Bioscene

Bioscene
Volume- 22 Number- 02
ISSN: 1539-2422 (P) 2055-1583 (O)
www.explorebioscene.com

Effects of Sodium Hydroxide Concentration, Sodium Silicate to Sodium Hydroxide Ratio and Heat Curing on fly ash-based Inorganic Polymer Mortar

Ishan Tank[1], Sanjay Kumar Sharma[1], Amit Goyal[1]

[1] National Institute of Technical Teachers Training and Research (NITTTR), Department of Civil Engineering, Chandigarh, India

Abstract: Some studies have reported using sodium silicate solution to sodium hydroxide (SS/SH) with low calcium fly ash to develop inorganic polymer concrete or mortar. However, little in-depth analysis of setting time and mechanical properties has been reported.This study investigates the effects of sodium hydroxide (NaOH) concentration, sodium silicate to sodium hydroxide (SS/SH) ratio, and curing temperature on fly ash-based geopolymer mortar's setting time and compressive strength. Twenty mix designs were evaluated with NaOH concentrations ranging from 8M to 16M, SS/SH ratios from 1.5 to 3.0, and curing temperatures of 30°C, 60°C, and 90°C. The standard consistency was determined to be 28%. Experimental results indicated that increasing NaOH concentration from 8M to 14M generally enhanced compressive strength, attaining 38.85 N/mm^2 at 28 days with an SS/SH ratio of 2.0 cured at 90°C. However, concentrations above 14M resulted in reduced strength. The SS/SH ratio improved strength up to an optimal point of 2.0, beyond which a decline was observed. Higher curing temperatures significantly accelerated setting times, with initial setting times recorded at 317 minutes for a 14M NaOH solution at an SS/SH ratio of 1.5 at 30°C, compared to rapid polymerization at 90°C. These findings contribute to understanding geopolymerization processes, increasing the use of recycled materials in construction projects, and encouraging the adoption of low-carbon cement alternatives. **Significance Statement:** This study investigates the effects of the ratio of sodium silicate solution to sodium hydroxide (SS/SH), sodium hydroxide (NaOH) solution concentration, and curing temperature on fly ash-based inorganic polymer mortar'ssetting time and compressive strength.

Keywords: fly ash, sodium silicate to sodium hydroxide ratio, sodium hydroxide concentration, inorganic polymer mortar, heat curing, compressive strength, setting time, eco-friendly materials.

Introduction

The cement industry is a significant source of greenhouse gas emissions, contributing approximately 1.35 billion tons of carbon dioxide annually (Kumar et al., 2005; Patankar et al., 2014; Yousuf et al., 2020). This is mainly due to the escalating demand for Portland cement, which has surpassed one billion tons per year as the construction industry expands. Concurrently, fly ash, a byproduct from coal combustion in thermal power plants, poses environmental challenges related to its disposal, affecting land use and public health. Its management is crucial, as it has an estimated global production of about 780 million tons of fly ash annually (Pandey et al., 2011). Fly ash's lightweight and airborne nature can lead to serious health issues, including respiratory ailments.

Recent advancements in material science have introduced geopolymer technology, wherein fly ash is fully activated using alkaline solutions. This state-of-the-art approach eliminates the need for conventional cement, thus addressing both fly ash disposal issues and the environmental impact of cement production. The study by Davidovits (2005) pinpoints the necessity for low-CO_2-emission cementitious materials and highlights the role of geopolymer concrete as a viable alternative for sustainable construction practices. The geopolymerization process involves (i) dissolution of aluminosilicate materials in a concentrated alkaline solution, (ii) formation of silica-alumina oligomers, (iii) polycondensation to create inorganic polymers, and (iv) bonding of undissolved particles into the final geopolymer structure (Yuan et al., 2023). Curing occurs at 60–100°C for 20–36 hours to enhance the reaction (Duxson et al., 2006).

Numerous research studies have been carried out on the properties and applications of fly ash in concrete. Suri (2012) highlighted the potential of fly ash for developing innovative building materials, mainly through its complete utilization in geopolymer concrete formulations. Rangan et al. (2005) demonstrated that fly ash-based geopolymer concrete offers excellent durability and low creep. Furthermore, Yellaiah (2016) analytically investigated the influence of factors such as water content and the ratios of alkaline activators, offering valuable insights into optimizing the performance of geopolymeric materials.The literature indicates a growing recognition of fly ash as a sustainable construction material. Geopolymer technology signifies a promising solution to traditional cement, helping to reduce the environmental footprint of construction while enhancing the performance and durability of concrete. It also provides significant benefits by recycling waste materials as binders.This study aims to develop an understanding by investigating the influence of NaOH concentrations, AAS ratio, and heat curing on the properties of fly ash-based inorganic polymer mortar, further contributing to the development of sustainablebuilding materials. Moreover, this approach reduces the environmental impact of construction materials and promotes industrial waste recycling, contributing to a circular economy.

Experimental Program

2.1. Materials

This study used low-calcium processed fly ash as the binder procured from Ultratech RMC Plant, Mohali, Punjab, India.Standard sand was used as the fine aggregatesize fractions were combined in equal proportions to meet the grading specifications outlined in IS 650:1991. Laboratory-grade SH in flake form (98% purity) and SS solution (50.72% solids) were utilized for the alkaline activator solution. The concentration of SH solutions varied from molarities of 8 M, 10 M, 12 M, 14 M, and 16 M, and Sodium silicate solution-to-sodium hydroxide ratios of 1.5, 2.0, 2.5, and 3.0 were considered. Curing was conducted in an oven at temperatures of 30°C, 60°C, and 90°C for 24 hours, and ambient cured afterward till the days of testing.

2.2. Specimen Preparation and Casting

Inorganic polymer mortar mixes were formulated by entirely substituting traditional cement with an equivalent mass of fly ash, subsequently activated using a combination of sodium hydroxide and sodium silicate solutions. SH solution was chosen for this research due to its widespread availability and lower cost than KOH(potassium hydroxide). The AAS (alkaline activator solution), comprising a blend of SH and SS solutions in varying concentrations, was prepared in the laboratory. The SH was mixed with the SS solution before mortar preparation to ensure its reactivity. SS aims to facilitate the development of geopolymer precursors and enhance the polymerization process (Xu et al., 2000).

All materials were mixed in a pan mixer and dried for 5 minutes. After this initial mixing, the activator solution (i.e., AAS) was added within the stated concentration range of 8 to 16M, and the mixture was blended for another 10 minutes. The geopolymer paste was then cast into cube molds measuring 70.6 mm × 70.6 mm × 70.6 mm immediately following mixing. The casting was conducted in two layers, adhering to the IS 4031 (Part VI)-1981 standard, with each layer tamped 25 times using a rod. All prepared specimenswere vibrated for 2 minutes to eliminate air voids and rapidly moved to a curing oven for respective curing regimes, i.e., 30°C, 60°C, and 90°C, for 24 hours after casting. After curing, the samples were demolded and placed in the room for ambient curing undisturbed until the testing day. The reported strengths represent the average outcomes of three tests at 3, 7, and 28 days using a Compression Testing Machine. Details of the mix proportions can be found in Table 4.2, while the specimens cast for various mix compositions are depicted in Photographs.

Methodology and Testing Procedures:

An experimental investigation is designed to quantify the fly ash-based inorganic polymer mixtures' setting response and compressive strength. The primary variables considered in this study include:

- SH- concentration
- SS/SH ratio - Sodium Silicate Solution-to-Sodium Hydroxide Solution
- Curing regime (30 °C, 60 °C and 90 °C)

Fly Ash Geopolymer Paste:

Standard Consistency, Setting Time, and Compressive Strength

The fly ash-based geopolymer paste's standard consistency and setting times are determined following IS: 4031 (Part-4, 5& 6)-1988, which outlines the procedure for assessing the consistency,setting time, and compressive strength of Portland cement paste, respectively.

3 Results and Discussion:

The standard consistency of the fly ash-based geopolymer paste was determined to be 28% after various trials with varying percentages of alkaline activator solution. After that, a total of twenty mixtures were prepared, each with different concentrations of NaOH solution and varying ratios of AAS (Na_2SiO_3 to NaOH by weight) to examine their effect on the setting times and compressive strength at curing regimes of 30°C, 60°C, and 90°C. The experimental results for setting time and compressive strength at 3, 7, and 28 days (averaged from three specimens) are presented in Tables 3 and 4, and their respective graphs are plotted in Figures 1 and 2.

Table 1: Setting Times and Compressive Strength Details of Inorganic Polymer Mortar

Sample Identity	**Mix composition of geopolymer**		Setting times at various combinations of mixtures						**Compressive strength (N/mm²)**								
			Initial setting time in minutes			Final setting time in minutes			3days	3days	3days	7days	7days	7days	28days	28days	28days
	NaOH Conc.	**SS/SH ratio**	**30°C**	**60°C**	**90°C**	**30°C**	**60°C**	**90°C**	**30 °C**	**30 °C**	**30 °C**	**60°C**	**60°C**	**60°C**	**90°C**	**90°C**	**90°C**
FGPM1	8M	1.5	195	78	39	355	184	62	2.38	6.10	11.50	4.60	9.51	15.55	8.32	16.08	23.11
FGPM2	8M	2.0	188	74	42	290	134	98	4.87	6.51	11.95	5.45	13.20	16.11	11.00	15.98	25.97
FGPM3	8M	2.5	141	62	44	257	122	63	3.67	6.52	11.22	4.82	12.10	15.34	10.25	15.66	22.00
FGPM4	8M	3.0	85	62	41	215	102	62	2.41	3.22	6.07	3.27	6.28	12.32	4.85	9.17	17.50
FGPM5	10M	1.5	217	115	42	435	231	87	3.81	6.98	12.04	6.95	12.84	15.78	9.31	17.02	23.90
FGPM6	10M	2.0	205	92	41	338	185	71	8.02	8.51	13.65	8.50	13.91	17.32	11.12	18.31	27.85
FGPM7	10M	2.5	160	85	46	265	155	68	6.31	8.07	11.98	7.52	13.00	17.00	11.10	17.51	23.51
FGPM8	10M	3.0	185	98	47	315	170	72	3.61	3.87	8.21	4.34	8.25	15.63	7.12	11.91	24.38
FGPM9	12M	1.5	291	150	40	427	238	85	3.95	7.88	12.12	7.98	16.02	18.22	9.93	16.95	30.01
FGPM10	12M	2.0	258	124	42	355	190	55	9.24	9.32	13.23	10.42	16.21	19.41	12.04	18.67	30.82
FGPM11	12M	2.5	180	105	45	325	178	59	8.22	9.21	13.01	8.95	16.32	17.90	11.15	18.10	29.95
FGPM12	12M	3.0	158	68	42	297	144	62	5.10	7.44	9.21	5.42	14.02	16.71	8.32	16.84	25.21
FGPM13	14M	1.5	317	152	40	514	265	98	5.01	11.05	13.75	9.52	18.54	21.32	12.21	21.23	34.05
FGPM14	14M	2.0	305	125	41	475	280	86	12.21	12.90	17.21	12.85	19.32	26.44	15.51	25.51	38.85
FGPM15	14M	2.5	242	105	41	372	188	62	9.10	10.01	14.05	11.21	17.85	22.01	13.32	24.50	34.20
FGPM16	14M	3.0	108	60	41	254	124	62	5.98	9.52	13.21	6.85	17.21	20.42	11.52	20.52	28.98
FGPM17	16M	1.5	272	125	45	467	192	84	3.31	9.21	12.10	6.84	18.00	20.04	11.02	19.24	30.85
FGPM18	16M	2.0	215	57	38	347	141	80	8.92	10.07	14.21	10.02	19.01	23.52	11.94	20.11	34.71
FGPM19	16M	2.5	222	102	44	365	166	62	8.20	7.81	12.42	10.04	17.11	20.11	11.21	17.93	31.45
FGPM20	16M	3.0	218	65	41	325	121	57	5.21	7.75	8.41	7.10	15.45	18.52	9.21	18.02	27.32

3.1 Effect of Sodium Hydroxide Solution Concentration

a) Setting Time

The setting time of geopolymer paste is crucial for its handling from mixing to placement. As shown in Figures 1 and 2, increasing the NaOH concentration from 8M to 14M (in 2M increments) at temperatures of 30°C, 60°C, and 90°C results in an increased setting time for SS/SH ratios of 1.5, 2.0, and 2.5. However, a greater increase in NaOH concentration from 14M to 16M leads to a decrease in setting time. Notably, at an SS/SH ratio of 3.0, a rise in setting time is observed when increasing NaOH concentration from 8M to 10M, but a subsequent increase from 10M to 16M decreases the setting time. Thus, while higher NaOH concentrations initially extend the setting time, this effect diminishes at extreme concentrations. This behavior aligns with findings reported by Yellaiah (2016) but is dissimilar to results reported by Hardjitoet al (2005), where different materials influenced the outcomes.

b) Compressive Strength

Figures 3 to 4 illustrate that an increase in NaOH concentration from 8M to 14M enhances the compressive strength of the geopolymer mortar at numerous SS/SH ratios and temperatures ranging from 25.97 N/mm^2 at 8M to 38.85 N/mm^2 at 14M. On the other hand, a concentration increase from 14M to 16M reduces strength, i.e., 34.71 at 16M. The observed strength improvements from 8M to 14M can be linked to the dissolution of fly ash, which facilitates the materialization of the geopolymer network. However, the 11% strength reduction from 14M to 16M suggests potential over-activation effects or incomplete polymerization at extreme concentrations.These findings align with the research findings by Hardjitoet al. (2005) and Yellaiah (2016).

3.2 Effect of Sodium Silicate to Sodium Hydroxide Ratio (SS/SH)

a) Setting Time

Figures 1 to 3 indicate that increasing the SS/SH ratio from 1.5 to 3.0 (in 0.5 increments) leads to decreased setting times for NaOH concentrations of 8M, 12M, and 14M across curing temperatures of 30°C to 90°C.

For instance, at 8M NaOH and 30°C curing, increasing the SS/SH ratio from 1.5 to 3.0 decreased the initial setting time from 195 to 85 minutes (FGPM1 to FGPM4). Similarly, at 14M NaOH and 60°C curing, the final setting time reduced from 265 to 124 minutes when increasing the ratio from 1.5 to 3.0 (FGPM13 to FGPM16).

Furthermore, at a NaOH concentration of 10M, a decrease in setting time is noted when increasing the SS/SH ratio from 1.5 to 2.5, while a further increase to 3.0 results in longer setting times. The effects observed with the 16M NaOH solution show alternating trends in setting time as the SS/SH ratio increases. This relationship is most likely linked to Si/Al ratio variations, as Yellaiah (2016) observed.

b) Compressive Strength

Figures 1 to 3demonstrate that compressive strength increases with the SS/SH ratio, particularly from 1.5 to 2.0 across NaOH concentrations. For example, at 14M NaOH and 90°C curing, 28-day strength peaked at 38.85 N/mm^2 for SS/SH 2.0 (FGPM14), compared to 34.05 N/mm^2 at 1.5 (FGPM13) and 28.98 N/mm^2 at 3.0 (FGPM16). This pattern held consistently, with 12M NaOH showing maximum strength of 30.82 N/mm^2 at SS/SH 2.0 (FGPM10) versus 25.21 N/mm^2 at 3.0 (FGPM12),resulting in decreased strength. This trend aligns with previous research by Hardjito et al., indicating that a higher SS/SH ratio can enhance compressive strength. Interestingly, this contradicts findings from Hardjito and Tsen's study, where a higher ratio of silicate to hydroxide was associated with decreased compressive strength.

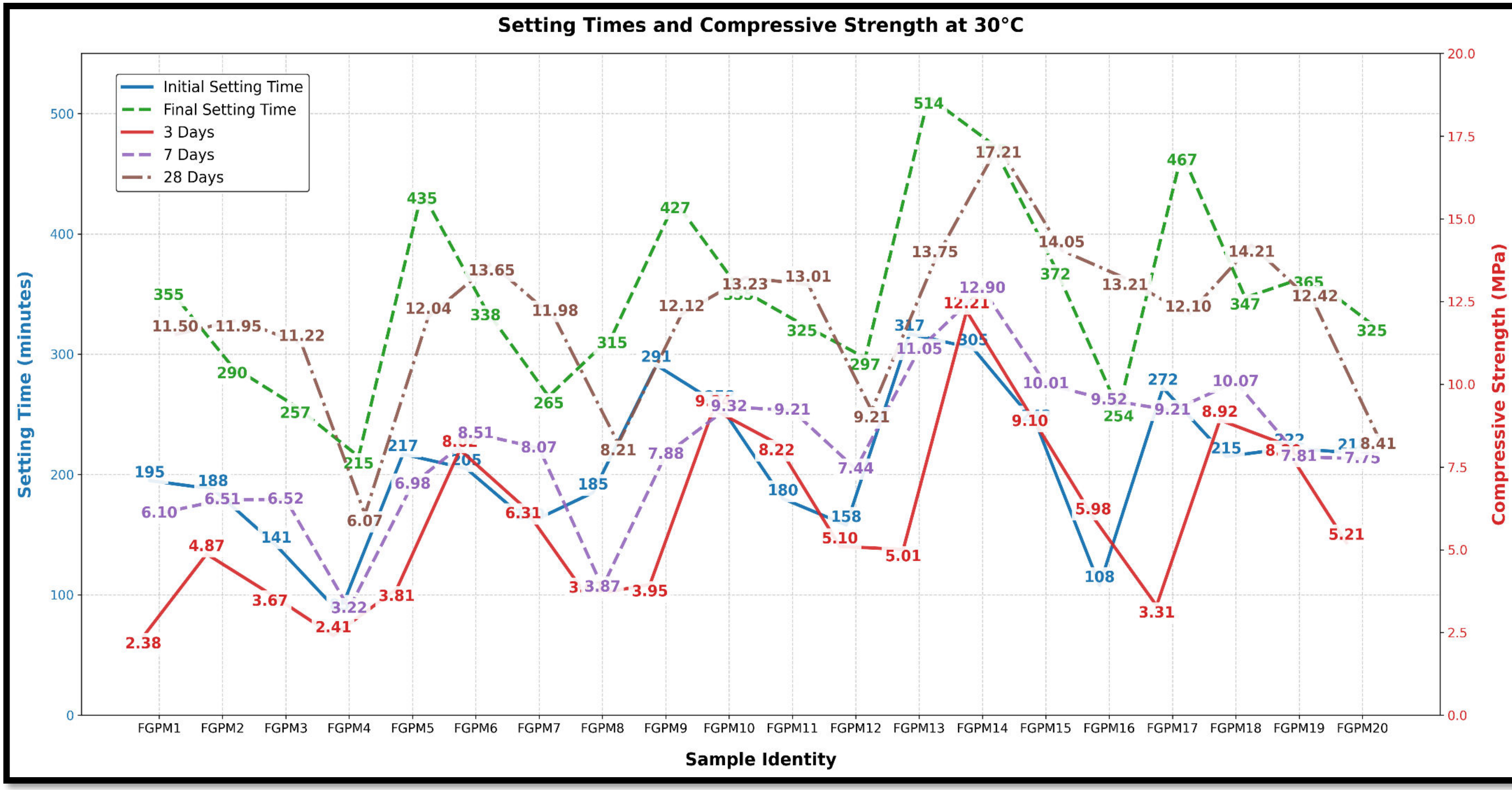

Figure 1 Setting Time & Compressive Strength Trends at 30°C

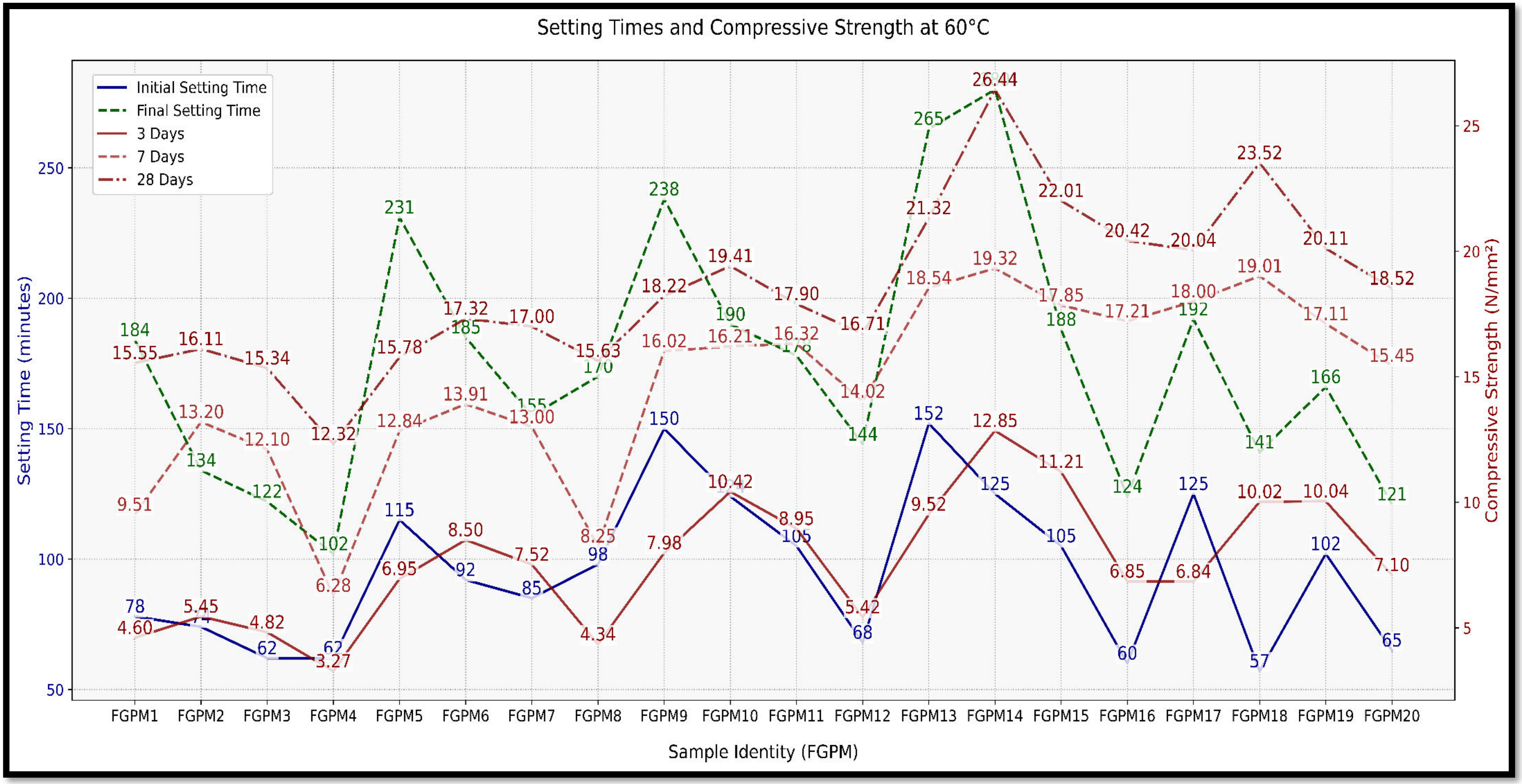

Figure 2 Setting Time & Compressive Strength Trends at 60°C

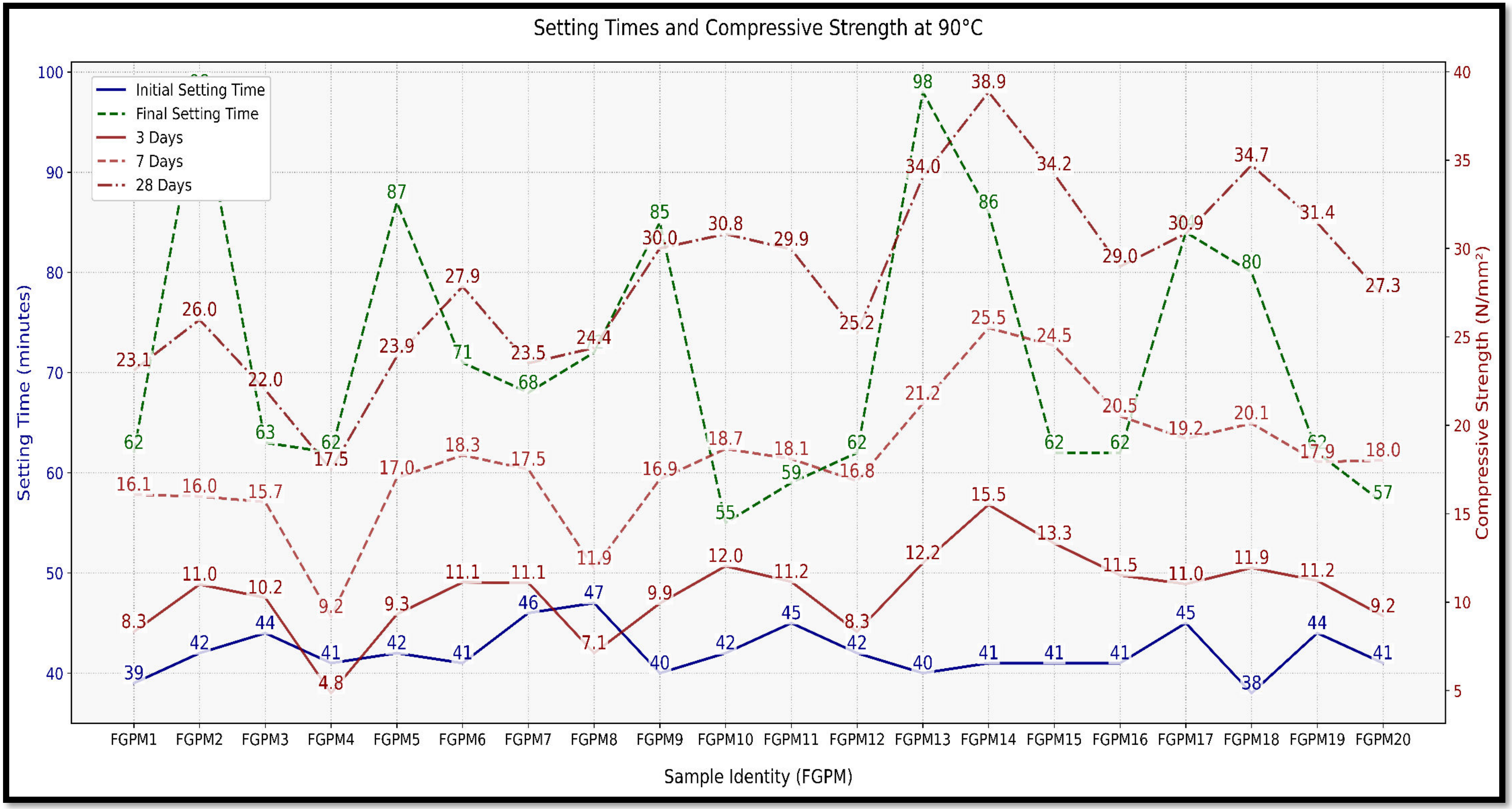

Figure 3 Setting Time & Compressive Strength Trends at 90°C

3.3 Effect of Curing Temperature

a) Setting Time

The curingregime significantly impacts the geopolymerization process. As Figures 1 to 3 illustrate, increased curing temperatures correlate with decreased setting times. Initial setting times at 30°C are notably longer, suggesting delayed polymerization, while rapid polymerization occurs at 90°C. Hardjito(2005) and Yellaiah (2016) noted similar results. The maximum recorded initial and final setting times were 317 minutes and 514 minutes, respectively, for a 14M NaOH concentration at an SS/SH ratio of 1.5 under 30°C. Overall, final setting time reduced from ~300–500 min (30°C) to ~50–100 min (90°C), confirming thermal activation.

b) Compressive Strength

Figures 1to 3demonstrate that the compressive strength of inorganic polymer mortar rises with increasing curing regime between 30°C and 90°C. Samples cured at 90°C exhibit rapid strength development compared to those cured at lower temperatures. The strength at 28 days ranges from 5.21 MPa for the 8M NaOH solution with an SS/SH ratio of 3.0 at 30°C to 39.76 MPa for the 14M solution with an SS/SH ratio of 2.0 at 90°C. These variations highlight the importance of NaOH concentration, SS/SH ratio, and curing temperature on strength development, with inadequate polymerization observed at lower strengths.

4. Conclusion

The findings of this study elucidate the critical roles that sodium hydroxide concentration, SS/SH ratio, and curing temperature play in determining the setting time and compressive strength of fly ash-based inorganic polymer mortar. The research contributes valuable insights for optimizing geopolymer formulations, ultimately advancing the development of sustainable building materials. The following conclusionscan be drawn from the research presented: Increasing the concentration of sodium hydroxide (NaOH) from 8M to 14M generally enhances the compressive strength of fly ash-based inorganic polymer mortar. Still, excessive concentrations (14M to 16M) can lead to reduced strength.A balanced increase in the SS/SH ratio improves compressive strength up to a certain point (1.5 to 2.0), beyond which strength tends to decline, indicating an optimal ratio for performance.Higher curing temperatures significantly accelerate both setting times and strength development. Mortars cured at 90°C exhibit rapid polymerization and greater compressive strength than those cured at lower temperatures.The setting time of geopolymer mortar is influenced by both NaOH concentration and SS/SH ratios. Increased NaOH concentration can initially extend setting times but may decrease them at higher concentrations.The study emphasizes the need to optimize the combination of SH concentration, SS/SH ratio, and curing temperature to achieve desirable properties in geopolymer mortar for construction applications.The findings contribute to developing more sustainable construction materials by utilizing fly ash, thereby addressing environmental concerns related to cement production.

This study offers significant insights, but it is important to acknowledge several limitations. Using low-calcium fly ash as the primary binder material may limit the applicability of findings to other types of fly ash or alternative pozzolanic materials.The study focused on a limited range of curing temperatures (30°C to 90°C). The effects of lower and higher extreme temperatures on geopolymer performance were not explored.The study examined compressive strength only at 28 days. Long-term durability and performance beyond this period remain to be investigated.The research was limited to sodium hydroxide and sodium silicate as alkaline activators. Exploring other activators could provide a broader understanding of the geopolymerization process.The study did not consider the influence of environmental conditions, such as humidity and air circulation, which could affect setting time and strength development in practical applications.

Future research on geopolymers should explore a broader range of binder materials, including fly ash from different sources and alternative pozzolanas, to validate the study's potential. In-depth durability studies, such as extreme curing temperatures, long-term durability in aggressive environments, and other conditions, such as humidity, should be conducted to understand thegeopolymer's performance. Furthermore, different alkaline activators must be studied to offer insights into optimizing the geopolymerization process withsignificant applications and environmental impacts.

Statements and Declarations

Competing Interests:

The authors declare that no competing interests related to the work submitted for publication are financial or non-financial. This research was conducted independently, and no external funding was received that could influence the outcomes presented in this manuscript. The study was conducted as part of ongoing academic efforts to explore using inorganic polymers and fly ash to enhance construction materials.

Ethical Considerations:

This study did not involve any human participants or animal subjects and, therefore, does not require ethical approval.

References:

1. Ahn, S. J., &Graczyk, D. (2012). Understanding energy challenges in India: Policies, players, and issues. International Energy Agency (IEA).
2. Davidovits, J. (1999). Geopolymer ‘99: 2nd International Conference, Saint-Quentin, France, pp. 9–39.
3. Davidovits, J. (2005). Proceedings of the World Congress Geopolymer 2005, Saint-Quentin, France, pp. 9–15.
4. Duxson, P., Fernández-Jiménez, A., Provis, J. L., Lukey, G. C., Palomo, A., & van Deventer, J. S. J. (2006). Geopolymer technology: The current state of the art. Journal of Materials Science, 42, 2917–2933.
5. Fongjan, J., & Ludger, L. (2005). Effect of high silica content materials on fly ash-based geopolymeric cement materials. In Proceedings of the Geopolymer 2005 World Congress: Green Chemistry and Sustainable Development Solutions, pp. 107–111.
6. Hardjito, H., &Rangan, R. V. (2005). Development and properties of low-calcium fly ash-based geopolymer concrete. Research Report GC1. Curtin University of Technology.
7. Kumar, S., Mucsi, G., Kristály, F., & Pekker, P. (2017). Mechanical activation of fly ash and its influence on micro- and nano-structural behaviour of resulting geopolymers. Advancesin Powder Technology, 28(5),805-813.
8. Kumar, V., Mathur, M., Sinha, S. S., &Dhatrak, S. (2005). Fly ash: An environmental savior. In Fly Ash UtilisationProgramme (FAUP), TIFAC, DST, pp. IV1.1–IV1.4, Fly Ash India, New Delhi, India.
9. Malhotra, V. M., &Ramezanianpour, A. A. (1994). Fly ash in concrete. Canada Centre for Mineral and Energy Technology (CANMET).
10. Pandey, V. C., Singh, J. S., Singh, R. P., Singh, N., &Yunus, M. (2011). Arsenic hazards in coal fly ash and its fate in the Indian scenario. Resources, Conservation and Recycling, 55(9-10), 819-835.
11. Patankar, Subhash V., Ghugal, Yuwaraj M., Jamkar, Sanjay S., Effect of Concentration of Sodium Hydroxide and Degree of Heat Curing on Fly Ash-Based Geopolymer Mortar, Indian Journal of Materials Science, 2014, 938789, 6 pages, 2014.
12. Rangan, B. V., Hardjito, D., Wallah, S. E., &Sumajouw, D. M. J. (2005). Studies on fly ash-based geopolymer concrete. In Proceedings of the Geopolymer 2005 World Congress: Green Chemistry and Sustainable Development Solutions, pp. 133–137.
13. S. B. Suri, “Fly ash based innovative building products for construction-part-II,” CE & CR, vol.25, pp.134-142,2012.

14. Xu, H., & Van Deventer, J. S. J. (2000). The geopolymerisation of aluminosilicate minerals. International Journal of Minerals Processing, 59(3), 247–266.
15. Yellaiah, P. (2016). Development of geopolymer-based ferrocement panels under flexural loading (Doctoral dissertation, Punjab University, Faculty of Engineering and Technology). National Institute of Technical Teachers Training and Research, Chandigarh.
16. Yousuf, A., Manzoor, S. O., Youssouf, M., Malik, Z., & Khawaja, K. (2020). Fly ash: Production and utilization in India—An overview. Journal of Materials and Environmental Science, 11, 911-921.
17. Yuan, P., Zhang, B., Yang, Y., Jiang, T., Li, J., Qiu, J., & He, H. (2023). Application of polymer cement repair mortar in underground engineering: A review. Case Studies in Construction Materials, 19, e02555.
18. IS 4031 (Part 4):1988 Indian Standard Methods ofDetermination of Consistency of Standard Cement PasteBureau of Indian Standards, New Delhi.
19. IS 4031 (Part 5):1988 Indian Standard Methods ofDetermination of Initial and Final Setting TimeBureau of Indian Standards, New Delhi.
20. IS 4031 Part 6:1988 Indian Standard Methods of Physical Tests for Hydraulic Cement Bureau of Indian Standards, New Delhi.
21. IS 4031 (Part 7):1988 Indian Standard Methods ofDetermination of Compressive Strength of Masonry CementBureau of Indian Standards, New Delhi.
22. Indian Standards: 2250-1981 "International Standard Code of Practice for Preparation and Use of Masonry Mortars", Bureau of Indian Standards, New Delhi, India.