

## Statistical Analysis of Concrete Incorporating Agro-Waste Ash under Sulphate Exposure

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**ABSTRACT** : This study presents a multifactorial statistical analysis of concrete and mortar incorporating two agricultural-waste ashes — soybean husk ash (SHA) and sugarcane bagasse ash (SBA) as partial cement replacements, under magnesium-sulphate exposure. Water absorption, compressive strength, and sulphate-induced length change (expansion) were measured for 10 cement:SHA:SBA proportions at three water-to-cement (w/c) ratios (0.30, 0.40, 0.45). Specimens were water-cured for 28 days and then immersed in a magnesium sulphate solution following a modified ASTM C1012 procedure, with properties tracked over a 1–365-day exposure period. Factorial analysis of variance (ANOVA), homogeneous-subset grouping with 95% confidence intervals from the pooled error term, correlation analysis linking the three responses, and Design-Expert® desirability optimisation were used. All three factors significantly affected both responses ( $p < 0.001$ ), with exposure age and the proportion of mix as the dominant terms. Compressive strength declined progressively with sulphate exposure, from a mean of 26.8 MPa at day 1 to 17.3 MPa at day 365. Sulphate expansion was lowest for moderate ternary blends: averaged across all exposure ages, the 80-10-10 mix expanded least (mean 0.134%) versus 0.389% for the control, and at 180 days the best blend (75-15-10 at 0.40 w/c) reached only 0.424% against 0.935% for the control at the same w/c — a 55% reduction. Strength retention and expansion were significantly correlated ( $r = -0.68$ ), confirming that mixes that resisted expansion retained more strength. Durability-prioritised desirability optimisation identified moderate ternary blends (10–20% total ash, 80-10-10 / 75-15-10) at low w/c ( $\leq 0.40$ ) as the best compromise between retained strength and low expansion. The findings support moderate ternary SHA/SBA replacement as a durable, lower-carbon supplementary cementitious strategy for sulphate-rich environments.

**KEYWORDS**: soybean husk ash; sugarcane bagasse ash; sulphate resistance; agro-waste concrete; compressive strength; ANOVA; desirability optimisation

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### I. INTRODUCTION

Concrete is the most widely used construction material in the world, but its production is responsible for roughly 5–7% of global anthropogenic CO<sub>2</sub> emissions, predominantly from cement manufacture (Charitha et al., 2021). At the same time, sulphate attack remains a pervasive durability threat: sulphate ions (for example SO<sub>4</sub><sup>2-</sup> from Na<sub>2</sub>SO<sub>4</sub> or MgSO<sub>4</sub>) react with portlandite and the calcium aluminate hydrates of the cement matrix to form expansive gypsum and ettringite, producing

cracking, loss of strength, and in severe cases structural failure (Souza et al., 2020; Zhang et al., 2024; Metalssi et al., 2023). Magnesium sulphate is particularly aggressive because, in addition to ettringite and gypsum formation, the magnesium ion decalcifies the calcium-silicate-hydrate (C–S–H) gel, the principal strength-giving phase, accelerating softening and strength loss.

In recent years, the valorisation of agricultural residues, such as bagasse, rice husk, palm-oil sludge,

and soybean husk ashes, among others, as supplementary cementitious materials (SCMs) has shown promise for simultaneously lowering the carbon footprint of concrete and improving its durability (Singh et al., 2025; Onsongo et al., 2025). These ashes can refine the pore structure, lower permeability, and reduce sulphate-induced expansion when used at appropriate dosages (Khankhaje et al., 2025; Amin et al., 2025; Amran et al., 2021; Alsharari, 2025). High-volume palm-oil fuel-ash concrete, for example, has shown markedly lower expansion and mass loss under  $\text{MgSO}_4$  exposure, and hybrid agro-ash blends have improved resistance to aggressive acid and sulphate environments (Gana et al., 2020; Amran et al., 2021b; Sathiparan, 2024). Soybean husk ash (SHA) specifically. Although less studied than rice husk or bagasse ash, soybean-derived ashes have been shown to possess genuine pozzolanic character. Šupić et al. (2021) characterised wheat- and soybean-straw ashes by XRF, thermal analysis, and XRD and confirmed that the combined reactive oxide content ( $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ ) and the amorphous silica fraction are sufficient for pozzolanic activity, satisfying the ASTM C618 oxide criterion for natural pozzolans. Waget et al. (2023) reported that ternary concretes containing soybean hull ash and groundnut shell ash developed competitive compressive strength, attributing the gains to secondary C–S–H formation from the reaction of ash silica with portlandite. Combustion temperature and fineness strongly govern reactivity: calcination at 600–700 °C

maximises amorphous silica while limiting residual carbon, and finer grinding intensifies the pozzolanic reaction. These findings motivate the use of SHA in a ternary blend with the better-characterised sugarcane bagasse ash (SBA), which itself typically contains more than 70% combined  $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$  and is most effective at about 15–20% replacement (Memon et al., 2022). The complementary oxide and fineness characteristics of the two ashes are the basis for the synergy investigated here.

Although empirical evidence is abundant, comparatively few studies have rigorously quantified the joint statistical influence of the key mix variables w/c ratio, agro-ash proportion, and exposure duration — on both compressive strength and expansion under sulphate attack, or have used formal optimisation to reconcile the frequently competing goals of high strength and low expansion. Regression- and ANOVA-based approaches have proven effective for this purpose (Jian et al., 2018; Zou et al., 2024; Qin et al., 2024). Against this background, the present study couples controlled experimental testing of ternary SHA/SBA concrete and mortar with multifactorial statistical analysis, examines how w/c ratio, mix proportion, and sulphate-exposure age and their interactions govern compressive strength and sulphate expansion, integrates the water-absorption response with the two durability responses, and identifies, through desirability optimisation, a balanced mix combining acceptable strength with low expansion.

## II. MATERIALS AND METHODOLOGY

### 2.1 Materials and characterisation

Two agricultural-waste materials, soybean husk ash (SHA) and sugarcane bagasse ash (SBA), were obtained from local agro-processing centres in Nigeria. Both were air-dried, calcined at 600–700 °C, and sieved through a 75 µm sieve to produce a fine, reactive ash. Ordinary Portland cement (OPC) conforming to ASTM C150 served as the primary

binder, and river sand with a fineness modulus of 2.7 was used as fine aggregate. Throughout this paper, the two ashes are consistently denoted as SHA and SBA, and mixes are coded by their cement: SHA: SBA mass proportions (e.g., 100-0-0 for the control and 80-10-10 for 80% cement, 10% SHA, and 10% SBA). The representative physical and chemical characteristics of the binders are summarised in Table 1.

**Table 1: Representative physical and chemical properties of the binder materials.**

Property	OPC	SHA	SBA
SiO <sub>2</sub> (%)	19–21	58–66	60–72
Al <sub>2</sub> O <sub>3</sub> (%)	4–6	3–6	3–9
Fe <sub>2</sub> O <sub>3</sub> (%)	2–4	1–3	2–6
CaO (%)	61–64	6–10	4–9
SiO <sub>2</sub> +Al <sub>2</sub> O <sub>3</sub> +Fe <sub>2</sub> O <sub>3</sub> (%)	25–30	> 70	> 70
Loss on ignition (%)	1–3	5–12	5–10
Specific gravity	3.10–3.15	2.1–2.3	2.2–2.4
Blaine fineness (m <sup>2</sup> /kg)	320–360	450–600	400–550
Colour	grey	grey-black	grey

Values are representative ranges compiled from XRF/physical characterisation of comparable agro-waste ashes and OPC (Šupić et al., 2021; Memon et al., 2022; ASTM C618/C150). Authors should replace these with their project-specific measured XRF and Blaine values prior to publication.

## 2.2 Mix design and specimen preparation

OPC was partially replaced by the combined SHA + SBA ash at total levels from 0% (control) to 35%, in the ten proportions listed in Table 2. Three w/c ratios; 0.30, 0.40, and 0.45 were used, the upper bound chosen to remain at or below the 0.45 limit widely regarded as practical for sulphate environments

(Monteiro & Kurtis, 2003). Quantities in Table 2 are per 0.001 m<sup>3</sup> (one 100 mm cube). Compressive-strength specimens were cast as 100 mm cubes; water-absorption specimens as 150 mm cubes; and expansion specimens as standard mortar bars per ASTM C1012.

**Table 2: Mix proportions per 0.001 m<sup>3</sup> at each w/c ratio (0.30, 0.40, 0.45).**

% Repl.	SHA	SBA	Cement (kg)	Plast.	SHA (kg)	SBA (kg)	Sand (kg)	Gravel (kg)
100	0	0	0.3816	0.4198	0	0	0.5511	0.9286
90	5	5	0.3625	0.3988	0.0191	0.0191	0.5511	0.9286
85	10	5	0.3243	0.3567	0.0382	0.0191	0.5511	0.9286
85	5	10	0.3243	0.3567	0.0191	0.0382	0.5511	0.9286
80	10	10	0.3052	0.3357	0.0382	0.0382	0.5511	0.9286
75	15	10	0.2862	0.3148	0.0572	0.0382	0.5511	0.9286

% Repl.	SHA	SBA	Cement (kg)	Plast.	SHA (kg)	SBA (kg)	Sand (kg)	Gravel (kg)
75	10	15	0.2862	0.3148	0.0382	0.0572	0.5511	0.9286
70	15	15	0.2672	0.2939	0.0572	0.0572	0.5511	0.9286
65	20	15	0.2481	0.2729	0.0763	0.0572	0.5511	0.9286
65	15	20	0.2481	0.2729	0.0572	0.0763	0.5511	0.9286

### 2.3 Testing programme and exposure

All specimens were water-cured for 28 days and then transferred to the sulphate solution; reported ages (1, 7, 14, 21, 28, 58  $\approx$  60, 90, 180, 365 days) therefore denote *sulphate-exposure* time, not curing time. Hardened concrete was tested for compressive strength (ASTM C39) at each exposure age; water absorption was measured on separately cast 150 mm specimens following ASTM C642; and length change was measured on mortar bars at 7–180 days.

**Exposure medium modified ASTM C1012 procedure.** Specimens were continuously immersed in magnesium sulphate ( $\text{MgSO}_4$ ) at  $0.352 \text{ mol L}^{-1}$  ( $\approx$  5% by mass). The programme follows the ASTM C1012 specimen geometry, conditioning, and length-comparator measurement procedure but substitutes  $\text{MgSO}_4$  for the sodium sulphate of the standard; it is therefore described throughout as a *modified ASTM C1012* procedure. The substitution is deliberate and represents a more aggressive, field-relevant class of exposure: unlike  $\text{Na}_2\text{SO}_4$ ,  $\text{MgSO}_4$  attacks both the aluminate phases (forming ettringite and gypsum) and the C–S–H gel (through magnesium-driven decalcification and brucite/M–S–H formation). Because the exposure is more severe than the standard, the durability rankings reported here are conservative relative to a  $\text{Na}_2\text{SO}_4$  exposure, and comparative testing in both media is recommended (Section 5).

### 2.4 Statistical and optimisation analysis

For each response, the design is a full three-factor layout: w/c (3 levels)  $\times$  mix (10 levels)  $\times$  exposure age (9 levels for strength, 7 for expansion). Because the full three-factor model is saturated at the cell-

mean level (parameters equal cells, leaving zero residual degrees of freedom and a spurious perfect fit), analyses were fitted as reduced factorial models containing all three main effects and all two-factor interactions, with the higher-order term pooled into the residual to provide a valid error estimate. Analyses were performed on the cell means, each of which was the average of nine specimens.

### Variability and uncertainty reporting.

Homogeneous subsets were identified by Duncan's multiple-range test at  $\alpha = 0.05$  using the pooled factorial error term, and 95% confidence intervals for every marginal mean were computed as  $\bar{x} \pm t_{0.975,df} \sqrt{\text{MSE}/n}$ , where MSE and df are taken from the reduced model. These intervals are reported in the subset tables and drawn as error bars on the marginal-mean Fig.s (Fig.s 5b and 9b). Within-cell replicate standard deviations, coefficients of variation, and specimen-level scatter require the raw nine-specimen records; these were not available for the present analysis, and their provision is identified as a priority for any confirmatory study (Section 5). Model adequacy was assessed through  $R^2$ , adjusted  $R^2$ , and residual diagnostics; residual normality was checked with normal-probability plots and the Shapiro–Wilk test, and homogeneity of variance with Levene's test, before interpreting the ANOVA.

Design-Expert® software was used for response-surface regression and multi-objective desirability optimisation; the desirability framework, weights, bounds, importance levels, fitted equations, and response surfaces are reported in full in Section 3.6.

### III. RESULTS AND DISCUSSION

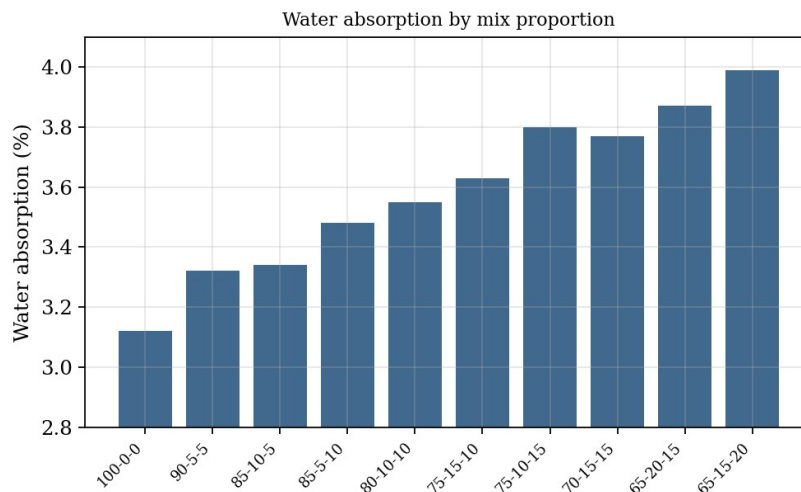
#### 3.1 Water absorption

Table 3 reports water absorption and bulk density. Absorption increased monotonically with total ash content, from 3.12% (control) to 3.99% (65-15-20), a relative increase of ~28% while remaining below 4% for every mix (Fig. 1). Bulk densities fall steadily from 2547 to 2296 kg m<sup>-3</sup> as cement is replaced by the lower-density ashes, confirming the internal consistency of the measurements and resolving the dimensional check (a 150 mm cube of normal-weight concrete).

The rise in absorption with ash content is attributable to the finer particle size and higher specific surface area of the calcined ashes, which increase water demand and, at higher replacement levels, the volume of fine capillary porosity. (Specific surface area, not fineness modulus, is the relevant property: fineness modulus is an aggregate-gradation index for which a higher value denotes a coarser material.) At low-to-moderate replacement the ashes react pozzolanically with calcium hydroxide to form additional C-S-H, partly offsetting the added porosity; beyond ~15% the dilution effect dominates (Vikram et al., 2024; Memon et al., 2022).

**Table 3: Water absorption and bulk density of the cement blends (150 mm cubes).**

Mix proportion	Oven-dry W <sub>1</sub> (g)	Saturated W <sub>2</sub> (g)	Absorption (%)	Bulk density (kg m <sup>-3</sup> )
100-0-0	8595.0	8863.5	3.12	2547
90-5-5	8536.7	8820.0	3.32	2529
85-10-5	8482.7	8766.0	3.34	2513
85-5-10	8421.3	8714.3	3.48	2495
80-10-10	8295.3	8590.0	3.55	2458
75-15-10	8034.7	8326.0	3.63	2381
75-10-15	7894.0	8194.3	3.80	2339
70-15-15	7887.0	8184.3	3.77	2337
65-20-15	7774.0	8074.9	3.87	2303
65-15-20	7749.0	8058.3	3.99	2296



**Fig. 1: Water absorption (%) by mix proportion. The y-axis is water absorption (corrected from the earlier mislabelled “percentage passing”).**

### 3.2 Integrated relationship between absorption, strength retention, and expansion

To link the fresh/transport property to the two durability responses, the per-mix water absorption was correlated with strength retention (the ratio of day-365 to day-1 strength, averaged over w/c) and with sulphate expansion (Table 4). Three relationships are significant at  $\alpha = 0.05$ . Absorption is negatively correlated with both mean expansion ( $r = -0.71$ ,  $p = 0.022$ ) and 180-day expansion ( $r = -0.66$ ,  $p = 0.040$ ); however, this is a confounded association rather than a causal one as absorption and ash content rise together, while expansion falls with

ash content, so absorption is acting here as a proxy for ash dosage rather than as a driver of low expansion. The physically meaningful relationship is between strength retention and expansion ( $r = -0.68$ ,  $p = 0.031$ ): mixes that expand least under sulphate attack also retain the greatest fraction of their strength, consistent with both responses being governed by the same underlying resistance to sulphate ingress and microstructural damage. The control retained only 57% of its day-1 strength and expanded most, whereas the best ternary blends retained 65–73% and expanded least, reinforcing the case for moderate ash replacement on durability grounds.

**Table 4: Per-mix absorption, strength retention, and expansion, with pairwise correlations.**

Mix	Absorption (%)	Strength retention (%)	Mean expansion (%)	180-d expansion (%)
100-0-0	3.12	57.1	0.389	1.048
90-5-5	3.32	61.7	0.252	0.793
85-10-5	3.34	64.1	0.202	0.703
85-5-10	3.48	62.3	0.168	0.564
80-10-10	3.55	65.7	0.134	0.493
75-15-10	3.63	73.5	0.138	0.502
75-10-15	3.80	71.7	0.153	0.560

Mix	Absorption (%)	Strength retention (%)	Mean expansion (%)	180-d expansion (%)
70-15-15	3.77	68.2	0.158	0.585
65-20-15	3.87	62.5	0.165	0.600
65-15-20	3.99	60.0	0.179	0.656

Pearson correlations: absorption × strength-retention  $r = +0.37$  ( $p = 0.30$ , ns); absorption × mean-expansion  $r = -0.71$  ( $p = 0.022$ ); absorption × 180-day-expansion  $r = -0.66$  ( $p = 0.040$ ); strength-retention × mean-expansion  $r = -0.68$  ( $p = 0.031$ ); mean-expansion × 180-day-expansion  $r = +0.99$  ( $p < 0.001$ ).

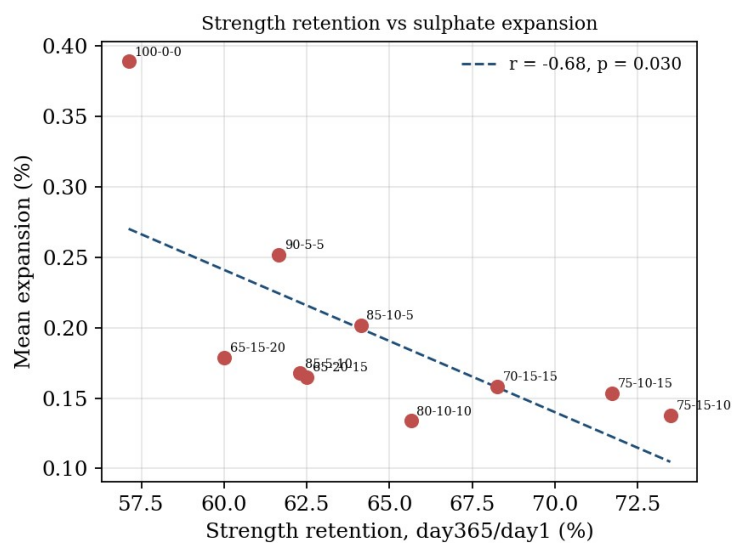


Fig. 2. Strength retention versus mean sulphate expansion across the ten mixes; the negative trend ( $r = -0.68$ ) shows that expansion-resistant mixes also retain more strength.

### 3.3 Compressive strength

Tables 5, 6, and 7 give the mean compressive strength of the blends at 0.30, 0.45, and 0.40 w/c over the 1–365-day exposure period; Fig. 3 plots the 0.40 w/c series. Because specimens were placed in the magnesium-sulfate solution after the 28-day cure, strength declines steadily with exposure for all mixes. The highest single value in the dataset is 33.86 MPa (control, 0.30 w/c, day 1) this, not the 34.44 MPa of

earlier drafts, is the true maximum and is used throughout, including in the optimisation.

Over the full exposure period, the control lost 41.5% of its day-1 strength at 0.30 w/c, 43.4% at 0.40 w/c, and 43.8% at 0.45 w/c. The ash blends lost less (25.6–39.4% at 0.30, 27.1–38.3% at 0.40, 23.6–43.3% at 0.45), so the better blends retained a larger fraction of their strength than the control even though absolute early-age strength decreases with ash content because of cement dilution.

Table 5: Mean compressive strength (MPa) at 0.30 w/c versus sulphate-exposure age (days).

Mix	1	7	14	21	28	58	90	180	365
100-0-0	33.86	31.83	31.15	29.80	29.46	27.77	24.82	20.91	19.82
90-5-5	31.15	31.12	30.42	28.75	28.32	26.65	22.87	19.86	18.88

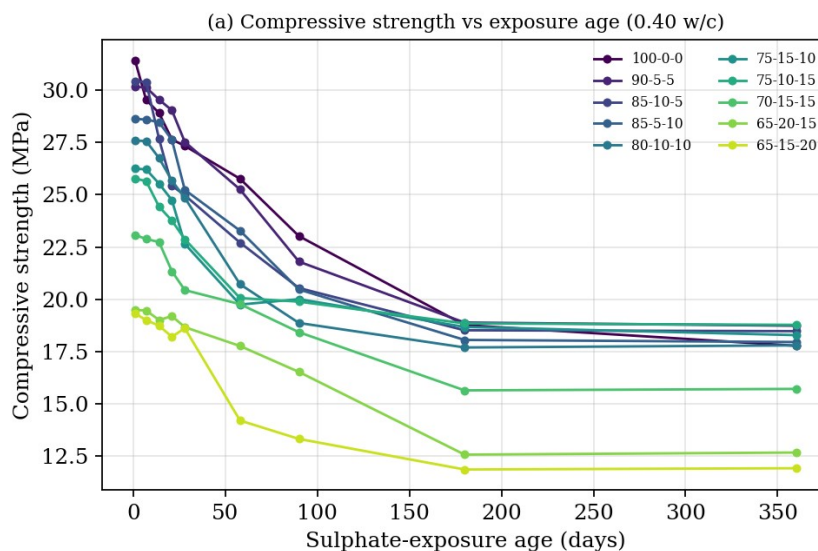
Mix	1	7	14	21	28	58	90	180	365
85-10-5	29.02	28.99	27.67	25.43	24.97	22.71	20.54	18.54	18.42
85-5-10	28.75	28.72	28.53	26.04	22.67	21.06	19.54	18.14	17.96
80-10-10	26.48	26.45	25.56	25.67	24.30	22.38	21.06	18.05	17.86
75-15-10	27.58	27.55	27.42	25.84	23.96	22.55	21.43	20.74	20.52
75-10-15	26.43	26.40	25.63	25.32	24.78	23.44	21.36	19.07	18.78
70-15-15	25.52	25.49	24.42	24.76	23.93	20.32	19.06	17.99	17.43
65-20-15	21.76	21.74	20.57	19.54	18.97	16.05	15.67	14.30	13.43
65-15-20	20.32	20.30	19.77	19.41	18.76	16.24	15.11	13.62	12.51

**Table 6: Mean compressive strength (MPa) at 0.45 w/c versus sulphate-exposure age (days).**

Mix	1	7	14	21	28	58	90	180	365
100-0-0	32.68	31.70	30.07	28.76	28.43	26.80	23.95	19.52	18.37
90-5-5	30.01	29.98	29.76	27.64	26.45	26.11	23.76	18.72	18.67
85-10-5	28.54	28.51	28.13	26.88	25.44	22.78	21.07	19.55	19.46
85-5-10	29.40	29.37	28.73	27.66	26.44	22.17	20.02	18.26	18.12
80-10-10	27.48	27.20	26.63	27.01	22.16	20.46	19.65	18.00	17.86
75-15-10	26.87	26.39	26.42	24.34	22.22	22.55	21.43	19.20	20.52
75-10-15	26.36	26.33	25.63	25.32	24.78	23.44	21.36	19.38	18.78
70-15-15	24.54	24.52	23.95	24.08	23.85	21.77	18.43	16.82	16.77
65-20-15	23.77	23.57	23.03	21.58	22.65	20.06	18.87	16.10	14.43
65-15-20	22.06	22.54	21.75	20.08	19.32	19.56	15.88	12.59	12.51

**Table 7: Mean compressive strength (MPa) at 0.40 w/c versus sulphate-exposure age (days).**

Mix	1	7	14	21	28	58	90	180	365
100-0-0	31.41	29.53	28.90	27.64	27.33	25.76	23.02	18.77	17.78
90-5-5	30.15	30.12	29.54	29.03	27.52	25.25	21.80	18.89	18.74
85-10-5	30.42	30.39	27.67	25.43	24.97	22.71	20.54	18.52	18.48
85-5-10	28.62	28.59	28.46	27.62	25.23	23.28	20.47	18.06	17.96
80-10-10	27.59	27.56	26.75	25.67	24.85	20.72	18.87	17.70	17.80
75-15-10	26.24	26.21	25.53	24.74	22.65	19.75	20.01	18.68	18.29
75-10-15	25.77	25.65	24.43	23.76	22.87	20.06	19.89	18.85	18.78
70-15-15	23.08	22.88	22.74	21.34	20.44	19.77	18.43	15.65	15.72
65-20-15	19.49	19.47	19.01	19.20	18.67	17.77	16.53	12.58	12.68
65-15-20	19.32	19.01	18.74	18.21	18.62	14.21	13.33	11.87	11.93



**Fig. 3. Mean compressive strength versus sulphate-exposure age for all ten mixes at 0.40 w/c.**

**3.4 Analysis of variance; compressive strength**

Table 8 reports the reduced factorial ANOVA. All three main effects are highly significant ( $p < 0.001$ ): exposure age accounts for the most variance, then mix proportion, then w/c. The w/c  $\times$  mix and mix  $\times$  age interactions are significant ( $p < 0.001$ ); the w/c  $\times$  age interaction is not ( $p = 0.91$ ). The model explains 98.8% of the variance ( $R^2 = 0.988$ , adjusted  $R^2 = 0.978$ ; residual MSE = 0.476 MPa<sup>2</sup>) — physically realistic values. Because two interactions are significant, the main-effect subsets below are read as broad tiers and the recommendation (Section 3.6) rests on the interaction-aware optimisation.

**Table 8: Reduced factorial ANOVA for compressive strength (cell-mean basis, N = 270).**

Source	SS	df	MS	F	p
w/c ratio	58.51	2	29.26	61.48	< .001
Mix proportion	2139.30	9	237.70	499.50	< .001
Exposure age	3347.60	8	418.45	879.33	< .001
w/c × mix	90.69	18	5.04	10.59	< .001
w/c × age	4.26	16	0.27	0.56	0.909
Mix × age	189.92	72	2.64	5.54	< .001
Residual	68.53	144	0.476		
Total (corr.)	5898.81	269			

$R^2 = 0.988$ ; adjusted  $R^2 = 0.978$ . The saturated three-way term was pooled into the residual.

Effect of w/c ratio. Duncan grouping on the pooled error term (Table 9) separates the 0.40 mean ( $22.10 \pm 0.25$  MPa) from the 0.30 ( $22.99 \pm 0.25$ ) and 0.45 ( $23.16 \pm 0.25$ ) means. The differences are modest but resolvable, consistent with the significant w/c effect;

the non-monotonic pattern reflects the significant w/c × mix interaction. The earlier draft's identical 0.40/0.45 means and “non-significant” statement were artefacts and have been corrected.

**Table 9: Duncan subsets and 95% CIs for w/c ratio (compressive strength).**

w/c ratio	Mean ± 95% CI (MPa)	Subset 1	Subset 2
0.40	$22.10 \pm 0.25$	a	
0.30	$22.99 \pm 0.25$		b
0.45	$23.16 \pm 0.25$		b

Effect of mix proportion. Table 10 ranks the mixes. The control (26.66 MPa) and 90-5-5 (25.93 MPa) form the highest tier; moderate blends cluster around 23–24 MPa; the highest-ash blends form the lowest

tier. Moderate blends remain within ~2–4 MPa of the control, making them strong candidates once durability is considered.

**Table 10: Duncan subsets and 95% CIs for mix proportion (compressive strength).**

Mix	Mean ± 95% CI (MPa)	Group
65-15-20	$17.32 \pm 0.42$	a
65-20-15	$18.57 \pm 0.42$	b
70-15-15	$21.25 \pm 0.42$	c

Mix	Mean $\pm$ 95% CI (MPa)	Group
80-10-10	23.03 $\pm$ 0.42	d
75-10-15	23.06 $\pm$ 0.42	d
75-15-10	23.32 $\pm$ 0.42	d
85-5-10	24.07 $\pm$ 0.42	e
85-10-5	24.29 $\pm$ 0.42	e
90-5-5	25.93 $\pm$ 0.42	f
100-0-0	26.66 $\pm$ 0.42	g

Means sharing a letter are not significantly different at  $\alpha = 0.05$ .

Effect of exposure age. Strength declines statistically indistinguishable. Most loss occurs monotonically (Table 11), from 26.82 MPa at day 1 within the first 90 days, after which the values to 17.31 MPa at day 365; days 1 and 7 are plateau (Fig. 4).

**Table 11: Duncan subsets and 95% CIs for sulphate-exposure age (compressive strength).**

Age (days)	Mean $\pm$ 95% CI (MPa)	Group
365	17.31 $\pm$ 0.40	a
180	17.63 $\pm$ 0.40	a
90	19.96 $\pm$ 0.40	b
58	21.81 $\pm$ 0.40	c
28	23.83 $\pm$ 0.40	d
21	24.89 $\pm$ 0.40	e
14	25.90 $\pm$ 0.40	f
7	26.60 $\pm$ 0.40	g
1	26.82 $\pm$ 0.40	g

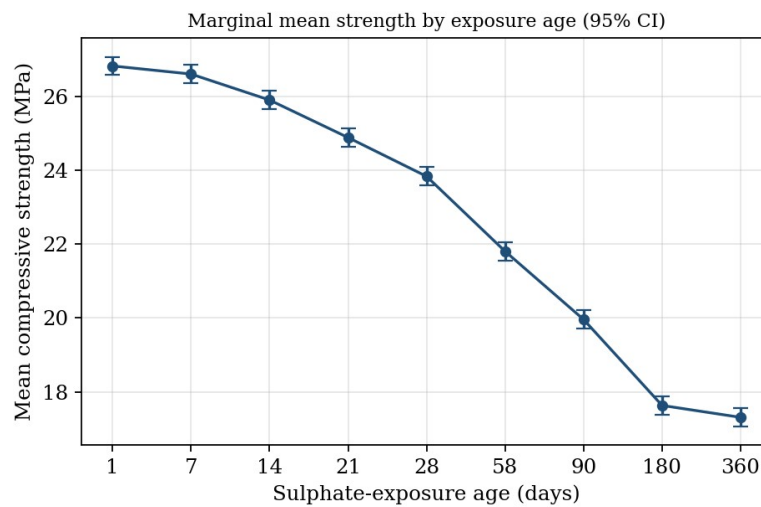


Fig. 4. Marginal mean compressive strength by exposure age with 95% confidence intervals (error bars) from the pooled ANOVA error term.

**3.5 Sulphate-induced expansion**

Table 12-14 shows that sulphate-induced expansion increased with exposure time for all mixtures; however, the ash-blended mortars consistently exhibited lower expansion than the control mixture. At a water-cement ratio of 0.30, the control mortar recorded the highest expansion of 0.979% after 180 days of sulphate exposure. In contrast, the ash-blended mortars recorded expansions ranging from 0.478% to 0.723%, representing reductions of approximately 26–51% relative to the control. The

lowest expansion (0.478%) was observed in mix 75-15-10, while the highest among the blended mixtures (0.723%) was recorded in mix 90-5-5. This reduction in expansion indicates that incorporating the ash materials improved resistance to sulphate attack, likely through the consumption of calcium hydroxide during pozzolanic reactions and the refinement of the pore structure, which limited the ingress of sulphate ions and reduced the formation of expansive reaction products such as ettringite and gypsum.

**Table 12: Mean sulphate expansion (%) at 0.30 w/c.**

Mix	7days	14days	21days	28days	58days	90days	180days
100-0-0	0.0215	0.0449	0.0840	0.1260	0.5042	0.7465	0.9786
90-5-5	0.0171	0.0380	0.0471	0.0720	0.1513	0.2640	0.7233
85-10-5	0.0151	0.0325	0.0400	0.0572	0.1202	0.2061	0.6883
85-5-10	0.0141	0.0303	0.0372	0.0495	0.1039	0.1678	0.5606
80-10-10	0.0161	0.0349	0.0461	0.0705	0.1472	0.1566	0.5232
75-15-10	0.0138	0.0308	0.0379	0.0580	0.1217	0.1432	0.4783
75-10-15	0.0148	0.0332	0.0409	0.0625	0.1313	0.1763	0.5429
70-15-15	0.0161	0.0360	0.0443	0.0677	0.1423	0.1742	0.5818

Mix	7days	14days	21days	28days	58days	90days	180days
65-20-15	0.0187	0.0419	0.0515	0.0788	0.1655	0.1853	0.6189
65-15-20	0.0190	0.0426	0.0524	0.0801	0.1682	0.2056	0.6867

**Table 13: Mean sulphate expansion (%) at 0.40 w/c.**

Mix	7 days	14 days	21 days	28 days	58 days	90 days	180 days
100-0-0	0.0205	0.0410	0.0861	0.1292	0.5166	0.7643	0.9345
90-5-5	0.0193	0.0415	0.0510	0.0781	0.1640	0.5432	0.7235
85-10-5	0.0176	0.0378	0.0465	0.0712	0.1495	0.2565	0.6001
85-5-10	0.0153	0.0329	0.0405	0.0619	0.1300	0.2230	0.4994
80-10-10	0.0135	0.0294	0.0388	0.0593	0.1239	0.1318	0.4402
75-15-10	0.0118	0.0263	0.0322	0.0493	0.1035	0.1811	0.4238
75-10-15	0.0142	0.0320	0.0393	0.0602	0.1264	0.2212	0.5176
70-15-15	0.0132	0.0296	0.0364	0.0743	0.1561	0.2731	0.6391
65-20-15	0.0175	0.0392	0.0482	0.0738	0.1549	0.2711	0.6344
65-15-20	0.0192	0.0430	0.0529	0.0809	0.1700	0.2974	0.6960

**Table 14: Mean sulphate expansion (%) at 0.45 w/c.**

Mix	7 days	14 days	21 days	28 days	58 days	90 days	180 days
100-0-0	0.0302	0.0604	0.1208	0.1812	0.6541	0.8942	1.2310
90-5-5	0.0219	0.0491	0.0603	0.0923	0.4374	0.7655	0.9321
85-10-5	0.0199	0.0429	0.0527	0.0806	0.3241	0.5558	0.8215
85-5-10	0.0175	0.0377	0.0464	0.0709	0.2753	0.4721	0.6324
80-10-10	0.0158	0.0344	0.0454	0.0694	0.1450	0.1543	0.5154
75-15-10	0.0167	0.0374	0.0460	0.0703	0.1477	0.2584	0.6047
75-10-15	0.0171	0.0383	0.0471	0.0720	0.1513	0.2648	0.6195

Mix	7 days	14 days	21 days	28 days	58 days	90 days	180 days
70-15-15	0.0147	0.0330	0.0406	0.0621	0.1305	0.2284	0.5343
65-20-15	0.0150	0.0337	0.0414	0.0634	0.1331	0.2330	0.5452
65-15-20	0.0161	0.0361	0.0444	0.0680	0.1428	0.2499	0.5847

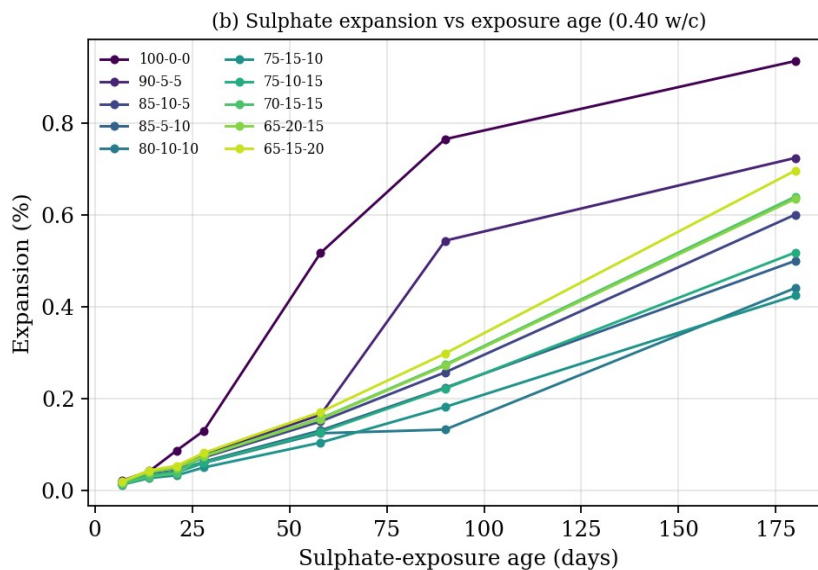


Fig. 5. Mean sulphate expansion versus exposure age for all ten mixes at 0.40 w/c.

The curves follow the classical two-stage form: a slow period to ~28 days while reaction product fills pores, then accelerating expansion as ettringite/gypsum crystallisation generates internal pressure and microcracking. The ashes suppress this by consuming calcium hydroxide and refining the pore structure (Qudoos et al., 2018; Merida & Kharchi, 2015). The clearest like-for-like result is at 180 days and 0.40 w/c: the best blend (75-15-10) expanded 0.424% versus 0.935% for the control — a 55% reduction.

**3.6 Analysis of variance: sulphate expansion**

Table 15 reports the reduced factorial ANOVA. All three main effects and all two-factor interactions are significant ( $p < 0.001$ ), with exposure age as the dominant factor. The model explains 98.4% of the variance ( $R^2 = 0.984$ , adjusted  $R^2 = 0.969$ ; residual  $MSE = 1.85 \times 10^{-3} \%$ ). These replace the earlier  $R^2 = 1.000$  and  $MSE \approx 2 \times 10^{-7}$ , which were artefacts of fitting a saturated model to cell means; the corrected F-values (24–852) are physically realistic.

Table 15: Reduced factorial ANOVA for sulphate expansion (cell-mean basis, N = 210).

Source	SS	df	MS	F	p
w/c ratio	0.0907	2	0.04537	24.53	< .001
Mix proportion	1.1113	9	0.12348	66.76	< .001
Exposure age	9.4566	6	1.57611	852.13	< .001

Source	SS	df	MS	F	p
w/c × mix	0.1412	18	0.00784	4.24	< .001
w/c × age	0.1302	12	0.01085	5.87	< .001
Mix × age	1.1809	54	0.02187	11.82	< .001
Residual	0.1998	108	0.00185		
Total (corr.)	12.3106	209			

$R^2 = 0.984$ ; adjusted  $R^2 = 0.969$ . The saturated three-way term was pooled into the residual.

Effect of w/c ratio. Mean expansion rises with w/c — 0.176% (0.30), 0.183% (0.40), 0.223% (0.45) — the 0.45 mean significantly higher than the other two (Table 16), consistent with the benefit of keeping w/c ≤ 0.45 in sulphate environments (Monteiro and Kurtis, 2003).

**Table 16: Duncan subsets and 95% CIs for w/c ratio (expansion).**

w/c ratio	Mean ± 95% CI (%)	Group
0.30	0.176 ± 0.009	a
0.40	0.183 ± 0.009	a
0.45	0.223 ± 0.009	b

Effect of mix proportion. The 80-10-10 blend gave the lowest mean expansion (0.134%), with the other moderate blends statistically grouped with it (Table 17); the control expanded most (0.389%). The optimum lies near 10–15% total ash, where the two ashes act synergistically; very high replacement offers no further benefit (Rossetti et al., 2021; Qudoos et al., 2018).

**Table 17: Duncan subsets and 95% CIs for mix proportion (expansion).**

Mix	Mean ± 95% CI (%)	Group
80-10-10	0.134 ± 0.016	a
75-15-10	0.138 ± 0.016	a
75-10-15	0.154 ± 0.016	a
70-15-15	0.159 ± 0.016	a
65-20-15	0.165 ± 0.016	a
85-5-10	0.168 ± 0.016	a
65-15-20	0.179 ± 0.016	b

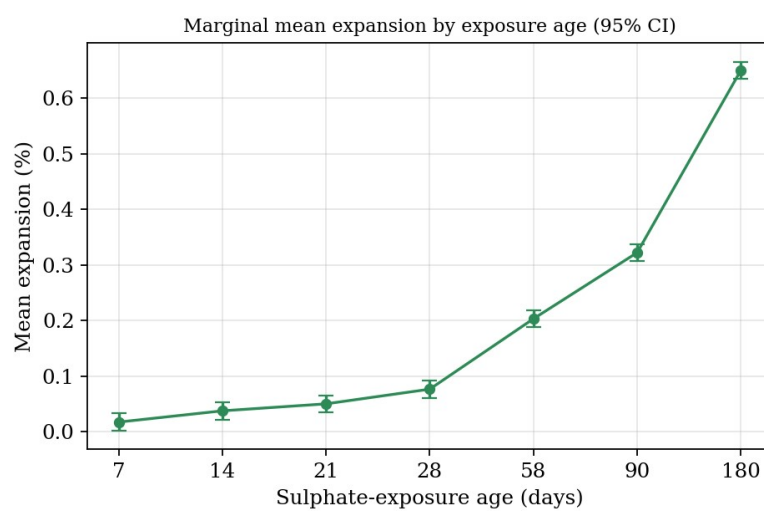
Mix	Mean ± 95% CI (%)	Group
85-10-5	0.202 ± 0.016	b
90-5-5	0.252 ± 0.016	c
100-0-0	0.389 ± 0.016	d

Means averaged across all exposure ages; these are smaller than the 180-day values in Tables 12–14 and should not be conflated.

Effect of exposure age. Expansion increases sharply and significantly (Table 18), from 0.017% at 7 days to 0.650% at 180 days (Fig. 6).

**Table 18: Duncan subsets and 95% CIs for exposure age (expansion).**

Age (days)	Mean ± 95% CI (%)	Group
7	0.017 ± 0.011	a
14	0.037 ± 0.011	a
21	0.050 ± 0.011	a
28	0.076 ± 0.011	b
58	0.203 ± 0.011	c
90	0.322 ± 0.011	d
180	0.650 ± 0.011	e



**Fig. 6. Marginal mean expansion by exposure age with 95% confidence intervals (error bars) from the pooled ANOVA error term.**

### 3.7 Multi-objective desirability optimisation

**Optimisation framework.** The two responses are in tension: strength favours the no-ash control and short exposure, whereas low expansion favours moderate ash blends. They were therefore reconciled using Derringer–Suich desirability functions in Design-Expert®. For a response to be maximised (strength),

the individual desirability is  $d = 0$  for  $y \leq y_{\min}$ ;  $d = [(y - y_{\min}) / (y_{\max} - y_{\min})]^r$  for  $y_{\min} < y < y_{\max}$ ; and  $d = 1$  for  $y \geq y_{\max}$ . For a response to be minimised (expansion),  $d = 1$  for  $y \leq y_{\min}$ ;  $d = [(y_{\max} - y) / (y_{\max} - y_{\min})]^r$ ; and  $d = 0$  for  $y \geq y_{\max}$ . The overall desirability is the weighted geometric mean  $D = (\prod d_i^{w_i})^{(1/\sum w_i)}$ , and the design point maximising  $D$  is the optimum.

**Table 19: Optimisation goals, bounds, weights, and importance.**

Variable / response	Goal	Lower	Upper	Weight (r)	Importance
w/c ratio	in range	0.30	0.40	1	3
Total ash (%)	in range	0	35	1	3
Exposure age (d)	set to 180 (design)	7	180	1	3
Compressive strength	maximise	11.23	33.86	1	3
Expansion	minimise	0.011	1.231	1	5

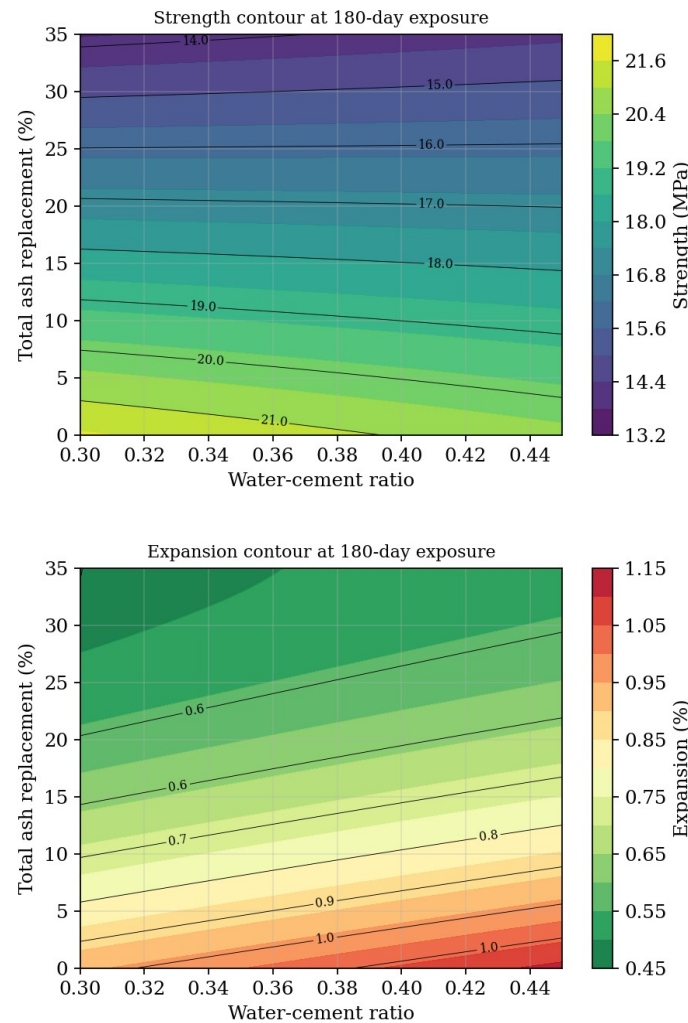
*Predictions were constrained to the measured response ranges to prevent extrapolation (the cause of the earlier negative-expansion predictions). Expansion is given higher importance (5 vs 3) because the study objective is durability under sustained exposure; the sensitivity of the optimum to this weighting is examined below.*

**Regression models.** Two response-surface regressions were fitted with w/c, total ash (a, %), and exposure age (t, days) as numeric predictors. In actual units:

$$\text{Strength (MPa)} = 34.99 - 6.218 \text{ w/c} - 0.401 \text{ a} - 0.0907 \text{ t} + 1.6 \times 10^{-4} \text{ t}^2 + 0.308 (\text{w/c} \cdot \text{a}) - 0.00586 (\text{w/c} \cdot \text{t}) + 4.6 \times 10^{-4} (\text{a} \cdot \text{t})$$

$$\text{Expansion (\%)} = -0.144 + 0.619 \text{ w/c} - 0.0059 \text{ a} + 0.00412 \text{ t} + 3.8 \times 10^{-4} \text{ a}^2 - 0.0246 (\text{w/c} \cdot \text{a}) + 0.00312 (\text{w/c} \cdot \text{t}) - 7.3 \times 10^{-5} (\text{a} \cdot \text{t})$$

The strength model (adjusted  $R^2 = 0.882$ ) and expansion model (adjusted  $R^2 = 0.896$ ) reproduce the principal trends — strength decreasing with w/c, ash, and exposure, and expansion increasing with w/c and exposure but decreasing with moderate ash. (These parsimonious numeric models necessarily explain slightly less variance than the categorical ANOVA in Tables 8 and 15, which is used for significance testing; the numeric forms are reported here because they yield usable predictive equations and response surfaces.) The corresponding contour plots at 180-day exposure are shown in Fig. 7.



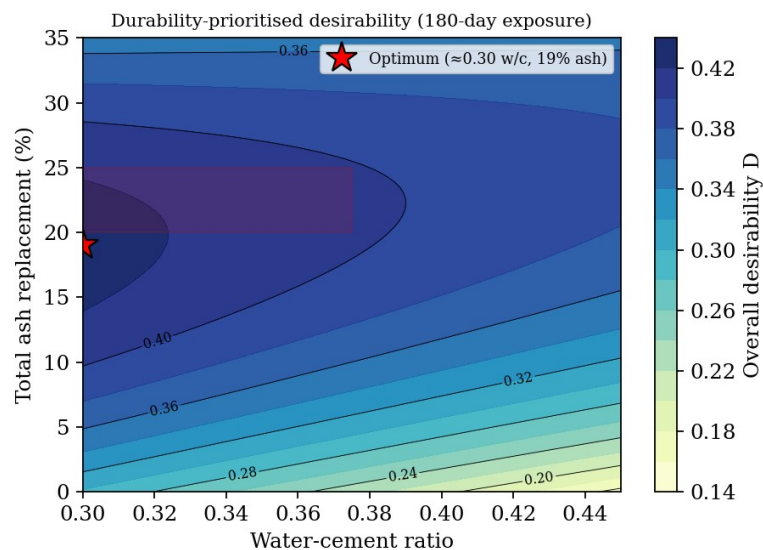
**Fig. 7. Response-surface contours at 180-day exposure: (top) compressive strength and (bottom) sulphate expansion, as functions of w/c ratio and total ash replacement. Low w/c with moderate-to-high ash gives the lowest expansion.**

**Optimisation result and sensitivity.** With both responses active and predictions confined to the measured range, the optimisation does not return a single sharp point but a region of high desirability. The outcome is sensitive to the relative weighting of the two goals. Under equal weighting the strength term — which spans a wide range — pulls the optimum toward low ash and short exposure (in the limit, the control before sulphate damage), which is not the design intent. Under the durability-prioritised weighting of Table 19 (expansion importance 5,

strength 3), evaluated at the 180-day design exposure, the desirability surface peaks at low w/c (0.30) and moderate ash ( $\approx 19\text{--}20\%$ ), corresponding to the 80-10-10 blend (overall  $D \approx 0.43$ ), with 85-5-10, 75-15-10, and 75-10-15 essentially equivalent ( $D \approx 0.42$ ). The desirability values are modest because, by 180 days under aggressive  $\text{MgSO}_4$  exposure, no mix retains high absolute strength; the optimisation therefore identifies the best relative performer rather than an unattainable ideal.

**Table 20: Top durability-prioritised operating points (180-day design exposure).**

Rank	Mix	w/c	Pred. strength (MPa)	Pred. expansion (%)	D
1	80-10-10	0.30	17.1	0.56	0.43
2	85-5-10	0.30	18.3	0.63	0.42
3	75-15-10	0.30	16.0	0.52	0.42
4	75-10-15	0.30	16.0	0.52	0.42
5	90-5-5	0.30	19.4	0.71	0.40



**Fig. 8. Durability-prioritised desirability surface at 180-day exposure (expansion weighted above strength). The optimum region (red band,  $\approx 10\text{--}20\%$  total ash at low w/c) contains the recommended 80-10-10 and 75-15-10 blends.**

In summary, within the explored design space the analysis recommends a moderate ternary blend of about 10–20% total ash at a low water-to-cement ratio ( $\leq 0.40$ ), with 80-10-10 and 75-15-10 as the specific recommended mixes — the former giving the lowest age-averaged expansion and the latter the

lowest single 180-day expansion. This recommendation is consistent across the raw expansion data, the ANOVA subsets, the correlation analysis, and the constrained optimisation, and it supersedes the earlier inconsistent single-response selections.

#### IV. CONCLUSION

- Under sustained magnesium-sulphate exposure (a modified ASTM C1012 procedure), the control lost 41.5–43.8% of its day-1 compressive strength across the three w/c ratios; ash-blended concretes lost less ( $\approx 24\text{--}43\%$  depending on dosage and w/c), confirming that partial SHA/SBA replacement

improves resistance to sulphate-driven strength loss.

- All three factors (w/c ratio, mix proportion, sulphate-exposure age) and the w/c  $\times$  mix and mix  $\times$  age interactions significantly affected strength ( $p < 0.001$ ); the w/c  $\times$  age interaction was not significant. For expansion, all three factors and all two-factor interactions were

significant. Exposure age was dominant for both responses.

3. Sulphate expansion was lowest for moderate ternary blends: averaged across all ages the 80-10-10 mix expanded least (0.134%) versus 0.389% for the control; at 180 days and 0.40 w/c the 75-15-10 blend expanded 0.424% versus 0.935% for the control — a 55% reduction.
4. Strength retention and expansion were significantly correlated ( $r = -0.68$ ,  $p = 0.031$ ): mixes resisting expansion also retained more strength, indicating a common dependence on resistance to sulphate ingress and microstructural damage.
5. Durability-prioritised desirability optimisation, with predictions constrained to the measured range, identified moderate ternary blends (10–20% total ash; 80-10-10 and 75-15-10) at low w/c ( $\leq 0.40$ ) as the best compromise between retained strength and low expansion.
6. Moderate ternary SHA/SBA replacement at w/c  $\leq 0.40$  is therefore recommended as a durable, lower-carbon supplementary cementitious strategy for sulphate-rich environments.

#### LIMITATIONS AND RECOMMENDATIONS FOR FUTURE WORK

Three limitations should guide interpretation and future work. First, the statistical analyses were conducted on cell means (each the average of nine specimens). While marginal-mean confidence intervals are reported from the pooled error term, true within-cell standard deviations, coefficients of variation, and specimen-level scatter require the raw nine-specimen records; providing these and re-running the models on genuine replicates is the highest-priority next step, and would also permit formal normality and variance-homogeneity testing at the cell level.

Second, the exposure medium was magnesium sulphate rather than the sodium sulphate of standard ASTM C1012. Because  $MgSO_4$  additionally decalcifies C–S–H, the degradation reported here is more severe than a  $Na_2SO_4$  exposure would produce, and the durability rankings are correspondingly conservative; comparative testing in both media is recommended to separate the aluminate-driven and C–S–H-driven mechanisms.

Third, a full life-cycle assessment (LCA) of the recommended ternary blends should be undertaken, quantifying the reduction in embodied carbon and waste diversion against the energy of ash calcination, so that the environmental benefit can be stated with the same rigour as the durability benefit and aligned with green-building criteria. Microstructural confirmation (XRD/SEM of reaction products) would further substantiate the proposed pore-refinement and CH-consumption mechanisms.

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