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Article

Optimization of Sisal Content in Geopolymer Mortars with Recycled Brick and Concrete: Design and Processing Implications

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Abstract

Geopolymer mortars were produced from construction-and-demolition waste using a binary binder of recycled brick powder/recycled concrete powder (RBP/RCP = 70/30 wt%), activated with a hybrid alkaline solution (NaOH/Na₂SiO₃/KOH) and reinforced with sisal fibres at 0–2 wt%. Mechanical performance (compression and three-point bending) and microstructure–phase evolution (XRD, FTIR, SEM-EDS) were assessed after low-temperature curing. Sisal addition delivered a strength–toughness trade-off with a reproducible optimum at ~1.0–1.5 wt%; at 2.0 wt%, fibre clustering and connected porosity reduced the effective load-bearing section, penalising flexure more than compression. Microstructural evidence indicates coexistence and co-crosslinking of N-A-S-H and C-(A)-S-H gels—enabled by Ca from RCP—leading to matrix densification and improved fibre–matrix anchorage. Fractographic features (tortuous crack paths, bridging, and extensive pull-out at ~1.5 wt%) are consistent with an extended post-peak response and higher fracture work without compromising early-age strength. The study (i) identifies a practical reinforcement window for sisal in RBP/RCP geopolymers, (ii) links gel chemistry and interfacial phenomena to macroscopic behaviour, and (iii) distils processing guidelines (rain addition, workability control, gentle deaeration, constant A/S) that support reproducibility. These outcomes provide a replicable, low-embodied-CO₂ route to fibre-reinforced geopolymer mortars derived from CDW for non-structural and semi-structural applications where flexural performance and post-peak behaviour are critical.

Keywords: geopolymer mortar; recycled brick powder (RBP); recycled concrete powder (RCP); sisal fibre; alkali activation; construction and demolition waste (CDW); N-A-S-H; C-(A)-S-H; SEM-EDS; XRD; FTIR

1. Introduction

The construction sector is a structural driver of materials consumption, energy demand, and greenhouse-gas emissions. Its heavy reliance on Portland cement amplifies the embodied-CO₂ of the built environment, motivating the development of low-carbon binders and circular strategies that valorize construction and demolition waste (CDW) as secondary raw materials [3,4]. In the Peruvian context, sustained growth of construction activity and a specific regulatory framework for CDW and solid-waste management create an opportunity to close material loops and divert CDW from informal dumping to higher-value uses [14,22–24,30,45].

Within this agenda, alkali-activated materials (AAMs) and geopolymers have emerged as promising alternatives: aluminosilicate networks can form at ambient or moderately elevated temperatures from a broad spectrum of precursors (industrial by-products and mineral residues), delivering mechanical performance and durability comparable to ordinary Portland cement (OPC) with lower embodied CO₂ when precursors, alkalinity, and curing are properly tuned [13,66,67,69–71]. Among CDW-derived precursors, recycled brick powder (RBP) contributes reactive glassy

fractions rich in Si–Al, whereas recycled concrete powder (RCP) supplies residual Ca; in combination, they facilitate the coexistence/co-crosslinking of N-A-S-H and C-(A)-S-H gels at early ages, favouring consolidation and strength development when workability and setting are controlled [63,79,82].

As with other brittle cementitious systems, geopolymers exhibit limited flexural toughness, with sensitivity to crack initiation and propagation. Fiber reinforcement is a proven route to enhance damage tolerance via crack bridging, deflection, and frictional pull-out, increasing post-peak energy dissipation [18,35,66]. Among natural fibers, sisal (*Agave sisalana*) stands out for its high specific stiffness, regional availability, and reasonable alkali compatibility after conditioning; in mortar-like matrices, multiple studies report dosage windows where gains in flexure/toughness outweigh porosity penalties—provided dispersion, workability, and deaeration are ensured [28,42,49,60,72,89].

Aim and contribution. This work formulates RBP/RCP = 70/30 wt% geopolymer mortars, activated with a NaOH/Na₂SiO₃/KOH system and reinforced with sisal fibers (0–2 wt%), to: (i) quantify the strength–toughness compromise in compression (ASTM C109) and flexure (ASTM C348), and (ii) link microstructure to properties using XRD/FTIR/SEM-EDS, thereby rationalizing the optimum fiber content from hybrid-gel chemistry and fiber–matrix interfacial interactions. The contribution is threefold: (a) delineate a reproducible, process-aware dosage window for sisal (property–processability) in RBP/RCP systems; (b) explain post-peak behaviour through fractography and the co-evolution of N-A-S-H/C-(A)-S-H; and (c) translate findings into design/processing guidelines (late “rain” addition, flow control, gentle deaeration, constant A/S) that facilitate scalability of CDW-derived geopolymers with lower embodied CO₂ [13,18,28,35,49,60,63,66,67,69–72,79,82,89].

2. Materials and Methods

2.1. Raw Materials and Preparation

Construction-and-demolition waste (CDW) from a disposal site in Trujillo (Peru) was processed to obtain recycled brick powder (RBP; Spanish RBP) and recycled concrete powder (RCP). Both precursors were crushed, milled, and sieved to pass No. 400 (≈38 μm). Their oxide compositions (XRF) are listed in Table 1 (major oxides: SiO₂, Al₂O₃, CaO, Fe₂O₃, Na₂O, K₂O, MgO; LOI). Prior to mixing, powders were equilibrated in a desiccator (23–25 °C) for ≥24 h.

Table 1. Chemical characterization (oxides compound) of RPC and RBP.

RPC		RBP	
Óxides	wt%	Óxides	wt%
SiO ₂	51.21	SiO ₂	55.21
CaO	23.78	Al ₂ O ₃	19.17
Al ₂ O ₃	10.45	Fe ₂ O ₃	8.21
Fe ₂ O ₃	5.01	K ₂ O	2.74
MgO	2.46	MgO	1.79
K ₂ O	2.14	CaO	1.59
Na ₂ O	1.27	Na ₂ O	1.87
SO ₃	1.98		
TiO ₂	0.81		
P ₂ O ₅	0.51		
MnO	0.27		

2.2. Fibres and Alkaline Reagents

Sisal fibres (*Agave sisalana*) were sourced locally (Moche, Trujillo), extracted by water retting and manual pressing, cut to ≈1 cm, dried to constant mass at ambient conditions, and stored in sealed bags. No chemical surface treatment was applied.

Alkaline activator: A ternary activator NaOH/Na₂SiO₃/KOH with a mass split 0.10/0.60/0.30 was used at A/S = 0.485, i.e., 485 g of activator per 1000 g of binder (RBP+RCP) (Table 2). Pellets NaOH (≥98 wt%) and KOH (≥90 wt%) were dissolved separately in deionized water and then mixed with the commercial Na₂SiO₃ solution (nominal Ms ≈ 3.2, reference ρ ≈ 1.38 g·mL⁻¹ at 20 °C). Per 1000 g of binder, the component masses were: NaOH 48.5 g (effective 47.53 g), Na₂SiO₃ 291.0 g, KOH 145.5 g (effective 130.95 g). From these declared masses (purity-corrected), the strong-base charge is:

- n(NaOH)=47,53/40,00=1,188 mol
- n(KOH)=130,95/56,1056=2,334 mol
- n(OH⁻) =3,522 por 1000 g de aglomerante.

Table 2. Mix compositions of geopolymer mortars (reference batch normalised to 1,000 g of binder). Binder:sand = 1:3 (by mass). Activator-to-solids ratio (A/S) = 0.485 with respect to the binder. Mass split of the ternary activator: 0.10 NaOH: 0.60 Na₂SiO₃ (commercial solution): 0.30 KOH.

Mix ID	Sisal (wt% of binder)	RBP in binder (wt%)	RCP in binder (wt%)	Binder (g)	Sisal (g)	Sand (g)	A/S (by binder mass)	Activator total (g)	NaOH (g)	Na ₂ SiO ₃ (g)	KOH (g)
M1SF0	0	70	30	1000	0	3000	0.485	485	48.5	291	145.5
M2SF10	1	70	30	1000	10	3000	0.485	485	48.5	291	145.5
M3SF15	1.5	70	30	1000	15	3000	0.485	485	48.5	291	145.5
M4SF20	2	70	30	1000	20	3000	0.485	485	48.5	291	145.5

Notes: (i) A/S is defined with respect to the binder (RBP + RCP), excluding sand and fibres; (ii) NaOH/KOH pellets are dissolved separately and then mixed with Na₂SiO₃; the listed masses refer to the activator components as prepared.

2.3. Mix Design and Experimental Plan

The binary binder was RBP/RCP = 70/30 wt%. Sisal fibre dosages (by binder mass) were 0.0, 1.0, 1.5, and 2.0 wt%. The activator-to-solids ratio (A/S) was kept constant at 0.485 for all series. Binder-to-sand ratio was 1:3 (by mass). For each condition, n = 5 specimens were fabricated per test. Table 2 summarises the compositions; Figure 1 outlines the workflow.

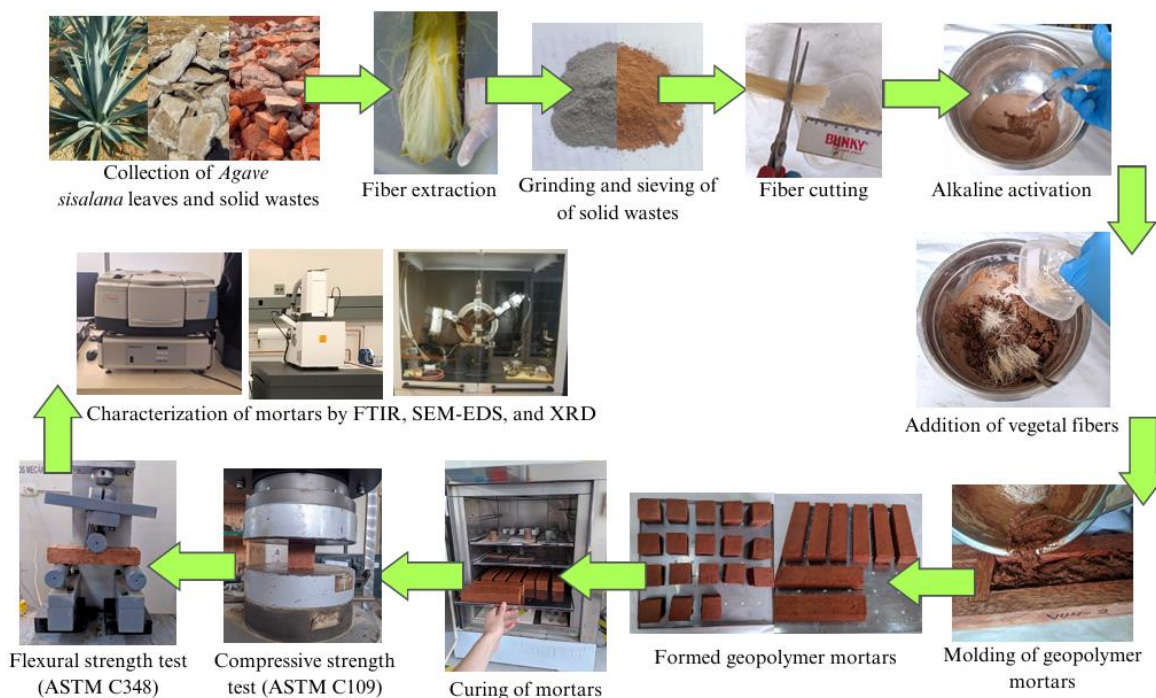


Figure 1. Experimental workflow: activator preparation (dissolution of NaOH/KOH, addition of Na_2SiO_3 , resting) → dry mixing of RBP/RCP (70/30 by mass) → gradual addition of the activator ($A/S = 0.485$) → “rain” addition of 0–2 wt% sisal (of the binder) during the final third of mixing (dispersion, minimising bundles) → casting and deaeration (50 mm cubes; $40 \times 40 \times 160$ mm prisms) → curing at $40^\circ\text{C} \times 72$ h and sealed storage to 7/14/28 d → mechanical testing (ASTM C109, ASTM C348) → characterisation (XRD, FTIR-ATR, SEM-EDS using Axia ChemiSEM).

2.4. Mixing, Casting, and Curing

Mixing was carried out in a mortar mixer. Sequence: (1) dry-blend RBP–RCP for ~2 min; (2) add the activator gradually (~2 min) and continue until uniform consistency; (3) add fibres “in rain” during the final third of mixing to promote dispersion and minimise bundling. Specimens: $50 \times 50 \times 50$ mm cubes (compression) and $40 \times 40 \times 160$ mm prisms (three-point bending). Gentle tapping/brief vibration was used for deaeration. Demoulding at 24 ± 2 h. Curing: 40°C for 72 h in an oven (low-temperature curing), then sealed storage (bags) to the test ages 7, 14, and 28 days.

2.5. Mechanical Testing

Compression (ASTM C109): 50 mm cubes; loading rate $\approx 0.5 \text{ MPa s}^{-1}$; ages 7, 14, and 28 days. Maximum stress and failure mode were recorded.

Three-point bending (ASTM C348): $40 \times 40 \times 160$ mm prisms; span $L = 120$ mm; loading rate 50 N s^{-1} . Flexural strength (MOR) was computed as:

$$\sigma_f = \frac{3PL}{2bd^2} \quad (1)$$

where P is the maximum load, L the span, and b and d are the specimen width and height, respectively. Results are reported as mean $\pm$ SD ($n = 5$) with error bars in the figures (Tukey superscripts where applicable). Flexural and compression tests were performed on a semi-automatic ADR Touch SOLO 2000 machine with digital display (ELE International), capacity 2000 kN ($\approx 450,000$ lbf).

2.6. Stereomicroscopy of Fracture Surfaces

A Euromex NexiusZoom NZ.1902-S stereomicroscope (10×) with incident and oblique illumination (~20–40°) was used to observe flexural fracture surfaces. Samples were handled dry, cleaned with oil-free air, and imaged under constant lighting per batch to highlight relief, active fibres (bridging/pull-out), and porosity while avoiding specular glare (diffuser as needed).

2.7. X-Ray Diffraction (XRD): Grinding, Mounting, and Scan

Representative matrix fragments (avoiding fibres) from broken specimens were dried at 60 ± 2 °C for 24 h, cooled in a desiccator (1 h), and ground to pass No. 200 (≈ 75 μm) to reduce preferred orientation. Measurements used Cu K α radiation ($\lambda = 1.5406$ Å), 5–70° (2 θ), step $\sim 0.02^\circ$, and a counting time per step sufficient for an adequate signal-to-noise ratio. Crystalline phases were identified and the amorphous hump (~ 20 – 35° 2 θ) was analysed (position/FWHM) as a proxy for gel polymerisation. Powders were stored sealed with silica gel until analysis.

2.8. Fourier-Transform Infrared Spectroscopy (FTIR)

Spectra were collected in 4000–400 cm^{-1} , resolution ~ 4 cm^{-1} , ≥ 32 scans, using ATR. We monitored O–H/H–O–H bands (~ 3400 and ~ 1640 cm^{-1}), the T–O–T band (T = Si, Al) near ~ 1000 cm^{-1} (downshift/broadening associated with polycondensation), and carbonates (~ 1450 and ~ 870 cm^{-1}). Sampling, drying, grinding, and storage followed Section 2.7 to ensure consistency.

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

Polished cross-sections were prepared from representative fragments (bent prisms): diamond saw cutting, vacuum impregnation (low-viscosity epoxy), and progressive polishing (P320→P4000; finish with alumina 1.0/0.3 μm). When required, a conductive coating (C or Au) was applied.

Instrument: Thermo Scientific Axia ChemiSEM (real-time EDS integration), 15 kV accelerating voltage, working distance ~ 10 mm. Secondary- and backscattered-electron images and EDS maps (Si, Al, Ca, Na, K) were acquired to distinguish N-A-S-H/C-(A)-S-H domains and the interfacial transition zone (ITZ). Micrographs include scale bars and, where relevant, annotations of bridging/pull-out/debonding.

2.10. Data Treatment and Statistical Analysis

For each (fibre content $\times$ age) combination, $n = 5$ replicates were tested. Normality (Shapiro–Wilk) and homoscedasticity (Levene) were verified. Group comparisons by fibre content used one-way ANOVA, followed by Tukey's HSD ($\alpha = 0.05$). Along with mean $\pm$ SD, 95% confidence intervals (95% CI) and effect size (η^2) are reported. In figures, superscript letters denote statistically distinct groups; p-values and η^2 are provided in supplementary tables.

For bending, MOR was computed as in Section 2.5; load–deflection curves were recorded to qualitatively discuss post-peak response (area under the curve). Ambient conditions during testing were 23–25 °C to minimise temperature/humidity variability.

3. Results

3.1. Mechanical Properties

3.1.1. Compressive Strength (7–14–28 Days)

Compressive strength of 50 mm cubes increased with curing age for all mixtures (RBP/RCP = 70/30 wt%; sisal = 0.0–2.0 wt%), consistent with progressive gel formation and matrix densification. The effect of fibre content was non-linear: relative to 0.0%, strength peaked at 1.0% sisal and then declined at 2.0% (Figure 2). This behaviour is attributed to two competing effects: (i) crack-bridging and frictional load transfer at the fibre–matrix interface at low–moderate dosages; and (ii) loss of

packing and connected porosity due to fibre clustering at high dosages, which weakens matrix continuity.

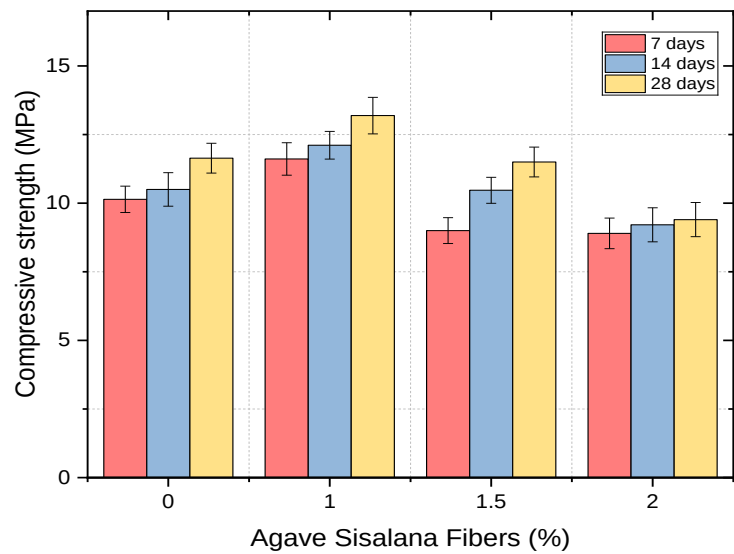


Figure 2. Compressive strength at 7, 14, and 28 days for RBP/RCP mortars with 0.0–2.0 wt% sisal (mean ± SD; n = 5).

Group statistics (mean ± SD, n = 5) and 95% CI are reported in Table 3 (Tukey superscripts at $\alpha = 0.05$). One-way ANOVA showed significant differences among fibre contents at all ages; effect sizes (η^2) indicated large practical magnitude.

Table 3. Compressive strength (MPa) — mean ± SD [95% CI]; Tukey letters by age.

Age	Sisal content	Compression (MPa) — Mean ± SD [95% CI]	Tukey letter
7d	0%	10.14 ± 0.35 [9.71, 10.57]	b
7d	1.0%	11.61 ± 0.52 [10.96, 12.26]	a
7d	1.5%	9.00 ± 0.38 [8.53, 9.47]	b
7d	2.0%	8.90 ± 0.53 [8.24, 9.56]	c
14d	0%	10.50 ± 0.40 [10.00, 11.00]	b
14d	1.0%	12.11 ± 0.44 [11.56, 12.66]	a
14d	1.5%	10.47 ± 0.51 [9.84, 11.10]	b
14d	2.0%	9.21 ± 0.43 [8.68, 9.74]	c
28d	0%	11.64 ± 0.39 [11.16, 12.12]	b
28d	1.0%	13.19 ± 0.50 [12.57, 13.81]	a
28d	1.5%	11.50 ± 0.49 [10.89, 12.11]	b
28d	2.0%	9.40 ± 0.47 [8.82, 9.98]	c

3.1.2. Flexural Strength (Three-Point Bending)

For 40 × 40 × 160 mm prisms tested at L = 120 mm, bending confirmed a beneficial dosage window (1.0–1.5 wt%), with the best response around 1.5% (Figure 3). At 2.0%, performance decreased, in line with induced defects (porosity, fibre bundles) that reduce the effective section and are particularly detrimental under bending (Figure 4). At ~1.5%, load–deflection curves exhibited a longer post-peak segment and gradual decay, consistent with crack bridging and progressive pull-out of embedded fibres. Group statistics (mean ± SD, n = 5) with 95% CI and Tukey letters ($\alpha = 0.05$) are reported in Table 4.

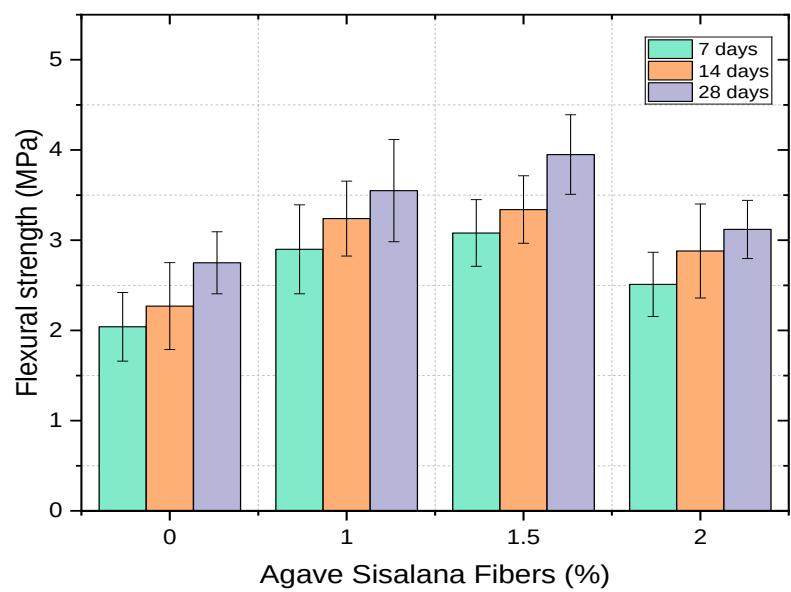


Figure 3. Flexural strength (MOR) at 7, 14, and 28 days (mean ± SD; n = 5).

Table 4. Flexural strength, MOR (MPa) — mean ± SD [95% CI]; Tukey letters by age.

Age	Sisal content	Flexural strength, MOR (MPa) — Mean ± SD [95% CI]	Tukey letter
7d	0%	3.28 ± 0.16 [3.09, 3.48]	c
7d	1.0%	4.52 ± 0.25 [4.21, 4.84]	a
7d	1.5%	4.42 ± 0.10 [4.29, 4.54]	a
7d	2.0%	3.83 ± 0.15 [3.65, 4.02]	b
14d	0%	3.42 ± 0.08 [3.32, 3.52]	c
14d	1.0%	4.88 ± 0.10 [4.76, 5.01]	a
14d	1.5%	4.96 ± 0.38 [4.49, 5.43]	a
14d	2.0%	4.12 ± 0.11 [3.98, 4.26]	b
28d	0%	3.94 ± 0.24 [3.65, 4.23]	b
28d	1.0%	5.38 ± 0.20 [5.13, 5.63]	a
28d	1.5%	5.54 ± 0.26 [5.22, 5.86]	a
28d	2.0%	4.16 ± 0.46 [3.58, 4.73]	b

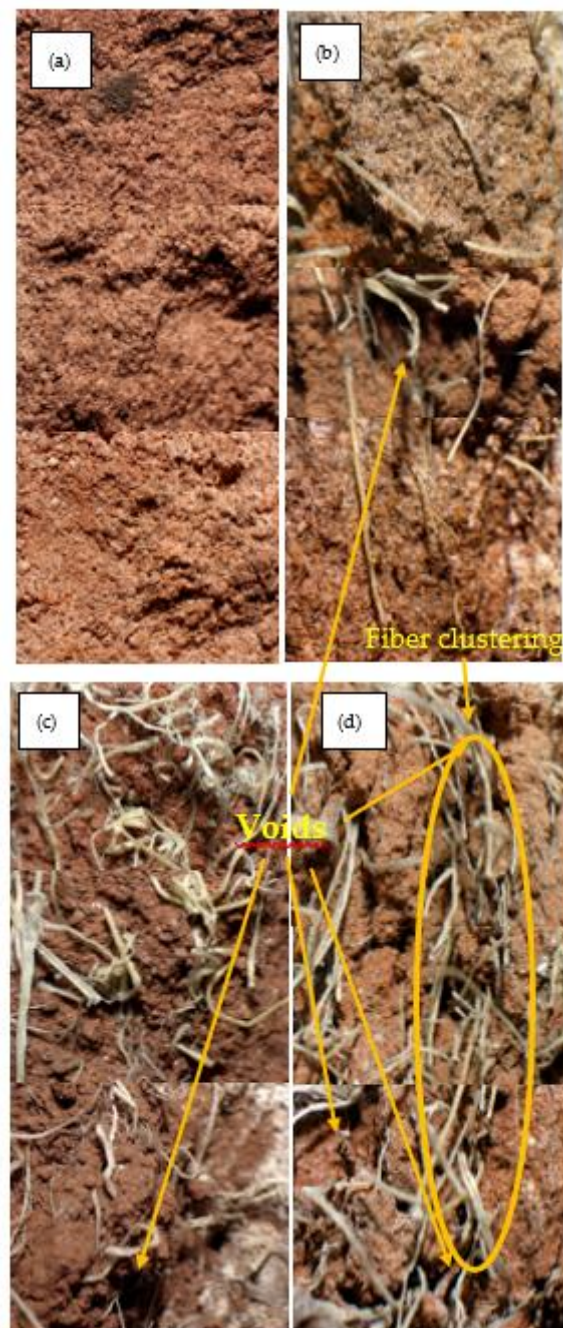


Figure 4. Representative flexural fracture cross-sections: (a) 0.0%; (b) 1.0%; (c) 1.5%; (d) 2.0%.

3.1.3. Compression–Flexure Correlation

Averaged by mixture, the relative gain was greater in flexure than in compression for 1.0–1.5% fibres, indicating that sisal is most effective in tension-dominated mechanisms (crack opening). The decline at 2.0% was more pronounced in flexure, consistent with the stronger penalty of connected porosity and fibre clustering when tensile responses govern.

3.2. Fracture Morphology in Bending (28 Days)

Macroscopic observations (Figure 4) show a systematic evolution of the fracture mode with fibre content:

- 0.0%: straight fracture plane, low tortuosity, minimal branching;
- 1.0%: more sinuous crack path and moderate pull-out;

- 1.5%: high density of active fibres (bridges), secondary branching, visible extraction lengths;
- 2.0%: fibre agglomerates and connected pores near the fracture plane.

These features align with the increased toughness and post-peak capacity at 1.5%, and with the loss of performance at 2.0% due to processing-induced defects.

The higher density of active bridges and visible extraction lengths at ~1.5 wt% mirrors interfacial conditions reported for sisal after mild alkaline conditioning, where pull-out-dominated failure increases post-peak toughness; the trend reverses when over-treatment or excessive fibre content introduces defects.

3.3. X-Ray Diffraction (XRD)

Diffraction patterns (Figure 5) showed a broad amorphous hump at $2\theta \approx 20\text{--}35^\circ$, typical of alkali-activated matrices, together with quartz/cristobalite peaks inherited from RBP and minor residual phases from RCP. From 7 to 28 days, the hump stabilised/intensified without the emergence of dominant new crystalline phases, suggesting greater aluminosilicate network polymerisation. The Ca contribution from RCP is consistent with hybrid gels C-(A)-S-H coexisting with N-A-S-H, which rationalises the age-related strength gain. Differences between 0.0% and 2.0% fibres were subtle in crystalline mineralogy, though small changes in disorder (hump position/width) are plausible.

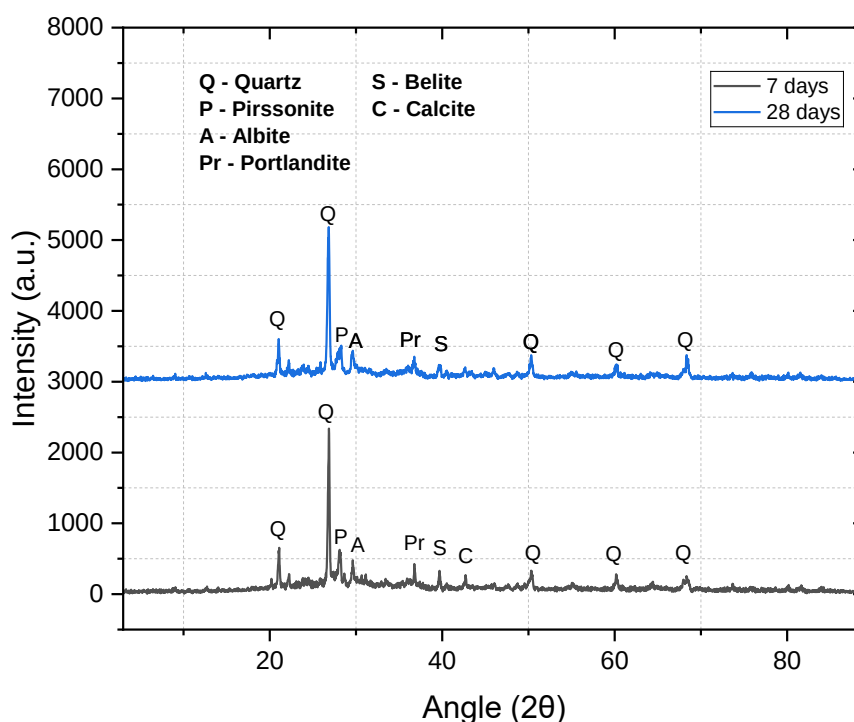


Figure 5. XRD ($5\text{--}70^\circ 2\theta$) of RBP/RCP mortars at 7–28 days.

3.4. Fourier-Transform Infrared Spectroscopy (FTIR)

Spectra (Figure 6) displayed O–H/H–O–H bands (~ 3400 and 1640 cm^{-1}) from structural/surface water and carbonates (~ 1450 and 870 cm^{-1}) due to superficial carbonation in alkaline media. The T–O–T band (T = Si, Al) centred near $\sim 1000\text{ cm}^{-1}$ downshifted and broadened with age, in line with polycondensation and increased short-range order of the gel. Features compatible with C-(A)-S-H ($\approx 700\text{--}600\text{ cm}^{-1}$) support the hybrid nature of the system (consistent with XRD). No new bands attributable to deleterious fibre–matrix reactions were observed.

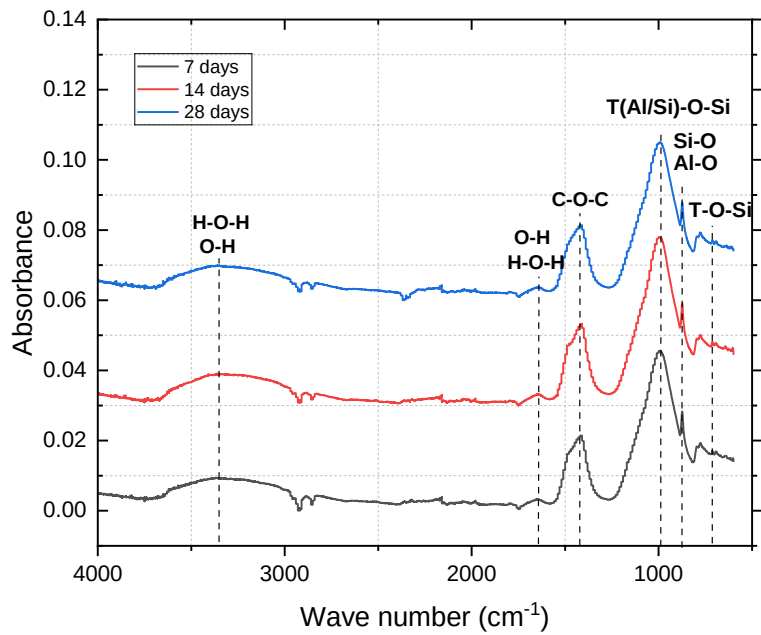


Figure 6. FTIR-ATR (4000–400 cm⁻¹) at 7–14–28 days.

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

Micrographs (Figure 7) revealed a transition from a heterogeneous texture with gel domains and residual particles (7 days) to a denser, more continuous microstructure (28 days). EDS maps (Si, Al, Ca, Na, K) showed distributions consistent with N-A-S-H/C-(A)-S-H domains and an interfacial transition zone (ITZ) that appeared tighter at 28 days. No thick amorphous coatings on fibres or evidence of significant sisal degradation were observed at the tested ages.

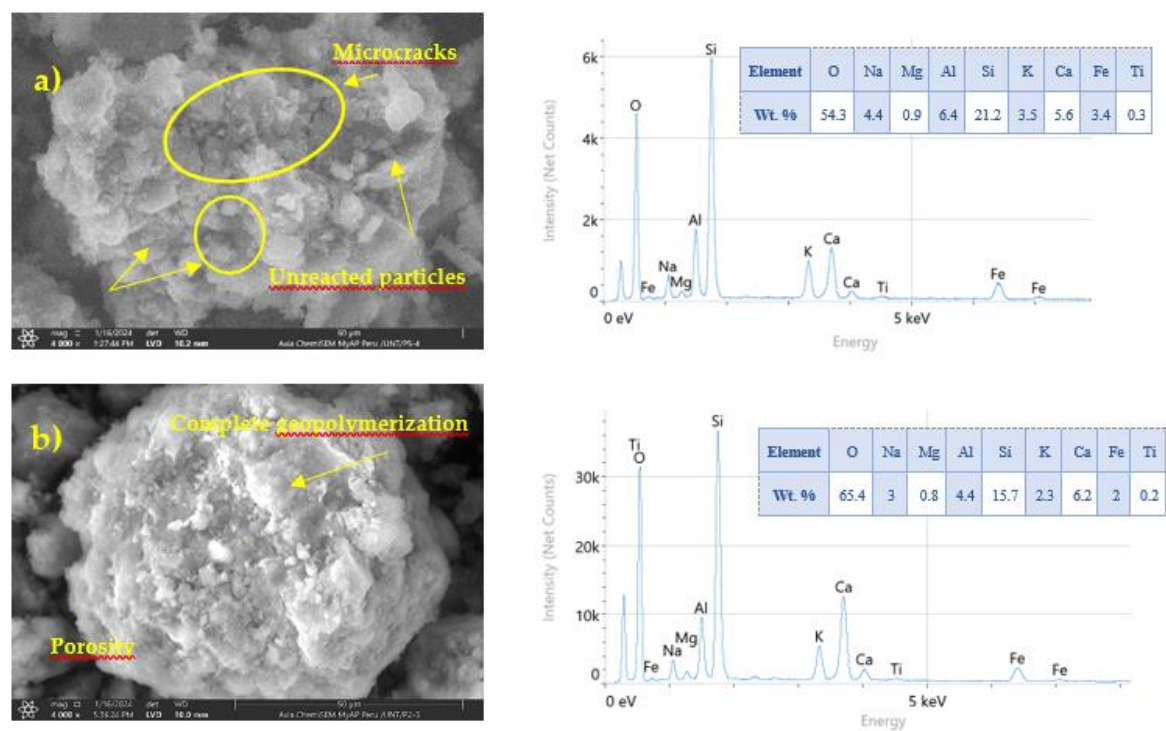


Figure 7. SEM-EDS microscopies of geopolymers mortars. a) 7 days; b) 28 days.

3.6. Statistical Summary (Assumptions, ANOVA, Tukey)

Parametric assumptions were satisfied (Shapiro–Wilk, $p > 0.05$; Levene, $p > 0.98$). Fibre content significantly affected compression and flexure at 7–28 days (one-way ANOVA, $p < 0.001$; $\eta^2 \approx 0.89$ – 0.93).

Tukey's HSD ($\alpha = 0.05$) confirmed patterns consistent with the mechanical/fractographic reading:

- Compression (all ages): 1.0% (a) $>$ 0.0% (b) = 1.5% (b) $>$ 2.0% (c).
- Flexure: 1.0% = 1.5% (a) $>$ 2.0% (b) $>$ 0.0% (c) at 7–14 days; and 1.0% = 1.5% (a) $>$ 2.0% = 0.0% (b) at 28 days.

Complete means $\pm$ SD, 95% CI and Tukey letters are given in Tables 3–4 (per age), with p-values and η^2 detailed in the Supplementary Tables.

4. Discussion

4.1. Optimal Sisal Dosage: A Bridging–Porosity Compromise

The mechanical trends delineate a clear reinforcement window at ~ 1.0 – 1.5 wt% sisal. In this range, crack-bridging and progressive pull-out dissipate energy and stabilise the post-peak response, producing larger relative gains in flexure (MOR) than in compression—consistent with the fibre's action under tension-dominated mechanisms (crack opening) [41–47,61,69]. At 2.0 wt%, workability decreases and fibre bundles (clustering) with connected porosity appear, reducing the effective load-bearing section; these defects penalise bending more than compression and explain the MOR drop [61,65,69]. This bridging–porosity balance also rationalises why 1.0 wt% tops compressive strength, whereas ~ 1.5 wt% maximises flexural performance: at low–moderate contents, frictional load transfer at the fibre–matrix interface dominates; beyond the threshold, shadowing between bundles, air occlusion and loss of packing undermine matrix continuity [41–47,61,65,69]. Macroscopic fracture images (Figure 4)—more tortuous crack paths and a higher density of active bridges at 1.5%, versus agglomerates/pores near the crack plane at 2.0%—and the statistical hierarchy (ANOVA/Tukey, Tables 3–4; Supplementary S1–S4) support this interpretation.

Recent literature on sisal-reinforced geopolymers shows that, at moderate fibre contents, crack bridging and pull-out account for the increase in ductility/toughness and the stabilisation of the post-peak response; moreover, calibrated constitutive parameters (microplane model) are available to reproduce this behaviour and support the dosage window identified here [70].

In parallel, comparative reviews indicate that natural fibres in geopolymeric matrices improve damage tolerance relative to unreinforced matrices, reinforcing the need to limit the fibre dosage to avoid packing defects [71].

4.2. Hybrid Gel Chemistry Enabled by RBP/RCP (70/30)

The binary precursor RBP/RCP = 70/30 (wt%) favours the co-existence and co-reticulation of N-A-S-H (fed by the amorphous, Al–Si-rich fraction in RBP) with C-(A)-S-H (promoted by Ca carried by RCP) [34–40]. This synergy explains: (i) the stabilisation/intensification of the amorphous hump in XRD from 7 to 28 days without dominant new crystalline phases; (ii) the downshift/broadening of the T–O–T FTIR band consistent with increased polycondensation; and (iii) the denser texture and a tighter ITZ at 28 days in SEM-EDS [34–40,85,90,94]. Prior work on alkali-activated systems with ceramic residues and concrete fines reports similar hybrid gel behaviour and early-age strength gain driven by Ca, together with improved interfacial anchoring for fibre pull-out, while the crystalline assemblage remains broadly comparable across fibre dosages [34–40,85,90,95].

Reference chemical and mineralogical data explain the observed co-reticulation: RCP supplies CaO and cementitious phases (calcium silicates, portlandite), favouring C-(A)-S-H domains, whereas RBP/CBP, rich in SiO_2 – Al_2O_3 , promotes N-A-S-H formation; this combination is consistent with an amorphous hump at $2\theta \approx 20$ – 35° and with denser microstructures [72].

4.3. Microstructure–Property Integration

The spectroscopic (FTIR), diffraction (XRD) and microscopic (SEM-EDS) signatures converge with the mechanical curves: increases in short-range order of the gel and microstructural continuity match the 7→28 day rise in both compressive and flexural strengths [34–40,85,90,97]. Fractographically, ~1.5 wt% exhibits higher crack tortuosity, secondary branching and visible extraction lengths, consistent with a longer post-peak segment and higher work of fracture; at 2.0 wt%, connected porosity and bundles near the crack plane coherently explain the lower MOR despite broadly similar crystalline assemblages across fibre contents [41–47,61,65,69,85,90,97].

Studies with optimized RCP/CBP (≡RBP) replacements report strength gains and densification due to filler effects and residual reactivity (e.g., 25 wt% RCP increases compressive strength by ~15% vs. the control), whereas high replacement levels dilute the reactive binder and increase porosity, degrading the matrix — a finding consistent with our sensitivity to workability and entrapped air as the fibre content rises [72].

4.3.1. Mechanistic Evidence from Sisal-Reinforced Polymer Composites and Relevance to RBP/RCP Mortars

Studies on sisal-reinforced thermoset laminates show that a mild alkaline treatment (~6 wt% NaOH) enhances fibre wettability and micro-roughness, increasing frictional sliding and the work of pull-out; as a result, tensile/flexural/impact strengths improve and the post-peak response stabilises. In contrast, stronger alkali (~10 wt% NaOH) damages the fibre surface and reduces properties [NN]. Although our matrix is inorganic (geopolymer RBP/RCP), the interfacial mechanism is transferable: a denser ITZ—supported here by hybrid N-A-S-H / C-(A)-S-H gel co-reticulation—promotes anchorage and controlled debonding/pull-out, which is consistent with our tortuous crack paths and higher bridge density at ~1.5 wt% sisal (Figures 3–4) and the extended post-peak segment in MOR. Conversely, the drop at 2.0 wt% is coherent with bundle formation and connected porosity, which penalise bending despite broadly similar crystalline assemblages. These mechanistic links motivate future exploration of mild mercerisation (≤ 6 wt% NaOH, short times) for sisal prior to mixing, provided compatibility with the alkaline activator and gel chemistry is verified [98].

4.4. Design and Processing Implications (Addressing Reviewers' Requests)

To sustain the optimum around ~1.5 wt% and improve reproducibility and processing robustness, the data support the following good practices:

- Dispersion control. Add sisal “rain-wise” in the final third of mixing and break up tufts before incorporation to minimise clustering [41–47,61,65].
- Workability & de-airing. Tune rheology (e.g., alkali-compatible plasticiser) and apply short vibration to limit entrapped air and connected porosity, which is particularly critical for MOR [61,65,69].
- Geometry control. Keep fibres short and consistent (~1 cm) to balance bridging efficiency with mix processability [41–52].
- Matrix control. Maintain A/S and curing (T/RH) as specified; the degree of gelation governs ITZ quality and thus the pull-out vs. fibre-rupture balance [34–40,85,90,94].
These measures align with the statistical evidence (large effect sizes η^2 ; significant differences among dosages), the fracture morphologies, and the mechanistic map requested by reviewers: mechanism → statistics → practical guidance (Figures 2–4; Tables 3–4; S1–S4).
- At the rheological level, RCP—and more markedly RBP/CBP—increase water/activator demand and absorption due to their fineness and porosity; therefore, tuning the A/S ratio and applying gentle de-airing are critical to prevent connected porosity and to preserve the optimal fibre window [72].

- Regarding durability, CBP ($\equiv$ RBP) has been reported to show better acid resistance, whereas RCP contributes thermal stability; both effects are consistent with our observations of a denser matrix and a more effective fibre–matrix interface [72].
- Surface treatment (future work). Consider mild mercerisation (≤ 6 wt% NaOH, short exposure) to enhance interfacial anchorage without fibre damage, by analogy with sisal composites; verify compatibility with the N-A-S-H / C-(A)-S-H hybrid gel chemistry of the RBP/RCP system before scaling [98].

4.5. Limitations and Scope

This study used $n = 5$ per condition, focused on 7–28-day performance of mortar-scale specimens under quasi-static loading. Random fibre orientation and length variability add dispersion. While internal validity is strengthened by the agreement among mechanics, XRD/FTIR/SEM-EDS, and fracture morphology, extrapolation to larger elements and aggressive environments (wet–dry cycles, chlorides, sulfates, carbonation) requires further work. Future extensions should quantify the density of active bridges (image analysis on fracture), explore sisal surface treatments (alkaline/silane) and assess durability [41–52,85,90,97].

Evidence on RBP/WCBP-based geopolymers in pastes and mortars (30–100 MPa under optimal conditions) and on concretes using brick powder as an SCM supports the technological feasibility of C&D-waste routes, although the replacement level and curing govern the workability–strength trade-off [99].

4.6. Relevance for Low-Carbon Construction

Identifying a reproducible reinforcement window in RBP/RCP-based geopolymer mortars provides a practical route to lower- CO_2 , fibre-toughened binders derived from construction and demolition waste. The ~ 1.0 – 1.5 wt% range delivers an efficient strength–toughness balance with processing guidance to avoid porosity pitfalls, making these mortars promising for non-structural/semi-structural components where flexural capacity and post-peak behaviour are decisive [22–52,85,90,97].

5. Conclusions

- System viability (RBP/RCP). Geopolymer mortars with RBP/RCP = 70/30 wt%, activated with NaOH/ Na_2SiO_3 /KOH at A/S = 0.485, develop strength from 7→28 days, evidencing a consolidated, workable matrix suitable for low-carbon construction uses.
- Non-linear sisal effect and optimal window. Sisal reinforcement shows a non-linear response with an optimal window at ~ 1.0 – 1.5 wt%:
 - Compression: peak performance at ~ 1.0 wt% versus 0%.
 - Flexure (MOR) and toughness: peak at ~ 1.5 wt%, with a longer post-peak segment and gradual load decay.
- Penalty at 2.0 wt%. At 2.0 wt%, fibre clustering and connected porosity reduce the effective load-bearing section, penalising flexure more than compression, confirming that the net benefit hinges on dispersion and de-airing control.
- Hybrid gel chemistry and densification. The RBP/RCP blend promotes coexistence and co-reticulation of N-A-S-H and C-(A)-S-H gels (Ca contributed by RCP), consistent with: (i) a better-defined amorphous hump in XRD without dominant new crystalline phases; (ii) downshift/broadening of the T–O–T FTIR band indicating greater polycondensation; and (iii) a denser microstructure and tighter ITZ in SEM-EDS at 28 days.
- Fractography–mechanics correlation. At ~ 1.5 wt%, tortuous crack paths, bridging and visible pull-out explain the higher fracture work and post-peak capacity; at 2.0 wt%, bundles/pores near the crack plane align with the observed MOR decrease.

- Robust statistical support. Assumptions were met (Shapiro–Wilk, Levene). Fibre content significantly affected the response (ANOVA, $p < 0.001$; large η^2). Tukey patterns matched the mechanical reading:
 - Compression: 1.0% (a) > 0% (b) = 1.5% (b) > 2.0% (c).
 - Flexure: 1.0% = 1.5% (a) > 2.0% (b) > 0% (c) (7–14 d) and 1.0% = 1.5% (a) > 2.0% = 0% (b) (28 d). (See Tables 3–4 and Supplementary S1–S4.)
- Key practical takeaway. For RBP/RCP matrices under this study's conditions, ~1.5 wt% sisal is recommended when flexure/toughness is critical; ~1.0 wt% is advantageous for compression. Performance depends on proper dispersion, brief de-airing, and ~1 cm fibre length, while keeping A/S and curing constant.
- Contribution to circularity. The system valorises C&D waste (RBP and RCP) and provides a reproducible route to fibre-toughened, lower-CO₂ geopolymer mortars, suitable for non-structural/semi-structural elements where flexural capacity and post-peak behaviour are decisive.

Within the present scope (n = 5, 7–28 days, mortar-scale specimens, quasi-static loading), these conclusions are consistent and traceable to the mechanical results, XRD/FTIR/SEM-EDS, and the statistical analysis. Extrapolation to larger elements and durability under aggressive environments will require dedicated future work.

Supplementary Materials: The following supporting information can be downloaded at the website of this paper posted on Preprints.org.

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Abbreviations

The following abbreviations are used in this manuscript:

AAM — Alkali-Activated Materials

ANOVA — Analysis of Variance

A/S — Activator-to-Solids mass ratio

ATR — Attenuated Total Reflectance (in FTIR-ATR)

BSE — Backscattered Electrons (SEM imaging mode)

C-(A)-S-H — Calcium (Aluminum) Silicate Hydrate gel

C&DW — Construction & Demolition Waste

CI — Confidence Interval (e.g., 95% CI)

CRedit — Contributor Roles Taxonomy
EDS — Energy-Dispersive X-ray Spectroscopy
FTIR-ATR — Fourier Transform Infrared Spectroscopy with ATR
ITZ — Interfacial Transition Zone
KOH — Potassium Hydroxide
MOR — Modulus of Rupture (flexural strength)
Ms — Silicate Modulus of sodium silicate (SiO₂/Na₂O)
Na₂SiO₃ — Sodium Silicate (commercial solution)
NaOH — Sodium Hydroxide
N-A-S-H — Sodium Alumino-Silicate Hydrate gel
OPC — Ordinary Portland Cement
RBP — Recycled Brick Powder
RCP — Recycled Concrete Powder
RH — Relative Humidity
SD — Standard Deviation
SE — Secondary Electrons (SEM imaging mode)
SEM-EDS — Scanning Electron Microscopy with EDS
Shapiro–Wilk — Normality test (W statistic)
Tukey HSD — Tukey’s Honestly Significant Difference post hoc test
wt% — Weight Percent
XRD — X-ray Diffraction.

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