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Chemical and Microstructural Behaviour of Rice Husk Ash Blended Cement

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ABSTRACT

This study investigates the chemical and microstructural behaviour of rice husk ash (RHA)-blended cement, focusing on the oxide compositions (XRF) and microstructural evolution at 28 days. RHA sourced from five major rice processing mills in Nigeria was analyzed for its specific gravity and chemical composition, to select the best RHA based on silica content for incorporation into cement at 10–50% replacement levels. XRF analysis confirmed that all RHA samples met ASTM C618 (2019) pozzolanic criteria on the summation of major oxides, with SiO_2 content ranging from 70.11% to 84.14%. The selected RHA sample was inter-ground with OPC-clinker and 5% gypsum to produce RHA-blended cement (M10–M50). Results revealed that increasing RHA replacement led to higher silica (SiO_2) and reduced calcium oxide (CaO) contents, altering the hydration chemistry of cement. The microstructural assessment at 28 days showed that 10–30% RHA replacement improved pore refinement and C-S-H gel formation, reducing calcium hydroxide (CH) content. Optimal densification occurred at 30% RHA (M30), exhibiting a highly compact microstructure with minimal CH residue. However, 40–50% RHA replacements (M40–M50) resulted in excessive porosity and disrupted C-S-H gel connectivity, indicating diminishing structural benefits. Findings suggest that 30% RHA replacement is the optimal threshold, balancing strength, durability, and sustainability at 28 days. This level effectively enhances cementitious properties while significantly reducing clinker consumption and associated CO_2 emissions. However, higher replacement levels compromise hydration efficiency, necessitating careful mix proportioning. Further studies on long-term durability and field performance are recommended. The study reinforces RHA's viability as a sustainable SCM, with practical applications in low-carbon cement production.

INTRODUCTION

Cement is one of the most widely used construction materials globally, playing a crucial role in infrastructure development and urbanization. However, its production is highly energy-intensive and contributes significantly to greenhouse gas emissions. The cement industry alone is responsible for approximately 8% of global CO_2 emissions (IPCC, 2022; Minx *et al.,* 2021), making it one of the largest industrial sources of carbon dioxide. The manufacturing of one ton of Ordinary Portland Cement (OPC) emits between 750–1000 kg of CO_2 (Kamau *et al.,* 2018), primarily due to the high-temperature calcination of limestone ($CaCO_3$) into clinker, the key binding component in cement. Recognizing the urgent need for sustainable solutions, international climate policies, including the United Nations' emission targets, call for a reduction in CO_2 emissions to approximately 475–375 kg per ton of cement by 2030 (Yussuf, 2023).

To achieve these emission reductions while meeting the increasing demand for cement, researchers and industry stakeholders have explored various sustainable alternatives. One widely adopted strategy is the partial replacement of clinker with supplementary cementitious materials (SCMs), which not only reduces CO_2 emissions but also enhances certain properties of concrete. SCMs such as fly ash (FA), silica fume (SF), ground granulated blast furnace slag (GGBS), and metakaolin (MK) have been successfully incorporated into cement formulations to enhance durability and sustainability (Adamu *et al.,* 2024 and Massouni *et al.,* 2025). However, reliance on industrial byproducts like fly ash and slag is becoming increasingly uncertain due to declining coal-fired power plants and steel production (Abdul-Wahab *et al.,* 2021). This has led to increased interest in agro-industrial waste-derived SCMs, particularly Rice Husk Ash (RHA), due to its high silica content, pozzolanic reactivity, and abundant availability in rice-producing regions (Fapohunda *et al.,* 2017; Sharma *et al.,* 2022).

RHA is a byproduct of rice milling, typically discarded as waste, contributing to land pollution and air contamination through open-field burning (Khan *et al.,* 2025). However, controlled combustion of rice husks at 500–800°C produces highly reactive amorphous silica (SiO_2), often exceeding 80% (Fapohunda *et al.,* 2017; Siddique and Cachim, 2018). This silica-rich ash exhibits strong pozzolanic activity, enabling it to react with calcium hydroxide (CH) from cement hydration, forming additional calcium silicate hydrate (C-S-H) gel, which improves strength, durability, and pore structure refinement (Sanou *et al.,* 2019). Furthermore, replacing clinker with RHA can reduce water demand, shrinkage, and heat of hydration, making it a viable SCM for sustainable cement production (Raheem and Kareem, 2017; Tulashie *et al.,* 2021).

Equation 1 shows how Calcium Hydroxide $Ca(OH)_2$ dissociates in water (H_2O) to produce Calcium ions (Ca^{2+}) and Hydroxide ions (OH^-). Equation 2 shows the reaction between the amorphous SiO_2 in RHA and Hydroxide ions (OH^-) to form silicate anions $(SiO_3)^{2-}$. Equation 3 shows that amorphous silica in the RHA can react with $Ca(OH)_2$ in the secondary hydration reaction to form C-S-H gel, which has a floc-like morphology with a porous structure and large specific surface area (Thomas, 2018). The formation of the additional C-S-H in the secondary hydration reaction contributes to both strength development and enhanced durability of concrete (Siddique and Cachim, 2018). Additionally, utilizing RHA in cement production helps address environmental disposal issues, as rice husk waste often contributes to land pollution if not managed properly.

$$Ca(OH)_2 \rightarrow Ca^{2+} + 2OH^- \quad (1)$$

$$SiO_2 + 2OH^- \rightarrow [SiO_3]^{2-} + H_2O \quad (2)$$

$$SiO_2 + Ca(OH)_2 \rightarrow CSH(gel) + H_2O \quad (3)$$

Several studies have examined RHA's influence on cement properties. Research indicates that partial replacement (10–30%) enhances compressive strength, durability, and sulfate resistance, while higher replacement levels (beyond 30%) may lead to increased porosity, reduced early strength, and incomplete hydration (Al-Alwan *et al.,* 2024; Li *et al.,* 2024). The efficiency of RHA as a pozzolan depends on its particle size, silica content, and combustion conditions, which can significantly impact hydration kinetics (Abdulwahab *et al.,* 2021; Fapohunda *et al.,* 2021; Raheem and Anifowose, 2023).

Moreover, studies indicate that the optimum RHA content varies depending on factors such as particle fineness, burning conditions, and replacement levels (Aniceto *et al.,* 2023; Seyed *et al.,* 2017). While 10–30% RHA

replacement has been widely reported as beneficial for strength development, higher replacement levels (40% or more) tend to show reduced early strength but enhanced long-term durability (Liu *et al.,* 2017; Abdulwahab *et al.,* 2021; Al-Alwan *et al.,* 2024). However, despite these advancements, most studies focus on mechanical and durability aspects, with limited investigations into the detailed chemical and microstructural behaviour of RHA-BC. A deeper understanding of oxide composition variations, hydration transformations, and microstructural development is critical for optimizing RHA usage in cementitious systems (Barbhuiya *et al.,* 2023).

This study aims to bridge this gap by evaluating the chemical composition and microstructural characteristics of RHA-blended cement using X-ray fluorescence (XRF) analysis and microstructural observations. The research will compare the oxide compositions of pure RHA, normal OPC, and five RHA-blended cement variants, providing insights into how RHA incorporation alters the chemical reactivity and hydration mechanisms of the resulting cementitious system. By correlating XRF results with microstructural developments, this study will contribute to optimizing RHA replacement levels for sustainable cement production with lower CO_2 emissions, reduced energy consumption, and improved material performance.

METHODOLOGY

This study investigates the chemical and microstructural behaviour of RHA-blended cement by evaluating the oxide compositions (XRF) and microstructural characteristics at 28 days. The methodology includes the procurement, preparation, and characterization of materials, cement production, and microstructural assessment.

Materials

The primary materials used in this study were Rice Husk Ash (RHA), Ordinary Portland Cement (OPC)-clinker, gypsum, fine aggregates (CEN standard sand), and potable water. The OPC-clinker, standard sand, and water were obtained from Lafarge, Ewekoro, Ogun State, Nigeria, where the experiments were conducted. The OPC clinker was sourced from Lafarge, Ewekoro, Nigeria. Rice Husk Ash (RHA) was obtained from five major rice processing mills in Nigeria, identified as SP1, SP2, SP3, SP4, and SP5. The RHA was collected from the dumpsites of these mills, ensuring minimal contamination, and stored in airtight polythene bags to maintain its original state for chemical characterization. Gypsum, sourced from Lafarge, was maintained constant at 5% content across all cement mixes. The Fine aggregate used was CEN standard sand to the specification BS EN 197-1 (2021) for the preparation of mortar for microstructural analysis after 28 days. Portable water sourced from the Physical Laboratory of Lafarge was used for mixing and curing all mortar specimens.

Preparation of RHA Samples

The raw RHA samples underwent milling and sieving to achieve particle sizes below 75 µm, enhancing their pozzolanic reactivity. The process involved drying the collected RHA at 105°C for 24 hours to remove moisture and was followed by grinding in a ball mill to improve fineness. A targeted fineness of 75 µm particle sizes was achieved through sieving (No. 200 ASTM sieve) to obtain uniform particles. The processed RHA samples were then labelled as (SP1–SP5) for further testing. The objective was to select the RHA sample with the highest reactive silica content for cement blending.

Characterization of Materials

To determine the oxide compositions of the cement materials, an XRF-based chemical characterization approach was used. However, it will be incomplete not to determine the Loss on Ignition (LOI) and specific gravity, both of which are classified as physical characterization.

Chemical Characterization of RHA and OPC-Clinker

The oxide compositions of the five RHA samples and OPC-clinker were determined using XRF spectroscopy (Model: EWL-71A-0010) at Lafarge Cement Wapco. The analysis followed ASTM E1621 (2021) standards, ensuring accurate elemental quantification.

Physical Characterization

The Specific Gravity was measured using the Le Chatelier Flask Method according to ASTM C188 (2014) to determine the density and reactivity of RHA in comparison with OPC. The LOI was conducted at 950°C in a muffle furnace, following ASTM D7348 (2021) to assess the volatile content present in the materials.

Production of RHA-Blended Cement

The RHA sample with the highest reactive silica content was selected and used to produce RHA-blended cement (RHA-BC) by inter-grinding it with OPC-clinker and 5% gypsum, which was kept constant. The production process includes batching and inter-grinding by ball-milling. The batching of the six cement blends was prepared by inter-grinding OPC clinker with varying RHA percentages (10–50%) while maintaining 5% gypsum in all formulations as presented in Table 1.

Table 1: Batch Plan for OPC and RHA-Blended Cement

Mix ID	RHA Content (% Wt.)	OPC-Clinker Content (% Wt.)	Gypsum Content (% Wt.)	Total-Content (% Wt.)
M0	0	95	5	100
M10	10	85	5	100
M20	20	75	5	100
M30	30	65	5	100
M40	40	55	5	100
M50	50	45	5	100

In the milling process, the mixtures were milled using a pilot-scale ball mill (BB27-7170/13) at Lafarge Cement, Ewekoro, with a grinding time of 60–90 minutes, depending on clinker grindability. The ball sizes of the mill are 90 mm (8.2 kg), 80 mm (10.0 kg), 60 mm (10.0 kg), 50 mm (14.0 kg), and 30–40 mm (12.8 kg) while the mill speed is 44 RPM with 95±1% grinding efficiency. The control OPC (M0) and RHA-BC (M10–M50) were produced to achieve similar fineness levels, ensuring comparable performance.

Characterization of RHA-Blended Cement

The chemical characterization of the RHA-BC was XRF-based and targeted at determining the effect of RHA replacement on the chemical composition of the blended cement. The oxide compositions of OPC (M0) and RHA-BC (M10–M50) were determined via XRF spectroscopy, following ASTM E1621 (2021). Some of the key oxides analyzed included SiO_2, CaO, Al_2O_3, Fe_2O_3, MgO, and SO_3 to assess chemical changes due to RHA replacement.

Microstructural Assessment

To carry out the SEM analysis, mortar production and curing must be carried out within 28 days. The subsection below describes the mortar preparation and microstructural imaging that was carried out.

Mortar Specimen Preparation

Mortar specimens in the size of 40 x 40 x 160mm prism were produced for all cement samples including that of control at a 1:3 (binder-to-sand) ratio, following BS EN 196-1 (2005) and a consistency-based water-to-binder ratio was adopted for the RHA-BC (Bhowmik and Pal, 2022), while OPC mortar (M0) had a fixed 0.5 water/cement ratio as described by BS 5628-1 (1992). All mortar specimens were cured in water for 28 days at a controlled temperature of 20 $^{\circ}$C before testing.

Microstructural Imaging

The microstructural imaging was performed using a Hitachi SU 3500 SEM at the Department of Chemical and Petroleum Engineering, Petroleum Reservoir Laboratory in Afe Babalola University, Ado-Ekiti (ABUAD) to study the morphology of the sample specimens. The focus was on C-S-H gel formation, pore structure and density, unreacted particles and calcium hydroxide presence. The analysis was conducted on all mortar samples produced from the different types of cement after 28 days of curing.

RESULTS AND DISCUSSIONS

This section discusses the results of laboratory tests carried out in the course of studying the chemical and microstructural behaviour of OPC and RHA-BC mortar after 28 days of curing in water by complete immersion.

Chemical Properties of RHA

This section evaluates the specific gravity and oxide compositions (XRF analysis) of RHA sourced from five major rice processing mills in Nigeria, highlighting its potential as an SCM.

Specific Gravity of RHA Samples

The specific gravity of the RHA samples ranged between 2.19 and 2.27 g/cm³, which is lower than the 3.105 g/cm³ typically reported for OPC in Table 2. This lower density results in a higher volume per unit mass, influencing workability, porosity and durability in blended cement. A lower specific gravity increases cement paste volume, enhancing workability and internal curing. However, higher porosity in RHA affects water demand and early strength development, requiring optimized mix proportions to balance strength and durability. The observed values align with previous studies like Hussin and Parasuraman (2018) and Tulashie *et al. (*2021), suggesting that RHA's specific gravity is consistent.

XRF Analysis of RHA Samples

The oxide compositions of RHA samples as shown in Table 3, confirm SiO_2 as the dominant component, ranging from 70.11% (SP1) to 84.14% (SP2). ASTM C618 (2019) specifies that the combined SiO_2, Al_2O_3 and Fe_2O_3 must exceed 70% for a material to qualify as a pozzolan, all samples exceed this threshold, confirming their suitability for cementitious applications.

High SiO_2 content enhances pozzolanic reactivity by reacting with $Ca(OH)_2$ to form additional C-S-H gel and also improves later-age strength and durability (Das *et al.,* 2022). While the highest SiO_2 content in SP2 (84.14%) suggests it has the strongest pozzolanic potential, it also recorded the lowest LOI value of 8.22%, which increases

its conformity with the ASTM C 618 (2019), hence the most suitable RHA sample. The high LOI values, which exceed 10%, suggest unburnt carbon, which can affect workability and early hydration (Chen *et al.,* 2019).

Table 2: Specific Gravity Results of the Selected RHA

S/N	Sample	Specific Gravity (g/cm^3)
1	SP1	2.25
2	SP2	2.19
3	SP3	2.25
4	SP4	2.23
5	SP5	2.27

Table 3: XRF Analysis of RHA Samples (% by weight)

Compounds	SP1	SP2	SP3	SP4	SP5
SiO_2	70.11	84.14	78.65	77.51	78.05
Al_2O_3	0.54	3.35	0.47	0.23	0.27
Fe_2O_3	0.49	1.02	0.45	0.17	0.31
CaO	0.3	0.43	0.42	0.14	0.24
LOI	26.9	8.22	18.76	20.74	20.08

Chemical Composition of OPC-Clinker and RHA-Blended Cement

The chemical composition results of OPC clinker and RHA-BC variants were assessed by an XRF-based test approach and the results are hereby discussed.

XRF analysis of OPC-Clinker

The oxide composition of OPC-clinker as presented in Table 4 conforms to BS EN 197-1 (2021) specifications. SiO_2 (21.04%), Al_2O_3 (3.40%), Fe_2O_3 (4.20%), and CaO (67.07%) dominate the clinker composition. These results confirm the suitability of the clinker for cement production.

XRF analysis of OPC and RHA-Blended Cement

The oxide compositions of OPC (M0) and RHA-blended cement (M10–M50) presented in Table 5 showed a gradual increase in SiO_2 and a corresponding decrease in CaO from 62.06% (M0) to 30.91% (M50) as RHA replacement increases, confirming clinker dilution. Likewise, the LOI was found to increase with RHA, which is attributable to the high organic content of RHA.

Table 4: XRF Analysis of OPC-Clinker

Compounds	**Content (%)**	**EN 197-1 (%)**
SiO_2	21.04	19-23
Al_2O_3	4.35	2-6
Fe_2O_3	4.61	4-8
CaO	65.26	60-67
LOI	0.96	0-5

While the increased SiO_2 content in the RHA-BC M50 (21.43-53.20%) for M0-M50 improves pozzolanic activity and enhances long-term strength, the reduction in CaO content affects early strength gain which will require optimized curing conditions to meet up with strength requirements (Wang *et al.,* 2024). The increasing LOI is consistent with the high LOI of raw RHA (8.22%).

Table 5: XRF Analysis of OPC and RHA-Blended Cement

Compounds	**Mo**	**M10**	**M20**	**M30**	**M40**	**M50**
	Content (%)					
SiO_2	19.93	21.43	33.36	38.19	46.52	53.20
CaO	62.06	56.18	50.32	43.94	37.45	30.91
Fe_2O_3	3.10	2.92	2.62	2.40	2.20	2.01
Al_2O_3	5.16	4.83	4.69	4.47	4.30	4.20
LOI	3.50	3.70	3.94	4.11	4.46	5.37

Microstructural Assessment of OPC and RHA-Blended Cement Mortars

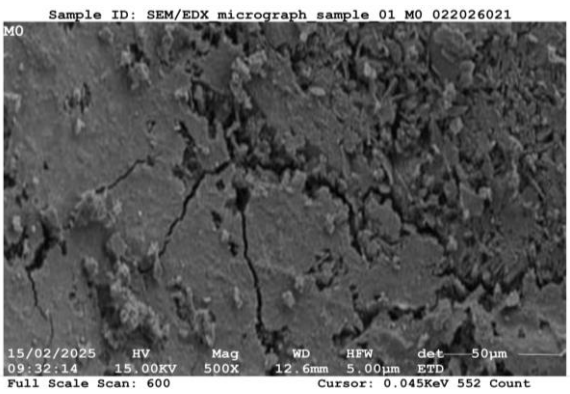
This section discusses the observations from the micrographs of OPC and the variants of RHA-BC mortars produced by partially replacing OPC-clinker with RHA after curing in water for 28 days.

Microstructural assessment

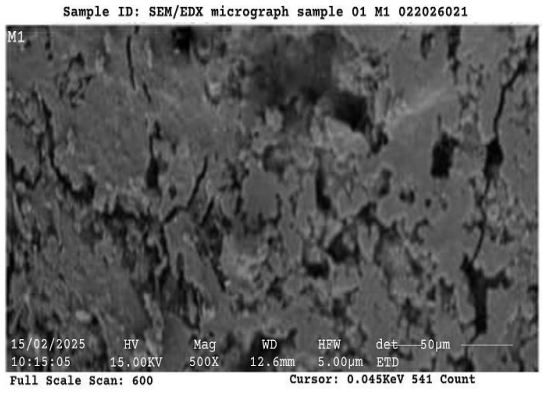
Figure 1 (a–f) presents the SEM micrographs of OPC (M0) and RHA-BC mortars (M10–M50) after 28 days of curing. The M0 mortar representing the OPC as control is given in Figure 1a, where a dense structure with large CH crystals is visible. The presence of large CH crystals corroborates limited or no pozzolanic activity, as expected, hence minimal porosity. Pores are present, however, the matrix remains strong. The M10 mortar, represented by the micrograph in Figure 1b, depicted improved particle packing with increased C-S-H gel formation, hence, reduced CH crystals. The pore structure is refined compared to M0; however, there is a presence of microcracks. The micrograph of the M20 mortar presented in Figure 1c revealed a denser matrix as a result of

further CH consumption as a result of pozzolanic reaction, leading to C-S-H gel formation. The observed pore structure is better than that of M10, which informs enhanced strength and durability. The micrograph of M30 showed a good C-S-H gel formation, indicating an improved pozzolanic formation, as can be observed in Figure 1d. There is minimal CH crystal presence, which also suggests a strong ongoing pozzolanic reactivity. In addition, a more refined pore structure is observed, which has been reported to enhance durability properties. The micrograph of M40 in Figure 1e indicates increased porosity in some areas, which will diminish the strength benefits. This may suggest excessive RHA, which reduces hydration efficiency. Unlike the micrograph of other RHA-BC mortars, that of M50 displayed in Figure 1f indicated a highly porous and disconnected microstructure, indicating a reduced hydration efficiency. This will diminish the strength potential and it suggests an over-replacement of clinker in the RHA-BC.

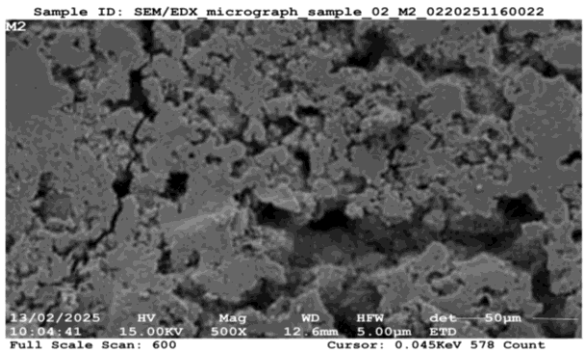
Overall, practically, the RHA-BC mortars containing 10-30% RHA partially replacing OPC-clinker displayed C-S-H gel formation and pore refinement after 28 days of curing, making the durable cementitious material. The 20% replacement level showed the optimal properties after which pozzolanic reactivity reduced as described by the less cohesive microstructure as a result of diminishing C-S-H gel formation noticed from 40% and 50%, suggesting 20% RHA as the optimal practical upper limit for strength development at 28 days. However, 30% showed promising microstructural behaviour, but not as good as those of 10 and 20% at 28 days. In closing, while the M10–M30 showed improved pore refinement, higher C-S-H content, and reduced CH, leading to enhanced mechanical and durability properties, the M40 and M50 mortars exhibited increased porosity, indicating a threshold beyond which RHA content negatively impacts cement structure. These observations are tallied with Fapohunda *et al.* (2017), Raheem and Kareem (2017), Abiodun and Jimoh (2018) and Tulashie *et al.,* (2021).



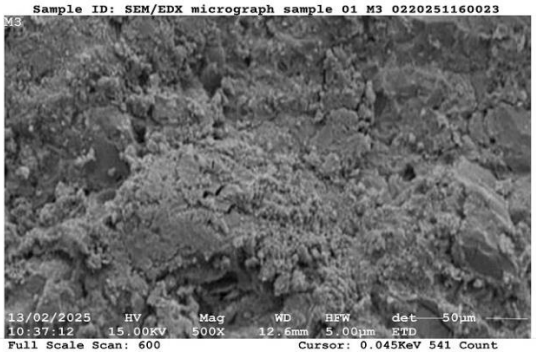
(a-M0)



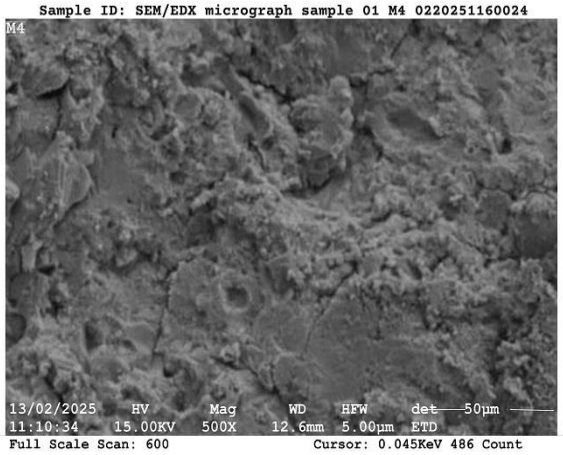
(b-M10)



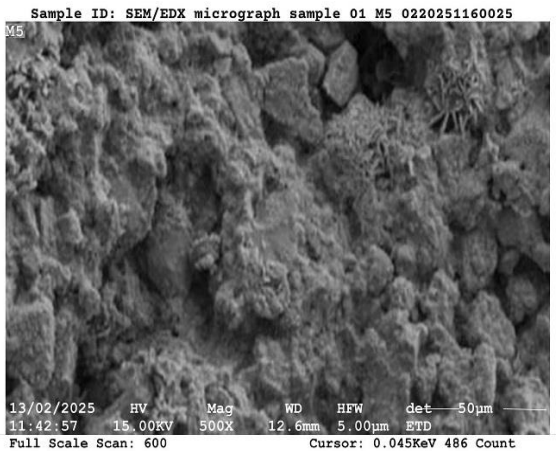
(c-M20)



(d-M30)



(e-M40)



(f-M50)

Figure 1 (a-f): Micrographs of OPC and RHA-BC Mortar after 28 Days of Curing

CONCLUSIONS AND RECOMMENDATIONS

The study investigates the chemical and microstructural behaviour of RHA-BC, focusing on the oxide compositions (XRF) and microstructural evolution (SEM) at 28 days. The findings highlighted the pozzolanic potential of RHA and its impact on cement hydration, strength development and durability. The conclusions drawn and recommendations from the study are given;

Conclusions

SiO_2 content in RHA from five selected Nigerian sources ranged from 70.11 to 84.14%, exceeding the ASTM C618 (2019) pozzolanic threshold. Higher RHA content led to increased SiO_2 and decreased CaO in RHA-BC, influencing hydration and strength gain. RHA replacement for OPC-clinker enhanced C-S-H gel formation, refining pore structure and reducing CH presence and the optimal densification was observed at 20%, where a well-packed microstructure formed with reduced CH at 28 days. The pozzolanic activity of RHA contributes to enhanced durability, making it suitable for long-term cement applications. Excessive RHA content occurred at RHA content greater than 30% as evidenced by increased pore structure and of course, possible reduced strength limiting its practical application in structural concrete.

Recommendations

I. RHA is recommended for eco-friendly cement production as it can replace up to 30% of clinker with good strength, durability and microstructure properties comparable to OPC at 28 days, while the 40 and 50% RHA blends are not recommended for short-term construction applications as they may require extended curing time to enhance hydration.

II. Further studies should investigate the long-term microstructural properties of RHA-blended cement mortar at later curing ages. This will help understand the behaviour of both low and high RHA content concerning strength, durability and performance under aggressive conditions. Such research will provide valuable insights for the construction industry on best practices for using RHA as an SCM in cementitious products.

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