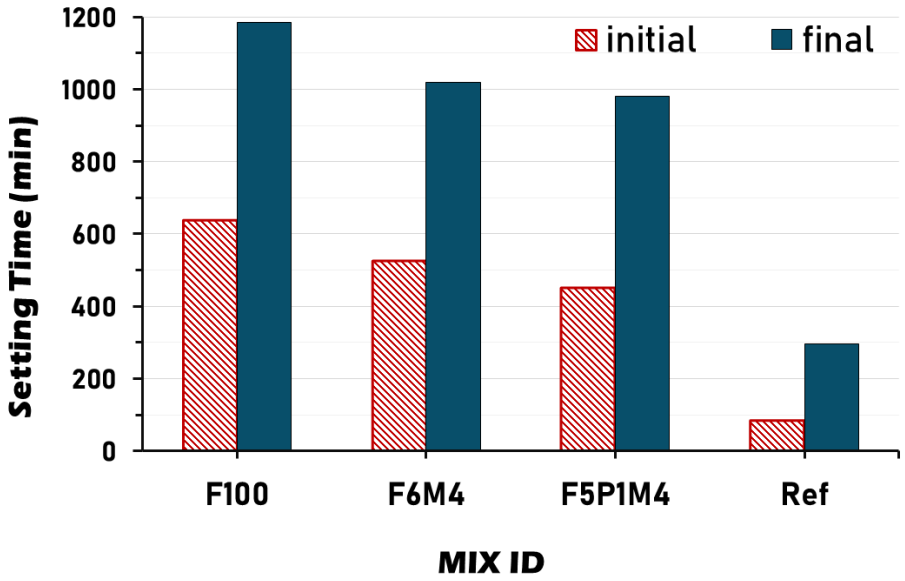

Fig. 9. Initial and final setting time results of alkali-activated mortars.

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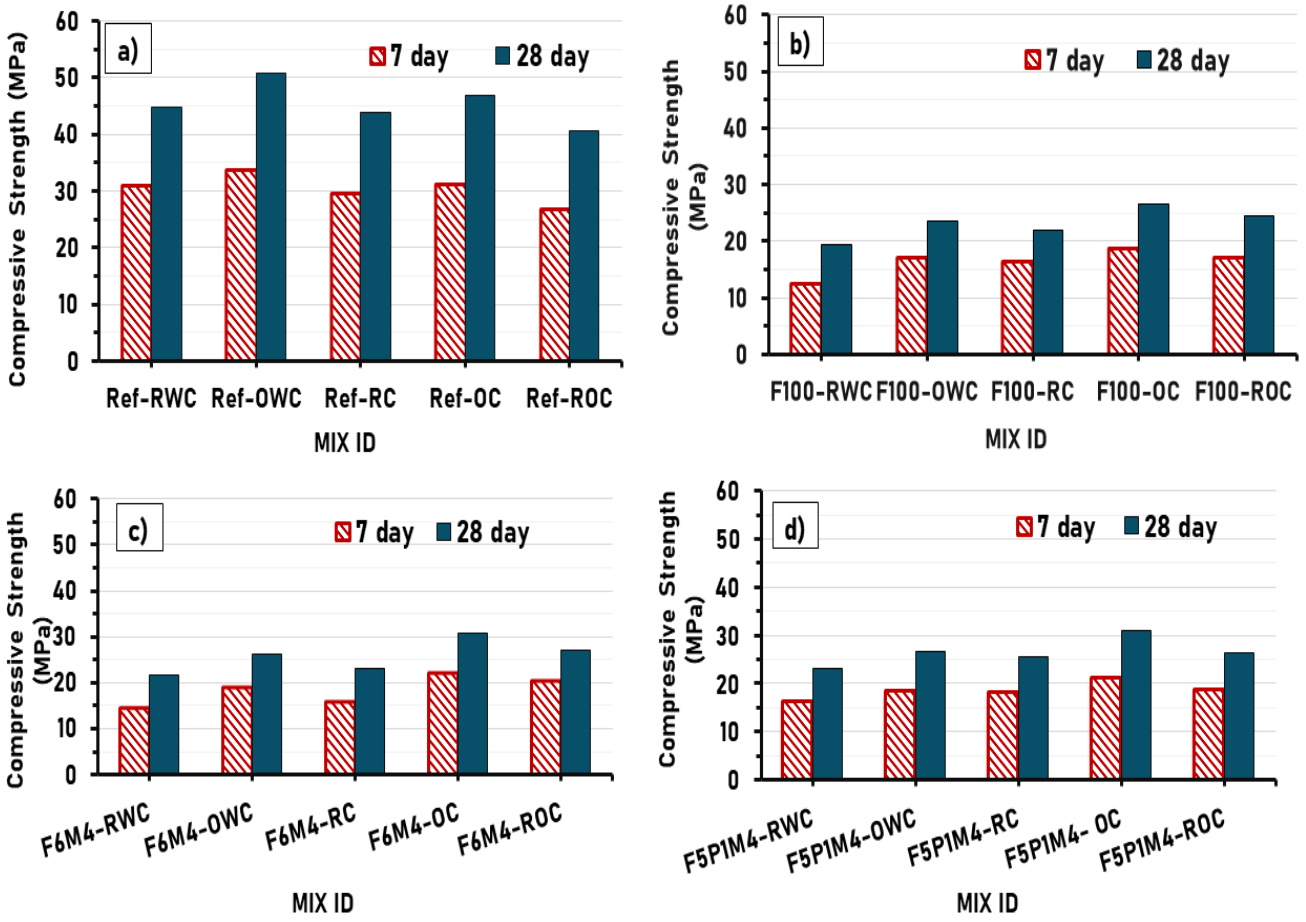

Fig. 10. 7- and 28-days compressive strength test results of Geopolymer and reference mortars in different curing regimes: a) Reference mixes, b) Mix F100, c) Mix F6M4, d) Mix F5P1M4.

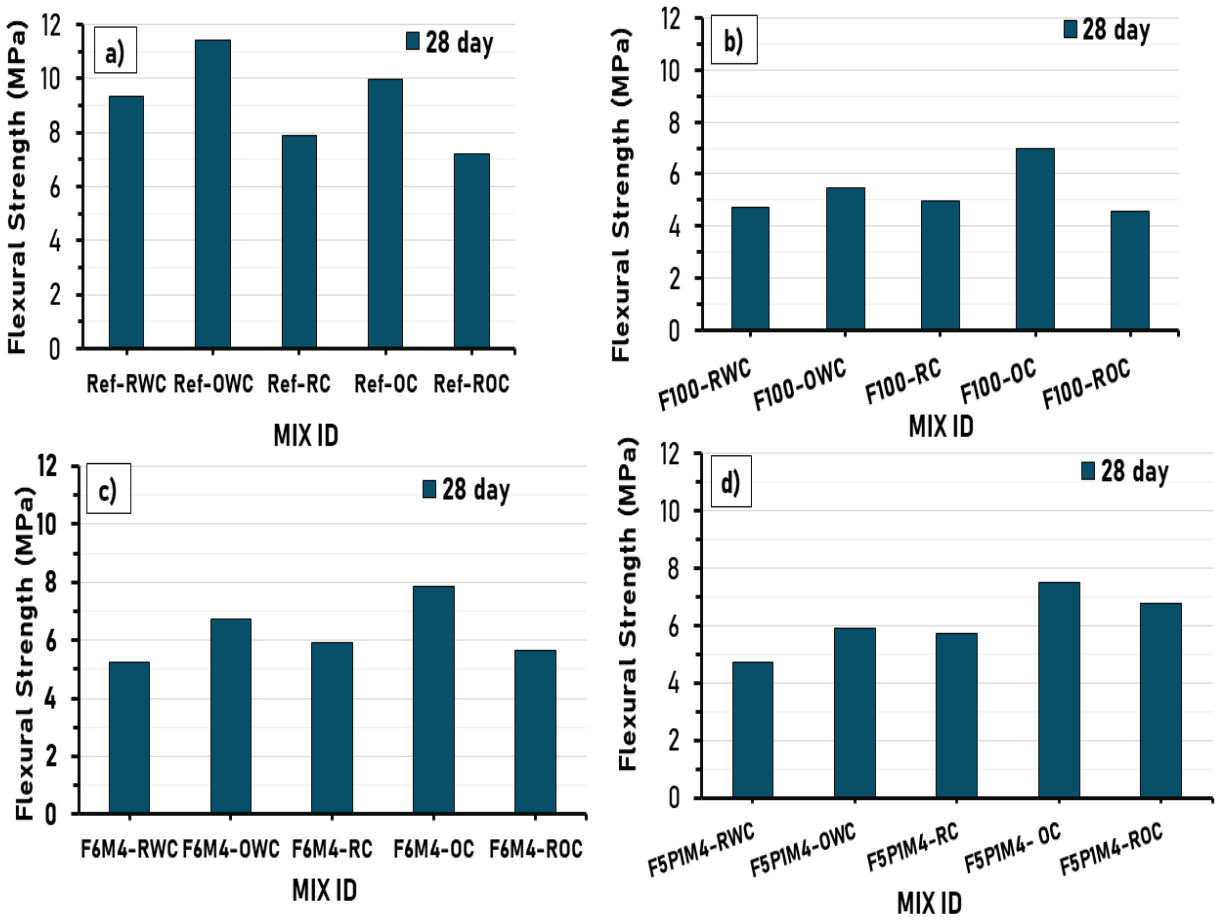

Fig. 11. Flexural strength test results of mortars at 28 days in different curing regimes: a) Reference, b) Mix F100, c) Mix F6M4, d) Mix F5P1M4.

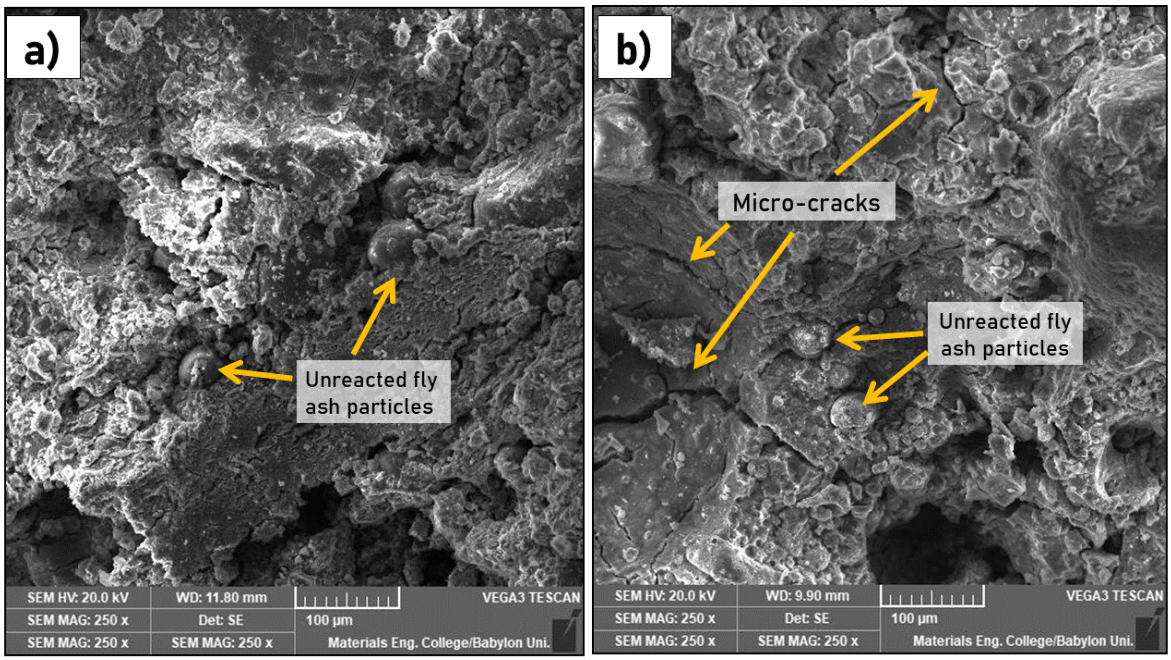

Fig. 12. SEM analysis test results of oven cured mortars at 28-day age a) Mix F6M4, b) Mix F100.

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