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Curing Condition and NaOH Concentration on the Mechanical Properties of Fly Ash Based Geopolymer Mortars

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Abstract: This paper describes the effect of different curing conditions on the mechanical properties of fly ash-based geopolymer mortars activated by different NaOH solution molarity. The influence of different curing initial curing temperatures (40℃,50℃,60℃), initial curing time (4h,8h,12h), and NaOH solution concentration (8mol, 10mol, 12mol) was studied by orthogonal design and the single-variable method. Results indicated that the fluidity of the geopolymer mortar sample followed the opposite trend with NaOH molarity increase. The most sensitive factor is NaOH molarity without considering curing age based on the data from orthogonal experiments. Furthermore, the study revealed a tendency to increase the flexural and compressive strength with rising NaOH concentration, initial curing temperature, and time, despite the mechanical strength development speed at different ages being various, and the optimum NaOH molarity may be 10mol while NaOH pellets dosage and CO_2 emission were considered.

Keywords: Geopolymer mortars, fly ash, curing condition, mechanical properties

1. Introduction

With an annual use of 1 m^3 per person, concrete is the most frequently utilized building material on the planet (Gartner, 2004). The bonding part that affords conventional concrete considerable strength consists of Portland cement. (Provis et al., 2014). Nevertheless, cement manufacture contributes conservatively 5-8% of global anthropogenic CO_2 emissions (Sousa et al., 2021). As a consequence of this predicament, the hunt for ways to achieve a compromise between increased building and environmental protection has escalated. The use of geopolymers, which are inorganic polymers, to substitute cement-based concrete attempts to limit emissions of carbon dioxide caused by cement manufacture (Davidovits, 1991). Geopolymers are inorganic materials rich in silicon-aluminum oxide that react with alkaline activators to form cementitious materials. NaOH and water glass (Na_2SO_3) were the most accepted alkaline activator considering their economy and applicability. During the preparation process of 1-ton geopolymer cement, the carbon emission is 180 kg, which is only 1/5 of that of ordinary silicate cement (Habert et al., 2011). Geopolymer also has great mechanical and durability properties.

Al-mashhadani et al. (2020) investigate the impact of various NaOH concentrations (8mol and 12 mol) on the properties of geopolymer. The compressive strength results for the 28th day indicated an increase of nearly 3%. Rekha et al. (2021) investigate the mechanical properties (compressive strengths, splitting tensile strengths, and flexural strengths) at 1 to 28 days of FA-GGBS based geopolymer concrete with five types of sodium hydroxide concentration (4mol, 6mol, 8mol, 10mol, and 12mol). The result illustrates that the ultimate strength was achieved up to 57.53MPa at 28 days for 8mol geopolymer concrete, Chen et al. (2021) have a similar set of NaOH concentrations while the

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compressive strength generally increases with increasing the molarity of NaOH concentrations. Nagral et al. (2014) find that a longer curing period of one day at the curing temperature of 90 °C provides the optimum mechanical properties of geopolymer, while other research reveals various results (Bah et al., 2022; Gorhan et al., 2014; Suppiah et al., 2022; Verma et al., 2022). The comprehensive impact of various variables on the mechanical properties of geopolymer was seldom investigated in a single design of tests prepared using a single source of fly ash.

A great deal of previous research has stated that CO_2 emission values for geopolymer concrete are 80% to 25% lower than those for OPC (Duxson et al., 2007; McLellan et al., 2011; Shi et al., 2021; Teh et al., 2017; van Deventer et al., 2010). Nevertheless, the energy expended during the manufacturing of the alkaline activators and elevated temperature curing of geopolymers has not been considered regularly. Therefore, the contribution to CO_2 emission from curing temperature and usage of alkaline activators should be noted in geopolymer manufacture.

This study aimed to examine the mechanical properties of fly ash based geopolymer mortars prepared in different curing conditions concerning temperatures from 40 to 60°C, curing time from 4 to 12 hours, and NaOH solution concentration from 8 to 12mol. We also discussed the correlation between strength properties' increase rate and CO_2 emission.

2. Experimental Program

2.1 Experimental Materials

Fly ash (collected from Datang Thermal Power Plant, Gongyi (China)), which is mainly composed of SiO_2 (56.004 wt%) and Al_2O_3 (30.267 wt%), and the percentage of CaO is less than 10% (2.36 wt%) (Table 1). This allows us to classify the fly ash as ASTM class F.

Table 1 - Chemical composition of materials

	SiO_2	Al_2O_3	Fe_2O_3	K_2O	MgO	CaO
Fly ash	56.004	30.267	4.361	1.71	0.237	2.36
Standard sand	91.734	3.178	0.238	0.819	0.198	0.243

Fig. 1c depicts the microstructure of FA. The FA particles are spherical with smooth surfaces. Fig.1d presents the XRD analysis of fly ash. The particle size distribution of fly ash, conducted by laser particle size analyzer (Malvern Mastersizer 2000), is given in Fig 1b. The medium diameter (D50) of FA was 17.254μm.

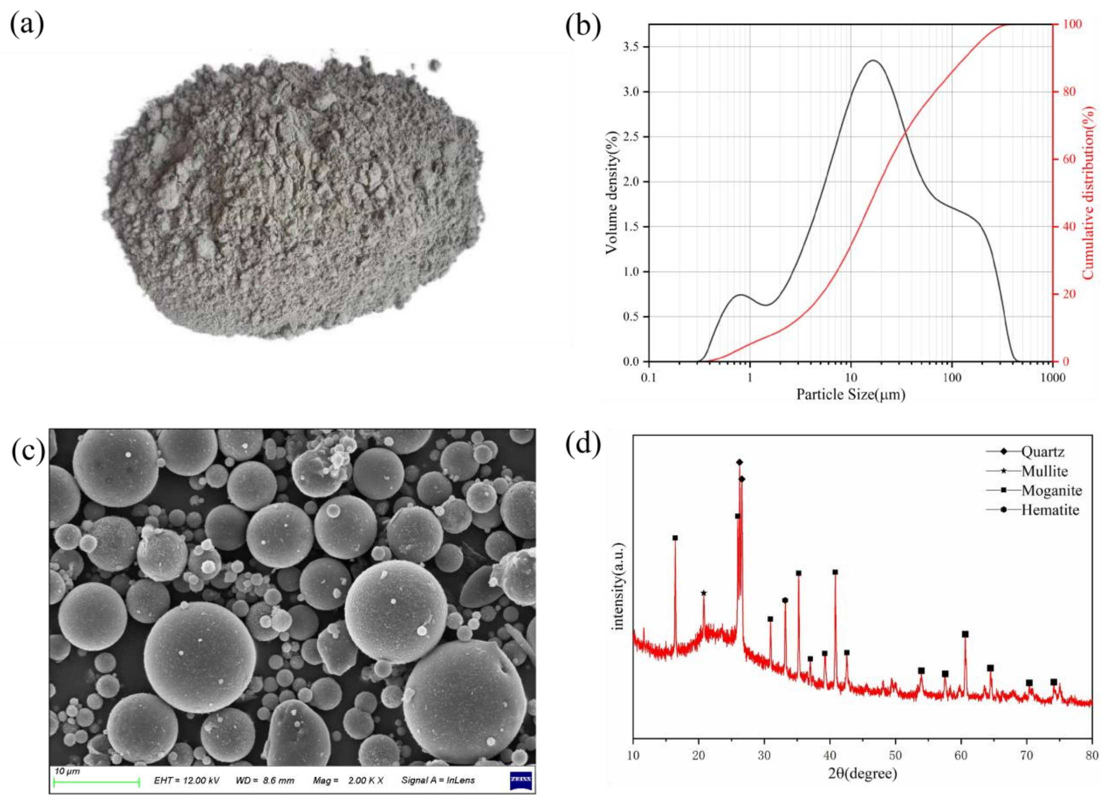

Fig. 1 - (a) Image of fly ash; (b) Particle size distribution of fly ash; (c) SEM image of fly ash; (d) XRD patterns of fly ash

NaOH pellets (purity>96%) and water glass (sodium silicate solution, Na_2O, ~10.2%, SiO_2, ~26.1%) were sourced from a Chinese chemical company from Ningxia and Sichuan. Three different concentrations of NaOH: 8mol, 10mol, and 12mol were arranged, where 'mol' stands for the molarity of the solution concentration. The alkaline activator was prepared by a blend of NaOH solutions and water glass in the mass ratio of 1:2 one day before mixing. The standard sand was purchased from Xiamen ISO Standard Sand Co., Ltd.

2.2 Experimental Procedure

Design mixes of geopolymer mortars based on orthogonal design and the single-variable method are given in Table 2, and the used sodium hydroxide concentration was 8mol, 10mol, and 12mol.

Table 2 - Design mixes of geopolymer mortars

Fly ash	sodium hydroxide solution	sodium silicate solution	standard sand
450g	78.3g(8mol)	156.7g	1350g
450g	78.3g(10mol)	156.7g	1350g
450g	78.3g(12mol)	156.7g	1350g

The specimens were prepared based on the Chinese Transportation Trade Standard JTG 3420-2020. The alkali-activator and fly ash was placed in the cement mortar mixer for 30-second slow stirring, with sand addition, 30s slow stirring,30s rapid stirring,90s break, and then 60s rapid stirring. Subsequently, the mixture was poured into the rectangular lubricated steel molds with 40×40×160 mm in two layers, and each one was pounded 60 times. Afterward, the samples were cured in an oven with different temperatures and initial curing times, then demolded by the next day, followed by further curing at room temperature for 3,7,28 days. The central apparatus operating in the experimentation consists of the mixer for mixing mortars (JC/T-681), jolting table for compacting mortars specimen (JC/T-682), NLD-3 apparatus for the fluidity of cement mortar, and YAW-300 pressure testing machine produced by Jinan Zhongluchang Instrument Equipment Co., Ltd. The sample preparation and the mix proportions of geopolymer mortars are shown in Fig 2 and Table 2.

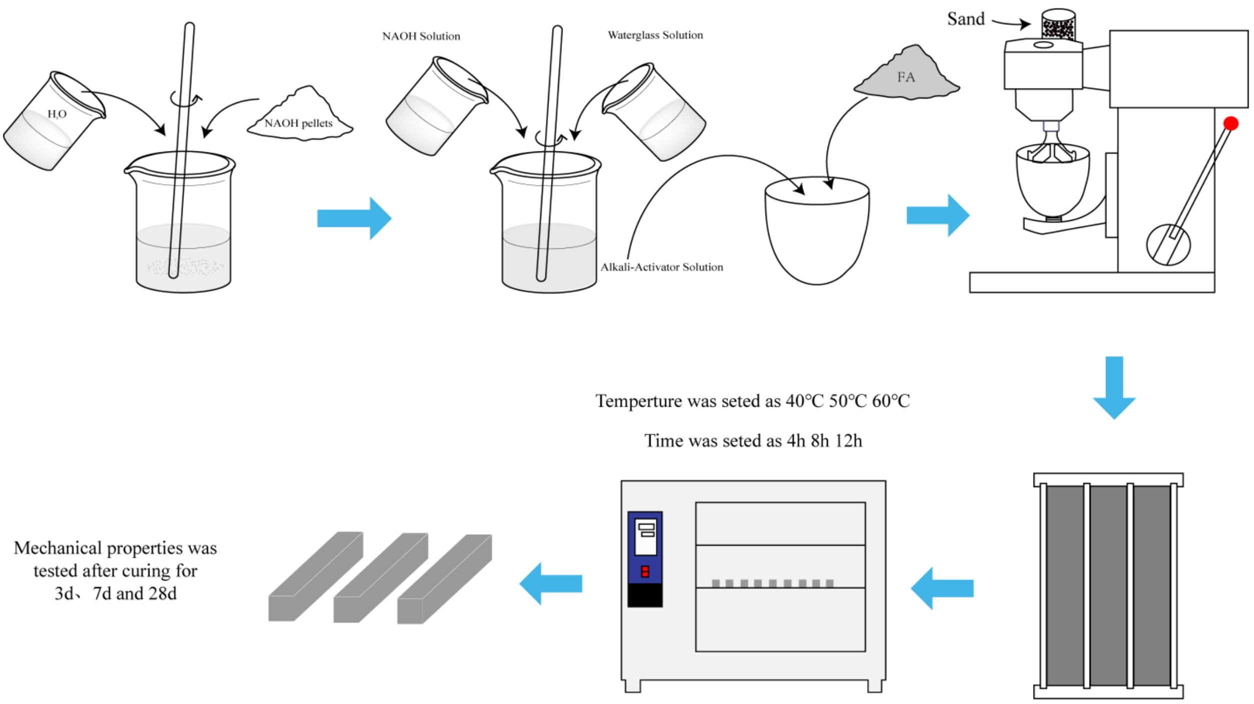

Fig. 2 - Experimental procedure

2.3 Design of Orthogonal Experiments

The best mechanical properties of geopolymer mortars were determined by the orthogonal test of four factors (the molarity of the NaOH solution, initial curing temperature, initial curing time, and curing age) and three levels. The test level and factors are illustrated in Table 3.

Table 3 - Factors and level table of L9 (3^4) orthogonal test

Level	Level 1	Level 2	Level 3
NaOH molarity	8mol	10mol	12mol
Initial curing temperature	40℃	50℃	60℃
initial curing time	4h	8h	12h
curing age	3d	7d	28d

3. Results and Discussion

3.1 Fluidity

In order to ensure that the dosage of the Alkali-activator does not change with the difference in NaOH solution concentration, the fluidity was compliant. After several attempts, the dosage of the Alkali-activator was set as 235g. However, the fluidity of geopolymer mortars decreased with increasing NaOH solution concentrations which can be observed in Fig 3. This is due to the decrease in water phase content.

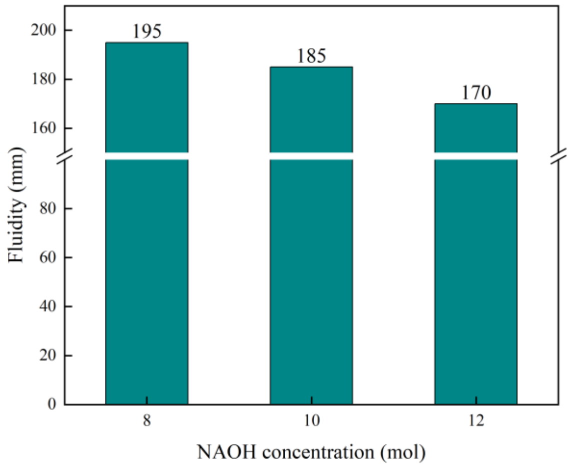

Fig. 3 - Effect of NaOH concentration on fluidity of geopolymer mortars

3.2 Result of Orthogonal Experiments

As shown in Table 4, the best curing condition is plan 8. According to the development law of strength, It can be presumed that the best curing condition is NaOH:12mol, initial curing temperature:60℃, initial curing time:12h, and curing age is 28d. To measure the sensitivity of each factor to the intensity index, The size of the extreme difference of the experimental index under each factor can be calculated in orthogonal trials. It can be concluded from the results, which are shown in Table 4, that the range of curing age is the largest, which is the main factor. The extreme difference in NaOH molarity is relatively tiny, a secondary factor.

Table 4 - The result of orthogonal experiments

Factor	NaOH molarity	Initial curing temperature	Initial curing time	Curing age	Flexural strength	Compressive strength
Plan1	8mol	40℃	4h	3d	3Mpa	7.6Mpa
Plan2	8mol	50℃	8h	7d	5.7Mpa	16.3Mpa
Plan3	8mol	60℃	12h	28d	7.8Mpa	31.6Mpa
Plan4	10mol	40℃	8h	28d	8.3Mpa	32.5Mpa
Plan5	10mol	50℃	12h	3d	5.4Mpa	19.8Mpa
Plan6	10mol	60℃	4h	7d	5.8Mpa	19.1Mpa
Plan7	12mol	40℃	12h	7d	6.2Mpa	22.8Mpa
Plan8	12mol	50℃	4h	28d	9.4Mpa	36Mpa
Plan9	12mol	60℃	8h	3d	5.7Mpa	26.8Mpa
Range (flexural strength)	1.6	1	0.5	3.8	/	/
Range (compressive strength)	10.033	4.866	4.3	15.3	/	/

3.3 Effect of NaOH Concentration

Testing the mechanical properties of fly ash-based geopolymer mortars with NaOH concentration as a single variable. The initial curing temperature was 50℃, and the initial curing time was 12h. The results are shown in Fig 4.

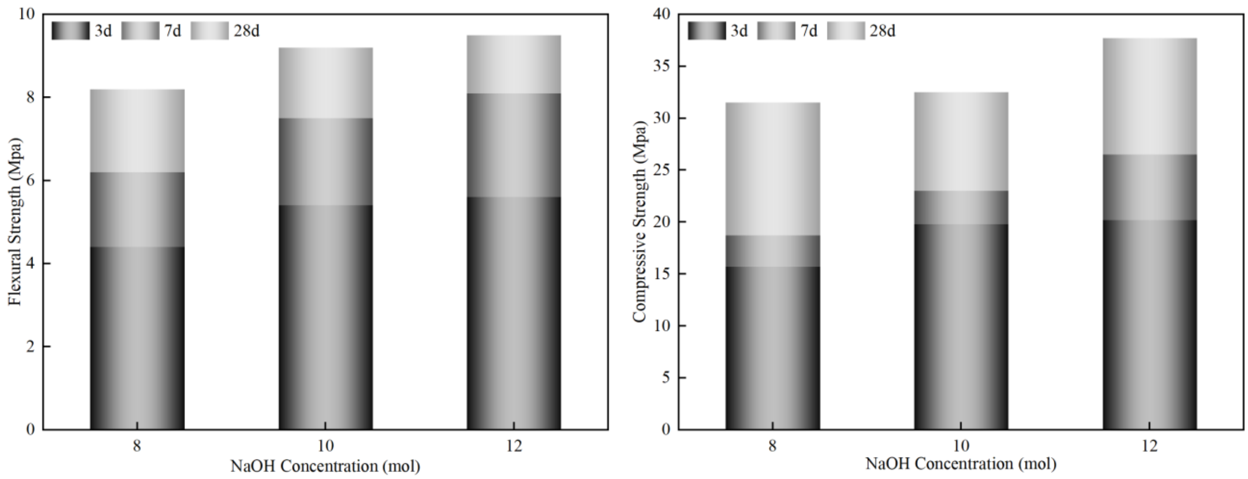

Fig. 4 - Mechanical properties of geopolymer mortars with NaOH molarity increase

Flexural and compressive strength results were obtained at 3d,7d, and 28 days. What can be seen apparently in Fig 4 is the growth of flexural and compressive strength with the increase of NaOH concentration, sample 12mol has the best 3d's mechanical properties, but the sample 8mol has the most considerable percentage of strength development, which 28d's strength, almost the double of 3d's, furthermore, the flexural strength results of 10mol were very close to the ones of 12mol samples. Hence, we can infer that higher NaOH concentration does promote geopolymerization resulting in a higher flexural and compressive strength. In addition, the dosage of NaOH pellets increases by 19.6% and 41.8% from 8mol to 10mol and 12mol, respectively. The contribution of CO_2 emissions from raw materials to the preparation of 1 group of geopolymer mortars is about 0.305kg to 0.321kg (Turner et al., 2013).

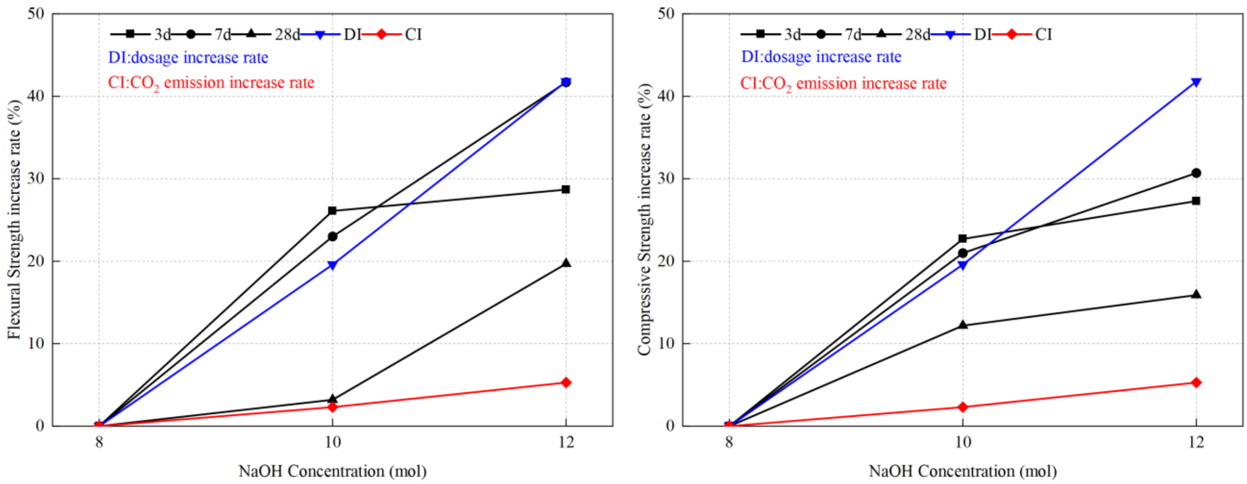

Fig. 5 - Strength increase rate compare with dosage and CO_2 emission increase rate at different NaOH molarity

Fig 5 compared the increase rate of 3d, 7d, and 28d's strength, NaOH pellets dosage, and CO_2 emission from 8mol to 10mol and 12mol, respectively. It was observed that increases in NaOH molarity from 8mol to 10mol led to a greater early strength increase rate than the dosage of NaOH pellets. However, this relationship was reversed with an increase in NaOH molarity from 10mol to 12mol. For the 28d's strength, it appears to be more cost-effective when NaOH molarity is 8 mol for the reason that the NaOH pellets increase did not provide an equivalent strength increase. On the other hand, CO_2 emission did not appear to increase significantly with the NaOH pellets, and strength increased.

3.4 Effect of Curing Condition

The mechanical properties test was carried out with initial curing temperature and initial curing time as a single variable. The initial curing time and temperature were set as 12h and 60℃, respectively, NaOH solution was 10mol. The flexural and compressive strength development results of geopolymers at different ages with curing condition changes have been plotted in Fig 6.

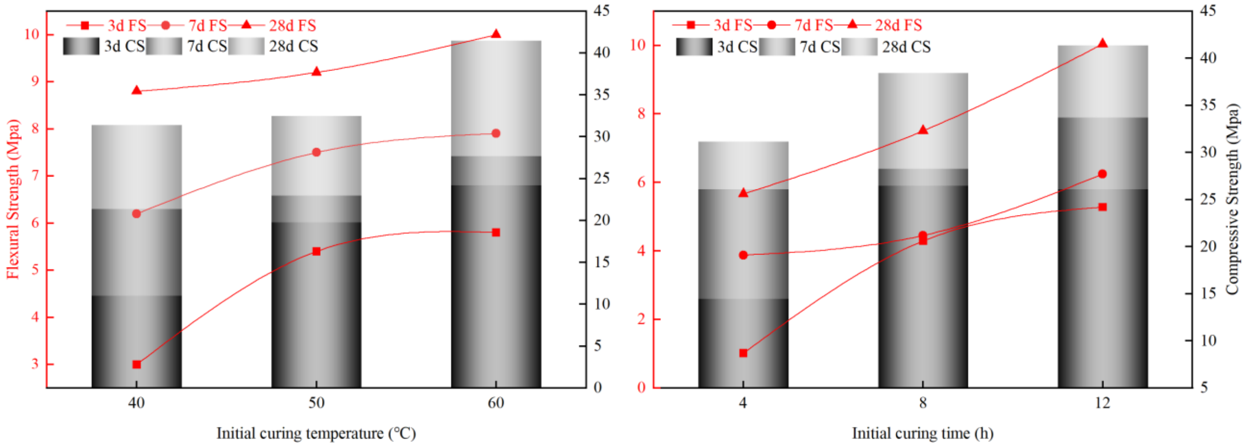

Fig. 6 - Mechanical properties of geopolymer mortars with initial curing temperature and time

Increases in curing time led to compressive and flexural strength development in the geopolymer mortars at different ages. Fig 4a revealed that the flexural strength development of all mixtures increased rapidly with initial curing temperature from 40℃ to 50℃, followed by a further increase in flexural strength at slower rates with initial curing temperature from 50℃ to 60℃. Fig 4b shows that there has been a steady increase in the mechanical properties of samples 4h and 12h relatively while the late strength of sample 8h increased rapidly.

3.5 Microstructural Analysis

After mechanical testing, the geopolymer mortar samples of 10mol NaOH (initial curing time: 12h, curing age: 7 days) were prepared for microstructural analysis. The fracture surface of samples at the magnification of 10k is shown in Fig 7.

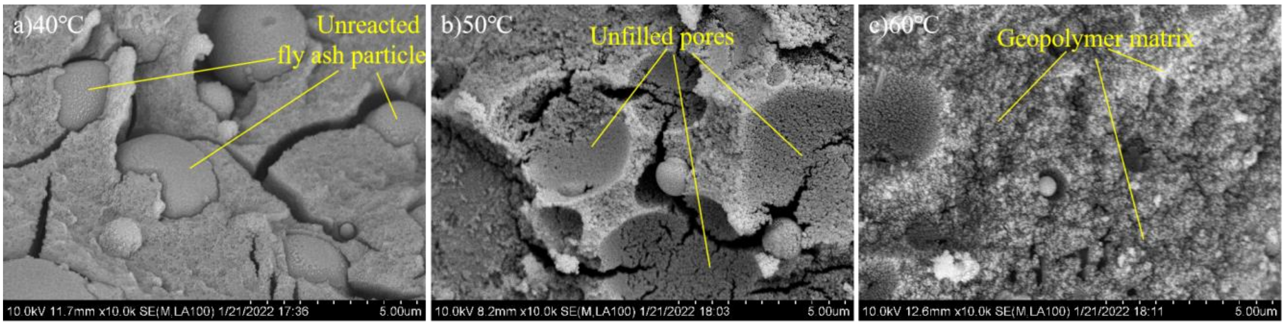

Fig. 7 - SEM micrographs of geopolymer mortars

It is obvious that the development of binding gels has been considerably impacted by the various initial curing temperature. Some unreacted fly ash particles were observed in the micrographs. However, partially dissolved fly ash particles were found in Fig. 7b-c. As the temperature increases, the geopolymerization process develops, leading the unreacted fly ash to dissolve and leaving behind unreacted inner voids in the geopolymer. This may be owing to the fact that there is inadequate heat to break the chemical bonds of Si-O and Al-O bonds in fly ash (Chindaprasirt et al., 2017). As shown in Fig.7c, during thermal hardening at 60℃, the bulk of the fly ash particles had dissolved in the alkaline activator with little unreacted or partially reacted fly ash particles remaining. Compressive strength and flexural strength values follow a similar trend.

4. Conclusion

The current research investigated the effects of NaOH concentration and curing conditions on fly ash-based geopolymer mortars' strength and microstructure. The following conclusions can be inferred based on the present study:

1. The increase in NaOH molarity did not lead to an increase in the fluidity of geopolymer mortars. On the contrary, it followed the opposite trend.

2. Based on the obtained data from orthogonal experiments, the sensitivity factor of geopolymer mortar mechanical strength is in the order: curing age > NaOH molarity > initial curing temperature > initial curing time. Therefore, the NaOH molarity and initial curing temperature should be attention in the preparation of geopolymer mortars.

3. The higher concentration of NaOH showed significantly better mechanical strength results. The optimum NaOH molarity may be 10mol while NaOH pellets dosage and CO_2 emission were taken into account. The longer initial curing time and higher initial curing temperature showed better 28d ages mechanical strength despite the mechanical strength development speed at 3d, and 7d ages being different.

4. The micrographs of geopolymer mortars showed that the increases in mechanical properties followed a similar trend of the geopolymerization process with the increase in temperature.

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References

Al-mashhadani, M. M. & Canpolat, O. (2020). Effect of Various Naoh Molarities and Various Filling Materials on the Behavior of Fly Ash Based Geopolymer Composites. *Construction and Building Materials,* 262, 1-9.

Bah, A., Feng, D. L., Kedjanyi, E. A. G., Shen, Z. Y., Bah, A. & Li, F. H. (2022). Solidification of (Pb-Zn) Mine Tailings by Fly Ash-Based Geopolymer I: Influence of Alkali Reagents Ratio and Curing Condition on Compressive Strength. *Journal of Material Cycles and Waste Management,* 24, 351-363.

Chen, K. L., Lin, W. T. & Liu, W. D. (2021). Effect of Naoh Concentration on Properties and Microstructure of a Novel Reactive Ultra-Fine Fly Ash Geopolymer. *Advanced Powder Technology,* 32, 2929-2939.

Chindaprasirt, P. & Rattanasak, U. (2017). Characterization of the High-Calcium Fly Ash Geopolymer Mortar with Hot-Weather Curing Systems for Sustainable Application. *Advanced Powder Technology,* 28, 2317-2324.

Davidovits, J. (1991). Geopolymers. *Journal of thermal analysis,* 37, 1633-1656.

Duxson, P., Provis, J. L., Lukey, G. C. & Van Deventer, J. S. J. (2007). The Role of Inorganic Polymer Technology in the Development of 'Green Concrete'. *Cement and Concrete Research,* 37, 1590-1597.

Gartner, E. (2004). Industrially Interesting Approaches to "Low-CO_2" Cements. *Cement and Concrete Research,* 34, 1489-1498.

Gorhan, G. & Kurklu, G. (2014). The Influence of the Naoh Solution on the Properties of the Fly Ash-Based Geopolymer Mortar Cured at Different Temperatures. *Composites Part B: Engineering,* 58, 371-377.

Habert, G., de Lacaillerie, J. B. D. & Roussel, N. (2011). An Environmental Evaluation of Geopolymer Based Concrete Production: Reviewing Current Research Trends. *Journal of Cleaner Production,* 19, 1229-1238.

McLellan, B. C., Williams, R. P., Lay, J., van Riessen, A. & Corder, G. D. (2011). Costs and Carbon Emissions for Geopolymer Pastes in Comparison to Ordinary Portland Cement. *Journal of Cleaner Production,* 19, 1080-1090.

Nagral, M. R., Ostwal, T. & Chitawadagi, M. V. (2014). Effect of Curing Temperature and Curing Hours on the Properties of Geo-Polymer Concrete. *International Journal of Computational Engineering Research,* 4, 1-11.

Rekha, M. S. & Sumathy, S. R. (2021). A Study on Cement-Free Geopolymer Concrete Incorporated with Industrial Waste Cured at Open Environment for Different Molarities of Sodium Hydroxide. *Global NEST Journal,* 23, 265-274.

Shi, X. S., Zhang, C., Liang, Y. C., Luo, J. Q., Wang, X. Q., Feng, Y., Li, Y. L., Wang, Q. Y. & Abomohra, A. (2021). Life Cycle Assessment and Impact Correlation Analysis of Fly Ash Geopolymer Concrete. *Materials,* 14, 7375.

Sousa, V. & Bogas, J. A. (2021). Comparison of Energy Consumption and Carbon Emissions from Clinker and Recycled Cement Production. *Journal of Cleaner Production,* 306,127277.

Suppiah, R. R., Nermoggan, P. & Shafiq, N. (2022). Experimental Investigation of the Effects of Temperature on the Morphological Characteristics of Geopolymer Binders. *Journal of Petroleum Exploration and Production Technology,* 1-8.

Teh, S. H., Wiedmann, T., Castel, A. & de Burgh, J. (2017). Hybrid Life Cycle Assessment of Greenhouse Gas Emissions from Cement, Concrete and Geopolymer Concrete in Australia. *Journal of Cleaner Production,* 152, 312-320.

Turner, L. K. & Collins, F. G. (2013). Carbon Dioxide Equivalent (CO_2-E) Emissions: A Comparison between Geopolymer and Opc Cement Concrete. *Construction and Building Materials,* 43, 125-130.

van Deventer, J. S. J., Provis, J. L., Duxson, P. & Brice, D. G. (2010). Chemical Research and Climate Change as Drivers in the Commercial Adoption of Alkali Activated Materials. *Waste and Biomass Valorization,* 1, 145-155.

Verma, M. & Dev, N. (2022). Effect of Ground Granulated Blast Furnace Slag and Fly Ash Ratio and the Curing Conditions on the Mechanical Properties of Geopolymer Concrete. *Structural Concrete,* 23,2015-2029.