



Physicochemical and Mineralogical Characterisation of Nigerian Rice Husk Ash and a Portland Limestone Cement-Based Binder Containing 30% Rice Husk Ash

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ABSTRACT

Rice husk ash (RHA) is a potential supplementary cementitious material whose suitability depends on its chemical composition, mineralogical structure and physical characteristics. This study characterises RHA obtained from Imota Rice Mill, Ikorodu, Lagos State, Nigeria, and compares the chemical, mineralogical and physical properties reported for a control Portland limestone cement (PLC) and a binder designated as containing 30% RHA. X-ray fluorescence analysis indicated that the ash contained 91.84% SiO₂, while the combined SiO₂, Al₂O₃ and Fe₂O₃ content was 92.94%. The supplied X-ray diffraction interpretation estimated an amorphous fraction of 88.6% and identified quartz, graphitic carbon, calcite and hematite. Compared with the control PLC, the 30% RHA blend had higher Blaine fineness (4,179 versus 3,992 cm²/g), lower specific gravity (2.80 versus 3.15), higher standard consistency (33% versus 27%) and longer initial and final setting times (230 and 290 min, respectively, compared with 130 and 200 min). Soundness was reported as 0 mm for both binders. These findings show that the investigated ash was silica-rich and that its incorporation was associated with measurable changes in binder properties. They do not independently demonstrate pozzolanic reactivity or improved strength and durability.

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INTRODUCTION

Reducing clinker content is one approach being investigated to lower the resource and environmental burden associated with cement production. Supplementary cementitious materials can partially replace clinker and modify the chemical, mineralogical and physical characteristics of a binder. Their suitability depends on the properties of the particular material, its processing history and the way it is incorporated into the binder.

Rice husk ash (RHA) is produced by burning rice husks, an agricultural by-product. Depending on combustion conditions, cooling, grinding and storage, RHA may contain substantial quantities of silica in amorphous or crystalline form. Amorphous silica can react with calcium hydroxide released during cement

hydration; however, total silica content alone does not establish pozzolanic activity. Mineralogy, fineness, residual carbon, water demand and binder formulation must also be considered.

Previous studies have examined the use of RHA in blended cement and concrete. Ganesan, Rajagopal and Thangavel (2008) investigated strength and permeability, while Habeeb and Mahmud (2010) characterised RHA and its use as a cement replacement material. Research has also considered the influence of ash fineness and content on consistency and setting time (Raheem and Anifowose, 2023). Such findings highlight the importance of characterising each ash source rather than assuming that results from one source apply universally.

This study examines RHA obtained from Imota Rice Mill, Ikorodu, Lagos State, Nigeria. It

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combines XRF chemistry, XRD-based mineralogical interpretation and physical testing of a control PLC and a binder designated as containing 30% RHA. The objectives were to determine the reported oxide composition, describe the identified mineralogical phases, compare selected physical properties, and discuss the implications and limitations for further binder development.

MATERIALS AND METHODS

Materials and binder designation

RHA was obtained from Imota Rice Mill, Ikorodu, Lagos State, Nigeria. Portland limestone cement (PLC) was used as the reference material, and the characterisation programme also included PLC clinker. The comparative binder is referred to throughout as the 30% RHA blend, following the designation in the supplied record. The percentage basis should be confirmed from the original batch records because replacement of clinker, finished PLC and total binder represent different formulations.

Gypsum was also reported as a constituent used in preparing the blended binder. The available record does not unambiguously establish the gypsum dosage basis or the final batch proportions. These details should be checked against the laboratory records before the formulation is reproduced or compared with standard cement compositions.

X-ray fluorescence analysis

X-ray fluorescence (XRF) was used to determine the major and minor oxide composition of the RHA, PLC clinker, control PLC and 30% RHA blend. Reported constituents included SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , K_2O , Na_2O , SO_3 , TiO_2 , P_2O_5 and Mn_2O_3 , together with loss on ignition (LOI). The combined $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ content was calculated by summing the reported percentages of these three oxides.

X-ray diffraction analysis

X-ray diffraction (XRD) was used to identify crystalline phases and examine features interpreted as amorphous material in the RHA,

PLC clinker, control PLC and 30% RHA blend. The reported interpretation considered alite (C_3S), belite (C_2S), tricalcium aluminate (C_3A), tetracalcium aluminoferrite (C_4AF), calcium carbonate, quartz and phases assigned to the RHA.

The supplied record does not state the diffractometer model, radiation source, scan range, step size, scan rate or phase-quantification procedure. Accordingly, phase percentages are treated as reported estimates rather than independently verified quantitative results. In Table 6, the listed crystalline phases for the 30% RHA blend sum to 75.5%; together with the reported 24.5% amorphous phase, these values total 100.0%.

Physical tests

The physical properties reported were Blaine fineness, residue on the 45 μm sieve, specific gravity, standard consistency, initial and final setting times, soundness and LOI.

RESULTS

Chemical composition of the RHA

Table 1. Reported oxide composition of the rice husk ash

Constituent	Reported content (%)
SiO_2	91.84
K_2O	2.54
CaO	1.36
P_2O_5	0.89
MgO	0.81
Al_2O_3	0.68
Fe_2O_3	0.42
Loss on ignition (LOI)	3.48

As shown in Table 1, silicon dioxide was the dominant reported oxide in the Imota Rice Mill RHA, at 91.84%. The combined SiO_2 , Al_2O_3 and Fe_2O_3 content was 92.94%, calculated from the reported values. These results establish a silica-rich bulk composition but do not quantify the proportion of silica that is amorphous or directly measure pozzolanic reactivity.

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The reported LOI was 3.48%. LOI represents mass loss under the specified ignition conditions and may reflect moisture, residual carbon and other volatile constituents; it is not a direct measurement of unburnt carbon. The RHA also contained 2.54% K_2O , which contributes to the alkali content of a binder incorporating the ash. Its practical significance depends on the full binder chemistry and intended application. The composition is broadly consistent with published characterisations of RHA, although source and processing differences can produce substantial variation (Ganesan *et al.*, 2008; Habeeb and Mahmud, 2010; Thiedeitz *et al.*, 2020).

Mineralogical characteristics of RHA

Table 2. Reported XRD phase estimates for the rice husk ash

Reported phase	Estimated content (%)
Amorphous fraction	88.6
Quartz	6.2
Graphitic carbon	2.0
Calcite	1.6
Hematite	1.6

The supplied XRD interpretation, summarised in Table 2, assigned 88.6% of the RHA to an amorphous fraction and identified quartz (6.2%), graphitic carbon (2.0%), calcite (1.6%) and hematite (1.6%). A broad diffuse scattering feature in the approximate 20° – 30° 2θ region is commonly associated with amorphous material, whereas discrete reflections are associated with crystalline phases. Independent confirmation requires the original diffractogram and instrument output.

An amorphous silica-rich fraction may be more reactive than well-crystallised quartz, but the reliability of the reported amorphous fraction depends on the quantification procedure. Quantitative amorphous-content determination generally requires a validated method, such as an internal-standard approach combined with appropriate diffraction analysis (De La Torre *et al.*, 2001).

Chemical composition of PLC clinker

Table 3. Reported XRF composition of PLC clinker

Oxide	Reported content (wt.%)
CaO	65.25
SiO ₂	20.86
Al ₂ O ₃	4.60
Fe ₂ O ₃	4.33
MgO	2.11
SO ₃	0.79
K ₂ O	0.22
Na ₂ O	0.37
TiO ₂	0.32
Mn ₂ O ₃	0.11
P ₂ O ₅	0.09
LOI	0.95
Total	100.00

Table 3 identifies CaO as the predominant oxide in the PLC clinker (65.25%), followed by SiO₂ (20.86%), Al₂O₃ (4.60%) and Fe₂O₃ (4.33%). The supplied oxide values, including LOI, sum to 100.00 wt.%.

Mineralogical composition of PLC clinker

Table 4. Reported XRD phase estimates for PLC clinker

Reported phase	Estimated content (%)
Alite (C ₃ S)	59.4
Belite (C ₂ S)	20.6
Tricalcium aluminate (C ₃ A)	8.2
Tetracalcium aluminoferrite (C ₄ AF)	7.4
Calcium carbonate	3.2
Quartz	1.2

The phase estimates in Table 4 identify alite as the largest reported phase (59.4%), followed by belite (20.6%), C₃A (8.2%) and C₄AF (7.4%). Calcium carbonate (3.2%) and quartz (1.2%) were also listed.



Chemical changes associated with the 30% RHA blend

Table 5. XRF composition of control PLC and the 30% RHA blend

Oxide	Control PLC (wt.%)	30% RHA blend (wt.%)
SiO ₂	19.85	38.53
Al ₂ O ₃	5.30	4.07
Fe ₂ O ₃	3.72	3.20
CaO	61.84	44.34
MgO	2.24	1.84
SO ₃	2.43	1.84
K ₂ O	0.28	0.92
Na ₂ O	0.37	0.29
TiO ₂	0.28	0.35
P ₂ O ₅	0.08	0.34
Mn ₂ O ₃	0.12	0.16
LOI	3.49	4.15
Total	100.00	100.00

The XRF results in Table 5 show that the reported oxide composition changed substantially after RHA incorporation. SiO₂ increased from 19.85 wt.% in the control PLC to 38.53 wt.% in the blend, a rise of 18.68 percentage points (approximately 94.1% relative to the control). This increase is consistent with the introduction of silica-rich RHA. However, XRF reports total oxide composition and does not distinguish reactive amorphous silica from crystalline silica; therefore, the result alone does not quantify pozzolanic reactivity. The oxide comparison is illustrated in Figure 1.

CaO decreased from 61.84 to 44.34 wt.%, a reduction of 17.50 percentage points (approximately 28.3%). The combined SiO₂, Al₂O₃ and Fe₂O₃ content increased from 28.87 wt.% in the control to 45.80 wt.% in the blend, primarily because of the increase in SiO₂ despite reductions in Al₂O₃ and Fe₂O₃. This combined value is descriptive and should not be used alone to claim compliance with a pozzolan specification or to prove pozzolanic activity.

MgO decreased from 2.24 to 1.84 wt.% and SO₃ from 2.43 to 1.84 wt.%. K₂O increased from 0.28 to 0.92 wt.%, while Na₂O decreased slightly from 0.37 to 0.29 wt.%. P₂O₅, TiO₂ and

Mn₂O₃ also increased modestly. These differences describe the measured composition; their implications for hydration, alkali-related reactions or performance cannot be determined from the oxide values alone.

The XRF composition in Table 5 reports LOI values of 3.49 wt.% for the control PLC and 4.15 wt.% for the 30% RHA blend. The physical-property table separately records LOI as 3.50% and 4.11%, respectively. The XRF-reported blend value is retained as 4.15 wt.% in the chemical-composition results; the difference between the XRF and physical-test records should be checked against the original laboratory sheets. LOI does not identify the constituent responsible for mass loss and should not be interpreted as a direct measurement of residual carbon.

Mineralogical changes in the blended binder

Table 6. Reported XRD phase estimates for control PLC and the 30% RHA blend

Reported phase	Control PLC (%)	30% RHA blend (%)
Alite (C ₃ S)	52.6	37.1
Belite (C ₂ S)	18.4	14.0
C ₃ A	8.0	5.8
C ₄ AF	7.2	5.0
Calcium carbonate	10.6	8.1
Quartz	3.2	5.5
Amorphous phase	-	24.5

As summarised in Table 6, the reported alite content decreased from 52.6% in the control PLC to 37.1% in the 30% RHA blend, while belite decreased from 18.4% to 14.0%. C₃A and C₄AF also decreased, from 8.0% to 5.8% and from 7.2% to 5.0%, respectively. These trends may be consistent with dilution of clinker-derived phases, but phase proportions alone do not establish hydration rate or strength development.

Quartz increased from 3.2% in the control PLC to 5.5% in the blend. The blend was also reported to contain a 24.5% amorphous fraction. This fraction may include disordered silica associated with RHA, but it must not automatically be interpreted as 24.5% amorphous

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SiO₂. The chemical identity and reactivity of the amorphous material require supporting evidence. The six crystalline phase values listed for the blend sum to 75.5%, and the reported amorphous fraction is 24.5%, giving a combined reported total of 100.0%. The values are retained as supplied, while the phase assignments and quantification procedure remain to be verified against the original XRD output. The mineralogical findings provide a basis for further investigation but do not independently prove the extent of pozzolanic reaction.

Physical properties of the control PLC and 30% RHA blend

Table 7. Physical properties of control PLC and the 30% RHA blend

Property	Control PLC	30% RHA blend
Blaine fineness (cm ² /g)	3,992	4,179
Residue on 45 µm sieve (%)	11.00	10.34
Specific gravity	3.15	2.80
Standard consistency (%)	27	33
Initial setting time (min)	130	230
Final setting time (min)	200	290
Soundness (mm)	0	0
LOI (%)	3.50	4.11

The table reports LOI values of 3.50% and 4.11% for the control and blend, respectively, as recorded for the physical-property results. These remain distinct from the XRF composition results in Table 5, where the blend LOI is 4.15 wt.%; the two records should be reconciled with the laboratory sheets.

Fineness

Blaine fineness increased from 3,992 cm²/g for the control PLC to 4,179 cm²/g for the blend, an increase of approximately 4.68%. Residue on the 45 µm sieve decreased from 11.00% to 10.34%, a reduction of 0.66 percentage

points. Together, these results indicate a modest shift towards finer material. Blaine fineness estimates specific surface area, whereas sieve residue measures the fraction retained above a specified particle-size threshold; the measures are complementary rather than interchangeable.

Specific gravity

Specific gravity decreased from 3.15 to 2.80, a reduction of approximately 11.11%. The lower value is consistent with incorporating RHA, which generally has a lower particle density than Portland cement constituents. Equal masses of the two binders would therefore occupy different solid volumes, a consideration when proportioning blended cementitious materials. This difference does not by itself demonstrate improved strength or durability.

Standard consistency

Standard consistency increased from 27% for the control PLC to 33% for the blend, a rise of 6 percentage points or approximately 22.22% relative to the control. This indicates a greater water requirement to achieve the prescribed consistency. RHA particle characteristics, including angularity and microporosity, may contribute to increased water demand; a similar effect has been discussed by Habeeb and Mahmud (2010). However, particle morphology and porosity were not independently measured in this study.

Setting time

Initial setting time increased from 130 to 230 min, a rise of 100 min (approximately 76.92%). Final setting time increased from 200 to 290 min, a rise of 90 min (45.00%). The blend therefore took longer to reach both initial and final setting. Possible explanations include clinker dilution and the greater water requirement, but their respective contributions cannot be distinguished from the available measurements. The trend is consistent with the relevance of RHA content and fineness to setting behaviour reported by Raheem and Anifowose (2023).

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Soundness

Soundness was reported as 0 mm for both materials. No measurable expansion was recorded under the reported test conditions. This finding should be interpreted within the limits of the test and does not establish dimensional stability under all exposure or service conditions.

Loss on ignition

The physical-property table reports LOI values of 3.50% for the control PLC and 4.11% for the blend, a difference of 0.61 percentage points. LOI measures mass loss during heating under specified conditions and does not identify the responsible constituents. Because the XRF table reports slightly different values, the relevant laboratory records should be consulted before the final manuscript is submitted.

Implications and limitations

Overall, the results indicate that the investigated Imota RHA had a silica-rich bulk composition and a substantial amorphous fraction according to the supplied XRD interpretation. Relative to the control PLC, the binder designated as containing 30% RHA had higher Blaine

fineness, lower specific gravity, higher standard consistency and longer setting times. These findings demonstrate the value of source-specific chemical, mineralogical and physical characterisation.

The available data do not directly demonstrate pozzolanic activity, secondary calcium silicate hydrate formation, equivalent or improved compressive strength, reduced permeability or enhanced chemical durability. Such outcomes should not be inferred from total silica content or a diffuse XRD feature alone. Further work should include a verified XRD quantification procedure, direct pozzolanic activity assessment, hydration or microstructural analysis, and systematic mechanical and durability testing at defined ages. Reproducibility would also be improved by reporting instrument settings, test standards, replicate information and the exact binder batch proportions.

Figure 1 compares the reported XRF oxide contents of the control PLC and 30% RHA blend. Figure 2 summarises the relative changes in selected physical properties between the two binders. Both figures are inserted below and are cited in the relevant discussion.

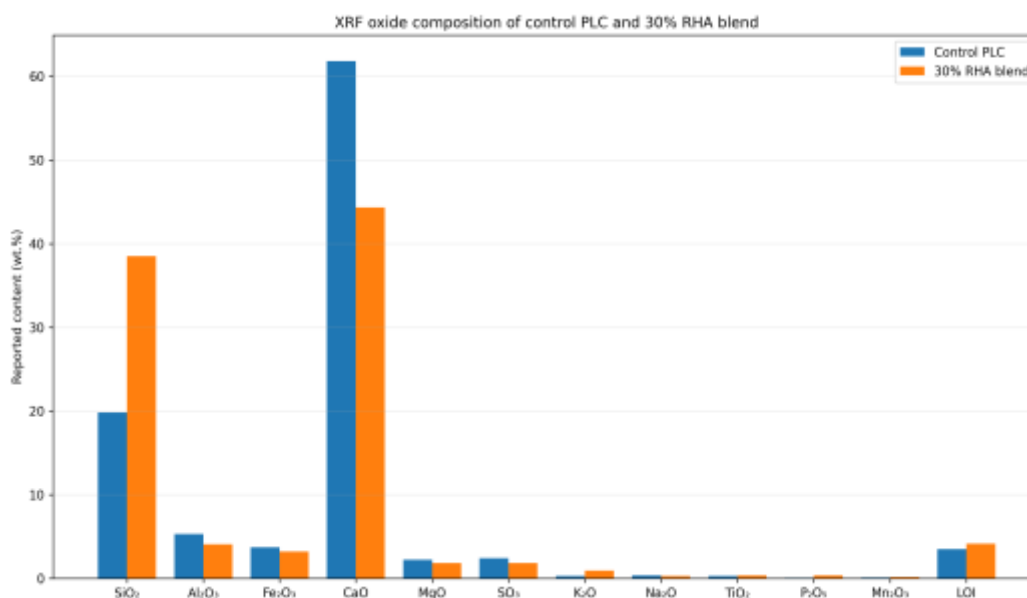


Figure 1. Comparison of the reported XRF oxide contents of the control PLC and the 30% RHA blend. The 30% RHA blend LOI is shown as 4.15 wt.% according to the XRF composition table.

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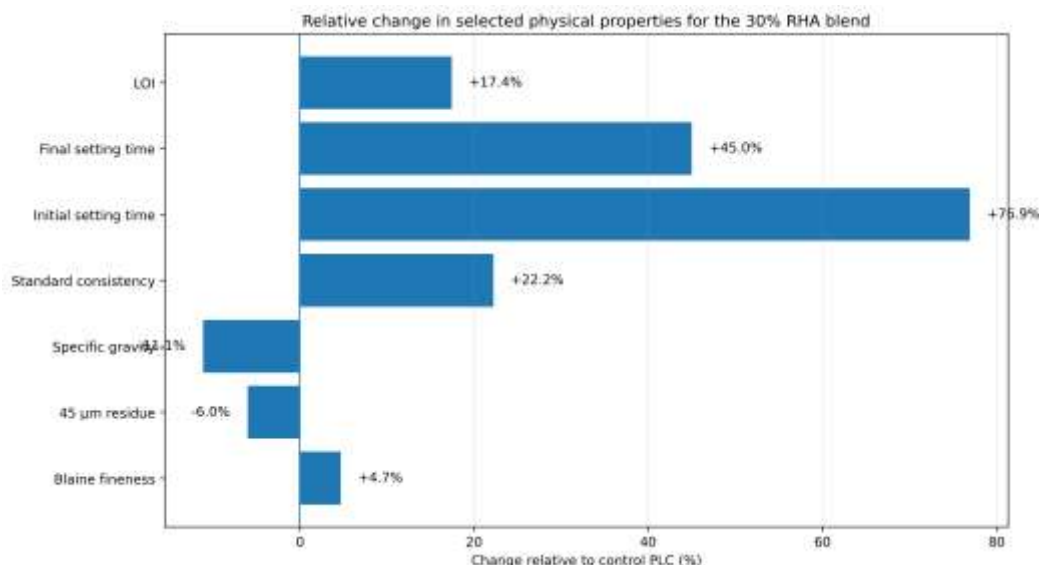


Figure 2. Relative percentage change in selected physical properties of the 30% RHA blend compared with the control PLC. Changes are calculated from the values in Table 7; the plotted LOI change uses the physical-property values (3.50% and 4.11%).

CONCLUSIONS

This study characterised RHA obtained from Imota Rice Mill, Ikorodu, Lagos State, Nigeria, and compared the reported chemical, mineralogical and physical properties of a control PLC and a binder designated as containing 30% RHA. The principal conclusions are as follows:

The RHA contained 91.84% SiO_2 , and the combined SiO_2 , Al_2O_3 and Fe_2O_3 content was 92.94%. These results establish a silica-rich bulk composition but do not independently quantify reactive silica. The supplied XRD interpretation reported an estimated amorphous fraction of 88.6%, alongside quartz, graphitic carbon, calcite and hematite for RHA. The phase assignments and amorphous-content estimate require verification against the original XRD data.

Compared with the control PLC, the 30% RHA blend had higher Blaine fineness (4,179 versus 3,992 cm^2/g), lower specific gravity (2.80 versus 3.15), higher standard consistency (33% versus 27%) and longer initial and final setting times (230 and 290 min versus 130 and 200 min). Soundness was reported as 0 mm for both materials under the test conditions; this should not be extended to a claim of long-term dimensional

stability. The findings justify further evaluation of the ash as a potential supplementary cementitious material but do not establish pozzolanic reactivity or improved strength and durability. These require direct testing and verified mineralogical analysis.

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