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Article

Calcium Effect in PLR–PCR Geopolymers: Peak Compressive Strength at 30% PCR and Evidence of C-A-S-H/N-A-S-H Synergy

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Abstract

Valorization of construction and demolition waste (CDW) through alkali activation enables the formulation of geopolymers with a lower carbon footprint. This study evaluates binary pastes prepared with powdered recycled concrete (PCR) and powdered recycled brick (PLR) in seven proportions (0–100% PCR), activated with NaOH/Na₂SiO₃/KOH and cured for 7, 14, and 28 days. Compressive strength, microstructure (SEM-EDS), and chemical structure (XRD, FTIR) were characterized. Mixtures with 30% PCR reached the highest 28-day strength, associated with a denser microstructure and lower porosity. EDS analysis evidenced an increase in Ca with increasing PCR, promoting C-A-S-H gels coexisting with N-A-S-H; XRD showed a reduction of non-reactive crystalline phases, and FTIR an intensification of T–O–Si bands, both consistent with higher polymerization. Higher PCR contents diluted reactive aluminosilicates and reintroduced microdefects, leading to a drop in strength. Altogether, an optimal window at ~30% PCR is identified to maximize densification and mechanical performance without compromising the aluminosilicate network. These results support the combined use of PCR/PLR as geopolymer precursors to manage CDW and produce sustainable construction materials.

Keywords: geopolymers; construction and demolition waste (CDW); recycled concrete powder (PCR); recycled brick powder (PLR); alkali activation; microstructure; compressive strength

1. Introduction

The construction industry is seeking innovative, sustainable solutions in response to rising activity and mounting pressure to manage waste efficiently [1]. Construction and demolition waste (CDW) originates from debris and poor practices (deficient planning, inadequate handling) and, owing to its chemical nature, is not amenable to aerobic/anaerobic digestion, composting, or chemical degradation; it often ends up in informal dumps, creating environmental liabilities. In 2018 the global average was estimated at 604 kg·capita^{−1}·year^{−1}, with a projected +60% increase by 2050 [2–4]. These volumes exacerbate soil and water pollution and contribute to climate change through GHG emissions [2,3].

In Peru, construction is a major waste source; Lima reported 3,881,000 t of municipal waste in 2020 (+7.4% vs. 2019), and in La Libertad building activity grew 3.8% per year (2013–2022), reflecting expansion driven by self-construction and housing/infrastructure demand [5,6]. This context increases CDW and strains management capacity; hence recycling and valorization strategies that integrate CDW into material value chains are required [7]. The Peruvian legal framework (Law No. 1278 and its regulations; PLANRES; municipal ordinances) promotes minimization, segregation, material/energy recovery, traceability, and extended producer responsibility (EPR), establishing the basis for safely and standardly integrating CDW into construction materials [8–10].

Leveraging CDW in materials reduces landfill demand and extraction of non-renewable resources (the sector consumes ≈ 3 billion tonnes of raw materials per year), enabling circular-economy pathways [11,12]. The literature proposes supplementary cementitious materials (silica fume, fly ash, quicklime, ceramic wastes, palm-oil by-products), with effects on strength, durability, and waste-management cost [4]. In La Libertad (2017 census), 58.8% of dwellings use brick/concrete block, making PLR and PCR relevant fractions in the CDW stream [13].

Within CDW, recycled brick powder (PLR)—rich in Si–Al—has been used as a cement replacement, with mechanical improvements around $\approx 15\%$ substitution; some studies report no significant losses with 15% PLR and 30% recycled aggregates [14–16]. Recycled concrete powder (PCR)—rich in Ca—is the most abundant residue globally, but its structural reuse is limited by abrasion, bulk density, and absorption, restricting it to non-structural applications [17,18].

Geopolymers emerge as a lower-carbon alternative to Portland cement: alkali activation of aluminosilicates forms N-A-S-H networks and, in the presence of Ca, C-A-S-H domains, achieving competitive mechanical and durability performance [19–21]. Activation of Si–Al–Ca-rich materials (e.g., ceramic–cementitious CDW) shows favorable results at $T \geq 60^\circ\text{C}$ and low L/B ratios, consistent with dissolution $\rightarrow$ condensation $\rightarrow$ gelation/crystallization (Si–O–Al linkages) [22–26]. Activator chemistry is critical: $\text{Na}_2\text{SiO}_3/\text{NaOH}$ (and solid variants Na_2CO_3 , $\text{Na}_2\text{SiO}_3 \cdot 5\text{H}_2\text{O}$) allows tuning workability, microstructure, and compressive strength (fc); the silicate modulus ($\text{SiO}_2/\text{Na}_2\text{O}$), NaOH molarity, and the use of KOH modulate strength and network densification [27–36].

Evidence with PLR and ceramic tiles (BCR) shows that curing at $80\text{--}90^\circ\text{C}$ with 8–14 M NaOH yields 40–60 MPa, with clear influences of fineness and porosity of the precursor [37,38]. For PCR, single-precursor systems typically underperform ($<10\text{--}27$ MPa) unless co-activated with metakaolin or clinker and cured at 70°C ; typical limitations are a low $\text{SiO}_2/(\text{Al}_2\text{O}_3 + \text{CaO})$ ratio and secondary carbonation [38,39]. Various binary PLR+PCR studies report synergies: 20–50% PCR densifies the matrix and improves fc, whereas excess PCR reduces reactive Si–Al species; optima around $\approx 10\text{--}20\%$ PCR have been observed (lot/activator/curing dependent) [40–42].

Despite advances, the literature shows two cross-cutting limitations: (i) inconsistent process variables (activator, L/S, curing) hinder inter-study comparisons and quantification of the pure effect of %PCR; (ii) scale decoupling: many mechanical results lack the multi-technique support (XRD/FTIR/SEM-EDS) needed to build convergent mechanistic arguments. Therefore, this article proposes to assess whether an optimal PCR fraction exists that maximizes compressive strength by enabling C-A-S-H/N-A-S-H synergy, and whether this can be demonstrated using independent tracers (FWHM in XRD, Δv in FTIR, and Ca (at.%) in SEM-EDS) under constant activator and curing conditions, thereby validating the role of Ca and gel coexistence reported for CDW systems [14–18,25–34].

Accordingly, the general objective is to evaluate the effect of PCR/PLR proportion on the microstructure and compressive strength of geopolymer pastes, employing constant activator and curing, and triangulating XRD, FTIR, and SEM-EDS. We expect to demonstrate that an intermediate %PCR (with moderate Ca) maximizes network densification through C-A-S-H/N-A-S-H coexistence, translating into a peak compressive strength, as reported in [19–44].

The contributions of this research are: isolation of the net effect of %PCR under constant activator and curing (uncommon in the CDW literature); multi-technique triangulation (fc–XRD–FTIR–SEM-EDS) to build a convergent argument on the Ca effect; a reproducible methodology (cylinder geometry, loading rates, normalized metrics) that facilitates transfer to other CDW families; and mapping of an optimal design window as a function of %PCR, supporting scale-up and quality-control roadmaps under feedstock variability [1–5,35–45].

2. Materials and Methods

2.1. Materials

Recycled concrete powder (PCR/RCP) and recycled brick powder (PLR/RBP) were collected from construction and demolition waste (CDW) generated in Trujillo, Peru. Both were used as aluminosilicate precursors in alkali-activated geopolymer pastes.

Activator. Sodium hydroxide (NaOH) flakes/pellets, ≥98 wt%, ACS (Macron), and potassium hydroxide (KOH) pellets, ≥90 wt%, ACS (Macron), were used. Sodium silicate (Na₂SiO₃) was a commercial solution with silica modulus (M_s = SiO₂/Na₂O) = 3.2, water as balance (reference density ~1.38 g mL⁻¹ at 20 °C). Deionized water was used to prepare the activator.

2.2. Activator Preparation

The ternary activator was formulated at a mass ratio of 0.10 NaOH : 0.60 Na₂SiO₃ : 0.30 KOH. For preparation, the hydroxides were dissolved separately in deionized water, allowed to cool to 23–25 °C, and then the Na₂SiO₃ solution was added under stirring to reach the target volume. Prior to mixing with the precursors, the activator was homogenized (5 min) and rested (≥15 min) to stabilize temperature and degassing. We report the formulation in mass fractions and the Na₂SiO₃ modulus (M_s = 3.2) as a sufficient chemical specification to reproduce the activator chemistry without resorting to molarities.

2.3. Mix Design and Paste Preparation

Seven formulations were produced by replacing PLR with PCR at 0, 10, 30, 50, 70, 90, and 100 wt% of the precursor (PLR provides the complementary fraction to 100% in each mix). The liquid-to-binder ratio (L/B) was fixed at 0.15 for all compositions; details are shown in Table 2.

Powder conditioning. PCR and PLR were dried at 105 °C for 24 h, ground, and sieved to <37 μm (400 mesh) to minimize granulometry effects on early reactivity. Table 1 reports the chemical composition of the raw materials.

2.4. Mixing Procedure

The dry powders (PCR, PLR) were pre-mixed for 2 min to homogenize. The activator was then added and mixed for 3 min at low speed in a Hobart paste mixer. Pastes were cast into metal cylinders, 25 mm diameter × 50 mm height, using compression molding (500 psi). Demolding was performed at 24 h. Curing was carried out in an oven at 40 °C for 72 h; specimens were then cooled and stored at room temperature until the testing ages (7, 14, and 28 days). Table 2 lists the mixes produced.

Table 1. Chemical characterization (oxides) of PCR and PLR.

PCR		PLR	
Óxido	wt%	Óxido	wt%
SiO ₂	49.16	SiO ₂	53.45
CaO	24.11	Al ₂ O ₃	20.52
Al ₂ O ₃	10.85	Fe ₂ O ₃	7.80
Fe ₂ O ₃	4.93	K ₂ O	2.63
MgO	2.86	MgO	1.85
K ₂ O	2.08	CaO	1.62
Na ₂ O	1.55	Na ₂ O	1.50
SO ₃	2.87		

TiO ₂	0.77
P ₂ O ₅	0.53
MnO	0.30

Table 2. Binary PCR/PLR Mix Matrix (L/B = 0.15).

Mixture	PRC (%)	PRB(%)	NaOH (%)	Na ₂ SiO ₃ (%)	KOH (%)	l/b
P ₁ PRC ₀	0	100	10	60	30	0.15
P ₂ PRC ₁₀	10	90	10	60	30	0.15
P ₃ PRC ₃₀	30	70	10	60	30	0.15
P ₄ PRC ₅₀	50	50	10	60	30	0.15
P ₅ PRC ₇₀	70	30	10	60	30	0.15
P ₆ PRC ₉₀	90	10	10	60	30	0.15
P ₇ PRC ₁₀₀	100	0	10	60	30	0.15

2.5. Compressive Strength Test

Compressive strength was measured on a Tecnotest Modena universal testing machine (F060/EV) with parallel platens and verified alignment. Tests were run under displacement control at 1.0 mm·min⁻¹, with n = 5 specimens per age (7, 14, and 28 days). Strength is reported as mean ± SD.

2.6. X-ray Diffraction (XRD)

Phase identification was performed on a Bruker D8 Advance powder diffractometer using Cu K α radiation (λ = 1.5406 Å, 40 kV, 40 mA), θ –2 θ mode. The scan covered 5–80° 2 θ , step 0.020°, count time 1.0 s/step, with sample spinner enabled. Finely ground powders were mounted on low-background (Si-zero) holders. A Rietveld-type (semiquantitative) refinement was applied to estimate residual amorphous gels.

2.7. Fourier-Transform Infrared Spectroscopy (FTIR)

Spectra were acquired in ATR mode (Thermo Fisher Scientific iS50, diamond crystal) over 4000–400 cm⁻¹, 4 cm⁻¹ resolution, 32 scans per spectrum, with background correction prior to each measurement. Samples were dried at 60 °C for 24 h before analysis to limit physically adsorbed water.

2.8. Scanning Electron Microscopy and EDS (SEM-EDS)

Microstructure and local chemistry were examined on a Thermo Scientific Axia ChemiSEM (real-time EDS integration). Imaging was performed in high vacuum using SE (topography) and BSE (compositional contrast) detectors, with typical operating conditions of 5 kV (SE) and 15 kV (BSE), working distance 9–12 mm. At least five fields of view per sample (representative areas) were recorded, with magnification and scale reported on each micrograph.

2.9. Statistical Treatment and Quality Control

Results are reported as mean ± standard deviation (n = 5 per age). Normality was checked with Shapiro–Wilk; when applicable, one-way ANOVA was used (α = 0.05). Instrument QA/QC: XRD was verified with Si standard (NIST SRM 640d); FTIR with a polystyrene film (reference bands check); SEM-EDS with internal/commercial standards (gain adjustment and drift correction). Figure 1 shows the experimental workflow.

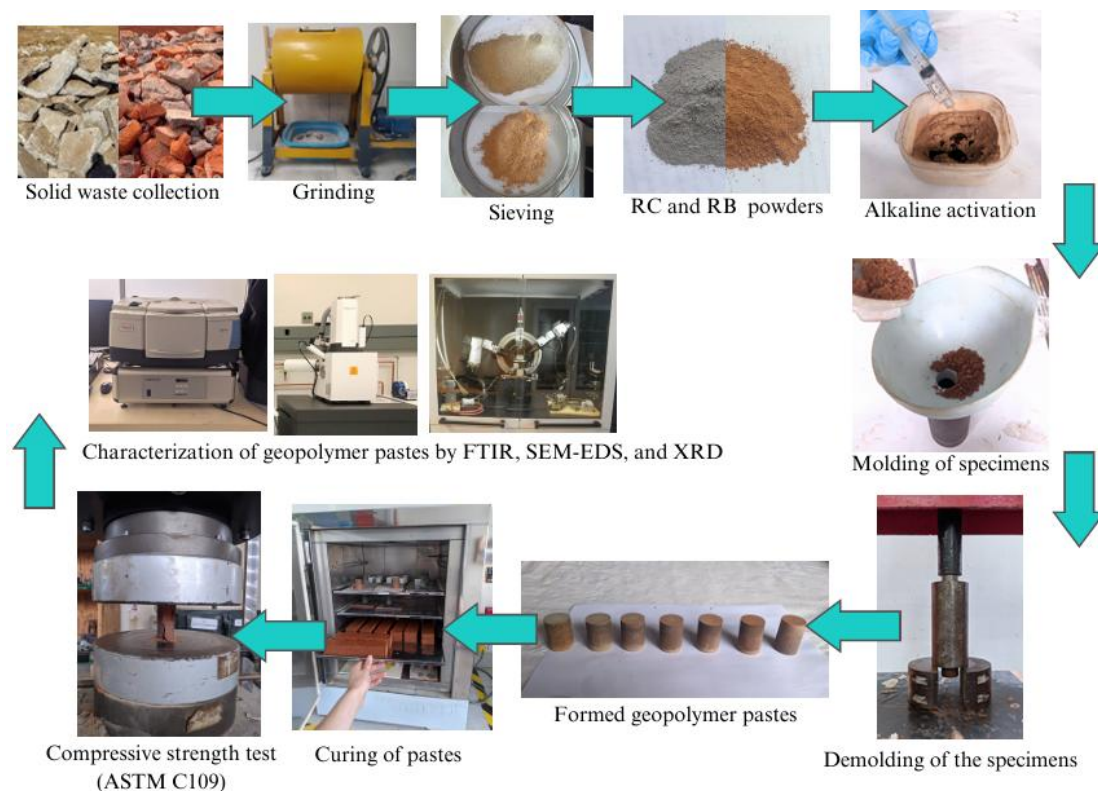


Figure 1. Experimental procedure developed.

3. Results

3.1. Compression Resistance

Figure 2 shows the f_c of PLR–PCR pastes at 7, 14, and 28 days for PCR contents between 0–100%. A systematic increase in f_c was observed between 0–30% PCR, followed by a loss for substitutions $\geq 50\%$. The 0% PCR (100% PLR) mix reached 13.2, 13.9, and 14.2 MPa (7, 14, and 28 d). With 10% PCR, values were 13.5, 14.3, and 15.6 MPa (+2.27%, +2.88%, and +9.86% vs. 0% PCR). The local maximum occurred at 30% PCR: 14.4, 14.7, and 16.2 MPa (+9.09%, +5.76%, and +14.08%). At 50% PCR, f_c dropped to 13.5, 13.55, and 13.8 MPa (–2.27%, –2.52%, and –2.82%), with larger decreases at 70% PCR (9.5, 9.7, and 10.8 MPa; –28.03%, –30.21%, and –23.94%) and 100% PCR (7.6, 8.2, and 10.1 MPa; –42.00%, –41.01%, and –28.87%). Error bars (SD) reflect the dispersion typical of alkali-activated systems.

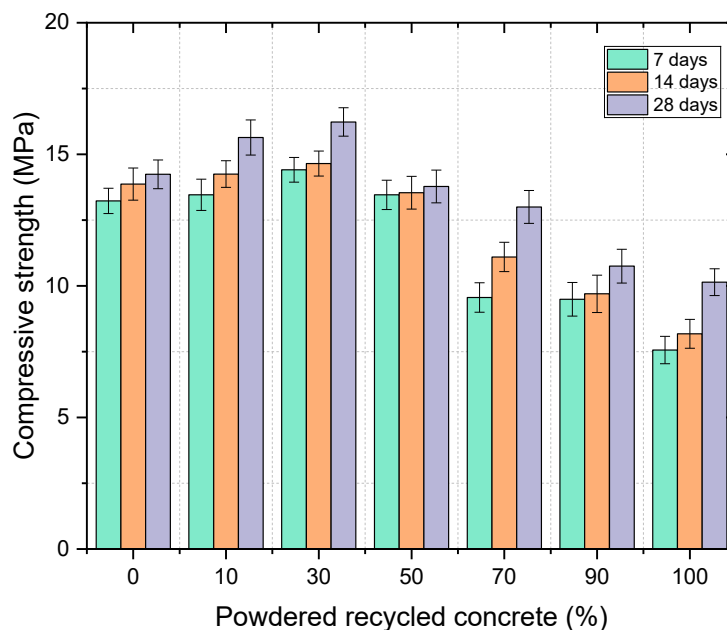


Figure 2. Compressive strength values.

3.2. X-ray Diffraction (XRD): Compositional Effect

Figure 3 compares 28-day XRD patterns for 0, 10, 30, and 100% PCR. 0% PCR (pure PLR): broad set of crystalline phases (kaolinite, quartz, albite, belite, calcite, octahedrite, gehlenite, anorthite), with quartz dominant. 10–30% PCR: portlandite ($\sim 36.6^\circ 2\theta$) appears (indicating hydration of residual clinker from PCR); the variety of PLR phases decreases while quartz ($\sim 26.7^\circ 2\theta$) remains. At 30% PCR the relative intensity of albite decreases, consistent with aluminosilicate consumption and a larger amorphous fraction associated with reaction gels. 100% PCR: simplified pattern dominated by quartz and portlandite, with lower crystalline diversity, indicating the preeminence of Ca-rich products and reduced aluminosilicate contribution.

3.3. XRD at Curing Ages

Figure 4 (30% PCR, 7 vs. 28 d) shows stable peak positions (quartz, albite, portlandite, belite, calcite) with a slight decrease in portlandite/belite intensities at 28 d, consistent with reaction progress and reorganization toward amorphous phases of the geopolymer network.

3.4. SEM-EDS: Microstructure and Local Chemistry

Figure 5 compares 28-day microstructures. 0% PCR: noticeable porosity and microcracks; unreacted particles present. EDS dominated by Si–Al (N–A–S–H matrix) with low Ca. 10% PCR: greater compaction with less perceptible microcracks; EDS shows higher Si and a slight rise in Ca, consistent with PCR addition. 30% PCR: denser matrix, no visible porosity/microcracks, and extensive amorphous regions; EDS evidences higher Ca (vs. 0–10%), indicative of C–A–S–H gels co-existing with N–A–S–H. 100% PCR: microdefects reappear (pores/microcracks) despite locally compact zones; EDS with higher Ca and lower relative Al–Si, suggesting heterogeneity and incomplete consolidation of a continuous aluminosilicate network.

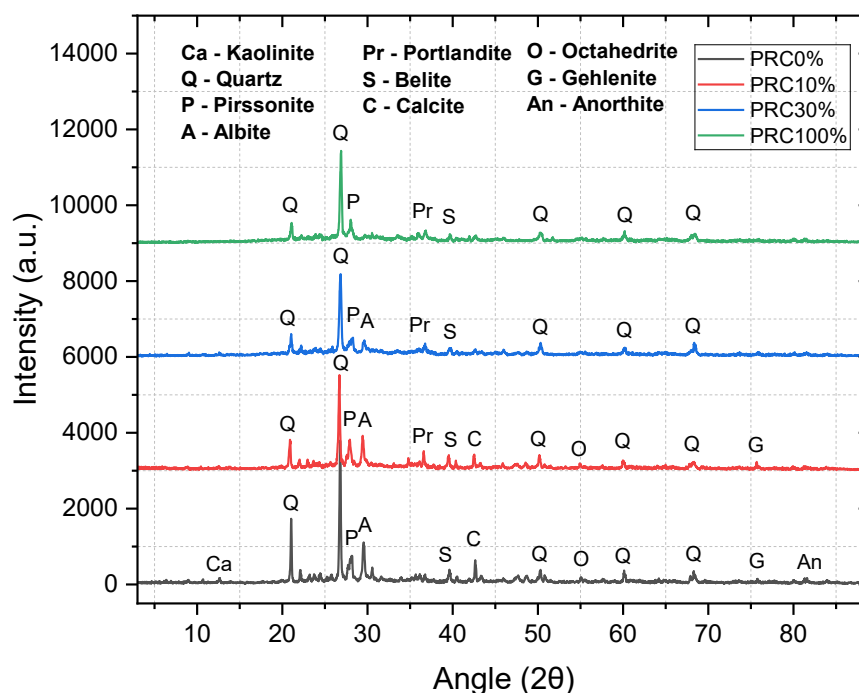


Figure 3. XRD of pastes with different compositions at 28 days.

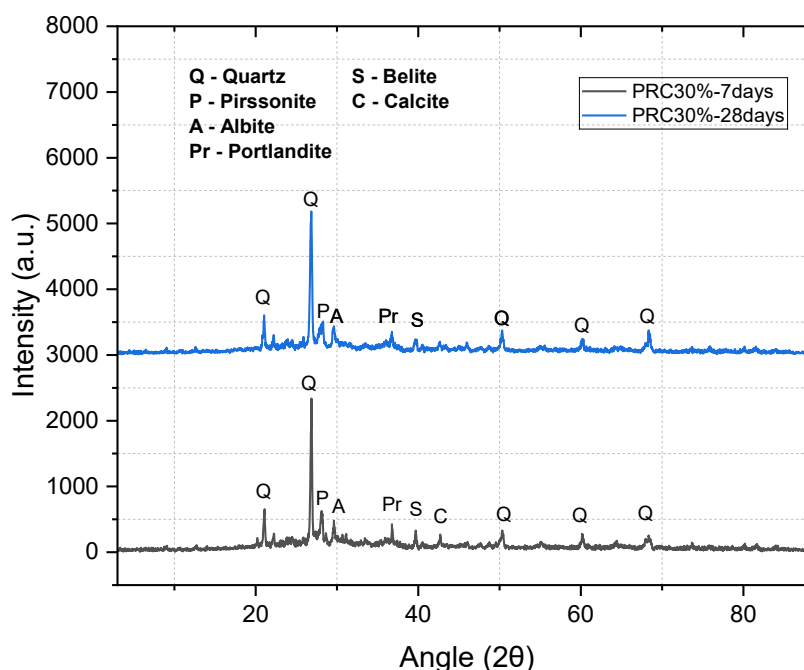


Figure 4. XRD comparison of pastes at different curing ages.

3.5. FTIR: Network Signatures and Carbonates

Figure 6 (0, 10, 30, and 100% PCR; 28 d) shows: (a) O–H/H–O–H ($3440\text{--}3320$ and $1690\text{--}1600\text{ cm}^{-1}$): structural/interlayer water associated with gels. (b) Carbonates (C–O) ($\approx 1460\text{--}1380\text{ cm}^{-1}$): more pronounced with higher Ca (PCR), consistent with secondary carbonation. (c) T–O–Si (Si/Al) ($\approx 1020\text{--}958\text{ cm}^{-1}$) and Si–O–(Si/Al) ($\approx 725\text{--}707\text{ cm}^{-1}$): intensify at intermediate PCR, consistent with greater network polymerization. Figure 7 (30% PCR, 7–14–28 d) shows a sustained increase of the $1020\text{--}958\text{ cm}^{-1}$ band, evidencing advancing condensation and time-dependent densification of the matrix.

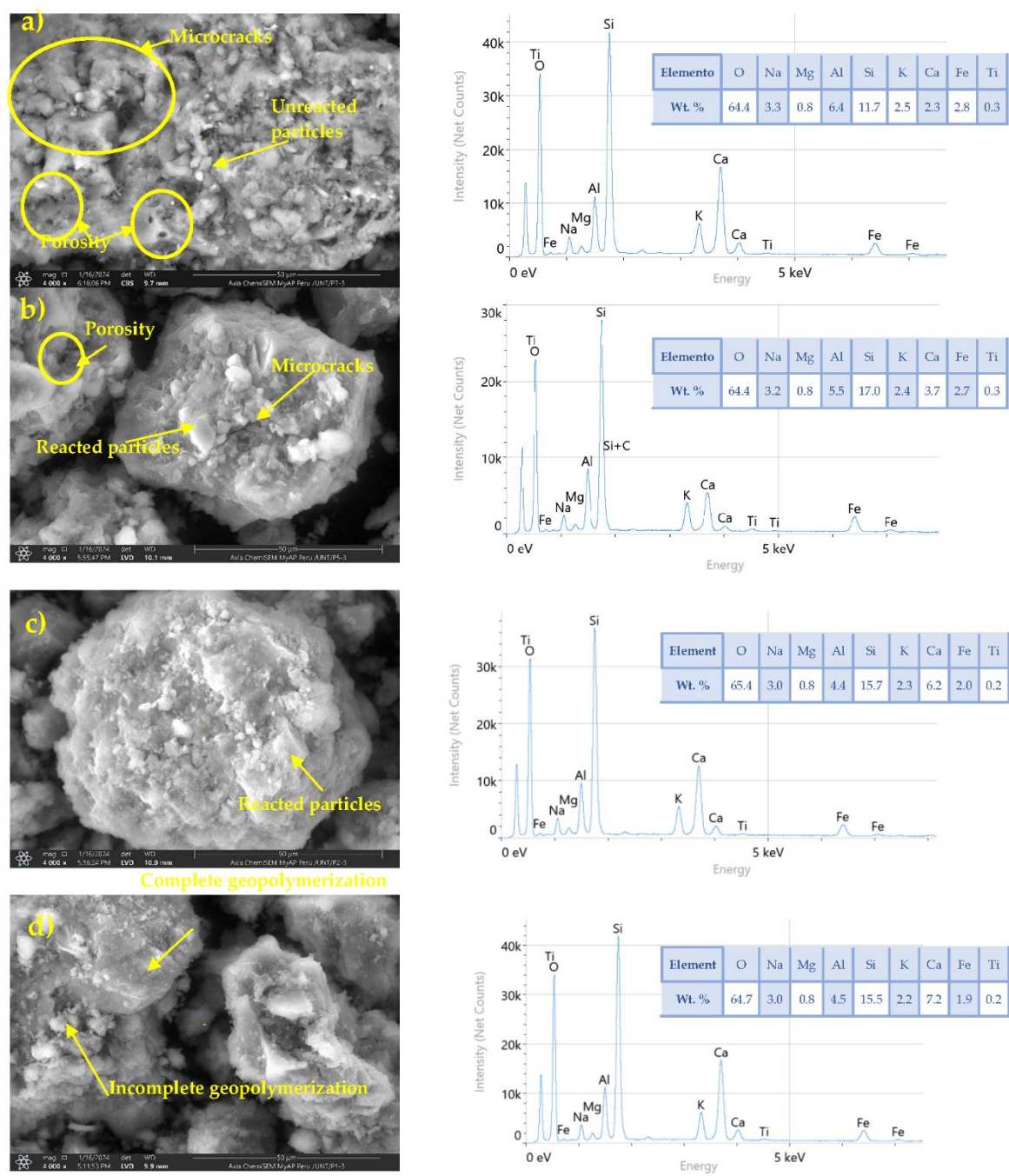


Figure 5. SEM-EDS of pastes with different compositions at 28 days: a) PCR 0%, b) PCR 10%, c) PCR 30% and d) PCR 100%.

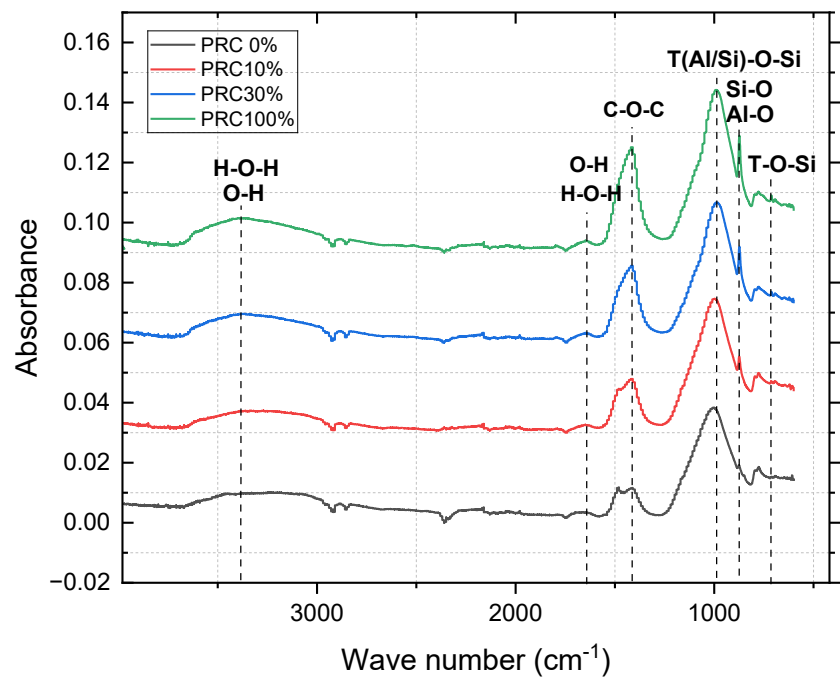


Figure 6. FTIR of pastes with different compositions at 28 days.

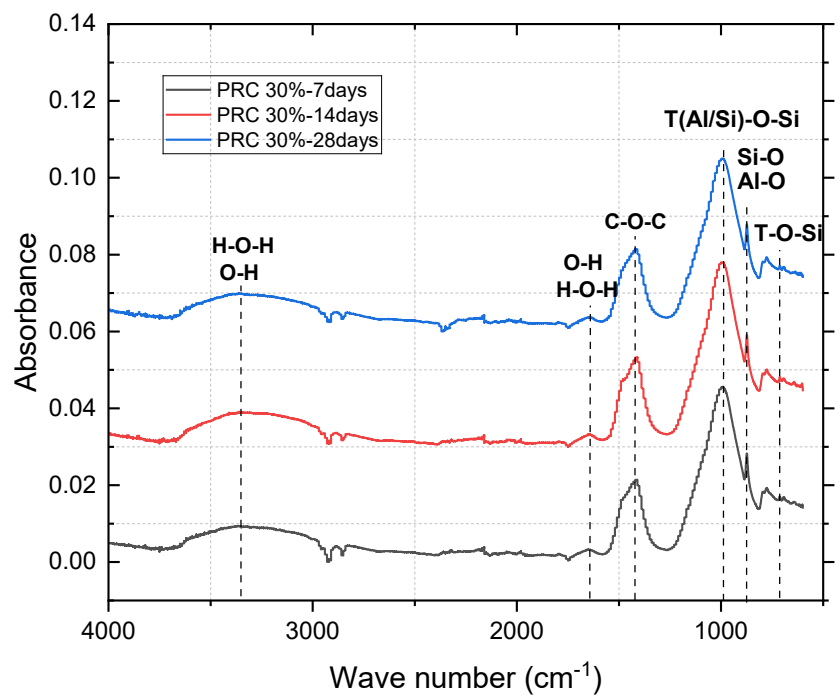


Figure 7. FTIR of pastes at different curing ages.

4. Discussion

4.1. Reinforcement Mechanism: 30% PCR Mix

The mechanical data (Figure 2) show a non-linear response with a peak f_c at $\approx 30\%$ PCR and progressive deterioration for substitutions $\geq 50\%$. This behavior is explained by a gel synergy whereby Ca provided by PCR nucleates C–A–S–H domains that bridge and densify the N–A–S–H network originating from the aluminosilicates in PLR [43–46]. While the Ca input remains moderate, the C–A–S–H/N–A–S–H co-existence increases connectivity, decreases effective porosity, and enhances load transfer; in contrast, once the PCR fraction exceeds that threshold, Ca^{2+} overloading together with dilution of reactive Si–Al species interrupts aluminosilicate crosslinking and activates competing routes (accelerated C–S–H formation and/or secondary carbonation), leading to a drop in f_c [42,47–49]. This trend is consistent with systems where high PCR substitutions or Ca-rich fillers (calcite) dilute reactive aluminosilicates and penalize strength [48,49].

4.2. Diffractometric Evidence: Mineralogical Simplification and Growth of the Amorphous Fraction

The 28-day XRD patterns (Figure 3) show that pure PLR (0% PCR) exhibits a broad crystalline palette (kaolinite, quartz, albite, belite, calcite, octahedrite, gehlenite, anorthite), in line with the ceramic nature of the precursor [51,52]. Upon introducing 10–30% PCR, portlandite ($\sim 36.6^\circ 2\theta$) appears and the diversity of PLR phases decreases (e.g., albite), while quartz around $26.7^\circ 2\theta$ remains. This reduction of crystalline phases together with the emergence of Ca-rich phases indicates consumption/dilution of PLR crystalline aluminosilicates and growth of the amorphous fraction associated with N–A–S–H/C–A–S–H gels, in line with the f_c increase at 30% PCR [54,55]. At 100% PCR, the pattern simplifies and is dominated by quartz/portlandite, evidencing Ca predominance and limitations of the aluminosilicate network, consistent with strength loss [56,57].

The correlation of FWHM and the amorphicity index over $18\text{--}38^\circ 2\theta$ maximizes at 30% PCR, whereas $I_{\text{Q}}(26.7^\circ)$ and $I_{\text{Pr}}(36.6^\circ)$ behave consistently with greater polymerization at intermediate Ca fractions and with crystalline simplification at 100% PCR ($r(f_c, \text{FWHM}) \approx +0.86$; $r(f_c, \text{Amorphicity}) \approx +0.88$) [54–57].

4.3. Spectroscopic Evidence (FTIR): Strengthening of T–O–Si Linkages and Secondary Processes

The 28-day FTIR spectra show reinforcement of the T–O–Si band ($\approx 1020\text{--}958\text{ cm}^{-1}$) in mixes with intermediate PCR, consistent with greater network extent and densification (in agreement with XRD and f_c). The carbonate signal ($\approx 1460\text{--}1380\text{ cm}^{-1}$) becomes more pronounced in Ca-rich compositions, compatible with secondary carbonation that may locally stiffen but, at the network scale, interferes with aluminosilicate crosslinking when Ca is excessive [42,47–49,65–72]. The time evolution (30% PCR, 7→28 d) with increasing T–O–Si confirms condensation progress under the applied curing protocol [50].

4.4. Microstructure (SEM-EDS): Densification, Heterogeneity, and Chemical Balance

28-day SEM micrographs show a transition from a porous matrix with microcracks (0% PCR) to a compact microstructure with minimal defects (10–30% PCR), and then back to a heterogeneous state with reintroduced microdefects (100% PCR). EDS supports this narrative: the increase of Ca (at./wt.%) from 0→30% PCR underpins nucleation and bridging of C–A–S–H domains, whereas at 100% PCR high Ca with a lower relative Al–Si fraction is observed, consistent with less continuous networks and lower f_c [62–65]. In sum, the triple triangulation (XRD–FTIR–SEM/EDS) supports the performance maximum around 30% PCR.

4.5. Kinetics and Curing: Initial Acceleration and Consolidation

Thermal pre-curing (40°C , 72 h) accelerates dissolution–condensation of dissolved species and favours early gel formation; subsequently, room-temperature curing enables network reorganization

and maturation, as reflected by the fc growth from 7→14→28 d and the intensification of structural FTIR bands [37,38,50]. This sequence reproduces the directionality reported for ceramic–cement alkali-activated systems, provided the Ca/Si–Al balance remains moderate [37,38].

4.6. Role of Quartz and Albite: from Inherited Phase to Partial Reactive Precursor

Quartz ($26.7^\circ 2\theta$) behaves mainly as an inherited phase and a marker of residual crystallinity: higher intensities associate with lower amorphicity and lower fc (mild-to-moderate negative correlation in our metrics). Albite ($29.54^\circ 2\theta$), by contrast, acts as a source of Al/Si; its relative decrease at 30% PCR suggests consumption toward more extensive amorphous networks (N–A–S–H/C–A–S–H), matching the fc peak and reports for PLR-dominant matrices under controlled Ca inputs [47,54,55].

4.7. Targeted Comparison with the Literature and Positioning of the Contribution

In PLR/BCR systems cured at 80–90 °C with 8–14 M NaOH, 40–60 MPa have been achieved; temperature and precursor fineness/porosity are decisive [37,38]. For PCR as a single precursor, <10–27 MPa values are frequent unless co-activated with MK or clinker and cured at 70 °C [38,39]. Our binary results confirm the synergy for low-to-intermediate PCR fractions, with a mechanical optimum emerging when Ca is sufficient to percolate C–A–S–H domains but not so high as to dilute the aluminosilicate stock and simplify mineralogy (quartz/portlandite) [40–42,54–57]. This behavior aligns with studies reporting improvements at 20–50% PCR and penalties at higher substitutions, reinforcing that the origin of the maximum is not a mere “filler effect” but a co-crosslinking of C–A–S–H/N–A–S–H governed by chemical balance [40–46,48,49,55].

4.8. Activator Design and L/B Ratio: Robustness and Bounds

The NaOH/Na₂SiO₃/KOH activator ($M_s \approx 3.2$) and L/B = 0.15 provide sufficient chemistry to deploy the synergy around 30% PCR. A higher L/B could improve workability but would likely raise porosity and lower fc; a higher M_s could increase polymerization (more available Si), albeit with risks of viscosity and early gelation; a lower M_s and/or higher KOH would tend to loosen the network and shift the optimal percentage, as inferred from activator–chemistry literature [27–36] and from our tracers (FWHM/FTIR) at 30% PCR. However, to isolate the role of %PCR, the present work kept these variables constant, which is part of its methodological contribution.

4.9. Pore Architecture and Defectology: Origin of Deterioration at High PCR

The reappearance of microdefects (pores, microcracks) at 100% PCR suggests: (i) precipitation/growth of Ca-rich products (C–S–H, portlandite) that interrupt network co-continuity; (ii) mismatch of thermo-mechanical moduli among domains of different composition; and (iii) preferential secondary carbonation in high-Ca matrices, which may occlude fine pores but open connectivity via local stresses [42,47–49,56,57]. Altogether, this reduces effective load-bearing area and increases microcracking susceptibility, leading to a loss in fc.

4.10. Practical Implications: Quality Control and Scale-Up

The results delineate a design window centered at ≈30% PCR that maximizes densification and performance. For industrial scale-up, we recommend: (i) fast tracers (FTIR 1020–958 cm^{−1} band and XRD FWHM) as process control; (ii) EDS quantification of Ca (at.%) to verify the Ca/Si–Al balance on receipt; (iii) ensuring adequate fineness and controlled inherited phases (quartz, albite) in PLR/PCR batches; and (iv) maintaining the curing protocol that guarantees the observed kinetic directionality [37,38,50,54–57].

4.11. Limitations and Future Work

Activator and L/B were fixed, and neither fineness nor compositional variability of CDW was varied. Next steps include mapping (i) M_s and effective molarity of the activator, (ii) granulometry and mineralogy of PLR/PCR, and (iii) curing windows (T–time–RH), to tune C–A–S–H/N–A–S–H co-crosslinking and mitigate secondary carbonation, while preserving inter-study comparability [37–39,43–49,54–57].

5. Conclusions

5.1 ~30% PCR fraction. Under NaOH/Na₂SiO₃/KOH activation ($M_s \approx 3.2$) and L/B = 0.15, the binary PLR–PCR pastes exhibited a local maximum in 28-day compressive strength of 16.2 MPa at 30% PCR, higher than 0% PCR (14.2 MPa) and clearly above PCR-rich formulations ($\geq 50\%$), which showed systematic decreases (e.g., 13.8 MPa at 50%, 10.8–10.1 MPa at 70–100%).

5.2. Mechanism: N–A–S–H/C–A–S–H co-crosslinking. Ca supplied by PCR nucleates C–A–S–H domains that bridge and densify the N–A–S–H network (derived from PLR aluminosilicates), increasing connectivity and load-bearing capacity up to intermediate PCR fractions. Above ~30–50%, excess Ca²⁺ and reduced availability of reactive Si–Al species dilute the aluminosilicate network, promote C–S–H and/or secondary carbonation, and degrade fc.

5.3. XRD at ~30% PCR. At 28 days, 0% PCR exhibits a broad crystalline suite (kaolinite, quartz, albite, belite, calcite, gehlenite, anorthite). With 10–30% PCR, portlandite ($\sim 36.6^\circ 2\theta$) emerges and the relative intensity of albite ($29.54^\circ 2\theta$) and other PLR phases decreases, indicating consumption/dilution of crystalline aluminosilicates and growth of the amorphous fraction associated with reaction gels; this is consistent with the fc peak at 30% PCR. At 100% PCR the pattern simplifies (quartz/portlandite dominant), reflecting Ca predominance and loss of crystalline diversity.

5.4. FTIR evidence of increased polymerization. For 0–10–30–100% PCR (28 d), the T–O–Si band (~ 1020 – 958 cm^{-1}) intensifies at intermediate compositions, indicating greater network extent and densification; carbonates (~ 1460 – 1380 cm^{-1}) increase with Ca, consistent with more prominent carbonation at high PCR. At 30% PCR, the 7→14→28 d evolution shows a sustained growth of T–O–Si, confirming condensation progress.

5.5. SEM–EDS microstructure: densification at 30% PCR. 28-day micrographs show a transition from porosity and microcracking (0% PCR) to a compact matrix with minimal defect population (10–30%), and then reappearance of microdefects at 100% PCR. EDS follows this trend: Ca (wt.%/at.%) increases from 0→30% PCR (favoring C–A–S–H) and is high at 100% PCR alongside a lower relative Al–Si fraction, consistent with heterogeneity and reduced co-continuity of the network.

5.6. Kinetics and curing. Pre-curing at 40 °C/72 h accelerates initial dissolution–condensation and gel formation; subsequent ambient curing consolidates the network, as reflected by the fc increase (7→14→28 d) and intensification of structural FTIR bands. The response depends on the Ca/Si–Al balance: beneficial around 30% PCR and counterproductive at high PCR contents.

5.7. Formulation and control implications. For reproducibility and process control, we recommend: (i) maintaining L/B = 0.15 and an activator with $M_s \approx 3.2$; (ii) ensuring fineness < 75 μm for PLR/PCR to homogenize reactivity; (iii) monitoring T–O–Si (FTIR) and FWHM/amorphicity index (XRD) as rapid tracers of polymerization; and (iv) verifying Ca (EDS) in incoming batches to sustain the chemical balance that enables N–A–S–H/C–A–S–H synergy.

5.8. Scope and primary limitation. The study isolated the effect of %PCR by keeping activator and L/B constant; thus, it demonstrated that the best performance stems from chemical synergy and microstructural densification around 30% PCR, whereas deterioration at high PCR is associated with excess Ca, mineralogical simplification, and microdefect development. Future work should map M_s /molarity, granulometry/mineralogy, and curing windows to refine the optimal range and mitigate secondary carbonation.

The binary valorization of PCR/PLR enables geopolymeric binders with a lower carbon footprint and competitive mechanical/microstructural performance when the Ca contribution from PCR is moderated around ~30%. In that range, N-A-S-H/C-A-S-H co-formation maximizes matrix densification and compressive strength; outside it, Ca overloading dilutes reactive aluminosilicates, increases carbonates/C-S-H, and penalizes performance. These results provide transferable design and control criteria for CDW management in alternative cementitious materials.

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Abbreviations

The following abbreviations are used in this manuscript:

Materials and waste

- RCD: Construction and Demolition Waste (Spanish acronym).
- CDW: Construction and Demolition Waste (English equivalent of RCD).
- PCR: Recycled Concrete Powder.
- PLR: Recycled Brick Powder.
- BCR: Recycled Ceramic Tiles (when citing tile-based literature).
- MK: Metakaolin

Activators and chemistry

- NaOH: Sodium hydroxide.
- KOH: Potassium hydroxide.
- Na₂SiO₃: Sodium silicate (commercial solution).
- M_s: Silica modulus of sodium silicate (M_s = SiO₂/Na₂O).
- L/B or l/a: Liquid-to-binder ratio (liquid/binder).
- N-A-S-H: Sodium Aluminosilicate Hydrate (sodium aluminosilicate gel).
- C-A-S-H: Calcium Aluminosilicate Hydrate (calcium aluminosilicate gel).
- C-S-H: Calcium Silicate Hydrate (calcium silicate gel).

Properties and metrics

- f_c: Compressive strength (MPa).
- SD: Standard deviation.
- FWHM: Full Width at Half Maximum (in XRD).
- I_Q(26.7°): Intensity of the quartz peak around 26.7° 2θ.
- I_{Pr}(36.6°): Intensity of the portlandite peak around 36.6° 2θ.
- d: Days (curing age, e.g., 7, 14, 28 d).
- MPa: Megapascal (unit of stress).

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