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Research Article

Effect of different activations of supplementary cementitious materials on the early-age compressive strength of mortars based on quaternary binders

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ABSTRACT

In recent years, building materials have seen the widespread use of supplementary cementitious materials (SCMs) due to important environmental considerations. However, most SCMs exhibit slow development of compressive strength in the early stages. This study aims to evaluate and compare three combined activation strategies of SCMs, namely natural pozzolana (NP), blast furnace slag (BFS) and limestone filler (LF), incorporated with a replacement level of 50% of cement. SCMs activation was achieved through three approaches: mechanical (grinding to Blaine fineness values of 3800 and 6000 cm²/g), thermal (curing at 20 and 40 °C), and chemical (addition of 1% NaOH by mass of binder). Early compressive strength (2 and 7 days) was measured and analyzed using a fractional factorial design. The results demonstrate that all investigated combinations of SCM activations generate synergistic effects by significantly improving the mechanical performance of early-age quaternary mortars, due to an acceleration of hydration reactions and an improvement in the densification of the material's microstructure. Statistical analysis (ANOVA) revealed that curing temperature is one of the most influential parameters on early compressive strength. Increasing the temperature from 20 to 40°C accelerates hydration and pozzolanic reactions, thus promoting faster calcium silicate hydrate C-S-H gel formation and a significant improvement in strength gain. Furthermore, the binder composition and the degree of substitution of SCMs strongly influence mechanical performance. Formulations containing adequate proportions of BFS and NP exhibit higher reactivity during activation.

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1. Introduction

Cement production is among the most energy-intensive industrial processes, accounting for 3% of annual global energy production and contributing to about 8%

of anthropogenic greenhouse gas emissions (Miller and Moore 2020; UNEP 2026; Yu et al. 2024). In the context of the energy transition, political, regulatory and technological pressures are leading to a reconsideration of traditional manufacturing pathways and underline the ur-

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gency of low-carbon cement manufacturing approaches. Indeed, one of the proposed and effective solutions consists in partial replacement of Portland cement clinker by alternative materials exhibiting pozzolanic or latent hydraulic properties.

Various Supplementary Cementitious Materials (SCMs), such as blast furnace slag (BFS), fly ash (FA), metakaolin (MK), silica fume (SF), natural pozzolan (NP) and limestone filler (LF), have been widely used in mortar and concrete mixtures (Khan et al. 2022; Pang et al. 2022; Berkak et al. 2025; Al-Safi et al. 2025; Chenafi et al. 2026). The impact of SCMs on the future of the cement industry will depend on multiple factors, including the availability, production efficiency and profitability of these SCMs. Their effects on mortar performance are also strongly dependent on replacement level and binder composition (Mohamad et al. 2025).

In addition, the development of quaternary composite cements combines scientific and technological advances in the fields of cement and concrete. It effectively meets mechanical performance requirements while promoting sustainable development and environmental preservation (Alcan et al. 2024; Makhloufi et al. 2012; Vignesh and Rahim 2022; Makhloufi et al. 2015). In quaternary mixtures, an optimal LF replacement level in the binder of between 8 and 17% leads to an early-age reaction equivalent to or greater than that of OPC and to a compressive strength at 28 days comparable to that of binary mortar mixtures (Mounanga et al. 2011). The detrimental effect of silica fume (SF) on the workability of fresh mortar can be compensated by the simultaneous use of SF and LF (Zelić et al. 2009; Li et al. 2019). Moreover, mortars and concretes containing a quaternary binder, composed of cement simultaneously incorporating fly ash (FA), LF and metakaolin (MK), may also be more efficient than CEM I cement in terms of compressive strength after 28 days of curing, but they have a low initial strength due to the dilution effect of LF; furthermore, when the LF content increases from 5% to 10%, the reduction in strength becomes significant (Lin et al. 2021). According to work of Rojo-López et al. (2020), quaternary binder mortars containing granulated blast furnace slag (GGBS) and MK have better performance in terms of permeability and compressive strength than those using LF.

The pozzolanic reaction occurs through the chemical interaction between the amorphous silica or silica and alumina present in SCMs and calcium hydroxide ($\text{Ca}(\text{OH})_2$), generated during cement hydration. This reaction leads to the formation of additional hydration products, mainly C-S-H and C-A-S-H gels, which contribute to the enhancement of mechanical strength (Allahverdi and Salem 2010). Dave et al. (2016) reported that quaternary blended cements, incorporating metakaolin (MK) at a high replacement level of 50% with fly ash (FA) and silica fume (SF), can achieve compressive strength comparable to that of the control mortar at early ages. Moreover, the compressive strength was found to increase significantly during the early age as the MK content in the quaternary system increases. This enhancement is primarily attributed to the activation of tricalcium aluminate (C_3A) and tricalcium silicate (C_3S) phases in Portland cement (Dave et al. 2017).

Nevertheless, the use of SCMs, especially with high substitution rates, is often limited by a slow strength development during the early ages of cement hydration (Dong et al. 2002). An improvement in its responsiveness is therefore required through the use of SCM activation techniques (Dong et al. 2002; Sajedi and Razak 2011a, 2011b). Several methods have been implemented to increase the reactivity of pozzolanic materials in the short term. They can be classified into three categories: mechanical (grinding), thermal (thermal curing), and chemical (Go et al. 2010; Allahverdi and Mahinroosta 2013; Ababneh et al. 2025; Li et al. 2013; Elbar et al. 2018). Each method of SCM activation has advantages and limitations.

Mechanical activation, induced by grinding, results from a set of transformations: particle fracture, various physicochemical changes in volume and surface (Kumar and al. 2008), while generating defects (dislocations, surface amorphization) (Sobolev 2005). The accumulated mechanical energy during grinding increases the specific surface area and reactivity of the material, thereby enhancing early-age strength through the greater availability of reactive silica for the formation of calcium silicate hydrate (C-S-H) (Zivanovic et al. 2002; Indrawati and Manaf 2008; Ababneh et al. 2025), hence the sustained interest and numerous studies on the subject (Kumar et al. 2008; Oner et al. 2003; Fu et al. 2008).

Allahverdi and Mahinroosta (2013) reported that all the results show that mechanical activation is an effective approach, used in addition to other techniques, to improve the performance of cementitious systems containing a high proportion of chemically activated slag. However, extended milling duration increases energy consumption, which can limit large-scale adoption. Moreover, the cost-effectiveness of mechanical activation remains a concern.

Moreover, thermal curing at high temperature inevitably induces significant changes in the physicochemical and mechanical properties of SCMs (Sajedi and Razak 2011a; Allahverdi and Saffari 2011). Increasing the curing temperature accelerates hydration kinetics in accordance with Arrhenius's law, thereby promoting the development of higher early-age mechanical strength in slag cement mortars (Ezziane 2010). When the curing temperature exceeds 30 °C, a reduction in the final setting time is generally observed, thus reflecting an acceleration of the curing process of pozzolanic mortars (Ababneh et al. 2025).

However, the application of thermal activation remains poorly adapted to conventional concretes, mainly due to its high energy consumption and technical constraints related to practical conditions. The prolonged exposure to high temperatures can cause thermal shrinkage phenomena and promote the appearance of cracks, particularly in mass concrete structures (Ababneh et al. 2025).

Chemical activation involves the incorporation of alkaline activators, such as sodium hydroxide (NaOH) or sodium sulfate (Na_2SO_4), aimed at giving the material a cementitious character and/or activating its latent hydraulic properties (Bellmann and Stark 2009; Sajedi and Razak 2011b). The type of alkaline activator can also significantly influence the resulting mechanical perfor-

mance (Ünal and Canbaz 2025). In concretes incorporating pozzolans, chemical activators (e.g., NaOH, Na₂SO₄), introduced either during SCM grinding or at the mixing stage, accelerate the pozzolanic reaction and enhance strength development (Shi and Day 2001).

Although the introduction of one or more activators increases the overall cost of the materials, the cost relative to the enhancement in mechanical strength obtained decreases (Shi and Day 2001).

However, this activation method remains limited by the long-term stability of the reaction products and by the environmental impacts associated with the use of chemical reagents. Moreover, the effectiveness of chemical activation depends closely on the composition of the pozzolan, which differs from one region to another (Ababneh et al. 2025).

1.1. Scope and significance

SCMs, such as natural pozzolan and blast furnace slag, generally develop lower compressive strengths at early ages compared to Portland cement, especially at high replacement levels (Ghrici 2007). Despite the extensive research devoted to the activation of a SCM by a single and isolated approach, studies on the combined influence of mechanical, chemical and thermal activations on the physico-mechanical properties of mortars with quaternary binders and high substitution rates remain insufficient. Moreover, the lack of systematic experimental approaches limits the understanding of both individual and interaction effects of these activation strategies. In the context, the present study aims to fill this gap by evaluating, using a fractional factorial plan, the joint effect of these three SCM activation methods on the compressive strength of quaternary binder mortars at early-age with a substitution rate of 50% of Portland cement. A fractional factorial design is employed to not only assess the overall effect of these activation methods but also to quantify their main and interaction effects on early-age compressive strength. This approach provides new in-

sights into the synergistic behavior of multiple activation strategies in highly substituted quaternary systems.

2. Materials and Experimental Procedures

2.1. Raw materials

Binders used in this research include ordinary Portland cement (OPC) and SCMs, such as limestone fillers (LF), blast furnace slag (BFS) and natural pozzolan (NP). The chemical and mineralogical compositions of OPC and SCMs, determined by X-ray fluorescence (XRF), are presented in Table 1. The densities of OPC, LF, BFS, and NP are 3.0, 2.7, 2.8, and 2.6 g/cm³, respectively. The Blaine specific surface area (cm²/g) of the cement is 3500 while that of the SCM is 3800 and 6000 (± 100). X-ray diffraction analyses of the SCMs used are shown in Fig. 1.

Crushed limestone sand was used. The filler content passing through the 80 µm diameter sieve is 8.25 %, which is acceptable according to standard NF P 18-301 (1983). The physical characteristics of the sand are reported in Table 2. The fineness modulus is 2.30 and the sand equivalent is 76.46%, indicating clean and negligible clay fraction.

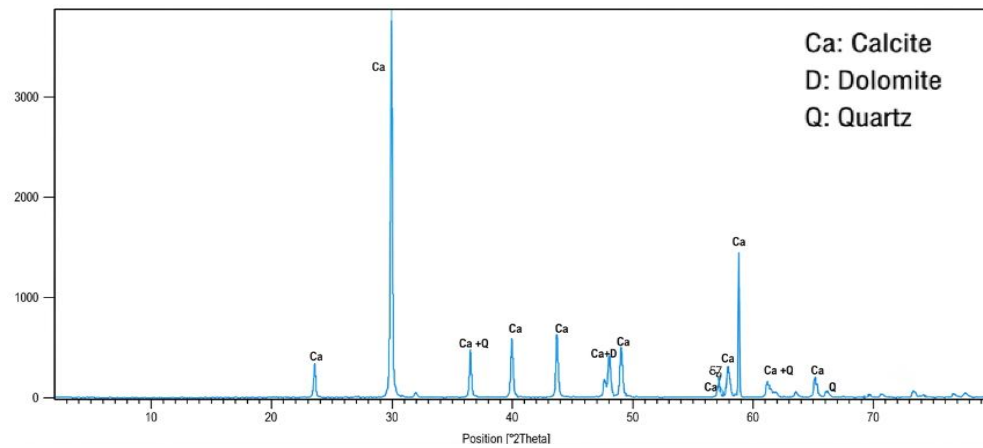
2.2. Design of mixtures

In order to identify the most suitable SCMs activation process for quaternary mixtures, a Design of Experiments (DOE) approach based on the fractional factorial design was applied. Four main factors were considered in the DOE for the proportions of the mixture: (i) the compositions of the cementitious binders (noted from C1 to C7), (ii) mechanical activation, (iii) thermal activation and (iv) chemical activation. Each factor was evaluated at two levels, namely -1 (low level) and +1 (high level), except that the composition of the binders which was tested with 7 levels, in accordance with the modalities mentioned in Table 3.

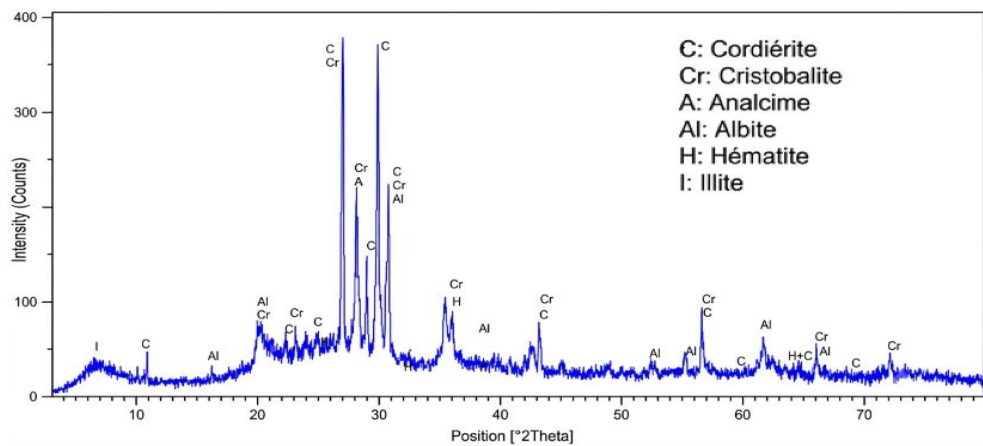
Table 1. Chemical and mineralogical composites of different compounds (%).

Composition	OPC	LF	NP	BFS
SiO ₂	38.21	0.76	10.57	39.38
Al ₂ O ₃	5.59	0.41	15.82	5.64
Fe ₂ O ₃	3.21	0.23	6.16	2.3
CaO	65.26	54.9	5.95	40.3
MgO	1.72	0.61	2.09	4.50
K ₂ O	0.47	0.24	2.0	0.46
Na ₂ O	0.19	0.04	1.1	0.13
SO ₃	0.56	0.61	0.28	0.90
Cl ⁻	0.02	0.005	1.40	–
LOI*	0.58	36.3	1.2	0.8
Cement mineralogy (%)	C ₂ S	C ₃ S	C ₃ A	C ₄ AF
	64.15	97.60	9.39	9.76

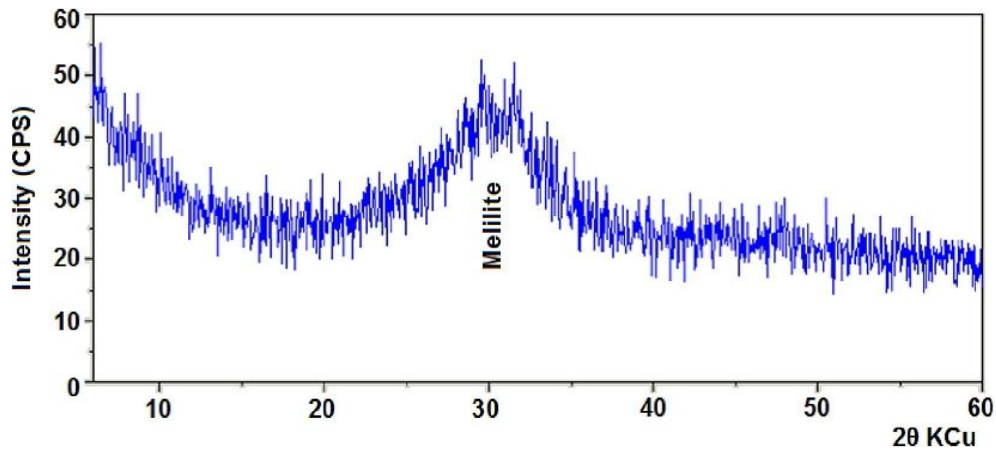
*Loss on ignition.



(a) Limestone Filler



(b) Natural pozzola



(c) Blast furnace slag

Fig. 1. X-ray diffraction of SCMs used.**Table 2.** Physical properties of sand used.

Physical characteristics	Values
Specific density (g/cm ³)	2.70
Apparent density (g/cm ³)	1.53
Fineness modulus	2.30
Compactness (%)	56.70
Porosity (%)	43.30
Absorption coefficient (%)	4.30
Visual sand equivalent (%)	34.71
Piston sand equivalent (%)	46.76

Table 3. Factors and levels used in the fractional factorial design (2^{3-1}).

Factor	Description	Level 1 (-1)	Level 2 (+1)
F	Mechanical activation (fineness) (cm^2/g)	3800	6000
T	Thermal activation ($^{\circ}\text{C}$)	20	40
A	Chemical activation (% NaOH)*	0	1

*% in mass of binder.

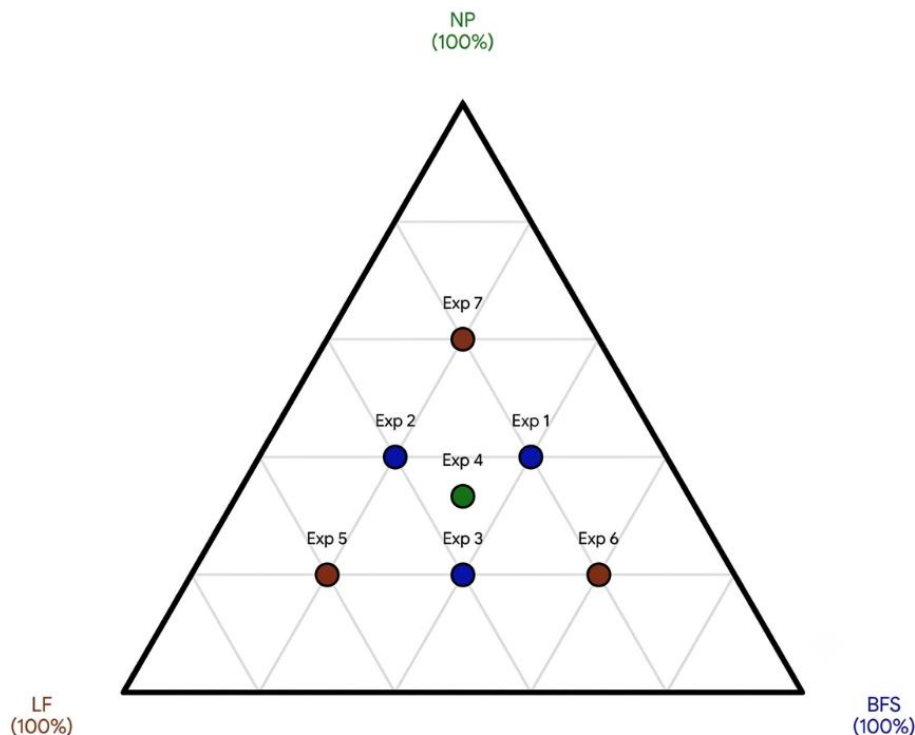
In the regression analysis, composition variable “Comp” was treated as a categorical (nominal) factor comprising seven levels (C1 to C7). To incorporate this factor into the regression model and avoid multicollinearity, the categories were transformed into numerical variables using dummy coding. Specifically, each level was represented by a binary variable (0 or 1), with category C1 defined as the reference baseline level to ensure proper model estimation.

For the three activation factors, a fractional factorial design 2^{3-1} was adopted in order to reduce the number of experimental runs while still allowing estimation of the main effects and selected interaction effects. In this design, the defining relation was selected so that the most influential effects could be estimated with minimal confounding; thus, main effects were considered unaliased

with each other, whereas higher-order interactions were assumed to be negligible.

The effects of the studied factors (responses) on compressive strength at early-age (2 and 7 days) were examined, in order to evaluate the early-age performance of the quaternary binders. Each response was analyzed separately. The experimental results were statistically processed using analysis of variance (ANOVA), which made it possible to identify the significant factors and interactions and to assess their relative contribution to strength development.

On the other hand, the binder compositions (Fig. 2) were prepared using a mixture design of experiments, by substituting 50% of OPC by SCMs. The composition of the various binders, including the reference mortar, is presented in Table 4. Thus, the total number of experiments carried out for each response is 36.

**Fig. 2.** Triangular representation of seven compositions studied.**Table 4.** Composition of binders.

Composition (%)	C0	C1	C2	C3	C4	C5	C6	C7
Clinker	95.0	47.5	47.5	47.5	47.5	47.5	47.5	47.5
Gypsum	5.0	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Limestone	0	10	20	20	16.67	30	10	10
Slag	0	20	10	20	16.67	10	30	10
Pozzolana	0	20	20	10	16.67	10	10	30

2.3. Mix proportions and specimens preparation

All the mixtures prepared were produced with a water to binder ratio (W/B) of 0.50, where the binder is defined as the sum of cement and SCMs forming the quaternary system. For the reference mortar (100% cement), the W/B ratio is indeed equal to the water-to-cement ratio (W/C = 0.50). In addition, a sand-to-binder ratio of 3 was used and the dosage of the superplasticizer is determined so as to ensure appropriate handling of the mixtures, while keeping the W/B ratio constant. It should be noted that the dosage of the superplasticizer varied from

1.1 to 2.9% by weight of the binder. The results, illustrated by Fig. 3, revealed that the superplasticizer saturation doses were significantly higher than those recommended by the manufacturer for common concretes and mortars (1.5% by weight of the binder).

Fig. 4 compares the water demand of the control mortar with and without superplasticizer. It is clearly apparent that the incorporation of the superplasticizer allows a significant reduction in the water/cement ratio while retaining equivalent workability, which results in a notable improvement in the mechanical properties.

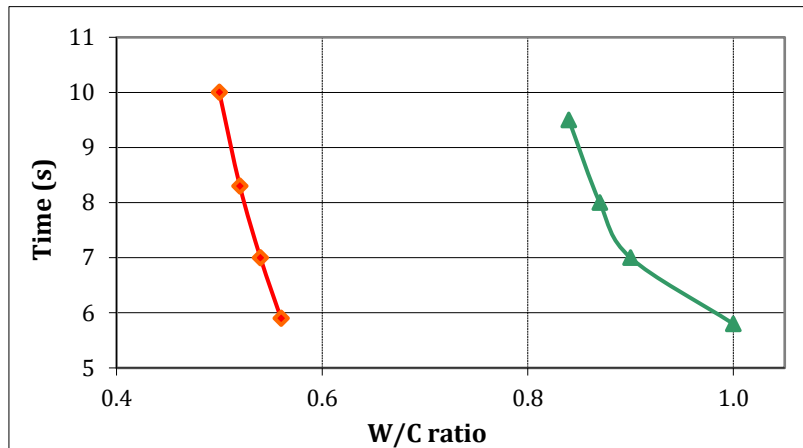


Fig. 3. Evolution of flow time of reference mortar as a function of the W/C ratio.

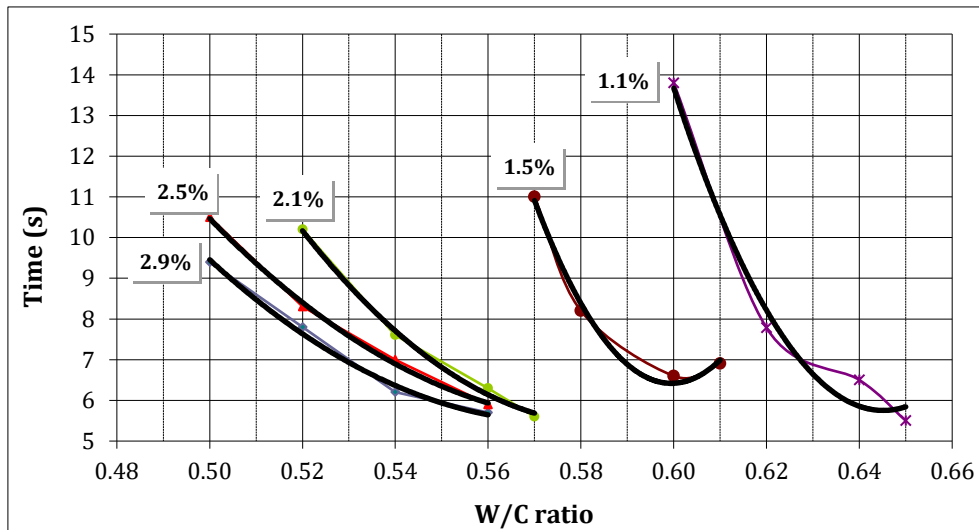


Fig. 4. Comparison of flow-time as a function of W/C ratio for control mortar with and without superplasticizer.

The processes for mixing and preparing the samples, with dimensions of $40 \times 40 \times 160 \text{ mm}^3$, were defined in accordance with standard NF EN 206-1 (2004). The prepared mortar samples were covered with a polyethylene film and maintained in their molds for 24 h under laboratory conditions. After demolding, they were immersed in water for an additional 24 h to ensure uniform early hydration. Subsequently, the samples were cured at $20 \pm 2^\circ\text{C}$ with 65% RH in laboratory condition until the time of testing. This curing procedure was adopted to combine reproducible early-age curing with storage conditions repre-

sentative of the practical exposure targeted in this study.

The chemical activator NaOH was used as an alkaline activator at a dosage of 1% of the weight of the binder. To do this, it was dissolved in the mixing water. Finally, for thermal activation, two ambient temperatures were selected (20°C and 40°C). These temperatures in the cool state were controlled using an electrically regulated enclosure. After demolding, the specimens were stored either under laboratory conditions at 20°C or in an oven maintained at 40°C , depending on the temperatures previously selected in the design.

2.4. Test methods

In order to improve the reactivity of the SCMs used, this study aims to analyze the effect of different activation processes, namely mechanical, thermal or chemical, on the evolution of mechanical performance at early-age, especially the compressive strength at 2 and 7 days. Compressive strength tests were conducted in accordance with the standard EN 196-1 (2005). For each mix and testing age, six replicate measurements were performed. The reported value was calculated as the arithmetic mean of these measurements. If one result deviated by more than $\pm 10\%$ from the mean, it was discarded, and a new average was calculated based on the remaining five values.

3. Results and Discussion

3.1. 2-day compressive strength

Fig. 5 presents the results of the compressive strength at 2 days of the control mortar C0, based on 100% OPC, as well as those of the activated and non-activated quaternary binder mortars. The main result shows that the C0 reference mortar develops a higher compressive strength at 2 days (7.5 MPa), compared to mortars with non-activated quaternary binders, whose strength does not exceed 3.5 MPa. This decrease corresponds to a reduction rate of more than 53%. In addition, all activated quaternary binder mortars show a clear improvement, both compared to the reference mortar and compared to non-activated quaternary binder mortars. This behavior highlights the beneficial effect of activation processes, regardless of the type of activation used.

Moreover, it is clear that the combined activation process « T:40 F:6000 A:1 » is the most efficient process for all the mortars studied, in comparison with the other activation processes. This improvement is often due to the acceleration of hydration reactions and the increase in microstructure by synergistic mechanisms: (i) the increased fineness of SCMs improves Interfacial Transition Zone (ITZ), reduces capillary porosity and accelerates the precipitation of hydration products; (ii) the high temperature (20–40 °C) increases the rate of hydration, resulting in a higher early-age strength; (iii) the NaOH activator (0–1 %) increases pH and increases the dissociation of the vitreous network from the slag as well as the solubility of pozzolan.

While the two activation modes « T:20 F:3800 A:0 » and « T:40 F:6000 A:1 » have fairly similar compressive strengths. This can be explained by compensation between effects such as reaction acceleration, increased porosity and the formation of early-age hydration products with lower intrinsic strength.

It is also observed that mortars C1 and C6, containing 20% and 30% of blast furnace slag respectively, develop very high compressive strengths at 2 days. This improvement is particularly marked for the two activation modes « T:40 F:3800 A:0 » and « T:40 F:6000 A:1 ». The 2-day compressive strengths of C1 and C6 under the activation mode “T:40 F:6000 A:1” reach 20 and 21 MPa, respectively, compared to 2.5 and 2 MPa for the corresponding non-activated mortars (i.e., the same formulations without activation), corresponding to increases of approximately 700% and 950%, respectively. This is attributed to the combined effect of activation processes on the hydration reaction and slag activation (Biricik and Karapinar 2021).

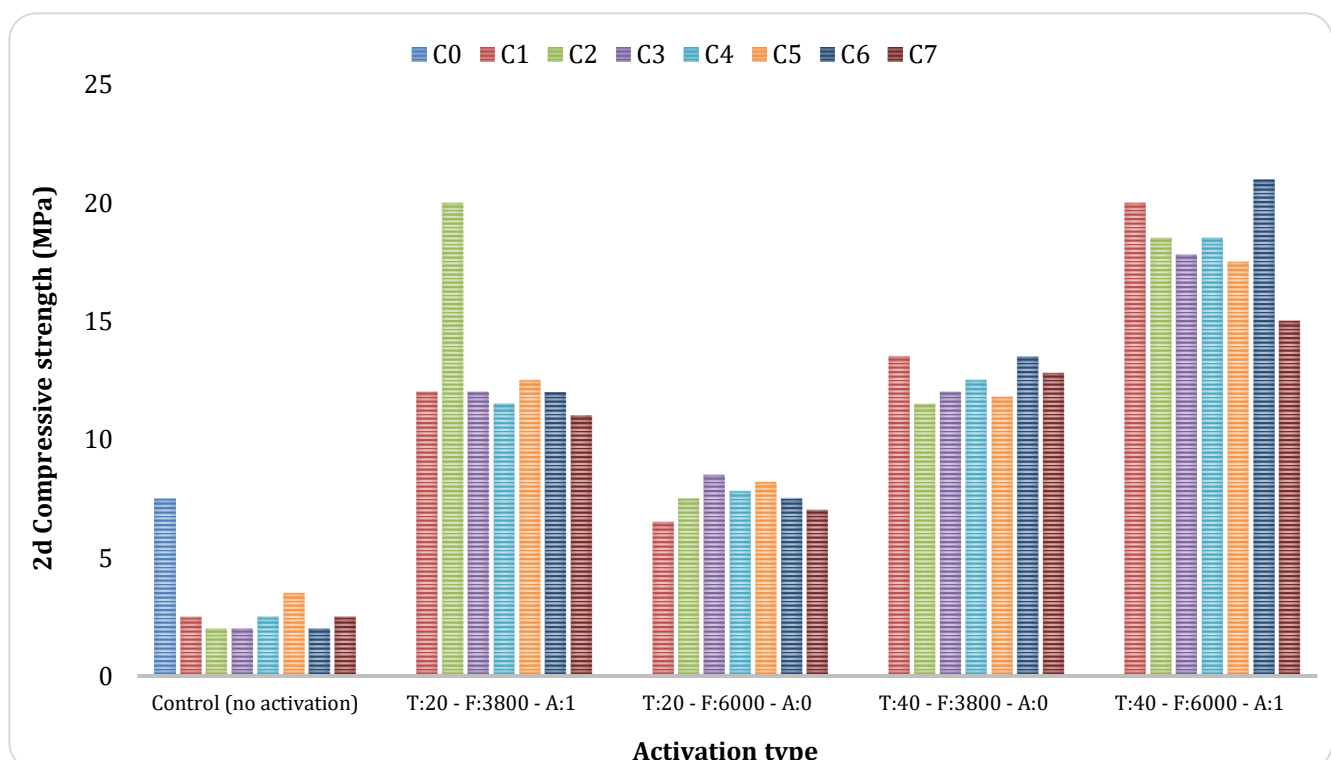


Fig. 5. Compressive strength at 2 days of activated and non-activated quaternary binder mortars.

A fractional factorial experimental design (2^{3-1}) was used to identify the SCM activation processes that exert a statistically significant effect on the 2-day compressive strength of quaternary binder mortars. This choice has reduced the experimental trials, time and costs, while ensuring high-quality results. The observed and predicted compressive strengths at 2 days of the activation

modes studied are presented in Table 5, making it possible to evaluate the effect of each variable studied.

The main factors influencing the compressive strength of 2-day quaternary binder mortars were determined by an analysis of variance (ANOVA). The values of « Prob > F » below 0.0500 indicate that the terms of the model are significant (Table 6).

Table 5. Factorial design matrix with experimental and predicted responses.

Run	Coded values of independent variables				2-day compressive strength (MPa)		7-day compressive strength (MPa)	
	Composition	T	F	A	Observed	Predicted	Observed	Predicted
1	C1	-1	1	-1	12.66	12.12	16.0	15.32
2		-1	1	1	6.72	8.25	19.0	17.66
3		1	-1	-1	13.44	13.02	19.5	19.85
4		1	-1	1	20.31	18.59	21.0	23.66
5	C2	-1	-1	1	19.84	20.00	16.0	16.50
6		-1	1	1	6.88	7.12	15.0	16.73
7		1	-1	-1	11.25	11.88	20.5	21.26
8		1	1	-1	18.28	18.50	23.0	24.50
9	C3	-1	-1	-1	12.19	11.70	19.0	19.03
10		-1	1	1	8.59	7.83	17.0	16.70
11		1	-1	1	12.50	12.60	22.0	21.23
12		1	1	-1	18.12	18.17	26.0	25.04
13	C4	-1	-1	1	11.88	11.70	19.5	19.25
14		-1	1	-1	8.12	7.83	14.0	14.83
15		1	-1	-1	12.19	12.60	18.0	17.50
16		1	1	1	18.59	18.17	24.5	25.52
17	C5	-1	-1	-1	12.66	11.63	17.5	17.50
18		-1	-1	1	8.12	7.76	16.0	16.27
19		1	1	-1	11.88	12.52	21.0	20.81
20		1	1	1	16.56	18.09	28.5	25.04
21	C6	-1	-1	-1	12.19	13.27	16.5	16.70
22		-1	1	1	7.66	7.50	15.0	14.57
23		1	-1	1	13.59	13.50	17.5	19.03
24		1	1	-1	21.09	19.73	22.0	22.91
25	C7	-1	-1	-1	10.94	10.58	16.5	16.27
26		-1	1	-1	7.81	6.71	14.5	14.57
27		-1	1	1	12.97	11.47	20.0	21.23
28		1	-1	-1	14.84	17.04	21.0	22.91

Table 6. Analysis of variance (ANOVA) for selected factorial model for 2-day compressive strength.

Source	Sum of squares	DF	Mean square	F-value	Prob>F	Status
Model	475.21331	13	36.5549	28.3743	<0.0001	Significant
Comp.	10.78033	6	1.79672	1.3946	0.2836	Not significant
T	53.04381	1	53.04381	41.1732	<0.0001	Significant
F	18.13714	1	18.13714	14.0782	0.0021	Significant
A	124.03125	1	124.03125	96.2744	<0.0001	Significant
T*F	14.22004	1	14.22004	11.0377	0.0050	Significant
T*A	7.20857	1	7.20857	5.5954	0.0330	Significant
F*A	9.71524	1	9.71524	7.5411	0.0158	Significant
T*F*A	7.03125	1	7.03125	5.4577	0.0349	Significant
Residual	18.03633	14	1.2883	R ²		0.963434
Cor. Total	493.24964	27	/	Adj. R ²		0.929479
RMSE	1.135037			Pred. R ²		0.881613
Mean	12.85357					
Coefficient of variation (%)	8.83					
Predicted residual error of sum of squares	58.39426					

The ANOVA analysis shows the developed model is highly significant, as evidenced by a high F-value of 28.37 and a p-value of less than 0.0001. The reliability of the model is confirmed by an excellent determination coefficient ($R^2 = 0.9634$), indicating that the factorial design accounts for over 96% of the response variance. Furthermore, the adjusted R^2 (0.9295) and the predicted R^2 (0.8816) are in strong agreement (difference < 0.2), demonstrating that the model possesses a robust predictive capability without overfitting the experimental data.

Regarding the specific design parameters, the main effects of variables T, F, and A are statistically significant ($p < 0.05$). Notably, all two-way interactions ($T \times F$, $T \times A$, $F \times A$) as well as the three-way interaction ($T \times F \times A$) are also significant. This highlights the presence of complex synergistic effects between these factors governing the early-age strength development of the matrix. Finally, the low coefficient of variation ($CV = 8.83\%$) underscores the high precision and reproducibility of the experimental testing procedures.

To understand the physical meaning behind these statistical interactions, it is essential to examine the synergistic mechanisms between the activation parameters. For ($T \times F$) interaction, the NaOH activator alone increases the pH to initiate the dissociation of the vitreous network of the slag, its chemical efficiency is significantly amplified when coupled with a higher temperature (40 °C). The added thermal energy helps overcome the activation energy barrier for the dissolution of the SCMs, creating a strong synergistic effect. Similarly, the interaction ($F \times A$) is physically manifested by the fact that an increased fineness (6000 cm^2/g) exposes a much larger specific surface area to the alkaline solution. This ampli-

fies the chemical attack and accelerates the precipitation of hydration products far more effectively than if the activator were applied to coarser particles.

The statistical significance of the three-way interaction ($T \times F \times A$) highlights a profound multi-physics synergy. The chemical activation provided by the alkaline solution (A) is drastically amplified by the thermal curing (T), which accelerates the reaction kinetics. However, this thermo-chemical acceleration can only translate into massive early-age strength development if there is simultaneously a highly extended specific surface area (F) available for the rapid precipitation of hydration products. This three-way interdependency confirms that the exceptional performances of the 'T:40 F:6000 A:1' mode are the result of eliminating all kinetic bottlenecks during the early stages of hydration. This profound interdependence explains the exceptional compressive strength peaks and the massive improvement rates observed in mortars with high slag content, such as C1 and C6, under the combined 'T:40 F:6000 A:1' mode (Humad et al. 2019).

The simplified mathematical model obtained is represented by Eq. (1).

$$\begin{aligned} \text{CS}_{2d} = & 13.73 + 1.83T + 0.96F + 3.65A - 1.18 T \cdot \\ & F - 0.59 T \cdot A + 1.03 F \cdot A - 1.23 T \cdot F \cdot A \end{aligned} \quad (1)$$

3.2. 7-day compressive strength

Fig. 6 illustrates the results of the compressive strength at 7 days of the reference mortar C0, as well as those of the mortars with quaternary binders, whether they are activated or not.

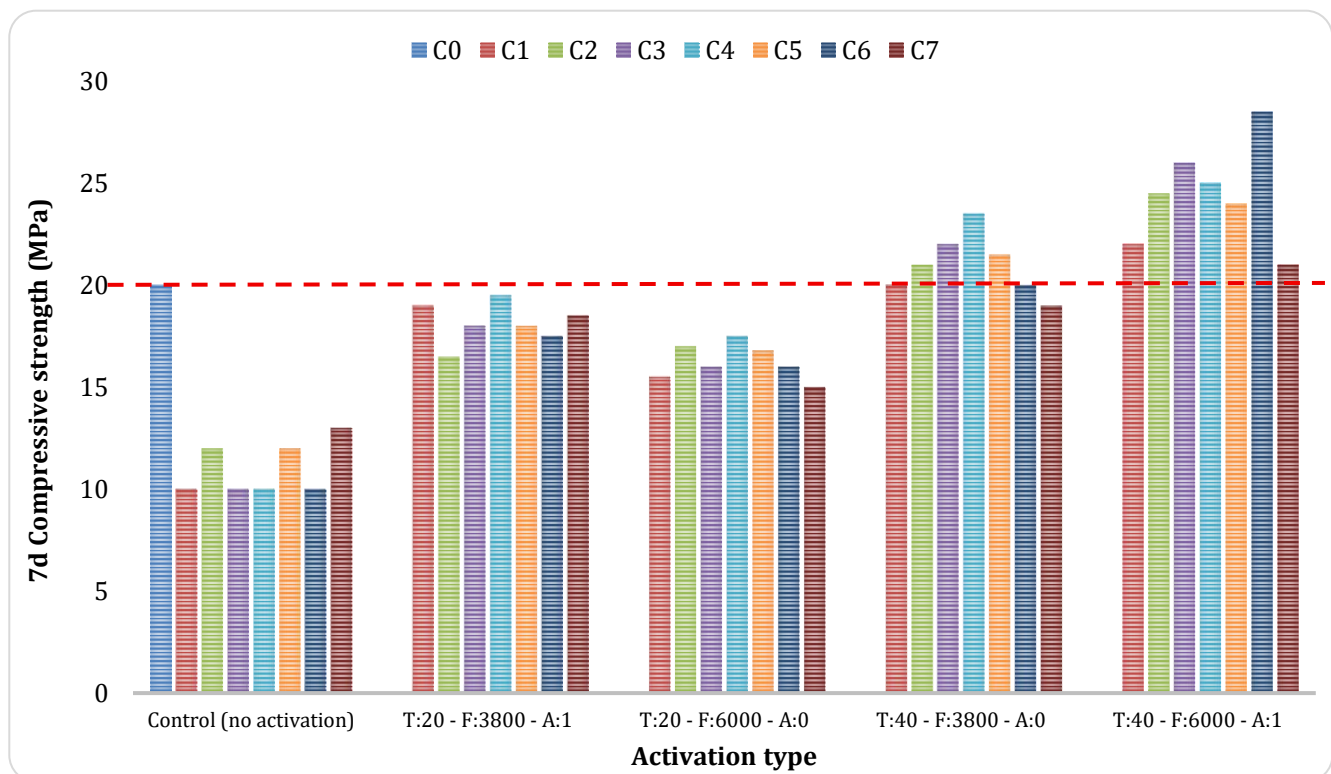


Fig. 6. Evolution of the 7-day compressive strength of activated and non-activated quaternary binder mortars.

The results of Fig. 4 clearly show that the compressive strengths at 7 days of all the non-activated quaternary mixtures are lower than that of the C0 reference mortar, which confirms that the partial replacement of the cement by non-activated SCMs leads to a remarkable decrease in the compressive strength at an early-age. On the other hand, the activation of SCMs significantly improves the compressive strength at 7 days for all the quaternary compositions studied, thus demonstrating the effectiveness of the activation processes in compensating for the initial loss of performance and optimizing the early development of the mechanical properties.

The activation of the SCMs for a temperature of 20 °C. remains insufficient for all the compositions tested, despite the increase in the fineness of the SCMs and the incorporation of 1% of NaOH. In fact, the increase in the fineness of the SCMs to 6000 cm²/g at 20 °C. improves the mechanical strength, but this improvement remains limited due to the low curing temperature.

On the other hand, the increase in the curing temperature from 20 to 40° C. markedly increases the compressive strength at 7 days of the mortars containing quaternary binders, which can be interpreted by the acceleration of the pozzolanic reactions and the accelerated and denser formation of the C-S-H gels in the system studied. It appears that the increase in temperature is a crucial factor for improving the compressive strength, thus making it possible to obtain better performance.

Furthermore, this effect is particularly marked for C3,

C4, C5 and C6, which have a particularly favorable response to the activation mechanisms, with the highest strength values. The difference in compressive strength between formulations C1 to C7, under the same activation conditions, demonstrates that the nature of the SCMs and their degree of substitution significantly influence the mechanical performance of the mortar. The results also indicate that some SCMs, especially pozzolan and slag, exhibit better reactivity to thermomechanical activation mechanisms, thus promoting the development of early-age strength.

Moreover, the combination of three activation modes leads to a notable improvement in the compressive strengths at 7 days, which exceed those of the reference mortar C0 for all the mortars with a quaternary binder. This activation method thus appears to be the most optimal mode for improving mechanical performance at an early-age (7 days), indicating the synergistic effect between the curing temperature, the high fineness and the alkaline activation.

These results thus show that the combined activation constitutes an effective strategy for compensating for the initial loss of strength of the mortars with non-activated quaternary binders.

An analysis of variance (ANOVA) also identified the main parameters influencing the compressive strength of 7-day quaternary binder mortars. Model terms with a « Prob > F » value less than 0.0500 are considered statistically significant (Table 7).

Table 7. Analysis of variance (ANOVA) for selected factorial model for 7-day compressive strength.

Source	Sum of squares	DF	Mean square	F-value	Prob>F	Status
Model	295.49890	13	22.7307	9.8861	<0.0001	Significant
Comp.	16.81033	6	2.80172	1.2185	0.3536	Not significant
T	128.97524	1	128.97524	56.0942	<0.0001	Significant
F	0.06095	1	0.06095	0.0265	0.8730	Not significant
A	5.28125	1	5.28125	2.2969	0.1519	Not significant
T*F	2.98289	1	2.98289	1.2973	0.2738	Not significant
T*A	2.57524	1	2.57524	1.1200	0.3078	Not significant
F*A	2.57524	1	2.57524	1.1200	0.3078	Not significant
T*F*A	0.03125	1	0.03125	0.0136	0.9088	Not significant
Residual	32.18967	14	2.2993	R ²		0.963434
Cor. Total	327.68857	27	/	Adj. R ²		0.929479
RMSE	1.516332			Pred. R ²		0.881613
Mean	19.95714					
Coefficient of variation (%)	7.60					
Predicted residual error of sum of squares	85.48427					

The ANOVA analysis shows the overall model remains highly significant ($p < 0.0001$), with a strong coefficient of determination ($R^2 = 0.9469$). The acceptable difference of less than 0.20 between the adjusted R^2 (0.8976) and the predicted R^2 (0.7391) confirms the model's adequate predictive capability for this curing age.

A critical observation at 7 days is the shift in the significance of the experimental factors compared to the early-age results. The main effect of factor T remains highly significant and becomes the dominant driver of

compressive strength ($p < 0.0001$). Conversely, the previously significant factors (F and A) and all of their interaction terms (T*F, T*A, F*A, and T*F*A) are statistically insignificant at this stage. This microstructural evolution suggests that while complex synergistic activation mechanisms govern initial hydration kinetics, the intermediate-term strength development of the mortar matrix is primarily controlled by parameter T. Furthermore, the reduced coefficient of variation ($CV = 7.60\%$) reflects the stabilization of the matrix and the high reliability of the crushing test protocol.

At early ages (2 days), the synergy of high fineness and chemical activation is critical to overcome the activation energy and dissolve the slag's vitreous network. By 7 days, however, the reactive surfaces are coated with hydration products and the activator is consumed (Bernal et al. 2011; Ke et al. 2019). Consequently, the initial advantages of fineness and chemical activation lose statistical dominance, and thermal curing becomes the sole driving factor, providing the necessary thermodynamic energy to accelerate ionic diffusion through the dense matrix (Bakharev et al. 1999).

After elimination of the non-significant terms ($p > 0.05$), the reduced model selected comprises the factors Comp, T and the interaction T×F. The simplified mathematical model obtained is represented by Eq. (2).

$$CS_{7d} = 19.91 + 3.07T - 0.07F + 0.81A + 0.7225T \cdot F + 0.43T \cdot A - 0.43F \cdot A + 0.06T \cdot F \cdot A \quad (2)$$

4. Conclusions

This research examines the combined effects of the three SCMs activation methods on the compressive strength of quaternary binder mortars at early-age, while proposing a statistical analysis of the main effects and their interactions. The results obtained provide a better understanding of the synergistic role of the different activation techniques, thus contributing to the optimization of the early mechanical performance of quaternary cement systems. The main results of this study are presented below:

- Partial replacement of Portland cement with non-activated SCMs leads to a significant decrease in the compressive strength of quaternary binder mortars at an early-age, due to their relatively low initial reactivity compared to cement.
- The SCM activation processes significantly improve the mechanical performance of quaternary mortars at early ages (2 and 7 days), by compensating for the early-age strength loss observed in the non-activated formulations, indicating an acceleration of hydration reactions and an improvement in the densification of the material microstructure.
- Statistical analysis (ANOVA) revealed that curing temperature is one of the most influential parameters on the compressive strength of quaternary binder mortars.
- Increasing the temperature from 20 °C to 40 °C accelerates hydration reactions and pozzolanic reactions, thus promoting faster formation of C–S–H gels and a significant improvement in mechanical strength.
- The combination of several activation processes, including the increase in the fineness of the SCMs, the increase in the curing temperature and the alkaline activation, leads to a notable improvement in the mechanical performance of the mortars studied. This combined activation generates synergistic effects that accelerate chemical reactions and improve the microstructure of the cement material.
- The composition of the binder and the degree of substitution of the SCMs strongly influence the mechanical performance. The formulations containing suitable

proportions of blast furnace slag and pozzolan exhibit a higher reactivity under activation, leading to higher compressive strengths than the other formulations studied.

- Future work should investigate the mechanical performance of these quaternary binder systems at intermediate and long cure ages (28 and 90 days) to better understand the delayed pozzolanic reactions and the long-term microstructural evolution of activated SCM binders.
- A limitation of this study is the absence of microstructural characterization. Future work will incorporate SEM and XRD analyses to directly validate the proposed mechanisms and provide deeper insight into hydration processes in quaternary binder systems.

These large amounts of experimental data can also be used to develop machine learning models capable of predicting the mechanical behavior of quaternary binder systems. Such data-driven approaches can contribute to improving the design of cementitious formulations and accelerating the development of high-performance, sustainable cementitious materials.

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No AI-based tools were used in the preparation of this manuscript.

Author Contributions

All authors made substantial contributions to the conception and design of the study, acquisition of data, analysis and interpretation of data; drafted or critically revised the manuscript for important intellectual content; and approved the final version to be published.

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