Study of raw and chemically treated Sansevieria ehrenbergii fibers for brake pad application

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Abstract
The replacement for synthetic fiber and other materials with natural fiber in the brake pad has become a key research area in many industries. Many production industries are always focused on natural fiber over synthetic fibers because of its advantage of biodegradability, less expensive, availability, and environment friendly. However, natural fibers cannot be used directly in composites because of its poor adhesion behavior between matrix and fiber and observe high water content in the fiber. Further, the excess water content in the inside fiber has been removed by using alkaline, benzoylation, and acetylation treatment. It has been achieved by the same chemical concentration but with different time duration. The Fourier-transform infrared spectroscopy (FT-IR) analysis revealed that the amount of cellulose content after chemical treatment was improved and decrease in other substances like hemicellulose, lignin, and wax content. X-Ray Diffraction (XRD) analysis study confirmed that the rise in the crystalline index (70.41 from 52.7) of alkali-treated SEFs at 30 min. The thermogravimetric analysis study has confirmed that the increase in the degradation temperature of Benzoyl-treated SEFs (387 °C) at 60 min when compared with untreated fiber (320 °C). The Scanning Electron Microscope (SEM) results have confirmed that the elimination of impurities from the fiber surface after chemical treatment. Further, the acetyl- treated fibers have shown that better hydrophobic properties than other treated fibers. In the above tests have been concluded that the chemically treated fibers, as one of the suitable materials for the development of the brake pad application.

1. Introduction
Brake pads play a vital role in automotive vehicles. Every vehicle requires brakes to retard or to stop the vehicle. The braking system mainly consists of a drum and pads. When the brakes are applied, the pad comes in contact with the drum, and the friction is generated between brake drum and padding, the vehicle gets retarded. The mechanism of the brake pad is to convert kinetic energy to heat energy through friction. Earlier brake pads were manufactured by using asbestos embedded in a matrix with several other materials. However, due to its carcinogenic nature, the use of asbestos in brake pad has been stopped [1]. Many researchers studied and developed many materials for brake pad applications, which are free from asbestos-like Semi-metallic, ceramics, glass as a fiber. Usually, brake pads contain various combinations of ingredients of some 5–30 materials, mainly used like abrasives, reinforcement, binders, and friction modifiers for adequate friction and wear performance. Among these, reinforcement plays a vital role as its properties decide the performance of the brake pad. Thus It is essential to choose perfect reinforcement for the brake pad. Reinforcement helps to make the brake pad reliable.

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Mainly, fibers are used as a reinforcement because it has frictional property, excellent thermal resistance, etc. There are two types of fibers used, natural and synthetic. Synthetic fibers like PVC, potassium Octatitanate, Polyethylene fibers, etc are used in the brake pads and have performed well. Nevertheless, due to health and environmental issues, the usage of these fibers was reduced. The natural fiber was preferred to avoid the usage of synthetic fiber.

Natural fiber plays a vital role as a raw material in many upcoming industries. Due to an increase in environmental issues, Natural fibers have been proved to be better raw materials in many sectors, mainly in the automotive industry. The most common materials used in automotive sectors are synthetic and glass [2]. However, researchers are very much focused on studying the natural fibers as it provided the desired properties like less expensive, lower density, high toughness, harmless, sustainable, soft, and environment friendly features [3]. Many natural fibers like fibers of Banana, Bamboo, Coconut, Kenaf, etc are used in the day to day applications.

Sansevieria ehrenbergii fibers are obtained from Sansevieria ehrenbergii plant leaves. It is composed of hemicellulose, cellulose, and lignin. The presence of a maximum amount of cellulose content is essential as it helps in efficient reinforcement. Hemicellulose is very hydrophilic and easily gets dissolved in alkali. Lignin is very amorphous and hydrophobic [4]. The raw fibers cannot be used as the reinforcement in brake pad composite because it does not have the required property like high thermal stability, good strength, etc. To achieve it, we need to eliminate the impurities like hemicellulose, wax, lignin, pectin, etc from the SEF. Thus these fibers are treated with chemicals to enhance their properties. Many researchers have conducted chemical treatment on the fibers. Chemicals like alkaline, acetylation, silane, permanganates acrylation, etc have been done [5]. In this study, performed three different chemical treatments, mainly Alkaline, Acetylation, and Benzylation by varying the soaking time for 30 min interval (i.e., 30, 60, 90, 120, 150 min). The effect of various chemical treatments on natural fiber was studied with the help of various testing like TGA, XRD, FTIR, SEM, and water absorption. From this study, it can observe which chemical treatment provides the required result with the duration.

### 2. Methodology

#### 2.1. Materials

The Sansevieria ehrenbergii plant leaves were collected from the Erode region, Tamil Nadu, South India. This plant is called a Snake grass plant and comes under the Sisal plant family [6]. Untreated Sansevieria ehrenbergii fibers (SEFs), Sodium hydroxide pellets, concentrated Hydrochloric acid, and deionized water is used in the alkali treatment [7]. Glacial acetic acid, acetic anhydride, and sulphuric acid are used for Acetylation treatment. Benzyol chloride and deionized water are used for Benzylation treatment [8].

#### 2.2. Fiber extraction

The various process is adopted from the extraction of natural fibers from plants. Retting is a common technique used for fiber extraction. Retting is also called a degumming process. It is an effective method for the separation of fiber from the stem and leaves by removing the outer layer and gum-like material from the surface of the leaves. Different Retting techniques process is available like water retting process, Chemical retting, Spray enzyme retting, and Bio-innovative retting process [9–11]. By using a sharp knife, the Sansevieria ehrenbergii leaves are chopped from Sansevieria ehrenbergii plant. The Sansevieria ehrenbergii leaves were kept inside the water for four days as a part of the soaking process. Then resin-like material and outer layer of the leaf are removed, and single fiber is obtained by manual peeling. The collected fiber is dried under sunlight for 8 h.

#### 2.3. Chemical treatment

##### 2.3.1. Alkaline treatment

For natural fibers to be used as reinforcement in composite, alkaline treatment is commonly used. To increase the surface roughness of the fiber, the alkaline treatment, separates hydrogen bonds from the grid structure. The removal of minor constituents like lignin, hemicellulose, and amorphous cellulose from the outer surface of the fiber to a certain extent [7]. The hydroxyl group present in natural fiber is ionized to the alkoxide group after the treatment [12]. This treatment improves fiber matrix interlocking and increase cellulose content in fiber which increase adhesion property, reduce water absorption and thermal stability [13]

\[
\text{SEF} - \text{OH} + \text{Na}^+ \text{OH}^- \rightarrow \text{SEF} - \text{O}^- \text{Na}^+ + \text{H}_2\text{O}
\]

Initially, for this treatment, SE fibers were soaked in 5% (w/v) NaOH solution. The immersion period for fiber in NaOH solution was 30 min, 60 min, 90 min, 120 min, and 150 min. The fiber samples each weighing 6 g are soaked in 200 ml of water containing 10 g of sodium pellets. A total of 5 samples were prepared, and each
sample is taken out for 30 min. Then it neutralized by using a few drops of HCl for 3–5 min to prevent a further chemical reaction. These fibers are then oven-dried by placing it in an electric oven and heated at the temperature of 80 °C for 6 h [14].

2.3.2. Acetylation treatment
The acetylation process was the best method to modify the surface properties of the fiber and also making the fiber hygroscopic. This treatment is most used adequately in textile goods as the rate and extent of swelling is highly reduced [15, 16]. The process is applied to the wood cellulose because it improves dimensional accuracies, increases the stability of the cell wall, and also safe environmental degradation [17]. It is an esterification method in which reaction takes place between an acetyl functional group (CH₃COO−) and hydroxyl group (−OH). This process can take place with or without the help of a catalyst. During this process, the plasticization of the cellulosic fibers takes place. For the acetylation process, acetic anhydride (CH₃COOCOCH₃) was preferred more than acetic acid because acetic acid does not react adequately with cellulose [16]. However, before treating with a dioxide, it is treated with acetic acid as anhydride is a poor swelling agent for cellulose. Also, acetic acid increases the rate of the chemical reaction.

The reaction that takes place between the acetic anhydride and the hydroxyl group is the removal of the hydroxyl group. Those are minor constituents like lignin, hemicellulose, and amorphous cellulose [18]. The reaction does not take place with the crystalline cellulose as it is complicated to remove the strong interchain bonding between them, which is very difficult for any chemicals to react. Also, this reaction results in the generation of by-product acetic acid, which is to be removed before the fiber being along with the lignocellulosic material [19]. The hygroscopic nature of natural fibers is reduced, which results in dimensional stability. Water absorption mainly depends on factors like fibers hydrophilic nature, temperature, the orientation of the fiber, protection of the surface, exposed surfaces area, loading of the fiber, diffusivity, and amount of void content of the individual components [5]. This treatment also improves the fiber–matrix adhesion property, which also improves the tensile and thermal properties of the composites [20]. The chemically treated fibers had developed a very rough surface and also developed the amount of voids present, which provides better mechanical interlocking with the polystyrene matrix [21]. The chemical reaction is given by

\[
\text{SEF} - \text{OH} + \text{CH}_3 - \text{CO} - \text{O} - \text{CO} - \text{CH}_3 \rightarrow \text{SEF} - \text{O} - \text{C} - \text{O} - \text{CH}_3 + \text{CH}_2\text{COOH}
\]

Initially, for this treatment, SE fibers are soaked in 5% (w/v) NaOH solution. Then acetylation treatment was applied for each group of SE fibers in glacial acetic acid concentration solution of 5%. The immersion period for fiber in glacial acetic acid solution was 30 min, 60 min, 90 min, 120 min, and 150 min. The samples each weighing 6 g are to be treated with NaOH and then is neutralized by using HCl concentration [22]. Then the fibers sample is soaked in 200 ml glacial acetic acid and 2–4 drops of acetic anhydride solution. A total of 5 samples were prepared, and each sample is taken out for 30 min. Then it neutralized by using a few drops of conc. H₂SO₄ for 3–5 min to prevent further chemical reaction [23]. These fibers are then oven-dried by placing it in an electric oven and heated at a temperature of 80 °C for 4 h. Figure 1 shows the process of neutralizing the acid-treated fiber. Figure 2 portrays the oven dried fiber.

2.3.3. Benzoylation treatment
Benzoylation treatment is essential and effective in fiber treatment. Usually, Benzoyl chloride was used for the treatment. It contains a benzoyl group, which helps to decrease the hydrophilic property of the fiber. At the beginning of Benzoyl treatment, the fiber is treated with NaOH so that it will trigger the hydroxyl groups, which are present in cellulose and lignin of the fiber [24]. This treatment improves the adhesion property, reduces water absorption, and increase thermal stability. The following reaction takes place during chemical treatment:

\[
\text{SEF} - \text{OH} + \text{NaOH} \rightarrow \text{SEF} - \text{O} - \text{Na}^+ + \text{H}_2\text{O}
\]

\[
\text{SEF} - \text{O} - \text{Na}^+\text{C}_6\text{H}_5 = \text{OCI} \rightarrow \text{SEF} - \text{C}_6\text{H}_5\text{C} = \text{O}_2 + \text{NaCl}
\]

For this treatment, SE fibers are soaked in 5% (w/v) NaOH solution. Then treated SE fibers are soaked in 5% v benzoyl chloride solution. The immersion period for fiber in benzoyl chloride solution was 30 min, 60 min, 90 min, 120 min, and 150 min.50 ml of benzoyl chloride is added on 30 grams of SEFs in 1 liter of water for 150 min. At each interval of 30 min, 6 grams of SEFs were taken out and neutralized with deionized water and were cleaned with water and dried in the oven for 4 h at 70 °C [25]. Figure 3 illustrates the sample that is cleaned after soaking in Benzyl chloride. Figure 4 displays the sample that is dried in an oven.

2.4. FTIR
The Fourier transform infrared analysis of SEFs is done using a Shimadzu IRAffinity-1 spectrometer. In this study, samples are prepared by chopping it approximately for 2–3 mm. The test is conducted in the region from
400 to 4200 cm\(^{-1}\) wavenumber at room temperature of 25 °C and relative humidity of 60%. A total of 16 samples were taken for each example, a resolution between 0.5–16 cm\(^{-1}\). The chopped 3 mg of dried fiber is mixed with potassium 150 mg of bromide and pelletized. Each sample pellet is kept to record spectra.

2.5. XRD

X-ray Diffraction analysis is generally used to check the number of cellulosic-crystals present in a mixture. The XRD result was used to determine the percentage of crystallinity and also the crystalline index. The XRD analysis was carried out using a D8 advance machine with the source of Cu-anode ceramic tube and Ni filter as a beta filter. The test was carried out for the finely chopped fiber samples, each weighing 400 mg at room temperature 25 °C and relative humidity of 60%. The machine was operated at the range of 5–90° (2\(\theta\) angle range). Materials crystalline index (C.I.) and percentage of crystallinity (%CR) were calculated using the equations (1) and (2) respectively, where I22 is the most significant intensity peak of the sample which indicates the crystalline cellulose material peak in the range of 20°–23° and I18 is the smallest peak which means the amorphous crystalline material peak in the range of 16°–19° for the natural fiber [26].

\[
CI = \frac{\text{Area of Crystalline peaks}}{\text{Total area under XRD plot}}
\]  
\[
%CR = \frac{I22}{I22 + I18} \times 100\%
\]

The %CR only calculates the crystalline area under the curve of the XRD graph, whereas CI is used to calculate the relative amount of crystallinity present in the cellulose [27].

2.6. TGA

It is useful to determine the degradation of raw and chemically treated SEFs concerning temperature. The thermograms of SEFs were executed using an SDT Q600 V20.9 Build 20 Jupiter apparatus in a Nitrogen atmosphere to avoid the oxidation, with a heating rate of 20 °C min\(^{-1}\) over a range from 30 °C–800 °C. The experiment can also be done with other noble gases which are not relative.

2.7. SEM

To evaluate the modification made on the SE fiber surface of untreated and after chemical treatment. SEM is recorded with an instrument which is having an accelerated voltage of 10-kilo Volt, a scale of 20 micrometers, and 500 \times magnification for visualizing the surface morphology of SEFs. Fibrous samples were gently secured on an SEM sample holder with carbon tape. A gold layer 5 nm thick was sputter-coated onto the surface of the sample to improve image resolution and to avoid an electrostatic charge. A total of 16 samples were taken.

2.8. Water absorption

A water absorption test was performed to check the quantity of water that fiber can absorb under specific conditions. Many factors affect water absorption of fibers like the exposure of fiber in water, temperature, etc. For the test, the fibers were dried in an oven. Each sample of 3 g of fibers was kept in 0.3 l of water at 23 °C for 24 h or until equilibrium. Fibers were removed, squeezed the excess water using the cloth, and weighed. This experiment was carried out only once. The calculation can be done using the given below equation.

\[
\text{Water Absorption}\% = \frac{W_a - W_b}{W_b} \times 100
\]

Where \(W_a\) is the weight of fiber after absorption, and \(W_b\) is the weight of fiber before absorption.
3. Results and discussion

3.1. FTIR
FTIR analysis, IR light is sent through each sample material. Some of the infrared light is absorbed in the sample, a wavenumber from 400 to 4200 cm\(^{-1}\) was recorded. It gives information to identify and characterize the functional groups that exist in the sample. The FTIR analysis is done for the untreated and chemically treated SE fibers. FTIR spectrum for Raw, alkaline, Acetyl, and Benzoyl are shown in figures 5–7. Cellulose, lignin, and hemicellulose are the major vibration bands present in both treated and untreated fibers. The broad and deep peak at 3312 cm\(^{-1}\) represents the hydroxyl group with hydrogen bond (refers to cellulose) characteristics of intermolecular bonded hydroxyl (O–H) groups [28]. The peak intensity of cellulose increases after each chemical treatment. The peak at 2916–2852 cm\(^{-1}\) refers to C–H stretching vibration in the CH\(_2\) group and CH group present in hemicellulose and cellulose components [29]. The peak of the hemicellulose component noticed only in the raw fiber but perished in chemical-treated fiber spectra. Further, this proves the elimination of hemicellulose content on the SE Fibers surface after chemical treatment. The sharp peak at 1642 cm\(^{-1}\) presences lignin with the carboxylic acid group and C=O vibration [30].
The short peak 1421 cm\(^{-1}\) represents minor constituents like lignin, hemicellulose, and amorphous cellulose with CH\(_2\) bending vibration. The intensities this peak varied for each chemical treatment. In acetyl and benzyl treatment, the intensity of the peaks was much lower than the untreated sample. C–H group bending vibration in the aromatic ring of hemicellulose and lignin in peak 1020 cm\(^{-1}\) [31]. To increase in cellulose content in SEF proved that the capability of these three chemical treatments, and during the chemical treatment, most of the gangue partials are eliminated. The fibers are changed to hydrophobic nature from hydrophilic after chemical treatment [32]. Thus chemical treatment improves the adhesion property, reduces water absorption, and increases thermal stability.

3.2. XRD

The structure of the fibers and its properties are determined mainly by the amount of cellulose and non-cellulosic constituents present in the natural fiber. These also influence the crystallinity of the fiber [33]. The graphs obtained for the untreated fiber and chemically treated fibers are shown in figures 8–11. Generally, from the figures, we can identify that for untreated and fibers treated with acetic acid, there was the presence of 2 peaks at the 2θ angle of 18° and 22°, for alkaline and benzyl treated fibers there was the presence of 3 peaks at the range of 2θ angles.
of 13°, 15° and 23°. However, these irregular distributed amorphous peak after chemical treated latter gets merged [34]. The difference in peak values from the analysis is because of chemical treatment, which causes the generation of disorder in the structure of material [35].

Many researchers have already studied the effect of chemical treatment on the lignocellulosic material. Nobuo shiraishi et al [36] from their observation reported that the chemical reaction takes place with the amorphous region, and the latter proceeds with the rate of diffusion and rate of reaction. Since it cannot diffuse the crystalline region, it reacts with the chain ends, which are on the surface of the crystallites. This results in the opening of some of the bonds of the hydrogen–cellulosic chain and produces amorphous cellulose content. This eventually results in the reduction of crystallinity. Zafeiropoulos et al [37] observed that chemical treatment increased the crystalline cellulose content of flax fiber because of the removal of impurities and amorphous contents Tserki et al [38] in their study, observed that even when the flax, hemp, and wood fibers were treated with different chemicals, they still showed the same crystalline index.

Using the equations (1) and (2), the CI and crystallinity % are represented in table 1. From the table 1, we can identify that for raw fiber the CI and crystallinity % were 0.53 and 63% whereas the chemically treated showed maximum values at a different period, i.e., for alkaline treated fibers the maximum CI and crystallinity % of 0.70 and 68% were identified at 30 min whereas for acetyl treated fibers the maximum CI and crystallinity % of 0.69 and 65% were determined at 90 min whereas for benzyl treated fibers the maximum CI and crystallinity % of 0.67 and 68 were identified at 30 min.
From the table 1, we can identify that CI values for all chemically treated fibers, i.e., alkaline, acetyl, and benzyl treated, were nearly the same. This was mainly because when the fibers are treated with different chemicals for the different soaking period, it was found that there was the difference in the concentration in the removal of several impurities, amorphous cellulose, hemicellulose, lignin which were studied in previous observations [35–37]. It was found that alkaline had a maximum crystalline index and the percentage of crystallinity. Also, when compared with the untreated fiber, it showed an increase in values because when the fibers are chemically treated, there was an increment in the amount of crystalline cellulose, which helps in achieving high thermal stability. And also, the removal of non-crystalline materials like lignin, hemicellulose, etc [39].

### 3.3. TGA

Typically, three degradations were observed in various stages during the study of SEF. Firstly, the degradation occurs at 70 °C to 130 °C because of the evaporation of water and other volatile substance present in the SEFs. The other significant degradation (130 °C–275 °C) corresponds to the thermal decomposition of hemicellulose and the degradation of cellulose. The final stage linked to the deterioration of lignin in SEFs. From the table 2, we can observe that the 1st degradation, i.e., the amount of water evaporation of treated fiber gets decreased when compared to raw fiber. Besides, it can be witnessed from figures 12–14 and also from table 2 that the lignin content gets reduced after the chemical treatment. It is also evident that it improved the thermal stability of SEF after chemical treatment as the raw fiber gets degraded at 320 °C. Still, after the chemical treatment, the

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**Table 1.** Peaks intensities, crystallinity and crystallinity index of raw, NaOH, acetylation and benzoylation treated SEFs.

| Sample  | i22  | I18  | %Cr | CI  | Treatment   |
|---------|------|------|-----|-----|-------------|
| Raw     | 1111 | 643  | 63  | 0.53|             |
| ERM-30  | 2002 | 963  | 68  | 0.70| Alkaline    |
| ERM-60  | 1941 | 941  | 67  | 0.69|             |
| ERM-90  | 2070 | 1020 | 67  | 0.62|             |
| ERM-120 | 1950 | 900  | 68  | 0.62|             |
| ERM-150 | 2135 | 993  | 68  | 0.63|             |
| EOA-30  | 2125 | 1023 | 67  | 0.66| Acetylation |
| EOA-60  | 1958 | 1044 | 67  | 0.67|             |
| EOA-90  | 1551 | 838  | 65  | 0.69|             |
| EOA-120 | 2158 | 1041 | 67  | 0.64|             |
| EOA-150 | 1827 | 890  | 67  | 0.62|             |
| BT-30   | 2070 | 1051 | 68  | 0.67| Benzylation |
| BT-60   | 1982 | 1020 | 66  | 0.65|             |
| BT-90   | 1795 | 848  | 66  | 0.64|             |
| BT-120  | 2070 | 999  | 67  | 0.63|             |
| BT-150  | 2100 | 1054 | 67  | 0.62|             |
Figure 10. Benzoyl Treated.

Figure 11. Acetic acid Treated.

Figure 12. Alkaline treated fiber.
degradation temperature gets increased up to 330 °C–380 °C. In Alkaline treated fiber, ERN-60 (60 min) shows a better result when compared with others. In Benzoyl treated fiber, BT-150 (150 min) sample showed a better effect than others as it proves that the treated SEF has more hydrophobicity property compared to the raw fiber and increase in degradation temperature up to 387 °C. In Acetyl treated fiber, EOA-90 (90 min) provides a better result than others in terms of the degradation temperature. Still, water content gets reduced, and it is proved in the water absorption test. It can be observed that the % CR of many samples in XRD is the same; however, the degradation temperature varies, as %CR only indicates a crystalline area in the graph and has no relation with the thermal stability.

Thermal stability only depends on CI and crystalline size [40]. From XRD and FTIR, we witness that there is still a presence of some amorphous content in fibers. Therefore, the major amount of amorphous cellulose region bonds becomes degraded during the thermal treatment, similar to the breaking of C–C bond and other bonds in an amorphous region or some elimination reactions, etc, when compared to those bonds within the crystalline. Besides, it also depends on the packing of the cellulose chain. Further, due to the chemical treatment, the presence of H-bond intensity near the cellulose chains can be seen, and it varies with the different chemical
treatments and its soaking period. It makes the cellulose structure more packed, which in turn acts as an obstacle and makes it difficult to break the bond, and thus there is a requirement of high temperature to break it \[41, 42\].

3.4. SEM

SEM provides the surface morphology of the natural fiber surface. Figure 15 shows the surface image of untreated SEFs. Figures 16–18 give the images of SEFs of alkali-treated, Benzyl, and acetylation treated fiber, respectively. The white blanket over the surface of fiber shows the existence of impurities like waxes and other impurities \[24\]. By treating SEF with chemical, take out waxes and oil from the outer surface of the fiber and also removal of minor constituents like lignin, hemicellulose, and amorphous cellulose from the outer surface of the fiber to a certain extent and improve roughness on the surface of fibers compared with untreated fiber. Small traces of unwanted materials can be found after alkaline treated fiber, but after Benzyl and acetylation treatment, it removes the unwanted materials from the fiber surface. With an increase in time, the chemical harms the surface of SEF seen on acetyl treated fiber after 60 min. After treatment with benzyl, it is understood that impurities were effectively removed from fiber surface without damaging. The treatment also improves the fiber–matrix adhesion property, which also improves the tensile and thermal properties of the composites \[20\]. The chemically treated fibers had developed a very rough surface and also developed amount of voids present which provides good mechanical interlocking with the matrix \[43\].

![Figure 15. Untreated fiber.](image)

**Table 2.** Degradation % and temperature in TGA of raw, NaOH, acetylation and benzoylation treated SEFs.

| Chemical     | S. No | Sample | 1st Degradation (%) | 2nd Degradation (%) | 3rd Degradation (%) | Temp (°C) |
|--------------|-------|--------|---------------------|---------------------|---------------------|-----------|
| Raw          | 1     | RAW    | 16.63               | 71.01               | 8.75                | 320       |
| Alkaline     | 2     | ERN-30 | 15.03               | 82.01               | 4.39                | 361       |
|              | 3     | ERN-60 | 9.32                | 68.94               | 5.08                | 361       |
|              | 4     | ERN-90 | 14.01               | 66.51               | 4.71                | 361       |
|              | 5     | ERN-120| 15.1                | 61.87               | 3.69                | 351       |
|              | 6     | ERN-150| 15.59               | 63.13               | 5.93                | 343       |
| Benzoylation | 7     | BT-30  | 9.21                | 79.24               | 5.93                | 385       |
|              | 8     | BT-60  | 7.59                | 90.66               | 1.61                | 387       |
|              | 9     | BT-90  | 7.23                | 72.4                | 5.51                | 386       |
|              | 10    | BT-120 | 5.91                | 88.82               | 6.92                | 382       |
|              | 11    | BT-150 | 8.76                | 71.37               | 8.12                | 366       |
| Acetylation  | 12    | EOA-30 | 4.3                 | 73.95               | 4.6                 | 336       |
|              | 13    | EOA-60 | 3.42                | 71.3                | 4.45                | 338       |
|              | 14    | EOA-90 | 6.352               | 64.59               | 2.67                | 339       |
|              | 15    | EOA-120| 3.4                 | 54.77               | 4.9                 | 330       |
|              | 16    | EOA-150| 9.16                | 66.99               | 8.28                | 335       |
3.5. Water absorption

Table 3 shows the water absorption test of raw, NaOH, acetylation and benzoylation treated SEFs. From the table 3, we can observe that the hydrophobic property of treated fibers is improved (decreased) compared to raw fiber. The reason is due to the removal of hemicellulose with the help of hydrophilic chemicals. In Alkaline treated fiber, ERN-120 (120 min) shows good results of 53.3% absorption compared to others. In Acetylation treated fiber, EOA-120 (120 min) shows 33.3% of absorption, which is better than others. Furthermore, in Benzoyl-treated fiber, BT-60 (60 min) show 80% of absorption, and it is a good result comparing to others. In general, we can say that Acetylation treated fibers exhibit better hydrophobic properties than others. Figure 19 displays the water absorption graph.

Figure 16. Alkaline treated fiber.

Figure 17. Acetyl treated fiber.
4. Conclusion

In this research work, the crystalline property, thermal stability, and surface morphology of SEFs were studied. The FT-IR results showed that chemical treatment removed some contents of impurities and also amorphous cellulose, hemicelluloses, lignin, etc from the SEFs. The thermal stability of the brake pad depends on the crystallinity index of the reinforcement. From the results of the x-ray Diffraction test, it has been found that for alkali-treated fiber, especially when treated for 30 min, showed the crystalline index of 70%. Through TGA, it was determined that the Benzoyl-treated SEFs provided better thermal resistance compared to raw fiber with the thermal stability of 387 °C. From the water absorption test, Acetyl- treated SEFs displayed better hydrophobic property compared to others. The outcome of the results has been confirmed that the chemically treated SEFs
more useful for improving the physical, chemical, and thermal properties of the brake pad application. It can be used as a suitable fiber in the composite materials and also the replacement for synthetic fibers in the development brake pad application.

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| SAMPLE | Before absorption | After absorption | Water Abs |
|--------|-------------------|------------------|-----------|
| RAW    | 3                 | 7                | 1.333 333 |
| ERN-30 | 3                 | 5                | 0.67     |
| ERN-60 | 3                 | 4.9              | 0.633 333 |
| ERN-90 | 3                 | 4.8              | 0.6      |
| ERN-120| 3                 | 4.6              | 0.533 333 |
| ERN-150| 3                 | 5                | 0.666 667 |
| EOA-30 | 3                 | 4.8              | 0.6      |
| EOA-60 | 3                 | 4.2              | 0.4      |
| EOA-90 | 3                 | 4.5              | 0.5      |
| EOA-120| 3                 | 4                | 0.333 333 |
| EOA-150| 3                 | 4.6              | 0.533 333 |
| BT-30  | 3                 | 5.6              | 0.866 667 |
| BT-60  | 3                 | 5.4              | 0.8      |
| BT-90  | 3                 | 5.5              | 0.833 333 |
| BT-120 | 3                 | 5.8              | 0.933 333 |
| BT-150 | 3                 | 5.6              | 0.866 667 |
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