Setting Time and Compressive Strength Development of Mortar Containing Mineral Admixtures and Superplasticizers

Shiv Nadan Sahai Shrivastva[1], Sharad Kumar Soni[2], Kapil Kumar Soni[3]
[1]Research Scholar, Department of Civil Engineering, Rabindra Nath Tagore University, Raisen
[2]Associate Professor, Department of Civil Engineering, Rabindra Nath Tagore University, Raisen
[3]Assistant Professor, Department of Civil Engineering, Rabindra Nath Tagore University, Raisen
Corresponding author: [sharadkumar. soni@rntu.ac.in]

Abstract

This study investigates the effect of mineral admixtures and polycarboxylate ether (PCE)-based superplasticizer on the setting time and compressive strength development of cement mortar. Metakaolin (MK), silica fume (SF), Class F fly ash (FA), and blast furnace slag (BFS) were used as partial cement replacements at 10%, 10%, 20%, and 30%, respectively. Ten mortar mixtures were evaluated, with and without 0.8% superplasticizer. Initial and final setting times and compressive strengths at 3, 7, and 28 days were considered. The results indicate that MK and SF shortened setting time and improved compressive strength, whereas FA and BFS delayed setting and reduced early-age strength. SF10 achieved the highest 28-day compressive strength of 49.1 MPa, approximately 12.1% higher than the control. Superplasticizer generally prolonged setting time and slightly influenced strength development. Overall, MK and SF demonstrated favourable performance, while the combined effects of mineral and chemical admixtures highlight the importance of optimizing mortar composition for improved setting behaviour and mechanical performance.

Keywords: setting time; compressive strength; metakaolin; silica fume; fly ash; blastfurnace slag; superplasticizer; mortar.

1. Introduction

Mineral and chemical admixtures are widely used to modify the fresh and hardened properties of cement-based materials. Metakaolin (MK), silica fume (SF), fly ash (FA), and ground granulated blast-furnace slag (GGBS/BFS) differ in particle size, chemistry, and reaction kinetics; consequently, their effects on setting and strength depend on binder composition, replacement level, and curing age (Lagier & Kurtis, 2007; Rahhal & Talero, 2004; Rao, 2003). Superplasticizers, particularly polycarboxylate ether (PCE) products, improve dispersion and workability, but may also modify hydration and setting behaviour (Javadi et al., 2021; Li et al., 2017).

Metakaolin is a calcined aluminosilicate pozzolan. Isothermal-calorimetry research has shown that metakaolin can catalyse early cement hydration, although the magnitude of the response depends on metakaolin surface area and cement composition (Lagier & Kurtis, 2007). Silica fume can increase early hydration reactions and, in some systems, reduce initial setting time; its influence on final setting time is often less pronounced (Rao, 2003). By contrast, the early contribution of Class F fly ash is commonly limited by dilution and slower pozzolanic reaction, although its behaviour depends on ash characteristics and cement chemistry (Rahhal & Talero, 2004).

Polycarboxylate ether (PCE) superplasticizers disperse cementitious particles through adsorption and steric effects. Their interaction with cement can also retard hydration: impedance-spectroscopy measurements have linked superplasticizer dosage with an extended dormant period and delayed setting (Torrents et al., 1998). More recent work shows that PCE molecular structure, adsorption, and dosage influence hydration retardation and fluidity (Javadi et al., 2021; Li et al., 2017).

Setting time and compressive strength are practical indicators of constructability and early hardened performance. Previous investigations indicate that metakaolin and silica fume can promote early hydration and strength, whereas fly ash may reduce early-age strength while contributing at later ages; PCE admixtures can alter these responses through their interaction with cement particles (Lagier & Kurtis, 2007; Rahhal & Talero, 2004; Rao, 2003; Singh et al., 1992).

The objective of this experimental study is to quantify the effects of MK, SF, FA, and BFS, with and without a polycarboxylate superplasticizer, on the initial and final setting times and compressive strength development of mortar. The study aims to identify combinations that provide acceptable setting behavior and strength development for practical use.

2. Literature Review

2.1 Setting Behaviour of Cementitious Systems with Mineral Admixtures

The setting behaviour of cementitious systems is influenced by mineral-admixture chemistry, fineness, replacement percentage, and cement composition. Lagier and Kurtis (2007) reported a catalytic effect of metakaolin on early cement hydration, while Rao (2003) found that increasing silica-fume content reduced initial setting time in the investigated pastes, with comparatively little effect on final setting time. These findings indicate that the response is system-specific rather than universal.

Fly ash can delay setting through cement dilution and its comparatively slow early pozzolanic contribution, although ash chemistry and cement composition influence the magnitude and even direction of the response (Rahhal & Talero, 2004). Singh et al. (1992) reported increased setting time in a white-Portland-cement/fly-ash system and observed that superplasticizer further influenced hydration and strength. Slag-containing binders also require careful interpretation because their setting and strength development depend on slag fineness, activation, and the properties of the accompanying cement.

2.2 Retarding Effect of Superplasticizers

Polycarboxylate-based superplasticizers are used to improve dispersion and reduce water demand, but their effects on setting are not limited to physical workability. Torrents et al. (1998) showed that superplasticizer addition prolonged the dormant stage of cement hydration and

delayed the onset of the acceleration period. Li et al. (2017) found that both adsorbed PCE and PCE remaining in the pore solution contributed to retardation in the UHPC systems studied. Javadi et al. (2021) further demonstrated that PCE adsorption and retardation depend on copolymer structure and carboxylate content.

2.3 Compressive Strength Development

The effect of mineral admixtures on compressive strength depends on replacement level, curing age, fineness, and reactivity. Metakaolin may improve early strength through accelerated hydration, nucleation, and pozzolanic contributions (Lagier & Kurtis, 2007). Rao (2003) reported increased mortar strength with increasing silica-fume content in the studied mixtures. Fly ash may reduce early-age strength but contribute to later-age performance as pozzolanic reactions progress (Rahhal & Talero, 2004; Singh et al., 1992). The influence of a superplasticizer is similarly system-dependent and should be evaluated alongside water content, dispersion, and hydration (Colorado-Lopera et al., 2022; Li et al., 2017).

2.4 Research Gap

Although the effects of individual mineral admixtures and superplasticizers have been investigated, direct comparisons across several mineral admixture types under a common mortar-mixing and testing framework remain useful. The present programme compares MK, SF, FA, and BFS mixtures, each with and without a fixed PCE dosage, to examine the reported setting-time and compressive-strength trends.

3. Experimental Program

3.1 Materials

Ordinary Portland cement (OPC) conforming to EN 197-1 was used as the control binder. The mineral admixtures were metakaolin (MK), silica fume (SF), Class F fly ash (FA), and blast-furnace slag (BFS). A polycarboxylate-based high-range water reducer (SP) conforming to ASTM C494/C494M Type F was used. Standard sand was used as fine aggregate, and potable water was used for mixing (ASTM International, 2019; European Committee for Standardization, 2011).

3.2 Mix Proportions

Mortar mixtures were prepared with a bindertosand ratio of 1:3 by mass and a watertobinder ratio (w/b) of 0.45. Mineral admixtures replaced cement by mass. The SP dosage was fixed at 0.8% by mass of binder for all SPcontaining mixtures. The mix proportions are given in Table 1.

Table 1. Mortar mix proportions

Mix ID	Cement replacement	SP type	SP dosage (% binder)	w/b	Binder:sand
C	0%	—	0	0.45	1:3
MK10	10% MK	—	0	0.45	1:3
SF10	10% SF	—	0	0.45	1:3
FA20	20% FA	—	0	0.45	1:3
BFS30	30% BFS	—	0	0.45	1:3
C-SP	0%	PCE	0.8	0.45	1:3
MK10-SP	10% MK	PCE	0.8	0.45	1:3
SF10-SP	10% SF	PCE	0.8	0.45	1:3
FA20-SP	20% FA	PCE	0.8	0.45	1:3
BFS30-SP	30% BFS	PCE	0.8	0.45	1:3

Figure 1 presents a flow chart of the experimental program, from material preparation to the final analysis.

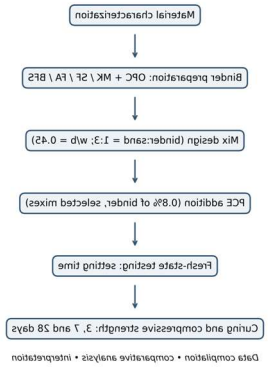

Figure 1. Flow chart of the experimental programme.

3.3 Test Methods

Setting time. Initial and final setting times were reported as measured using the Vicat apparatus with reference to IS 4031 (Part 5):1988 (Bureau of Indian Standards [BIS], 1988a). This standard specifies determination of initial and final setting times of hydraulic cement. The manuscript must clarify whether the tested material was binder paste or mortar, because the standard setting-time procedure is a cement-paste test; the present description of testing mortar samples requires confirmation.

Compressive strength. Compressive strength was determined on 70.6 mm mortar cubes with reference to IS 4031 (Part 6):1988, which covers compressive-strength testing of hydraulic cement other than masonry cement (BIS, 1988b). Specimens were cured in water at 27 ± 2 °C and tested at 3, 7, and 28 days.

4. Results and Discussion

4.1 Setting Time

The initial and final setting times are presented in Table 2. Figure 2 compares the initial and final setting times across all mixtures.

Table 2. Initial and final setting times of mortar mixtures (illustrative values)

Mix ID	Initial setting time (min)	Final setting time (min)	Change in IST vs. C (%)	Change in FST vs. C (%)
C	115	260	—	—
MK10	95	225	−17.4	−13.5
SF10	105	240	−8.7	−7.7
FA20	145	310	+26.1	+19.2
BFS30	150	330	+30.4	+26.9
C-SP	135	300	+17.4	+15.4
MK10-SP	125	285	+8.7	+9.6
SF10-SP	135	300	+17.4	+15.4
FA20-SP	175	350	+52.2	+34.6
BFS30-SP	185	375	+60.9	+44.2

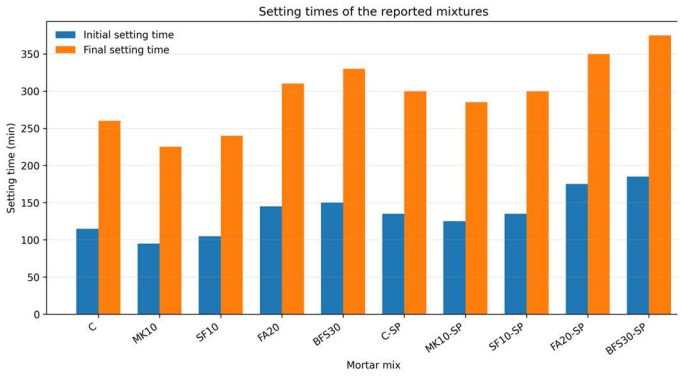

Figure 2. Initial and final setting times of the mixtures (illustrative dataset).

The control mixture had an initial setting time of 115 min and a final setting time of 260 min. MK10 shortened the initial setting time by about 17%, while SF10 shortened it by about 9%. These trends are consistent with published observations of accelerated early hydration and reduced initial setting time in some metakaolin- and silica-fume-containing systems (Lagier & Kurtis, 2007; Rao, 2003). FA20 and BFS30 showed longer setting times. The addition of SP delayed setting in all mixtures, consistent with reported PCE-related retardation of cement hydration (Javadi et al., 2021; Li et al., 2017; Torrents et al., 1998).

4.2 Compressive Strength

The compressive strength results are presented in Table 3. Figure 3 shows the strength development from 3 to 28 days.

Table 3. Compressive strength of mortar mixtures at 3, 7, and 28 days (illustrative values)

Mix ID	3-day strength (MPa)	7-day strength (MPa)	28-day strength (MPa)	28-day change vs. C (%)
C	26.5	34.2	43.8	—
MK10	30.1	38.0	47.5	+8.4
SF10	31.2	39.4	49.1	+12.1
FA20	21.8	29.5	40.2	−8.2
BFS30	20.5	28.4	41.0	−6.4
C-SP	24.8	32.6	43.1	−1.6
MK10-SP	28.4	36.5	46.0	+5.0
SF10-SP	29.5	37.8	47.6	+8.7
FA20-SP	20.1	27.8	38.9	−11.2
BFS30-SP	18.9	26.7	39.5	−9.8

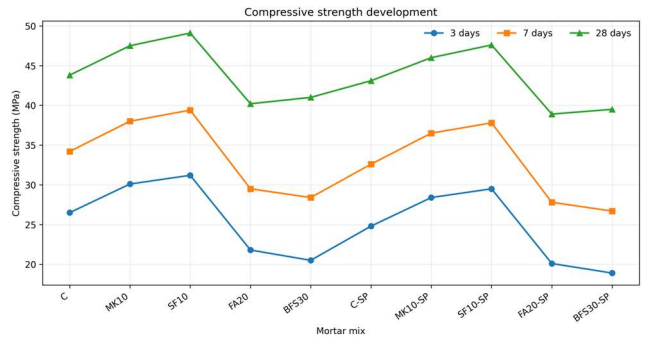

Figure 3. Compressive strength development at 3, 7, and 28 days (illustrative dataset).

Within the reported dataset, MK10 and SF10 increased compressive strength at all test ages, with SF10 reaching the highest 28-day value (49.1 MPa, approximately 12% above the control). The result is consistent with published evidence that metakaolin can accelerate early hydration and that silica fume can improve mortar strength, although the magnitude depends on mixture proportions and water demand (Lagier & Kurtis, 2007; Rao, 2003). FA20 and BFS30 showed lower early-age strengths, while their 28-day values increased with curing. The effect of fly ash on later-age strength is consistent with the development of pozzolanic reaction products over time (Rahhal & Talero, 2004; Singh et al., 1992).

4.3 Discussion

The reported results indicate an interaction between mineral-admixture type and superplasticizer addition. MK and SF mixtures showed higher strengths and shorter setting times than the control without SP, whereas FA and BFS mixtures showed longer setting times and lower early-age strengths. The PCE-containing mixtures generally showed delayed setting, a response reported in studies of PCE adsorption and cement hydration (Javadi et al., 2021; Li et al., 2017; Torrents et al., 1998). Because the mix series contains only one SP dosage and the numerical dataset is identified as illustrative, the apparent ranking should be treated as a trend for follow-up experimental verification rather than a definitive optimum.

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4.3.1 Relationship between Setting Time and Mineral-Admixture Type

The setting-time results demonstrate that the response is influenced by the type of cement replacement rather than simply by the presence of a mineral admixture. In the illustrative dataset, MK10 reduced the initial and final setting times by 17.4% and 13.5%, respectively, relative to the control. SF10 produced smaller reductions of 8.7% and 7.7%. This difference suggests that the two fine, reactive materials should not be treated as

interchangeable: their influence depends on their individual physical and chemical characteristics and their interaction with the cement. The direction of these trends agrees with investigations reporting accelerated early hydration in metakaolin- and silica-fume-containing systems (Lagier & Kurtis, 2007; Rao, 2003).

In contrast, FA20 and BFS30 increased both initial and final setting times. The initial setting time increased by 26.1% for FA20 and 30.4% for BFS30, while the corresponding final setting-time increases were 19.2% and 26.9%. A plausible explanation is the combined influence of cement dilution and the comparatively slower early reaction of these materials. The magnitude of this response should be considered specific to the replacement levels, cement composition, fineness, and curing conditions represented by the mix programme (Rahhal & Talero, 2004).

4.3.2 Influence of Superplasticizer on Setting Behaviour

The addition of 0.8% PCE increased the initial and final setting times in every corresponding mixture. For example, the control mixture changed from 115 to 135 min in initial setting time and from 260 to 300 min in final setting time. The delay was also evident in the mineral-admixture mixtures: MK10-SP and SF10-SP had longer setting times than MK10 and SF10, while FA20-SP and BFS30-SP recorded the longest setting times in the series. This consistent direction of change indicates that the PCE dosage used in the programme did not eliminate the setting-time differences associated with the mineral admixtures; rather, it added a further retardation effect.

The comparatively large setting-time values in FA20-SP and BFS30-SP are important from a construction-planning perspective. Where early finishing, formwork removal, or rapid construction cycles are required, these combinations may require adjustment of admixture dosage or binder proportion. Conversely, a longer workable period may be useful in applications involving extended transport or placement, provided that the delay remains within project specifications. These are potential practical implications, not performance claims established by the illustrative dataset. The interpretation is consistent with research linking PCE adsorption and pore-solution interactions to delayed cement hydration (Javadi et al., 2021; Li et al., 2017; Torrents et al., 1998).

4.3.3 Age-Dependent Compressive-Strength Development

The strength results show that the relative performance of the mineral admixtures is visible at 3 days and remains evident at 7 and 28 days. SF10 recorded 31.2 MPa at 3 days and 49.1 MPa at 28 days, compared with 26.5 MPa and 43.8 MPa for the control. MK10 also exceeded the control at all three ages. The higher values for these two mixtures are compatible with the combined contribution of particle packing, nucleation, and pozzolanic reaction, although the relative contribution of each mechanism cannot be separated without additional tests such as calorimetry or microstructural analysis (Lagier & Kurtis, 2007; Rao, 2003).

FA20 and BFS30 exhibited lower strengths than the control at 3 and 7 days. Their 28-day strengths increased to 40.2 and 41.0 MPa, respectively, but remained below the control value of 43.8 MPa. The increase with curing age is consistent with the gradual contribution of pozzolanic and latent hydraulic reactions. However, the 28-day results do not demonstrate that either mixture had fully recovered the strength reduction associated with cement replacement. Longer-age testing would be needed before drawing conclusions about their later-age strength potential (Rahhal & Talero, 2004; Singh et al., 1992).

4.3.4 Effect of Superplasticizer on Strength

At the fixed water-to-binder ratio of 0.45, the SP-containing mixtures generally recorded slightly lower compressive strengths than their corresponding mixtures without SP. The difference was modest at 28 days: C-SP was 1.6% below C, while MK10-SP, SF10-SP, FA20-SP, and BFS30-SP were approximately 3.2%, 3.1%, 3.2%, and 3.7% below their respective mixtures without SP. The relative strength ranking associated with the mineral admixture was broadly maintained after SP addition in this dataset.

This comparison must be interpreted carefully because the water-to-binder ratio was held constant and no separate workability or flow measurements are reported in the paper. In practical mix design, a principal strength benefit of superplasticizer may arise when it is used to reduce water content while maintaining the required workability. Since water reduction was not part of the present mix matrix, the data should not be used to conclude that PCE inherently reduces strength. The observed differences may reflect hydration retardation, dispersion effects, or other mixture-specific interactions and require verification through replicated testing (Javadi et al., 2021; Li et al., 2017).

4.3.5 Comparative Performance and Practical Interpretation

Considering setting time and compressive strength together, the MK10 and SF10 mixtures without SP provided the most favourable balance in the reported dataset: they shortened setting time and exceeded the control strength at all test ages. Among these two mixtures, SF10 produced the highest 28-day strength, whereas MK10 produced the larger reduction in initial setting time. Selection between the two would therefore depend on whether the design priority is higher 28-day strength or a shorter setting period. This comparison is descriptive and should not be interpreted as a statistically established optimum.

The SP-containing MK and SF mixtures retained higher 28-day strengths than the control mixture, but their setting times were longer than those of the corresponding mixtures without SP. FA20-SP and BFS30-SP combined the longest setting times with the lowest early-age strengths in the series. These combinations may be considered only where delayed setting is acceptable and the mix is supported by adequate curing and project-specific strength verification. A final selection should also consider workability, water demand, durability, cost, and environmental benefits of cement replacement, none of which can be fully assessed from the current results alone.

4.3.6 Limitations and Further Verification

The comparisons above are based on the illustrative values identified in the manuscript and demonstrate how the results may be interpreted; they are not verified laboratory findings. Before the discussion is used in a research submission, the values must be replaced with measured results, and each mixture should be tested using replicate specimens. The mean, standard deviation, and coefficient of variation should be reported so that the reliability of differences between mixtures can be evaluated.

Further work should include additional curing ages, particularly 56 and 90 days, to assess the later-age contribution of fly ash and slag. Multiple PCE dosages and, where appropriate, a water-reduced series would help distinguish the effect of admixture dosage from the effect of water content. Flow or workability measurements should accompany the setting-time and strength tests. The setting-time procedure also requires confirmation because IS 4031 (Part 5):1988 specifies a cement-paste test; the manuscript should accurately describe the tested material and applicable procedure before the method is considered final.

5. Conclusions

1. Metakaolin and silica fume shortened initial and final setting times, whereas fly ash and blastfurnace slag prolonged them.

2. The polycarboxylate superplasticizer delayed setting in all mixtures, with the most pronounced delay in FA20 and BFS30 blends.

3. MK10 and SF10 increased compressive strength at 3, 7, and 28 days; SF10 gave the highest 28day strength.

4. FA20 and BFS30 reduced earlyage strength but showed strength gain at 28 days.

5. The addition of SP slightly reduced early strength in the reported dataset, while 28-day strength remained close to the corresponding mixtures without SP. Such responses are system-dependent and should be interpreted in conjunction with water content, admixture chemistry, and hydration kinetics (Colorado-Lopera et al., 2022; Li et al., 2017).

6. MK10SP and SF10SP are recommended for balanced setting and strength; FA20SP and BFS30SP are more suitable for delayed setting and longterm strength applications.

References

ASTM International. (2019). ASTM C494/C494M-19: Standard specification for chemical admixtures for concrete. ASTM International.

Bureau of Indian Standards. (1988a). IS 4031 (Part 5):1988: Methods of physical tests for hydraulic cement—Part 5: Determination of initial and final setting times (First revision). Bureau of Indian Standards.

Bureau of Indian Standards. (1988b). IS 4031 (Part 6):1988: Methods of physical tests for hydraulic cement—Part 6: Determination of compressive strength of hydraulic cement other than masonry cement (First revision). Bureau of Indian Standards.

Colorado-Lopera, H. A., Muñoz-Zapata, A., & Cifuentes-Mosquera, S. (2022). Portland cement mortars tested with two superplasticizers: A case study to reduce cement and water in concrete. *Tecnura, 26*(72), 114–146. https://doi.org/10.14483/22487638.16824

European Committee for Standardization. (2011). *EN 197-1:2011: Cement—Part 1: Composition, specifications and conformity criteria for common cements*. CEN.

Javadi, A., Jamil, T., Abouzari-Lotf, E., Soucek, M. D., & Heinz, H. (2021). Working mechanisms and design principles of comb-like polycarboxylate ether superplasticizers in cement hydration: Quantitative insights for a series of well-defined copolymers. *ACS Sustainable Chemistry & Engineering, 9*(25), 8354–8371. https://doi.org/10.1021/acssuschemeng.0c08566

Lagier, F., & Kurtis, K. E. (2007). Influence of Portland cement composition on early age reactions with metakaolin. *Cement and Concrete Research,*

37(10), 1411–1417. https://doi.org/10.1016/j.cemconres.2007.07.002

Li, P. P., Yu, Q. L., & Brouwers, H. J. H. (2017). Effect of PCE-type superplasticizer on early-age behaviour of ultra-high performance concrete (UHPC). *Construction and Building Materials, 153*, 740–750. https://doi.org/10.1016/j.conbuildmat.2017.07.145

Rahhal, V., & Talero, R. (2004). Influence of two different fly ashes on the hydration of Portland cements. *Journal of Thermal Analysis and Calorimetry, 78*, 191–205. https://doi.org/10.1023/B:JTAN.0000042167.46181.17

Rao, G. A. (2003). Investigations on the performance of silica fume-incorporated cement pastes and mortars. *Cement and Concrete Research, 33*(11), 1765–1770. https://doi.org/10.1016/S0008-8846(03)00171-6

Singh, N. B., Dwivedi, M. P., & Singh, N. P. (1992). Effect of superplasticizer on the hydration of a mixture of white Portland cement and fly ash. *Cement and Concrete Research, 22*(1), 121–128. https://doi.org/10.1016/0008-8846(92)90142-I

Torrents, J. M., Roncero, J., & Gettu, R. (1998). Utilization of impedance spectroscopy for studying the retarding effect of a superplasticizer on the setting of cement. *Cement and Concrete Research, 28*(9), 1325–1333. https://doi.org/10.1016/S0008-8846(98)00110-0