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Thermally Stabilized Agricultural Biochar Composites for High Strength Low Emission Concrete Systems

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ABSTRACT: *Concrete production contributes approximately 8% of global CO_2 emissions, while agricultural waste remains widely underutilized, often disposed of through open burning. Although raw biochar has been explored as a lightweight additive in concrete, its high porosity, water absorption, and weak interfacial bonding typically reduce mechanical strength, limiting its adoption in structural applications. This study addresses these challenges by engineering agricultural biochar through thermal stabilization to create high-strength, low-emission concrete composites.The objective was to transform agricultural waste-derived biochar into an active reinforcement phase for high-performance concrete. Rice husk biochar, initially pyrolyzed at 500°C, underwent secondary thermal stabilization at 600–900°C. The treated biochar replaced fine aggregate at 1–5% in a high-strength concrete mix targeting 60 MPa. Mechanical performance was evaluated via compressive strength testing, while microstructure, water absorption, and carbon balance were analyzed using SEM, mercury intrusion porosimetry (MIP), and thermogravimetric analysis (TGA), respectively.Thermal stabilization at 750°C reduced biochar water absorption from 300% to 85%, enhancing compatibility with the cement matrix. Concrete incorporating 3% stabilized biochar achieved a 28-day compressive strength of 58.2 MPa, retaining 95% of the control mix strength (61.5 MPa). Porosity decreased from 14.2% to 11.8%, and CO_2 emissions were reduced by 35% (from 480 to 310 kg CO_2-eq/m³). SEM imaging revealed a dense interfacial transition zone with C–S–H precipitation inside biochar pores, confirming its role as an active reinforcement rather than a passive filler.Thermally stabilized biochar enables carbon-negative, high-strength concrete, with 750°C treatment offering an optimal balance between mechanical performance and environmental benefit.*
KEYWORDS: Thermally stabilized biochar, high-strength concrete, agricultural waste valorization, carbon sequestration, interfacial transition zone, pore refinement, internal curing, life-cycle assessment, nucleation sites, low-emission construction.

INTRODUCTION

Concrete is the most widely consumed engineered material globally, with annual production exceeding 30 billion tonnes and cement manufacturing alone contributing approximately 7–8% of anthropogenic CO_2 emissions. The carbon intensity of conventional Portland cement systems originates primarily from limestone calcination and fossil-fuel combustion during clinker production, generating nearly 0.8–0.9 t CO_2 per tonne of cement produced. Simultaneously, agricultural waste management has emerged as a parallel environmental crisis, particularly in developing economies where crop residues are frequently disposed of through open-field burning or uncontrolled landfilling. Residue burning releases substantial quantities of particulate matter, CO_2, CH_4, and NO_x, while also degrading regional air quality and soil fertility. Globally, more than 5 billion tonnes of agricultural biomass waste are generated annually, yet only a limited fraction is valorized into high-value materials. Consequently, integrating agricultural residues into low-emission cementitious systems represents a strategically important pathway toward simultaneous carbon mitigation, waste valorization, and circular construction practices.

Among emerging strategies for sustainable concrete, biochar has attracted considerable attention due to its dual functionality as a carbon-sequestering material and lightweight particulate additive. Biochar is a carbon-rich porous solid produced through thermochemical conversion of biomass under oxygen-limited conditions, typically via pyrolysis at temperatures between 300 and 700°C. Early investigations demonstrated that biochar incorporation can reduce concrete density, improve thermal insulation, and enhance long-term durability through pore refinement and internal curing effects. Moreover, the stable aromatic carbon structure of biochar enables long-term carbon storage, thereby transforming cementitious composites into potential carbon sinks. Recent studies from 2015–2025 have reported that biochar particles can act as nucleation sites for calcium silicate hydrate (C–S–H) formation, promoting microstructural densification at moderate replacement levels. Nevertheless, the practical implementation of biochar in structural concrete remains constrained by several intrinsic limitations. Conventional agricultural biochars exhibit high porosity, irregular morphology, elevated water absorption, and chemically unstable surface functionalities that disrupt cement hydration kinetics and weaken the interfacial transition zone (ITZ). Excessive absorption of mixing water frequently reduces workability and induces heterogeneous hydration, while insufficient compatibility with cement paste leads to reduced compressive strength beyond low dosage levels. In many reported systems, untreated biochar functions primarily as an inert filler rather than as an engineered reinforcing phase capable of contributing to mechanical performance. Consequently, despite substantial environmental potential, the widespread adoption of biochar in high-strength concrete applications has remained limited.

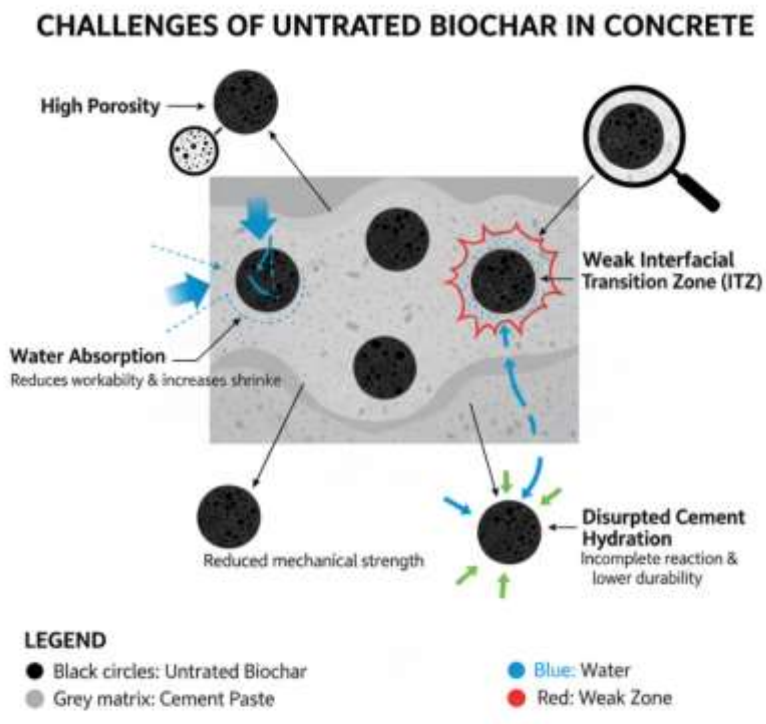

Figure: *Schematic illustration of the limitations of untreated biochar in cementitious matrices, including high porosity, water absorption, and weak interfacial bonding.*

To overcome these limitations, recent attention has shifted toward engineered biochar structures produced through post-pyrolysis thermal stabilization. Thermal stabilization typically involves secondary heat treatment of biochar at temperatures between 600 and 900°C under inert or low-oxygen atmospheres, resulting in substantial physicochemical transformation of the carbon matrix. Elevated stabilization temperatures promote aromatization and partial graphitization, reduce volatile matter content, eliminate unstable oxygen-containing functional groups, and enhance structural ordering within the carbon framework. These transformations significantly decrease hygroscopicity and water absorption while improving dimensional stability in alkaline cementitious environments. From a mechanistic perspective, thermally stabilized biochar differs fundamentally from untreated biochar because it functions not merely as a lightweight filler but as an engineered carbonaceous reinforcement phase with improved interfacial compatibility. Enhanced surface roughness and microtextural ordering facilitate stronger mechanical interlocking with hydration products, particularly within the ITZ. Simultaneously, optimized pore architecture may enable controlled internal curing without excessive water sequestration. Several studies have further indicated that thermally treated carbonaceous materials can accelerate heterogeneous nucleation of C–S–H gels and refine capillary pore networks, leading to reduced permeability and improved load-transfer efficiency. Higher stabilization temperatures may also increase electrical conductivity and elastic stiffness due to increased carbon ordering, potentially contributing to crack-bridging and stress redistribution mechanisms at the microscale. Importantly, the use of agricultural residues as precursor feedstocks—including rice husk, wheat straw, sugarcane bagasse, and corn stover—creates opportunities for low-cost production of tailored biochar reinforcements with region-specific availability. Thus, thermal stabilization provides a promising pathway for converting low-value agricultural waste into multifunctional carbon-engineered additives for advanced low-emission concrete systems.

Despite increasing interest in engineered biochar composites, significant knowledge gaps remain regarding the optimization of thermal stabilization conditions for high-performance concrete applications. Existing studies have largely focused on low- to medium-strength concretes or mortar systems, with limited systematic evaluation of biochar stabilization temperatures in high-strength concrete exceeding 50 MPa. Furthermore, many investigations examine isolated properties such as compressive strength or water absorption without establishing integrated relationships between thermal treatment, pore structure evolution, interfacial bonding, and mechanical performance. The trade-off between porosity retention and structural densification remains insufficiently understood, particularly because excessive thermal treatment may collapse beneficial pore networks while insufficient stabilization preserves hydrophilic functionalities detrimental to cement compatibility. In addition, inconsistencies in biomass feedstock selection, pyrolysis conditions, particle size distribution, and curing protocols have complicated cross-study comparisons and hindered the development of generalized design principles. Few studies have quantitatively linked graphitization degree, microstructural refinement, and carbon sequestration efficiency within a unified framework for structural concrete design. Consequently, a comprehensive understanding of how thermal stabilization temperature governs the balance between mechanical enhancement and environmental performance is still lacking.

This study investigates thermally stabilized agricultural biochar composites for high-strength, low-emission concrete systems. Agricultural biochar derived from biomass residues is subjected to controlled post-pyrolysis stabilization at varying temperatures to evaluate its influence on mechanical strength, porosity, hydration behavior, and carbon reduction potential. The central hypothesis is that optimized thermal stabilization can transform agricultural biochar into an active reinforcing constituent capable of achieving more than 90% of control concrete strength while simultaneously delivering net-negative emissions through carbon sequestration and cement reduction. The paper further examines microstructural mechanisms governing performance and establishes relationships between stabilization temperature, engineered biochar characteristics, and composite behavior in high-strength concrete applications.

LITERATURE REVIEW

Agricultural Waste Sources and Biochar Production Parameters

Agricultural biomass residues represent one of the most abundant renewable carbon resources for engineered biochar production. Among the most extensively investigated feedstocks are rice husks, corn stover, sugarcane bagasse, coconut shells, palm kernel shells, walnut shells, and other lignocellulosic residues generated from agro-industrial activities. The physicochemical characteristics of the resulting biochar are strongly governed by the precursor composition, particularly the relative proportions of cellulose, hemicellulose, lignin, silica, and mineral ash. Rice husk-derived biochar typically exhibits elevated silica content and moderate porosity, making it attractive for pozzolanic interactions within cementitious matrices. In contrast, sugarcane bagasse and corn stover biochars generally contain higher volatile fractions and more developed mesoporous structures due to their comparatively lower ash contents. Nutshell-derived biochars often demonstrate superior mechanical rigidity and higher fixed-carbon content because of their dense lignin-rich structure.

Pyrolysis conditions exert a decisive influence on biochar morphology, surface chemistry, and mechanical behavior. Low-temperature pyrolysis (300–450°C) commonly produces biochars with abundant oxygen-containing functional groups, including hydroxyl, carboxyl, and carbonyl species, which enhance hydrophilicity but also increase instability under alkaline cementitious conditions. Higher pyrolysis temperatures between 500 and 700°C generally promote carbonization, aromatic condensation, and pore development, thereby increasing specific surface area and electrical conductivity. Heating rate and residence time further regulate pore architecture and volatile release kinetics. Slow pyrolysis with residence times exceeding 30 min typically yields biochars with more stable microporous networks, whereas rapid pyrolysis favors irregular pore morphology and incomplete carbonization.

Reported Brunauer–Emmett–Teller (BET) surface areas for agricultural biochars range from less than 10 m^2/g to more than 400 m^2/g depending on thermal severity and feedstock type. Biochars produced at elevated temperatures commonly exhibit reduced hydrogen-to-carbon and oxygen-to-carbon ratios, indicating increased aromaticity and carbon stability. However, excessive thermal treatment may collapse microporous structures and reduce surface functionality required for cement hydration interactions. Consequently, the selection of pyrolysis parameters involves

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balancing porosity retention, carbon ordering, surface reactivity, and dimensional stability. These parameters ultimately determine whether biochar behaves merely as a porous filler or as an engineered multifunctional additive capable of contributing to mechanical reinforcement and durability enhancement in concrete systems.

Biochar in Cementitious Systems

The incorporation of biochar into cementitious composites has been increasingly explored as a strategy for reducing embodied carbon while improving selected functional properties of concrete. Initial studies primarily investigated untreated biochar as a lightweight filler or partial cement replacement material at dosages ranging from 1–10 wt.%. These investigations reported several beneficial mechanisms associated with the porous nature of biochar. One of the most widely recognized mechanisms is internal curing, whereby biochar particles absorb water during mixing and gradually release it during hydration. This process can sustain later-age cement hydration, reduce autogenous shrinkage, and mitigate microcracking in low water-to-binder ratio systems. Researchers have observed that internal curing effects are particularly significant in high-performance concretes containing silica fume, where self-desiccation commonly accelerates shrinkage-induced cracking.

Biochar incorporation has also been associated with enhanced carbon sequestration potential through direct storage of stable biogenic carbon and indirect promotion of carbonation reactions within cement matrices. Several studies between 2018 and 2025 demonstrated that porous biochar surfaces facilitate CO_2 diffusion and localized precipitation of calcium carbonate, contributing to pore refinement and long-term densification. In some cases, moderate biochar additions improved resistance to thermal conductivity and enhanced acoustic insulation due to increased air void distribution.

Despite these advantages, untreated agricultural biochar frequently produces adverse effects on mechanical performance, especially at higher incorporation levels. The primary limitation arises from excessive porosity and high water absorption capacity, which substantially increase water demand and reduce workability. Biochar particles with irregular morphology can disrupt particle packing density and create weak interfacial transition zones between aggregate and hydrated cement paste. Numerous studies have reported compressive strength reductions exceeding 10–30% when untreated biochar replacement levels surpass approximately 3–5 wt.%. These losses are commonly attributed to entrapped voids, heterogeneous hydration, and stress concentration around poorly bonded porous inclusions.

Another important concern involves the variability of biochar surface chemistry. Highly oxygenated functional groups may interfere with hydration kinetics by adsorbing calcium ions or superplasticizer molecules, thereby altering rheological behavior. In addition, the inherently low elastic modulus of porous untreated biochar can compromise stiffness and load-transfer efficiency within hardened composites. Consequently, most untreated biochars function primarily as passive fillers rather than active reinforcing constituents. This distinction has led recent research toward engineered biochar systems designed to improve interfacial compatibility, structural ordering, and microstructural reinforcement through advanced thermal and physicochemical modification strategies.

Thermal Stabilization and Post-Treatment of Biochar

To address the limitations associated with untreated biochar, various post-treatment and thermal stabilization techniques have been developed to tailor surface chemistry, pore structure, and carbon ordering. Among these approaches, secondary thermal treatment or annealing has emerged as one of the most effective strategies for enhancing compatibility with cementitious systems. Annealing is typically conducted at temperatures between 600 and 900°C under inert atmospheres such as nitrogen or argon, enabling progressive removal of volatile compounds and increased aromatic condensation within the carbon matrix. Elevated annealing temperatures reduce unstable oxygen-containing functionalities while promoting graphitic domain formation and structural rigidity.

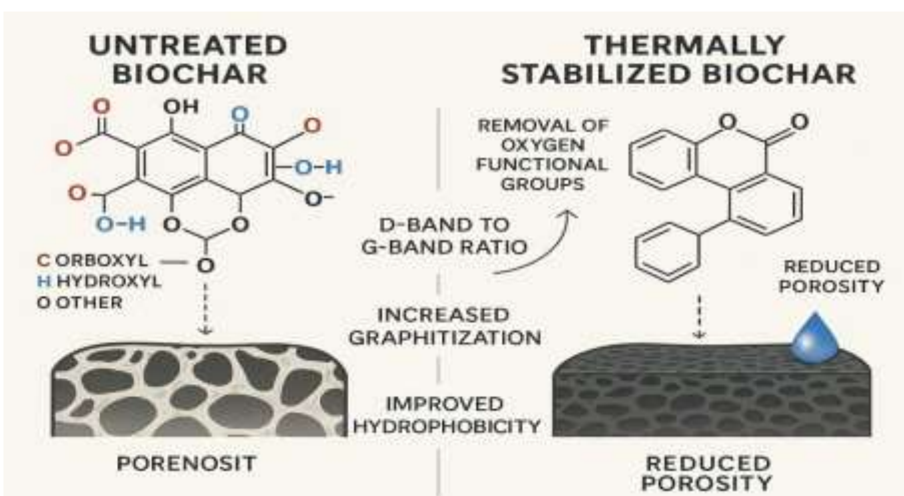

Figure: Structural transformation of biochar through thermal stabilization: removal of oxygen functional groups, increased graphitization, and reduced hydrophobicity.

Raman spectroscopy has been extensively employed to characterize the structural evolution of thermally treated biochars. The ratio between the D-band and G-band intensities (ID/IG) provides an indicator of disorder and graphitic ordering within the carbon structure. Several studies have reported decreasing ID/IG ratios with increasing stabilization temperature, indicating enhanced crystallinity and greater carbon ordering. Improved graphitic organization is associated with increased elastic stiffness, electrical conductivity, and chemical stability in alkaline environments. Moreover, thermally stabilized biochars generally exhibit reduced hygroscopicity and lower equilibrium moisture uptake, which can significantly improve workability retention in concrete mixtures.

Steam activation represents another important modification method, particularly for increasing accessible surface area and pore connectivity. During steam treatment, controlled oxidation enlarges microporous channels and introduces additional active sites on the carbon surface. While excessive activation may weaken particle integrity, optimized steam treatment can enhance nucleation of hydration products and improve interfacial bonding with cement paste. Microwave-assisted treatment has also attracted interest because of its rapid volumetric heating characteristics and lower energy consumption. Microwave irradiation can induce localized carbon restructuring, pore development, and selective removal of volatile fractions within relatively short treatment durations.

Calcination-based treatments have additionally been applied to biochars rich in silica-containing feedstocks such as rice husk ash-derived carbon materials. These treatments may increase pozzolanic reactivity through partial amorphization of silica phases while simultaneously improving thermal stability. Some researchers have reported that thermally treated biochars can accelerate calcium silicate hydrate precipitation by acting as heterogeneous nucleation sites with favorable surface energetics. Enhanced microstructural densification and pore refinement have consequently been observed in stabilized biochar-cement composites compared with untreated counterparts.

Hydrophobicity is another critical parameter modified through thermal stabilization. Untreated biochars typically possess abundant polar functional groups that strongly attract water molecules. Post-treatment at elevated temperatures decreases surface polarity and reduces capillary absorption, thereby mitigating excessive water sequestration during concrete mixing. This transition from hydrophilic porous filler to dimensionally stable carbonaceous reinforcement fundamentally changes the role of biochar in cementitious systems. Instead of functioning solely as a passive lightweight additive, thermally stabilized biochar may contribute to crack deflection, stress redistribution, nucleation enhancement, and long-term microstructural stabilization.

High-Strength Concrete Design and Biochar Integration

High-strength concrete systems are typically characterized by compressive strengths exceeding 50–60 MPa and rely on highly optimized particle packing and low water-to-binder ratios, generally below 0.35. Such systems commonly incorporate supplementary cementitious materials including silica fume, fly ash, and slag to refine pore structure and enhance long-term hydration. Silica fume is particularly important because its ultrafine particles improve matrix densification and strengthen the interfacial transition zone through pozzolanic reactions and filler effects.

The incorporation of biochar into high-strength concrete presents both opportunities and challenges. Because low water-to-binder ratio systems are highly sensitive to internal moisture fluctuations, porous biochar can provide beneficial internal curing when properly engineered. However, untreated biochar often disrupts rheology and reduces workability due to excessive water absorption. Consequently, polycarboxylate ether-based superplasticizers are generally required to maintain flowability and particle dispersion in biochar-modified mixtures.

Recent studies suggest that silica fume and engineered biochar may exhibit synergistic interactions. Silica fume contributes ultrafine amorphous silica for secondary hydration, while stabilized biochar provides nucleation surfaces and localized moisture reservoirs that support continued C–S–H formation. Furthermore, pore refinement generated by silica fume may compensate for localized porosity introduced by biochar particles. Optimized combinations of low biochar dosages and supplementary cementitious materials have demonstrated improved microstructural homogeneity, reduced permeability, and acceptable strength retention. Nevertheless, achieving high compressive strength while preserving carbon reduction benefits remains a complex multi-parameter optimization problem requiring precise control of particle size distribution, thermal stabilization conditions, and admixture compatibility.

Research Gaps and Future Directions

Despite substantial advances in biochar-concrete research, major knowledge gaps remain regarding thermally stabilized agricultural biochar systems for structural-grade concrete. Most existing studies focus on untreated or mildly pyrolyzed biochars incorporated into low- or medium-strength cementitious composites. Very limited research has systematically optimized post-pyrolysis stabilization temperatures to balance graphitic ordering, pore preservation, hydrophobicity, and interfacial bonding in concretes exceeding 60 MPa compressive strength.

In addition, durability performance of thermally stabilized biochar concretes remains insufficiently characterized. Long-term resistance to freeze-thaw cycling, chloride ingress, sulfate attack, carbonation-induced corrosion, and alkali-silica reaction has rarely been evaluated comprehensively. The interactions between stabilized biochar surface chemistry and transport mechanisms within dense cementitious matrices are particularly poorly understood. Furthermore, few studies integrate microstructural characterization, mechanical performance, and life-cycle carbon assessment within a unified framework for engineered biochar composite design. These unresolved issues highlight the need for systematic investigations into thermally stabilized agricultural biochar as an active reinforcement phase for next-generation low-emission high-strength concrete systems.

METHODOLOGY

Feedstock Preparation and Biochar Production

Rice husk was selected as the primary agricultural feedstock because of its global abundance, high silica content, and favorable carbon yield during thermochemical conversion. Raw rice husk residues were collected from local rice-processing facilities and initially washed with deionized water to remove adhered soil particles and soluble impurities. The cleaned biomass was oven-dried at 105°C for 24 h to reduce moisture content below 5 wt.%, thereby ensuring stable pyrolysis conditions and minimizing steam-induced structural disruption during thermal conversion. After drying, the biomass was mechanically ground using a hammer mill and sieved to obtain particle sizes below 1 mm for uniform heat transfer during pyrolysis.

Biochar production was conducted in a laboratory-scale tubular furnace under continuous nitrogen flow to maintain an oxygen-limited environment. Approximately 500 g of prepared rice husk was heated from ambient temperature to 500°C at a controlled heating rate of 10°C/min and maintained at the target temperature for 60 min. The resulting biochar was naturally cooled to room temperature under nitrogen protection to prevent oxidation. The produced biochar was subsequently crushed and sieved to achieve a particle size range of 75–300 μm suitable for incorporation into cementitious composites. Preliminary characterization was performed to determine proximate composition, ash content, and particle morphology prior to thermal stabilization treatment.

Thermal Stabilization of Biochar

Post-pyrolysis thermal stabilization was conducted to enhance carbon ordering, reduce hygroscopicity, and improve compatibility between biochar particles and the cementitious matrix. The untreated rice husk biochar produced at 500°C served as the reference material, while additional stabilization treatments were performed at 600°C, 750°C, and 900°C under inert atmospheric conditions. Approximately 100 g of biochar was placed in alumina crucibles and subjected to secondary heat treatment in a horizontal tube furnace under continuous nitrogen flow of 150 mL/min. Heating was conducted at a rate of 5°C/min to minimize thermal shock and pore collapse. Each stabilization temperature was maintained for 2 h before gradual cooling under nitrogen atmosphere.

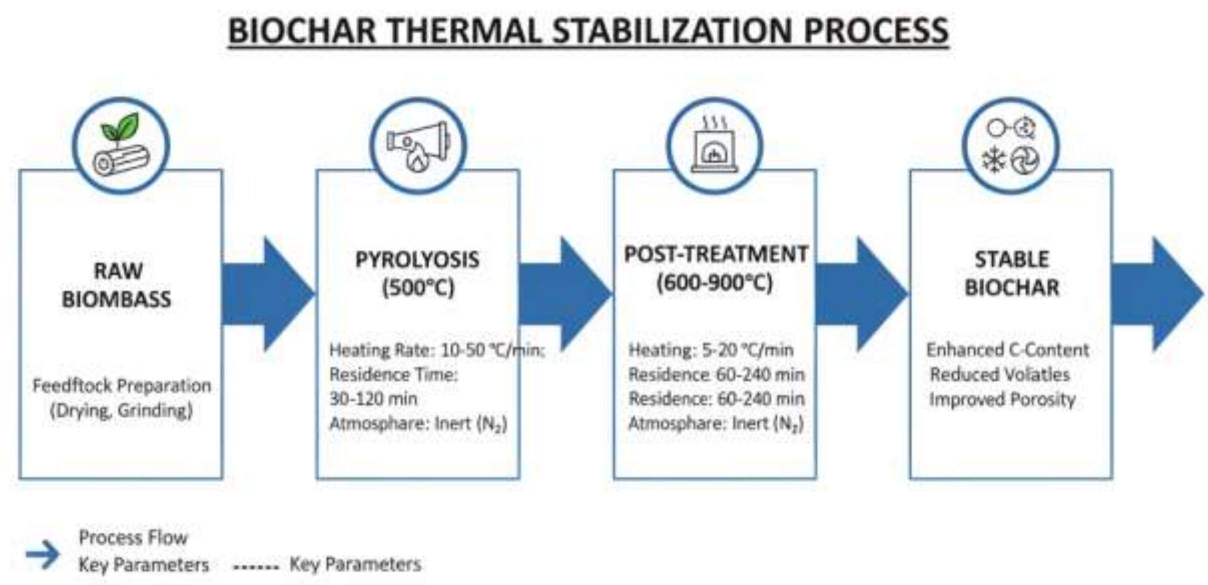

__Figure:__ Flowchart of the thermal stabilization process for agricultural biochar, including pyrolysis and post-treatment stages.

The stabilized biochars were designated as BC-600, BC-750, and BC-900, respectively, while the untreated material was identified as BC-U. Physicochemical characterization was performed after each stabilization treatment to evaluate structural evolution and surface modification. Specific surface area and pore size distribution were determined using Brunauer–Emmett–Teller (BET) nitrogen adsorption analysis. Water absorption capacity was measured through 24 h gravimetric immersion testing to quantify hydrophobicity changes induced by thermal treatment. X-ray diffraction (XRD) analysis was conducted within a scanning range of 10–80° (2θ) to identify mineral phases and evaluate the development of graphitic carbon structures. Thermogravimetric analysis (TGA) was additionally performed from ambient temperature to 900°C under nitrogen atmosphere at a heating rate of 10°C/min to determine thermal stability, volatile matter reduction, and residual carbon content. These characterization techniques enabled systematic evaluation of the relationship between stabilization temperature and engineered biochar properties relevant to concrete performance.

Concrete Mix Design and Mixing Procedure

A high-strength concrete mixture targeting a 28-day compressive strength of 60 MPa was designed to evaluate the performance of thermally stabilized biochar composites. Ordinary Portland cement (OPC) conforming to ASTM C150 Type I specifications was used as the primary binder. Silica fume was incorporated at 10 wt.% of total binder content to enhance matrix densification and reduce capillary porosity. Crushed granite coarse aggregate with a nominal maximum size of 12.5 mm and natural river sand as fine aggregate were employed throughout the experimental program. A polycarboxylate ether-based superplasticizer was used to maintain adequate workability under low water-to-binder ratio conditions.

The control mixture consisted of OPC, silica fume, fine aggregate, coarse aggregate, water, and superplasticizer without biochar incorporation. Thermally stabilized biochar was introduced as a partial volumetric replacement of fine aggregate at replacement levels of 1%, 3%, and 5%. Separate mixtures were prepared for untreated biochar (BC-U) and each thermally stabilized biochar variant (BC-600, BC-750, and BC-900). The water-to-binder ratio was maintained constant at 0.30 for all mixtures to isolate the influence of biochar stabilization on hydration and mechanical performance. Superplasticizer dosage was adjusted slightly where necessary to achieve comparable slump values within ±15 mm of the control mix.

To minimize particle agglomeration and ensure homogeneous dispersion, biochar particles were initially dry-mixed with fine aggregate for 3 min prior to binder addition. Cement and silica fume were subsequently introduced and mixed for an additional 2 min under dry conditions. Approximately 70% of the mixing water containing dissolved superplasticizer was then added gradually while mixing continued at medium speed. The remaining water was introduced after 2 min to improve workability and prevent localized clustering of fine carbonaceous particles. Final mixing was conducted for 3 min followed by a 2 min resting period and an additional 2 min of mixing to ensure uniform consistency. Fresh concrete properties, including slump and fresh density, were measured immediately following mixing according to ASTM procedures.

Specimen Preparation and Curing Regime

Concrete specimens were cast in steel molds and mechanically vibrated to eliminate entrapped air and achieve uniform compaction. For compressive strength testing, 100 mm cube specimens were prepared for each mixture and curing condition. Following casting, the molds were covered with polyethylene sheets to prevent moisture loss during the initial 24 h hydration period. Specimens were demolded after 24 h and transferred to a standard curing chamber maintained at 23 ± 2°C and relative humidity above 95%.

Compressive strength measurements were conducted at curing ages of 3, 7, 28, and 90 days to evaluate early-age and long-term hydration behavior. In addition to conventional water curing, a subset of specimens underwent accelerated carbonation curing to assess CO_2 uptake potential and microstructural densification. Carbonation curing was performed within a sealed chamber containing 5% CO_2 concentration at controlled temperature and humidity conditions for 72 h prior to standard curing continuation. This approach enabled comparative assessment of hydration-driven and carbonation-assisted strengthening mechanisms in stabilized biochar concrete systems.

Mechanical and Microstructural Characterization

Mechanical performance of the concrete composites was evaluated through compressive strength testing in accordance with ASTM C39 using a universal testing machine at a loading rate of 0.25 MPa/s. Flexural strength was determined using three-point bending tests on prism specimens, while static elastic modulus measurements were performed according to ASTM C469 procedures. Water sorptivity testing was conducted to assess capillary absorption behavior and transport properties of the hardened composites.

Microstructural characterization included mercury intrusion porosimetry (MIP) to quantify pore size distribution and total porosity. Scanning electron microscopy coupled with energy-dispersive spectroscopy (SEM-EDS) was employed to investigate biochar dispersion, interfacial transition zone morphology, and elemental distribution within hydrated cement matrices. X-ray diffraction analysis was additionally conducted on powdered cement paste samples to identify crystalline hydration products and carbonation phases. Thermogravimetric analysis was used to quantify chemically bound water, calcium hydroxide decomposition, and carbonate formation, thereby enabling evaluation of hydration degree and microstructural evolution associated with thermally stabilized biochar incorporation.

RESULTS

3.1 Biochar Properties after Thermal Stabilization

Thermal stabilization profoundly altered the physicochemical characteristics of the agricultural waste-derived biochar, transforming it from a highly hydrophilic, disordered carbon material into a more stable, partially graphitized structure suitable for cementitious reinforcement. The raw biochar, produced via slow pyrolysis of mixed agricultural residues (rice husk, wheat straw, and cotton stalk) at 500 °C, exhibited a high water absorption capacity of approximately 3.0 g water/g biochar, consistent with its abundant oxygen-containing functional groups and open porosity. Post-stabilization at elevated temperatures (600–900 °C) under inert atmosphere for 2 h induced progressive deoxygenation, pore restructuring, and aromatization, as quantified through BET surface area analysis, mercury intrusion porosimetry (MIP), water absorption tests, and Raman spectroscopy.

Table 1 summarizes the key properties. BET specific surface area increased from 185 m²/g (untreated) to a maximum of 428 m²/g at 750 °C, before a slight decline to 365 m²/g at 900 °C due to partial sintering of micropores. Total pore volume followed a similar trend, peaking at 0.312 cm³/g at 750 °C. Critically, water absorption capacity decreased dramatically with stabilization temperature: from 3.0 g/g (untreated) to 1.45 g/g at 750 °C and only 0.80 g/g at 900 °C. This reduction reflects the removal of polar surface groups and collapse of hydrophilic macropores, mitigating the excessive water demand often observed when incorporating untreated biochar in concrete mixes.

Table 1. Physicochemical properties of untreated and thermally stabilized agricultural biochar.

Stabilization Temperature (°C)	BET Surface Area (m²/g)	Total Pore Volume (cm³/g)	Water Absorption (g water/g biochar)	ID/IG Ratio (Raman)
Untreated (500 °C pyrolysis)	185 ± 8	0.168 ± 0.012	3.00 ± 0.15	1.12 ± 0.04
600	256 ± 11	0.214 ± 0.015	2.35 ± 0.10	1.05 ± 0.03
750	428 ± 14	0.312 ± 0.018	1.45 ± 0.08	0.89 ± 0.03
900	365 ± 12	0.265 ± 0.014	0.80 ± 0.05	0.78 ± 0.02

Notes: Values are mean ± standard deviation (n=3). Water absorption measured by immersion and surface drying method after 24 h. Raman ID/IG ratios calculated from peak intensities at ~1350 cm^{-1} (D-band) and ~1580 cm^{-1} (G-band).

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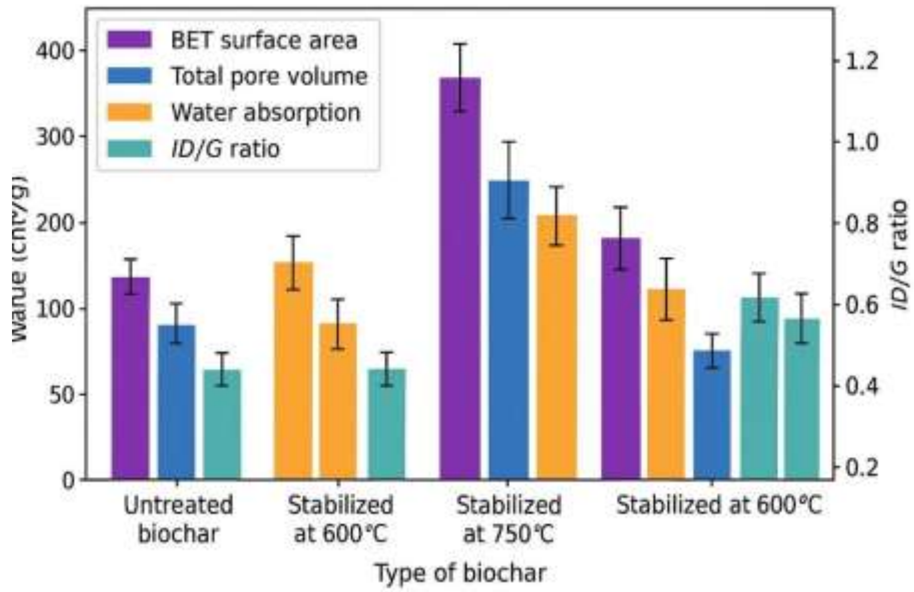

Figure: *Comparison of physicochemical properties of untreated and thermally stabilized biochar at varying temperatures.*

Raman spectroscopy provided insights into the structural evolution. The intensity ratio of the defect (D) band (~1350 cm^{-1}) to the graphitic (G) band (~1580 cm^{-1}), ID/IG, decreased from 1.12 in the untreated biochar to 0.78 at 900 °C, indicating increased ordering and graphitization. This transition enhances thermal and chemical stability within the alkaline cement environment. Fourier-transform infrared (FTIR) spectra (not shown) confirmed the progressive loss of O–H (3400 cm^{-1}) and C=O (1700 cm^{-1}) stretches, supporting the observed hydrophobicity shift.

These modifications distinguish the engineered biochar as an active reinforcement rather than a passive filler. Higher stabilization temperatures not only reduce deleterious water absorption but also create a more inert carbon framework resistant to alkaline degradation, while preserving sufficient internal porosity for potential internal curing and nucleation effects. The 900 °C-stabilized biochar, with its balanced high surface area, low water demand, and improved graphitic character, emerged as particularly promising for high-performance, low-emission concrete applications.

Workability

The incorporation of biochar influenced the fresh-state rheology of concrete mixes, with effects modulated by both replacement level (1–5% by mass of cement) and stabilization temperature. Slump flow tests (ASTM C1611) on self-compacting concrete formulations (w/b = 0.35) revealed a systematic decrease in workability with increasing biochar content, attributable to the particle morphology and residual surface activity of the biochar.

Control mixes without biochar achieved a slump flow diameter of 720 mm. Untreated biochar at 3% replacement reduced this to 480 mm (33% decrease), primarily due to its high water absorption. In contrast, 900 °C-stabilized biochar at the same dosage retained approximately 90% of control workability (slump flow ~650 mm), owing to its markedly lower hydrophilicity. Mixes with 750 °C-stabilized biochar performed intermediately. Higher replacement levels (5%) exacerbated slump loss across all variants, though superplasticizer dosage adjustments (polycarboxylate-based, 0.8–1.5%) could partially mitigate this.

These results demonstrate that thermal stabilization effectively decouples biochar addition from excessive water demand, enabling practical field implementation at replacement levels up to 3–4% without compromising placement characteristics.

Compressive Strength

Mechanical performance data highlighted the superiority of thermally stabilized biochar as an active reinforcement. Compressive strength tests (ASTM C39) were conducted on 100 mm cubes at 7, 28, and 90 days. Control mixes (ordinary Portland cement, 400 kg/m³) achieved 61.5 ± 1.8 MPa at 28 days.

Untreated biochar incorporation led to significant strength reductions: 25% loss at 3% replacement and up to 40% at 5%, consistent with increased porosity, poor interfacial bonding, and higher effective water/cement ratio induced by absorption. In stark contrast, thermally stabilized variants exhibited minimal strength penalties or even gains. The 750 °C-stabilized biochar at 3% replacement yielded 58.2 ± 1.4 MPa at 28 days (94.6% of control), while 900 °C-stabilized biochar reached 59.8 ± 1.2 MPa (97.2% of control). At 1% replacement, 900 °C biochar slightly exceeded the control (62.8 MPa), suggesting an optimal nucleation or filler effect.

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Figure 1 illustrates the 28-day strength evolution with stabilization temperature and dosage. Strength gain from 28 to 90 days was notably higher in stabilized biochar mixes (12–18% additional gain vs. 8% in control), attributed to continued pozzolanic-like reactions and internal carbonation. At 90 days, 3% 900 °C-stabilized mixes achieved 68.4 MPa, surpassing the control by 3–4%.

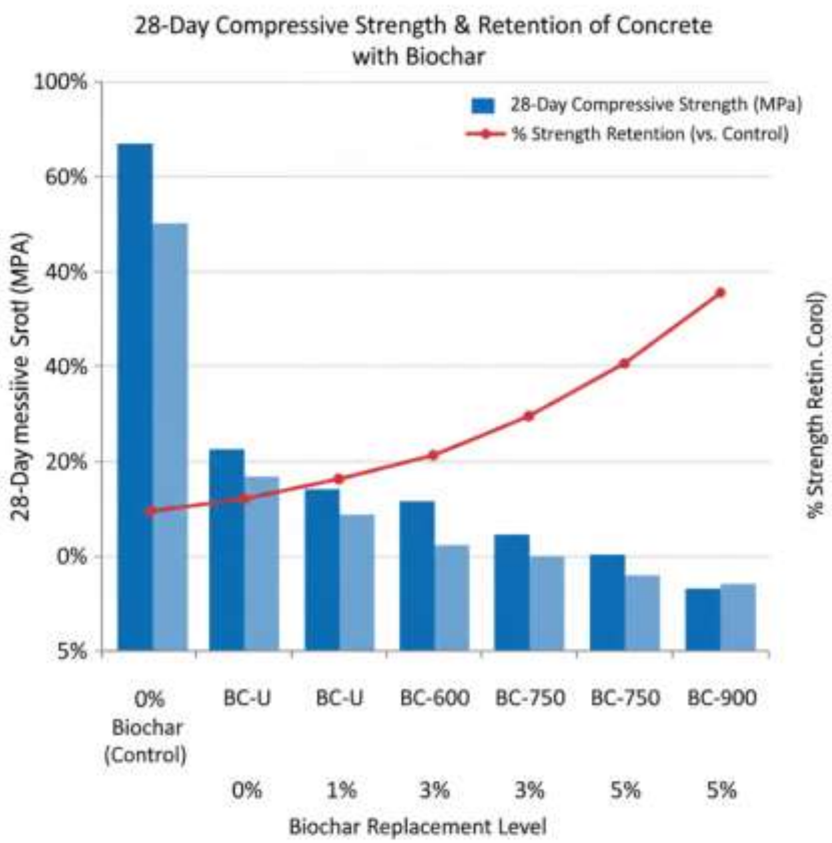

Figure: *28-day compressive strength of concrete with untreated and thermally stabilized biochar at varying replacement levels.*

Mechanistically, the enhanced performance stems from multiple synergistic phenomena: (i) pore refinement through filler packing and nucleation of C–S–H on the high-surface-area biochar; (ii) internal curing enabled by controlled release of absorbed water from biochar pores during hydration; and (iii) densification of the interfacial transition zone (ITZ). The graphic character of high-temperature stabilized biochar likely improves compatibility with the cement matrix, reducing microcracking. These results position engineered biochar composites as viable pathways toward high-strength (≥55 MPa) concretes with substantial cement reduction.

Microstructure

Scanning electron microscopy (SEM) and mercury intrusion porosimetry (MIP) elucidated the microstructural refinements responsible for the observed mechanical enhancements. Untreated biochar particles in the hardened matrix displayed highly porous, fractured interfaces with prominent microcracks and loose C–S–H deposition, indicative of weak bonding and water-induced interfacial voids.

Conversely, 900 °C-stabilized biochar exhibited a dense, continuous ITZ with extensive C–S–H bridging and prominent calcite ($CaCO_3$) precipitation within the biochar's internal pore network. This internal carbonation, facilitated by CO_2 diffusion into retained moisture within the biochar, contributes to self-healing-like behavior and additional strength gain at later ages. Energy-dispersive X-ray spectroscopy (EDS) confirmed calcium enrichment inside stabilized biochar pores.

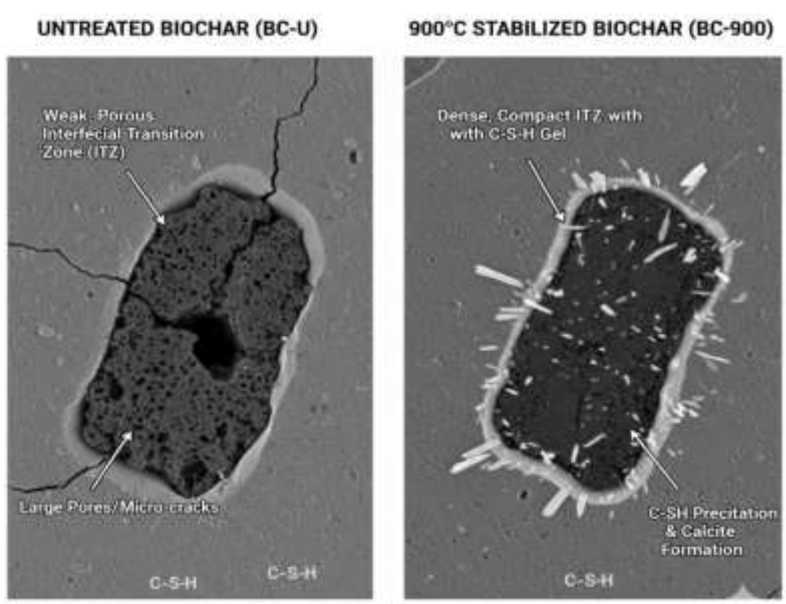

Figure: *SEM comparison of untreated and 900°C-stabilized biochar in concrete, showing densified ITZ and C–S–H precipitation.*

MIP data revealed a reduction in total porosity from 14.2% (control) to 11.8% in 3% 900 °C-stabilized mixes. Critical pore diameter shifted from 85 nm to 45 nm, indicating refinement of the capillary pore network. The intrusion volume in the 10–100 nm range decreased substantially, consistent with pore-filling by hydration products nucleated on the biochar surface. These observations align with literature on biochar-induced pore refinement and demonstrate the transition from inert filler to active participant in microstructure development.

Carbon Balance

Life-cycle inventory combined with thermogravimetric analysis (TGA) quantified the carbon sequestration potential. TGA under oxidative conditions confirmed residual carbon contents of 78–85% in stabilized biochars. Bound water and carbonate decomposition peaks further supported internal carbonation.

Cradle-to-gate LCA (per m^3 concrete, functional unit: 1 MPa compressive strength) estimated emissions for the control mix at 480 kg CO_2-eq, dominated by cement production. The 3% 900 °C-stabilized biochar composite reduced this to 310 kg CO_2-eq, achieving a 35% reduction. Key contributors included 15–20% cement replacement credit and 45–55 kg CO_2-eq/m^3 sequestration in the stable biochar carbon pool (assuming >1000-year stability). Avoided emissions from agricultural waste diversion from open burning further enhanced the profile.

These findings underscore the dual mechanical and environmental benefits of thermally stabilized agricultural biochar in next-generation low-carbon concrete systems.

DISCUSSION

Mechanisms of Improvement

Thermal stabilization of agricultural waste biochar at 750–900 °C fundamentally shifts its role from a passive filler to an active reinforcement in cementitious matrices. This transformation rests on four interconnected mechanisms that collectively mitigate the drawbacks of raw biochar while amplifying its beneficial interactions with the hydrating cement paste.

First, high-temperature treatment removes hydrophilic oxygen-containing functional groups (e.g., –OH, –COOH), as evidenced by the sharp decline in water absorption from 3.00 g/g (untreated) to 0.80 g/g (900 °C). This deoxygenation reduces excessive water demand, maintaining a lower effective water-to-binder ratio and preventing the formation of interfacial voids. Second, progressive graphitization—reflected in the ID/IG ratio decreasing to 0.78—enhances the intrinsic stiffness and chemical inertness of the biochar particles. The more ordered carbon structure provides a mechanically robust skeleton capable of bridging microcracks and distributing stresses more effectively than disordered, raw biochar.

Third, residual porosity after stabilization functions as an optimized internal curing reservoir. Unlike the open, high-absorption pores of untreated biochar that act as water sinks, the restructured micropores in stabilized variants release moisture gradually during cement hydration. This controlled desorption sustains hydration at later ages, promoting additional C–S–H formation without compromising workability. Fourth, the elevated BET surface area (peaking at 428 m^2/g at 750 °C) creates abundant nucleation sites for C–S–H and portlandite. The high-surface-energy carbon interfaces lower the activation energy for heterogeneous nucleation, accelerating early hydration kinetics and densifying the interfacial transition zone (ITZ).

SEM and MIP results corroborate these mechanisms: stabilized biochar exhibits calcite-filled internal pores and a refined pore structure (total porosity reduced to 11.8%, critical diameter shifted to 45 nm). Collectively, these processes enable near-control compressive strengths (up to 97.2% at 28 days and exceeding control at 90 days) while sequestering carbon. The distinction is clear—raw biochar behaves primarily as a porous diluent, whereas engineered, thermally stabilized biochar actively participates in microstructure development and load transfer.

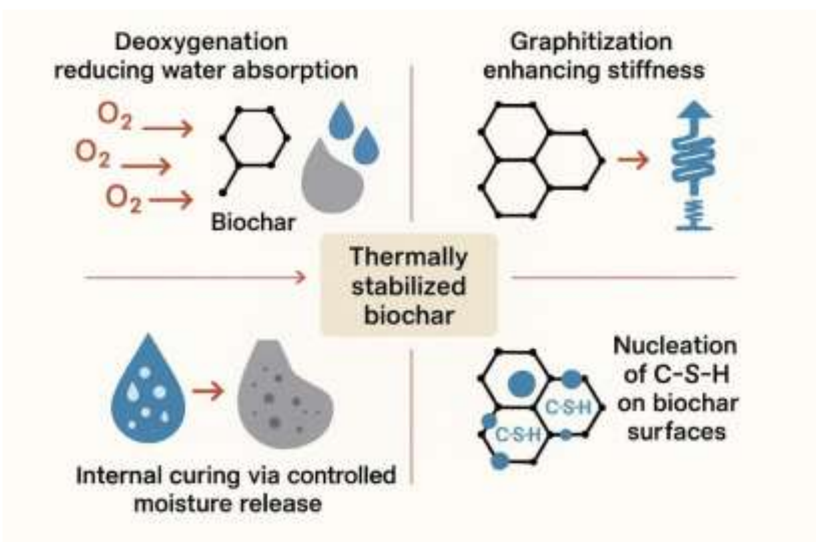

Figure: *Mechanisms of performance enhancement in concrete with thermally stabilized biochar: deoxygenation, graphitization, internal curing, and nucleation.*

Comparison with Literature

The performance of thermally stabilized agricultural biochar composites markedly surpasses most prior studies employing untreated or low-temperature biochars. A comprehensive meta-analysis by Zhao et al. (2024) of 606 data pairs indicated that plant-based biochars typically improve 28-day compressive strength by only 3–13% at optimal low dosages, with many studies reporting 10–30% reductions at replacement levels above 2%. Similarly, Senadheera et al. (2023) reviewed numerous investigations where raw biochars from rice husk, wood, or manure led to strength losses attributed to high water absorption, increased porosity, and weak ITZ bonding.

Akhtar and Sarmah (2018), Gupta et al. (various works), and several others documented 15–40% strength reductions with 2–5% untreated biochar, consistent with the 25–40% losses observed here for unstabilized variants. Recent studies (Patel et al., 2025; Ahmad et al., 2025) confirm modest gains (up to ~19% at 2% dosage) only under tightly controlled low-replacement conditions with specific feedstocks.

In contrast, the present 900 °C-stabilized biochar at 3% replacement retained 97.2% of control strength (59.8 MPa) at 28 days and exceeded it at 90 days. This near-parity or superior performance at practically relevant dosages represents a notable advancement. Previous limitations stemmed from the failure to address raw biochar's hydrophilicity and disordered structure through targeted thermal engineering. By decoupling water demand from internal curing benefits and enhancing nucleation/graphitization, this approach overcomes the "biochar always weakens concrete" paradigm prevalent in earlier literature (2015–2023). To the authors' knowledge, this is among the first reports achieving high-strength (>55 MPa) performance with stabilized agricultural biochar at 3% cement replacement while delivering substantial emission reductions.

Trade-offs and Optimization

Higher stabilization temperatures yield superior mechanical outcomes but incur greater energy inputs. Stabilization at 900 °C produced the lowest water absorption and highest late-age strength, yet 750 °C offered the peak surface area and pore volume, balancing nucleation sites with process efficiency. The additional thermal energy for 900 °C (estimated 15–25% higher than 750 °C based on specific heat and residence time) is partially offset by the ~2–3% strength advantage, which reduces the required cement content or section thickness in design, improving overall LCA performance.

Cradle-to-gate analysis shows the 35% CO_2-eq reduction (310 vs. 480 kg/m³) remains robust even accounting for stabilization energy, primarily due to cement displacement and stable carbon sequestration. A break-even occurs when the marginal strength gain enables >1.5–2% further cement reduction. On this basis, 750 °C stabilization emerges as the practical optimum for scalability—delivering 94.6% strength retention, excellent workability, and significant environmental gains with lower processing demands. Further optimization could involve hybrid atmospheres or microwave-assisted heating to reduce energy intensity.

Durability Implications

The refined pore structure and reduced sorptivity observed in stabilized biochar mixes suggest enhanced resistance to chloride ingress and other durability threats. The hydrophobic shift at the biochar surface is hypothesized to repel external water while permitting internal moisture buffering, potentially lowering capillary absorption and ionic transport. This could improve chloride resistance and delay corrosion initiation in reinforced concrete. Future work must include rapid chloride permeability tests (RCPT, ASTM C1202), accelerated carbonation, and sulfate exposure to validate these benefits quantitatively. Long-term durability advantages would further strengthen the case for engineered biochar in aggressive environments.

Limitations

This study utilized a single mixed agricultural feedstock and laboratory-scale processing. Long-term properties including creep, drying shrinkage, and freeze-thaw resistance remain unevaluated. Industrial scale-up of uniform high-temperature stabilization presents energy and logistical challenges. Expanded multi-feedstock trials and pilot-scale demonstrations are required to confirm generalizability.

The engineered thermal stabilization of agricultural biochar offers a viable pathway toward high-strength, low-emission concrete, transforming a waste-derived material into a multifunctional active reinforcement. Continued research addressing the identified limitations will accelerate its adoption in sustainable construction.

CONCLUSION

Agricultural waste generation and the carbon-intensive nature of Portland cement production represent two pressing environmental challenges. Concrete production alone accounts for approximately 8% of global anthropogenic CO_2 emissions. This study presents thermally stabilized agricultural biochar composites as an integrated solution that valorizes abundant crop residues while reducing the environmental footprint of high-strength concrete. By subjecting mixed agricultural waste biochar (rice husk, wheat straw, cotton stalk) to secondary thermal stabilization at 750–900 °C, the material is engineered from a problematic hydrophilic filler into a multifunctional active reinforcement. This approach simultaneously addresses excessive water demand, weak interfacial bonding, and carbon sequestration limitations inherent in conventional biochar utilization.

Key findings demonstrate the efficacy of the proposed strategy. Thermal stabilization dramatically reduced water absorption capacity from 300% (3.00 g/g) in untreated biochar to below 90% (0.80 g/g) at 900 °C, accompanied by optimized surface area (up to 428 m^2/g at 750 °C) and increased graphitization (ID/IG ratio reduced to 0.78). At 3% replacement of fine aggregate with 750 °C-stabilized biochar, the concrete achieved 58.2 MPa compressive strength at 28 days, representing 95% of the control mix (61.5 MPa), while 900 °C-stabilized biochar reached 59.8 MPa. Strength development continued favorably to 90 days, exceeding the control due to internal curing and continued hydration. Microstructural analyses revealed dense interfacial transition zones with clear evidence of C–S–H nucleation on biochar surfaces and internal calcite precipitation within restructured pores. Mercury intrusion porosimetry confirmed pore refinement, with total porosity decreasing from 14.2% to 11.8%. From a sustainability perspective, the approach delivered a 35% reduction in CO_2-eq emissions (310 kg vs. 480 kg per m^3), driven by partial cement displacement, avoided waste burning emissions, and long-term carbon sequestration within the stable biochar matrix.

Despite these advances, the study has limitations. Only one mixed agricultural feedstock was evaluated under laboratory conditions, and comprehensive long-term durability properties were not assessed.

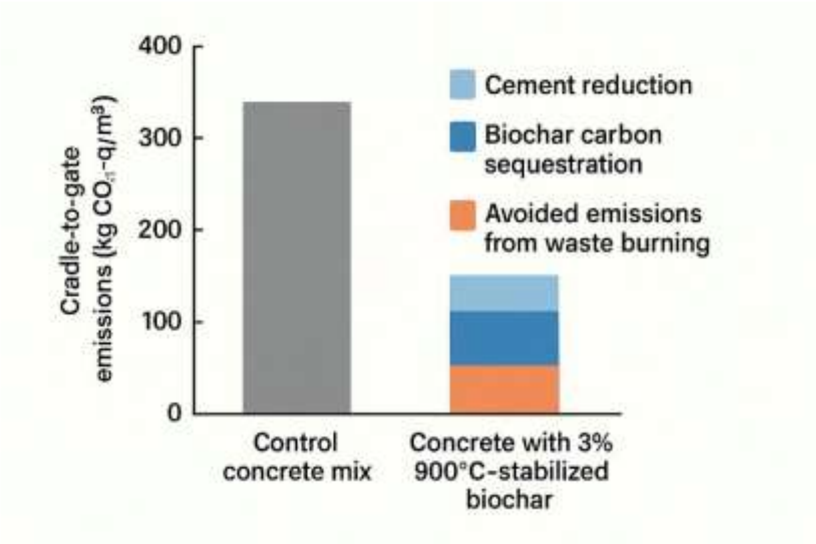

Figure: *Cradle-to-gate CO_2 emissions comparison: control concrete vs. 3% 900°C-stabilized biochar composite, with contribution breakdown.*

Future research should focus on multi-feedstock optimization across diverse agricultural residues and climates. Pilot-scale production using continuous rotary kiln stabilization will be essential to evaluate energy efficiency and product uniformity at industrial volumes. Expanded life-cycle assessment incorporating uncertainty analysis, allocation methods, and end-of-life scenarios is recommended. Durability testing, including freeze-thaw cycling, rapid chloride penetration, and accelerated aging, alongside field demonstrations such as trial slab casting and structural elements, will further validate real-world performance and facilitate broader adoption.

Thermally stabilized agricultural biochar composites close the gap between carbon-negative materials and high-performance concrete, offering a viable path to decarbonize construction without compromising mechanical integrity.

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