

Surface-modified *Agave vilmoriniana* fibers for enhanced thermo-mechanical and interfacial performance in composites

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Abstract

This study examines *Agave vilmoriniana* leaf fibers after consecutive alkali, silane, and starch treatments for polymer composites. The treatments reduce hemicellulose, moisture absorption and improve fiber stability. X-ray diffraction Analysis showed intensified (002) peaks that confirm improved crystalline ordering, while Fourier Transform Infrared spectra showed the reduction of hemicellulose and lignin and after alkali and silane treatments by the peaks around 1730 cm⁻¹ band and the peak 750 cm⁻¹ confirms the emergence of siloxane linkages. Subsequently, thermal analysis using Thermogravimetry showed improved thermal stability with onset and maximum degradation temperatures rising from ~285 to 305 °C and ~340 to 355 °C respectively. Mechanical testing shows starch coating increases stiffness to 23.42 GPa. Scanning Electron Microscopic image observations confirmed the gradual surface modifications. The result makes that the fibers were more suitable for high-performance, moisture-resistant and polymer composites.

Keywords: *Agave vilmoriniana* leaf fibers, alkali treatment, silane coupling, starch coating, mechanical properties.

Data Availability: All data supporting the findings of this study are available from the corresponding author upon request.

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1. Introduction

Global usage of fiber-reinforced composite's continues to gradually expand which is driven by their elevated strength-to-weight ratio, corrosion resistivity and design versatility. Traditionally, alternative synthetic fibers took over the market which were accounting for more than 90% of the 12 million tonnes of fiber reinforcements produced annually worldwide. While these materials deliver excellent mechanical performance, their production is energy-intensive, non-biodegradable, and heavily reliant on petrochemical resources, leading to significant environmental concerns. For example, the carbon footprint of glass fiber production can exceed 1.5 kg CO₂ per one kilo fiber and end-of-life disposal often results in landfill accumulation or incineration. Alternatively, natural fibers are renewable, biodegradable, lightweight, and often created with far reduced energy inputs. Recent life-cycle studies show that natural fiber composites can cut energy use during production by up to 60% and greenhouse gas emissions by more than 50% compared to synthetic composites. To meet the European Union's End-of-Life Vehicle Directive (2000/53/EC), which requires 95% material recovery and recyclability, the European automotive industry has already used natural fiber composites in door panels, dashboards, and interior trims^[1-4].

Natural fibers are good for the environment in some ways, but they also have some fundamental issues that make them hard to utilize in high-performance applications.

Because they have a lot of cellulose and hemicellulose in them, they are hydrophilic, which means they hold too much water, swell, and lose dimensional stability. These challenges are particularly evident in *Agave vilmoriniana* fibers, which represent a promising yet underexplored reinforcement material. However, their high lignin content and the presence of surface impurities restrict effective compatibility with polymer matrices^[5,6].

Natural fibers are inherently hydrophilic because of the abundance of hydroxyl groups in their composition structure. This characteristic adversely affects dimensional stability and weakens fiber-matrix adhesion. Moisture uptake leads to fiber swelling, the development of internal stresses, and the formation of interfacial microcracks, thereby reducing the efficiency of stress transfer^[7-9]. The polarity mismatch between fibers-matrices paves the way to poor wettability, low resin impregnation, and void formation. These defects act as sites of stress concentration and adversely affect the tensile, flexural, and fatigue performance of composite materials^[8,10,11]. Repeated cycles of moisture absorption and desorption accelerate interfacial debonding and degradation, leading to a progressive reduction in material durability. To address these limitations, surface treatments such as alkali treatment, silane modification, and starch coating have been extensively investigated to reduce hydrophilicity and enhance fiber-matrix compatibility^[12,13].

These modifications enhance interfacial adhesion by reducing polarity and promoting chemical interaction with the matrix. However excessive treatment can modify the fiber structure or compromise environmental sustainability. The primary challenge lies in developing treatment strategies that achieve improved interfacial adhesion while preserving the inherent environmental benefits of natural fibers^[14,15].

Agave vilmoriniana leaf fiber (AVLF) has get attention as a promising plant based natural reinforcement owing to its higher tensile strength, lower density, and renewable origin. Although several studies have reported the influence of alkali treatment on the mechanical performance of Agave derived fibers, investigations specifically focused on *Agave vilmoriniana* remain limited^[16,17]. The combined impact of multi-treatments such as alkali treatment, silane functionalization, and starch coating has not been explored in detail. Some studies have indicated that treating *Agave* fibers with alkali solutions followed by silane agents improves mechanical properties and fiber–matrix adhesion^[18,19]. However, a complete analysis of the synergistic effects of these treatments for *Agave vilmoriniana* is yet to be reported. This research gap offers the potential to develop an optimized treatment approach to improve both mechanical performance and moisture resistance in *Agave vilmoriniana* fiber-reinforced composites^[3].

2. Materials and Methods

2.1 Materials used

AVLF's were manually extracted from *Agave vilmoriniana* plant leaves which were locally cultivated around chettipalayam, Coimbatore, Tamil Nadu, India. Sodium Hydroxide (NaOH) and Aminopropyltriethoxysilane (APTES) were purchased from Sigma-Aldrich Chemicals Private Limited, Bengaluru, Karnataka, India for surface modification and enhancement of AVLF's. For surface coating, Cassava Starch powder purchased from a local supplier for the AVLF's surface coating.

2.2 Alkali treatment of AVLFS

An alkali treatment with a 6% weight by volume of NaOH solution was used to improve the adhesion tendency and interfacial compatibility in *Agave vilmoriniana* fibres. AVLFS were immersed in the alkali at ambient temperature for 4 Hrs^[11,20]. This method eliminates surface-confined polysaccharides such as hemicellulose, waxes and residual lignin that makes a rougher fibre surface and more accessible OH groups for future modification^[21,22]. Subsequently, the AVLFS were thoroughly cleansed with distilled water until achieving a neutral pH which were indicating the complete removal of residual alkali. After that the AVLFS were air-dried for 48 hours at ambient temperature and stored in sealed moisture-free containers.

2.3 Silane treatment of AVLFS

Silane treatment was followed by NaOH treatment to improve interfacial binding and enhance the chemical interaction of AVLFS with polymer matrices. A 2% (v/v) APTES formulation was prepared using a solvent mixture of 95% ethyl alcohol and 5% water. The pH of the solution

were maintained at around 4.5 through hydrolysis induced by acetic acid. The pre-treated AVLFS were subsequently immersed in the silane solution and continuously agitated for 3 hours to facilitate bonding between silane reagent and hydroxyl groups on the fiber surface. AVLFS were cleansed with ethyl alcohol to eliminate residual silane and subsequently dried at ambient temperature for 48–50 hours. This alteration was designed to generate covalent bonds at the fiber-matrix interface^[23–26].

2.4 Cassava starch coating of AVLFS

A surface coating composed of cassava starch was applied to AVLF's after surface treatment to increase their hydrolytic stability and interfacial integrity. A 10% w/v cassava starch solution was prepared by dispersing the starch powder in distilled water and heating the mixture to 80 °C with continuous stirring until complete gelatinization was achieved. The silane-treated fibers were then encased in the heated starch mixture for 30 mins to ensure uniform surface coverage. After taking the fibers from the solution, the excess starch was allowed to drain and the coated AVLF's were oven dried at room temperature for 48Hrs. The starch film was intended to serve as a biodegradable interfacial modifier which was functioning as a hydrophilic–hydrophobic transition layer to reduce water absorption and improve fiber–matrix compatibility in composite manufacturing.

2.5 Characterization

2.5.1 Physical characteristics

The fiber is long, circular, usually tapered to a tip, and has a thicker side on one side. Particularly finer fibers come from the leaf's bottom side. Depending on the method of processing and the amount of time spent extracting the fiber, the colour of the AVLF can range from off-white to yellowish. Due to bacterial action, the treated fiber was darkened, which might be dyed with a naturally seeming light brown tint. The fiber seems robust and long-lasting. All leaf fibers have the stiff, harsh, gritty, and hard-surfaced characteristics of the dry AVLF. However, fibers become pliable, smooth, and slippery when they are wet. The diameter of the AVLF was calculated using a ZEISS (Sigma 360VP, Germany) Microscope. To take their difference in thickness into account along with single units, the dia was estimated for about 10–12 sections at 4–6 different sites of a single AVLF. The density of the AVLFS was evaluated using the conventional pycnometer methods for density measurement. Before being used, the AVLFS model was broken into pieces of 5–6 mm in length and dried out for five days in desiccators made of silica gel. In order to close the microscopic holes in the outer region, the AVLFS were then wrapped up in a container containing toluene for two hours.

2.5.2 Chemical behaviour

Chemical composition analysis is essential to assess the eradication of non-cellulosic components during fiber treatments and predict performance in composite materials. The Conrad technique was used to determine the wax content of *Agave vilmoriniana* leaf fiber, whereas the Kurschner–Hoffner method was used to quantify the cellulose content.

The NFT 12-008 test and the Klason method were used to measure hemicellulose and lignin. Moisture content was measured with a Sartorius MA45 analyser and ash content was measured according to ASTM E175501.

2.5.3 X-ray diffraction analysis

The crystallinity and structural alterations in raw AVLFs as well as in AVLFs following surface modification by alkali, silane and starch coating were investigated by XRD. The Analysis were conducted using a diffractometer (Malvern Panalytical, Empyrean Series III, Netherlands) fitted with Bragg Brentano HD optics and a quick response detector. It provides high resolution diffraction data which facilitates a thorough comparison of the crystalline structure among various surface-modified fibres. The instrument used Cu K α radiation with the wavelength of 1.5406 angstroms then it was powered by 45 kV and 40 mA. Diffraction traces were gathered throughout a 2θ range of 5° to 60°, enabling a thorough characterization of both crystalline and amorphous regions within the fiber structure. The degree of crystallinity was derived using the Segal empirical method. The crystallinity index is given by,

$$CI(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

where I_{002} is intensity of (002) crystalline peak, typically observed near $2\theta \approx 22^\circ$ – 23° , I_{am} is minimum intensity between (002) peak and amorphous background, usually near $2\theta \approx 18^\circ$.

2.5.4 Fourier transform infrared spectroscopy

The chemical structure of AVLFs were examined through FTIR both before and after surface modification through alkali and silane treatment then the starch coating. The spectra was acquired using a FTIR spectrophotometer (SHIMADZU IRSpirit, Japan) attached with a single reflectance ATR measurements were performed using a QATR S accessory equipped with a diamond crystal. Each measurement was made across the spectra were recorded over the range of 4000–400 cm^{-1} with a precision of 4 cm^{-1} . Each sample was averaged over 45 scans to improve signal clarity and reduce noise. The ATR sample region had a diameter of about 1.5 millimetres, which allowed for constant contact between the fibre surface and the ATR crystal. Before proceeding with the analysis, all recorded spectra were baseline adjusted with the built-in program.

2.5.5 Thermogravimetry (TG)

A Thermal Analyser (NETZSCH STA 449 F3 Jupiter, Germany) were used to look at *Agave vilmoriniana* fibres when it exposed thermally both before and after surface modification of AVLFs through alkali and silane treatment then the starch coating. For each thermal study, a fibre sample weighing around 8 to 10 mg was put in an alumina crucible and heated from 26°C to 500°C at a steady rate of 10°C per minute under inert nitrogen environment. This technique made it possible to record changes in weight and heat flow patterns at the same time using Thermogravimetry.

2.5.6 Mechanical behaviour

The tensile behaviour of single fibres were measured by universal testing equipment and adhering to ASTM D3379,

the tensile behaviour of raw and surface modified AVLFs were examined. A polarised light microscope was used to measure the fibre diameters which helps to the accurate stress calculations. The experiments were examined under the temperature of 25°C and relative humidity at 65°C. At least 15 fibers from both untreated and treated AVLFs were examined to make sure the results were statistically valid.

2.5.7 Surface morphology

A ZEISS (Sigma 360VP, Germany) SEM were used to examine the surface microstructure of the raw fibers as well as in AVLFs following surface modification by alkali, silane and starch coating. To improve conductivity and image clarity, raw AVLFs were spray-coated with an incredibly thin layer of gold before imaging. A secondary electron detector was used to record minute surface details during high vacuum inspections carried out at an accelerating voltage of 10 kilovolts. Each treatment produced distinct variations in surface texture, as observed in micrographs recorded at different magnifications. These observations provide visual confirmation of structural modifications and offer insight into potential enhancement of fiber–matrix adhesion in composite systems.

3. Results and Discussions

3.1 Physical characteristics

In terms of effective length, the bundles of AVLFs are longer than those of *Agave sisalana* fibres. The AVLFs are made up of as bundles of multiple sole fibres that are tightly bound together by some waxy and sticky substances. When the fibre is physically extracted from the whole length of the leaf sheaths, the fibres from the central sheaths may vary in length from 0.5 to 1.4 meters. Using a SEM, the fibers thickness were found between 85 and 170 μm as in Figure 1. The density value of AVLFs were determined using the density bottle method at constant temperature which is not above 25° C. It shows values that on an average of 1.32 g/cc.

3.2 Chemical composition

After treatment, the chemical composition of AVLFs transformed a lot. The cellulose content raised notably from 56% in untreated fibres to over 68% in fibres treated with silane and starch, while hemicellulose, lignin, and surface impurities such as waxes and ash were reduced. NaOH treatment successfully eliminated hemi-cellulose and lignin that were increasing fibre crystallinity and density to enhance fiber interlocking behaviour in composites. Silane treatment not only cleaned the AVLF surface but also enhanced fiber–matrix adhesion by introducing siloxane bonds which leads to reduce moisture uptake. Starch coating increased surface hydroxyl groups and fiber density but retained higher wax content that potentially increasing water absorption. These compositional alterations are anticipated to enhance interfacial adhesion and mechanical performance in composites, however moisture behaviour may differ. Silane-treated fibres exhibited the most efficient proportion of adhesion and hydrophobicity. A comprehensive analysis of the chemical composition among treatments is shown in the accompanying Table 1.

3.3 X-ray diffraction analysis

The XRD analysis patterns of the untreated and treated AVL F reveal the characteristic cellulose I structure, with a prominent (002) reflection at $2\theta \approx 22\text{--}23^\circ$ and a broad amorphous region around $18\text{--}19^\circ$. Untreated fibers carry more amorphous hemicellulose and lignin because it has lower (002) peak intensity. NaOH treatment raises the (002) peak and decreases the amorphous background, removing non-cellulosic components and aligning cellulose microfibrils. Crystallinity rises from 55% to 63%. Additional silane treatment increases crystallinity by 64%, revealing the cellulose structure is stable without affecting its core

crystalline phase. Fibers reach peak crystallinity ($\sim 66\%$) when coated with cassava starch after NaOH and silane treatment. This indicates that starch deposition does not damage the cellulose lattice and may even help order the surface fibers. The modest rise noted in silane-treated fibers is due to partial recrystallization that happens when starch retrogrades. The crystalline structure of cellulose I stays unaltered. These observations suggest that additional treatments enhance cellulose organization, aligning with the noted thermal stability and mechanical performance. The comparative XRD profiles of untreated and treated AVL F's are presented in Figure 2 for detailed analysis.

Table 1. Chemical composition of untreated, treated and starch coated AVL F's in wt. %.

Fiber Sample	Cellulose	Hemicellulose	Lignin	Ash & SiO ₂	Wax & Others	Moisture
Untreated	56.26 $\pm$ 1.3	18.32 $\pm$ 1.1	11.02 $\pm$ 0.9	3.12 $\pm$ 0.5	3.87 $\pm$ 0.4	8.56 $\pm$ 0.6
Alkali Treated	67.10 $\pm$ 0.8	10.20 $\pm$ 0.7	9.05 $\pm$ 0.6	2.45 $\pm$ 0.3	2.18 $\pm$ 0.3	5.25 $\pm$ 0.4
Silane Treated	68.25 $\pm$ 0.6	9.95 $\pm$ 0.5	7.75 $\pm$ 0.4	2.64 $\pm$ 0.2	1.85 $\pm$ 0.2	4.23 $\pm$ 0.3
Starch Coated	68.32 $\pm$ 0.7	9.82 $\pm$ 0.6	7.16 $\pm$ 0.5	2.76 $\pm$ 0.2	2.25 $\pm$ 0.3	5.10 $\pm$ 0.4

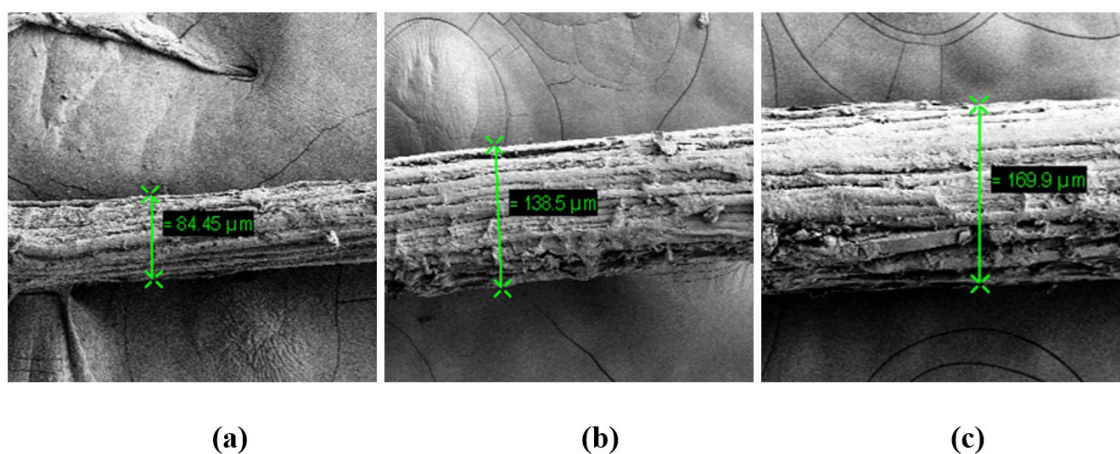


Figure 1. a), b), c) Different AVL Fiber's thickness.

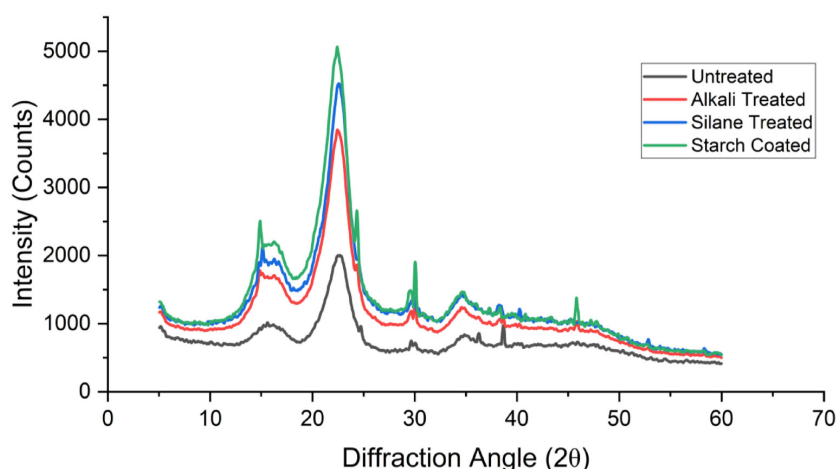


Figure 2. Comparison of XRD of untreated, treated and starch coated AVL F's.

3.4 Fourier transform infrared spectroscopy

The FTIR spectra of AVLFs before and after surface modification and coating show clear changes in crystalline structure. The treated fibers have a stronger broad absorption band around 3300 cm^{-1} that corresponds to O–H stretching. This means that the cellulose hydroxyl groups are more exposed in the treated fibers. The band near 2920 cm^{-1} that is linked to C–H stretching is in all of the samples. In untreated fibers, a clear peak at 1730 cm^{-1} shows hemicellulose and waxy ester parts. The evidence that this peak gets limited after alkali and silane treatments shows that non-cellulosic parts have been removed. The absorption bands at 1610 , 1235 , and 1020 cm^{-1} are due to the stretching vibrations of lignin and the C–O bonds in cellulose and hemicellulose. An additional sharp peak around 750 cm^{-1} appears in silane and starch treated fibers, suggesting the formation of new chemical linkages such as Si–O bonds or starch related functional groups. These spectral changes confirm effective surface modification and indicate enhanced fiber purity and functionalization. A comparative representation of the FTIR spectra is shown in Figure 3.

3.5 Thermogravimetry

This analysis of the untreated and treated *Agave vilmoriniana* fibers shows notable improvements in thermal stability after chemical modifications. The thermal degradation behavior of untreated and chemically treated AVLf was analyzed using TG and derivative TG (DTG). The onset degradation temperature (Tonset) was determined as an extrapolated value from TG curves using tangent intersection method at the main decomposition region. Accordingly, the Tonset of untreated fiber is around $285\text{ }^{\circ}\text{C}$, the NaOH treated fiber and the NaOH with silane treated fibers showed a Tonset of about 295 and $300\text{ }^{\circ}\text{C}$, while treated fiber with starch coated fibers exhibited a Tonset of around $300\text{ }^{\circ}\text{C}$.

The increase in Tonset for treated fibers suggests improved thermal stability and reduced moisture-related mass loss, indirectly indicating enhanced fiber surface modification. On otherhand, the maximum degradation temperature (Tmax) was directly obtained from the peak positions of the DTG curves, corresponding to the highest rate of mass loss. The DTG result shows Tmax values of approximately $340\text{ }^{\circ}\text{C}$ for untreated fibers, $350\text{ }^{\circ}\text{C}$ for NaOH treated-fibers and NaOH with silane treated fibers, $355\text{ }^{\circ}\text{C}$ for treated and starch coated fibers. This changes confirms that the chemically treated and coated fibers were enhanced thermal stability resulting from the removal of amorphous components and improved interfacial structure, consistent with enhanced fiber–matrix bonding rather than direct hydrophobicity assessment. A comparative analysis of thermal behaviour is clearly depicted in Figure 4.

3.6 Mechanical behaviour

The diameters and tensile properties such as tensile strength, Young's modulus, and the elongation at break in raw fibers and of AVLFs following surface modification by alkali, silane and starch coating are summarized in Table 2. That the result shows a clear reduction in fiber diameter after chemical treatment, with six percent sodium hydroxide proving to be the most effective concentration among those tested. The observed reduction in fiber diameter confirms the effective removal of non cellulosic constituents such as hemicellulose and surface impurities. For mechanical characterization, five specimens from each group were tested to ensure reliable averaging of the measured properties. The treated samples have higher tensile strength and greater elongation at break than the untreated fibers. The removal of amorphous phases has made the fibers more compact and better aligned on the level of molecular structure, which has led to these improvements. This kind of structural refinement makes it easier for loads to transfer and improves the tensile mechanical performance.

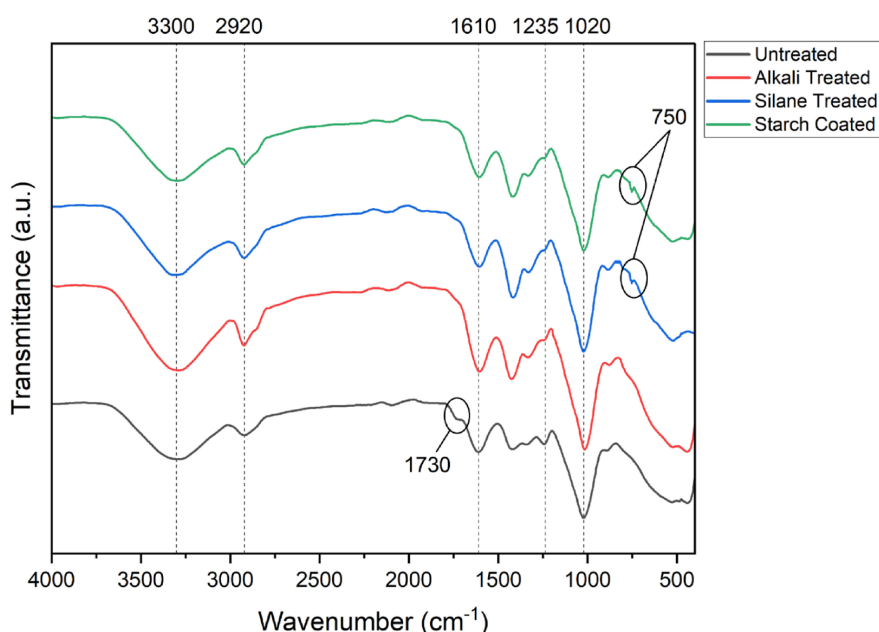


Figure 3. Comparison of FTIR analysis of untreated, treated and starch coated AVLf's.

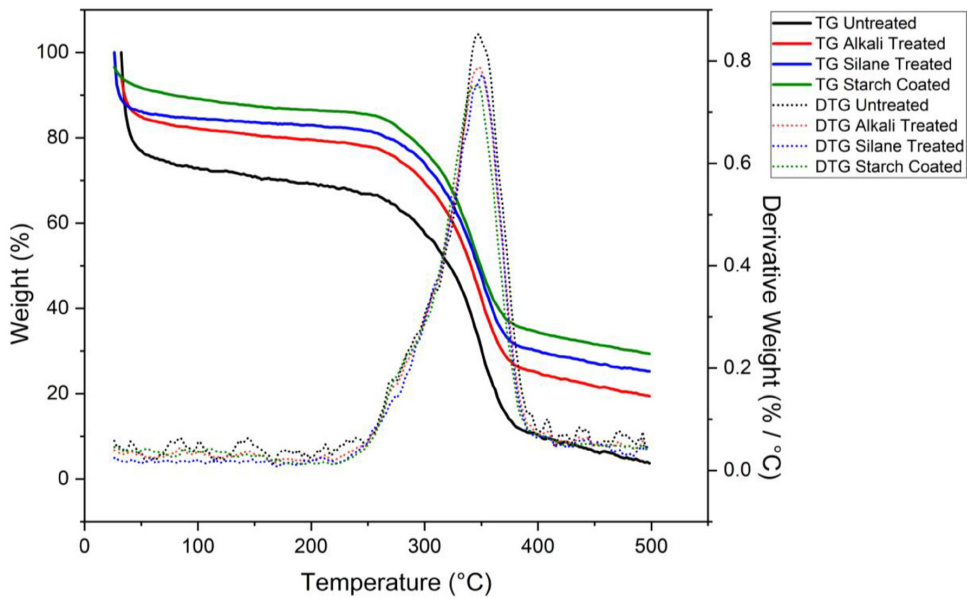


Figure 4. Comparison of TG and DTG of untreated, treated and starch coated AVLf's.

Table 2. Comparison of the tensile properties of untreated, treated and starch coated AVLf's.

Fiber Sample	Diameter (μm)	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation at Break (%)
Untreated	161 ± 16	358 ± 24	18.12 ± 1.1	3.17 ± 0.4
Alkali Treated	158 ± 12	456 ± 22	20.23 ± 0.9	3.81 ± 0.3
Silane Treated	160 ± 10	468 ± 20	21.56 ± 0.8	3.85 ± 0.2
Starch Coated	162 ± 11	471 ± 21	23.42 ± 0.9	4.12 ± 0.3

3.7 Scanning Electron Microscopy analysis

The SEM analysis of *Agave vilmoriniana* fibers shows that changes to the surface cause distinct modifications in the fiber. In the untreated fiber (Figure 5a), a thick layer of waxes, ash, and non-cellulosic materials masks the microfibrillar structure. This shows that the poor surface roughness and limited interfacial bonding potential in composites. After the alkali treatment (Figure 5b), some wax and other impurities are still there, but the surface is cleaner. This treatment breaks up the amorphous hemicellulose matrix, making the fibrillar cellulose more visible. Silane-treated fibers (Figure 5c) have surfaces that are much rougher and have visible cellulose microfibrils. This is because the silane coupling agent interacts with hydroxyl groups, which creates covalent bonding sites that help the matrix bind better. Starch-coated fibers (Figure 5d) have smoother, more continuous surfaces with fewer voids and microfibrils that are well-aligned. This means that the coating looks uniform and there are fewer surface defects which were drastic changes while comparing with the raw fiber (Figure 5a). The starch film probably makes things more compatible by forming hydrogen bonds with polar matrices.

4. Conclusions

The comprehensive study of *Agave vilmoriniana* fibers shows that certain chemical modifications have a significant

positive effect on their structural, thermal and mechanical characteristics, based on their applicability as eco-friendly reinforcing agents in polymer composites. The alkali treatment enhances cellulose, crystallinity, tensile strength, modulus, and thermal stability by removing the non-cellulosic materials, such as hemicellulose and lignin. Silane treatment is used to promote the development of covalent siloxane bonding, promote bonding of the fibers and the matrix have increase the composite durability and reduce the moisture affinity. The coating of starch increases the coverage and interfacial compatibility. Presumably, because of the limited starch retrogradation their effect on the total thermal and structural properties is not significant in the current conditions of processing. FTIR Spectroscopy and XRD postulates that the surface treatments lead to changes in the chemical and crystalline structure. The thermogravimetry has shown that the treated fibers are more stable at higher temperatures with the alkali treatment showing significant enhancement. SEM explains that the process gradually removes the surface contaminants and improve the surface roughness. This makes the composite matrix mechanically interlock with the surface. The physicochemical properties of *Agave vilmoriniana* fibers are increased by the treatment induced changes, which confirms its feasibility as a sustainable reinforcement material. It has been found that surface-engineered plant-based fibers can be effectively used as reinforcements in bio-composites that would require improved mechanical performance, durability, and thermal stability where used in engineering.

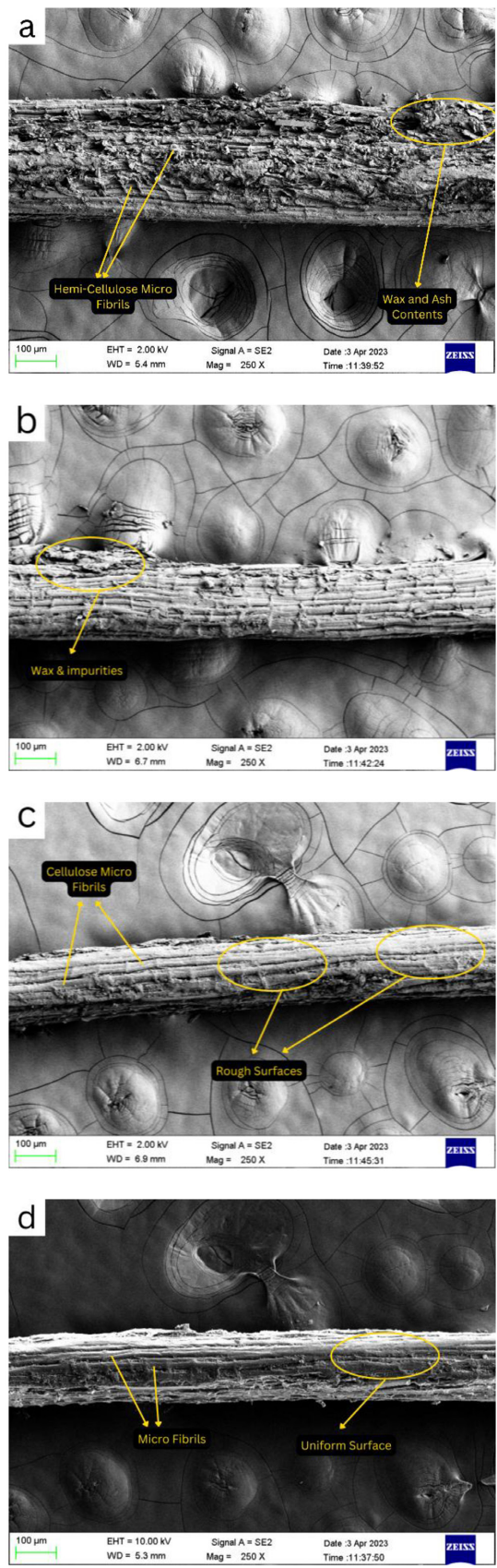


Figure 5. SEM image of a) untreated AVLF, b) NaOH treated AVLF, c) Silane treated AVLF and d) starch coated AVLF.

5. Author's Contribution

- **Conceptualization** – Gowtham Saravanan
- **Data curation** – Gowtham Saravanan
- **Formal analysis** – Gowtham Saravanan
- **Funding acquisition** – NA
- **Investigation** – Gowtham Saravanan
- **Methodology** – Gowtham Saravanan; Ramakrishnan Thirumalaisamy
- **Project administration** – Gowtham Saravanan
- **Resources** – Gowtham Saravanan; Ramakrishnan Thirumalaisamy
- **Software** – Gowtham Saravanan
- **Supervision** – Ramakrishnan Thirumalaisamy
- **Validation** – Ramakrishnan Thirumalaisamy
- **Visualization** – Gowtham Saravanan; Ramakrishnan Thirumalaisamy
- **Writing – original draft** – Gowtham Saravanan
- **Writing – review & editing** – Gowtham Saravanan; Ramakrishnan Thirumalaisamy

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