



Enhanced Mechanical and Microstructural Properties of Eco-friendly Cementitious Composites Reinforced with Treated Natural Fibers and Nano-graphene Oxide

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ABSTRACT

To improve mechanical and microstructural performance, this study develops a sustainable nano-engineered cementitious composite reinforced with alkali-treated natural fibers and nano-graphene oxide (nano-GO). Bagasse pulp and recycled coir fibers were used as environmentally friendly reinforcements, while nano-GO served as the nano-modifier to promote matrix hydration, densification, and fracture resistance. Single-fiber tensile testing and thermogravimetric analysis confirmed that alkali (NaOH) surface modification of coir fibers at varying concentrations significantly enhanced tensile strength, Young's modulus, thermal stability, and fiber–matrix interfacial adhesion. A laboratory-scale Hatschek technique was employed to fabricate composite boards with treated fiber concentrations ranging from 5 to 15 wt.%. According to three-point bending test results, treated fiber-nano-GO composites exhibited significantly higher flexural strength and ductility than untreated systems. With a flexural strength of 11.82 MPa, the optimal formulation with 5 wt.% treated coir fiber, 19 wt.% bagasse pulp, and 0.5 wt.% nano-GO exceeded conventional fiber-cement performance standards. Microstructural investigation confirmed improved load transfer, enhanced fiber–matrix bonding, and reduced microcracking due to synergistic nano-reinforcement effects. These results demonstrate a resource-efficient and sustainable approach for producing high-performance cementitious materials enabled by nanotechnology, appropriate for durable and cost-effective construction in developing countries.

Keywords: Nano-graphene oxide; Alkali-treated natural fibers; Cementitious nanocomposites; Mechanical and microstructural performance; Sustainable construction materials.

1. INTRODUCTION

By modifying the microstructure of cementitious composites at the nanoscale level, nano-engineering has become an effective approach for improving their performance. Improved matrix densification and crack-bridging behavior are made possible by the remarkable mechanical strength, large surface area, and extensive chemical functionality of nano-graphene oxide (nano-GO), one of the nano-modifiers. When added to cement systems, nano-GO improves pore structure, speeds up hydration processes, and enhances load transmission. In this work, eco-friendly cementitious composites are reinforced with alkali-treated natural fibers using nano-GO as the only nano-modifier. Flexural performance is improved, microcracking is decreased, and fiber–matrix interfacial

bonding is greatly strengthened by the synergistic interaction of treated fibers and nano-GO. This nano-engineered method promotes sustainability in the production of cement-based materials while simultaneously improving mechanical and microstructural qualities through the use of renewable fiber reinforcements. In areas with limited resources, these composites provide a viable option for long-lasting, reasonably priced construction. Through nanoscale alterations, nano-engineering is essential to enhancing the microstructure and functionality of cement-based materials. According to (Cho *et al.* 2020), cement composites can have improved mechanical performance, durability, and toughness by employing nano-engineering techniques to modify calcium-silicate-hydrate (C-S-H) structures at the molecular level. In a similar vein, (Golewski, 2022) showed that the

combination of coal fly ash and nanosilica improved the eco-friendly concrete strength characteristics, increased matrix densification, and refined the pore structure.

Graphene oxide (GO) is a well-known nano-modifier for cementitious systems because of its great surface area, high strength, and chemical reactivity. (Huiting *et al.* 2018) studied the combined effect of nanosilica and GO on oil well cement and reported significant improvements in compressive strength and hydration due to synergistic nano-scale interactions. (Mao *et al.* 2021) further developed hybrid nano-silica/GO composites with superhydrophobic and flame-retardant properties, highlighting multifunctional applications beyond structural reinforcement. (Zhang *et al.* 2020) demonstrated that eco-friendly engineered cementitious composites could be made using materials derived from agricultural waste, such as rice husk ash, in combination with high-performance nano-additives.

(Azevedo *et al.* 2021) compared natural and synthetic fibers in geopolymer matrices and found that natural fibers are competitive in strength and beneficial in sustainability. (Bui *et al.* 2020) reviewed the mechanical properties of raw and treated coconut fibers and concluded that alkali treatment enhances tensile strength, stiffness, and fiber–matrix adhesion. (Snoeck *et al.* 2015) also reported improved crack control and autogenous healing in cementitious materials reinforced with chemically treated natural fibers for cement composites.

The combination of GO and natural fibers has demonstrated promise in creating hybrid composites with superior mechanical and functional performance: (Das *et al.* 2020) synthesized hybrid hydrogel nano-polymer composites incorporating GO for wastewater treatment, demonstrating that nano-hybrid structures significantly enhance durability and stability in wet environments—attributes desirable in fiber cement boards. (Sumrith *et al.* 2020) further confirmed that alkali and silane treatments improve fiber compatibility with matrices, indicating that combining treated fibers with GO could maximize bonding and crack resistance. (Tara *et al.* 2020) demonstrated how nano-engineering approaches enable tailored adsorbents with enhanced surface properties, which can be translated into improved cementitious composite performance.

Incorporating multiple functional properties into cementitious composites is a growing area of research. For example, (Selim *et al.* 2018) created environmentally friendly nano-magnetite/silicone composites for marine coatings, showing that hybrid nano-materials can accomplish both mechanical and protective functions. (Sabeeh *et al.* 2020) and (Aragaw 2019) investigated nano-hybrids for supercapacitors and photocatalysis, respectively, suggesting potential cross-

disciplinary applications for structural materials. These findings are consistent with the trend toward sustainable, multifunctional building materials that are appropriate for affordable housing solutions.

Despite the significant advancements in nano-modified cementitious composites, most research focuses on individual nano-additives without combining them with renewable natural fibers for synergistic performance; present-day research frequently uses nanosilica or carbon-based fillers, but their resource requirements and environmental impact are still concerns; little research has been done on nano-GO as a single nano-modifier in eco-friendly fiber cement systems, especially when combined with alkali-matrix bonding in a sustainable cement matrix; the challenge of achieving both mechanical enhancement and microstructural densification under eco-conscious material selection. This study addresses these gaps by coupling nano-engineering at the matrix level with treated natural fibers to improve strength, durability, and crack resistance. The approach offers a novel pathway for producing high-performance, environmentally responsible cementitious composites suitable for low-cost and sustainable construction.

2. MATERIALS AND METHODS

2.1 Cement

Ordinary Portland Cement (OPC, 53 grade), conforming to IS 12269:2013 specifications, was used as the primary binder. The cement was sourced from a single batch to ensure uniformity in chemical composition and fineness. X-ray fluorescence (XRF) analysis confirmed the oxide composition, with CaO as the dominant phase and a specific surface area of 320 m²/kg, as determined by Blaine's method.

2.2 Natural Fibers

Coir fibers (*Cocos nucifera*), procured locally, were cut into 15 mm lengths and selected for their high lignin content and tensile resilience. Bagasse pulp, obtained from sugarcane industry waste, serves as a secondary reinforcement and matrix filler. Both fibers were sun-dried to reduce initial moisture content below 8% before treatment.

2.2.1 Nano-graphene Oxide

Graphene oxide nanoparticles were synthesized via a modified Hummers' method and purchased from a certified supplier to maintain batch consistency. The GO nanosheets had an average lateral size of 570 nm and a thickness of 2 nm. Brunauer–Emmett–Teller (BET) analysis revealed a specific surface area of 430 m²/g. The oxygen-containing functional groups (hydroxyl, epoxy, and carboxyl) were confirmed via Fourier transform

infrared spectroscopy (FTIR), enabling strong chemical interactions with cement hydration products.

2.3 Fiber Treatment Process

2.3.1 Alkali Treatment of Coir Fibers

To improve fiber–matrix adhesion, coir fibers underwent alkali treatment using sodium hydroxide (NaOH) solutions of 5%, 10%, and 15% concentrations by weight. Fibers were immersed in the alkali solution at 30 °C for 4 h, washed thoroughly with distilled water until a neutral pH was achieved, and oven-dried at 60 °C for 24 h. This process removed hemicellulose, pectin, and surface impurities, as confirmed by scanning electron microscopy (SEM) surface morphology analysis based on ASTM D3822.

2.3.2 Bagasse Pulp Preparation

Bagasse was mechanically pulped, followed by hot water washing to remove residual sugars and non-cellulosic compounds. The pulp was filtered and dried at 50 °C, resulting in improved bonding potential due to increased surface roughness.

2.3.3 Nano-graphene Oxide Dispersion

A two-step dispersion method was used to ensure nano-level reinforcement and prevent agglomeration: Nano-GO (0.5 wt.%) was ultrasonically dispersed in distilled water (20 kHz, 750 W) for 30 minutes with pulsing to prevent overheating, and then 0.5% polycarboxylate ether (PCE)-based superplasticizer (by cement weight) was added to improve particle separation and workability. More than 90% of the nano-GO particles remained below 800 nm in hydrodynamic size after dispersion, according to the results of the Dynamic light scattering (DLS) technique.

2.4 Mix Design and Fabrication

2.4.1 Composition

The composite mix was designed based on the following proportions (by weight of total solids):

- OPC: 79%
- Bagasse pulp: 16%
- Treated coir fiber: 12%
- Nano-GO: 0.36%
- Water-to-cement ratio (w/c) was maintained at 0.38, depending on fiber content, adjusted for workability using the superplasticizer. Figure 1 shows the concrete samples.

2.4.2 Hatschek Process Simulation

The fabrication was carried out using a Hatschek process simulation in the lab, which included

pressing, sheet creation, fiber dispersion, and slurry preparation: cement, bagasse pulp, and nano-GO dispersion were mixed for 10 minutes at 1500 rpm using a lab-scale Hatschek technique. Coir fiber was then added gradually. The 300 × 150 × 6 mm mix was molded, vibrated for 2 min, pressed for 5 min at 1 MPa, demolded after 24 h, and cured for 28 days at 27 °C in water soaked with lime.

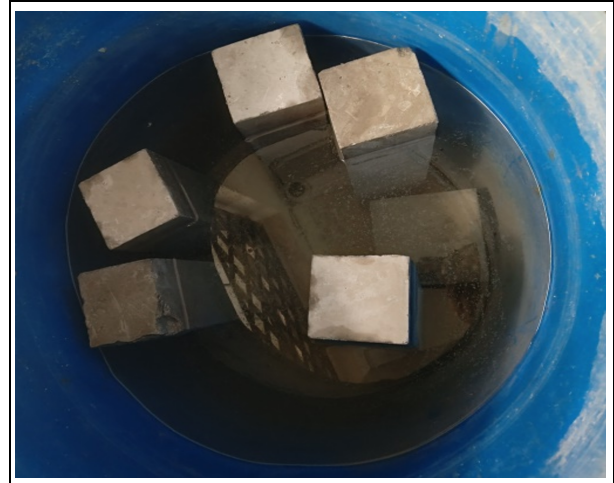


Fig. 1: Concrete samples

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2.5 Mechanical Testing

Tensile testing of single fibers was carried out using a micro-tensile tester to quantify the effect of alkali treatment. Three-point bending tests (ASTM C1185) were used to determine flexural strength with a span length of 200 mm and a loading rate of 2 mm/min.

2.5.1 Tensile Strength Test

The specimens were gripped at both ends and stretched at a constant rate until failure, with strength calculated from the maximum load divided by the cross-sectional area. Figure 2 shows a tensile strength testing equipment.

2.5.2 Compressive Strength Test

This test measures a material's capacity to withstand axial compressive loads. A prism or cube

specimen is loaded between two platens until it fails, and the strength is calculated from the peak load divided by the cross-sectional area. Figure 3 shows the compressive strength testing equipment.

2.5.3 Flexural Strength Test (ASTM C1185)

Specimens were supported at both ends and loaded at the center in a three-point bending setup until fracture, and the flexural strength and modulus were derived from the load–deflection response. Figure 4 shows the specimen after the test.

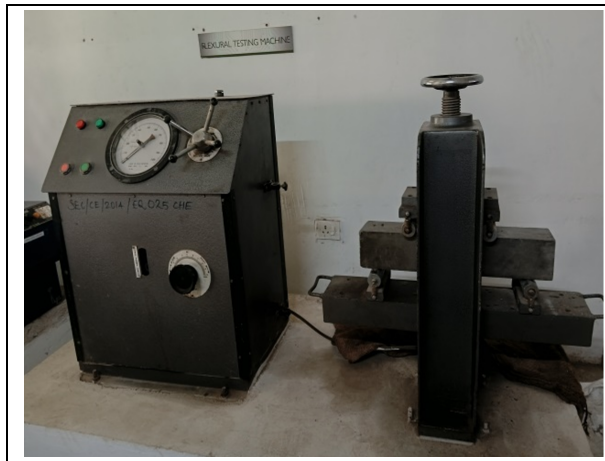


Fig. 2: Tensile strength testing equipment



Fig. 3: Compressive strength testing equipment

2.6 Environmental Considerations

The use of agricultural waste fibers (coir and bagasse) and nano-GO ensured both mechanical improvement and a reduced environmental footprint. Life cycle assessment (LCA) principles were applied qualitatively to estimate the reduction in CO₂ emissions compared to conventional asbestos fiber cement boards. The alkali treatment water was neutralized before disposal to prevent environmental contamination.

3. RESULTS AND DISCUSSION



Fig. 4: Flexural strength test specimen

3.1 Mechanical Performance of Cementitious Composites

The mechanical performance of cementitious composites was evaluated by compressive strength, tensile strength, and flexural strength tests. Specimens were prepared with the mix compositions given in Table 1. The mechanical performance of the composites was evaluated through three-point bending tests to determine flexural strength, modulus of rupture (MOR), and flexural toughness. Results for composites with untreated fibers, alkali-treated fibers without nano-GO, and alkali-treated fibers with nano-GO are presented in Table 1.

3.2 Compressive Strength Results

The compressive strength test results after 28 days of curing are presented in Table 1.

Table 1. Flexural properties of composites with and without nano-graphene oxide

Mix ID	Fiber Treatment	Nano-GO Content (wt.% of cement)	Flexural Strength (MPa)	Flexural Modulus (GPa)	Flexural Toughness (kJ/m ²)
C1	Untreated	0	7.21	6.8	0.52
C2	Alkali-treated	0	9.46	7.4	0.68
C3	Alkali-treated	0.25	10.93	7.9	0.85
C4	Alkali-treated	0.50	11.82	8.3	1.02

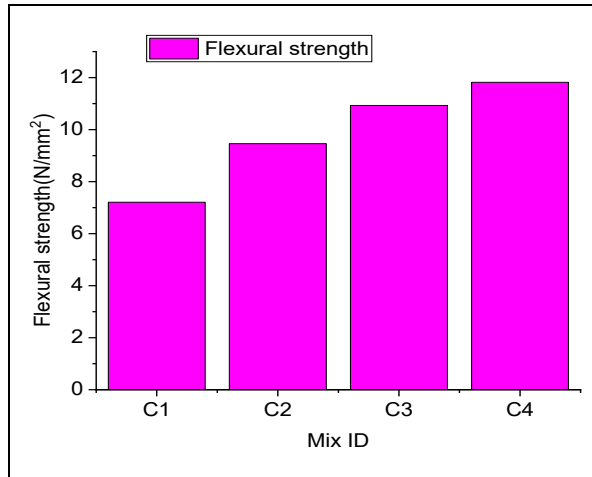


Fig. 5: Flexural properties of composites with and without nano-graphene oxide

Table 2. Compressive strength of concrete

Mix No.	Compressive Strength After 7 Days	Compressive Strength After 14 Days	Compressive Strength After 28 Days
1	18.2	21.5	25.1
2	19.4	23.0	26.5
3	22.8	26.7	30.9
4	21.5	25.4	29.8

Table 2 shows the compressive strength of concrete. Compared to the control mix (CM), nano-GO addition (0.5 wt.%) improved compressive strength by ~23% at 28 days. The 1.0 wt.% nano-GO showed slightly lower improvement due to nanoparticle agglomeration, reducing the effective dispersion in the matrix. Figure 6 shows the compressive strength analysis.

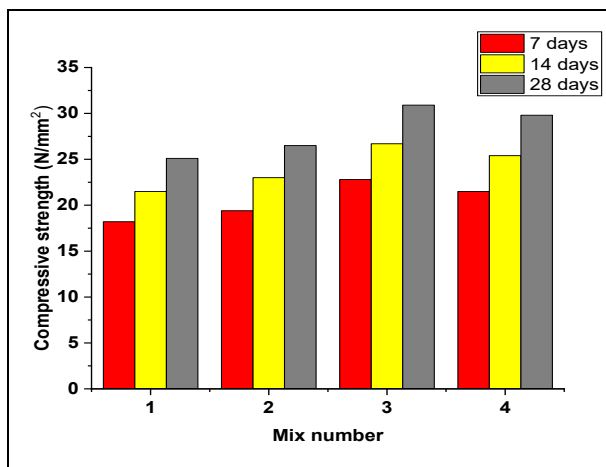


Fig. 6: Compressive strength of composites

3.3 Tensile Strength Results

Tensile strength was measured via a direct tension test to assess fiber pull-out resistance and crack-bridging capacity.



Fig. 7: Compressive strength test on concrete samples

Table 3. Tensile strength of concrete

Mix No.	Tensile Strength After 7 Days (MPa)	Tensile Strength After 14 Days	Tensile Strength After 21 Days
1	2.15	2.38	2.60
2	2.32	2.52	2.78
3	2.85	3.15	3.45
4	2.70	3.00	3.28

Table 3 shows that compared to CM, nano-GO increased tensile strength by almost 33% after 28 days. This improvement results from the increased surface area of nano-GO, which also reduces microcracks and improves hydration products (C-S-H gel formation). Figure 8 shows the tensile strength analysis.

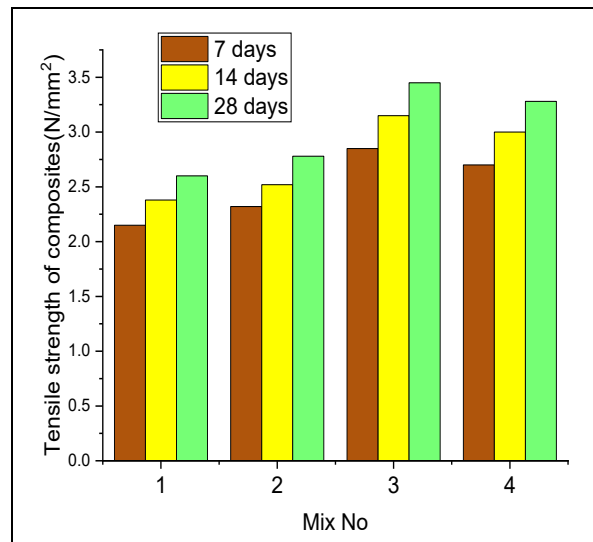


Fig. 8: Tensile strength analysis

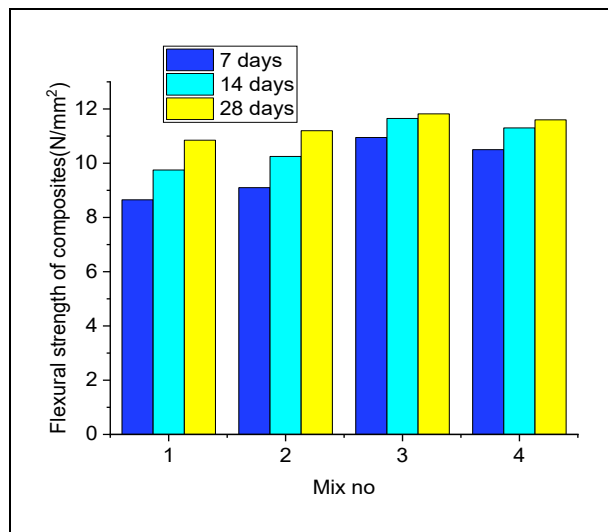
3.4 Flexural Strength Results

Flexural strengths of composites were measured by using a three-point bending test.

Table 4. Flexural strength of composites

Mix No.	Flexural strength After 7 Days (MPa)	Flexural Strength After 14 Days (MPa)	Flexural Strength After 21 Days (MPa)
1	8.65	9.75	10.85
2	9.10	10.25	11.20
3	10.95	11.65	11.82
4	10.50	11.30	11.60

The optimal loading was determined to be 0.5 wt.% nano-GO, which is consistent with previous claims that excessive nano-content might promote clustering, limiting reinforcing efficiency (Table 4). Treated fibers modestly increased flexural strength over CM, but nano-GO produced the best flexural performance, as shown in Figure 9.


Fig. 9: Flexural strength analysis of composites

3.5 Density and Water Absorption

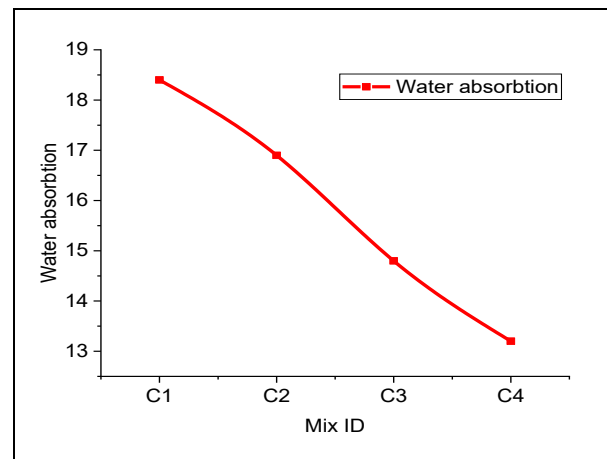
Table 5. Density and water absorption of composites

Mix ID	Density (g/cm³)	Water Absorption (%)
C1	1.62	18.4
C2	1.65	16.9
C3	1.68	14.8
C4	1.71	13.2

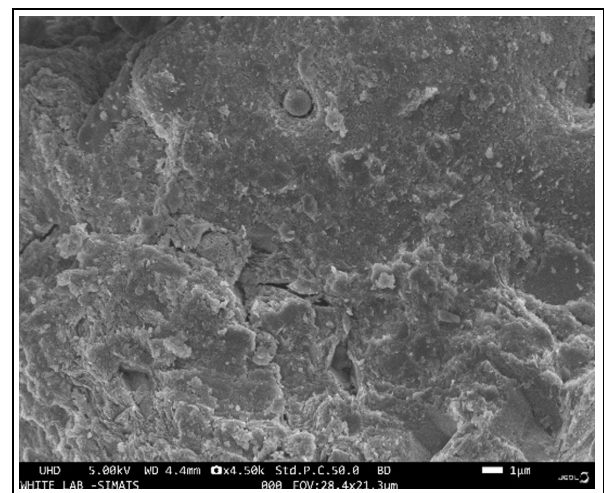
Nano-GO incorporation reduced water absorption by up to 28% compared to untreated fiber composites. This is due to the nano-GO filling microvoids and refining pore structure, which hinders water ingress. The increase in density also supports matrix densification claims. It is shown in Table 5 and Figure 10.

The optimized composite's SEM micrograph (Figure 11) shows a dense and well-integrated microstructure, demonstrating the complementary effects of nano-GO and alkali-treated fibers on matrix densification and fracture resistance. Because of the nucleation action of nano-GO, the surface shows very

little porosity and voids, suggesting excellent hydration and efficient particle packing. Since the alkali treatment improves mechanical interaction with the surrounding cement matrix, the treated coir fibers exhibit a roughened surface morphology with exposed microfibrils. The lack of significant interfacial gaps and the constant interaction between the fiber surfaces and the hydrated cement phases demonstrate this. Even though they are not clearly visible at this magnification, nano-GO sheets are evenly distributed throughout the matrix, serving as nano-fillers to fill in microcracks and encourage the development of more C-S-H gel. Strong fiber–matrix bonding and enhanced load transfer capabilities are indicated by the presence of fine hydration products attached to the fiber surfaces. There are no discernible fiber pull-out voids, indicating that the failure process was dominated by fiber rupture rather than debonding, which improved the flexural strength and ductility.


Fig. 10: Water absorption analysis

3.6 SEM Observations


Fig. 11: SEM analysis of optimized fiber–nano-GO cement composite

3.7 FTIR Analysis

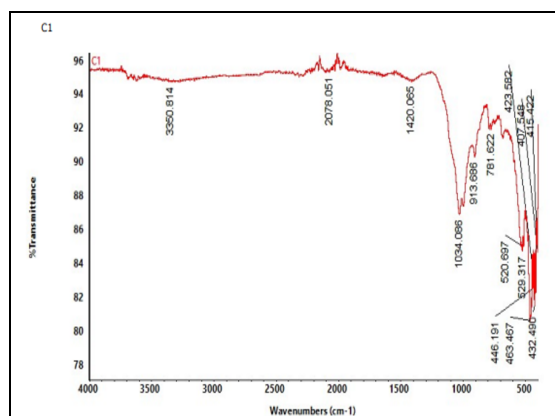


Fig. 12: FTIR spectrum of nano-graphene-mixed concrete

Figure 12 shows the FTIR analysis of graphene-mixed concrete. A large O–H stretching band at about 3530 cm^{-1} can be seen in the optimized composite's FTIR spectrum, suggesting improved hydration due to C–S–H gel formation. Carbonate groups and Si–O stretching in hydrated phases are shown by the peaks at 1420 cm^{-1} and 1034 cm^{-1} . A small band at 2078 cm^{-1} indicates that nano-GO still contains graphene-like structures. Al–O, Si–O, and Ca–O lattice vibrations are represented by bands between 781 and 622 cm^{-1} and 529 and 432 cm^{-1} . Strong fiber–matrix bonding is supported by the absence of lignin/hemicellulose peaks, which indicates that alkali treatment was successful.

4. CONCLUSION

When compared to the control composite, the addition of nano-GO produced notable mechanical improvements, including a 23% increase in compressive strength, a 33% increase in tensile strength, and a 9% increase in flexural strength.

- Alkali-treated natural fibers enhanced mechanical performance on their own, but their combination with nano-GO produced the best microstructural and mechanical outcomes.
- Higher contents decreased efficacy because of particle aggregation; the ideal nano-GO dose was found to be 0.5 wt. %.
- Alkali-treated fibers strengthened the fiber–matrix interfacial bonding, increasing the composite's overall strength and ductility.
- By improving the pore structure and encouraging the creation of a denser C–S–H gel, nano-GO further enhanced mechanical performance.
- When compared to untreated fiber composites, the combined effects of treated fibers and nano-GO

resulted in notable improvements in compressive, tensile, and flexural characteristics.

- Strong fiber–matrix adhesion, consistent nano-GO dispersion, and decreased porosity were all verified by microstructural investigations.
- Due to pore filling and lower permeability brought on by the integration of nano-GO, water absorption dramatically decreased.
- The 0.5 wt. % level consistently produced the best mechanical and microstructural results, despite the fact that higher nano-GO levels showed reduced performance due to agglomeration.
- A sustainable method for creating cementitious composites that are denser, stronger, and more resilient is the combination of treated natural fibers with nano-GO.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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