



Effect of Alkali on the Mechanical Properties of Sansevieria Fibre Bio-Composites

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ABSTRACT

This study investigated the efficacy of different alkali (sodium hydroxide (NaOH), calcium hydroxide (CaOH), and potassium hydroxide (KOH)) in removing hemicellulose and lignin from Sansevieria fibres (SF) while maintaining cellulose content. Employing a precise immersion method, the alkali treatment significantly enhanced the crystalline properties and tensile strength of the fibres. FTIR spectroscopy demonstrated alterations in functional groups, while thermogravimetric analysis (TGA) revealed improved heat resistance. X-ray diffraction (XRD) analysis confirmed the enhanced crystallinity and reduced amorphousity of the treated fibres. Moreover, tensile testing confirmed the superior tensile strength and overall mechanical properties of the NaOH-treated fibres, attributing this positive outcome to the preservation of cellulose structure and enhanced interfacial bonding with the matrix. This study found that alkali treatment increased the potential of sansevieria fibres as composite reinforcement.

1. Introduction

Industrial material requirements demand the use of materials that are strong, environmentally friendly, have low economic costs, and have strong competitiveness. Natural fibre composites offer high strength and low density, which can reduce vehicle weight and improve fuel efficiency, making them suitable for body panels and interior applications in the automotive industry. Lightweight and durable composites are an excellent choice for the packaging industry due to their ability to provide effective product protection at a low cost. Additionally, composites are well-suited for aircraft body applications due to their strength and ability to withstand aerodynamic loads. Natural fibre composites have good isothermal properties, which reduce energy consumption and increase efficiency. These composites are suitable for various sea usage applications [1]. Sansevieria fibre is an environmentally friendly, non-toxic, and durable natural material with good mechanical properties, including strength and moisture resistance. SF have significant potential in various industries, including manufacturing, automotive, and industrial applications [2].

In general, plant fibres have higher strength and durability. The natural fibre's properties are affected by several main factors, including fibre strength and stiffness, density, thermal stability, and chemical composition. Fibre strength and stiffness are affected by natural fibre density, with higher-density fibres

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tending to be stronger. The chemical composition of SF has a significant effect on its mechanical and chemical properties, including the content of cellulose, lignin, and hemicellulose. Cellulose provides strength to the fibres. In applications where natural fibres will be exposed to high temperatures, their thermal stability is crucial. Fibres with good thermal stability will remain strong and durable under extreme temperatures. It is important to note that this evaluation is objective and based on empirical evidence. Previous research has indicated that the characteristics of natural fibres are significantly impacted by the conditions in which the fibre crops are grown, including factors such as soil type, climate, harvesting method, and post-harvest treatment [3]. These factors affect the fibre's mechanical characteristics, including their density, composition, strength, surface morphology, and other mechanical characteristics [4]. Selecting the appropriate fibre source is crucial in obtaining fibres with the desired properties for a specific application, whether it be in the textile, construction, or other materials processing industries [5,6]. Sansevieria fibre (SF), as one of the natural fibres, has good potential as a composite reinforcement. Prior to the moulding process, an alkali treatment can enhance the interfacial bonding between the fibre and the polyester matrix [7]. According to morphological and mechanical studies, SF has a significant potential for tensile strength and contains cellulose, which strengthens and neatens the bonds between cell structures [8].

Previous research has investigated the chemical and mechanical characteristics of SF. The study assessed thermal stability, single fibre tensile strength, crystallinity qualities, and fibre surface hardness by analyzing the chemical composition, chemical functional groups, and microstructure [9-11]. Additionally, the research included a single-fibre tensile test, fibre optic testing, microstructural analysis, X-ray diffraction (XRD) analysis, functional group analysis with FTIR, thermal stability with TGA and DSC analysis, morphology analysis with SEM and AFM analysis of the SF [12]. Composites with a polyester matrix reinforced with SF were made using a compression molding machine. SF fibres play a strong role in strengthening composites, increasing interfacial tensile forces which improve dynamic properties. Natural fibres have significant potential for producing composite materials and enhancing their dynamic mechanical properties in general [2]. SF can have an increased interfacial bonding value with polyester matrix with the addition of alkali treatment before molding. [5,8]. Analysis of SF reveals their morphological and physical properties. The fibres possess a robust tensile strength, while the cellulose content enhances the bond between cell structures, resulting in a neater and stronger structure [10]. SF have great potential to be developed as composite reinforcement. Therefore, it is necessary to characterize the fibre before it is applied as a composite reinforcement. The application of SF in composites has the potential to be developed because it has suitable, strong, and environmentally friendly characteristics. The utilization of this fibre is expected to be a solution and an alternative material for various purposes. Chemical treatment, either calcium hydroxide, sodium bicarbonate, or sodium hydroxide, is thought to have an effect on improving the mechanical, physical, and chemical characteristics of SF. Raju et al., [13] conducted research that emphasized the significance of alkali treatment for natural fibre materials. The study's results demonstrated that alkali treatment effectively enhances various aspects of natural fibre material quality. A key finding was the increase in cellulose content, which is a crucial indicator for improving fibre strength and durability. Furthermore, the alkali treatment resulted in an increase in the density of the natural fibre material, leading to improvements in tensile strength, elastic modulus, and overall mechanical properties [13]. Furthermore, alkali treatment improved the physical, thermal, mechanical, and surface properties of the fibres [14,15]. Additionally, the weight ratio of fibres to matrix is determined in order to analyze the mechanical and physical properties of the composite. Making composites with the appropriate ratio of fibre weight fraction and matrix produces composites that have good properties and are environmentally friendly. This research examines the effect of alkali type and concentration on composite characteristics.

2. Methodology

2.1 Fibre Characterization

SF were given alkali treatment by the immersion method, i.e., immersion of fibres in alkali solution. Alkali base was prepared with three types of alkali, namely NaOH, KOH, and CaOH. Each alkali was given a concentration variation of 4%, 7%, 10%, 11%, and 15%. Calculation of alkaline compounds uses the weight fraction ratio between alkaline compounds and distilled water (H₂O).

Soaking was carried out for 2 hours to provide enough time for the alkaline solution to react with the fibre surface layer. Sufficient time allows the alkaline solution to break down the hemicellulose layer on the surface and the lignin layer in the fibre cavity. The selection of 2 hours is in accordance with previous research and the results of optimizing the appropriate time interval. Excess time has an impact on the destruction of the cellulose layer [16].

2.2 Single Fibre Tensile Strength Analysis

The alkalinized fibres were tested for tensile strength. This test is expected to provide valid data on the effect of fibre alkalization on the generation of residual stresses due to the loss of some of the fibre constituent structures. The single fibre tensile strength analysis test used a Zwickroell tensile testing machine with reference to ASTM E8 and DIN EN ISO 6892-1 standards. Preload was set at 0.5 N, speed 100 mm/min, and loading speed 150 mm/min [17,18].

2.3 FTIR Analysis

Fourier transform Infrared Spectroscopy (FTIR) is used to identify and analyse the chemical components of sansevieria fibres, determine their chemical structure, and monitor chemical changes that occur during fibre processing. The FTIR testing process involves exposing natural fibres to infrared radiation, which is then measured by a detector. The infrared spectral pattern resulting from the test provides information about the molecular vibrations in the sample, enabling analysis of the chemical components present. FTIR testing of sansevieria fibres confirms fibre quality, detects contaminants, and provides insight into the chemical properties of fibres that can affect their performance and applications. The VERTEX 80v FTIR spectrometer manufactured by Bruker in Germany was used to conduct the FTIR test. The fibres were ground into a fine powder, sifted to a particle size of 100 mesh, and mixed with KBr. The obtained samples were examined using an FTIR spectrometer, capturing light spectra within the wave number range of 4000 cm^{-1} to 500 cm^{-1} . The testing was conducted following the ASTM E 1252-98 criteria to achieve precise and consistent analysis outcomes. This technique enables precise determination of the chemical composition and molecular structure of the fibres, facilitating thorough investigation into the material's properties [19].

2.4 XRD Analysis

X-ray Diffraction (XRD) testing was used to identify the crystal structure of Sansevieria fibre. This method provides information about the mineral and crystalline compounds present in the fibre. The process involves shooting X-rays onto a sample of Sansevieria fibre and measuring the diffraction patterns of the scattered X-rays. The diffraction patterns offer traces that are interpreted to identify the minerals and crystal structures present in the Sansevieria fibre. XRD testing of Sansevieria fibres can provide an in-depth understanding of their mineral composition and crystal structure. This information is useful for comprehending the mechanical, thermal, and chemical properties of the fibres and can support the development of applications or manufacturing processes involving Sansevieria fibres.

2.5 TGA-DTA Analysis

Thermogravimetric (TGA) and Differential Thermal Analysis (DTA) are analytical techniques used to investigate the thermal properties and decomposition of SF. TGA measures the weight change of a sample as temperature increases, allowing for the identification of decomposition temperature, water loss, and mass changes associated with organic and inorganic components in SF. DTA analyzes the physical and chemical changes in SF samples by comparing the temperature difference between the sample under test and the same temperature over time. This temperature difference is recorded as a function of time or temperature and can provide information on the thermal properties, stability, and reaction kinetics of alkali-treated and non-alkali-treated SF samples. TGA and DTA testing are used to analyse the thermal properties of Sansevieria fibres, including their thermal stability, decomposition point, and thermal composition. This information can be useful in selecting SF materials for applications and developing manufacturing processes.

2.6 Surface Analysis

Morphological testing using Scanning Electron Microscopy (SEM) is employed to observe the surface structure and morphology of SF. The testing process involves transmitting electrons to the Sansevieria fibre sample and capturing images of the fibre surface using an electron detector. The result is a high-resolution, three-dimensional image that allows for detailed observation of surface structures, such as fibre morphology, porosity, and structural diversity. SEM morphology testing of Sansevieria fibres provides very useful visual information to understand the physical characteristics of SF fibres, including fibre size, pore distribution, and possible defects or contaminants.

2.7 Composite Production

The composites were created using the hand-lay-up method. The fibre and matrix compositions were determined using weight fraction calculations. The volume fraction of fibre to matrix is determined using the weight fraction calculation equation [20].

$$V_f = \frac{w_f / \rho_f}{w_f / \rho_f + w_m / \rho_m} \quad (1)$$

where W_f is fibre weight, ρ_f is fibre density, w_m is matrix weight, ρ_m is matrix density. V_f is the volume of fibre in percent. The volume fraction of the composite was made at 10%, 20%, 30%, 40%, 50%, and 60%. The composite molding tool is made of silent silicon to avoid sticking to the matrix. [9] The desired specimen size is 20 cm, 5 cm, and 1 cm thick according to the ASTM D638-03 standard [21].

2.8 Composite Molding

The printing process is carried out at room temperature (25 o C) without direct sunlight. The drying process is carried out in a room free from direct sunlight exposure, has sufficient air circulation, and has a standard humidity of 45%–64% (RH or relative humidity). The humidity of this room keeps the specimen in a stable condition to avoid the potential growth of microbes that can damage the fibre specimen [22].

2.9 Composite Specimen

Specimens are made with the ASTM E8 Tensile Test Standard, with specimen shapes according to ASTM D638-03. Its showed in Figure 1 [17,21].

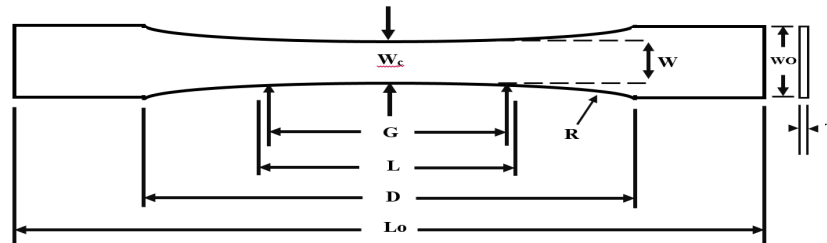


Fig. 1. Tensile Test Specimen

The tensile strength value is equal to the maximum tensile stress value obtained from Eq. (2):

$$\tau_{max} = \frac{F_{max}}{A} \quad (2)$$

where τ_{max} : Maximum tensile stress strength (N/mm²)
 F_{max} : Maximum tensile force (N)
 A : Cross-sectional area (mm²)

The tensile strain is calculated with the Eq. (3):

$$\varepsilon = \frac{\Delta L}{L_o} \quad (3)$$

where ε : Strain due to tensile force
 ΔL : Change in length (mm)
 L_o : Initial length (mm)

The tensile modulus of elasticity is calculated by the Eq. (4):

$$E = \frac{\sigma}{\varepsilon} \quad (4)$$

where E : Tensile modulus of elasticity (N/mm²)
 σ : Tensile stress (N/mm²)
 ε : Stretch

Tensile testing of the composite specimens was conducted at Politeknik Negeri Malang, a higher education institution in Malang known for its focus on vocational and technical education (POLINEMA) laboratory. Test specifications include accuracy $\pm 0.5\%$, elongation accuracy 0.001 mm, weight 1000 kg, poser AC 220V, 50 Hz, , display device = PC, and capacity 5000 kg.

3. Results and Discussion

3.1 Mechanical Properties of Single Fibre

The implementation of the single fibre specimen tensile test was carried out at the Sidoarjo Center for the Friendship Industry. A total of 50 specimens were taken to obtain accurate results. Single fibre tensile tests are carried out carefully, and considering that the fibre has low homogeneity, it requires accuracy in the selection of single fibre specimens to be tested in the laboratory [23]. Fibre selection is focused on getting fibres that have the best morphology and physical appearance, are flat, have the same dimensions, and are of uniform length. This is done to ensure the data is taken from the best and most homogeneous fibre specimens. The fibres used in this test have obtained a good homogeneity selection so that the data obtained can be calculated and analyzed properly. Tensile test measurements of the specimens obtained the following data (Figure 2).

In Figure 2, the tensile strength of single fibres resulting from alkali treatment provides varying data. KOH and NaOH, as strong bases, have a significant effect on tensile strength. Tensile strength decreased dramatically due to the alkali treatment. Alkaline solutions break down some of the fibre structure. This condition allows for a decrease in tensile strength. A considerable decrease occurred at alkali concentrations of 10% and 13%. The elevated alkaline concentration results in the elimination of the fibre's hemicellulose and lignin layer structures [24]. The erosion of the fibre surface layer due to alkalization causes residual stress in the rod. In addition, the reduction of part of the fibre structure will certainly reduce the mechanical strength. The surface hemicellulose contributes significantly to the tensile strength. The loss of some of the hemicellulose layer makes the fibre lose its strength and support on the outer layer. The removal of this cellulose layer is necessary in order to open the fibre pores for bonding with the matrix during composite molding. The surface layer, which consists of hemicellulose, can inhibit this bond between the fibre and matrix in the composite. To create an optimal composite structure, a strong bond between the cellulose (the main component of the fibre) and the polymer matrix is required. However, the hemicellulose layer on the fibre surface layer makes the matrix not bind perfectly with it. Hemicellulose is more hydrophobic, so it is difficult to bind with other elements, in this case, the matrix liquid. In addition to hemicellulose, the lignin structure within the cellulose pores can also impede the interaction between the matrix and the fibres in the composite. Alkali treatment also has the function of removing the lignin structure so that matrix and cellulose bonding can be maximized [25]. Excessive alkali treatment, both in concentration and duration of soaking or contact duration, will cause the decay of most cellulose, hemicellulose, and lignin structures. Figure 2 illustrates a decline in the tensile strength value resulting from increased alkali concentration. This shows that many fibre constituent structures are lost or decomposed. In CaOH (calcium hydroxide) alkali treatment, the tensile strength value tends to be higher. This is due to the fact that CaOH contains weak bases, so the CaOH-base reaction is not too large. In CaOH alkali treatment, the tensile strength value can still be maintained. At 7% CaOH alkali treatment, the tensile strength reached 12.16 N/mm².

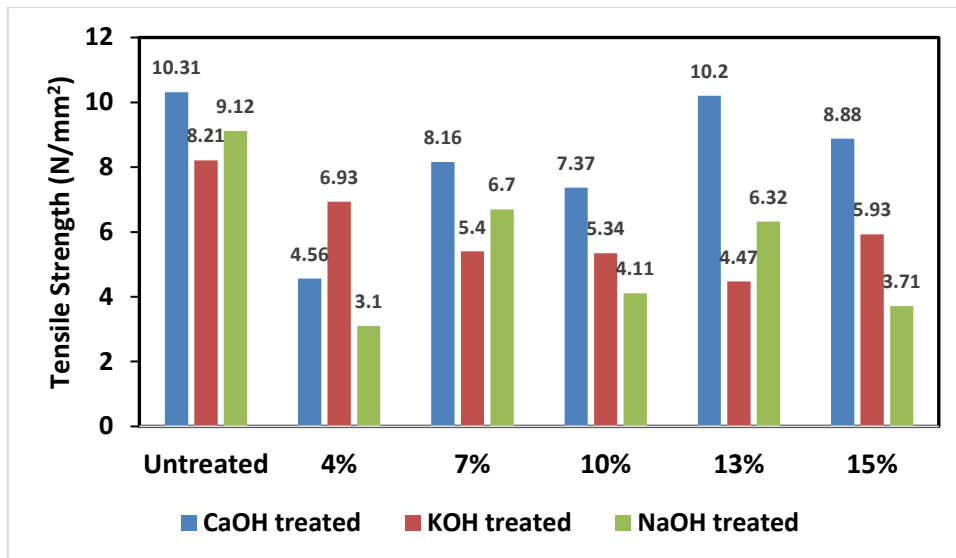


Fig. 2. Tensile Strength of untreated and alkali-treated single fibres

Elongation in fibres treated with 4 % alkali has the highest elongation value as shown in Figure 3. The limited number of hydroxyl groups in the alkaline solution's low base content hinders the formation of a strong chemical bond between the fibre surface and the solution, reducing the effectiveness of alkali treatment in enhancing fibre properties for composite applications. The elongation value indicates the material's capability to stretch without fracturing. A high elongation value makes the composite material have a high flexural test value. The measurements of fibre diameter and density were based on previous work. Sansevieria fibres were found to have a density of 1159 kg/m³ and an average single fibre diameter of 0.231 mm without alkali treatment. With a 5% NaOH alkali treatment, the density value increased to 1225 kg/m³ and the average diameter decreased to 0.115 mm for the treated fibres [26]. In a separate study, it was found that treating fibres with alkali can increase their tensile strength and elastic modulus, as well as improve the mechanical properties of *Acacia pennata* fibres. This indicates that alkali treatment has the potential to enhance the quality of natural fibres and broaden their applications in different fields, such as composite manufacturing [27], coconut fibre [28], jute fibre [29] and others. The study's sequence of work followed the process depicted in Figure 4.

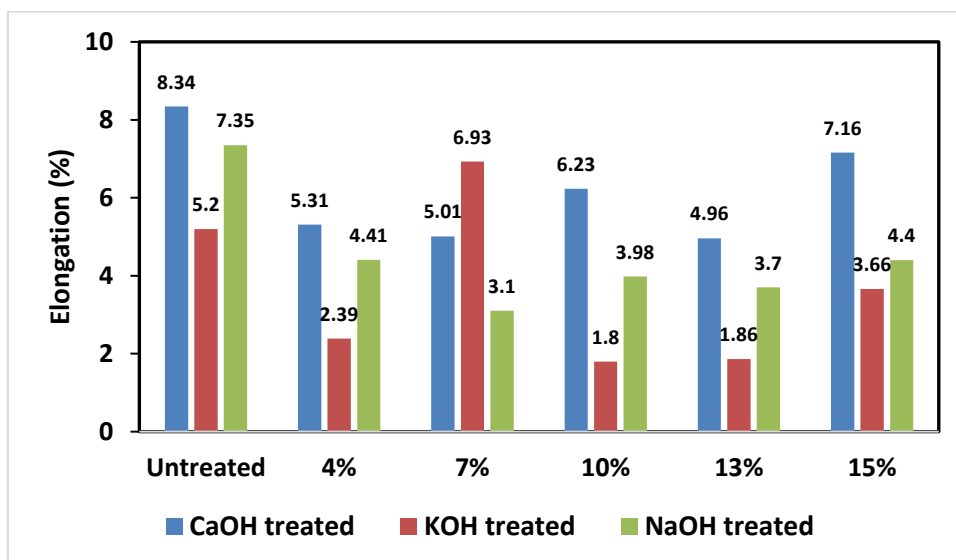


Fig. 3. Elongation of untreated and alkali-treated single fibres



Fig. 4. (a) Fresh sansevieria leaf; (b) Extracted Fibre of Sansevieria (c) NaOH alkalization processes; (d) Hand lay up Composite moulding (e) Composite of sansevieria fibre

3.2 Fibre Functional Group Analysis

Figure 5 compares the FTIR spectra of the alkali-treated SF and the untreated SF, highlighting their characteristic differences. Analysis of the FTIR spectra determined the functional group bands at $3600\text{--}3200\text{ cm}^{-1}$ and $2960\text{--}2819\text{ cm}^{-1}$. Both are associated with O-H, N-H [30] and CH hydrographic bond stretching vibrations [31]. The alkali-treated SF spectra showed identical peaks at 2807 , 2131 , 1904 , 1654 , 1505 , and 1302 cm^{-1} . The FTIR test results show a peak at 2807 cm^{-1} indicating the existence of CH stretching in the structures of cellulose and hemicellulose. This stretching is caused by a chemical bond between carbon and hydrogen atoms in the molecular chains of cellulose and hemicellulose. The similarity band at 1904 comes from the carbonaceous absorption ($\text{CO}=\text{}$) stretching of fatty acids found in the fibre [32]. The results show peaks at 1654 , 1505 , and 1302 cm^{-1} indicating the presence of aromatic skeleton vibrations ($=\text{CH}$) and CO stretching in the lignin structure. The CO stretching peak reflects the existence of carbonyl groups in the structure of lignin, while the aromatic skeleton vibrations ($=\text{CH}$) peak indicates the presence of single double bonds in the aromatic ring of lignin. Similar patterns were also found in previous studies in several studies. The bands at 1302 cm^{-1} can be attributed to C-H and O-H bending [26,31]. The small bands detected at 977 cm^{-1} and 891 cm^{-1} indicate the presence of glucosidic bonds in the structures of hemicellulose and cellulose. Glucosidic bonds are covalent bonds between hydroxyl groups on monosaccharide sugars, such as glucose, which form the polysaccharide chains of cellulose and hemicellulose. The detection of this band gives a strong indication of the existence of polysaccharide structures in SF samples. The FTIR spectra of SF treated with NaOH, CaOH, and KOH each showed significant changes in the shift of functional groups

towards untreated SF. All three have identical characteristics in the vibration of functional group band peaks. It can be seen that the 1755 cm^{-1} band peak seen in the untreated SF disappeared and shifted, indicating the elimination of hemicellulose and lumaic acid from the SF [3]. The high intensity of the treated SF is seen in the shift of the peak to 1654 cm^{-1} in CaOH alkali-treated SF. The decrease in band intensity after alkali treatment indicates a partial reduction of hemicellulose on the fibre surface and the removal of impurities. Alkali treatment of SF results in a decrease in band intensity in the FTIR spectrum due to the loss of non-cellulosic components such as lignin and hemicellulose, erosion of the fibre surface, and changes in the chemical structure of the fibre. This decrease in band intensity indicates changes in the chemical structure of SF fibres. However, the absence of new bands in the spectra indicates that no new functional groups are formed in the cellulose molecules during the alkali treatment process. Similar findings were found in bamboo fibre, while improvements remained on the surface of alkali-treated fibres [33].

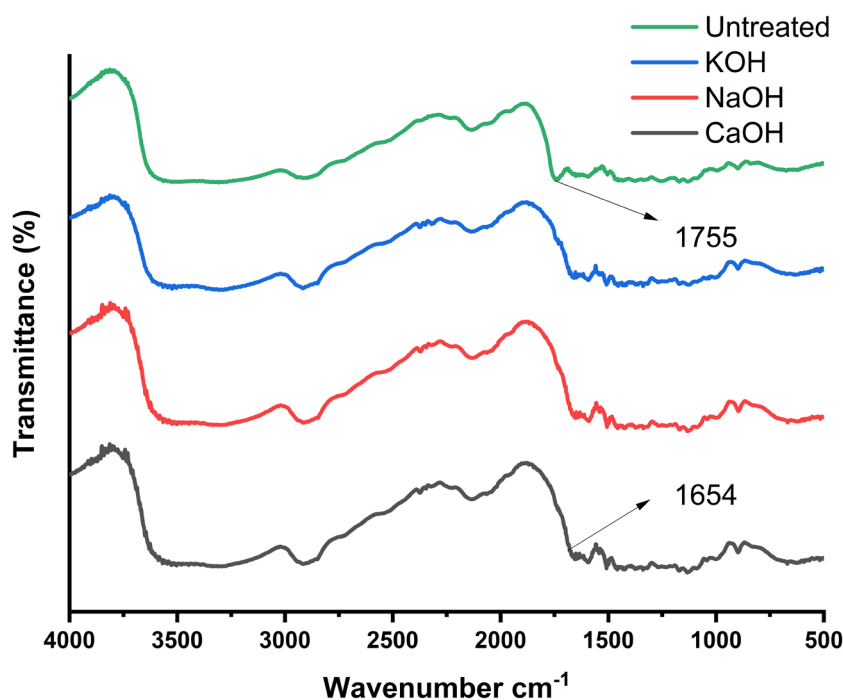


Fig. 5. FTIR spektrum of untreated and treated SF

3.3 Crystalline Properties Analysis

Figure 6 shows the x-ray diffractometry of NaOH, CaOH, and KOH alkali-treated SF fibres and untreated raw fibres. According to figure 5, the peaks at $2\theta = 160$ and 220 , respectively, appear from the cellulose (1 1 0) and (0 0 2) planes, as in previous research findings on *Cyrtostachys lace* fibres [34]. XRD tests revealed that prolonged exposure to alkali and high concentrations weakened the fibres due to damage to the cellulose crystal structure, polymer degradation, and changes in chemical structure. The crystallinity index increases with higher cellulose content, as cellulose has a more ordered crystal structure. The crystallinity index measures the proportion of crystals in the material structure, and cellulose is the main component in SF fibres that have a crystalline structure. A higher cellulose content indicates the presence of more crystals in the SF sample, which is reflected in an increase in the crystallinity index. The crystal structure of SF fibres is directly affected by the cellulose content, which determines the mechanical, thermal, and physical properties [9]. However, a high

base content causes a reduction in the crystallinity index. The alkaline hydrolysis process enhances the crystallinity index of SF fibres by purifying cellulose. The cellulose in SF fibres has a semi-crystalline structure that combines the strength and stiffness of the crystalline regions with the flexibility and elasticity of the amorphous regions. This results in fibres that are both strong and durable, yet flexible and adaptable to their environment. In contrast, hemicellulose and lignin have an amorphous structure. The alkaline hydrolysis process eliminates the amorphous cellulose content by dissolving it, thereby increasing the proportion of crystalline cellulose in the fibre. This increase in crystallinity index indicates a higher proportion of crystals in the cellulose structure. This can significantly alter their mechanical, thermal, and chemical properties [34]. This is because the ester and ether bonds contribute to increasing the cellulose content, which in turn increases the crystalline part of the cellulose structure. Cellulose is the dominant polysaccharide polymer in natural fibres and has a semi-crystalline structure consisting of ordered crystalline regions and disordered amorphous regions. Increased crystallinity enhances the strength and stiffness of the fibre, while also improving its thermal stability and chemical resistance. Ester and ether bonds, which are generally formed between cellulose polymer chains, can strengthen the crystalline structure and increase the proportion of crystals in the fibre. This is reflected in the XRD test results by an increase in the crystallinity index, indicating an increase in the proportion of crystals in the fibre structure. The ester and ether bonding that increases the cellulose content can significantly affect the mechanical, thermal, and chemical properties of SF fibres.

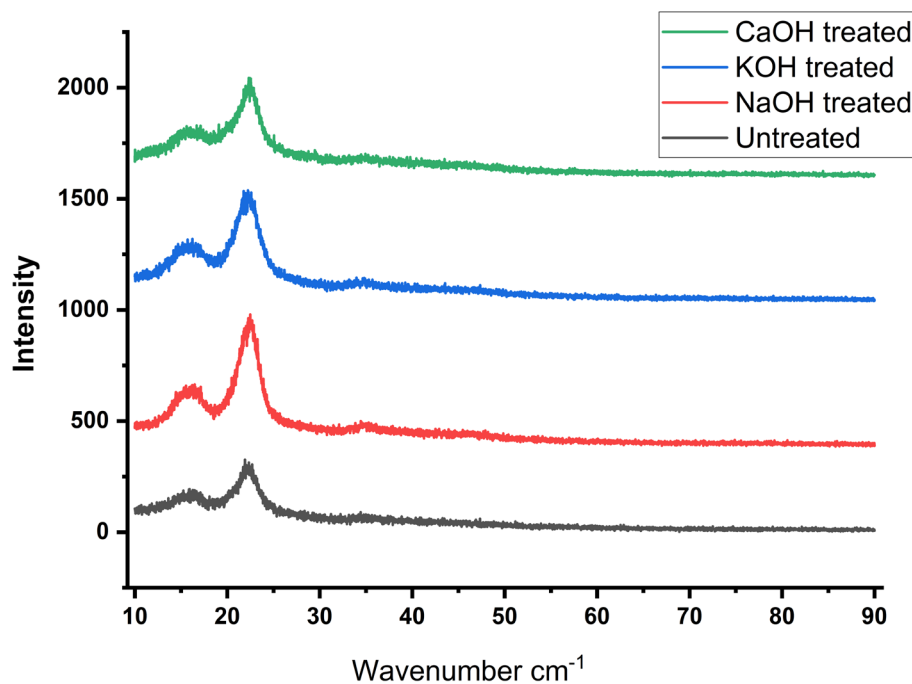


Fig. 6. XRD Analysis of untreated dan treated SF

3.4 Thermal Properties Analysis

Figure 7 shows the thermogravimetric curve of the sansevieria fibre. The TGV (thermogravimetric) curve shows the mass loss of SF fibres when subjected to temperature loading. The decrease in mass of SF samples between 30°C and 75°C is caused by the evaporation of water that is bound to the fibres. This water can be physically bound through adsorption or chemically

bound through hydrogen bonding. The heating process causes enough thermal energy to overcome the intermolecular forces that maintain water in liquid form. At this temperature increase, the water molecules begin to undergo more energetic translations and eventually break away from the sample surface in the form of vapor. This phenomenon provides data on the moisture and thermal stability of the SF sample and identifies the initial vaporization process that should be accounted for in further thermal analysis [35]. Untreated SF fibres started to decompose at 218 °C and ended at 428 °C. The CaOH-treated SF had a temperature stability value almost coinciding with the curve of SF without alkali treatment. This is because CaOH has a lower base value than NaOH or KOH. Thus, phisic changes due to the alkali hydrolysis process in the use of CaOH alkali did not have much impact on morphological changes or chemical changes in SF fibres. In the SF curves treated with NaOH and KOH alkali, it was seen that the SF fibre without treatment did not have the same character. The mass change process due to heating is recorded to have a sloping contour on the NaOH and KOH-treated SF fibres. Meanwhile, the curve of CaOH-treated SF fibre has a steep contour, indicating a considerable mass reduction when the SF fibre is subjected to temperatures from 30 °C to 100 °C. The thermal stability value of these fibres also increased. The decomposition of NaOH- and KOH-treated SF started at 226 °C, slightly higher than the decomposition of SF without alkali treatment. Differential thermogravimetric analytic (DTA) curves of thermal stability were observed in SF both with and without alkali treatment, as shown in Figure 8. Improved thermal stability is observed after the partial removal of hemicellulose, lignin, and impurities through alkali treatment. This process effectively eliminates non-cellulosic components, such as hemicellulose and lignin, and reduces the impurity content of the sample. The remaining material becomes more homogeneous and pure, resulting in improved thermal stability. The thermogravimetric curves reflect this phenomenon, showing either an increase in decomposition temperature or a slower decrease in sample mass when heated, indicating improved resistance to thermal degradation [36]. In NaOH-treated SF fibres, the decomposition occurred when the temperature reached 300 °C. The next peaks in CaOH and KOH-treated fibres occurred at 382 °C and 392 °C, respectively. These peaks correspond to the decomposition of cellulose and lignin [37]. The fibres treated with NaOH alkali showed the lowest residual weight, indicating optimal delignification. This process effectively removes most of the lignin from the fibre, which is a component susceptible to thermal degradation. Therefore, reducing the lignin content significantly improved the fibre's thermal stability. Based on this phenomenon, NaOH alkali treatment is considered an appropriate method to enhance the thermal stability and other properties of the fibres. This is because it produces less residue, resulting in purer fibres.

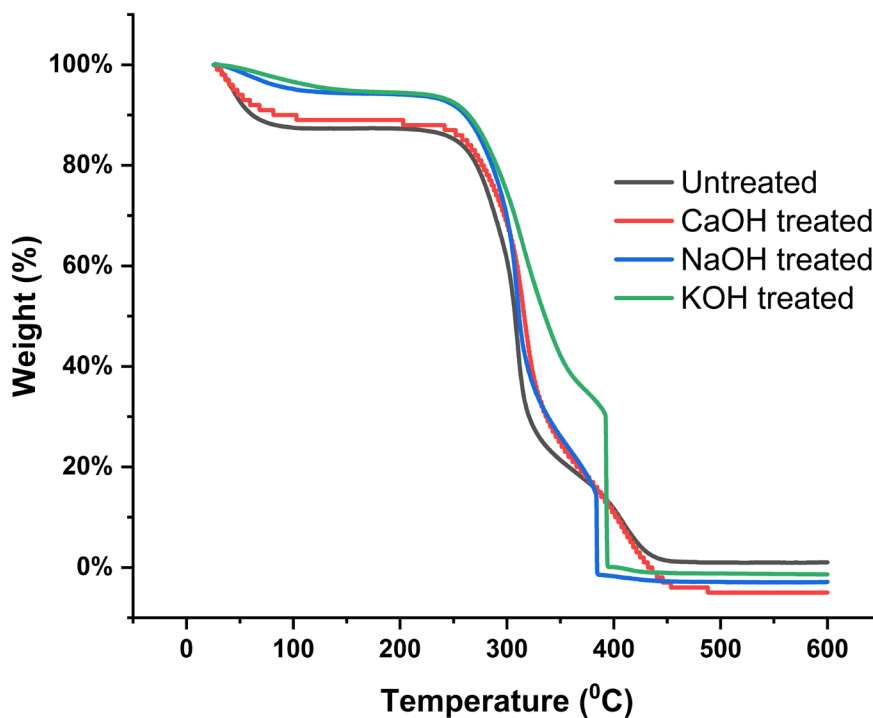


Fig. 7. Thermogravimetry Analitic/TGV of untreated dan treated SF

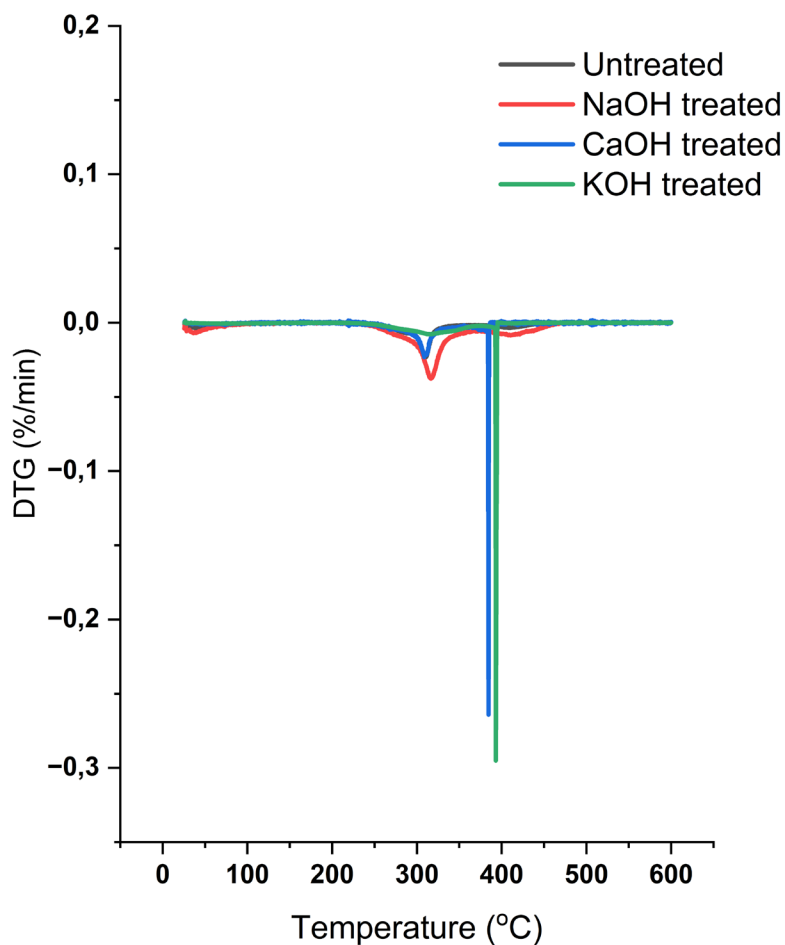


Fig. 8. Differential Thermogravimetry /DTA of SF treated and untreated

3.5 STF Surface Properties

SEM (Scanning Electron Microscopy) is used to analyse the SF morphology. Alkali treatment of SF was represented by NaOH alkalization of SF fibres with a 4 % alkali concentration. NaOH was assumed to be an illustration of the influence of bases that represent CaOH and KOH alkalis. Alkaline solutions, such as NaOH, KOH, and NaOH-KOH, significantly alter the morphology of SF fibres. These treatments effectively reduce hemicellulose and lignin content, increase surface accessibility, remove impurities such as wax and pectin, and improve fibre cleanliness and smoothness. The fibre surface layer is significantly reduced under 1000x magnification. The treatment clears the lignin surface, which normally covers the fibre, revealing the purer cellulose surface. The modification of the fibre structure is indicated by this change, resulting in the loss of the fibre surface's protective layer. This alteration can impact the fibre's mechanical, chemical, and physical properties. Figures 9 (1) and (2) show SF fibres without alkali treatment. The structure of SF fibres is characterized by elongated, rod-shaped granules that resemble blocks. Lignin and hemicellulose protect the cellulose structure. Lignin provides stiffness and strength to the fibres, while hemicellulose fills the spaces between cellulose fibrils and increases structural stability. Lignin and hemicellulose provide strength and protection to the fibres by coating and binding the cellulose fibres. This complex structure plays an important role in maintaining the integrity and strength of SF fibres. Figures 9 (3) and (4) show the changes in fibre surface morphology. The permeability of the parenchyma cells that make up the cellulose decays and causes erosion of the fibre surface structure [38]. Figure 9 illustrates that the alkali process, which removes hemicellulose and lignin, results in a rough and uneven fibre surface. The removal of hemicellulose and lignin, which normally fill the space between cellulose fibrils, exposes more of the fibre surface and causes it to become uneven. The wrinkles and cracks on the fibre surface cause the lignin to erode, increasing fibre stiffness but also making the fibre more prone to tangling and cracking. Additionally, the loss of hemicellulose and lignin, which are components of the fibre wall, causes the fibre diameter to decrease. Figures 9 (3) and (4) show the surface of the fibre that has been eroded. After the decomposition of lignin and cellulose, the outer layer of the fibre decays, resulting in the formation of holes in the protective layer. The impact of alkali treatment on fibre structure is evident in electron microscopy images, which show a rougher and more porous fibre surface. These changes significantly affect the fibres' physical and chemical properties, including increased water absorption, enhanced adhesion to the composite matrix, and improved mechanical strength and stiffness [39]. When cellulose fibres are exposed to air, changes occur on their surface, which makes them rougher. The microstructure created by the roughness of the fibre surface interlocks with the matrix, improving mechanical adhesion. Additionally, the increased fibre surface area provides more contact area between the fibre and the matrix, further enhancing mechanical adhesion. This improvement in mechanical adhesion results in better performance of natural fibre composites, particularly in terms of mechanical strength. The cellulose core of the fibre remains protected, maintaining its strong mechanical properties [40]. The removal of the surface layer on cellulose fibres enables direct exposure of the cellulose to the composite matrix, enhancing the wettability of the fibres. This, in turn, allows the cellulose to bind directly to the composite matrix, creating a stronger bonding structure. This process enhances the adhesion between the fibres and the matrix, facilitating the transfer of mechanical loads between the phases. The strengthened interface between the fibres and the matrix results in composites with improved strength and resistance to mechanical loads and external stresses. Optimising wettability and interfacial bonding greatly contributes to the improved performance of composite materials in various applications, from the automotive industry to construction.

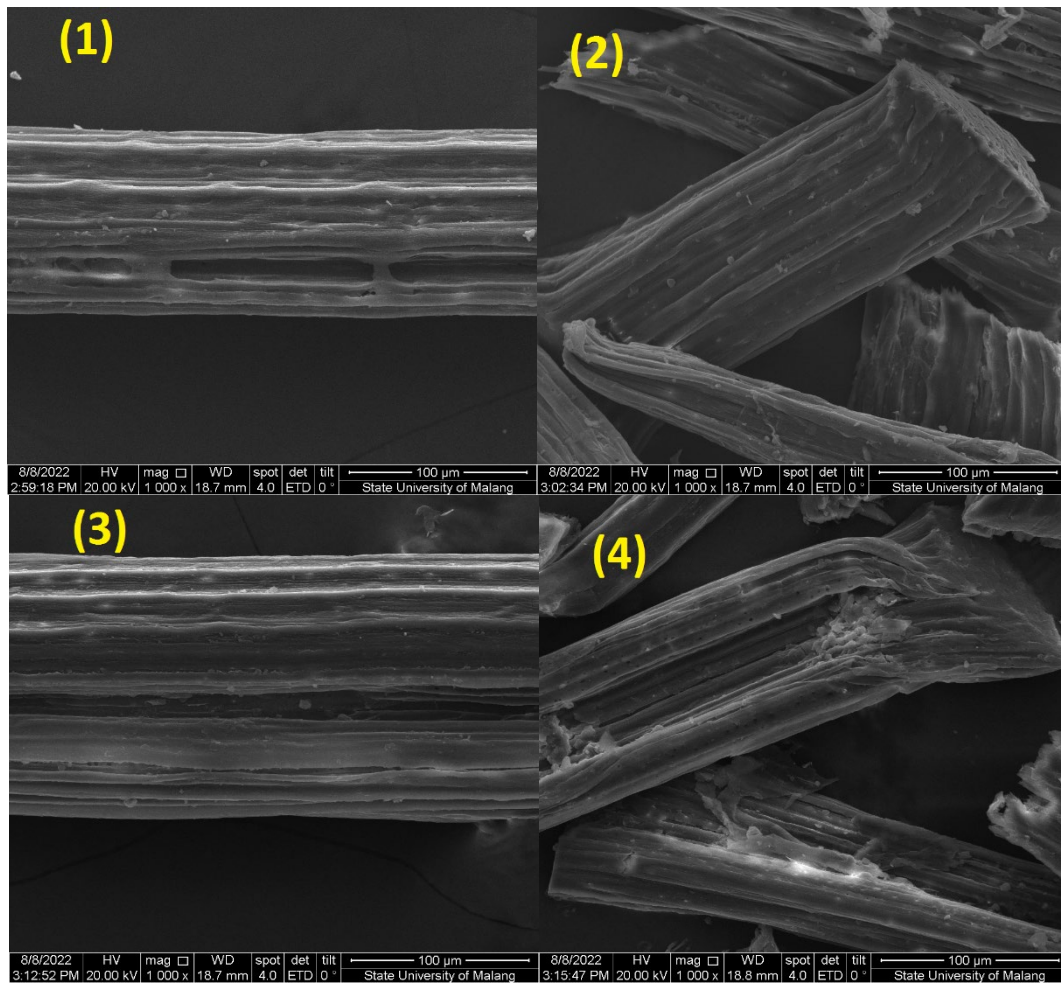


Fig. 9. Scanning Electron Microscope (1);(2) untreated SF; (3);(4) treated SF

3.6 Mechanical Properties of Composites Due to Changes in Weight Fraction

Composites were made by measuring their weight fraction. The calculation of weight fraction percentage is obtained by comparing the weight of fibre to resin using the respective density ratio in Eq. (1). The densities of fibre and resin have much different interval values, hence the need to use density ratios. The weight fraction equation serves to give a proportional percentage of fibre according to its density to the volume of resin, which has a much higher density. The results of the weight fraction calculation give the percentage of fibre volume in each specimen. Specimen 1 was 10%, specimen 2 was 20%, and so on up to specimen 7, with weight fractions of 30%, 40%, 50%, and 60%. The tensile testing results of the specimens were obtained according to the tensile strength graph in Figure 10.

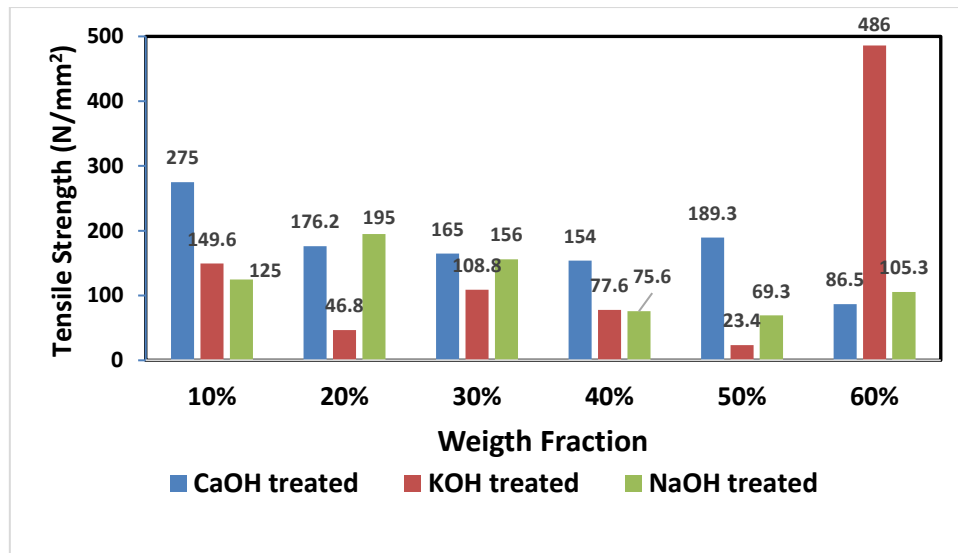


Fig. 10. Tensile Strength of composites by weight fraction

The tensile strength values of specimens 2 to 7 with different fibre alkali treatments can be observed in Figure 9. Composites using fibres treated with KOH alkali were found to have the highest value at a weight percentage of 60%. But the lowest is also owned by this specimen at a weight percentage of 50%. Other composites have values that tend to be uniform. The composite of KOH alkali-treated fibre has a non-uniform distribution of tensile strength. While the composites of NaOH and CaOH alkali fibres have different tensile strengths but have a relatively stable distribution. The tensile strength value of each weight fraction does not show a striking difference. While composites from KOH alkali-treated fibres have significantly different stability in each weight fraction presentation. Composites from fibres with CaOH alkali treatment have the highest value of tensile strength of 259.8 N/mm², while the lowest strength at 60% weight fraction percentage is 75.13 N/mm². In the alkaline fibre composite with NaOH solution, the highest value is 186 N/mm², and the lowest value is 68.14 N/mm² at a weight percentage of 60%. In composites using alkalized fibres from KOH solution, the maximum tensile strength of 486 N/mm² was achieved at a 60% weight fraction, whereas the minimum tensile strength of 46.8 N/mm² was recorded at a 20% weight fraction. Composites with CaOH alkali fibres showed the highest tensile strength overall, so they can be considered a good choice for applications with high strength requirements, while NaOH alkali treatment can be considered a more stable choice with a relatively consistent tensile strength distribution. NaOH is commonly used as an alkali in studies of jute fibre [41], bamboo [42], acacia tortilis [4] and other materials.

4. Conclusion

The difference in alkali in fibre treatment gives different characteristics to each fibre. CaOH alkali proved not to cause a decrease. The use of CaOH is highly recommended for alkaline treatments that minimize the partial dissolution of the parent material. CaOH has a lower ability to break down lignin and hemicellulose. This can be applied to natural fibres that are susceptible to an alkaline reaction. NaOH and KOH alkali treatments have a good ability to erode some of the fibre constituent materials. This can be seen from the results of the single fibre tensile test.

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Reference

- [1] Suharty, N. S., I. P. Almanar, K. Dihadjo, and N. Astasari. "Flammability, biodegradability and mechanical properties of bio-composites waste polypropylene/kenaf fiber containing nano CaCO₃ with diammonium phosphate." *Procedia Chemistry* 4 (2012): 282-287. <https://doi.org/10.1016/j.proche.2012.06.039>
- [2] Ashok, R. B., C. V. Srinivasa, and B. Basavaraju. "Dynamic mechanical properties of natural fiber composites—a review." *Advanced Composites and Hybrid Materials* 2 (2019): 586-607. <https://doi.org/10.1007/s42114-019-00121-8>
- [3] Sabarinathan, P., K. Rajkumar, V. E. Annamalai, and K. Vishal. "Characterization on chemical and mechanical properties of silane treated fish tail palm fibres." *International Journal of Biological Macromolecules* 163 (2020): 2457-2464. <https://doi.org/10.1016/j.ijbiomac.2020.09.159>
- [4] Dawit, Jonathan B., Yohannes Regassa, and Hirpa G. Lemu. "Property characterization of acacia tortilis for natural fiber reinforced polymer composite." *Results in Materials* 5 (2020): 100054. <https://doi.org/10.1016/j.rinma.2019.100054>
- [5] Ramanaiah, K., AV Ratna Prasad, and K. Hema Chandra Reddy. "Mechanical, thermophysical and fire properties of sansevieria fiber-reinforced polyester composites." *Materials & Design* 49 (2013): 986-991. <https://doi.org/10.1016/j.matdes.2013.02.056>
- [6] Widodo, Edi, Mulyadi Mulyadi, Prantasi Harmi Tjahjanti, and Fajar Syahril Kirom. "The sansevieria trifasciata fiber compatibility analysis for biocomposite reinforcement." *Turbo: Jurnal Program Studi Teknik Mesin* 10, no. 1 (2021). <https://doi.org/10.24127/trb.v10i1.1542>
- [7] Sreenivasan, V. S., D. Ravindran, V. Manikandan, and R. Narayanasamy. "Mechanical properties of randomly oriented short Sansevieria cylindrica fibre/polyester composites." *Materials & Design* 32, no. 4 (2011): 2444-2455. <https://doi.org/10.1016/j.matdes.2010.11.042>
- [8] Sreenivasan, V. S., D. Ravindran, V. Manikandan, and R. Narayanasamy. "Influence of fibre treatments on mechanical properties of short Sansevieria cylindrica/polyester composites." *Materials & Design* 37 (2012): 111-121. <https://doi.org/10.1016/j.matdes.2012.01.004>
- [9] Khandanlou, Roshanak, Mansor B. Ahmad, Kamyar Shameli, Mohd Zobir Hussein, Norhazlin Zainuddin, and Katayoon Kalantari. "[Retracted] Mechanical and Thermal Stability Properties of Modified Rice Straw Fiber Blend with Polycaprolactone Composite." *Journal of Nanomaterials* 2014, no. 1 (2014): 675258. <https://doi.org/10.1155/2014/675258>
- [10] Sreenivasan, V. S., S. Somasundaram, D. Ravindran, V. Manikandan, and R. Narayanasamy. "Microstructural, physico-chemical and mechanical characterisation of Sansevieria cylindrica fibres—An exploratory investigation." *Materials & Design* 32, no. 1 (2011): 453-461. <https://doi.org/10.1016/j.matdes.2010.06.004>
- [11] Rwwiire, Samson, and Blanka Tomkova. "Morphological, thermal, and mechanical characterization of Sansevieria trifasciata fibers." *Journal of Natural Fibers* 12, no. 3 (2015): 201-210. <https://doi.org/10.1080/15440478.2014.914006>
- [12] Sreenivasan, V. S., N. Rajini, A. Alavudeen, and V. Arumugaprabu. "Dynamic mechanical and thermo-gravimetric analysis of Sansevieria cylindrica/polyester composite: Effect of fiber length, fiber loading and chemical treatment." *Composites Part B: Engineering* 69 (2015): 76-86. <https://doi.org/10.1016/j.compositesb.2014.09.025>
- [13] Raju, Juvvi Siva Naga, Melvin Victor Depoures, and P. Kumaran. "Comprehensive characterization of raw and alkali (NaOH) treated natural fibers from *Symphirema involucratum* stem." *International Journal of Biological Macromolecules* 186 (2021): 886-896. <https://doi.org/10.1016/j.ijbiomac.2021.07.061>
- [14] Widodo, Teguh Dwi, Rudianto Raharjo, Redi Bintarto, Mirza Pramudia, Mohamad Irkham Mamungkas, and Arif Wahudiono. "Effect of alkalization treatment on the tensile strength and interface character matrix-fibber of bamboo Petung (*Dendrocalamus Asper*) reinforced polyester resin composite." In *IOP conference series: materials science and engineering*, vol. 494, no. 1, p. 012081. IOP Publishing, 2019. <https://doi.org/10.1088/1757-899X/494/1/012081>

- [15] Jaafar, Che Nor Aiza, Muhammad Asyraf Muhammad Rizal, and Ismail Zainol. "Effect of kenaf alkalization treatment on morphological and mechanical properties of epoxy/silica/kenaf composite." *Int. J. Eng. Technol* 7, no. 4 (2018): 258-263. <https://doi.org/10.14419/ijet.v7i4.35.22743>
- [16] Lassoued, Mouna, Taoufik Mnasri, Abdelmoumen Hidouri, and Rached Ben Younes. "Thermomechanical behavior of Tunisian palm fibers before and after alkalization." *Construction and Building Materials* 170 (2018): 121-128. <https://doi.org/10.1016/j.conbuildmat.2018.03.070>
- [17] ASTM International. "Standard Test Methods for Tension Testing of Metallic Materials1." *E8/E8M – 08*. 2000. <https://doi.org/10.1520/ignoring>.
- [18] ISO 113581. "INTERNATIONAL STANDARD ISO 6892-1." *International Organization for Standardization*. 2015.
- [19] Yang, Zengling, Linwei Cai, Lujia Han, Xia Fan, and Xian Liu. "Review of standards for near infrared spectroscopy methods." *Journal of Near Infrared Spectroscopy* 29, no. 6 (2021): 313-320. <https://doi.org/10.1177/09670335211042016>
- [20] Bommana, K., and G. C. Shanthakumar. "Study of sisal fibre attrition and characterization during direct extrusion compression moulding." *Materials Today: Proceedings* 5, no. 5 (2018): 13251-13257. <https://doi.org/10.1016/j.matpr.2018.02.316>
- [21] ASTM International D 638-03. "Standard Test Method for Tensile Properties of Plastics." *D 638 – 03 Standard*. 2000.
- [22] Aranda-García, Francisco J., R. González-Núñez, C. F. Jasso-Gastinel, and Eduardo Mendizabal. "Water absorption and thermomechanical characterization of extruded starch/poly (lactic acid)/agave bagasse fiber bioplastic composites." *International Journal of Polymer Science* 2015, no. 1 (2015): 343294. <https://doi.org/10.1155/2015/343294>
- [23] Mahzabin, Mst Sadia, Lim Jee Hock, Md Shabbir Hossain, and Lim Siong Kang. "The influence of addition of treated kenaf fibre in the production and properties of fibre reinforced foamed composite." *Construction and Building Materials* 178 (2018): 518-528. <https://doi.org/10.1016/j.conbuildmat.2018.05.169>
- [24] Widodo, Edi, and Prantasi Harmi Tjahjanti. "Characterization of Sansevieria Fiber with NaOH Alkalization to Increase Tensile Strength." In *IOP Conference Series: Earth and Environmental Science*, vol. 1104, no. 1, p. 012030. IOP Publishing, 2022. <https://doi.org/10.1088/1755-1315/1104/1/012030>
- [25] Gao, Chongfeng, Jie Yang, and Lujia Han. "Systematic comparison for effects of different scale mechanical-NaOH coupling treatments on lignocellulosic components, micromorphology and cellulose crystal structure of wheat straw." *Bioresource Technology* 326 (2021): 124786. <https://doi.org/10.1016/j.biortech.2021.124786>
- [26] Widodo, Edi, and Teguh Dwi Widodo. "Comprehensive investigation of raw and NaOH alkalized sansevieria fiber for enhancing composite reinforcement." *Case Studies in Chemical and Environmental Engineering* 9 (2024): 100546. <https://doi.org/10.1016/j.cscee.2023.100546>
- [27] Sheeba, KR Jaya, Jagadeesh Kumar Alagarasan, Jeyaprakash Dharmaraja, S. Anne Kavitha, Sutha Shobana, Sundaram Arvindnarayan, Manoharan Vadivel, Moonyong Lee, and Krishna Priya Retnam. "Physico-chemical and extraction properties on alkali-treated *Acacia pennata* fiber." *Environmental Research* 233 (2023): 116415. <https://doi.org/10.1016/j.envres.2023.116415>
- [28] Putra, Andi Erwin Eka, Ilyas Renreng, Hairul Arsyad, and Bakri Bakri. "Investigating the effects of liquid-plasma treatment on tensile strength of coir fibers and interfacial fiber-matrix adhesion of composites." *Composites Part B: Engineering* 183 (2020): 107722. <https://doi.org/10.1016/j.compositesb.2019.107722>
- [29] Saha, Prosenjit, Suvendu Manna, Sougata Roy Chowdhury, Ramkrishna Sen, Debasis Roy, and Basudam Adhikari. "Enhancement of tensile strength of lignocellulosic jute fibers by alkali-steam treatment." *Bioresource technology* 101, no. 9 (2010): 3182-3187. <https://doi.org/10.1016/j.biortech.2009.12.010>
- [30] Ong, Huei Ruey, Maksudur R. Khan, Abu Yousuf, Nitthiyah Jeyaratnam, and DM Reddy Prasad. "Effect of waste rubber powder as filler for plywood application." *Polish Journal of Chemical Technology* 17, no. 1 (2015): 41-47. <https://doi.org/10.1515/pjct-2015-0007>
- [31] Iskandar, Wan Mohd Eqhwan, Huei Ruey Ong, Md Maksudur Rahman Khan, and Ridzuan Ramli. "Effect of ultrasonication on alkaline treatment of empty fruit bunch fibre: Fourier Transform Infrared Spectroscopy (FTIR) and morphology study." *Materials Today: Proceedings* 66 (2022): 2840-2843. <https://doi.org/10.1016/j.matpr.2022.06.526>
- [32] Ong, Huei Ruey, Md Maksudur Rahman Khan, DM Reddy Prasad, Abu Yousuf, and M. N. K. Chowdhury. "Palm kernel meal as a melamine urea formaldehyde adhesive filler for plywood applications." *International Journal of Adhesion and Adhesives* 85 (2018): 8-14. <https://doi.org/10.1016/j.ijadhadh.2018.05.014>
- [33] Bai, Tian, Dong Wang, Jie Yan, Wanli Cheng, Haitao Cheng, Sheldon Q. Shi, Ge Wang, and Guangping Han. "Wetting mechanism and interfacial bonding performance of bamboo fiber reinforced epoxy resin composites." *Composites Science and Technology* 213 (2021): 108951. <https://doi.org/10.1016/j.compscitech.2021.108951>
- [34] Loganathan, Tamil Moli, Mohamed Thariq Hameed Sultan, Qumrul Ahsan, Mohammad Jawaid, Jesuarockiam Naveen, Ain Umaira Md Shah, and Lee Seng Hua. "Characterization of alkali treated new cellulosic fibre from

- Cyrtostachys renda." *Journal of Materials Research and technology* 9, no. 3 (2020): 3537-3546. <https://doi.org/10.1016/j.jmrt.2020.01.091>
- [35] Benítez-Guerrero, Mónica, Jorge López-Beceiro, Pedro E. Sánchez-Jiménez, and José Pascual-Cosp. "Comparison of thermal behavior of natural and hot-washed sisal fibers based on their main components: Cellulose, xylan and lignin. TG-FTIR analysis of volatile products." *Thermochimica Acta* 581 (2014): 70-86. <https://doi.org/10.1016/j.tca.2014.02.013>
- [36] Aristri, Manggar Arum, Muhammad Adly Rahandi Lubis, Raden Permana Budi Laksana, Rita Kartika Sari, Apri Heri Iswanto, Lubos Kristak, Petar Antov, and Antonio Pizzi. "Thermal and mechanical performance of ramie fibers modified with polyurethane resins derived from acacia mangium bark tannin." *journal of materials research and technology* 18 (2022): 2413-2427. <https://doi.org/10.1016/j.jmrt.2022.03.131>
- [37] AL-Oqla, Faris M., Mohammed T. Hayajneh, and M. Mu'ayyad. "Thermal stability analysis and mechanical performance of Mediterranean lignocellulosic fiber reinforced polypropylene sustainable composites." *Journal of Industrial and Engineering Chemistry* 134 (2024): 231-243. <https://doi.org/10.1016/j.jiec.2023.12.053>
- [38] Chen, Hong, Jieyu Wu, Jiangjing Shi, Wenfu Zhang, and Hankun Wang. "Effect of alkali treatment on microstructure and thermal stability of parenchyma cell compared with bamboo fiber." *Industrial Crops and Products* 164 (2021): 113380. <https://doi.org/10.1016/j.indcrop.2021.113380>
- [39] Manimaran, P., P. Sentharamaikannan, M. R. Sanjay, M. K. Marichelvam, and Mohammad Jawaid. "Study on characterization of Furcraea foetida new natural fiber as composite reinforcement for lightweight applications." *Carbohydrate polymers* 181 (2018): 650-658. <https://doi.org/10.1016/j.carbpol.2017.11.099>
- [40] Madhu, P., M. R. Sanjay, Mohammad Jawaid, Suchart Siengchin, Anish Khan, and Catalin Iulian Pruncu. "A new study on effect of various chemical treatments on Agave Americana fiber for composite reinforcement: Physico-chemical, thermal, mechanical and morphological properties." *Polymer Testing* 85 (2020): 106437. <https://doi.org/10.1016/j.polymertesting.2020.106437>
- [41] Balan, G. Sakthi, and M. Ravichandran. "Study of moisture absorption characteristics of jute fiber reinforced waste plastic filled polymer composite." *Materials Today: Proceedings* 27 (2020): 712-717. <https://doi.org/10.1016/j.matpr.2019.11.260>
- [42] Sánchez, Martha L., William Patino, and Jhon Cardenas. "Physical-mechanical properties of bamboo fibers-reinforced biocomposites: Influence of surface treatment of fibers." *Journal of Building Engineering* 28 (2020): 101058. <https://doi.org/10.1016/j.jobbe.2019.101058>