DOI
https://doi.org/10.18690/um.fkkt.1.2026.4

ISBN
978-961-299-130-2

Utilisation of Industrial Red Gypsum for Sustainable Cementitious and Geopolymer Composites

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This study investigates the potential utilisation of industrial red gypsum (RG), a by-product of titanium dioxide production, as a sustainable component in cementitious and geopolymer composites. Cement pastes, mortars and metakaolin-based geopolymers were prepared with partial replacement of the main binder by RG (5–50 wt%) to evaluate its effects on workability, porosity, hydration and mechanical performance. The experimental results showed that small additions of RG (5 %) enhanced the mechanical strength of cement mortars slightly due to its micro-filling and nucleation effects, while higher contents reduced the strength and workability because of the increased water demand and porosity. In geopolymers, RG acted mainly as an inert filler, with strength decreasing significantly beyond 10 % replacement. Response Surface Methodology (RSM) confirmed an optimal composition range at low RG dosages. The results demonstrate that limited RG incorporation can contribute to a circular economy strategy in construction, promoting waste valorisation and reducing cement consumption.

Keywords:
red gypsum,
cementitious composites,
geopolymers,
mechanical strength,
circular economy,
RSM optimisation

University of Maribor Press

1 Introduction

Cement is among the most extensively produced materials on Earth, and, by mass, is second only to water in global consumption (Environment et al., 2018). However, the production of each ton of ordinary Portland cement results in the emission of approximately one ton of carbon dioxide into the atmosphere (Hamada et al., 2021). Consequently, the scientific community has focused on developing alternative construction materials capable of replacing cement partially or fully, aiming to reduce its consumption while maintaining the overall quality and performance of concrete.

Supplementary cementitious materials (SCMs) are inorganic substances that enhance the performance of concrete. They can be classified as inert, latent hydraulic, or pozzolanic. Inert SCMs act primarily as fillers, providing nucleation sites that accelerate the hydration and hardening of cement while, simultaneously, filling voids and promoting microstructural densification in cementitious systems. Latent hydraulic SCMs, which are rich in calcium or reactive silicates, react with water to form calcium silicate hydrate (C–S–H) gels, either independently, or in combination with ordinary Portland cement (OPC) (Rashad et al., 2013; Zhang & Ye, 2012). Incorporating SCMs enhances the mechanical performance, durability and microstructural density of cement composites. Favourable results were provided by incorporating bentonite (Al-Hammood et al., 2021), biomass ash (Thomas et al., 2021), metakaolin (Kravanja & Knez, 2023), volcanic ash (Calderoni et al., 2024) and fly ash (Podnar et al., 2025). Natural SCMs showed greater potential for replacing cement than artificial ones, thereby reducing the energy demand and environmental impact (Fode et al., 2023).

Despite that, the limited research on both the short-term and long-term effects of incorporating emerging SCMs into concrete is one of the main factors preventing their widespread adoption in industrial applications (Jhatial et al., 2023).

Red gypsum (RG) is an industrial by-product generated during titanium dioxide production via the sulphate process. It is most often landfilled, which leads to environmental and spatial concerns due to the large volumes produced (Ju et al., 2023; Sotiriadis et al., 2024). However, because of its mineralogical composition, RG has attracted attention as a potential secondary raw material in sustainable construction. Previous studies demonstrated that RG can be used in cementitious and gypsum-based binders, even providing photocatalytic and self-cleaning properties (Xie et al., 2024). The valorisation of RG therefore represents an opportunity to support the principles of the circular economy by reducing industrial waste and transforming it into a valuable construction resource.

In addition to cement-based systems, geopolymer technology has emerged as a promising alternative binder system capable of achieving high mechanical strength (Castillo et al., 2021) and chemical stability (Lingyu et al., 2021) with significantly lower CO_2 emissions (Huang et al., 2023). Geopolymers are formed through the alkaline activation of aluminosilicate precursors, and their reaction mechanisms allow for the inclusion of various industrial residues, such as fly ash, slag, or red gypsum, as reactive or partial filler components. Their amorphous gel structure and dense matrix often result in enhanced durability and chemical resistance compared to traditional Portland-based binders (Das et al., 2022).

To ensure efficient material design and property optimisation, the use of Design of Experiments (DOE) has gained attention in material research. These statistical approaches allow systematic evaluation of multiple parameters—such as curing time, temperature, water-to-binder ratio and additive content—while minimising the number of experiments required (Jiju, 2023). Applying DOE to red gypsum–based cementitious and geopolymer systems can thus provide valuable insight into the interaction effects between material variables and their influence on mechanical and durability performance.

Therefore, this study aims to evaluate the potential utilisation of industrial red gypsum as a supplementary or partial replacement material in sustainable cementitious and geopolymer composites. The research focuses on understanding how the RG incorporation affects the binding behaviour, durability, porosity and mechanical properties of both material types. Furthermore, the study explores the applicability of RSM for optimising the mixture design and performance prediction. The findings are expected to contribute to the broader goal of developing low-carbon construction materials and advancing the sustainable management of industrial by-products.

2 Materials and Methods

2.1 Raw Materials

The Red gypsum (RG) used in this study was obtained from Cinkarna Celje d.d. (Slovenia), where it is produced as a by-product of TiO_2 manufacture via the sulphate process. The excess sulphuric acid is neutralised with limestone and lime milk, yielding a reddish-brown residue composed mainly of $CaSO_4 \cdot 2H_2O$, with minor Fe and Ti oxides, quartz, Mg hydroxides, and traces of heavy metals.

The as-received material, delivered as moist pressed cakes (~49 wt% moisture), was dried at 40 °C, ground and sieved (<90 μm) according to SIST EN 196-6, to ensure homogeneity and reproducibility.

For cementitious mixtures, Ordinary Portland Cement (CEM I 52.5 R, SIST EN 197-1) was used to isolate the effect of the RG on hydration and strength.

For geopolymer synthesis, the metakaolin (MK) was activated by $NaOH–Na_2SiO_3$ solutions.

2.2 Sample Preparations

The cement pastes were prepared by replacing the OPC partially with 5 %, 10 %, 15 %, and 20 % RG by mass, while maintaining the total binder content constant. The mixtures were designed and tested in accordance with SIST EN 196-3, which specifies procedures for determining the standard consistency, setting time and soundness of cement pastes.

The water content was adjusted to achieve a Vicat penetration between 5 mm and 7 mm. The RG was previously dried, ground finely (< 90 μm), and stored in sealed containers to prevent moisture re-absorption. All the samples were cured at 20 ± 1 °C and 98 % RH until testing.

The cement mortars were prepared following SIST EN 196-1, using a cement : sand mass ratio of 1 : 3 and a fixed water-to-cement ratio (w/c) = 0.5. Standard quartz sand conforming to SIST EN 196-1, Section 5.1.2 was used to ensure reproducibility.

The cement in the reference mix was replaced progressively with 5 %, 10 %, and 15 % RG by mass. The mixtures were cast into 40 × 40 × 160 mm prisms, compacted and cured under controlled conditions (20 ± 1 °C, RH > 95 %).

The workability was determined using the mini-slump test (ASTM C1437), and the porosity was measured according to SIST EN 1015-7. The compressive and flexural strengths were evaluated after 1, 7, and 28 days, as specified in SIST EN 196-1.

To analyse the influence of the RG content and curing time, the Response Surface Methodology (RSM) was applied to the experimental results.

The geopolymer mixtures were designed using a Design of Experiments (DOE) approach, to evaluate the effect of RG incorporation on the mechanical and microstructural performance. The mixtures were based on metakaolin replaced partially by RG at 0%, 10%, 25%, and 50% by mass, using the activator ratios Na_2SiO_3 : NaOH = 2 : 1(B1 mix) and 1 : 1 (B2 mix). Each mix contained NaOH solutions of 8 M, 9 M, or 10 M, with a water-to-binder ratio of approximately 0.1 and a sand-to-binder ratio of 2 : 1. Two curing regimes were applied:– ambient curing at room temperature, and accelerated curing at 60 °C for 24 h, followed by 7 days of laboratory ageing. This procedure enabled evaluation of the influence of RG on the geopolymerisation, setting behaviour and mechanical performance.

2.3 Characterisation methods

2.3.1 Determination of the Binding Properties and Durability of the Cement Pastes

The binding behaviour of cement pastes with and without red gypsum (RG) was evaluated following SIST EN 196-3. The tests included:

- standard consistency, determined using the Vicat apparatus to achieve a penetration depth of 5–7 mm,
- setting time, measured with the Vicat needle (cross-section 1 mm²), where the initial set corresponds to a penetration 3–5 mm from the mould bottom and the final set to ≤ 1 mm,

- soundness (dimensional stability), assessed using the Le Chatelier method, where the expansion of the specimen after boiling in water was measured to ensure stability (< 10 mm).

All the pastes were prepared at 20 ± 1 °C and 98 % relative humidity and tested immediately after curing.

2.3.2 FTIR–ATR Analysis of the Cement Pastes

The Fourier Transform Infrared Spectroscopy with Attenuated Total Reflectance (FTIR–ATR) technique was applied to identify functional groups and reaction products in the hydrated cement pastes.

Spectra were collected in the 4000–600 cm^{-1} range using a diamond ATR crystal. The characteristic peaks were interpreted as follows:

- Broad bands near 3400 cm^{-1} → -OH stretching from the bound water;
- Signals between 2000–2400 cm^{-1} → CO_2 adsorption and carbonate formation;
- Peaks around 1400 cm^{-1} → carbonate groups (CO_3^{2-}) associated with $CaCO_3$;
- Absorptions near 1000 cm^{-1} → sulphate groups (SO_4^{2-}) from the gypsum;
- Features near 700–850 cm^{-1} → mineral phases common to all hydrated systems.

The analysis enabled qualitative comparison of the hydration and carbonation processes in pastes containing varying RG contents.

2.3.3 Mortar Characterisation (Workability, Porosity, Strength)

The workability of fresh mortars was determined using the mini-slump test (ASTM C1437), where the average spread diameter (in mm) indicates the flowability.

The porosity was measured according to SIST EN 1015-7, by determining the air content of fresh mortar using a 1 L calibrated pressure vessel.

The mechanical testing followed SIST EN 196-1. Mortar prisms (40 × 40 × 160 mm) were cured for 1, 7, and 28 days, and tested for flexural and compressive strength using a standard testing machine.

The results were calculated according to Equations (1-2):

$$\sigma_f = \frac{3\,FL}{2b^3} \tag{1}$$

$$\sigma_c = \frac{F}{A} \tag{2}$$

where σ_f is the flexural strength (MPa), F is the maximum load (N), L is the span length (mm), b is the side length of the square cross-section (mm), and σ_c is the compressive strength (MPa), with A as the loaded area (mm^2). Both tests were performed for cement-based and geopolymer mortars, to ensure comparability of strength development and durability.

2.4.4 Application of the RSM Approach

Response Surface Methodology (RSM) was employed to analyse and optimise the influence of the key parameters.

A quadratic polynomial model was fitted to the experimental data, to evaluate the combined and interactive effects of the independent variables.

For cement mortars, RSM was used to model the relationship between the RG content, porosity, workability and compressive strength.

The RSM approach allowed efficient visualisation of nonlinear trends and identification of the optimum mixture compositions while minimising the number of experimental runs.

3 Results and discussion

3.1 Cement pastes with red gypsum

The influence of red gypsum (RG) on the binding behaviour and stability of cement pastes was examined systematically through standard consistency, setting time, soundness and FTIR–ATR analyses. Ordinary Portland cement (CEM I 52.5 R) was replaced partially by 5 %, 10 %, 15 %, and 20 % RG by mass, while a reference mix without RG served as the control.

The results showed that the incorporation of RG increased the water demand to reach standard consistency, with the reference mixture requiring 122 g of water and the 15 % and 20 % RG pastes requiring 149 g and 158 g, respectively. This higher water requirement is attributed to the fine particle size and high surface area of the RG, which enhances water absorption and reduces the fluidity of the paste.

Table 1 summarises the effect of the red gypsum (RG) addition on the setting time of the cement pastes. The data show that a small RG content (5 %) delayed both the initial and final set slightly compared to the reference mixture, while the higher RG contents (15–20 %) accelerated setting significantly. This indicates a transition from a mild retardation effect at low dosages to a pronounced acceleration of hydration at higher sulphate concentrations.

Table 1: Initial and final setting time in hours.

	REF	RS-5%	RS-10%
Initial setting time	2:00	2:30	2:00
Final setting time	2:35	3:20	2:45

The Le Chatelier test confirmed excellent dimensional stability across all the mixtures, with expansions between 0 and 1.5 mm, well below the permissible 10 mm limit, indicating that RG does not induce volumetric instability, and may even contribute to matrix densification.

The FTIR–ATR spectra supported these findings further, revealing broad -OH stretching bands near 3400 cm^{-1} that intensified with the RG content due to the increased bound water, as well as carbonate-related peaks around 1400 cm^{-1} and sulphate-associated absorptions near 1000 cm^{-1}, confirming the coexistence of carbonate and sulphate phases in the hydration matrix.

Overall, the partial replacement of cement with up to 15 % RG maintained suitable workability and stability, modifying the hydration kinetics slightly, while promoting a denser microstructure.

3.2 Cement mortars with red gypsum

The performance of cement mortars containing red gypsum (RG) was assessed in terms of workability, porosity and mechanical properties. The mortars were prepared with 5 %, 10 %, and 15 % replacement of cement by RG at a constant water-to-binder ratio (w/c = 0.5).

3.2.1 Workability and porosity

The results demonstrated that the incorporation of RG reduced the workability of fresh mortars progressively, as indicated by the mini-slump spread decreasing from 165 mm for the reference mixture to 157.5 mm, 117.5 mm, and 107.5 mm for 5 %, 10 %, and 15 % RG, respectively. The reduced flowability is attributed primarily to the increased water demand and higher surface area of the RG particles, which lower the effective water available for lubrication. The porosity measurements revealed a similar trend, with a slight reduction at 5 % RG (2.5 %) compared to the reference (3.0 %), followed by an increase to 3.5 % and 4.5 % at 10 % and 15 % RG. This suggests that small amounts of RG can improve packing density, while higher levels introduce additional voids due to the altered rheology and air entrainment.

3.2.2 Mechanical properties

The compressive and flexural strength of cement mortars with red gypsum (RG) after 1, 7 and 28 days are shown in Figures 1 and 2. A small RG addition (5 %) enhanced both strengths slightly, while higher contents reduced them. After one day, the reference mix reached 30.57 MPa and the 5 % RG sample achieved a slightly higher value (33.59 MPa) due to accelerated early hydration. At 7 days, the 5 % RG mortar attained 52.92 MPa, exceeding the reference (48.96 MPa), whereas 10 % and 15 % RG reduced the strength to 42.34 MPa and 34.27 MPa. After 28 days, the 5 %

RG mixture maintained the best performance (61.56 MPa) compared to the reference (58.70 MPa), while the 10 % and 15 % RG dropped to 47.66 MPa and 38.75 MPa. The flexural strength followed the same pattern: 7.73 MPa for 5 % RG versus 7.5 MPa for the control, decreasing to 6.5 MPa and 5 MPa for 10 % and 15 % RG. Overall, a 5 % replacement level provides a beneficial micro-filling and nucleation effect, whereas higher RG contents cause dilution, greater porosity and loss of strength.

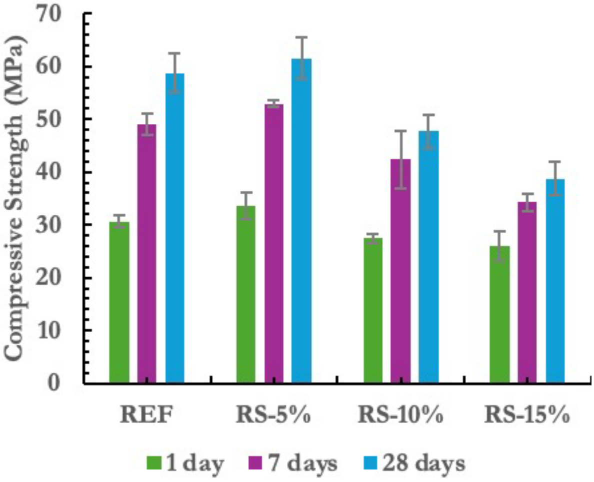

Figure 1: Compressive strength of cement composites after 1, 7, and 28 days.

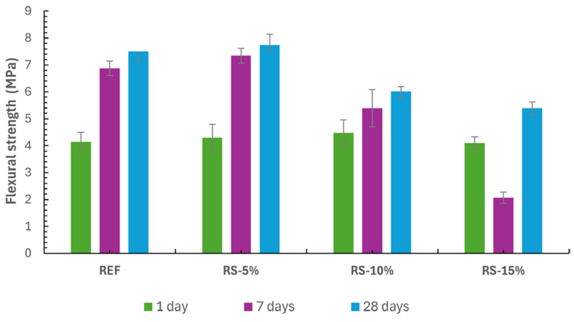

Figure 2: Flexural strength of cement composites after 1, 7, and 28 days.

The influence of red gypsum (RG) content and curing time on the mechanical behaviour of cement mortars was analysed further using Response Surface Methodology (RSM), as illustrated in Figures 3 and 4.

The 3D surface and contour plots show the nonlinear relationship between the RG percentage, curing duration, and the resulting compressive and flexural strengths. The models revealed that both properties initially increased with small additions of RG, reaching optimal values at approximately 5 %, followed by a gradual decline at higher replacement levels. This trend indicates a beneficial micro-filling and nucleation effect at low RG contents, enhancing early hydration and matrix densification, while excessive RG introduces dilution and increased porosity that weaken the composite structure. The curvature of the response surfaces also confirms that the development of mechanical strength is strongly time-dependent, with more pronounced differences observed after extended curing periods. The RSM analysis thus captured the combined and interactive effects of RG dosage and curing time effectively, allowing identification of an optimal composition range for achieving the best mechanical performance while maintaining mix stability.

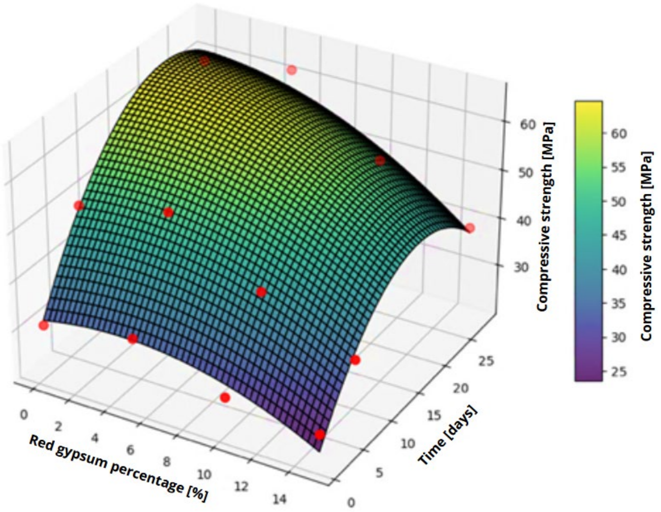

Figure 3: RSM visual representation of the effects of red gypsum content (%) and curing time (days) on compressive strength (MPa).

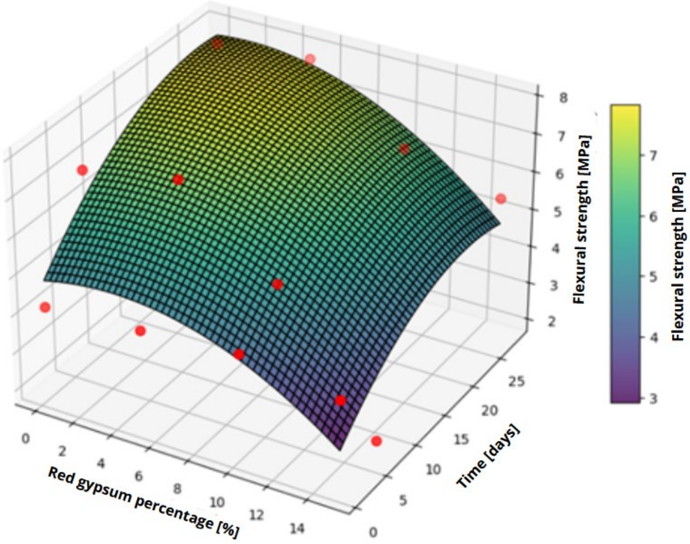

Figure 4: RSM visual representation of the effects of red gypsum content (%) and curing time (days) on flexural strength (MPa).

3.3 Geopolymers with red gypsum

Red gypsum (RG) was also evaluated as a partial replacement for metakaolin in geopolymer composites, to assess its effect on the workability and mechanical performance. As RG contains mainly calcium sulphate with limited aluminosilicate reactivity, its incorporation can influence geopolymerisation and matrix formation. This section examines how varying the RG contents (0–50 %) and activator ratios (Na_2SiO_3 : NaOH = 2 : 1 and 1 : 1) affect the fresh and hardened properties of geopolymer mixtures.

3.3.1 Workability and Porosity

All the geopolymer mixtures were designed using metakaolin (MK) as the main aluminosilicate precursor, replaced partially by 0–50 % of red gypsum (RG). High-alkali activating solutions composed of sodium silicate (Na_2SiO_3) and sodium hydroxide (NaOH) were used at ratios of 2 : 1 and 1 : 1. During mixing, it was observed that small additions of RG (up to 10 %) did not alter the consistency significantly, and the mixtures retained satisfactory workability suitable for moulding. However, as the RG content increased to 25 % and 50 %, the mixtures became noticeably stiffer, less cohesive and more difficult to compact, even with the increased liquid content. This was attributed to the inert nature of RG, its high sulphate content and limited aluminosilicate reactivity, which hindered the geopolymer gel formation and reduced binder cohesion. Additionally, higher RG levels led to a more open and porous structure, as confirmed by the visual and mechanical observations, indicating that excessive RG disrupts the packing and continuity of the geopolymer matrix.

3.3.2 Mechanical Properties

The compressive and flexural strengths of metakaolin-based geopolymers incorporating red gypsum (RG) are shown in Figures 5 and 6. The results revealed a pronounced decrease in both strengths with increasing RG replacement. After 7 days of curing at 60 °C, the reference mixture without RG reached a compressive strength of 20.63 MPa, while the 10 % RG sample achieved 13.49 MPa, the 25 % RG sample dropped to 2.66 MPa, and the 50 % RG mixture showed almost no load-bearing capacity (0.01 MPa). A similar trend was observed for the second activator ratio, where the compressive strength values were 21.09 MPa (0 % RG), 19.01 MPa (10 % RG), 11.25 MPa (25 % RG), and 0.95 MPa (50 % RG).

The flexural strength results followed the same pattern, decreasing sharply with the higher RG contents. For the Na_2SiO_3 : NaOH = 2 : 1, (60 °C) system (B1 mixes), the flexural strength declined from 6.00 MPa (0 % RG) to 4.75 MPa (10 %), 2.80 MPa (25 %), and 0.60 MPa (50 %). In the Na_2SiO_3 : NaOH = 1 : 1 (B2 mixes), the values were slightly higher at each corresponding RG level: 6.00 MPa, 5.10 MPa, 3.00 MPa, and 1.00 MPa, respectively.

These findings indicate that small RG additions (≈ 10 %) may still act as an inert filler, allowing acceptable mechanical performance, while higher replacement levels disrupt the geopolymerisation process significantly. The decline in strength at ≥ 25 % RG confirms that the limited aluminosilicate reactivity and high sulphate content of RG hinder gel formation and lead to a porous, poorly bonded microstructure.

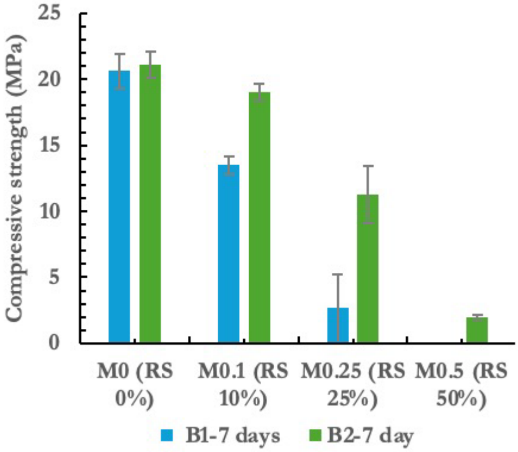

Figure 5: Compressive strength of geopolymer samples with metakaolin replaced partially by RG from 0% to 50%.

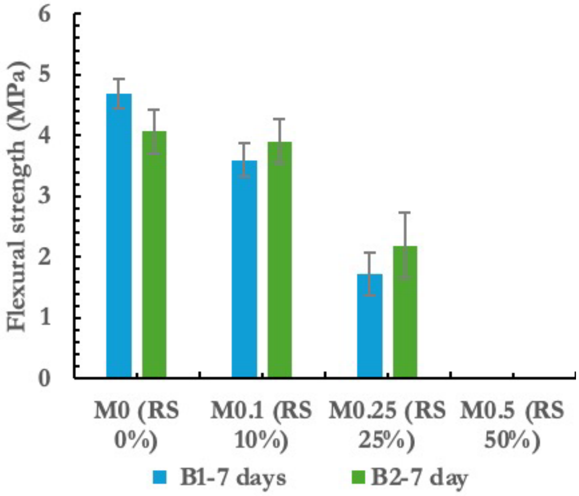

Figure 6: Flexural strength of geopolymer samples with metakaolin replaced partially by RG from 0% to 50%.

4 Conclusion

This research confirmed that industrial red gypsum (RG) can be valorised effectively as a secondary material in cement-based systems, with partial potential in geopolymer formulations. In cement pastes and mortars, RG additions up to about

5 % improved the matrix density and mechanical performance through filler and nucleation effects, while maintaining good dimensional stability. Higher RG contents increased the porosity and water demand, resulting in reduced strength and workability. The RSM analysis verified further that the optimum balance between compressive and flexural strength occurs at low RG replacement levels and longer curing times.

In geopolymer composites, the incorporation of RG limited geopolymerisation due to its low aluminosilicate reactivity, causing a rapid strength decline beyond 10 % replacement. Despite this, minor RG additions may serve as inert fillers or stabilisers within the alkali-activated matrix.

Overall, the study demonstrates that controlled use of red gypsum—particularly in low concentrations within cementitious systems—can support sustainable construction practices by reducing cement consumption, minimising industrial waste disposal, and advancing circular material use in the building sector. Nevertheless, the leaching behaviour of RG should be evaluated thoroughly prior to practical implementation, to ensure environmental safety and compliance with the regulatory Standards.

Acknowledgment

The authors acknowledge Cinkarna Celje d.d. for funding this industrial research project fully. The first author is a member of the research programme P2-0046, supported by the Slovenian Research and Innovation Agency (ARIS).

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