

Physical and Mechanical Evaluation of Concrete with Coffee Husk Ash and Coconut Fiber as Sustainable Additives

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Received July 19, 2025; Revised March 2, 2026; Accepted March 26, 2026

Cite This Paper in the Following Citation Styles

(a): [1] Cubas-Armas Marlon Robert, Muñoz Pérez Sócrates, Herhuay-Huaman David, Luna-Diaz Victor, "Physical and Mechanical Evaluation of Concrete with Coffee Husk Ash and Coconut Fiber as Sustainable Additives," *Civil Engineering and Architecture*, Vol. 14, No. 3, pp. 1494 - 1508, 2026. DOI: 10.13189/cea.2026.140308.

(b): Cubas-Armas Marlon Robert, Muñoz Pérez Sócrates, Herhuay-Huaman David, Luna-Diaz Victor (2026). *Physical and Mechanical Evaluation of Concrete with Coffee Husk Ash and Coconut Fiber as Sustainable Additives*. *Civil Engineering and Architecture*, 14(3), 1494 - 1508. DOI: 10.13189/cea.2026.140308.

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Abstract This study evaluates the combined addition of coffee husk ash (CHA) and coconut fiber (CF) to conventional concrete to enhance its physical and mechanical properties for structural applications. Motivated by the need for sustainable construction materials and utilizing agricultural waste, the study employs an applied experimental design with 200 test specimens made under controlled conditions using local materials. The CHA was calcined to meet ASTM C618 standards, and CF was alkali-treated to improve durability by increasing cellulose content and reducing hemicellulose and lignin. Three experimental mix designs with varying CHA and CF proportions were evaluated against a control mix, focusing on workability, compressive strength, tensile strength, flexural strength, modulus of elasticity, air content, and unit weight. Results revealed that the optimal mix—1.5% CHA combined with 0.65% CF—significantly improved compressive strength (+8.1%), flexural strength (+6.1%), and elasticity (+5.8%) without compromising workability or unit weight. Statistical analyses (ANOVA, Tukey, and Dunnett tests) confirmed the significance and synergy of this combination. Higher dosages resulted in diminished mechanical performance, indicating the importance of dosage control. The study also demonstrated that the physical properties of fresh concrete remained within standard specifications, ensuring the practical feasibility of these additives. This work contributes novel evidence supporting the sustainable reuse of agroindustrial

residues to produce mechanically enhanced concrete, aligning with environmental goals and promoting circular economy models. By validating the synergy between CHA and CF through rigorous chemical characterization, mix design, and statistical validation, this research advances knowledge of bio-based concrete additives and offers practical guidelines for their optimized incorporation in structural concrete.

Keywords Concrete, CHA, CF, Physical-Mechanical Properties, Sustainability, Agroindustrial Residues, DRX, TGA, SEM

1. Introduction

The need to seek sustainable alternatives in the construction industry has driven the evaluation of agro-industrial residues, such as coffee husk ash and coconut fiber in concrete formulation, in response to the growing demand for materials that combine mechanical performance with environmental responsibility [1]. The accumulation of these residues presents a management challenge, but their reuse as additives in concrete mixes offers technical benefits and contributes to the circular economy by reducing reliance on non-renewable resources [2].

Recent studies highlight that, when incorporated in suitable proportions, it can improve the strength and durability of concrete, although its effectiveness depends on dosage and processing conditions, such as calcination temperature [3]. Coconut fiber, on the other hand, has shown potential to increase compressive strength, flexural strength, and tensile strength of concrete, as well as to enhance toughness and reduce cracking [4], thus improving both the durability and the overall performance of the material [5]. However, the variability of these materials and the lack of standardization in their use require further research to identify optimal proportions and effective application conditions [6].

Ramesh K. [7], using an experimental approach, employed a design in which coconut fiber was added at 5% and coconut fiber ash at 15% by weight of cement. The study focused on the need to reduce costs associated with cement production, proposing the use of agricultural waste materials such as coconut fiber and ash, which are often discarded and contribute to environmental pollution [8].

The study follows an experimental design, in which coconut fiber was added in proportions of 0.5%, 1%, 1.5%, and 2% by weight of cement, evaluating compressive, tensile, flexural, and impact strength [1]. The optimal percentage identified ranged from 6% to 15% replacement of sand with SCG-HTC, with 6% being the most balanced in terms of mechanical performance [9].

In the international context, one of the world's top coffee producers, Brazil, faces environmental and waste management concerns arising from the accumulation of coffee husks, which, if disposed of improperly, can affect soil quality and water resources [10]. Recent research has demonstrated that using coffee husk ash as an additive in concrete is a viable option for reducing this waste and simultaneously improving key properties such as compressive strength and durability, provided calcination conditions and dosage are optimized [11]. Yet, variability in the chemical composition of the ash and lack of standard processing hinder its widespread use in Brazil's construction industry [12]. Logistics for the large-scale collection, processing, and distribution of this waste remains a challenge, limiting its environmental and economic impact. Therefore, research has focused not only on validating the technical benefits but also on analyzing the economic and logistical feasibility of large-scale implementation [13].

In India, the CF, abundant in the country's coastal regions, has been studied as a natural reinforcement in concrete mixes, showing improvements in tensile strength, flexural strength [14]. The addition of coconut fiber can reduce crack formation and increase the toughness of concrete, which is especially useful for infrastructure exposed to harsh climatic conditions [15].

In Perú, Jaén, this city, coffee production is a major

economic activity, generating large volumes of coffee husk as agricultural waste [16]. Accumulation of this material is an environmental issue, as it is often discarded in landfills or burned, affecting air quality and the local environment. Research in Jaén has focused on incorporating coffee husk ash into concrete to utilize this waste and improve the strength of concrete used in civil construction [17].

Coconut production here generates significant amounts of fiber waste, which has traditionally been discarded with no proper treatment [18]. Local research has aimed to evaluate the effects of incorporating coconut fiber in the physical and mechanical properties of concrete, seeking sustainable alternatives for waste management [19].

This research is focused on evaluating the combined addition of CHA and CF and its impact on the physical and mechanical properties of conventional concrete for structural design. The investigation aims to address the need for sustainable alternatives in the construction industry by utilizing agricultural residues that are commonly considered waste. The study is designed as applied research with a quantitative experimental approach, involving the preparation, characterization, and testing of concrete specimens incorporating different proportions of these natural additives.

The main objective is to determine the influence of adding CHA and CF on key concrete properties, such as compressive strength, tensile strength, flexural strength, modulus of elasticity, workability, unit weight, and air content. The project methodically examines not only the chemical and physical suitability of CHA and CF but also optimizes the mix design to meet the requirements of relevant standards. Three experimental treatments with variable combinations of CHA and CF are compared against a control mix, using locally sourced materials.

Laboratory tests are performed under standardized conditions to ensure valid comparisons, with particular emphasis on statistical validation of results. The research includes detailed chemical and physical characterization of both CHA and CF, using techniques, such as X-ray fluorescence and specialized treatment of fibers to enhance their structural performance. Statistical analyses, including tests for normality and homogeneity, as well as ANOVA and post hoc tests, are used to identify the most effective combination of additives.

Overall, the research aims to demonstrate that the controlled addition of coffee husk ash and coconut fiber can significantly enhance the mechanical properties and sustainability of concrete without compromising workability or other essential characteristics. The findings are positioned to contribute both to scientific understanding and to practical applications in sustainable building materials, potentially benefiting local agriculture and construction sectors while supporting environmental goals.

2. Materials and Methods

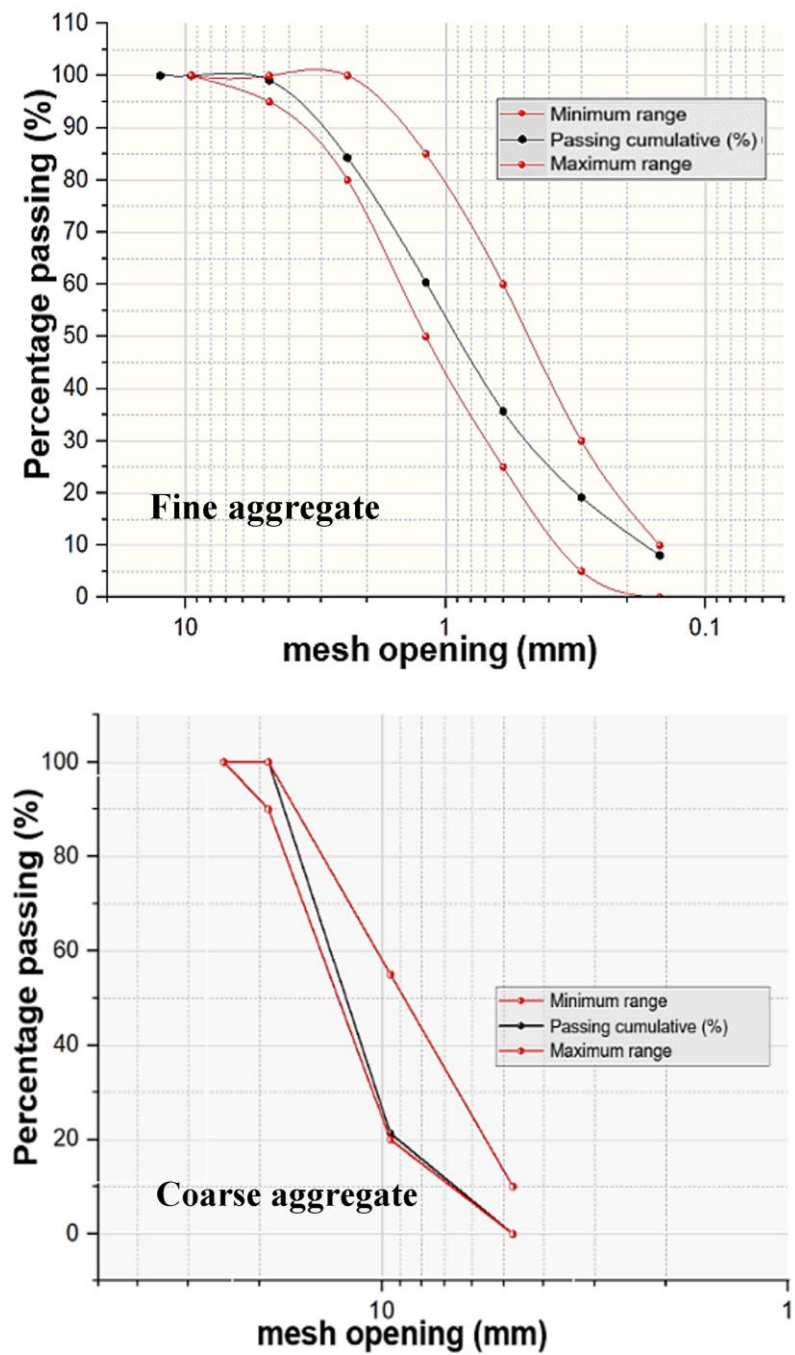
2.1. Materials

Below are the characteristics of the materials used in the conventional concrete mix, both without and with sustainable additives. Specifically, the following aspects are described: the particle size distribution of the fine and coarse aggregates, as they are locally sourced; the experimental additives; the mix proportions used in the research; and the standards that are met to obtain the

results.

2.1.1. Aggregates

In Figure 1, the sand gradation showed that the material complies with ASTM C 33, as 99.1% passes through the No. 4 sieve, exceeding the minimum requirement without surpassing the maximum allowed. For the No. 30 sieve, 35.7% is within the acceptable range, and only 8.1% passed the No. 100 sieve, remaining below the established limit. This ensured the material was suitable for use in concrete according to the ACI C-211 method.



Note: Comparison with ACI-211 limits.

Figure 1. Granulometry of coarse and fine aggregates

On the other hand, the coarse aggregate confirmed the absence of particles larger than 3/4" and smaller than the No. 4 sieve, characterizing it as a medium to small-sized material. The step-shaped curve indicates a less continuous gradation compared to the fine aggregate; however, it remains within the parameters required for ASTM C 33, Gradation #67. This curve was considered acceptable for the particle size distribution of the stone used in the concrete for the experiment.

2.1.2. Coffee Husk Ash (CHA)

Upon reviewing Table 1, both materials exhibited very different characteristics that influence their function in concrete. Type I OPC shows a high percentage of CaO, which allows it to set and gain strength when mixed with water, making it essential for structures that require structural strength. In contrast, CHA achieved $\text{SiO}_3 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 > 70\%$, but with a lower CaO content. This difference causes CHA to act as a pozzolanic material; that is, it does not set on its own but reacts with the cement compounds to form products that reinforce the mixture and improve the concrete's durability. Additionally, the ash demonstrated low levels of sulfates and moisture, which facilitates its use as a supplementary material without affecting the stability of the mix.

Table 1. Chemical characteristics of OPC vs. CHA

Requirements: ASTM C618-2025	OPC	CHA
$\text{SiO}_3 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 > 70\%$ (%)	28,74	80,43
$\text{SO}_3 < 4\%$ (%)	2,64	0,04
CaO (%)	63,15	5,73
$\text{Na}_2\text{O} + 0,658\text{K}_2\text{O} < 4\%$ (%)	0,64	2,31
Moisture content $< 3\%$ (%)	$< 1,00$	2,09
LOI $< 10\%$ (%)	3,08	4,47

Note: OPC refers to Type I Portland cement. Regarding CHA. The ash was calcined in a furnace at 750 °C for 3.5 hours.

Table 2. Physical characteristics of OPC vs CHA

Physical Requirements	OPC	CHA
Fineness (retained on #325) $< 34\%$	15-20*	32,68
Pozzolanic Index $> 75\%_{7\text{days}}$ (%)	N/A**	75,37
Apparent Density (g/cm^3)	3,15*	2,21
Specific Surface (m^2/g)	0,35*	0,42
Absorption (%)	N/A**	4,38

Notes: *Estimates based on typical specifications. **Not applicable, as the standard is based on pozzolanic materials used in concrete.

Table 2 shows that OPC has a higher density and is more compact compared to ash. Regarding fineness, both materials meet the regulatory requirements, although ash is close to the allowable limit, which may affect the reactivity and workability of the mix. The specific surface area of ash is slightly higher, providing a greater contact area for

chemical reactions within the concrete. The pozzolanic index and absorption do not apply to OPC because these properties are intended for supplementary materials such as fly ash, but by meeting the regulatory requirement, the pozzolanic reactivity within the concrete mixture is assured.

2.1.3. Coconut Fiber (CF)

According to Table 3, fiber treatment influenced its composition and therefore, its performance within the concrete. The increase in cellulose after alkali treatment (sodium hydroxide) indicates higher purity and a more suitable structure for reinforcing the cement matrix, since cellulose provides strength and flexibility. The reduction of hemicellulose and lignin is important because high levels negatively affect the adhesion of the fiber in concrete. The recommended values, taken from scientific publications, serve as a reference to ensure that plant fibers actually help improve the mechanical properties of the material. The treated fiber that met the recommended values demonstrates compatibility within the cement matrix of the concrete, ensuring durability.

The analysis of the data presented in Table 4 shows that the physical and mechanical properties of CF—such as length, diameter, density, elongation, and tensile strength—remain within ranges similar to those reported by recent studies from various parts of the world. For example, the average length of the fibers studied is close to values cited by leading researchers, indicating proper selection and characterization of the material. Likewise, the average diameter and density of the fibers in this research are consistent with documented intervals. Regarding elongation and tensile strength, the experimental values fall within international standards, suggesting that the coconut fiber used has strong potential to enhance the toughness and energy absorption capacity in concrete composite materials.

Upon analyzing Table 5 and Table 6, it was observed that the experimental mixtures incorporated different proportions of combined CHA and CF, while keeping the other components constant. This reflects mix design characteristics such as slump, maximum aggregate size, w/c ratio, and the volume of coarse aggregate, which remained within the ranges recommended by technical standards, ensuring comparability among the different tests of physical properties and of compressive strength ($f'c$), indirect tensile strength, and flexural strength. A water-to-cement ratio of 0.67 was selected to compensate for the high-water absorption capacity of the CF and the porous nature of the CHA. Studies indicate that lower w/c ratios in bio-based concretes can lead to drastic reductions in workability, hindering proper compaction and increasing porosity. This ratio ensures adequate rheology for the specific aggregate gradation used. The inclusion of CHA and CF did not significantly change the amount of water or the entrapped air, demonstrating that these

materials can be incorporated without affecting workability. Additionally, the temperature and other test conditions were kept controlled, which reinforces the validity of the results obtained.

The figure presents two key data sets regarding the experimental evaluation of concrete mixes modified with CHA and CF. The first table outlines four different concrete mix designs (D1–D4), indicating the specific dosages of each component per cubic meter. The control mix (D1) contains only OPC, sand, gravel, and water, with

no additives. The experimental mixes (D2, D3, D4) substitute portions of cement with increasing percentages of CHA (1.5%, 3.0%, 5.0%) and varying amounts of CF (0.65%, 0.45%, 0.35%). This systematic variation aims to assess the synergy of CHA and CF in enhancing concrete's performance while maintaining consistent quantities of sand, gravel, and water. Each formulation follows the ACI 211 standard for a target compressive strength of 21 MPa, using aggregates from a specific quarry in Lima and a commercial Type I cement brand.

Table 3. Chemical comparison of CF without and with treatment

Chemical requirements	Untreated	Treated (ASTM D6942)	Recommended range*
Cellulose	42,38	53,16	40-60
Hemicellulose	13,08	9,85	5-15
Lignin	39,62	34,19	20-35

Note: Recommended values for concrete based on international scientific reviews on plant fibers in cementitious materials, which suggest optimal ranges to maximize durability and fiber-to-matrix bonding in concrete: (Nabi Saheb and Jog [20]; Rajesh, Pitchaimani, and Rajini [21]; Castro Jerónimo et al. [22])

Table 4. Physical characteristics of the CF vs other research

Physical requirements	Research	Caruso et al. [6]	Kilani et al. [8]	Bindhu et al. [23]
Average length (mm)	42,38	50,00	30-60	40,00
Average diameter (mm)	136,74	100-450	100-450	100-450
Density (g/cm ³)	1,33	1,15-1,46	1,15-1,46	1,15-1,46
Elongation (%)	2,90	1,5-5,0	2,0-6,0	1,5-6,0
Tensile Strength (MPa)	310,00	175-270	131-228	131-228

Notes: Compared with the references: (1) (Caruso et al. [6]); (2) (Kilani et al. [8]); (3) (Bindhu et al. [23])

Table 5. Control and experimental mix designs

Design	Combination	OPC	Sand	Gravel	Water	CHA	CF
D1	0% (Control)	332,00	927,00	814,00	223,00	0,00	0,00
D2	1,5%CHA+0,65%CF	327,02	927,00	814,00	223,00	4,98	2,13
D3	3,0%CHA+0,45%CF	322,04	927,00	814,00	223,00	9,96	1,45
D4	5,0%CHA+0,35%CF	315,40	927,00	814,00	223,00	16,60	1,10

Notes: The ACI 211 mix design has been considered, using aggregates from the Trapiche quarry in Lima and Type I cement.

Table 6. Characteristics of the mix design values

Property	NTP	ASTM / International Standard
Slump (mm)	NTP 339.035	ASTM C143
Air content (%)	NTP 339.080	ASTM C231 (pressure), C173 (volumetric), C138 (gravimetric)
Unit weight (kg/m ³)	NTP 339.046	ASTM C138
Temperature (°C)	NTP 339.184	ASTM C1064
Compressive strength	NTP 339.034	ASTM C39
Splitting tensile strength	NTP 339.084	ASTM C496
Flexural strength	NTP 339.078	ASTM C78
Modulus of elasticity	NTP 339.186	ASTM C469

The second table specifies the standardized properties measured in the experimental program. Each key property (slump, air content, unit weight, temperature, compressive strength, splitting tensile strength, flexural strength, and modulus of elasticity) is associated with its corresponding NTP (Peruvian Technical Standard) and ASTM or international standard. This alignment ensures comparability and reliability of results by adhering to recognized methodologies. For example, slump is measured by NTP 339.035 and ASTM C143, and compressive strength by NTP 339.034 and ASTM C39. By structuring the experiment in this way, the study provides a comprehensive framework for evaluating the combined effect of pozzolanic ash and natural fiber on conventional concrete, ensuring both laboratory rigor and real-world relevance.

2.2. Experimental Methodology

The experimental process was structured according to Figure 2, beginning with the verification of the appropriate use of sustainable additives. Three experimental treatments were established with different combined proportions of ash and fiber additions (D2: 1.5% CHA + 0.65% CF; D3: 3% CHA + 0.45% CF; D4: 5% CHA + 0.35% CF), along with a control design, totaling 200 analysis units. The evaluation considered both physical properties (workability, unit weight, air content, and temperature) and mechanical properties: compressive strength, tensile strength, flexural strength, and elasticity, following standard regulatory procedures.

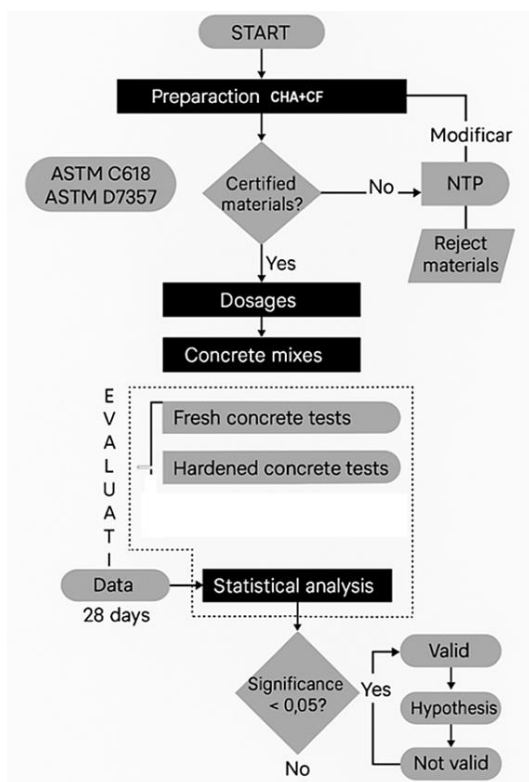


Figure 2. Methodological flowchart

2.3. Pretreatment of CHA and CF for Use as Concrete Additives

The CF was pretreated in a solution of distilled water with NaOH at a concentration of 5%, immersed for 3 hours, followed by oven drying at 100 °C, according to ASTM D7357 [24]. Figure 3-a depicts CF before and after the treatment.

Regarding the CHA, this material was produced by calcining recycled coffee husks from the coffee industry. Figure 4 illustrates the process: the husks were calcined at over 750 °C for 3-3.5 hours [10], [25], [26], cooled, and sieved to separate organic material. The sieved product was then ground with a manual mill to reduce its size, followed by further grinding in a high-powered electric grinder to achieve a particle size similar to cement (>66% passing through #325 mesh, see Figure 3-b).

The materials resulting from both the calcination process for CHA and the pretreatment of cellulose fibers from CF have been shown to comply with ASTM C618 and ASTM D7357 standards [24], [27], confirming their suitability as additives. Evidence regarding the process of generating CHA as a pozzolan is shown in the subprocesses of Figure 4.a,b,c,d.

This process is consistent with previous studies on ash production [3], [11], [28] for use as a replacement for OPC, as well as on CF pretreatment [13], [29], [30]. Following the processes shown in Figure 3 and Figure 4, the raw material for modifying the concrete is obtained. Physical tests are then performed prior to mechanical property testing (specifically compressive, splitting tensile, and flexural strength, as well as modulus of elasticity) on the specimens shown in Figure 5.

2.4. Microstructural and Thermal Characterization

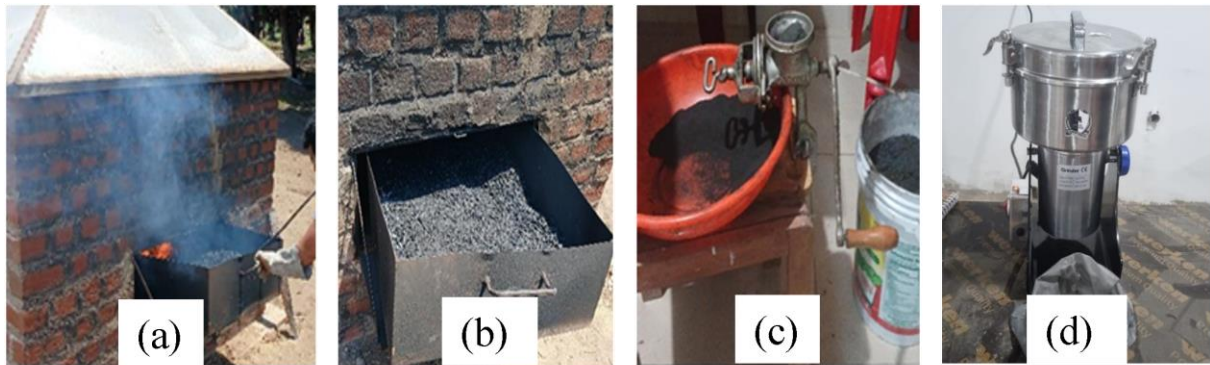
2.4.1. Thermal Analysis (TGA/DTA)

The thermal behavior and stability of the biomass was evaluated with the tests (a) Thermal Analysis by Differential Scanning Calorimetry (DSC)/ Differential Thermal Analysis (DTA) and (b) Thermogravimetric Analysis (TGA), by means of a simultaneous thermal analyzer TGA-DTA-DSC (SetSys Evolution, Setaram Instrumentation) at the Polymer Laboratory, Department of Materials Engineering, National University of Trujillo. A sample of approximately 19.5 mg was heated from room temperature (25 °C) to 900 °C at a heating rate of 20 °C/min in a nitrogen atmosphere with a flow rate of 10 mL/min. The objective of the test was to identify the thermal decomposition stages and determine the optimal calcination temperature range, following standard protocols for the characterization of CHA.



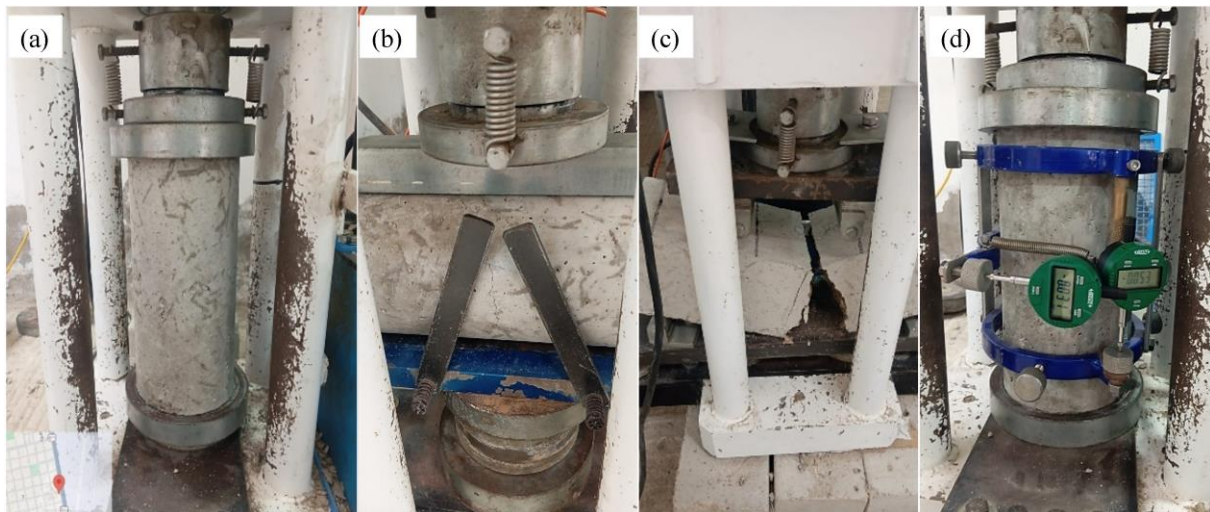
Notes: (a) Fibers before and after alkaline treatment; (b) OPC vs. CHA; from left to right

Figure 3. Inputs and pretreatment in research.



Notes: From right to left: a) calcination, b) cooling, c) grinding, and d) pulverization.

Figure 4. Production process of CHA as an OPC substitute



Notes: (a) compressive s., (b) indirect tensile s., (c) flexural s., (d) elasticity.

Figure 5. Tests for mechanical properties.

2.4.2. X-Ray Diffraction (XRD)

Mineralogical characterization of the CHA and the hardened concrete pastes (Control vs. Optimal Mix). It was performed with the X-ray diffractometer (Bruker equipment, model D8 Focus), with copper radiation ($\text{Cu-K}\alpha=0.15418 \text{ nm}$), 40 mA current and acceleration voltage of 40 kV, with a Lynxeye detector with energy selectivity. The analysis was performed at a range of

angles (2θ) from 5° to 60° in 0.02° steps. The time per step was 1 s. To calculate the composition of the crystalline phases and the amorphous part, the Reference Intensity Ratio (RIR) method was applied. The analysis focused on identifying amorphous silica humps in the ash and monitoring the consumption of portlandite (CH) in the hardened paste. The reduction of CH peaks served as a primary indicator of the pozzolanic reaction products

(C-S-H formation) generated by the CHA addition.

2.4.3. Scanning Electron Microscopy (SEM)

The morphology of the hydration products and the fiber-matrix interface were examined using a Scanning Electron Microscope (FEI, Model: Quanta 650). Fractured surfaces from the 28-day cured specimens were gold-coated to ensure conductivity before imaging. Special attention was given to the Interfacial Transition Zone (ITZ) between the treated coconut fiber and the cement matrix to visualize the physical interlocking achieved by the alkali treatment, as well as to observe the densification of the matrix attributed to the pozzolanic activity.

3. Results and Discussions

3.1. Thermal Decomposition Analysis (TGA/DTA)

Figure 6 illustrates the TGA and DTA curves for the raw husk. The TGA curve reveals a total mass loss of approximately 84% throughout the heating process, occurring in three distinct stages. The first stage, observed between 80 °C and 110 °C, corresponds to the evaporation of moisture and volatile components, indicated by an endothermic peak at 100 °C in the DTA curve. The second and most significant mass loss occurs in the range of 230 °C to 350 °C, attributed to the thermal degradation of hemicellulose and cellulose. The DTA curve shows a prominent exothermic peak around 390 °C, signaling the combustion of lignin and fixed carbon. Beyond 600 °C, the mass loss stabilizes, confirming that the selected calcination temperature of 750 °C is sufficient to remove all organic matter and obtain a silica-rich ash suitable for use as a pozzolanic material.

3.2. Physical Properties of the Control and Experimental Concrete Mixes

Figure 7 shows important changes when the concrete is modified with the addition of pozzolan together with fiber. The slump values in D2 showed that the CF when it is higher, affected the workability, due to the absorption of water and the surface characteristics of the ash; but it was recovered in D3 and D4, which shows that the reduction of the fiber does not affect workability. Regarding air content, a decreasing trend was observed as the proportion of CHA and CF increased, because these additions densify the

mixture and reduce the incorporated air. The unit weight showed a stable behavior in D2 and D3, but increased significantly in D4 due to the combined effect of higher ash density and fiber-enhanced compaction. The increase in temperature during mixing is due to the heat generated by the additional chemical reactions introduced by the pozzolanic ash.

3.3. Mechanical Properties of the Studied Concrete

Figure 8 comprehensively shows the results of the experimental designs as percentage variations with respect to the pattern (D1). It demonstrates that design D2 achieved the best mechanical performance integrating ash and fiber, recording favorable increases in f_c +8.08%, flexion +6.08%, and elasticity +5.81%, compared to conventional concrete observed at 28 days of curing. The synergy of the D2 combination strengthened the load-bearing capacity of the material and increased its stiffness and elastic deformation capacity, fundamental for concrete used in the design of structural elements. In contrast, D3 demonstrates that traction depends on the fiber, which contributes to increases in stiffness; however, the E data from D3 showed that the increase in CHA compromises the strength gain in concrete.

It has been demonstrated that the combined addition of CHA and CF in concrete, in optimal proportions, significantly improves the physical-mechanical properties of the material compared to the mix without additives. The results show that design D2 surpasses the pattern in compression (+8.08%), flexion (+6.08%) and elasticity (+5.81%) at 28 days of curing, which evidences the existence of a favorable synergy between both agricultural residues. This behavior coincides with the findings of de Almeida et al. [10] and the study by Atahu et al. [3], where the combination of coffee husk ash and fibers (polypropylene or coconut) increases f_c and flexion in similar ranges, provided that moderate dosing is respected. Additionally, research such as that by Asfaw et al. [28], Castro et al. [31], and Rafael [32] corroborates that the incorporation of agroindustrial ashes and vegetable fibers improves the mechanical resistance and durability of concrete, although they warn that excess additives can negatively affect the workability and stiffness of the material. The results of this research confirm that balance in dosing is key, since high doses (as in D4) compromise stiffness gain and can generate a less efficient internal structure.

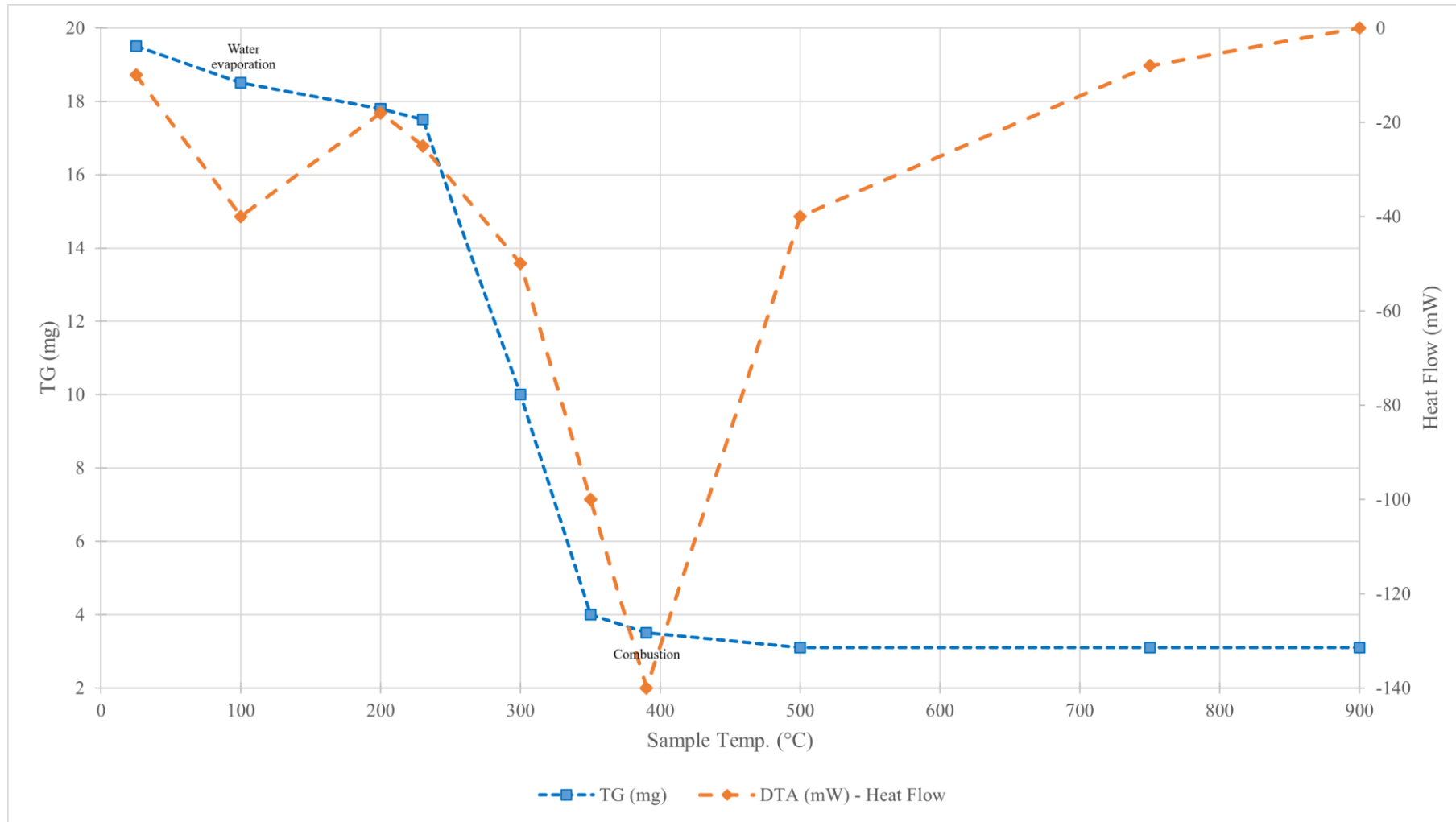


Figure 6. Thermogravimetric (TGA) and Differential Thermal Analysis (DTA) curves of the CHA

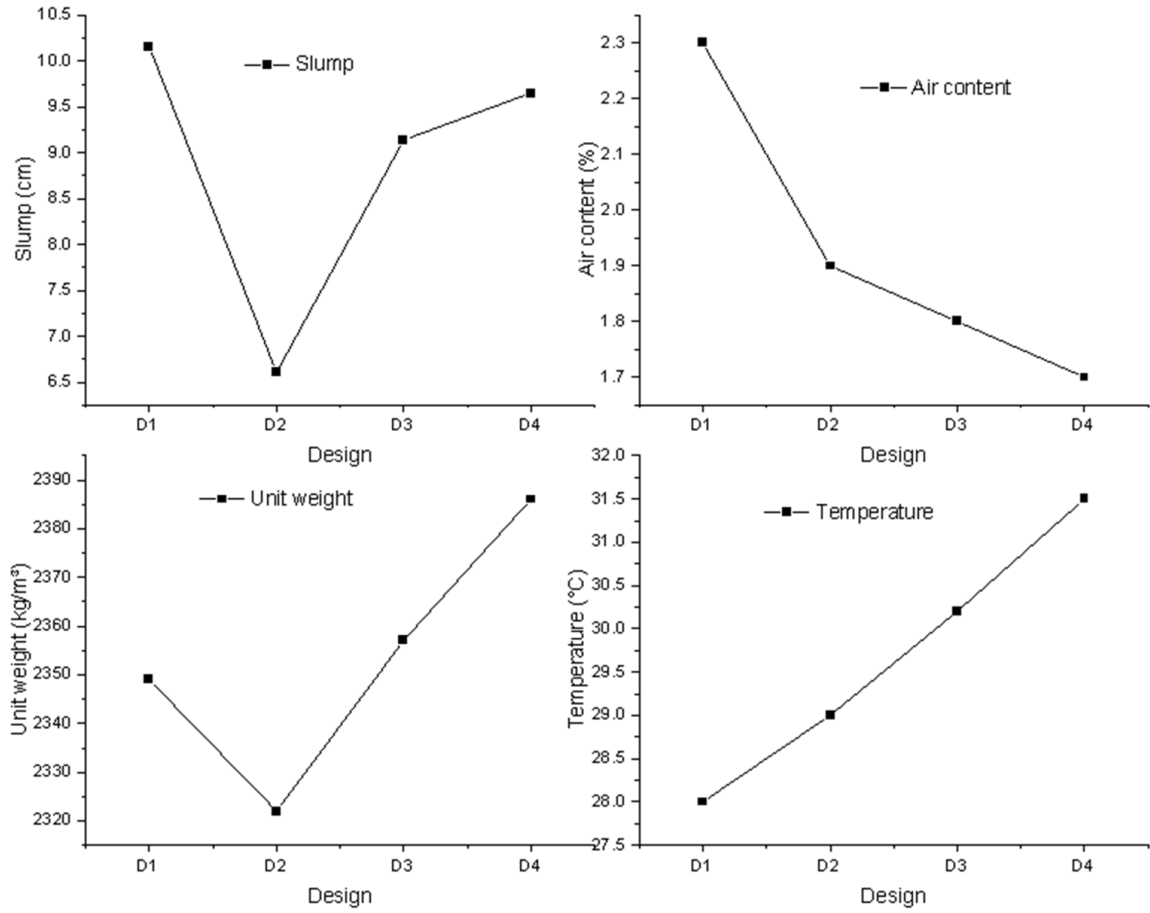


Figure 7. Physical properties of the control and experimental concrete mixes

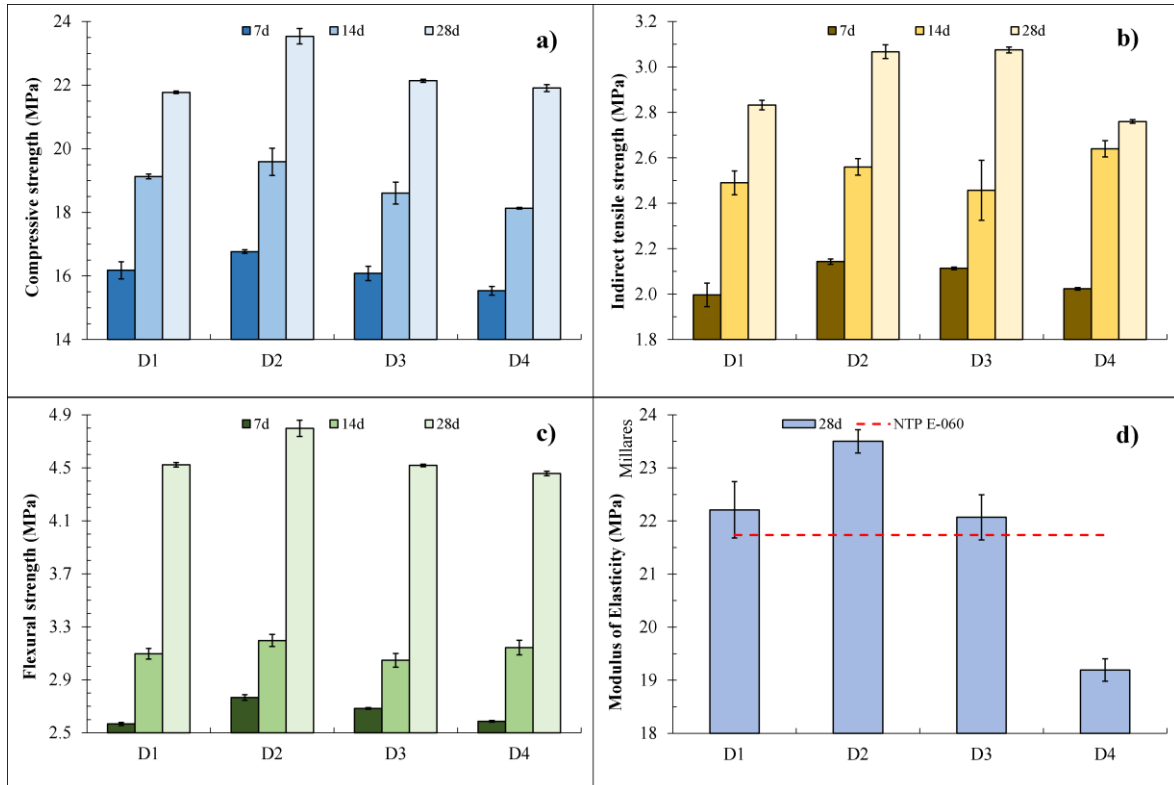


Figure 8. Mechanical properties of control and experimental concrete designs

3.4. Optimal Concrete Design Evaluation (ANOVA)

The boxplots (Figure 9) indicate that all mixes develop strength with curing age, but the combined CHA–CF modification yields a distinctly different dispersion pattern across properties. The optimal formulation (D2) shows a consistent upward shift in central tendency together with comparatively tighter interquartile ranges at later ages, suggesting a more homogeneous hardened matrix and reliable performance under the selected dosage window. This behavior is consistent with the expected pozzolanic contribution of coffee-residue ashes, which promotes matrix densification through secondary hydration products, and with the crack-bridging role commonly attributed to coconut fibers when properly treated and dosed. In contrast, the higher-addition mix (D4) exhibits a clear stiffness penalty and a less favorable distribution, supporting the interpretation that excessive solid additions can disrupt packing and continuity of the cementitious phase, offsetting the benefits of fiber reinforcement. Overall, the distributional evidence complements the ANOVA/Tukey

(Table 7) outcomes and reinforces the need for dosage control to maximize synergy while avoiding performance reversal at high replacement levels.

3.5. Microstructural Analysis

3.5.1. X-Ray Diffraction (XRD) Analysis

X-ray diffraction analysis (Figure 10) revealed significant mineralogical differences between the control (D1) and optimal (D2) mixes. Portlandite $[Ca(OH)_2]$ decreased from 5.6 wt% to 4.3 wt% (-23.2%), confirming pozzolanic reaction with CHA silica. The amorphous phase increased from 19.0 wt% to 21.7 wt%, indicating C-S-H gel formation. Quartz content rose from 29.0 wt% to 32.2 wt%, validating ash incorporation. Calcite diminished from 5.3 wt% to 2.7 wt%, suggesting secondary reactions. Crystalline hydrates (diopside, cordierite) were substantially reduced in D2, indicating a shift toward an amorphous gel-dominated matrix. These mineralogical changes directly account for the enhanced mechanical properties.

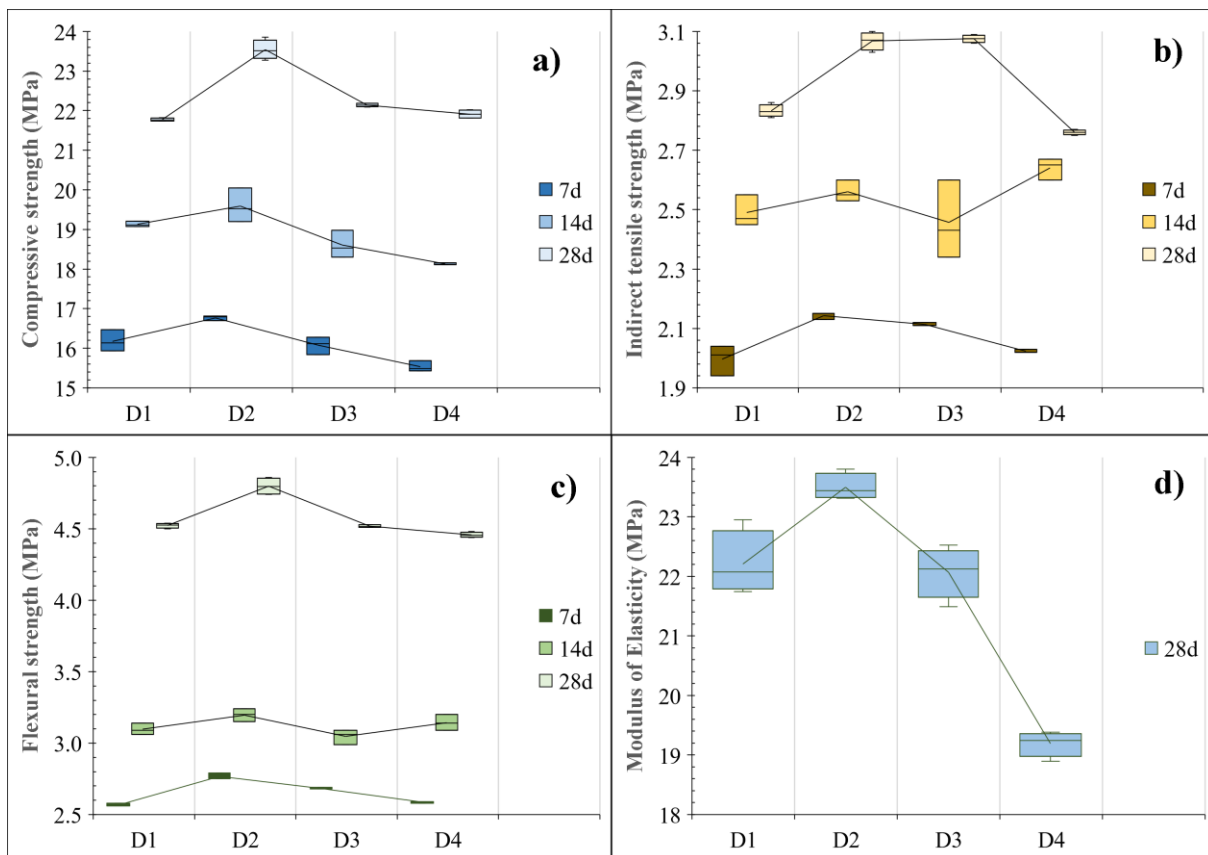
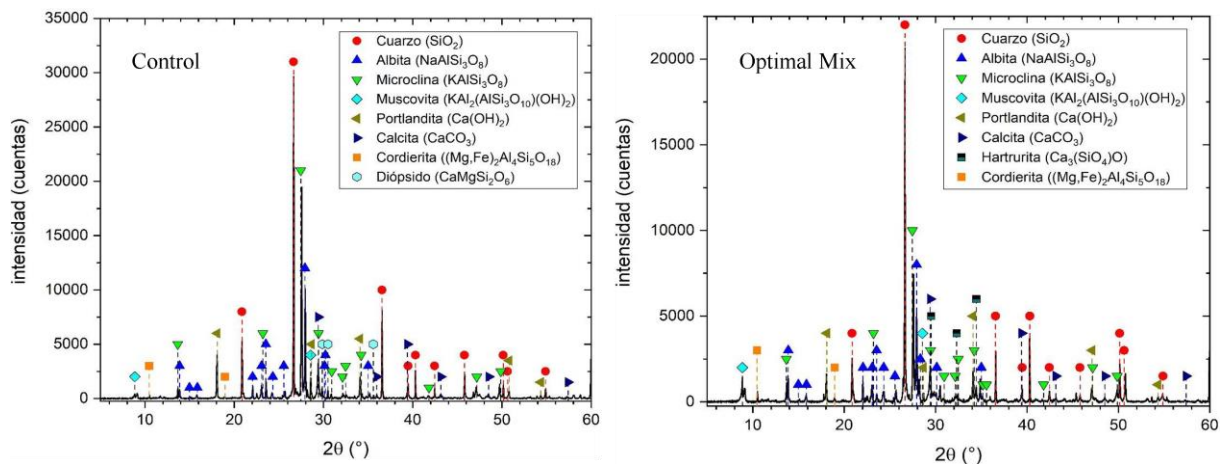


Figure 9. Boxplots of the mechanical properties of concrete mixes at different curing ages

Table 7. Inferential statistical analysis (ANOVA and Tukey HSD)

Property	Mix Design	Mean (MPa)	SD	ANOVA (F-value)	p-value	Post-hoc (Tukey)
CS	D1	21.78	0.04	100.95	<0.001	-
	D2	23.54	0.24			D2 > D1 (Sig)
	D3	22.14	0.05			D3 ~ D1 (NS)
	D4	21.91	0.11			D4 ~ D1 (NS)
FS	D1	4.52	0.02	35.84	<0.001	-
	D2	4.80	0.06			D2 > D1 (Sig)
	D3	4.52	0.01			D3 ~ D1 (NS)
	D4	4.46	0.02			D4 ~ D1 (NS)
ITS	D1	2.83	0.02	143.62	<0.001	-
	D2	3.07	0.03			D2 > D1 (Sig)
	D3	3.08	0.01			D3 > D1 (Sig)
	D4	2.76	0.01			D4 < D1 (Sig)
EM	D1	22,210	532	117.92	<0.001	-
	D2	23,500	220			D2 > D1 (Sig)
	D3	22,068	427			D3 ~ D1 (NS)
	D4	19,192	210			D4 < D1 (Sig)

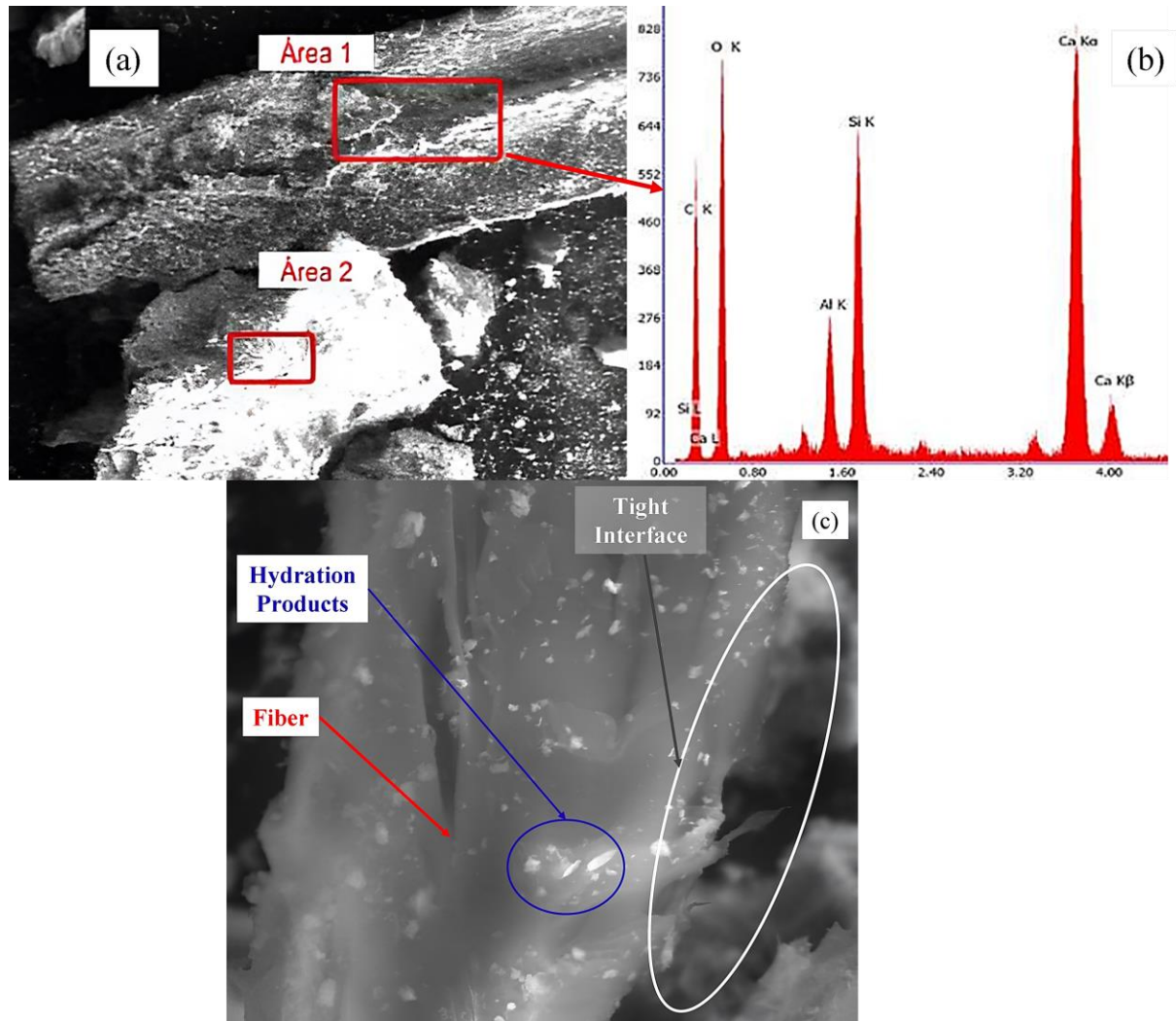
Notes: SD = Standard Deviation; Sig = Significant difference ($p < 0.05$); NS = Not Significant. Analysis performed on 28-day curing age data (n=4 samples per group)

**Figure 10.** X-ray diffractogram of the control sample and optimal mix

3.5.2. Interfacial Transition Zone (SEM)

SEM micrographs (Figure 11-a, c) show the CF embedded within the cementitious matrix, with adhered hydration products and a visibly dense Interfacial Transition Zone (ITZ), which is consistent with effective mechanical interlocking after alkaline treatment. The EDS spectrum from Area 1 (Figure 11-b) is dominated by Ca and O, with Si and Al contributions, supporting the presence of calcium-silicate hydration products (C-S-H

gel) at/near the interface, while C enrichment in fiber-rich regions confirms the organic reinforcement in intimate contact with the matrix. The lack of an evident debonding gap and the occurrence of matrix residues attached to the fiber surface support improved stress transfer and crack-bridging mechanisms, responsible for the enhanced post-cracking flexural strength (+6.2%) observed in the optimal mix.



Notes: (a) General distribution of fibers and aggregates in the matrix (40x). (b) Spectrum (EDS) obtained from the analysis in Area 1. (c) High-magnification view (1600x) of the fiber surface, showing surface roughness and adhesion of hydration products.

Figure 11. SEM micrographs of the fiber-reinforced concrete

4. Conclusions

The study demonstrates that the combined incorporation of CHA and CF can significantly improve the physical and mechanical properties of conventional concrete for structural design. The key findings are as follows:

- The mixture containing 1.5% CHA and 0.65% CF (D2) demonstrated the highest mechanical performance, achieving statistically significant increases ($p < 0.001$) in compressive strength (+8.1%), flexural strength (+6.2%), and modulus of elasticity (+5.8%) relative to the control.
- The mechanical gains are directly supported by physicochemical evidence. XRD analysis confirmed a 23.2% reduction in portlandite content, verifying the effective pozzolanic reactivity of the ash calcined at 750°C. Concurrently, SEM imaging revealed a dense Interfacial Transition Zone (ITZ) with no visible debonding gaps, indicating that the alkaline

treatment of the fibers promoted effective mechanical interlocking and stress transfer.

- Statistical analysis (Tukey HSD) identified a clear dosage threshold. While moderate substitutions improved performance, the highest dosage (D4: 5% CHA + 0.35% CF) caused a significant 13.6% reduction in stiffness (ME). This decline indicates that excessive inclusion of agro-waste fines disrupts particle packing and increases matrix porosity, negating the reinforcement benefits.
- The study validates that replacing 1.5% of cement with regional agro-industrial waste is technically viable for structural concrete applications ($f'_c > 21$ MPa).

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