

Performance Evaluation of Cement–Rice Husk Ash Hybrid Binder Systems for Mine Reclamation Backfill: Influence of Binder Composition and Material Type on Mechanical and Physical Properties

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Abstract: This study examined the mechanical and physical performance of marble-based backfill specimens prepared using two distinct binder strategies, cement-only formulations and hybrid cement–rice husk ash (RHA) systems, across four curing periods: 7, 14, 21 and 28 days. Three by-product marble fractions from the Oreke deposit in Ifelodun, Kwara State, Nigeria, were cast into cylindrical specimens (50 mm × 100 mm) using six binder configurations: cement alone at 20%, 40% and 60% by weight, and hybrid blends of 15% RHA/5% cement, 30% RHA/10% cement and 35% RHA/25% cement. Uniaxial compressive strength (UCS), dry density and water absorption were measured at four curing ages with $n = 3$ replicates per mix–age combination ($N = 216$). The 40% cement–aggregate mix (4B) achieved the highest 28-day UCS of 20.13 N/mm², while the 35% RHA/25% cement hybrid (6BH) reached 11.77 N/mm², exceeding the 0.7 MPa threshold for non-critical mine backfill. One-way ANOVA confirmed binder composition significantly affected compressive strength ($F(5,66) = 4.795$, $p = 0.001$), while material type did not significantly influence water absorption ($F(2,69) = 1.090$, $p = 0.342$). The 35% RHA/25% cement blend (mean UCS: 8.26 N/mm²) was statistically comparable to the 20% cement-only mix (8.78 N/mm²), demonstrating the viability of substantial cement replacement without compromising long-term structural performance.

Keywords: Mine Backfill; Rice Husk Ash; Hybrid Binder; Marble Aggregate; Compressive Strength; Pozzolanic Activity.

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I. INTRODUCTION

Backfilling in mines has, over many years, evolved from an elementary void-filling process to an advanced strategy for ground control and environmental management. Besides acting as structural supports, the current functions of backfilling include reducing surface subsidence, stabilising mined-out stopes, and providing a pathway for process waste recycling [1][2]. Within this context, the choice of binder plays a critical role in achieving the minimum required unconfined compressive strength (UCS) required for safe operation. As per the published guidelines, UCS values of at least 0.7 MPa and 4.0 MPa are required in passive, non-entry, and entry backfills, respectively, after 28 days [3][4].

Ordinary Portland Cement (OPC) has been the primary binder in the construction of CPB due to its well-known early strength properties and market availability. However, there are some challenges associated with its use, including its cost and carbon footprint. The cost of cement may account for about 75% of the total CPB cost [5]. In addition, each tonne of clinker produced releases about 1 tonne of CO₂ into the atmosphere, accounting for about 8% of global greenhouse gas emissions [6]. These factors have driven continued research into alternative binding materials to replace conventional OPC.

One such material is the rice husk ash (RHA). It has received significant attention as a supplementary

cementitious material because it contains very high levels of amorphous silica (SiO_2), with concentrations in the husk that may exceed 80% in the calcined state [7][8]. Silica present in the RHA reacts with calcium hydroxide (Ca(OH)_2) liberated during the hydration process of OPC, resulting in the formation of additional C-S-H gel. Laboratory experiments have revealed that replacement levels of RHA, ranging from 15–25% by cementitious material, can achieve UCS values equivalent to those of similar mixes containing only cement while decreasing permeability [9][10]. The above-mentioned characteristics of RHA are very favourable for mine backfilling applications, since in this case the loads act on the backfill after many months, providing adequate time for pozzolanic reactions [11].

Another potential source of materials for backfill application is the marble processing industry. Oreke marble quarry, located in Kwara State, Nigeria, produces large quantities of coarse aggregate, granular fragments, and dust as quarry waste [12]. Such aggregates possess mechanical benefits, including high density, low porosity, and good interlocking capabilities, among other things [13][14]. Their performance in combination with the RHA-OPC hybrid binders has not been investigated previously.

Although cement and RHA mixes have been widely studied as concrete and mortar mixtures, there are very few studies that focus on backfill applications, and even fewer that use marble quarry waste as aggregates and are conducted in the Nigerian context. The objective of this study is to fill this gap by conducting a comprehensive investigation of 18

mixes containing marble and/or RHA at four curing ages, using statistical analysis to separate the effects of binder composition and materials.

II. MATERIALS AND METHODS

➤ Materials

Marble materials were obtained from the Oreke Marble Quarry in Ifelodun Local Government Area, Kwara State, Nigeria (approximately $8^\circ 30' \text{N}$, $4^\circ 45' \text{E}$), belonging to the Precambrian Basement Complex of Western Nigeria [12]. The collected marble was divided into three size groups: fine particles ($< 1 \text{ mm}$), granular materials ($< 8 \text{ mm}$), and coarse aggregate ($< 10 \text{ mm}$). OPC Grade 42.5 from a single production batch was used as a binder. RHA was obtained from rice husks collected at the milling facility in Okeode, Ifelodun LGA, which were combusted at $80\text{--}95^\circ \text{C}$ and sieved through a $75 \mu\text{m}$ sieve to obtain fine pozzolan. Mixing water complied with BS EN 1008:2002 standard. The constant water-to-binder ratio of 0.50 was applied for all mixes.

➤ Mix Design

The experimental work was conducted based on the $2 \times 3 \times 3$ factorial plan having two types of binders (OPC only; hybrid OPC/RHA), three binder contents (20%, 40% and 60% by mix mass) and three marble types (fine, granular and aggregate), resulting in 18 mix codes (Table 1). In the case of hybrid binder series, three binder contents were used: 15% RHA + 5% cement (20% total), 30% RHA + 10% cement (40% total) and 35% RHA + 25% cement (60% total).

Table 1 Mix Design Codes, Binder Formulations and Marble Material Types (C = Cement; RHA = Rice Husk Ash).

Mix Code	Binder Type	Total Binder (%)	RHA (%)	Cement (%)	Material Type
2S	Cement only	20	—	20	Fine particles ($< 1 \text{ mm}$)
2M	Cement only	20	—	20	Granular material ($< 8 \text{ mm}$)
2B	Cement only	20	—	20	Coarse aggregate ($< 10 \text{ mm}$)
4S	Cement only	40	—	40	Fine particles
4M	Cement only	40	—	40	Granular material
4B	Cement only	40	—	40	Coarse aggregate
6S	Cement only	60	—	60	Fine particles
6M	Cement only	60	—	60	Granular material
6B	Cement only	60	—	60	Coarse aggregate
2SH	Hybrid (RHA–cement)	20	15	5	Fine particles
2MH	Hybrid (RHA–cement)	20	15	5	Granular material
2BH	Hybrid (RHA–cement)	20	15	5	Coarse aggregate
4SH	Hybrid (RHA–cement)	40	30	10	Fine particles
4MH	Hybrid (RHA–cement)	40	30	10	Granular material
4BH	Hybrid (RHA–cement)	40	30	10	Coarse aggregate
6SH	Hybrid (RHA–cement)	60	35	25	Fine particles
6MH	Hybrid (RHA–cement)	60	35	25	Granular material
6BH	Hybrid (RHA–cement)	60	35	25	Coarse aggregate

➤ Specimen Preparation and Curing

The component materials were weighed to $\pm 0.1 \text{ g}$ and dry-blended in the sequence: marble $\rightarrow$ RHA (if any) $\rightarrow$ OPC for five minutes in the mechanical mixer. Then, the water was added slowly over the next two minutes of the mixing process. The mixes were cast into the PVC cylindrical moulds ($50 \text{ mm} \times 100 \text{ mm}$) in two equal layers, compacted for 30

seconds on the vibrating table. After that, the moulds were sealed, left undisturbed for 24 hours and then demoulded. The specimens were water cured at $22 \pm 2^\circ \text{C}$ and $80 \pm 5\%$ relative humidity. Three replicates were prepared for each mix and each age, resulting in $N = 216$ specimens ($18 \text{ mixes} \times 4 \text{ ages} \times 3 \text{ replicates}$).

➤ Laboratory Testing

Dry density (kg/m^3) was measured by dividing the mass of oven-dry specimens (at 105°C to constant mass) by the calculated specimen volume. Water absorption (%) was evaluated by immersion of oven-dry specimens in water for 24 hours and expressing the mass increase as a percentage of the oven-dry mass. UCS was determined in accordance with ASTM C39 [21] using a hydraulic compression machine, with a net cross-sectional area of $\pi/4 \times 50^2 = 1963.5 \text{ mm}^2$. One-way ANOVA ($\alpha = 0.05$) and Tukey HSD post hoc test were performed using SPSS v28 to identify the effect of binder composition and material type on the response variables.

III. RESULTS AND DISCUSSION

➤ Compressive Strength Development

The mean UCS values for all 18 mixes across all four curing ages are presented in Table 2. Positive strength development was observed in all tested mixes during the curing period due to the time-dependent nature of OPC hydration and secondary pozzolanic reactions [15][22]. Nevertheless, the rate and extent of strength development varied across mixes, depending on the binder and material type.

For the cement-only mixes, the strongest UCS value at 28 days (20.13 N/mm^2) was achieved by 40% cement–

aggregate mix (4B). It should be noted that the 60% cement mixes (6B and 6M) do not outperform 4B. The 28-day UCS of the 6B mix was only 12.70 N/mm^2 , lower than that of the 4B despite its much higher cement content. One possible explanation for this result is that at 60% binder content, the volume of the coarse particles drops below a critical value for interlocking, thereby disrupting the mechanical framework formed by the aggregate rather than reinforcing it. At 40% binder content, the aggregate particles remain in contact and form a load-carrying framework that is effectively bound by the cement. The above-mentioned result is important for practical applications, as it indicates that increased cement content does not lead to a proportional increase in strength in aggregate-containing mixes.

In hybrid binder mixes, the 35% RHA/25% cement mix shows the best strength performance. The UCS of the aggregate mix (6BH) at 28 days reached 11.77 N/mm^2 , and the UCS of the 30% RHA/10% cement aggregate mix (4BH) reached 12.50 N/mm^2 , exceeding the 4 MPa limit for certain entry-level backfills. The average UCS of the 35% RHA/25% cement mix at 28 days, regardless of material type, reached 9.38 N/mm^2 , being better than the average of 4B mix (8.78 N/mm^2) and showing the fact that the formation of secondary C-S-H gel compensates for the reduced initial cement content [7][8][9][10].

Table 2 Mean Uniaxial Compressive Strength (N/mm^2) by Mix Code and Curing Age
($n = 3$ Per Cell; C = Cement; RHA = Rice Husk Ash).

Mix	Binder	Material	7 days	14 days	21 days	28 days
2S	20% Cement	Fine	0.70	0.70	0.70	1.97
2M	20% Cement	Granular	0.70	0.80	0.70	0.80
2B	20% Cement	Aggregate	4.70	6.60	8.90	7.17
4S	40% Cement	Fine	6.47	9.63	11.73	7.70
4M	40% Cement	Granular	0.97	2.13	1.60	1.83
4B	40% Cement	Aggregate	17.73	17.40	14.33	20.13
6S	60% Cement	Fine	14.40	16.40	18.30	16.60
6M	60% Cement	Granular	11.60	16.23	16.10	8.97
6B	60% Cement	Aggregate	11.00	16.20	10.90	12.70
2SH	15% RHA/5% C	Fine	0.70	1.30	1.17	1.80
2MH	15% RHA/5% C	Granular	2.47	4.50	3.23	4.60
2BH	15% RHA/5% C	Aggregate	2.03	2.57	3.80	4.23
4SH	30% RHA/10% C	Fine	1.20	3.23	5.43	3.37
4MH	30% RHA/10% C	Granular	2.83	4.50	4.93	5.70
4BH	30% RHA/10% C	Aggregate	8.80	8.20	14.50	12.50
6SH	35% RHA/25% C	Fine	3.70	7.40	8.83	9.07
6MH	35% RHA/25% C	Granular	5.20	10.57	9.30	7.30
6BH	35% RHA/25% C	Aggregate	7.10	11.80	11.80	11.77

- Note: Values are Means of $n = 3$ Replicates. Bold Values Indicate Mixes Exceeding 10 N/mm^2 at 28 Days.

➤ Dry Density

Table 3 gives the results of dry density development for all mixes. The dry density values ranged from $1,571 \text{ kg/m}^3$ (4SH at 28 days) to $2,320 \text{ kg/m}^3$ (2B at 14 days). The densest specimens were obtained from cement-only mixes with coarse aggregate, with a 28-day density of $2,213 \text{ kg/m}^3$ for the 4B

mix. Such density is determined by the high bulk density of coarse marble particles and OPC hydration products. The hybrid mixes showed consistently lower early-age densities due to the lower particle density of RHA and slower development of pozzolanic gel C-S-H [16][17]. However, by 28 days, this difference decreased: in the case of the comparison of 35% RHA/25% cement mixes with their cement-only counterparts, density differences at 28 days were 342 kg/m^3 for the fine material (6S vs. 6SH) and 202 kg/m^3 for aggregate (6B vs. 6BH).

Table 3 Mean Dry Density (kg/m^3) by Mix Code and Curing Age (n = 3 per cell).

Mix	Material	7 days (kg/m^3)	14 days (kg/m^3)	21 days (kg/m^3)	28 days (kg/m^3)
2S	Fine	1,906	1,808	1,912	1,810
2M	Granular	1,960	1,897	1,992	1,960
2B	Aggregate	2,283	2,320	2,085	1,972
4S	Fine	1,977	2,002	1,995	1,962
4M	Granular	1,942	1,913	1,937	1,878
4B	Aggregate	2,140	2,180	2,172	2,213
6S	Fine	1,997	1,962	2,010	1,972
6M	Granular	2,030	1,998	2,053	2,042
6B	Aggregate	1,963	2,042	2,060	1,997
2SH	Fine	1,665	1,732	1,880	1,660
2MH	Granular	1,828	1,875	1,933	1,877
2BH	Aggregate	1,852	1,848	1,855	1,932
4SH	Fine	1,633	1,695	1,700	1,571
4MH	Granular	1,898	2,122	1,927	1,900
4BH	Aggregate	1,800	1,898	1,817	1,873
6SH	Fine	1,585	1,728	1,722	1,630
6MH	Granular	1,673	1,741	1,700	1,870
6BH	Aggregate	1,750	1,733	1,813	1,795

➤ Water Absorption Behavior

Water absorption results are tabulated in Table 4. Rather than following a simple monotonic decline, most mixes exhibited a peak at or near 21 days, followed by a measurable reduction at 28 days, a pattern attributed to the transient opening of capillary pore channels during active hydration, which subsequently close as further reaction products accumulate [18]. The 21-day peak was most pronounced in higher-RHA hybrid mixes: 6SH reached 7.69% at 21 days, then fell to 5.20% at 28 days.

The most dramatic improvement was observed in 4SH, which fell from 5.49% at 21 days to just 0.96% at 28 days, the lowest 28-day absorption across the entire hybrid dataset. One explanation is that the fine packing geometry combined with delayed RHA reactivity creates a particularly effective pore-refinement effect at this binder loading. Cement-only aggregate mixes maintained consistently low absorption throughout; 4B averaged 2.07% across all four curing ages, underlining the effectiveness of the OPC-coarse aggregate combination in limiting capillary porosity [19].

Table 4 Mean Water Absorption (%) by Mix Code and Curing Age (n = 3 Per Cell).

Mix	Material	7 days (%)	14 days (%)	21 days (%)	28 days (%)
2S	Fine	2.40	3.13	5.44	0.60
2M	Granular	2.00	1.62	2.22	2.20
2B	Aggregate	2.00	1.40	3.56	2.27
4S	Fine	1.12	2.78	2.17	2.43
4M	Granular	1.57	1.95	3.11	3.57
4B	Aggregate	1.84	1.63	2.43	2.41
6S	Fine	2.21	2.10	3.25	2.28
6M	Granular	1.34	1.35	4.00	2.86
6B	Aggregate	2.71	2.97	3.78	3.29
2SH	Fine	1.43	2.40	2.67	1.44
2MH	Granular	1.42	3.29	3.68	2.30
2BH	Aggregate	2.03	2.53	2.35	2.10
4SH	Fine	3.85	4.55	5.49	0.96
4MH	Granular	1.41	2.33	1.17	2.64
4BH	Aggregate	1.99	2.67	4.52	2.10
6SH	Fine	3.39	1.65	7.69	5.20
6MH	Granular	1.62	1.40	5.70	2.90
6BH	Aggregate	1.11	1.50	4.53	3.26

- *Note: The Elevated Absorption at 21 Days in Several Hybrid Mixes Reflects Transient Capillary Pore Opening During Active Hydration before Pozzolan Pore-Filling at 28 Days.*

➤ Statistical Analysis

One-way ANOVA confirmed that binder composition had a statistically significant effect on compressive strength across the full dataset ($F(5, 66) = 4.795$, $p = 0.001$). The 60% cement formulation returned the highest mean UCS across all material types and curing ages combined (12.31 N/mm^2),

while the 15% RHA/5% cement blend produced the lowest (3.93 N/mm²). The Tukey HSD post hoc analysis (Table 5) identified two overlapping homogeneous subsets at $\alpha = 0.05$. The 60% cement group was statistically distinct from the

lower-performing compositions, while the 35% RHA/25% cement blend (8.26 N/mm²) and the 20% cement formulation (8.78 N/mm²) fell within the same subset, meaning they were statistically indistinguishable from each other.

Table 5a. One-Way ANOVA: Compressive Strength by Binder Composition.

Source	Sum of Squares	df	Mean Square	F	p-value
Between groups	569.589	5	113.918	4.795	0.001
Within groups	1568.006	66	23.758	—	—
Total	2137.595	71	—	—	—

Table 5b. Tukey HSD Post-HOC Test: Mean UCS (N/mm²) by Binder Composition.

Binder Composition	N	Mean UCS (N/mm ²)	Tukey Subset ($\alpha = 0.05$)
15% RHA / 5% Cement	12	3.93	1
40% Cement	12	5.20	1
30% RHA / 10% Cement	12	5.43	1
35% RHA / 25% Cement	12	8.26	1 & 2
20% Cement	12	8.78	1 & 2
60% Cement	12	12.31	2

- *Note: Groups Sharing the Same Subset Number are Not Statistically Distinguishable at $\alpha = 0.05$.*

Engineering-wise, the key conclusion from the statistical analysis is that replacing 60% of the binder weight with RHA does not adversely affect compressive strength, compared to the moderate-dose cement-only binder. In the case of water absorption, ANOVA showed that material type did not have a statistically significant effect ($F(2, 69) = 1.090$, $p = 0.342$), indicating that the binder system dominates the absorption behaviour of the sample. By contrast, the material type had a statistically significant effect on dry density ($F(2, 69) = 5.73$, $p < 0.01$): aggregate-based mixes had an average dry density equal to 1,999 kg/m³ compared to approximately 1,700 kg/m³ of fine-material hybrids with the same binder weight [13].

➤ Practical Implications for Mine Backfill Design

Summarising all results, a hierarchical selection approach becomes apparent. For zones requiring early strength development (≥ 4 MPa at 7 days), 40–60% OPC with coarse aggregates (4B, 6B) yields a 7-day UCS of 11.0–17.7 N/mm². For zones that allow deferred loading and have design controlled by 28-day parameters, the 35% RHA/25% cement-aggregate hybrid (6BH: 11.77 N/mm²) ensures adequate strength at one-quarter the cement content of equivalent high-dose cement-only mixes. Even in low-loading, non-structural zones, a 20% hybrid binder with aggregate (2BH: 4.23 N/mm²) provides a sufficient safety margin over the 0.7 MPa non-entry backfill requirement.

The substitution of 60% of the binder mass in 35% RHA/25% cement blend with RHA reduces the amount of the CO₂ emitted by approximately 55–60% according to the emission intensity of approximately 0.83 kg CO₂/kg for OPC clinker [6] and less than 0.05 kg CO₂/kg for RHA as an agricultural residue [20][23][24]. When the marble quarry waste is used as the main filling matrix, the proposed hybrid system represents a genuinely low-carbon backfill with no infrastructure changes at the site required.

IV. CONCLUSIONS

In the paper, the first systematic laboratory comparison of the properties of cement-only and RHA–cement hybrid binder systems in marble-based mine reclamation backfill was performed. The following conclusions can be made:

- The binder composition had a significant influence on the compressive strength ($F(5, 66) = 4.795$, $p = 0.001$). 35% RHA/25% cement hybrid (8.26 N/mm²) was statistically equivalent to the 20% cement-only formulation (8.78 N/mm²), demonstrating the feasibility of drastically reducing the amount of cement without compromising 28-day compressive performance.
- The geometry of the aggregate skeleton was the major factor affecting the peak strength. The 40% cement-coarse aggregate mix (4B: 20.13 N/mm²) outperformed even 60% cement mixes in both granular and fine-material formulations, demonstrating the leading role of skeleton geometry over the total binder content.
- All aggregates-based hybrid mixes surpassed the 0.7 MPa non-entry backfill criterion at 28 days, with 4BH (12.50 N/mm²) and 6BH (11.77 N/mm²) exceeding 10 MPa, which is enough for various partially load-bearing backfill designs.
- The material type did not have a significant effect on the water absorption ($p = 0.342$) but had a significant effect on the dry density ($p < 0.01$). All the hybrid mixes showed a decrease in water absorption between 21 and 28 days, indicating ongoing pozzolanic pore refinement at advanced curing ages.
- The 35% RHA/25% cement blend with coarse marble aggregate is suggested as the optimal sustainable backfill formulation, providing adequate compressive strength, lower consumption of the cement and significantly reduced carbon footprint without changing infrastructure.

Future work should focus on studying the long-term durability under simulated underground geochemical conditions, quantifying the impact of RHA calcination

temperature on its pozzolanic activity, and developing a machine learning-based approach for rapid mix-design optimisation within the larger combinatorial space of binder and aggregate grading combinations.

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