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Enhancing structural, thermal, and mechanical properties of Acacia pennata natural fibers through benzoyl chloride treatment for construction applications

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ABSTRACT

In recent years, there has been growing interest in exploring natural fiber reinforced composites as potential alternatives to conventional materials in various structural applications. The aim of this study on Acacia pennata fibers (APFs) and treating them with benzoyl chloride was to explore their potential as reinforcement in construction-related materials. The aim was to investigate the physico-chemical, thermal, and mechanical properties of these fibers to understand their suitability for applications in concrete reinforcement, retrofitting, roofing, and wall panels. By enhancing the understanding of the treated fibers' characteristics, this study contributes to the development of sustainable and high-performance construction materials. The fibers were extracted using both water retting and chemical retting methods. The physico-chemical properties of the fibers were assessed through X-ray diffraction (XRD) analysis, which determined a calculated crystalline index (CI) of 72.14% and a crystalline size of 2.6 nm. Thermo-gravimetric analysis was conducted to evaluate the thermal stability of the APFs, revealing a temperature of 366°C and a maximum degradation temperature of 226.7°C. Mechanical analysis included measurements of the APFs' tensile strength (467.86 MPa), tensile modulus (14.62 GPa), microfibrillar angle (14.79), and elongation at break (3.2%). The findings derived from these analyses suggest that the APFs that underwent treatment exhibit desirable mechanical characteristics, rendering them a viable option for utilization in construction-related materials like reinforcement in concrete, retrofitting, roofing and wall Pannels. This research presents a novel exploration of Acacia pennata fibers (APFs) treated with benzoyl chloride, aiming to establish their potential as reinforcements for construction materials. While natural fiber-reinforced composites have drawn interest, the unique application of APFs in construction and their treatment with benzoyl chloride to enhance properties remain relatively unexplored in the literature. This study fills a significant

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gap and contributes to the advancement of sustainable and high-performance construction materials by utilizing treated APFs. The results highlight the remarkable capability of these fibers to enhance the properties of composite materials in the construction industry.

1. Introduction

Today, experts from all around the world are focused on improving the sustainability and quality of eco-friendly products in order to conserve the environment and biodiversity. There has been a steady increase in public awareness of new items generated from renewable resources in recent years. Green marketing introduces new guidelines for recycling, modifies consumer values, and has a positive social impact on purchasing eco-friendly goods [1]. People are switching back to natural fibres in favour of synthetic and hazardous materials because of their bio-renewable qualities and eco-friendly practises [2]. Natural fibres can be found all over the world and are widely available because they grow naturally in the environment. They are numerous, widely available, need little to no energy to create, may be recovered from waste material, and can lessen environmental effect when employed in the building industry [3]. Fibres have long been employed in construction materials around the world. Furthermore, composite materials have a long history of using fibres as reinforcement to improve the properties of the materials [4]. Fibres can lower overall construction costs since they can be employed as reinforcement, substituting the traditional and energy-intensive methods of wiring mesh and steel reinforcement bars [5]. It is also possible to incorporate waste fibres into building materials. This would help the sector adopt sustainable working methods and reduce the quantity of garbage going to landfills. It would also save energy [6].

Natural fibres have a number of benefits, including the potential to combine strength and high specific stiffness, a suitable fibre aspect ratio, and convenience of obtaining them from natural sources [7]. The cellulosic century, in which a large number of plant resources for products have been identified, is the one we are currently living in. Primary fibres and secondary fibres are the two categories into which natural fibre plants are typically divided. Because of their low density, cost, and biodegradability, plant fibers are used for research purpose [8–10]. For more than 20 years, synthetic fibers including polyester, nylon, carbon, and glass have been employed extensively for a variety of purposes. They do, however, have certain negative aspects, notably with regard to the environment. Due to their extreme flammability and lack of biodegradability, synthetic materials raise questions about how they may affect ecosystems and how to manage trash. Recently, the use of natural fibres in place of synthetic fibres has gained popularity as a study issue [11].

Natural fibers provide a number of benefits that may help with these problems. First of all, natural fibers naturally degrade over time without producing long-term environmental impact since they are innately biodegradable. They are a more environmentally friendly choice than synthetic fibers, which are known to linger in the environment for long periods of time. Natural fibres are recyclable and friendly to the environment. As a result, at this point in technological development, they are widely used in our daily lives. They are raw material sources for clothing, textiles, and novel biomaterials due to their acceptable tensile strength, extensibility, toughness, chemical stability, electrical insulation properties, and biocompatibility [12–17]. Primary-fiber plants are typically cultivated for the specific purpose of obtaining fiber content, while secondary-fiber plants generate fiber as a by-product of their primary function. [18]. Researchers' focus has now turned in to the exciting and promising field of new natural fibres as environmental awareness has increased globally [19]. This is primarily because they are inexpensive, lightweight, accessible, biodegradable, noncorrosive, and environmentally friendly [20]. Synthetic fibres (like carbon, nylon, glass and polyester) have been used in applications for about more than 20 years. However, there are a number of drawbacks to these synthetic fibres, especially in terms of the environment [21–23]. Synthetic materials are very easily flammable and these fibers were can't break down biologically. The ability of synthetic fibres to absorb moisture and perspiration is inferior to that of natural fibres. Because they are not biodegradable and are produced using non-renewable resources, synthetic fibres are not sustainable. They don't naturally decompose in the environment. Pollution and damage to wildlife may result from this. Natural fibres are more durable than synthetic fibres. Many synthetic fibres are difficult to recycle and may wind up in landfills, increasing waste and pollution. But when compared to synthetic fibres, natural fibres have several advantages for the environment (such low impact farming, biodegradability, less water pollution, durability, and lifespan).

These problems can be resolved with natural fibers because of its low economic cost and degrading capacity [24]. The objective of numerous studies was to successfully replace synthetic fibres in structural materials with biodegradable natural fibres [25,26]. In current times, variety of applications, such as automobiles, industrial, and home appliances, use natural fibres [27,28]. Due to the increasing demand for natural fibers, it is necessary to look for natural fibre varieties with useful properties for reinforcement in making composites[29,30]. Thus, this study aims to investigate the characteristics of benzoyl chloride-treated Acacia pennata fibers through a comprehensive analysis involving XRD, FTIR, SEM, TGA, and mechanical characterization. By exploring the unique characteristics of Acacia pennata fibers and examining the effects of benzoyl chloride treatment, this research contributes to expanding the knowledge base and provides insights into the potential applications of these fibers in the field of construction. Acacia pennata fibers offer various possibilities in construction applications, it has an excellent mechanical properties and strength. One of the most prominent uses of these fibers is in concrete reinforcement, where they can enhance the strength of concrete and mortar used in constructing buildings, retrofitting and other infrastructural work [31–33]. Additionally, the fibers can be employed as a component of composite materials to create lightweight yet strong materials for use in wall panels, roofing tiles, and other building materials [34,35]. Acacia pennata fibers are also useful in soil stabilization, where they can be mixed with weak or unstable soil to improve its strength and stability, and in road construction, where they can be added to asphalt mixtures to increase their strength and durability.

Furthermore, these fibers can reinforce timber structures like beams, reduce cracking and water penetration in masonry walls, and serve as a component in geotextiles used in civil engineering applications for soil stabilization, erosion control, and drainage. There is a vast potential for Acacia pennata fibers in construction, and further research and development can explore innovative ways to utilize them in the field of construction. The hydrophilic nature of natural fibers absorbs water molecules, which causes the material's strength and thermal stability to degrade [36]. The ability of fibres to absorb water is decreased by chemical processing, and the surface qualities are improved, improving the compatibility of the fibres bonding capacity [37]. In fact, many researchers use benzoylation treatment and alkali treatment [38,39]. One chemical treatment procedure, known as benzoylation, was utilised to increase the interlocking between the fibre and bond strength while decreasing the hydrophilicity of the fibre, boosting the strength and thermal stability of the composite [40]. The hydroxyl group is activated using pre-treatment (NaOH) prior to the benzoylation process, which involves soaking the fibre in a solution of benzoyl chloride. Sisal fibres treated with benzoyl chloride have better adherence to the manufacturing of composites. Additionally, it greatly lowers the water absorption characteristics and improves thermal stability [41]. The sugar palm fibres treated with NaOH and benzoyl chloride did not increase the thermal stability, according to the thermal study done on the sugar palm fibres when compared to untreated sugar palm fibres [42].

This study marks the first utilization of Acacia pennata fibers for benzoyl chloride treatment, highlighting its pioneering nature in exploring the potential of these Fibers. This study stands out due to the novel selection of Acacia pennata fibers, driven by their exceptional strength and availability. This experiment holds significant importance as it involves the chemical treatment of alkaline pre-treated Acacia pennata fibers, resulting in enhanced thermal stability compared to untreated fibers. Building upon this approach, the present study aims to thoroughly investigate the characteristics of Acacia pennata fibers treated with benzoyl chloride. The study employs various analytical techniques, including X-ray diffraction, Fourier transform infrared spectroscopy, scanning electron microscopy (SEM) characterization, thermo-gravimetric analysis (TGA), and mechanical characterization, to provide comprehensive insights into the properties of benzoyl chloride-treated Acacia pennata fibers. There was no previous research has been carried out to the best of our ability on the physicochemical characteristics of bark fibers obtained from the Acacia pennata (AP) plant. This paper aims to describe the extraction process, determine APFs' structural characterization using XRD, FTIR, TGA, tensile analysis, and SEM surface roughness, and compare the results to other natural fibers. In this way, one could access the possibility of using the fiber in future construction applications, either as an individual fiber or as a supplement to other fibers that already exist.

1.1. Scope and objective

In this study, Acacia pennata fibers were used to study the characteristics with the help of benzoyl chloride treatment. Through treating this fiber, the scope of this research intends to accomplish the following:

- Investigate the physical and structural properties utilizing powder X-ray diffraction, Fourier transform infrared spectroscopy, and CHNS analysis.
- Using scanning electron microscopy, investigate the morphological alterations and surface characteristics of the AP fibres.
- Using TG and DTG experiments, examine mass changes and degradation at various temperatures.
- The endothermic and exothermic reactions are evaluated using differential scanning calorimetry, and the tensile strength and tensile modulus data are compared to other natural fibres.
- To investigate the quality of the AP fiber's structure which has a strong effect on the mechanical properties.

1.2. Acacia Pennata fibre in Civil Application

Acacia pennata fibers, with their desirable properties and characteristics, can be effectively utilized in various structural and retrofitting applications. Some potential applications include:

Reinforcement in Concrete: Acacia pennata fibers can be incorporated into concrete mixtures to enhance its mechanical properties, such as tensile strength and toughness. These fibers act as reinforcement, providing crack control and improving the overall durability of the concrete structures.

Fiber-Reinforced Polymer (FRP) Composites: Acacia pennata fibers can be used as reinforcements in FRP composites. These composites have high strength-to-weight ratios and can be used for structural strengthening, including the rehabilitation of bridges, columns, and beams[43,44]. The fibers can be embedded in a polymer matrix to form FRP sheets or bars that are applied externally or internally to enhance the load-carrying capacity of structural elements.

Retrofitting of Masonry Structures: Acacia pennata fibers can be used to strengthen and retrofit masonry structures, such as historic buildings or walls. By impregnating the fibers into the mortar joints or applying fiber-reinforced polymer composites on the surface, the flexural and shear strength of masonry elements can be significantly improved.

Geotechnical Engineering: Acacia pennata fibers can be utilized in geotechnical engineering applications, such as soil stabilization and reinforcement. When mixed with soil, the fibers enhance its mechanical properties, including shear strength and resistance to erosion. This can be particularly beneficial in slope stabilization, embankment construction, and erosion control projects.

Roofing and Wall Panels: Acacia pennata fibers can be incorporated into the production of roofing and wall panels to provide additional strength and impact resistance. These panels can be used in construction projects, offering lightweight and sustainable alternatives to traditional materials.

Composite Timber Products: Acacia pennata fibers can be combined with other materials, such as recycled plastic or wood particles, to produce composite timber products. These products offer improved strength, durability, and resistance to environmental

factors, making them suitable for applications such as decking, cladding, and structural components.

The utilization of Acacia pennata fibers in structural and retrofitting applications not only contributes to the development of sustainable and eco-friendly construction practices but also enhances the performance and longevity of various structures.

2. Materials and methods

2.1. Materials

The fibre was treated using distilled water, sodium hydroxide pellets (NaOH), and a benzoyl chloride solution. It was purchased from Premier Chemicals in Nagercoil, Tamilnadu, India. Acacia pennata (AP) is a gregarious plant that can reach a height of 100 m and has spreading branches that resemble flat, umbrellas. The Kerala border, which is close to the village of Vellarada in Kerala, India, is where the mature and healthy barks of AP were gathered. Fig. 2 represents the images of bark of the Acacia pennata plant and their respective fiber treatments.

2.2. Extraction methods

Acacia pennata fibers (APFs) were separated using a water retting and chemical retting processes. For the purpose of extracting fibre, AP barks were immersed in water for up to two weeks. The fibre was then exposed to a 0.1 M (ie., 0.1 molar concentration) alkali (NaOH) solution for roughly 30 min. Fibers that have been soaked are removed and left to dry.

- In the synthesis of organic compounds, benzoylation is a crucial transition [45]. Benzoyl chloride is most frequently used to treat fibres. Because of the presence of benzoyl (C6H5C═O) in benzoyl chloride, the treated fibre has less hydrophilicity and interacts more favourably with the hydrophobicity.
- By removing extra starch, cellulose, hemicellulose, lignin, etc., the benzoylation reduces surface roughness. This phenomenon makes the fibre more wettable, which leads to a strong connection with the fibre and boosts strength.

The dried fibres were submerged in a 100 ml of benzoyl chloride solution for about 20 min, followed by an hour-long ethanol wash. The APFs fibres were extracted with the help of extremely fine, metal teeth, cleaned with distilled water, and left to dry at room temperature around four days. Acacia pennata plant, untreated, and benzoyl chloride-treated AP fibres are depicted in Fig. 1(e) & (f).

2.3. X-ray diffraction analysis

Using a Bruker x-ray diffractometer (Bruker AXS-KAPPA APEX II CCD) system with monochromatic intensity of CuK radiation and wavelength of 0.154 nm recorded from 10° to 80°, the XRD analysis of APFs was investigated. The following expression (1) was used to

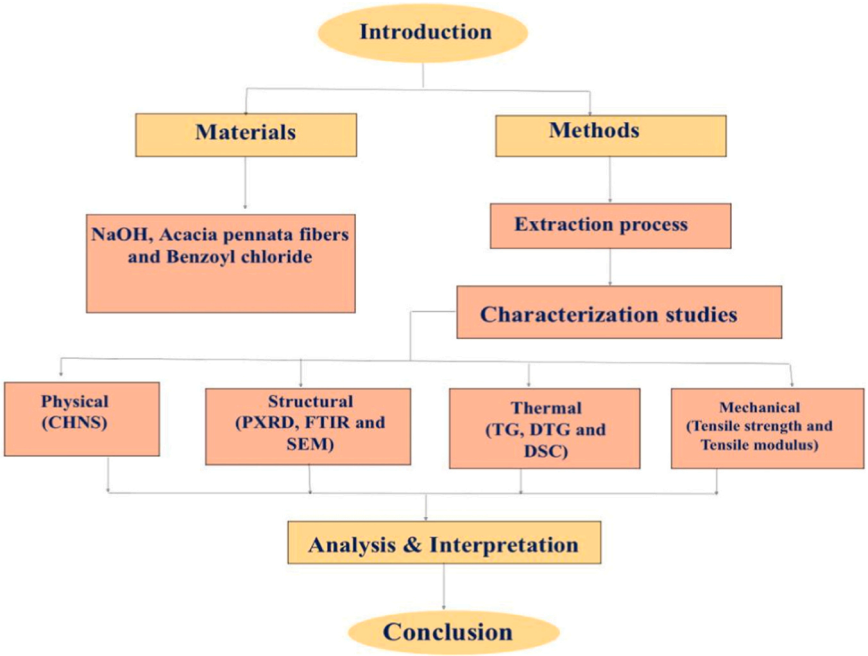

Fig. 1. Research flow chart.

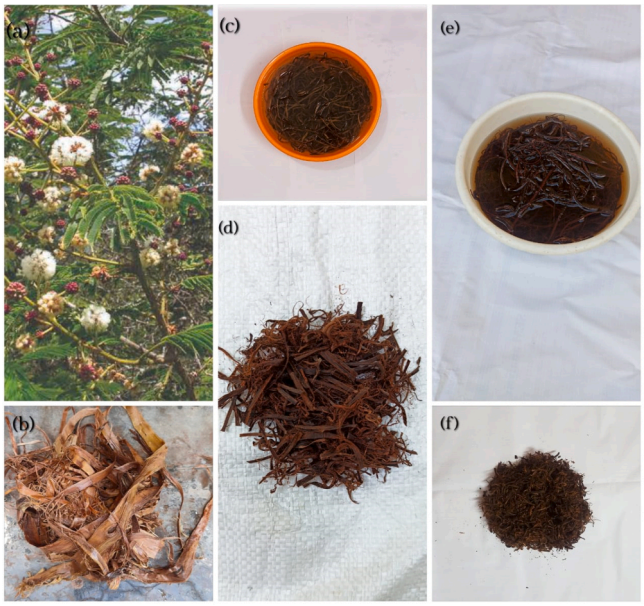

Fig. 2. Images of (a) Acacia pennata plant (AP), (b) AP fiber, (c) untreated AP fibers, (d) dried untreated AP fibers, (e) benzoyl treated AP fibers, (f) dried benzoyl treated AP fibers.

calculate the crystallinity index (CI).

$$CI = [(H_{Cr} - H_{am})/H_{Cr}] *100 \quad (1)$$

where; H_{Cr} and H_{am} are the heights of the peaks at $2\theta = 22.92^{\circ}$, 15.3°. According to Scherrer's formula (2), the diffraction pattern perpendicular to the lattice plane was used here, study to estimate the crystalline size (CS) of the AP fibers [46].

$$CS = K\lambda/\beta\cos\theta \quad (2)$$

where (K=0.89 is Scherrer's constant), β represents the full-width at half maximum value, and λ represents the wavelength.

2.4. FT-IR Spectrum Analysis

A general term for the process of breaking down any varying signal into its individual frequency components is "fourier spectroscopy." FTIR spectrometer Perkin Elmer was used to record the FT-IR spectrum of Acacia pennata natural fibers. The experiment was carried out on potassium bromide (KBr) pellets with a powdered fibre sample. The FTIR spectrum was recorded between the range of 4000 and 400 cm-1. An additional 32 scans were added to achieve an acceptable signal-to-noise ratio with a resolution of 2 cm-1.

2.5. SEM analysis

The morphology and cross-section of AP fibres were examined under the microscope using the SEM technique to study their microstructure. SEM analysis is used to analyse the surface roughness of a natural fibre. The necessary microscopic images of the Benzoyl-treated and untreated APFs were obtained using a JEOL JSM-6390LV scanning electron microscope. The electron source was heated tungsten filament.

2.6. EDX analysis

To ascertain the elemental composition of materials, an x-ray technique called Energy Dispersive X-Ray Analysis (EDX), also known as EDS or EDAX, is used [47]. The amount of elements (such as carbon, oxygen, nitrogen, and others) that are present on the fiber surface can be determined using this analytical technique (EDX). The distributions of the elements in the untreated and benzoyl-treated APFs were measured five times using the EDX (INCAPentaFETx3) attached to the scanning electron microscope, and the average values were recorded.

2.7. CHNS instrumentation

The type of combustion elemental analyzer (Elementar Vario EL) to use will be determined by the element type, sample size, and concentration of the analysis. Combustion elemental analyzers are manufactured in a variety of configurations to meet the needs of

various applications. All instruments need two types of gas: high purity oxygen (minimum 99.9995%) and an inert carrier gas (helium is recommended). The need to reduce the nitrogen "blank" contribution to a negligible level is related to the strict oxygen specification. The combustion section of the analyzer is designed to achieve both complete sample combustion and the conversion of nitrogen oxides to nitrogen gas (N2). The "static" system is preferred for slow-burning materials such as coals and cokes that require multiple oxygen additions for complete combustion. Depending on the combustion mode and sample size, the analyser's detection system can take on a variety of shapes. A variety of absorbents are used to remove these additional combustion products in addition to some of the principal elements, such as sulphur, if the determination of these additional elements is not necessary [48].

2.8. Thermogravimetric analysis

In recent years, TGA has become increasingly popular, particularly in the polymer processing industry, for failure analysis of finished parts as well as quality control and assurance of raw materials and incoming goods. To assess the degradation characteristics of APFs in a nitrogen atmosphere at a flow rate of 20 ml/min, TGA was carried out using an HITACHI-STA7300 thermal analyser. All measurements were made using a platinum crucible and a programmed temperature range of 30–500 °C with a heating rate 10 °C/min.

2.9. Mechanical analysis

The Zwick Roell instrument is used to test the single fiber's tensile properties, including strength, tensile modulus, and breaking elongation. This (Zwick Roel) universal testing device (ASTM D-3822) was used to test untreated, benzoyl-treated APFs (50 mm gauge length), and the results were recorded for interpretation analysis[49]. The crossing rate used for all tests was 0.1 mm/min. The Eq. (3) was used to calculate the microfibrile angles of the untreated and benzoyl-treated APFs [50].

$$\varepsilon = \ln(1+\Delta L/L_o) = -\ln(\cos\alpha) \quad (3)$$

where L_o is the gauge length (mm), L is the elongation at break, α- is the microfibrillar angle in degrees, and - is the overall deformation or strain (mm). The results of tensile tests on plant fibres are frequently inconsistent and depend on the bark of the plant from which the fibre was extracted, the method used to extract the fibre, the testing environment, variations in the diameter, and the presence of surface flaws. It is crucial to conduct analysis to determine the mean values of the tensile characteristics [51]. Weibull distribution was used by many researchers to examine the tensile characteristics of cellulosic fibres.

3. Results and discussion

3.1. XRD analysis

Fig. 3 shows two visible peaks at 15.3° (1 1 0) and 22.92° (0 0 2), which are typical plant fibre peaks. Indicating that the benzoyl chloride solution penetrated the fibre and dissolved the low molecular weight materials like hemicelluloses, wax, and lignin from the fiber's surface, revealing the cellulose, the peak (22.92°) of benzoyl treated APFs was magnified more than the peaks of untreated APFs. The raw and benzoyl-treated APFs' crystallinity index (CI) values, which are 46.52% and 72.14%, respectively, are higher than

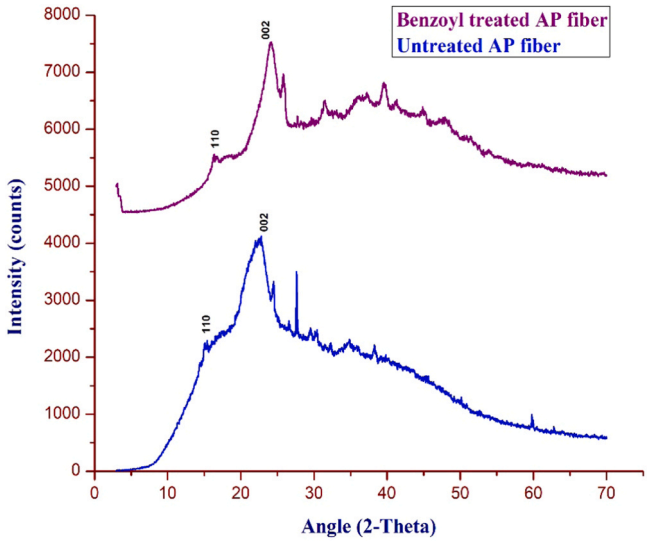

Fig. 3. X-ray diffraction pattern of untreated and benzoyl treated AP fiber.

those of Cordia dichotoma (50.20%) and Grewia tilifolia (41.7%) and lower than those of Ferula communis (68%) and Althaea officinalis L (68%) [52,53]. Untreated and benzoyl-treated APFs have crystallite size (CS) of 1.9 nm and 2.6 nm, respectively, which are smaller than Prosopis juliflora (15 nm) and larger than Ferula communis's (31.55 nm) respective sizes (1.6 nm) [54,55]. Based on this, the Crystallinity Index (CI) measurement may serve as a key indicator of the workability (strength and elasticity) of a fiber. Additionally, the amorphous portions offer water accessible volume, increasing the hygroscopicity of the fiber [56].

CI and CS values were tabulated (Table 1) below.

3.2. FTIR analysis

Fig. 4 provides an explanation of the FTIR spectram of untreated and benzoyl-treated APFs (a). Table 2 lists the peak positions and corresponding chemical functional group assignments for the untreated and benzoyl-treated APFs. Raw APFs have distinct peaks, and those at 3914.58 cm^{-1}, 3787.33 cm^{-1}, and 3437.02 cm^{-1}, respectively, are linked to OH stretching of hydrogen bonds [57]. The stretching of cellulose by CH is indicated by the following peak, which is located around 2923.1 cm^{-1}. A small peak at 2109.53 cm^{-1} and 1631.28 cm^{-1} is attributed to lignin's CC stretching. The CH bending is represented by the absorption peak around 1383.68 cm^{-1}. The CO stretching in lignin was associated with the peak at 1271.83 cm^{-1}. A noticeable peak at 776.35 cm^{-1} and 618.17 cm^{-1} is out of the OH bending plane and inclined to the saline content. In contrast, the peaks at 3431.91 cm^{-1} in the case of benzoyl-treated APFs were caused by the OH stretching in the hydrogen bond. The strongest and most pronounced peak is this one. The peak at 2923.81 cm^{-1} and 2853.6 cm^{-1} is due to cellulose and hemicellulose components stretching under the influence of CH and CH_2 [58]. The CC stretching vibration causes the absorption peak at 2134.98 cm^{-1}. The medium and sharp peak at 1643 cm^{-1} contained carbonyl groups of CO that were stretched. The CC group of lignin are responsible for the most noticeable peak, which is located at 1512 cm^{-1} [59]. In 1383.31 cm^{-1} and 1338.07 cm^{-1}, respectively, the vibrations of CH bending and CO stretching are noted. Due to the stretching of COH, around the peak at 1060.91 cm^{-1} was attributed to the presence of lignin[60,61]. The CO bonds in cellulose are what cause the peak to be seen at 1035.12. Finally, the saline content is responsible for the peak at 780.31.

3.3. SEM analysis

SEM is a great method for examining the surface morphology of Acacia pennata fibres that have been benzoyl treated and those that have not. To investigate the morphological alterations that happened after the treatment of the fibres, untreated APFs were looked at. Fig. 5 represents the SEM micrographs of untreated and benzoyl treated APFs, which is rougher and uneven.

According to this morphology, untreated APFs have helical fibrils, micro-voids, and hemicellulose content visible on their surface. It also reveals the presence of wax, pectin, lignin, and impurities on the surface of untreated APFs. Fig. 5a shows that untreated fibre has an extremely smooth surface with the presence of impurities (white spots in image), because amorphous materials are present. The AP fiber's surface roughness may be a sign of the presence of lignin [62]. After benzoylation, impurities (lignin, wax, pectin, and hemicellulose) were removed from the APF's surface [63]. This was also reported by Sherwani et al., (2022) in their investigation. The removal of pectin, lignin, oil, and other surface impurities during treatment, however, increased the roughness of the surface.

The surfaces of the benzoyl-treated APFs appears to be quite rough and due to the benzoyl chloride. The APF that has been benzoyl-treated improves the interface's bonding to resins. Because the primary walls of the untreated APFs were disintegrated away during the chemical treatments. On the fibre surface, a significant number of pinholes (crater-like structures) and a rough surface are readily apparent. This is the result of the amorphous components surrounding the fibre surface being removed. Again, during the benzoylation process, benzoyl groups replaced OH groups, improving the hydrophobic property of the fibre, as evidenced by literature from Nayak et al., (2019) [64]. Figure shows that wax, lignin, moisture, and hemicellulose contents were reduced in AP fibres treated with benzoyl chloride. This is caused by fibre separation from the bundle, which eliminates the aforementioned components and increases α–cellulose [65]. The contaminants on the surface of APF had been somewhat eliminated by the benzoylation process. The surface of the fibre was made rougher by the removal of lignin, wax, pectin, and hemicellulose, which improved the bonding with the fibre in composites [66]. SEM micrographs allowed researchers to observe the subtle structural changes in APFs.

3.4. EDX analysis

The distribution of compounds on the surface of untreated and benzoyl-treated APFs was shown in Fig. 6 in terms of atomic % and weight %. Several studies have employed EDX investigations to assess the level of chemical modification of fibre materials, as documented in the literature. EDX was used to analyse the elemental composition of APFs and chemical element distribution [67]. The major peaks of benzoyl-treated APFs are carbon and oxygen, potassium, sodium, and calcium, whereas the major peaks of untreated AP fibres are carbon, oxygen, potassium, sodium, and silicon. Cellulosic fibre is expected to result in C and O being dominant peak elements in the EDX spectrum of both untreated and benzoyl-treated APFs [68,69]. Particularly in the case of APF, the usual Si peak is

Table 1
CI and CS of untreated and benzoyl treated AP fiber.

Sample	Crystallinity Index (CI) (%)	Crystallite Size (CS) (nm)
Untreated AP fiber	46.52	1.9
Benzoyl treated AP fiber	72.14	2.6

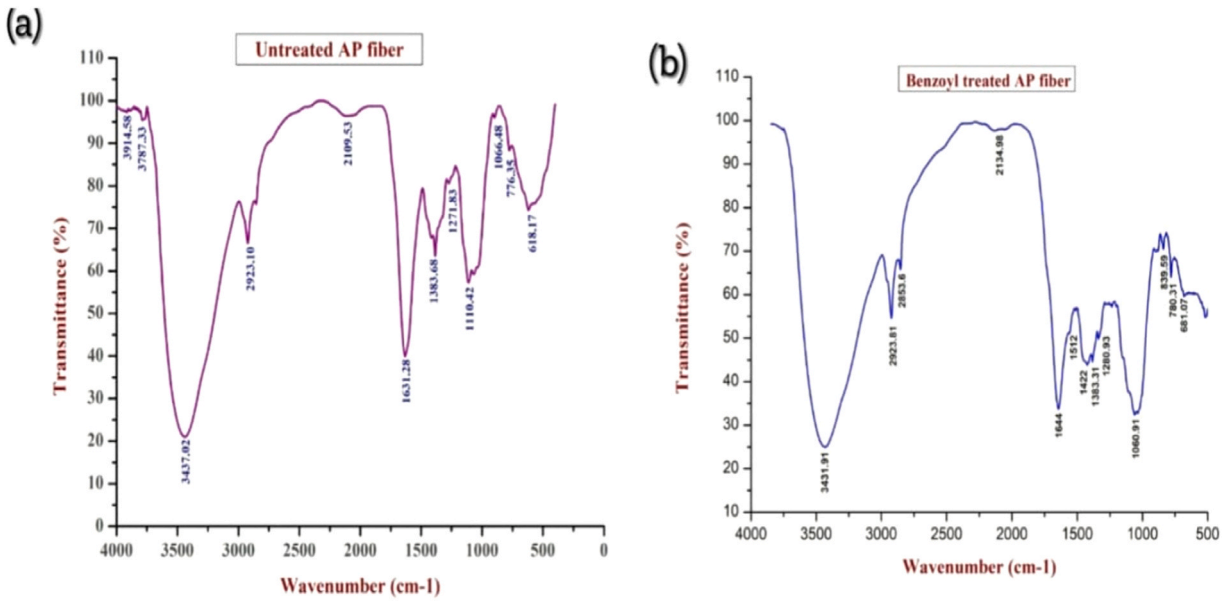

Fig. 4. FTIR curves of untreated and benzoyl treated AP fiber.

Table 2
FTIR Vibrational band assignments of untreated and benzoyl treated AP fiber.

Wave Number (cm^{-1})		Vibrational Band Assignment
Untreated	Benzoyl treated	
3914.58, 3787.33, 3437.02	3431.91	-OH Stretching in Hydrogen bond
2923.10	2923.81	-CH Stretching of Cellulose
-	2853.60	-CH_2 Stretching of cellulose and hemicelluloses
2109.53, 1631.28	-	-C═C Stretching
-	1643.04	-CO stretching in lignin
-	1512.13	C═C groups of lignin
-	1422.27	-CH_2 stretching of cellulose
1383.68	1383.31	-CH bending
1271.83	1280.93	-CO stretching in lignin
1110.42		-C═O═C stretching vibration of cellulose
-	1060.91	-COH stretching of lignin
	839.59	CO stretching of cellulose
776.35	780.31	Presence of saline content
618.17	681.07	Out of plane of -OH bonding

essentially absent from the picture, showing that some impurities connected to the material were eliminated during the benzylation process. The binding components' higher C/O atomic ratio further demonstrates how the mass fraction and degree of grafting of the benzyl groups were significantly improved under the chosen conditions [70]. It is confirmed that the removal of lignin and wax from the fibre surface as a result of surface treatment by the larger amounts of oxygen (O), carbon (C), and also the low amounts of elements like silicon (Si) and potassium (K) [71]. The findings also support a discernible alteration in the potassium (K) peak of untreated APFs, indicating that benzoyl-treated APFs have lower potassium contents than raw APFs because benzoyl treatment may have removed the treated fiber's outer layer, which was made up of wax, lignin, and impurities [72]. The atomic and weight percentages of untreated and benzoyl treated AP fibers were represented in Table 3.

3.5. CHNS analysis

A sample of a substance, mineral, or chemical compound is analysed for its elemental composition during the process of elemental analysis. It can be quantitative to determine how much of each element is present, or it can be qualitative to identify the elements that are present. The contents of carbon, hydrogen, nitrogen, sulphur, and oxygen are identified through CHNS (O) analysis. Impurities are simple to spot. Elemental analysis is a quick, easy, and low-cost method for figuring out chemical composition. It's crucial to weigh out samples that are all the same size. It is important to weigh the sample accurately and record the weight properly because the final results of the C, H, N, and S content are expressed as a percentage of the initial sample weight. Table 4 below shows the percentages of chemical compounds.

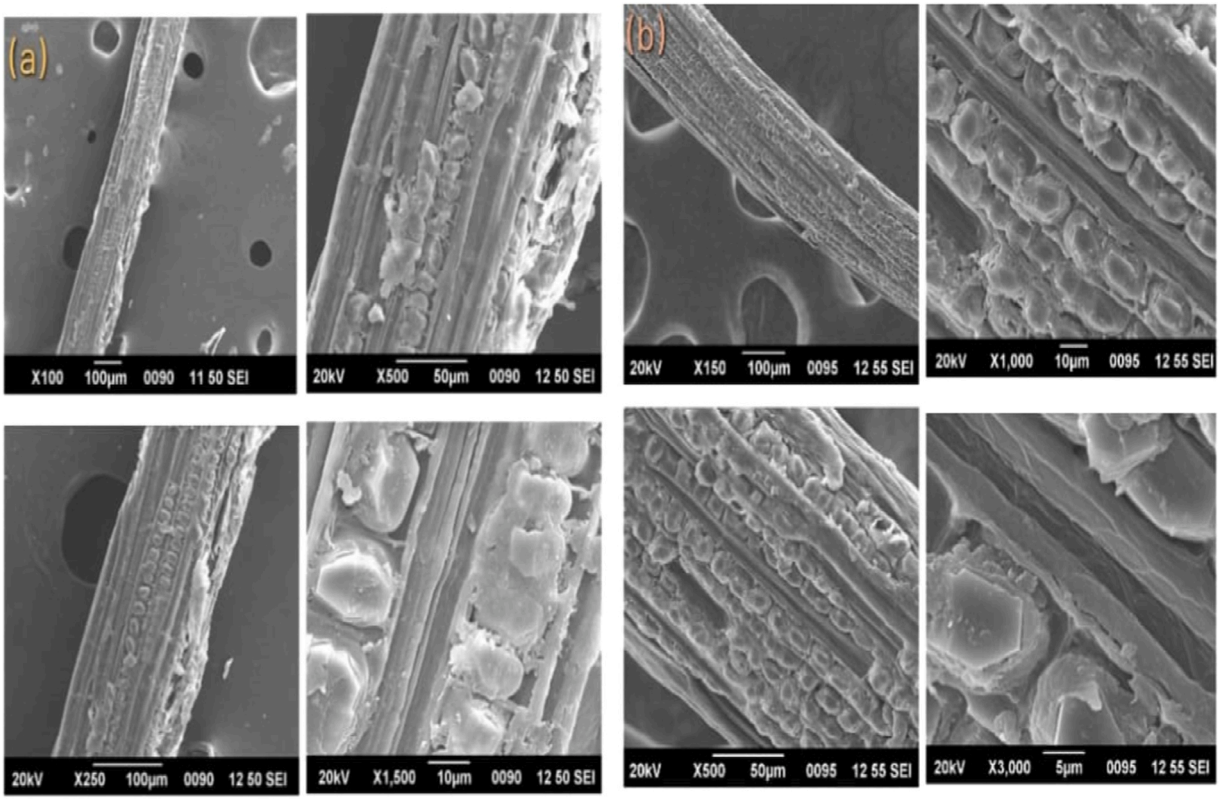

Fig. 5. SEM micrographs of untreated (a) and benzoyl treated (b) AP fiber under 100, 500, 1000, 3000 magnification fields.

According to the CHNS study, untreated AP fibres have a carbon content of 43.38%, but after treatment with benzoyl chloride, the carbon content drops to 38.93%. Low carbon content in natural fibres is one of the most important thing in modifying the mechanical characteristics of the final product. Because the carbon content in benzoyl-treated samples exceeds 40%, they can be used as conductive fillers in dielectric loss materials [73].

3.6. Thermo-gravimetric analysis

With the aid of TG, DTG, and DSC, researchers examined the thermal stability of various naturally occurring fibres that had been alkali-treated. The natural fiber's weight change (in%), thermal degradation temperature, thermal stability before and after the benzoyl treatment can be evaluated using the thermal analysis technique known as TGA [74]. Fig. 7 displays the TG curves of untreated and benzoyl-treated APF.

The different components of untreated and benzoylated APFs degrade in five stages. The thermal stability of benzoyl-treated AP fibres (366 °C) was slightly increased, according to results of Acacia pennata fibres treated with benzoyl chloride. Additionally, the weight loss percentage of the benzoyl-treated fibres were higher than the untreated AP fibres, indicating the removal of cementing agents. They also discovered that the alkali concentration and time of immersion had an impact on the AP fibres' thermal stability [75]. This resulted from the removal of lignin and hemicellulose following alkali treatment [76]. The five-phase characteristics of weight loss% with increasing temperature were discovered using TG results. The first phase is credited with removing moisture, and the second phase with removing cellulose and hemicellulose compounds. While the first phase of decomposition was attributed to the breakdown of hemicellulose and moisture loss, the second phase of decomposition was attributed to the degradation of cellulosic and lignin. However, the second-phase characteristics of weight loss percent against the temperature range for AP fibres [77], were different. Complete cellulose and hemicellulose content degradation is attributed to the third phase. The breakdown of pectin, wax, and other impurities is visible in the fourth and fifth phases. The thermal study of untreated, benzoyl-treated AP fibres and the mass loss at T-max of untreated, benzoyl-treated AP fibres are shown in Tables 5 and 6. AP fibres have higher heat stability and degradation temperatures when compared to sugar palm fibres. Less pronounced degradation peaks for benzoyl-treated APFs demonstrated the loss of several fibre components during treatment, including lignin and hemicellulose [78].

The TG/DTG analytical technique monitors the weight shift that takes place as a specimen is heated in order to evaluate the thermal stability and its fraction of volatile components. Thermal analysis is a test used to assess how a material would change chemically, physically, and structurally as a result of a temperature change [79]. Fig. 8 depicts the DTG curves for untreated and benzoyl-treated APF. The differential thermal analysis (DTG) explains how the different components of untreated and benzoyl-treated APFs degrade

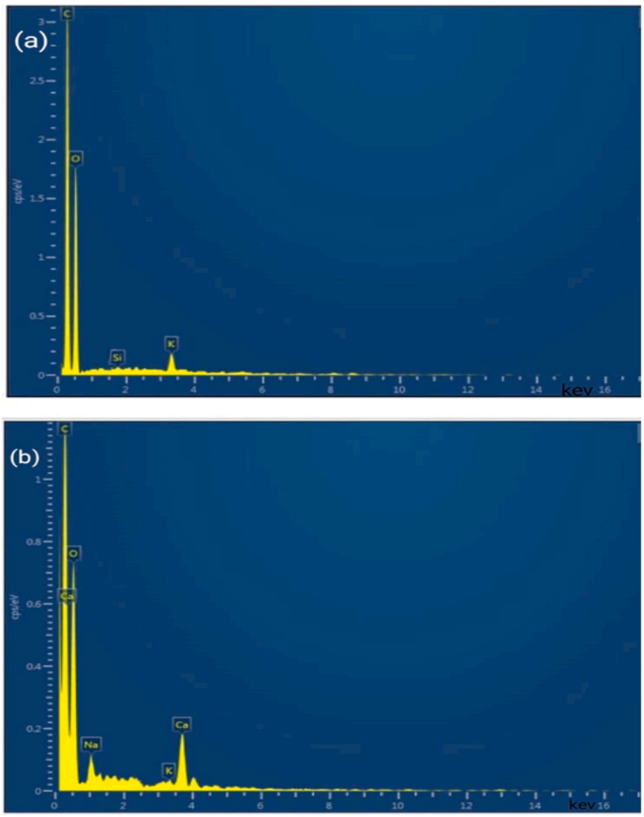

Fig. 6. EDX Pattern of (a) untreated and (b) benzoyl treated AP fiber.

Table 3
weight % and atomic % of various elements present in untreated and benzoyl treated AP fiber.

Element	Untreated		Benzoyl treated	
	wt%	Atomic %	wt%	Atomic %
C	55.03	60.03	54.61	62.77
O	42.93	38.02	40.91	35.31
K	1.74	1.21	0.21	0.08
Si	0.29	0.14	-	-
Na	-	-	1.47	0.88
Ca	-	-	2.79	0.96
Total	100	100	100	100

Table 4
CHNS elemental analysis of untreated and benzoyl treated AP fibres.

Fiber sample	N%	C%	S%	H%	Sample weight (mg)
Untreated AP fiber	0.76	43.38	0.51	6.75	14.35
Benzoyl treated fiber	0.82	38.93	ND	5.48	12.23

over time. First, weight has decreased at 89 °C and 185 °C at a rate of about 0.457 and 0.136 mg/min. Here, hemicelluloses and moisture were broken down. Mass has decreased with temperatures between 260.9 °C and 329.8 °C, 0.579 mg/min, and 1.215 mg/min. This results from the breakdown of cellulose's hemicelluloses, lignin, and glycol-sidic bonds [80]. The region (443.7 °C, 468.2 °C, and 500.2 °C) experienced mass losses (roughly 1.126 mg/min, 0.911 mg/min, and 0.691 mg/min) as a result of wax and lignin degradation. According to the thermal analysis' findings, both untreated and benzoyl-treated APFs make good reinforcement materials for making composites.

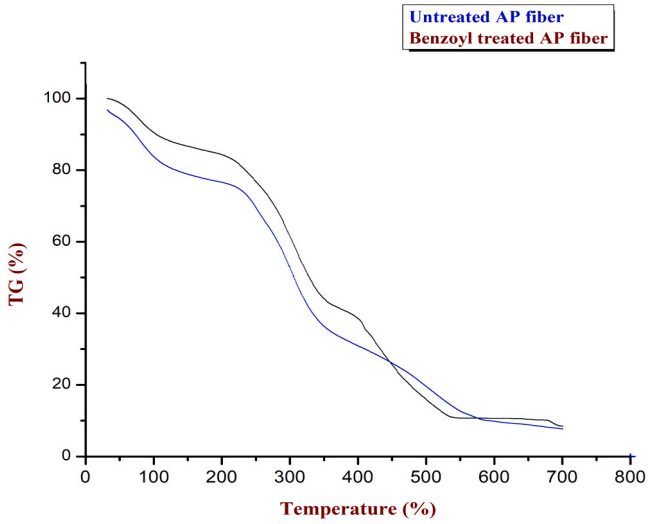

Fig. 7. TG curves of untreated and benzoyl treated AP fiber.

Table 5
Thermal study of untreated and benzoyl treated AP fibres.

Type of fiber	Temperature during loss (°C)	Weight loss (%)	Thermal stability (°C)	Residual char at 750 °C
Untreated AP fibers	40 – 120	14.27	328.95	0.072
	120–280	20.41		
	280–400	30.32		
	400–500	11.77		
	500–600	9.91		
Benzoyl treated AP fibers	40 – 120	11.8	366	0.081
	120–280	17.5		
	280–400	33.5		
	400–500	22.52		
	500–600	5.48		

Table 6
Mass loss at Tmax of untreated and benzoyl treated AP fibres.

Type of fiber	Total mass lost (%)					Max. Temperature limit (°C)
	1st stage	2nd stage	3rd stage	4th stage	5th stage	
Untreated AP fibers	14.27	34.68	65	76.77	86.88	226.3
Benzoyl treated AP fibers	11.8	29.3	62.8	85.32	90.8	226.7

3.7. Differential scanning calorimetric (DSC) analysis

Fig. 9(a and b) shows the DSC analysis curves of untreated and benzoyl treated APFs. This curve validates the reduction of hydrophilicity qualities in chemically treated fibres, the improvement in cellulose and amorphous fractions (hemicellulose and lignin) breakdown temperature, and also demonstrates overall high heat generation in these fibres [81]. The endothermic peak was seen at around 103.7°C and 100°C represented the loss of moisture from the AP fibres. The exothermic peaks at around 507°C and 733.9°C (for benzoyl treated), 325.2°C and 521.7°C (for untreated) support the complete decomposition of cellulose, hemicellulose, and lignin in benzoyl treated and untreated APFs.

3.8. Mechanical analysis

The Table 7 shows that the tensile (young's) modulus of untreated and benzoyl-treated APFs was 29.3 GPa and 14.62 GPa, respectively. These values are higher than those of Lygeum spartum L (13.27 GPa) [82] from and lower than those of Ferula communis (52.7 GPa) and Saharan Aloe vera (42.29 GPa), while the tensile strength of be (181.69 MPa). The tensile values are slightly higher than luffa cylindrical (385 Mpa) fibres and slightly lower than Ferula communis (475.6 Mpa) fibres [83]. Results revealed that Acacia pennata fibres had higher tensile and modulus values than sugar palm fibres (173.99 MPa and 6.64 GPa, respectively). The proportion

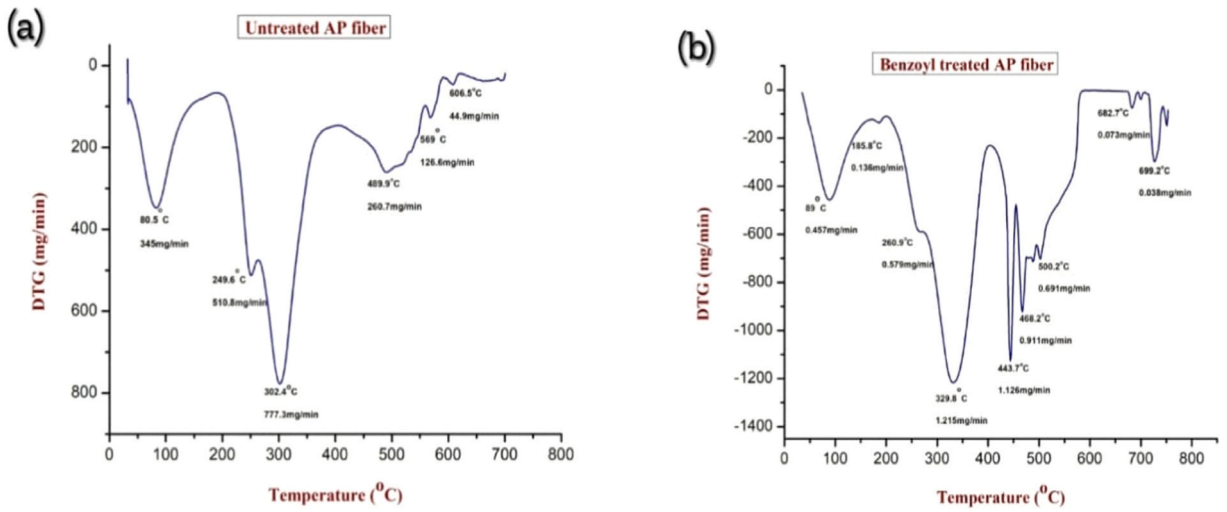

Fig. 8. DTG curves of untreated and benzoyl treated AP fiber.

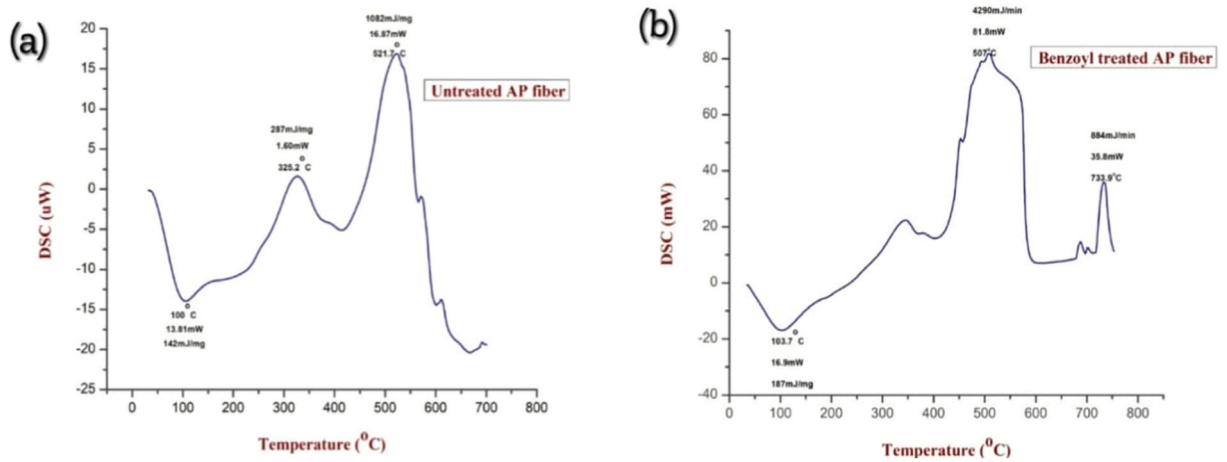

Fig. 9. DSC curves of untreated and benzoyl treated AP fiber.

of cellulose content in APF following the benzoylation treatment increases as a result of the efficient removal of lignin and hemicellulose. The reason why natural fibres are stronger is because they have a larger amount of cellulose [84]. This study's analysis of APF. The fact that all of the treated APF displayed increased tensile strength and modulus compared to the untreated showed the treatment's beneficial benefits [85]. Despite the fact that alkali treatment (NaOH) increased the tensile strength and modulus of APF in comparison to untreated APF, it was also noted that it only partially removed lignin and hemicellulose from the structure of natural fibre. As a result, further benzoyl treatment was used, which in this investigation improved the tensile strength and modulus of APF. Untreated and benzoyl treated APFs had an elongation at break of 6.2% and 3.2%, respectively. Microfibril angle (MFA) of natural fibers has an inverse correlation with tensile strength, while it is directly correlated with breaking elongation. Consequently, smaller MFA values in natural fibers are considered favorable characteristics for composite materials [86]. In this study, the MFA of benzoyl-treated and untreated APFs were measured as 19.67° and 14.79°respectively. The MFA of the benzoyl-treated and untreated APFs are respectively 19.67° and 14.79°. The calculated values of MFA and elongation at break were slightly supports the results of sisal fibers (10to 25) and kenaf (2.7–6.9%) fibers [87]. Resistance to breaking under tensile stress is one of the most important and frequently measured characteristics of materials used for structural application. To ensure reliable and accurate results, the tests were

Table 7
Mechanical measurments of untreated (raw) and benzoyl treated AP fiber.

Fiber sample	Breaking strength (gf)	Elongation at break (%)	Tensile modulus (GPa)	Micro-fibrillar angle (°)	Tensile strength (MPa)
Untreated AP fiber	1540	6.2	29.30	19.67	181.69
Benzoyl treated AP fiber	3340	3.2	14.62	14.79	467.86

conducted with approximately 3–4 repetitions. This repetition was necessary to achieve a good quality of results and to account for any potential variability or outliers in the data. By conducting multiple repetitions, it helps to establish consistency and validity in the obtained measurements.

4. Conclusion

Researchers have recently begun to pay more attention to natural fibres because of their biodegradability, low cost, abundance, etc. Despite of these advantages, the hydrophilic nature of these materials has severely limited their application in a variety of industries. The two chemical processes that are most frequently used are alkalization and benzoylation. At first the AP fibers were pretreated with alkali (NaOH) solution and then treated with benzoyl solution. The benzoyl treatment appears to be the most economical and effective way to increase moisture resistance and wetting properties. In this work, natural fibres extracted from Acacia pennata are characterised in terms of their structural, thermal, and mechanical properties.

For benzoylation, benzoyl chloride was used to improve the bonding between the fibre by decreasing the hydrophilicity of the Acacia pennata fibre. This has also been discovered to improve the AP fiber's resistance to high temperatures.

- The fibrillation effect observed in natural fibres treated with benzoyl increases the surface area and improves interfacial bonding with the structural materials, thereby enhancing the mechanical properties of the composites. The tensile properties and thermal stability of the composites are significantly influenced by the benzoyl chloride concentration, fiber loading percentage, and immersion time of the natural fibres.
- The XRD and SEM analyses demonstrate that benzoyl treatment increases the crystallinity index and crystalline size of Acacia pennata fibres, resulting in smoother surfaces. The FTIR analysis confirms the removal of cementing substances such as hemicellulose, lignin, and pectin in benzoyl-treated natural fibres.
- The TGA analysis indicates that the AP fibres treated with benzoyl exhibit second and third phase characteristics depending on the treatment conditions and immersion time. The TG analysis demonstrates that the benzoyl-treated Acacia pennata fibres have a thermal stability of up to 366 °C.
- The DSC results indicate that the first phase involves the evaporation of moisture, while the second phase involves the degradation of hemicellulose, lignin, and pectin, with both endothermic and exothermic reactions being visible. The SEM images confirm that the improved mechanical properties of the AP fibres after benzoyl treatment result in enhanced fibers structure which can be used as FRP composite.
- Pure Acacia pennata natural fibres possess high tensile strength and tensile modulus mechanical properties, which make them suitable for manufacturing composites for construction applications. The results suggest that benzoyl-treated fibres can be used in Concrete reinforcement, FRP composites, masonry retrofitting, geotechnical engineering, roofing/wall panels, owing to their improved mechanical and thermal properties.

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CRediT authorship contribution statement

K.R. Jaya Sheeba: Conceptualization, Data curation, Formal analysis, Methodology, Project administration, Software, Validation, Writing – original draft. **R. Krishna Priya:** Conceptualization, Formal analysis, Methodology, Project administration, Supervision, Validation, Visualization, Writing – review & editing. **Krishna Prakash Arunachalam:** Conceptualization, Formal analysis; Validation, Project administration, Visualization, Writing – review & editing. **Siva Avudaiappan:** Methodology, Project administration, Supervision, Funding acquisition, Validation, Visualization, Writing – review & editing. **Erick Saavedra Flores:** Project administration, Supervision, Funding acquisition, Validation, Visualization, Writing – review & editing. **Pavel Kozlov:** Formal analysis, Methodology, Validation, Visualization, Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

Data will be made available on request.

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