Research Article

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Alkali and Non-alkali Treated Coconut Coir Fiber-Reinforced Coconut Shell Powder/MWCNT-Filled Polyester Matrix Composite: An Experimental Comparison

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ABSTRACT

Engineering, biomedical, and medicinal applications effectively employ the extraction of natural resources. Natural and recycled natural fibers are highly advantageous for producing hard-core and soft-core composite materials and equipment. Natural fiber processing can yield diverse material characteristics. Particularly, processes such as chemical treatment, heat treatment, fine coating, and polishing (gold) composites can significantly enhance the characteristics of these materials. A further selection of non-synthetic solutions is designated for material application and environmentally friendly biodegradation to the earth. Due to the non-destructive nature of composites, many synthetic resources are incorporated into the environment, which can lead to corrosion and atmospheric pollution. Therefore, the remarkable movement toward replacing these compounds with natural coconut fiber, whether refined or untreated, shell powder, and composites incorporating multi-walled carbon nanotubes (MWCNT) contributes to establishing a more environmentally sustainable society. This work included rigorous mechanical testing to compare the effects of alkali and non-alkali treatments. In particular, the tensile strength is 17.06% higher, the flexural strength is 7.35% higher, and the hardness on Shore D is 5.81% higher than that of non-alkali-treated material. The SEM analysis revealed the alkali-treated and untreated composites' consecutive failure zones, regions, and matrix structures.

Keywords: Coconut coir/shell powder; NaOH treatment; MWCNT; Chemical treatments; Mechanical properties.

1. INTRODUCTION

This article extensively highlights the potential applications of natural fiber polymer composites in engineering, which is rapidly establishing itself. The intrinsic properties of natural fibers (NFs) present several obstacles that researchers must overcome to advance and use Natural Fiber Polymer Reinforced Composites (NFPRCs). These difficulties include incompatibility with polymer matrices, water absorption capacity, thermal stability, and fiber quality. Economic and ecological considerations drive emerging studies in NFRPCs (Khalid *et al.* 2021). Composites made of natural fibers are sourced from all over the world. Fiber, filler, and well-blended resins are all created using these resources efficiently. Chunks of fiber, treated fiber, fiber positioned at 0, 90, and 180 °, and sandwiched fibers are all fiber materials.

Similarly, filler materials are extracted using pyrolysis, heat treatment, chemical treatments, and mixing. Similarly, a simple solution is mixed with

various organisms and plant parts to create natural resin. There are problems with plant fiber because of the hydroxyl groups in lignocellulose. Plant fibers are hydrophilic, making them prone to moisture damage and mixing poorly with hydrophobic thermoplastics. These constraints cause problems when using plant fibers to reinforce polymers (Elfaleh *et al.* 2023). The classification of natural fiber reinforcement may be based on factors such as length, dimension, and orientation. Since natural fibers have a high strength-to-weight ratio and a low density, their properties make them well-suited for lightweight composites and reinforcing materials. Their microstructure and chemical compositions modify the mechanical features of fibers. Among all these variables, the diameter of the fiber is the most flexible and significantly affects its strength (Ramu *et al.* 2022a). The water absorption capacity of natural fibers is described as the presence of hemicellulose, which imparts hydrophilic characteristics to them, reducing their compatibility with any hydrophobic substrate. Greater levels of cellulose and crystallinity often lead to improved fiber strength characteristics, whereas lignin has the opposite effect (Karimah *et al.* 2021).

There are four separate but interconnected stages of fiber development. These are initiation, primary cell wall (PCW) formation for fiber extension, secondary cell wall (SCW), enzyme biosynthesis for cellulose accumulation and cell wall swelling, and maturity. After starting, fiber cells quickly and longitudinally extend, giving the fiber a final length of 22–35 mm and allowing a thin layer of the primary cell wall (PCW) to stick to it. Throughout this process, fibers form unidirectional bundles that only become separated from each other at the end of the fiber elongation cycle (Liu, 2018). Fibers can be categorized into two forms: continuous strands and discrete particles. The classification is based on the length-to-diameter (l/d) ratio, distinguishing between continuous and discontinuous (chopped) fibers.

Additionally, the structural organization of the fiber-reinforced phase is typically classified as either woven or non-woven, where woven fabrics are defined by the consistent interlacing of perpendicular strands in a regular pattern. Several intertwined fibers compose yarns, forming a cohesive structure. To a certain degree, the twist angle determines the fibers' cohesiveness and the yarn's strength. However, beyond this point, the maximum strength of the fibers diminishes due to an increase in obliquity (Bar *et al.* 2018). Furthermore, an increase in the angle at which the fibers twist directly correlates with reducing the link strength between the fibers and the resin. This loss in bond strength leads to poorer permeability and ultimately results in poor mechanical characteristics (Shah *et al.* 2013). One way to improve the adhesion between fibers and the polymer matrix and lower the amount of water absorbed is through chemical interaction; surface modifications are usually needed. Investigators have devised alkaline, silane, and other chemical treatments that boost fiber-matrix interface and laminate qualities (Fig. 1) (Gholampour *et al.* 2020). Blended characteristics are mainly attributable to differences in plant species, growing circumstances, and fiber extraction techniques. Furthermore, characteristics are type-specific and degree-dependent, depending on the fiber cell shape and degree of polymerization of the cellulose (Ho *et al.* 2012). Linear cellulosic macromolecules strongly interact with hemicellulose and lignin, providing rigidity to the fiber through hydrogen bonding. The cellulose present in the fiber cell wall plays a key role in binding the structural components together, thereby contributing to overall fiber integrity and strength (Chen *et al.* 2014; Hasan *et al.* 2021).

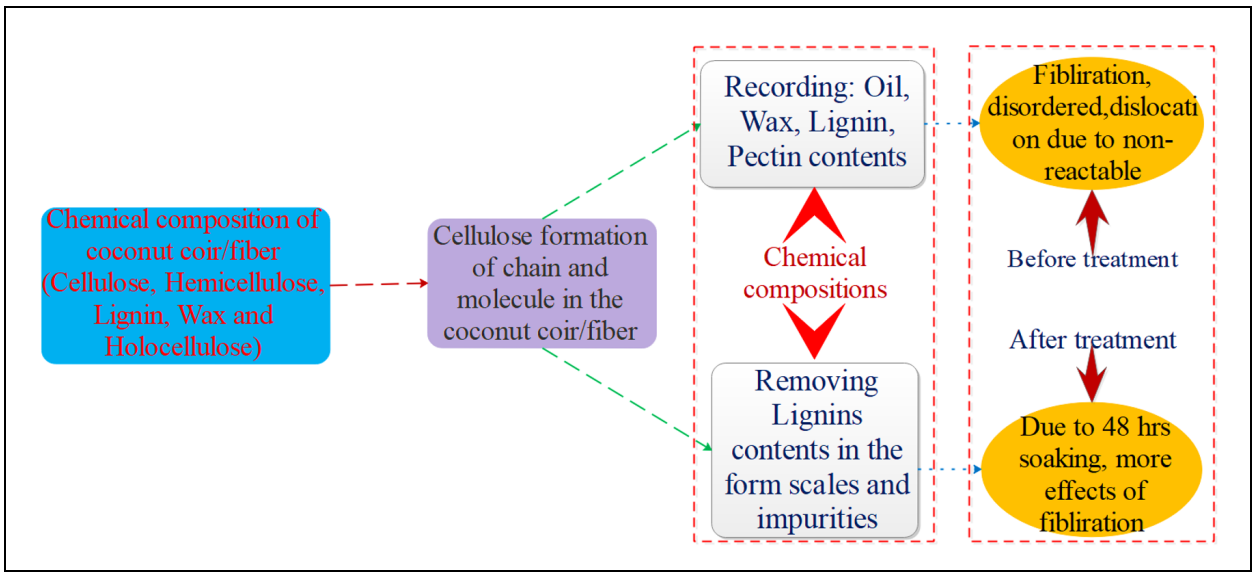


Fig. 1: Chemical composition influences the characteristics of the material (Ho *et al.* 2012; Chen *et al.* 2014; Gholampour *et al.* 2020)

Breaking down a novel polyurethane made from tung oil and composite material, including wood flour, was observed over 383 days while exposed to soil or vermiculite. In all cases, hydrolytic degradation was the predominant deterioration mechanism. The study of the biodegradability of polyurethanes derived from tung oil and a composite containing wood flour demonstrated their limited susceptibility to microbial assimilation (Hoang *et al.* 2022). Hydrolytic degradation is the primary method by which these materials break down when exposed to harsh conditions. The glass transition temperature of the materials increases when exposed to degrading agents due to the elimination of free or dangling pendant chains that soften the material (Aranguren *et al.* 2012). A honeycomb core made of a non-woven flax-epoxy composite was tested at three core densities: 100 g/m^2, 150 g/m^2, and 200 g/m^2. Acoustic emission (AE) techniques were used to assess honeycomb core deterioration during compression testing. A modified paper-making technique was employed to enhance reinforcement, and a custom-designed compression mould was used to transform flat thermoset prepregs into a honeycomb structure. The results indicate that increasing areal density leads to improved mechanical properties, particularly higher compressive strength and compressive modulus (Selmi *et al.* 2022). Table 1 displays several chemical treatments and their corresponding effects. However, most studies use peroxide treatments on natural fibers because of their simplicity and reasonably favourable mechanical qualities (Ali *et al.* 2018).

Table 1. Effects of chemical treatments (Kabir *et al.* 2012)

Treatment	Effects
Alkali	Fiber-filler bonding, thermal stability, energy, and heat resistance.
Acetylation	It enhances fiber quality and thermo-mechanical properties.
Enzyme	Lowering Lignin levels.
Isocyanate	Surface and internal structure commutations.
Mercerization	Reduce moisture and strengthen fiber/filler.
Silane	Chemical treatment is involved, and the surface appearance is altered.
Sodium Chloride	As a silane group, sodium chloride alters the material's internal and exterior structure to maintain integrity.

The PLA-based bio-composites are cost-effective, possess either complete or partial biodegradability depending on the reinforcing types, are lightweight, and exhibit excellent thermal and mechanical characteristics (Ebrahimi *et al.* 2022). The PLA-based composites have been identified as promising candidates for replacing conventional non-biodegradable and non-renewable synthetic materials. Enhancing the nano- and micro-scale characteristics of natural fibers and cellulose can significantly improve the mechanical and thermal properties of PLA-based composites (Trivedi *et al.* 2023).

The TAPPI test technique, the chlorination method, and the Kurschner-Haffner method were used to examine the chemical composition of pineapple leaf fiber holocellulose. The chemicals analyzed comprised

cellulose *via* the Kurzen-Haffner technique, holocellulose through the chlorination method, hemicellulose also utilizing the chlorination method, ash content assessed by the T21-om-93 method, lignin evaluated using the T22-om-98 method, and sodium hydroxide solubility determined by the T23-om-88 method (Daud *et al.* 2014). The dominant chemical makeup of pineapple leaf fiber was holocellulose, which constituted 85.7%, cellulose at 66.2%, hemicellulose at 19.5%, and lignin at 4.2%. The SEM investigation showed a diminished composition of fiber structure, rendering it a feasible alternative to pulp in the pulp production industry, thus promoting advancements in sustainable technology (Sethupathi *et al.* 2024). Table 2 presents a comparison of the physico-chemical, thermal, mechanical, and biological properties of natural and synthetic fibers.

Table 2. Comparison of Physio-Chemical-Thermal-Mechanical-Biological properties between natural and synthetic fiber (Kabir *et al.* 2012; Ebrahimi *et al.* 2022; Trivedi *et al.* 2023)

Properties	Synthetic Resources	Natural Resource	Identification of Properties
Density	High	Low	Physical
Specific strength	Moderate	High	Mechanical
Strength	High	Moderate	Mechanical
Thermal sensitivity/ stability	Low	High	Thermal
Utilization of energy	High	Low	Dielectric/ Thermal
Moisture analysis	Low	High	Physical/ Biological
Eco-friendly/ Biodegradable	No	Yes	Biological
Recyclable	Moderate	Good	Biological
Radiation and others	High	Low	Thermal/ Dielectric/ Biological
CO_2 emission	Yes	No	Thermal

The present investigation highlights coconut coir as the principal material, exploits filler particles (micro/nanofiller) to augment bonding inside the matrix, and synthesizes polyester resin to achieve flawless interlocking structures in the composite material.

2. MATERIALS AND METHOD

Materials used in this study were procured and processed from Metro Composite, Chennai. The constituents include coconut coir, powdered coconut shell microparticles, multi-walled carbon nanotubes (nanoparticles), sodium hydroxide solution, breather material, and distilled water. Fabrication was carried out using hot air weaving and vacuum layup techniques. Fig. 2 presents the material specifications and associated details.

3. EXPERIMENTAL WORK

Coconut coir, coconut shell powder, multi-walled carbon nanotubes (MWCNTs), and polyester resin were used to fabricate both treated and untreated composites for evaluating their thermo-mechanical properties. The fabrication process was carried out using a vacuum bagging technique. An experimental comparison was performed to study the effect of chemical treatment, in which sodium hydroxide treatment was applied to selected composite constituents. The NaOH-treated composite preparation is illustrated in Fig. 2. Composite laminates were developed by incorporating a balanced formulation of reinforcement materials within the polyester resin matrix to form a structural laminate system. The detailed composition and specifications of both treated and untreated composites are provided in Table 3. This approach ensured uniform dispersion of reinforcements and the formation of a stable composite structure.

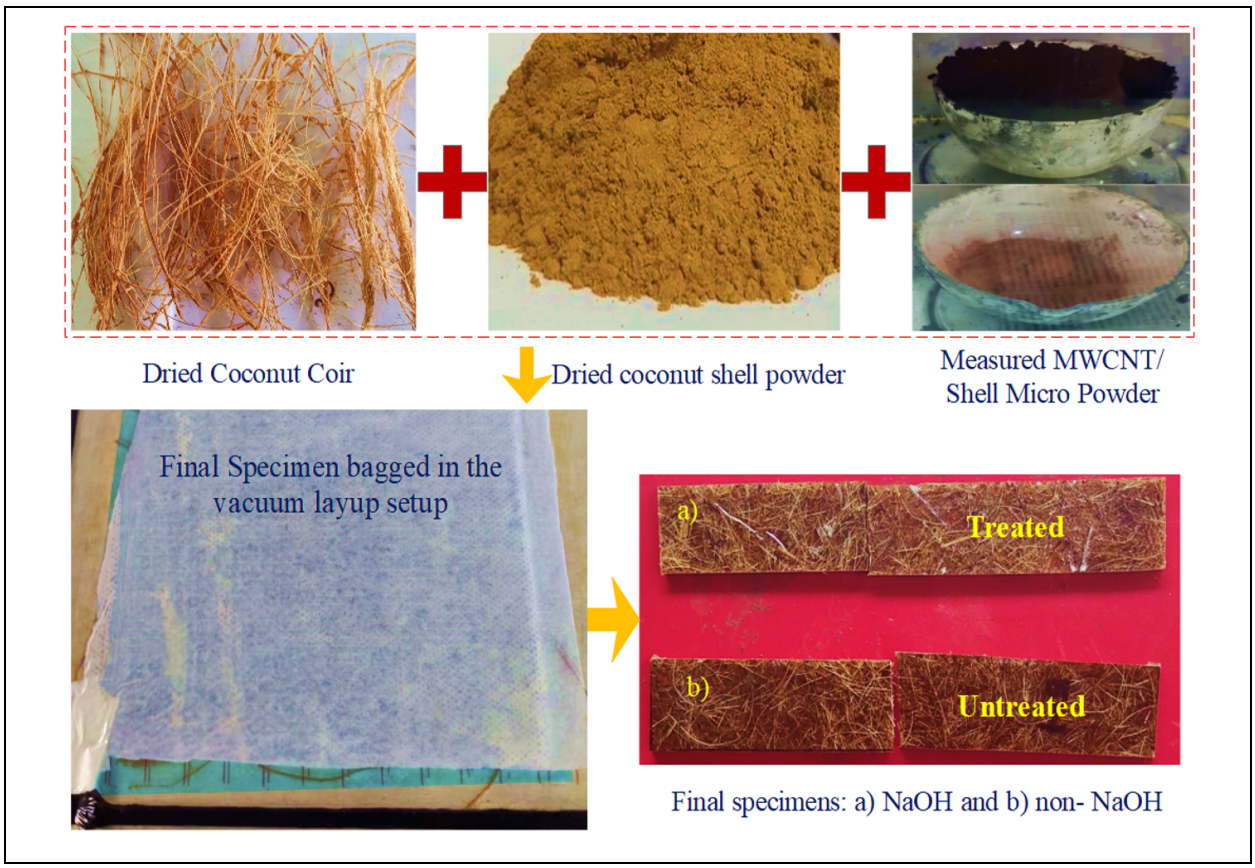


Fig. 2: Coconut fiber/filler and ultra-filler polyester matrix composite

Table 3. Laminated composite specifications

Treatment	Coconut coir in %	Coconut Powder in %	MWCNT in %	Polyester resin in %
NaOH	20	5	0.5	74.5
-	20	5	0.5	74.5

4. RESULTS AND DISCUSSION

The treated and untreated composites for the experimental comparison were fabricated using coconut coir, shell powder, and MWCNT-filled polyester nanocomposite. Table 4 specifies the treatment categories: tensile strength, flexural strength, and shore D hardness.

Table 4. Treated and untreated composite mechanical properties

Treatment	Tensile Strength (MPa)	Flexural Strength (MPa)	Hardness Shore D
NaOH	29	34	77.5
-	25.5	31.5	73

The untreated composite samples exhibit the most significant variations, not the treated ones. The ideal values for decreasing surface roughness during Inconel 625 machining were current (4 A), pulse on time (30 µs), and pulse off time (80 µs).

4.1 Mechanical Properties

Alkali treatment promotes the development of a hierarchical bonding structure within the composite material. During NaOH treatment, impurities and surface impurities are removed from the coconut coir, improving interfacial adhesion within the composite. Furthermore, the initial surface roughness and bleached layer of the coconut coir were removed during treatment. The untreated surface typically contributes to mechanical interlocking through frictional adhesion. The tensile characteristics of the coconut coir composite, modified with a 5% NaOH solution and immersed for 48 hours, were 12.06% greater than those of the untreated composite (Fig. 3). This process effectively enhances tensile strength by eliminating contaminants such as lignin and pectin from the coconut coir. By increasing the surface roughness, the treatment significantly improves the adhesion of the fibers.

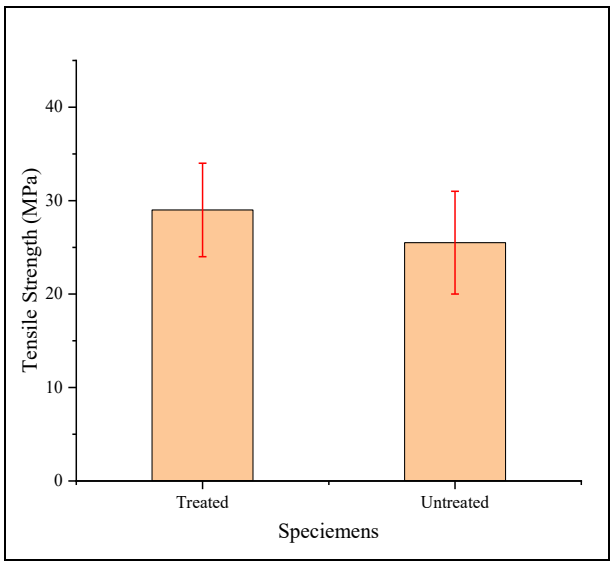


Fig. 3: Treated and untreated composite tensile strength deviation

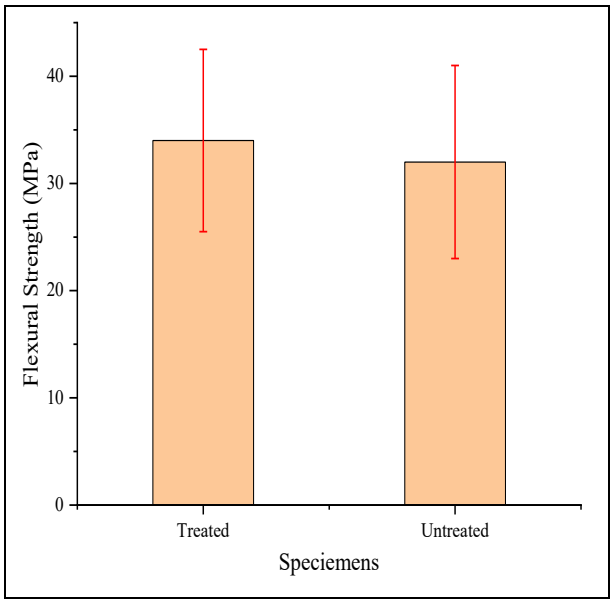


Fig. 4: Treated and untreated composite flexural strength deviation

The results from the three-point bending test indicate that the composite incorporating treated coconut coir fiber-reinforced shell powder and MWCNT-filled matrix polyester achieved a 7.35% improvement in flexural strength when compared to its untreated counterpart, as shown in Fig. 4, which is in accordance with a literature study (Benkhelladi *et al.* 2020). The NaOH composite has a higher maximum yield strength than the non-NaOH composite. Coconut coir is anticipated to exhibit a broad spectrum of cellulose content, while non-NaOH composites are expected to have elevated hemicellulose content.

The composite without NaOH treatment induces a more significant flattening of the fiber, increasing the probability of fiber pullout and diminishing the strength of the laminate construction. Additional noteworthy aspects are emphasized, suggesting that the bonding system in the untreated composite has deteriorated its mechanical properties (Ramu *et al.* 2022b). Fig. 5 shows the variation in hardness between the treated and untreated composites.

4.2 Surface Morphology

The SEM method examines the internal fractures and surface damage of the alkali- and non-alkali-treated coconut coir/shell powder/MWCNT-filled polyester matrix composite. The SEM examination identifies two contrasts (Hasan *et al.* 2021). The delamination and dissociation of fibers from the polyester matrix and woven mat structure are shown in Fig. 6a.

A universal testing machine facilitated the tensile and flexural testing of the composites not subjected to alkali treatment. The non-alkali-treated composites showed a coconut shell/MWCNT matrix structure that spread and broke over the composite surface. Furthermore, upon reaching its maximal loading condition, the filler matrix depletes its energy, leading to the delamination and deconstruction of the fiber from the weaved mat. Ultimately, the matrix and its positions are responsible for the material's failure.

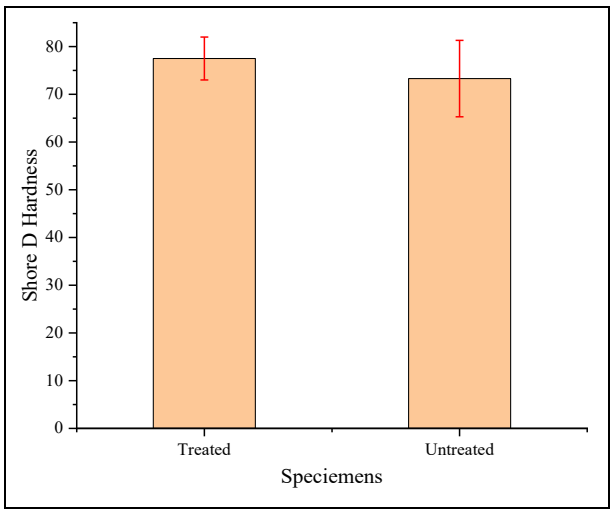


Fig. 5: Treated and untreated composite hardness deviation

Fig. 6b. shows that the failure zone of the fiber/matrix has achieved its highest loading level. Compared to non-alkali-treated composites, alkali-

treated fiber/filler polyester matrix composites have higher energy resistance. Scanning electron microscopy analysis showed that the filler matrix first broke into small crystal particles, making the coconut coir/shell powder stiffer in its protruding region. The interaction between coconut coir and fiber matrix structures demonstrates a greater yield strength.

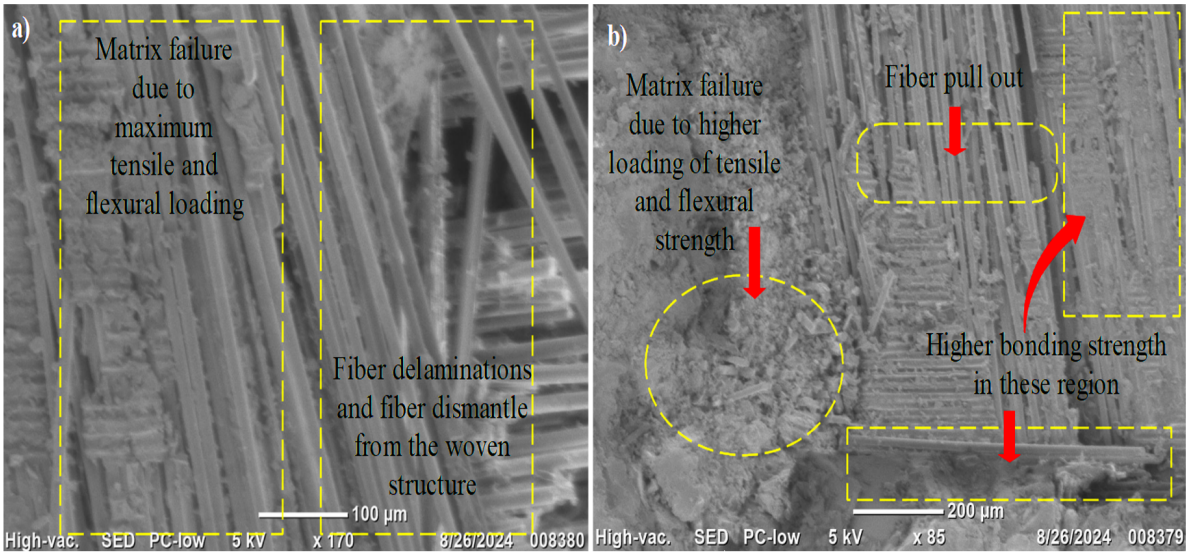


Fig. 6: (a) Non-alkali treated and (b) Alkali treated nano composition

5. CONCLUSIONS

The present study compares composites subjected to different chemical treatments and concentrations, emphasizing the role of eco-friendly materials such as coconut coir, shell powder, and MWCNTs in sustainable composites. NaOH treatment was found to significantly influence interfacial bonding, structural integrity, and failure behaviour of the fiber–matrix system. Experimental results show that alkali-treated composites exhibit improved mechanical performance compared to untreated samples, with increases of 12.06% in tensile strength, 7.35% in flexural strength, and 5.81% in hardness. Scanning electron microscopy (SEM) analysis of fracture morphology revealed distinct fiber bonding, fiber failure, and matrix failure zones in both conditions. Overall, the MWCNT–polyester composites reinforced with treated natural fibers demonstrated superior mechanical properties over their untreated counterparts.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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