



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 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 degrees, 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 (figure. 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). Note that linear cellulosic macromolecules strongly couple with hemicelluloses and lignin, giving the fiber its rigidity through hydrogen bonds. The cellulose in the fiber's cell wall binds the fibers together (Chen *et al.* 2014; Hasan *et al.* 2021).

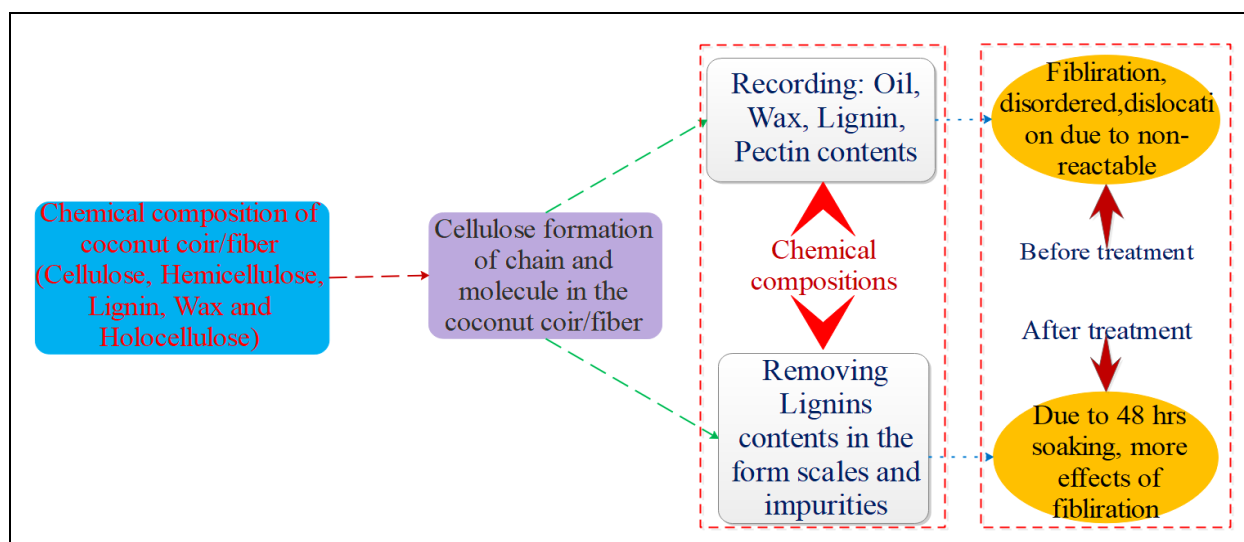


Fig. 1: Chemical composition influences the characteristics of the material (Ho *et al.* 2012; Chen *et al.* 2014; Gholampour *et al.* 2020)

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/m², 150 g/m², and 200 g/m². Acoustic Emission (AE) assessed honeycomb core deterioration via compression strength operation. A distinct paper making technique was employed to enhance reinforcement. A custom-designed compression mould was utilized to convert the flat thermoset prepregs into a honeycomb structure. The findings indicate that a rise in areal density correlates with improved mechanical properties, especially in stress and compressive modulus (Selmi *et al.* 2022). The table. 1 displays several chemical therapies and their corresponding effects now available on the market. 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 present investigation has identified PLA-based composites as the most outstanding candidates for substituting current non-biodegradable and non-renewable synthetic materials (table. 2). Strengthening the nano and micro dimensions of natural fibers and cellulose can significantly improve the mechanical and thermal characteristics of PLA (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 scanning electron microscopy (SEM) investigation unveiled 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. 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

2. RESEARCH GAP

The universal testing machine determines coconut coir as the composite material to assure its capacity to endure the highest yield stress according to defined load and deformation criteria. 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.

3. MATERIALS AND METHOD

Metro Composite in Chennai has gathered and processed the following ingredients: coconut coir, powdery coconut shells (microparticles), multi-walled carbon nanotubes (nanoparticles), sodium hydroxide solution, breather material, distilled water, hot air weaving, and vacuum layup setup modes (Ramu *et al.* 2023). This figure. 2 displays the material specification and supplementary information.

4. EXPERIMENTAL WORK

The acquisition of raw materials may begin the manufacturing process. Different kinds of coconut coir, shell powder, multi-walled carbon nanotubes

(MWCNT), and polyester resin were used in both treated and untreated composites to determine their thermo-mechanical properties that were important for each use. This work primarily investigates the fabrication process in a vacuum bagging setting. Through an experimental comparison, figure. 2 illustrates the treatment of the

NaOH composite (Ramu *et al.* 2023; Ramu *et al.* 2024). Table. 3 displays the composite concentrations and specifications of both the treated and untreated components. A well-balanced composition synthesized the composite resin as a lamination, creating the structural matrix.

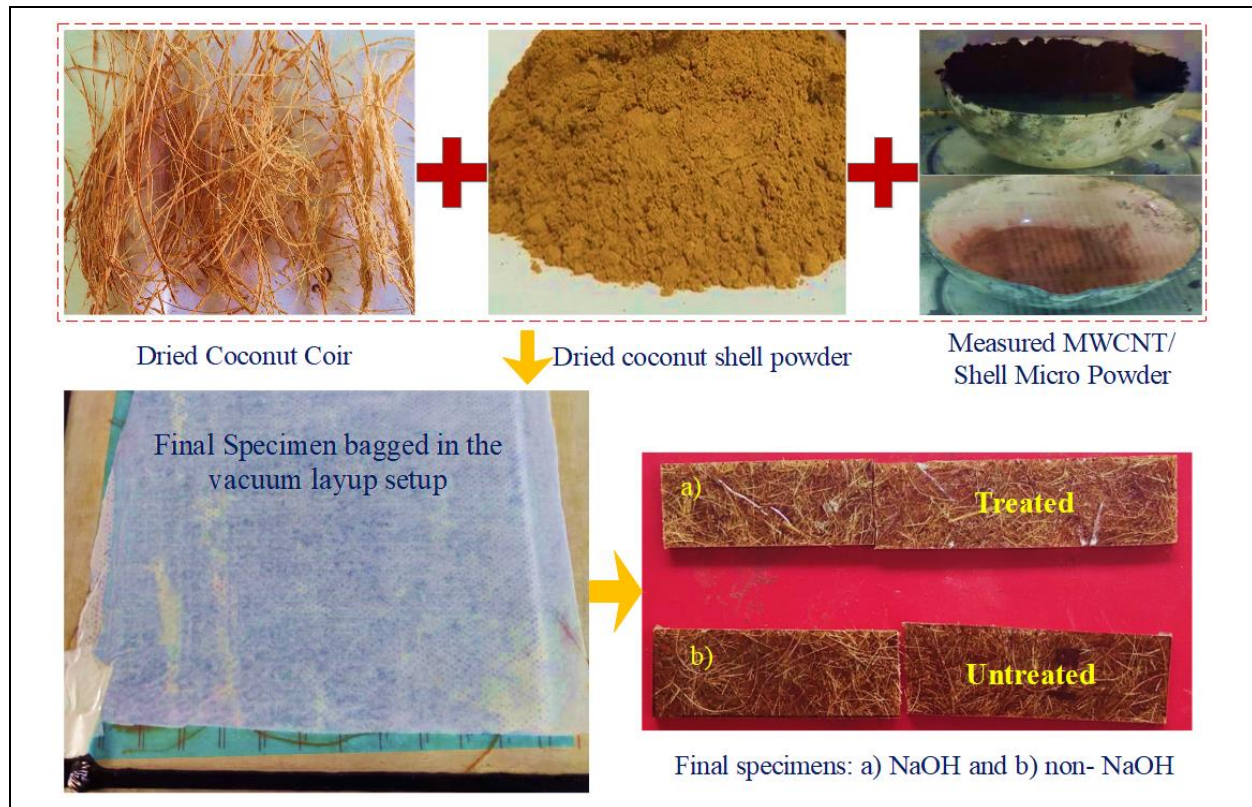


Fig. 2: Coconut fiber/filler and ultra-filler polyester matrix composite

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

5. RESULTS AND DISCUSSION

The treated and untreated composites for the experimental comparison are manufactured using coconut coir, shell powder, and MWCNT-filled polyester nanocomposite (Ramu *et al.* 2023). 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 mechanical characteristics of composites are the subject of profound debate. The ideal values for decreasing surface roughness during Inconel 625 machining were current (4 A), pulse on time (30 μ s), and pulse off time (80 μ s).

5.1 Mechanical Properties

The alkali treatment stimulates the formation of the bonding hierarchy inside the composite material. Impurities and scales are eliminated from the coconut coir during the NaOH cure. Furthermore, the initial roughness and bleached surface of the coir were removed. The initial surface induces mechanical adhesion via friction. 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 shown in the figure. 3 (Ramu *et al.* 2023). 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.

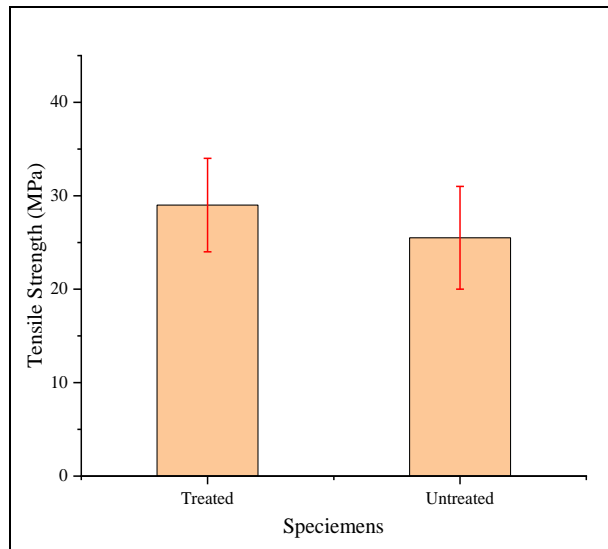


Fig. 3: Treated and untreated composite tensile strength deviation

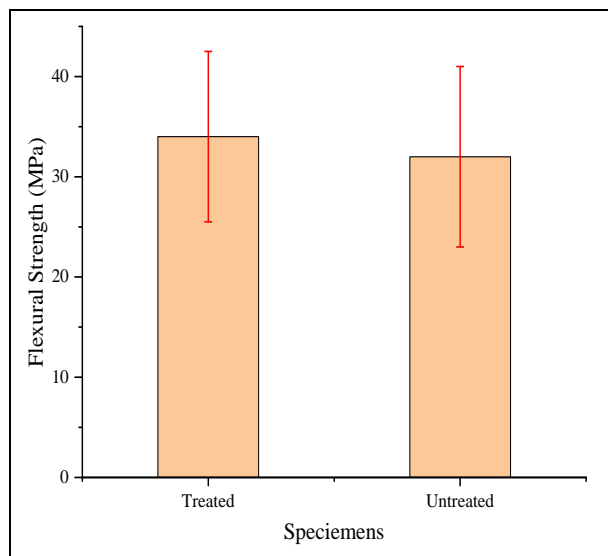


Fig. 4: Treated and untreated composite flexural strength deviation

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 Figure 4 (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).

5.2 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 Figure 6a.

A universal testing machine facilitated the tensile and flexural testing of the composites that had not been 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.

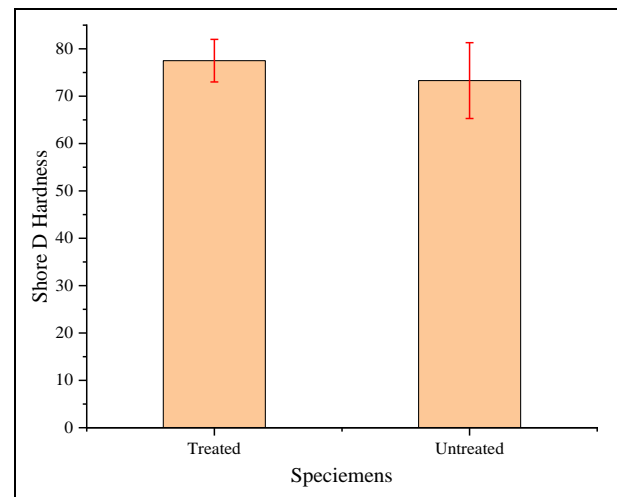


Fig. 5: Treated and untreated composite hardness deviation

Figure 6b. Evidence 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 (SEM) 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.

Finally, the alkali and non-alkali treated composites are subjected to scanning electron microscopy (SEM) analysis to record the structural

bonding between the matrix, the matrix's integrity, fiber breakages/ fractures, and delamination. The scanning electron microscopy (SEM) test revealed that the composite subjected to alkali treatment exhibited superior structural integration (fiber/filler/polyester matrix) compared to the composite untreated with alkali.

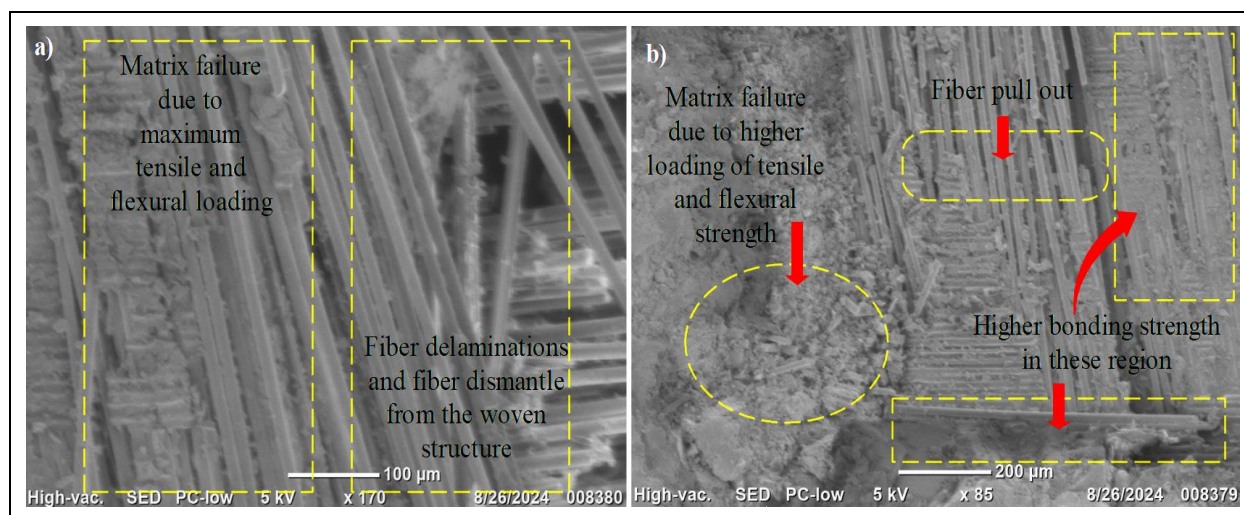


Fig. 6: (a) Non-alkali treated and (b) Alkali treated nano composition

6. CONCLUSIONS

The present experimental study undertakes a comparative analysis involving two separate chemical therapies and their varying quantities. The resource materials underscore the importance of ecologically sustainable biodegradation and the breakdown processes in the global environment. It is strongly recommended that reinforced materials be used for the coconut coir/shell powder/MWCNT. The use of NaOH treatment sparked much debate in the scientific community about composites' structural bonding, strength, integrity, retraction, and contractions, as well as the underlying reasons for the failure of the fiber/filler matrix. Experimental data analysis revealed that composites treated with alkali exhibited superior mechanical characteristics compared to composites not treated with alkali. The composite subjected to alkali treatment showed a 12.06% enhancement in tensile strength, a 7.35% improvement in flexural strength, and a 5.81% rise in hardness. The research focused on fracture morphology, which includes the fiber bonding zone, fiber failure zone, and matrix failure zone, and was analyzed through scanning electron microscopy (SEM) in both treated and untreated conditions with NaOH. The results of the experimental comparison revealed that the MWCNT polyester matrix composite reinforced with treated coconut coir/shell powder had significantly better mechanical properties than its untreated counterpart.

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CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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