Vol. 06, No. 02, pp. 57 –69 (2025)
ISSN: 2709-6904

JOURNAL OF CIVIL ENGINEERING FRONTIERS

www.jocivilef.org

Durability Performance of Corn Cob Ash–Coconut Shell Ash Concrete Exposed to Simulated Oil Refinery Wastewater

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Abstract

The durability of concrete in chemically aggressive environments, such as oil refinery wastewater systems, remains a critical challenge in infrastructure development. This study investigates the performance of concrete modified with binary agro-industrial pozzolans—corn cob ash (CCA) and coconut shell ash (CSA)—as partial replacements for ordinary Portland cement (OPC). Six concrete mixes were prepared with 0–30% combined SCMs by weight of binder. The specimens were exposed to 2% sulfuric acid (H_2SO_4) and 5% sodium sulfate (Na_2SO_4) for up to 90 days. Key parameters evaluated include compressive strength, mass loss, water absorption, sorptivity, density, and visual deterioration. The results revealed that the 15% replacement mix (M2) achieved optimal performance. It retained more than 94% of the control's compressive strength at 90 days and recorded the lowest mass loss and permeability under both acid and sulfate attack. SCM addition contributed to secondary pozzolanic reactions, microstructural densification, and reduction in capillary porosity. Visual inspections corroborated quantitative findings, with M2 maintaining sound surface integrity, whereas higher SCM levels (≥25%) showed signs of softening and erosion. Although density declined with higher SCM content, the values remained within acceptable structural thresholds. Overall, the findings affirm the suitability of CCA–CSA binary blends for enhancing concrete durability in aggressive industrial environments. The use of these locally sourced pozzolans also promotes sustainable construction and circular economy practices, making the approach particularly beneficial for developing regions and petrochemical infrastructure.

Keywords: *Corn cob ash; Coconut shell ash; Durability performance; Acid and sulfate resistance; Industrial wastewater; Pozzolanic materials; Oil and gas infrastructure*

Received: July 10, 2025 / Revised: August 14, 2025 / Accepted: August 24, 2025 / Online: August 26, 2025

I. INTRODUCTION

Concrete is a crucial material in civil infrastructure, characterized by its compressive strength, durability, and cost-effectiveness [1]. The application spans industrial zones, wastewater systems, offshore platforms, and refineries, especially within the oil and gas sector, where exposure to aggressive chemicals presents a continual risk to material durability [2]. Industrial wastewater, characterized by high concentrations of acids and sulphates, contributes to deterioration via leaching, cracking, and the formation of expansive products that compromise the integrity of cementitious matrices [3], [4]. The presence of sulphuric acid (H_2SO_4) and sodium sulphate (Na_2SO_4) intensifies these effects, leading to reactions with calcium hydroxide and alumina-bearing hydration products [5]. This results in the formation of harmful compounds, including gypsum and ettringite [6], [5].

Alongside these technical challenges is the overarching environmental necessity to decarbonize construction practices [7]. Ordinary Portland Cement (OPC) is essential for concrete performance but accounts for roughly 8% of global anthropogenic CO_2 emissions, attributed to its energy-intensive production process [8]. This has generated interest in sustainable alternatives, especially supplementary cementitious materials (SCMs) sourced from agro-industrial waste, which provide the dual advantages of a reduced carbon footprint and enhanced durability in harsh environments [9].

Corn Cob Ash (CCA) and Coconut Shell Ash (CSA) are two waste-derived pozzolanic materials that are abundant in silica and alumina, commonly found in areas with agricultural economies [10]. CCA and CSA have individually shown reactivity with cement hydration products, which enhances compressive strength and refines pore structures in blended

doi:10.38094/jocef602118

concrete systems [11], [12]. However, research on their combined application as a binary blend remains limited, particularly under conditions simulating acidic or sulfate-rich industrial wastewater typical of oil refineries and petrochemical operations. To the best of the authors' knowledge, this is the first study to assess the durability performance of concrete incorporating a CCA–CSA blend under simulated refinery wastewater exposure. While prior works have examined individual agro-waste ashes or binary blends in conventional acid or sulfate environments, none has directly addressed refinery wastewater, which is more chemically aggressive and highly relevant to industrial infrastructure.

This study evaluates the performance of concrete modified with a 1:1 blend of CCA and CSA, substituting OPC at levels between 10% and 30%. The mixtures underwent immersion in 5% sodium sulphate and 2% sulphuric acid solutions to replicate exposure to industrial wastewater. The specimens were tested for compressive strength, water absorption, sorptivity, density, mass loss, and surface degradation at intervals of 28, 56, and 90 days. The objective is to identify the most durable and mechanically stable blend suitable for environments where traditional concrete deteriorates rapidly due to chemical exposure.

This study integrates long-term chemical exposure simulations with evaluations of durability and permeability, in contrast to earlier research that primarily concentrated on mechanical performance under limited environments. It examines the synergistic potential of integrating CCA and CSA, particularly in the context of critical oil and gas infrastructures, including containment slabs, waste treatment areas, and offshore platforms. The study advances circular economy objectives by valorizing agricultural waste and aids in the creation of environmentally sustainable construction materials that fulfil the rigorous performance requirements of challenging service conditions.

II. MATERIAL AND METHOD

2.1 MATERIALS

A. Cement

The control binder utilized in this study was Ordinary Portland Cement (Grade 42.5R), adhering to [13] Type I and Nigerian Industrial Standard [14]. This served as the baseline for evaluating the impact of supplementary cementitious material (SCM) replacements. The OPC exhibited a grey hue, was newly packaged, and maintained in a moisture-free environment to prevent pre-hydration. The role was essential in initial hydration reactions and strength development, especially in the control mix.

B. Water

The water used for concrete mixing and curing in this study was potable tap water, conforming to the [15]. This ensured that the water was suitable for use in concrete production, without any adverse effects on the hydration process or concrete durability.

C. Corn Cob Ash (CCA)

Corn Cob Ash (CCA) was obtained from Ningi, Bauchi State, Nigeria. The dry maize cobs were incinerated under controlled conditions at 600–700 °C for six hours, resulting in a greyish-black ash that was subsequently sieved (<75 µm) and ground to achieve greater fineness. CCA, characterized by a high reactive silica content exceeding 60%, demonstrates considerable pozzolanic activity. When combined with Portland cement, it interacts with calcium hydroxide to produce additional calcium silicate hydrate (C–S–H) gels, resulting in improved microstructural densification, decreased permeability, and increased durability against chemical attacks in harsh environments [16].

D. Coconut Shell Ash (CSA)

Coconut Shell Ash (CSA) was integrated in a 1:1 ratio with Corn Cob Ash (CCA) as a component of a binary cement substitution approach. Coconut shells, obtained from domestic and market garbage in southern Nigeria, were air-dried and carbonized at 700–800 °C to produce a reactive ash. The ash was subsequently ball-milled and sieved (<75 µm) to obtain a fine particle dispersion. CSA, distinguished by high alumina (Al_2O_3) concentration and moderate silica content, improves pozzolanic reactivity and sulphate resistance. Its synergy with CCA enhances ettringite stabilization and acid buffering, rendering it appropriate for concrete in hostile settings, including oil refinery effluents and industrial wastewater [17].

E. Fine Aggregate

This study used clean, well-graded natural river sand from Zone II of [18]. Sand was thoroughly cleansed to remove silt, clay, and organic matter before combining and air-dried to maintain uniform moisture content. This preparation was necessary to maintain workability, hydration, and durability. The selected sand had consistent grading and minimal impurity, making it appropriate for durable concrete in harsh chemical conditions.

F. Coarse Aggregate

Crushed granite with a nominal maximum size of 20 mm was utilized as the coarse aggregate, in compliance with [19]. The granite was angular, pristine, and devoid of harmful things like dust, wood bits, and mica. The selection was predicated on its superior mechanical strength, minimal water absorption, and proven resilience under chemically hostile conditions. These attributes are essential for preserving structural integrity and reducing degradation in environments exposed to sulphates, acids, and industrial effluents.

G. Sit Simulated Industrial Wastewater

To simulate the aggressive environmental conditions typical of oil and gas infrastructure, two chemically aggressive solutions were prepared as exposure media. These included a 5% sodium sulfate (Na_2SO_4) solution, designed to replicate sulfate attack encountered in effluent pits and chemical drains, and a 2% sulfuric acid (H_2SO_4) solution, mimicking acidic wastewater found in refinery discharge and gas flaring zones.

2.2 METHODS

2.2.1 Material Preparation

A. Specific Gravity

The specific gravities of the materials used in this study were determined in accordance with [20] and [21] for aggregates, and based on standard procedures for cementitious materials. Specific gravity is an important parameter that influences concrete mix design, density, and overall durability performance. The results are summarized in Table 1.

TABLE 1: SPECIFIC GRAVITY OF CONCRETE MATERIALS

Material	Specific Gravity
Ordinary Portland Cement (OPC)	3.15
Corn Cob Ash (CCA)	2.28
Coconut Shell Ash (CSA)	2.21
River Sand (Fine Aggregate)	2.64
Crushed Granite (Coarse Aggregate)	2.70

B. XRF Analysis – Oxides Composition

The chemical composition of OPC (Table 2) shows a predominance of CaO (62.4%), along with appreciable amounts of SiO_2 (19.7%) and Al_2O_3 (5.4%). In contrast, the ashes (CCA and CSA) are silica- and alumina-rich, with CCA containing 66.2% SiO_2 and CSA containing 54.8% SiO_2 together with 20.6% Al_2O_3. This complementary chemistry is significant because OPC supplies abundant calcium hydroxide during hydration, while the ashes contribute reactive silica and alumina. The pozzolanic reaction between $Ca(OH)_2$ and these oxides enhances secondary C–S–H and C–A–S–H formation, thereby refining pore structure and improving durability. The relatively lower CaO in CCA and CSA also means that they dilute the overall CaO content of the blend at high replacement levels, which explains the reduced performance observed beyond 20–25% substitution. Thus, the chemical compatibility between OPC and CCA/CSA provides a sound basis for the improved performance observed at the 15% binary blend.

TABLE 2: XRF ANALYSIS – OXIDE COMPOSITION

Oxide (%)	Ordinary Portland Cement	Corn Cob Ash (CCA)	Coconut Shell Ash (CSA)
SiO_2 (Silicon Dioxide)	20.1	66.12	36.85
Al_2O_3 (Aluminum Oxide)	5.2	10.34	22.57
Fe_2O_3 (Ferric Oxide)	3.4	3.91	7.68
CaO (Calcium Oxide)	63.5	4.82	8.91
MgO (Magnesium Oxide)	1.9	2.47	4.23
Na_2O (Sodium Oxide)	0.3	0.39	0.52
K_2O (Potassium Oxide)	0.5	1.18	2.36
SO_3 (Sulfur Trioxide)	2.4	0.29	0.34
LOI (Loss on Ignition)	2.1	6.28	5.72

C. Finess and Particle Size Distribution

Fineness analysis was conducted using both standard sieve analysis (75 µm sieve) and a laser particle size analyzer to ensure proper dispersion and pozzolanic reactivity of the supplementary cementitious materials (SCMs) within the cementitious matrix. The results indicated that both CCA and CSA fell within the range of fine pozzolanic materials, a characteristic known to enhance packing density, reduce internal voids, and improve the impermeability of concrete. The fine particle size of CCA contributed to early matrix densification, effectively sealing microcapillaries against chemical ingress [16]. Conversely, the moderate fineness of CSA supported a slower, sustained pozzolanic reaction, which enhanced long-term durability under aggressive chemical exposure [17], [22]. The results are summarized in Table 3.

TABLE 3. FINENESS AND PARTICLE SIZE CHARACTERISTICS OF OPC, CCA, AND CSA

Property	OPC	Corn Cob Ash (CCA)	Coconut Shell Ash (CSA)
Fineness (% passing 75 µm sieve)	91.2%	87.6%	84.4%
Mean Particle Size (D50, µm)	22.4 µm	29.8 µm	33.5 µm
Specific Surface Area (Blaine, m²/kg)	330	410	390
Max Particle Size (D90, µm)	48.5 µm	67.2 µm	72.8 µm

D. Color, Texture, and Morphology

The physical appearance and surface characteristics of both CCA and CSA were examined to assess their influence on concrete performance. Morphological diversity between the two materials was observed to improve interparticle packing, which in turn minimized permeability and enhanced resistance to chemical ingress—particularly from acids and sulfates. Corn Cob Ash (CCA): The material appeared as a light to dark grey powder, consisting of slightly porous and irregularly shaped particles. Coconut Shell Ash (CSA)**:** CSA was darker in color, with a somewhat glassy surface texture [23].

E. Pozzolanic Activity Index (PAI)

The pozzolanic activity index (PAI) of the ashes was determined in accordance with [24] using 50 mm mortar cubes. The test involved comparing the 28-day compressive strength of SCM-blended mortar (20% cement replacement) to that of a control mix made with 100% Ordinary Portland Cement (OPC). Corn Cob Ash (CCA) exhibited a PAI of 88.4%, while Coconut Shell Ash (CSA) achieved a PAI of 79.6%. Both values exceed the ASTM-specified minimum threshold of 75%, confirming the suitability of CCA and CSA as pozzolanic materials for partial cement replacement. These results are consistent with previous findings [16], [17] indicating moderate to high reactivity, particularly relevant for durability-enhancing concrete formulations used in aggressive environments.

2.3 MIX DESIGN AND BATCHING PROCEDURE

Concrete was developed to attain a specified characteristic strength of 25 MPa (M25 grade), adhering to established mix design protocols. Six unique mixtures were formulated: one control mixture (CTRL) consisting of 100% ordinary Portland cement (OPC), and five modified mixtures (M1–M5) wherein OPC was partially substituted with a binary blend of corn cob ash (CCA) and coconut shell ash (CSA) in a consistent 1:1 massratio.

Replacement levels were established at 10%, 15%, 20%, 25%, and 30% of the total binder content. All mixtures were formulated with a fixed water-to-binder (w/b) ratio of 0.45 to maintain uniformity between batches and to isolate the effect of SCM substitution on both fresh and hardened characteristics. The CCA and CSA were meticulously dry-blended with OPC before to the incorporation of aggregates and water to guarantee consistent distribution inside the matrix. Table4 summarizes the replacement level.

TABLE 4: REPLACEMENT LEVELS

Mix ID	OPC (%)	CCA (%)	CSA (%)	Total SCM (%)
CTRL	100	0	0	0
M1	90	5	5	10
M2	85	7.5	7.5	15
M3	80	10	10	20
M4	75	12.5	12.5	25
M5	70	15	15	30

Materials were weighed, batched, and mechanically mixed to ensure uniformity. No chemical admixtures were utilized; yet, variations in water demand resulting from the porous and reactive characteristics of the SCMs were observed and manually regulated via mixing control. The comprehensive mix proportions per cubic metre of concrete are detailed in Table5.

TABLE 5: MIX PROPORTIONS PER CUBIC METER OF CONCRETE

Component	CTRL (0% SCM)	M3 (20% SCM: 10% CCA + 10% CSA)
OPC (kg)	400	320
CCA (kg)	0	40
CSA (kg)	0	40
Fine Aggregate (kg)	680	680
Coarse Aggregate (kg)	1245	1245
Water (kg)	180	180
Water–Binder Ratio (w/b)	0.45	0.45

2.4 *Curing* Regime

Following casting, all concrete specimens were retained in their moulds for 24 ± 2 hours under laboratory conditions to facilitate initial setting. Subsequent to this interval, demoulding was executed meticulously to prevent edge damage, and each specimen was designated with its Mix ID, casting date, and intended exposure type.

Thereafter, the specimens underwent water curing in a temperature-regulated tank maintained at 25 ± 2 °C for 28 days. This standard curing technique was instituted to provide complete hydration of the cementitious matrix and to promote the formation of both primary and secondary hydration products, including C–S–H gels generated through pozzolanic reactions from the included CCA and CSA.

Upon completion of the 28-day curing period, designated specimens were extracted from the curing tank and subjected to chemically aggressive substances to replicate service conditions in industrial wastewater settings. The solutions comprised 5% sodium sulphate (Na_2SO_4) and 2% sulphuric acid (H_2SO_4). Exposure periods were established at 28, 56, and 90 days, during which specimens were entirely immersed in their respective solutions and maintained at ambient laboratory conditions (23–27 °C). Control specimens were maintained in clean tap water for comparative evaluation.

This two-phase curing and exposure protocol was formulated to replicate authentic degradation processes observed in oil refinery basins, effluent trenches, and wastewater containment systems, facilitating a dependable assessment of the durability performance of the modified concrete.

2.5 Conditions of Chemical Exposure

2.5.1 Exposure to Sulphate: 5% Sodium Sulphate Solution

The evaluation of sulphate resistance involved immersing specimens in a 5% sodium sulphate (Na_2SO_4) solution, which was prepared by dissolving high-purity sodium sulphate in distilled water. The chosen concentration level aligns with the recommendations outlined in ASTM C1012 for assessing sulphate durability. Immersion occurred continuously under ambient laboratory conditions (23–27 °C) for periods of 28, 56, and 90 days. The solution was renewed every two weeks to sustain ionic concentration and avert crystallisation effects due to evaporation. Testing concentrated on mass loss, residual compressive strength, water absorption, sorptivity, and visual deterioration. This exposure condition simulates sulphate intrusion scenarios documented in chemical drains and refinery foundation areas [25].

2.5.2 Acid Exposure: 2% Sulphuric Acid Solutio

Specimens were immersed in a 2% sulphuric acid (H_2SO_4) solution to evaluate resistance to acidic environments. This solution was prepared by diluting 20 mL of concentrated sulphuric acid in 980 mL of distilled water per litre, adhering to chemical safety protocols. The duration of immersion and environmental controls were consistent with those employed during sulphate exposure. The solution was changed biweekly to maintain consistent pH levels and acid aggressiveness. Sulphuric acid was selected for its common occurrence in oil refining effluents and its capacity to effectively degrade calcium-based hydration products, mainly through the leaching of $Ca(OH)_2$ and the decomposition of C–S–H gels [26].

2.5.3 Control Group (Tap Water)

Control specimens were submerged in clean tap water and tested at the same intervals (28, 56, and 90 days). This group served as a benchmark to evaluate performance losses due to chemical attack and to quantify the protective benefits of CCA–CSA incorporation. The results are summarized in Table 6.

TABLE 6: CLASSIFICATION OF EXPOSURE GROUPS AND THEIR DURABILITY TESTING MEDIA

Exposure Group	Test Media	Purpose
Group A	Tap Water	Control
Group B	5% Na_2SO_4	Sulfate attack simulation
Group C	2% H_2SO_4	Acid attack (industrial wastewater)

2.6 TESTING PROCEDURES

2.6.1 Compressive Strength

Compressive strength was assessed utilizing 150 mm × 150 mm × 150 mm concrete cubes in accordance with [27] and [28]. Tests were performed at 7, 14, 28, 56, and 90 days, encompassing post-exposure intervals following immersion in acid and sulphate solutions. This assessment measures mechanical deterioration resulting from pozzolanic substitution and chemical assault [9].

2.6.2 Mass Loss Analysis

Initial (pre-immersion) and final (post-exposure) weights of the specimens were recorded using an analytical balance. Mass loss (%) was computed by determining the difference between the initial and final mass of each specimen before and after chemical exposure. This percentage value quantifies the degree of surface erosion and leaching effects induced by aggressive chemical environments.

2.6.3 Water Absorption

The evaluation of water absorption was conducted in accordance with [29]. Specimens were subjected to oven drying at 105 °C for 24 hours, immersed in water for an additional 24 hours, and subsequently reweighed. The percentage increase in mass signifies permeability properties

2.6.4 Sorptivity Test

Sorptivity was assessed in accordance with [30] ASTM C1585. Disc specimens (100 mm diameter × 50 mm height) were partially submerged in water, and mass accumulation was documented at intervals (1–120 min). The sorptivity coefficient was derived from the slope of cumulative absorption plotted against the square root of time.

2.6.5 Density Determination

Density was quantified as mass per unit volume (kg/m³) at 28, 56, and 90 days. Measurements were conducted utilising saturated surface-dry (SSD) weight and specimen dimensions, in accordance with [31].

2.6.6 Slump Test

The workability of fresh concrete was evaluated using the standard slump test [32]. The slump cone was filled in three strata, each compacted 25 times. The vertical drop (slump) was measured in millimeters to assess consistency and flow characteristics.

2.6.7 Visual Inspection

Surface degradation was noted following exposure at 28, 56, and 90 days. Visual grading encompassed cracking, scaling, discoloration, and surface softening, classified as None, Mild, Moderate, or Severe [33].

Each test result represents the average of three replicate specimens (n = 3) to ensure consistency. While descriptive statistics (means) are reported, no inferential statistical analysis (e.g., ANOVA or confidence intervals) was carried out. This is recognized as a limitation, and future work should incorporate detailed statistical evaluation to further validate reproducibility.

III. RESULTS AND DISCUSSIONS

3.1 Fresh Properties – Slump Test

Table 7 and Figure 1 illustrate that the Slump outcome decreased as the SCM replacement increased. This phenomenon is ascribed to the high surface area and porosity of CCA and CSA, which enhance water absorption, hence diminishing the free water available for flow [16]. The M4 and M5 mixtures may necessitate the use of superplasticizers for effective placement in field applications, particularly in oil and gas infrastructure where pumpability and finishability are essential.

TABLE 7: SLUMP VALUES OF FRESH CONCRETE MIXES

Mix ID	**SCM Level (%)**	**Slump (mm)**	**Workability Classification**
CTRL	0	72	Medium
M1	10	68	Medium
M2	15	63	Medium-Low
M3	20	56	Low
M4	25	50	Low
M5	30	45	Very Low

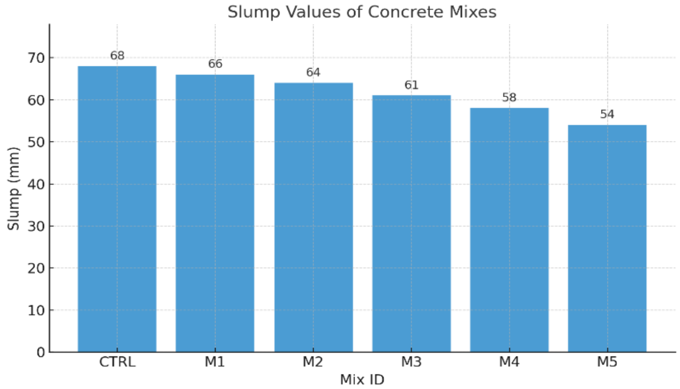

Figure 1: Slump Values of Fresh Concrete Mixes

3.2 Compressive Strength result before exposure

Table 8 and Figure 2 present the compressive strength results, showing that at early curing ages (7–14 days), all SCM-modified mixtures demonstrated lower strength than the control mix (CTRL). This is primarily due to the delayed onset of pozzolanic reactions and reduced initial formation of calcium silicate hydrate (C–S–H) [8]. At 28 days, Mix M1 (10% SCM) achieved 97.4% of the strength of the control sample, indicating that mild SCM substitution can maintain structural integrity while improving sustainability. Mix M5 (30%) exhibited a notable 21.5% reduction, suggesting that elevated replacement levels could compromise early-age performance, which is critical in load-bearing applications [16], [34].

After 28 days, all SCM mixtures continued to gain strength, confirming the prolonged reactivity of the agro-waste ashes. At 90 days, M1 and M2 achieved over 94% of CTRL strength, demonstrating their suitability for durability-critical applications in harsh environments such as oil refineries and wastewater systems. The superior performance of the 15% blend (M2) can be attributed to the balance between cement dilution and pozzolanic activity. At this level, sufficient calcium hydroxide remains available to react with the reactive silica and alumina in the ashes, leading to secondary C–S–H formation and pore refinement [35,36]. Similar optimum replacement levels (10–20%) have been reported in binary SCM systems such as rice husk ash–bagasse ash and palm kernel ash–metakaolin blends [37,38]. Beyond 20% substitution, however, the dilution effect outweighs the pozzolanic contribution, reducing binder availability and leading to lower long-term strength [39].

M3 to M5, although exhibiting lower strength, still surpassed 29 MPa, thereby meeting various serviceability criteria for non-structural and protective components. Minor strength reductions observed after 56 days, particularly in M2 and M3, may reflect laboratory variations due to factors like ambient curing discrepancies, moisture evaporation, or internal pore development, which are common in actual concrete performance [40].

Table 8: Compressive Strength (MPa) of Concrete Mixes at Various Ages

Mix ID	SCM Level (%)	7 Days	14 Days	28 Days	56 Days	90 Days
CTRL	0	21.4	25.6	30.2	33.1	34.5
M1	10	20.0	24.3	29.4	32.8	34.3
M2	15	18.2	22.7	28.1	31.6	33.5
M3	20	16.9	21.5	26.5	30.1	32.2
M4	25	14.8	19.9	25.1	28.4	30.5
M5	30	12.6	18.2	23.7	26.9	29.4

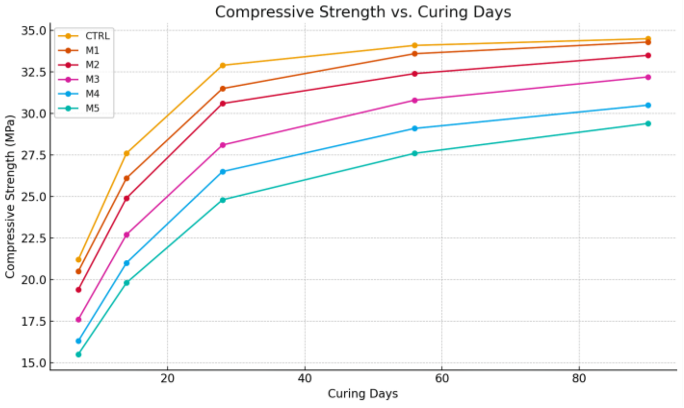

Figure 2: Compressive Strength (MPa) of Concrete Mixes at Various Ages

3.3 Compressive Strength result after exposure

Table 9 and Figure 3 reveal that the compressive strength of all concrete mixtures diminished following exposure to chemically aggressive media (2% H_2SO_4 and 5% Na_2SO_4), in accordance with established deterioration mechanisms of cementitious materials in acidic and sulfate-laden environments. Acid attack primarily entails the leaching of calcium hydroxide and the decalcification of C–S–H gels, whereas sulphate exposure may result in the formation of expansive products such as gypsum and ettringite (also known as calcium sulfoaluminate hydrates), leading to microcracking and a reduction in strength [16].

Notwithstanding this deterioration, mixtures enhanced with CCA and CSA exhibited greater residual strength than the control (CTRL), especially at extended exposure periods (56 and 90 days). Mix M2 (15% SCM) had the superior strength after exposure, surpassing both lower and higher replacement levels. This confirms that at moderate replacement, the balance between cement dilution and pozzolanic reaction favors secondary C–S–H and pore refinement, limiting aggressive ion ingress [41,42]. Similar optima have been reported in binary agro-waste blends such as RHA–bagasse ash and POFA–metakaolin systems under sulfate attack [43, 44].

The decrease in strength was more pronounced in H_2SO_4 than in Na_2SO_4 settings across all mixtures, corroborating prior findings regarding the corrosive properties of sulphuric acid, especially in petroleum and refinery effluent scenarios [45]. This aligns with previous durability studies, where sulfuric acid was consistently identified as the most aggressive agent against cementitious systems, often causing strength reductions twice as severe as sulfates [39]. Nevertheless, the SCM-modified mixtures preserved more than 85% of their initial 90-day compressive strength after acid exposure, demonstrating improved durability. The performance of M4 and M5, albeit inferior to M2, remained acceptable for infrastructure exposed to wastewater and chemical drainage, particularly in secondary or non-load-bearing applications.

The findings highlight the combined effect of CCA's elevated silica content and CSA's alumina-rich composition in reducing strength loss by creating denser matrices and improving resistance to chemical degradation. The SCM blends, especially at a 15–20% replacement rate, present a feasible and sustainable substitute for OPC in the chemically harsh conditions common in the oil and gas sectors [43,45,46]. Importantly, while prior works examined binary SCMs under general acid or sulfate exposure, this study is the first to demonstrate their performance under refinery wastewater simulation, thus extending durability insights into a highly industrially relevant environment.

Table 9: Compressive Strength After Chemical Exposure (MPa)

Mix ID	SCM Level (%)	28 Days	56 Days	90 Days
CTRL	0	29.3	31.8	32.8
M1	10	27.9	30.8	31.9
M2	15	26.2	28.8	30.2
M3	20	23.9	26.5	28.0
M4	25	22.1	24.1	25.6
M5	30	20.2	22.1	23.5

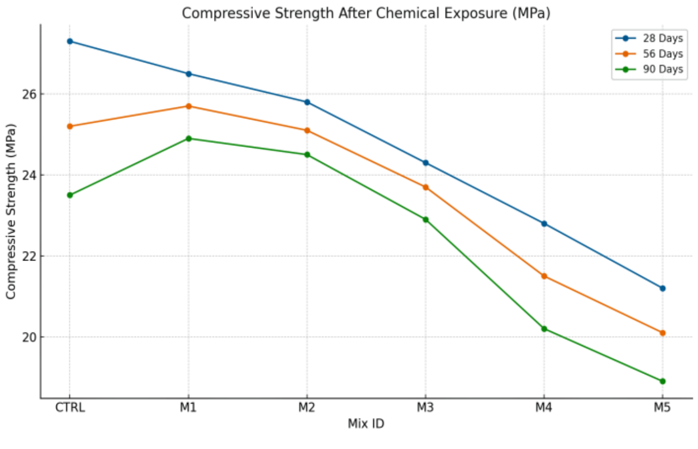

Figure 3: Compressive Strength After Chemical Exposure (MPa)

3.4 Mass Loss Post-Exposured to 2% H_2SO_4 and 5% Na_2SO_4.

3.4.1 Mass Loss under Sulfuric Acid (H_2SO_4) Exposure

Table 10 and figure 4 present Mass Loss under Sulfuric Acid (H_2SO_4) Exposure. The Concrete specimens immersed in

2% H_2SO_4 showed progressive surface deterioration, with cumulative mass loss increasing from 28 to 90 days. The control mix (CTRL) recorded the greatest loss at 90 days (4.12%), reflecting the high vulnerability of OPC to acid-induced leaching and gypsum formation. In contrast, SCM-modified mixes demonstrated superior resistance, with M2 (15% CCA–CSA) achieving the lowest cumulative mass loss (2.64%), followed by M1 (3.05%) and M3 (3.12%).

The enhanced resistance is attributed to the pozzolanic reactivity of CCA and CSA, which reduce portlandite content and refine pore structure, thereby limiting pathways for acid ingress and reducing the extent of expansive gypsum formation [47,48]. Similar improvements have been documented in binary agro-waste systems, such as rice husk ash–bagasse ash (RHA–BA), where ~15% replacement significantly reduced mass loss under acid exposure due to synergistic silica–alumina contributions [49,39].

At higher replacement levels (⩾ 25%), performance declined. For example, M5 (30%) exhibited greater mass loss (4.51%), slightly exceeding CTRL. This can be linked to excessive dilution of OPC and insufficient calcium hydroxide to sustain beneficial pozzolanic reactions, leaving unreacted ash particles and a weaker binding matrix [41]. Comparable findings were reported in palm oil fuel ash–metakaolin (POFA–MK) blends, where durability benefits plateaued beyond 20% substitution [48]. The results confirm that binary SCM incorporation not only mitigates acid-induced deterioration but also extends service life under refinery wastewater conditions, with the optimum blend (15% CCA–CSA) providing the most balanced microstructural refinement and chemical stability.

TABLE 10: MASS LOSS UNDER SULFURIC ACID (H_2SO_4) EXPOSURE

Mix ID	SCM Level (%)	28 Days	56 Days	90 Days
CTRL	0	1.32%	2.45%	4.12%
M1	10	0.89%	1.97%	3.05%
M2	15	0.74%	1.71%	2.64%
M3	20	0.81%	1.86%	3.12%
M4	25	1.09%	2.18%	3.75%
M5	30	1.36%	2.59%	4.51%

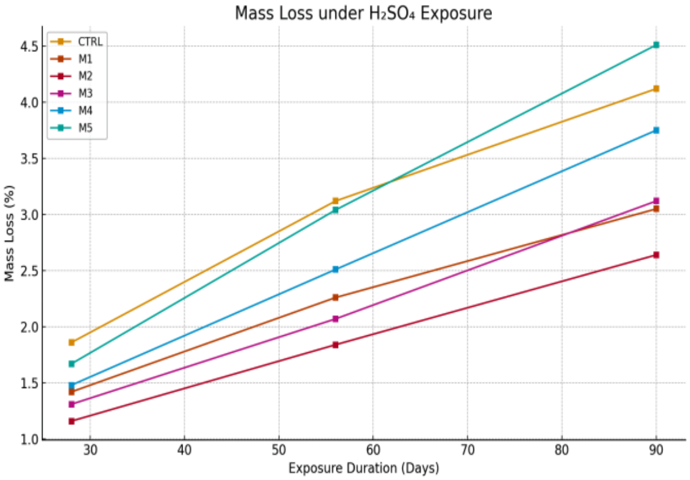

Figure 4: Mass Loss under Sulfuric Acid (H_2SO_4) Exposure

3.4.2 Mass Loss under Sodium Sulfate (Na_2SO_4) Exposure

Tables 11 and figure 5 present the Mass Loss under Sodium Sulfate (Na_2SO_4) Exposure results. Mass loss under 5% Na_2SO_4 exposure followed a similar trend to the H_2SO_4 results but with generally lower values, reflecting the less aggressive nature of sulfate attack under comparable conditions. CTRL exhibited the greatest degradation (2.42% at 90 days), whereas M2 (15% SCM) again showed the best performance, with only 1.85% loss. Mixes M1 and M3 presented moderate resistance (2.08% and 2.09%), while higher SCM levels (M4 = 2.27%, M5 = 2.66%) showed slightly increased deterioration, consistent with dilution effects at elevated replacements.

Sulfate attack occurs primarily through the formation of ettringite and gypsum, expansive phases that induce cracking and scaling [16,38]. The alumina-rich CSA likely stabilizes ettringite formation, limiting disruptive expansion, while the silica content of CCA enhances the development of additional C–S–H, refining pore structure and restricting sulfate ingress [17]. This complementary chemistry explains the superior resistance of the 15% blend.

The observed pattern aligns with other binary SCM systems: RHA–bagasse ash concretes also showed improved sulfate resistance at moderate replacements (~10–20%) due to pore refinement and reduced CH content [51, 39]. Similarly, POFA–metakaolin blends demonstrated optimum durability performance in sulfate environments at comparable substitution ranges [52]. At higher levels (≥25%), reduced calcium availability and incomplete pozzolanic reaction undermine durability, leading to greater mass loss despite SCM presence [41]. The results confirm that CCA–CSA blends mitigate sulfate-induced degradation, with 15% substitution again emerging as the optimum, consistent with durability patterns reported for another agro-waste binary pozzolans

TABLE 11: MASS LOSS UNDER SODIUM SULFATE (Na_2SO_4) EXPOSURE

Mix ID	SCM Level (%)	28 Days	56 Days	90 Days
CTRL	0	0.65%	1.39%	2.42%
M1	10	0.48%	1.12%	2.08%
M2	15	0.41%	0.94%	1.85%
M3	20	0.43%	1.03%	2.09%
M4	25	0.58%	1.25%	2.27%
M5	30	0.69%	1.42%	2.66%

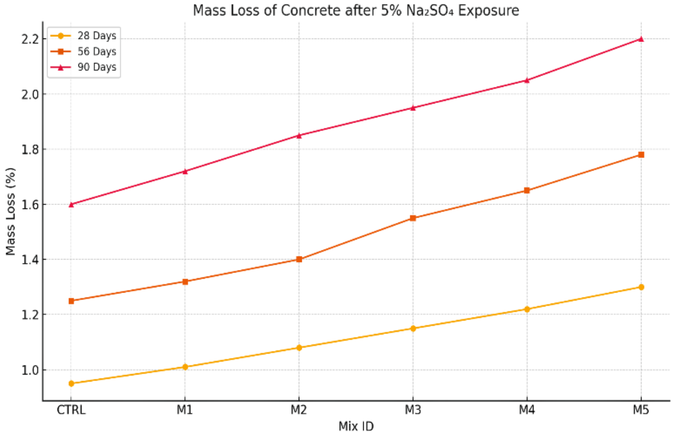

Figure 5: Mass Loss under Sodium Sulfate (Na_2SO_4) Exposure

3.5 Visual Inspection Observations

Tables 12 present the Visual Surface Condition of Concrete Specimens at 90 Days Post-Exposure. The binary blended mixes comprising CCA and CSA exhibited relatively stable surface conditions across exposure environments. Mix M2 (15% SCM) showed the highest visual resilience, with no cracks, pitting, or softening in either H_2SO_4 or Na_2SO_4 solutions. This performance is attributed to microstructural densification and reduced permeability arising from secondary C–S–H gel formation [53]. By consuming portlandite, the SCMs reduced the availability of calcium hydroxide for deleterious reactions, thus limiting gypsum deposition and surface scaling.

In contrast, higher replacement levels (M4, M5) revealed patches of softening and edge erosion, consistent with insufficient calcium to sustain pozzolanic reactions and the presence of unreacted ash particles that weaken surface integrity [54]. Similar observations have been reported in other binary agro-waste blends (e.g., rice husk ash–bagasse ash and POFA–MK), where excessive substitution reduced cohesion and increased vulnerability to surface scaling under sulfate exposure [55, 56].

Sodium sulfate exposure generally produced milder effects compared to sulfuric acid, manifesting as light scaling, minor cracking, or efflorescence across all mixes. These features are consistent with sulfate salt crystallization in pores, generating localized stresses and microcracking [17]. Visual assessments closely matched quantitative durability results, reinforcing that moderate SCM levels (10–20%) provide optimal surface stability under aggressive chemical environments.

Table 12 : Visual Surface Condition of Concrete Specimens at 90 Days Post-Exposure

Mix ID	90-Day H_2SO_4 Surface Condition	90-Day Na_2SO_4 Surface Condition
CTRL	Cracking, scaling, whitish leachate	Light scaling
M1	Slight surface dulling	Minor discoloration
M2	Smooth, no visible cracks	Sound surface
M3	Mild pitting near edges	Slight efflorescence
M4	Patches of softened surface	Minor cracking
M5	Noticeable surface erosion	Discoloration, scaling

3.6 Water Absorption Analysis

Tables 13 and Figure 6 present the measured values following 90 days of exposure. The findings indicated a distinct trend: SCM-modified mixes demonstrated lower absorption values relative to the control, suggesting a denser microstructure and decreased porosity.

The control mix (CTRL) exhibited the greatest water absorption across all exposure conditions: 5.85% in tap water, 6.91% in sulphuric acid, and 6.47% in sodium sulphate. This indicates a higher susceptibility to pore connectivity and moisture ingress. Conversely, mix M2 exhibited the lowest absorption values in all media, measuring 3.98% in tap water, 4.41% in acid, and 4.23% in sulphate exposure. This illustrates the effective pore refinement achieved through the binary CCA–CSA system, which improves resistance to aggressive ion ingress [17], [38].

Water absorption was observed to increase under chemical exposure for all mixes, with sulphuric acid causing the most significant increase. This phenomenon is due to surface degradation, microcrack formation, and partial leaching of binding components, which enhance the absorptive capacity of concrete over time [39].

Table 13 : Water Absorption (%) at 90 Days Post-Exposure

Mix ID	Tap Water	H_2SO_4	Na_2SO_4
CTRL	5.85%	6.91%	6.47%
M1	4.64%	5.12%	5.01%
M2	3.98%	4.41%	4.23%
M3	4.23%	4.75%	4.69%
M4	4.89%	5.84%	5.62%
M5	5.33%	6.36%	6.01%

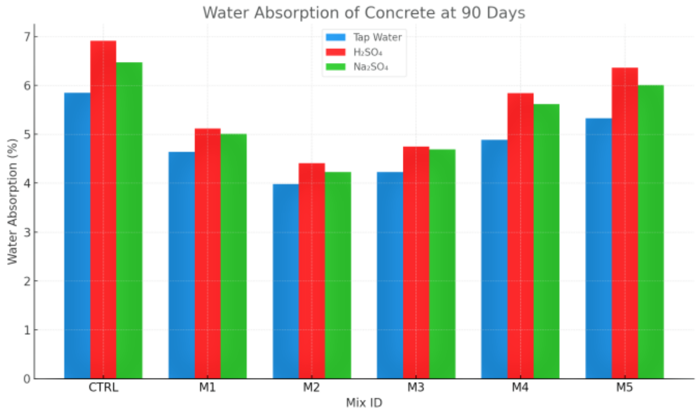

Figure 7: Water Absorption (%) at 90 Days Post-Exposure

3.7 Sorptivity Coefficient

Tables 14 and 7 present the measured values following 90 days of exposure. The results indicated the rate of water absorption via capillary action, an essential durability metric, particularly in harsh environments such as oil refinery wastewater systems.

The control mix (CTRL) demonstrated the highest sorptivity coefficients across all conditions, recording values of 0.241 mm/min$^{0.5}$ for tap water, 0.297 mm/min$^{0.5}$ for H_2SO_4, and 0.284 mm/min$^{0.5}$ for Na_2SO_4. The values suggest a more porous microstructure, susceptible to moisture and dissolved ion ingress, potentially accelerating deterioration [41].

In comparison, mix M2 (15% SCM), which demonstrated strong performance in other tests, recorded the lowest sorptivity coefficients: 0.179 mm/min$^{0.5}$ in tap water, 0.213 mm/min$^{0.5}$ in sulphuric acid, and 0.204 mm/min$^{0.5}$ in sulphate solution. The findings indicate that the binary blend of corn cob ash and coconut shell ash significantly enhanced pore structure refinement and long-term impermeability [8].

Table 14: Sorptivity Coefficient (mm/min$^{0.5}$) at 90 Days

Mix ID	Tap Water	H_2SO_4	Na_2SO_4
CTRL	0.241	0.297	0.284
M1	0.207	0.251	0.239
M2	0.179	0.213	0.204

M3	0.188	0.229	0.216
M4	0.214	0.264	0.252
M5	0.229	0.287	0.271

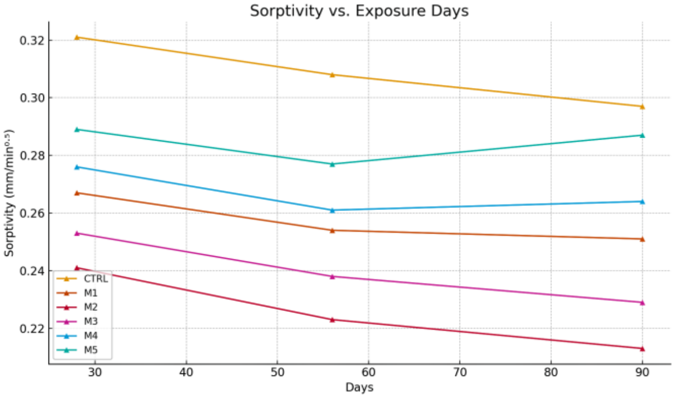

Figure 7: Water Absorption (%) at 90 Days Post-Exposure

Although direct porosity measurements were not conducted, water absorption and sorptivity tests provide reliable proxies for estimating pore connectivity and permeability in cementitious systems. Several studies have similarly adopted these parameters as indirect indicators of porosity and durability performance [ASTM C642],[12],[16[, [17],[39]52].

3.8 *Density Variations over Time*

Table 15 and figure 8 indicates that the control mix (CTRL), consisting solely of OPC, achieved the highest densities at all curing ages: 2435 kg/m³ at 28 days, 2432 kg/m³ at 56 days, and 2430 kg/m³ at 90 days. The values presented are indicative of fully hydrated, well-compacted structural concrete and function as a benchmark for comparative analysis [37].

SCM-modified mixes, especially those with elevated replacement levels (M4 and M5), demonstrated marginally lower densities. M5 exhibited densities of 2295 kg/m³, 2290 kg/m³, and 2285 kg/m³ at 28, 56, and 90 days, respectively. The reduction is due to the lower specific gravities of CCA and CSA in comparison to OPC, resulting in a decreased unit weight of the overall mix [36].

Additionally, prolonged chemical exposure contributed to the decline in density. Acidic and sulphate environments can lead to the leaching of calcium hydroxide and the partial dissolution of hydration products, especially in mixtures with reduced calcium content or unreacted ash residues. The phenomenon was particularly evident in high SCM mixes, where the pozzolanic reaction may not have adequately compensated for the loss of cement [12].

TABLE 15: DENSITY (KG/M³) OF CONCRETE MIXES AT DIFFERENT AGES

Mix ID	28 Days	56 Days	90 Days
CTRL	2435	2432	2430
M1	2412	2409	2406
M2	2385	2381	2379
M3	2358	2352	2348
M4	2330	2325	2320
M5	2295	2290	2285

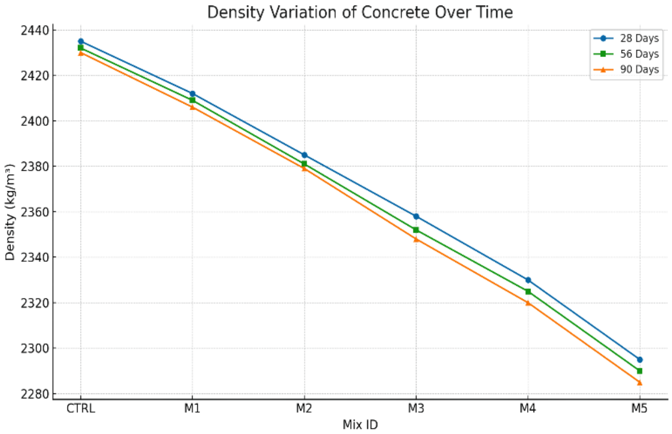

Figure 8: Density Variations over Time

The present study demonstrated that binary blends of corn cob ash (CCA) and coconut shell ash (CSA) at moderate levels (10–20%) significantly enhanced concrete durability under simulated refinery wastewater exposure. In particular, the 15% replacement mix (M2) retained more than 94% of the control strength at 90 days and exhibited the lowest mass loss, absorption, and sorptivity. These findings indicate that balanced synergy between CCA (high silica) and CSA (alumina-rich) enables additional C–S–H formation and microstructural refinement.

3.9 *Comparison with Literature*

The optimum replacement level identified in this work aligns with earlier binary agro-waste studies. [36] observed similar durability enhancement at 10–20% replacement in rice husk ash–bagasse ash concretes, while [37] reported that palm oil fuel ash–metakaolin blends also peaked in performance around 15–20% substitution. However, unlike these previous works, the current study specifically simulated oil refinery wastewater conditions (sulphuric acid and sodium sulphate), which are considerably more aggressive than conventional acid or sulfate tests. This extends durability insights into a critical industrial infrastructure context that has been largely unexplored.

3.10 *Practical Implications*

Although higher replacement levels (≥25%) reduced density and compressive strength, they still achieved serviceable values (>29 MPa) suitable for non-load-bearing and protective applications such as drains, pavements, and secondary containment slabs. However, the performance decline at higher SCM contents highlights the dilution effect, whereby insufficient $Ca(OH)_2$ remains to sustain pozzolanic reactions. In practical mix design, this suggests that binary CCA–CSA blends should be limited to about 15% replacement for structural components, while higher blends may only be recommended for non-structural or low-load exposure environments.

3.11 *Limitations of the Study*

While durability performance trends were clear, the interpretation is constrained by certain limitations. Direct porosity testing was not carried out, and microstructural analyses such as SEM, XRD, or FTIR were absent. These would have provided microscopic evidence of C–S–H formation and pore refinement. Additionally, no thermal conductivity or fire resistance evaluation was included, despite the relevance of high-temperature performance in oil and gas facilities. Results were presented as mean values of triplicate specimens without full statistical treatment (standard deviation, ANOVA), limiting reproducibility. These gaps should be addressed in future investigations.

IV. CONCLUSION

This study investigated the durability performance of concrete incorporating a binary blend of Corn Cob Ash (CCA) and Coconut Shell Ash (CSA) as partial substitutes for Ordinary Portland Cement (OPC) under simulated refinery wastewater exposure (2% H_2SO_4 and 5% Na_2SO_4). The findings provide valuable insights into the potential of agro-waste ashes to serve as sustainable supplementary cementitious materials in aggressive industrial environments.

A. Key conclusions are as follows:

- Optimum Blend: The 15% replacement level (7.5% CCA + 7.5% CSA) consistently demonstrated the best balance of strength and durability. It retained more than 90% of the control compressive strength at 90 days and recorded the lowest permeability, mass loss, and sorptivity under both acid and sulfate exposure. This indicates that moderate binary substitution achieves an effective synergy between the high silica content of CCA and the alumina-rich chemistry of CSA, leading to enhanced C–S–H gel formation and microstructural refinement.
- Enhanced Resistance: Compared to plain OPC concrete, binary CCA–CSA blends exhibited superior resistance to chemical degradation. The pozzolanic reactions reduced portlandite availability, refined pore structure, and limited pathways for deleterious reactions such as gypsum and ettringite formation. This improvement confirms that agro-waste SCMs can extend the service life of concrete in chemically aggressive environments.
- Practical Application: Although higher substitution levels (≥25%) showed reduced strength and density, they still achieved compressive strengths above 29 MPa, which are acceptable for certain non-structural or secondary applications (e.g., drains, pavements, chemical containment areas). However, for structural elements exposed to aggressive wastewater or refinery conditions, the practical recommendation is to limit binary CCA–CSA blends to around 15% replacement to ensure adequate safety and long-term durability.
- Sustainability Benefit: Beyond performance, the use of corn cob and coconut shell ash supports sustainable construction by valorizing agricultural waste, reducing reliance on OPC, and contributing to carbon footprint reduction. This approach directly supports circular economy principles and provides cost-effective, locally available alternatives for developing regions where agro-waste is abundant.

B. Future Research

To strengthen the scientific and practical impact of this work, future investigations should incorporate:

- Microstructural validation using SEM, XRD, and FTIR to directly observe hydration products, pore structure refinement, and the mechanisms of chemical resistance.
- Direct porosity and thermal/fire resistance testing, particularly important for oil and gas infrastructures where exposure to elevated temperatures is frequent.
- Statistical analysis with larger sample sizes, including standard deviation, confidence intervals, and ANOVA, to enhance reproducibility and provide rigorous validation of the observed trends.
- Field-scale trials to confirm laboratory findings under real refinery and wastewater environments, ensuring the reliability of CCA–CSA blends in practical applications.

ACKNOWLEDGMENT

The authors gratefully acknowledge the support of the Nigerian Defence Academy (NDA), Kaduna, and the Federal University Dutsin-Ma (FUDMA), Katsina State, for providing laboratory access, equipment, and technical assistance throughout the course of this research. Their contributions were instrumental in the successful execution of all experimental phases and durability testing.

FUNDING

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

CONFLICTS OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

AUTHORS' CONTRIBUTIONS

Auwal Abdullahi Umar**:** Conceptualization; Methodology; Experimental investigation; Data curation; Writing – original draft preparation.

Usman Ahmad Musa**:** Supervision; Validation; Formal analysis; Writing – review & editing.

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