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*Review*

# Effect of Mercerization/Alkali Surface Treatment of Natural Fibres and Their Utilization in Polymer Composites: Mechanical and Morphological Studies

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**Abstract:** Environmental pollution, such as air, water, and soil pollution, has become the most serious issue. Soil pollution is a major concern as it generally affects the lands and makes them non-fertile. The main cause of soil pollution is agro-waste. It may be possible to mitigate the agro-waste pollution by re-utilizing this agro-waste, namely natural fibres (NFs), by blending into polymer-based material to reinforce the polymer composite. However, there are pros and cons to this approach. Consequently, the polymer composite materials fabricated using NFs are inferior to those polymer composites that are reinforced by, e.g., carbon or glass fibres from the mechanical properties' perspectives. The limitations of utilizing natural fibres in polymer matrix are their high moisture absorption, resulting in high swelling rate and degradation, inferior resistance to fire and chemical, and inferior mechanical properties. In particular, the NF polymer composites exhibit inferior interfacial adhesion between the fibre and the matrix, which, if improved, ultimately overcome all the listed limitations and improve the mechanical properties of the developed composites. To improve the interfacial adhesion leading to the enhancement of the mechanical properties, optimum chemical treatment such as Alkalization/Mercerization of the fibres have been explored. This article discusses the Mercerization/Alkali surface treatment method for NFs and its effects on the fibres regarding the Mercerization/Alkali surface treatment method for NFs and its effect on the fibres regarding their utilization in the polymer composites, the morphological features, and mechanical properties of composites.

**Keywords:** polymer composites; surface treatment; Mercerization/Alkalization; mechanical properties; morphological properties; natural fibre



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## 1. Introduction

The main focus of this article is to discuss the difficulties that arise during the development of polymer composite materials reinforced by natural fibres (NFs), known as natural fibre-reinforced composites (NFRC). The main problem in the development of the NFRC is the hydrophilicity of the fibres. Hydrophilicity causes reduced adhesion ability and ultimately affects the mechanical properties of the composites. Chemical treatments are the most effective as they could remove the wax content, impurities from the fibre surface, resulting in a rough and hydrophobic surface. Chemical treatment improves the adhesion ability of the natural fibres with the polymer matrix and improves the mechanical properties of the composites. The polymers can be thermosets, thermoplastics, and biopolymers.

The benefits of using natural fibres (NFs) such as Coir, flax, hemp, and bast fibres as a reinforcement in polymer composites than synthetic fibres, are low cost, lower density, more recyclable and have greater "biodegradability" [1].

To give an idea of the mechanical properties of NF, we note that NFs possess strength, σ, ranging 99.8 ± 22.5 MPa (COIR fibres) to 639.5 ± 301.6 MPa (PALF fibres), and stiffness, E, ranging 0.5 ± 0.1 GPa (COIR fibres) to 7.1 ± 3.1 GPa (PALF fibres; NFs possess fracture strain, ε, ranging 0.11 ± 0.03 (PALF fibres) to 2.0 ± 1.3 (OPEFB fibres) [2]. Additionally of note is that, owing to the microscopic nature of these fibres, dedicated micromechanical testers capable of testing single fibre one at a time, have been used in evaluating the mechanical properties of the NF [3]. In general, NF consist of cellulose, hemicellulose, lignin pectin and waxy content [4]. From the microstructural point of view, the hierarchical structure of the fibre is very complex. The fibre is distributed in two cell walls; the outer cell wall consists of a single layer. The other comprises three layers organized as a concentric cylinder with a lumen in between [5]. However, it has been observed that the bio-composites developed by using natural fibres and conventional polymers (whether thermosets or thermoplastics) are not adequately environmentally friendly, as their matrix is non-biodegradable. Thermoplastics are now surpassing thermosets because of their recyclability and easy repairability.

Alkali treatment is used to breaking down the bundles of fibres into individual fibres. This process results in an increased aspect ratio of the smaller fibre particles and makes the fibre surface rough, which helps in increasing the interfacial bonding between fibre and the matrix material [6].

Hashim and co-workers have reported that alkali treatment or mercerization is the most effective chemical treatment method for enhancing the adhesion ability of the natural fibres with the polymer matrix. They performed the alkalization on the kenaf fibre. Three parameters they have selected for the alkali treatment. These are concentration level of the solution, immersion time of fibres in the solution, and immersion temperature. In this study, alkalization of the kenaf fibres is done at different concentrations, such as 2, 6, and 10 ($w/v$%) and at different temperatures (immersion) of 27, 60, and 100 °C. A small increase in the density of kenaf fibres was observed as compared to the untreated kenaf fibres. Additionally, a decrease in the diameter of treated kenaf fibre was observed than the untreated one [7].

Navin chand and co-workers reported that mercerization or alkalization changes the fibre structure, its dimensions, and improves mechanical properties. In this treatment, NaOH is used for the cleaning of the fibre surface and changes the fine structure of cellulose from cellulose I to cellulose III by depolymerization and developed short crystallites. The primary function of alkalization/mercerization lies in the disruption of the hydrogen bonds from the structure and making fibre surface rough for better compatibility [8].

Alkali treatment is an effective surface treatment method, helpful in removing some amount of lignin, impurities, which covers the fibre surface, depolymerize the cellulose structures [9]. The main problem in the development of the NF composite is their less adhesion ability with the matrix. Mercerization is one of the best ways to improve fibre-matrix adhesion [10]. Because of the hydrophilic nature of the fibre, Chemical treatment is found to be one of the great solutions that make the fibre surface rough and removes fatty deposits from the fibre called "xyloses". Alkali treatment results in the rough fibre surface and enhances the fibre surface area, leading to the excellent interlocking with the matrix [11]. A loss in mechanical properties was reported because of the fibre hydrophilic nature, which influences the adhesion to a hydrophobic matrix [12]. The hydrophobic nature of the fibre can be achieved by the different types of the chemical surface treatments. These are Alkalization, Acetylation, Anhydrides, Isocyanate, Permagnate, graft copolymerization and Silane coupling agents, etc. An improvement in the tensile properties (13%) was noticed by performing Alkalization on coir fibres (2 wt. %) over untreated fibre composites [13].

Nguyen and co-workers in 2010 reported that chemical treatment improves the contact surface area of bamboo fibres, providing a uniform dispersion of the fibre into the matrix. They also reported that alkalization enhances the tensile properties and the impact values of the composites [14].

Since the plants and polymer matrix have different chemical structures, coupling could be a major problem between the different phases, causing inadequate stress transfer at the composite interfaces. To avoid this, certain chemical treatments need to be performed on the fibre surface [15]. The alkaline process known as mercerization, is an efficient process that enhances the lignocellulosic fibres reinforced composite properties. The alkalization changes the colour from brown to dark brown, which shows that the anticipated modification has been achieved [16]. Jayabal and co-workers developed a composite using coir and optimize the alkali percentage concerning soaking time. They observed that 5% aqueous solution at 72 h increases tensile strength by 31% [17]. Jiang and co-workers reported that the alkali treatment ultimately reduces the spiral angle and improves the molecular orientation [18].

The interface plays a major role in deciding and establishing the strength and toughness of the fibre-reinforced composites. A stronger interface develops a material that shows the perfect strength and stiffness of the composite [19]. Another study performed by the Padmavathi and Naidu [20] reported that alkali treatment (18 wt %) showed improved mechanical properties such as tensile, impact strength and compression strength. Natural fibre treatment using alkalization/mercerization agitates the hydrogen bonding in the structure, and makes the fibre surface rough. These results demonstrate the excellent adhesion ability between the fibre and the matrix [21]. The modification of the fibres led to a change in the fibre surface properties and enhanced their adhesion ability with various matrix [22]. Ahmad and co-workers [23] reported that natural fibres generally have strong polar characteristic, which causes the incompatibility between the fibre and the matrix. To solve this, one needs to perform the chemical treatment on the fibres. Alkali treatment, an effective treatment amongst the available one. Shah and co-workers [24] performed the alkaline treatment on the bamboo fibres obtained by the stem explosion methods with various dissolved solid contents of 4, 6 and 8%. An enhancement in the tensile properties was observed with the highest alkaline treatment. Budtova and co-workers [25] reported that alkali perforates into the amorphous region resulting in the swelling of the cellulose fibres. The polymer chain then diffuses the alkali in the lateral direction and forms an alkali complex. After that, the alkali perforates into the crystalline region and develops an antiparallel crystalline soda complex known as Na-Cell II. Jamir and co-workers reported that the hydrophilic nature of the fibres resulted in higher moisture absorptions, which is a disadvantage of the natural fibres if considered for the composite development. A noticeable amount of H-bonds are available on the fibre surface and produce new H-bonds with water molecules, and moisture is absorbed by the fibre making a weak bonding between the fibre and the matrix [26]. Oladele and co-workers performed the alkali treatment on the bagasse fibres and reinforced it with the $CaCO_3$ into polypropylene and noticed that mercerization enhances the adhesion strength of the fibres with the matrix which ultimately enhances the mechanical properties [27]. The alkalization is a process which removes the waxy and oily contents from the fibre surface and makes it cleaner and increase roughness of the fibre. Roughness of the fibres provides greater surface area and improves the compatibility of the fibres with the matrix [28].

Sahu and Gupta have reported that because of the hydrophilicity of the natural fibres, it shows poor compatibility with the matrix material. Poor compatibility ultimately affects the composites' durability and mechanical properties, which is undesired in natural fibre composites applications. So, this hydrophilic nature of fibres is removed by chemical treatments. Alkalization/mercerization is the most effective and efficient chemical treatment which makes the fibre hydrophobic. It also removes the waxy contents, such as lignin, hemicellulose, pectin, in a small amount. Chemically treated fibre can be utilized to develop green composites, having distinct applications in various fields/areas.

In this article, various types of natural fibres, processing techniques of the natural fibre-reinforced composites, and chemical modification such as alkali treatment to improve the adhesion strength with the polymer matrix are reported. Additionally, the various mechanical and morphological properties if the natural fibre composites are also reported in the current chapter.

## 2. Natural Fibre Properties: A Brief Discussion

Natural fibres are an excellent replacement for the conventional fibres because of their high mechanical strength; they are the good options in many applications such as building and constructions, furniture, automotive, etc. The main problem of utilizing these fibres comes with their adhesion ability. Because of their hydrophilic nature, they are inferior to adhesion ability with the hydrophobic matrix. To solve this problem, various types of physical and chemical treatments are available. However, the chemical treatments are observed to be a good option to make them hydrophobic. Alkali treatment is the effective treatment that removes the wax and oil contents from the fibre surface and makes them rough, and enhances the adhesion strength with the matrix. Numerous natural fibres are available which are having excellent mechanical properties. Figure 1 shows the classification of natural fibres.

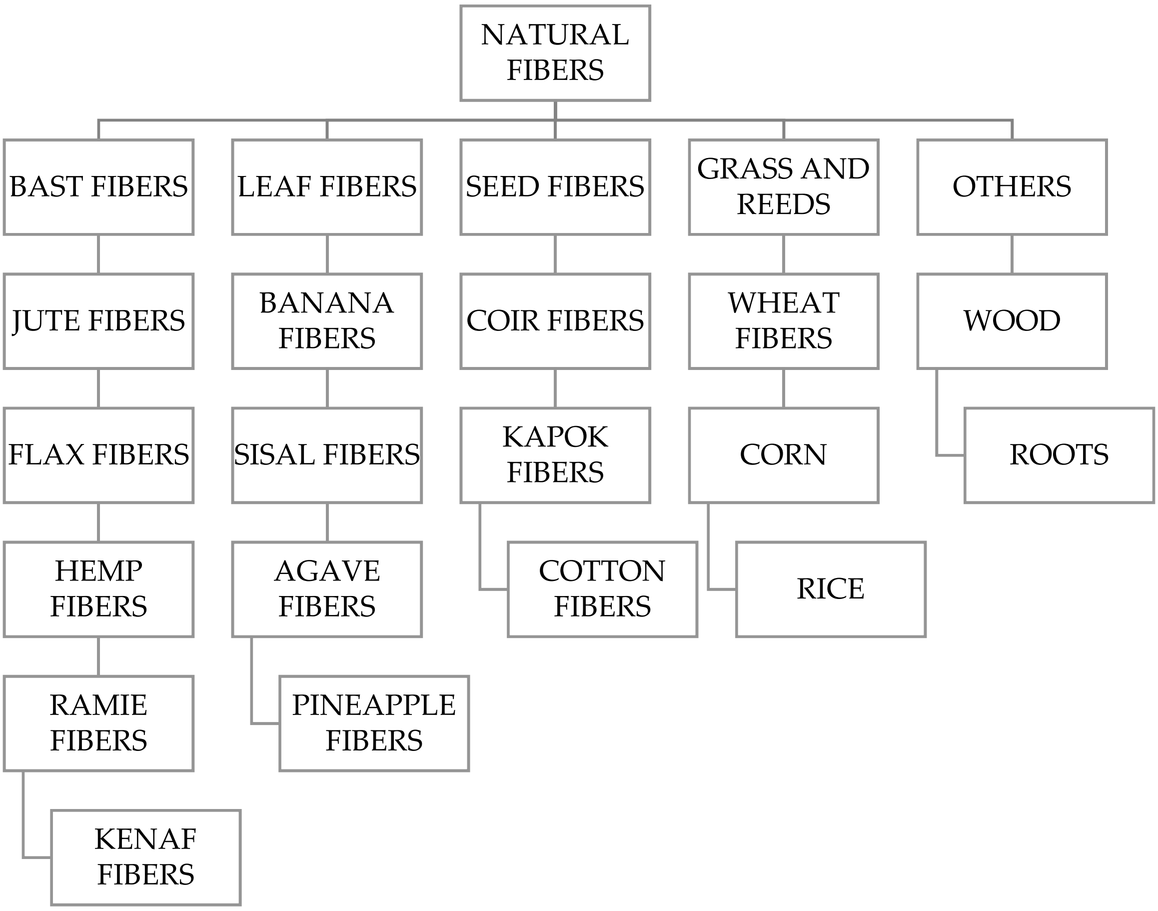



**Figure 1.** Classification of natural fibres.

### *2.1. Bast Fibres*

These fibres are found in the plant's inner bark and give structural strength and rigidness to the stem of the plant. These fibres remained as the bundles of the fibres and are placed parallel to the stem. The dimension of the bast strands varies, i.e., the length of the strands is up to 100 cm, and the width is about 1 mm [29].

- Hemp Fibre: Hemp belongs to the class of Genus cannabis. Globally, hemp is considered to be an anciently harvested fibre. The hemp fibre height is approximately between 1.2 to 5 m, and the diameter is approximately 4–20 mm [30].

- Ramie Fibres: Ramie comes under the nettle family Urticaceae. This plant grows in a hot and humid environment. The height of this plant is about 1–2.5 m.
- Flax Fibre: This fibre comes under the family of the Linaceae. This is utilized to make Linen. The fibre length varies from 9 to 70 mm.
- The width varies from 5 to 38 µm.
- Kenaf: It comes under the class of Hibiscus. The plant has a woody base, and its height varies from 1.5 to 3.5 m. The diameter of the stem varies from 1 to 2 cm. The length and width of the fibre vary from 2 to 6 mm and 14 to 33 µm [31]
- Jute: Jute comes under the Corchorus family. It comes in the second position by considering the amount produced after the cotton. The jute plants are around 2.5 to 3.5 m long. The fibres generally have 2 to 5 mm in length, and the width varies from 10–25 µm [32].

### *2.2. Leaf Fibres*

The following are fibres that can be acquired from plant leaves:

- Pineapple: It belongs to the family of Ananas comosus. The height of this plant varies from 1 and 1.5 m. Additionally, the leaves of this plant are around 30–100 cm long. The length of the ultimate fibre is approximately 61.7 mm, and the width is around 20 µm.
- Sisal: It belongs to Agave Sisalana, having sword-shaped leaves of around 1.5 to 2 m long. The width of the leaf is approximately 10–15 cm, and thickness is around 6 mm. The sisal fibre is around 1–8 mm long, and the width varies from 8 to 41 µm [29].

### *2.3. Seed Fibres*

- Cotton: Cotton belongs to the Gossypium family. The height of the plant varies from 0.5 to 1.5 m. Cotton fibres are categorized into two groups Lint fibres and Linters Fibres. Lint fibres are smooth fibres, and Linters are shorter fibres, i.e., around 2–7 mm in length. The fibre length varies from 15 to 40 mm, and the width varies from 15 to 40 µm.
- Kapok: It belongs to the family of Ceiba Pentandra. The length of the tree is around 60–70 m and hasa diameter of around 3 m. The length of the leaves is approximately 20 cm. Because of the buoyant nature and water-resistant property of the kapok fibre, these are mostly utilized to produce life jackets [33].
- Coir: The origination of the coir comes from the coconut palm tree. The coir husk fibres are placed between the husk and the outer shell of the coconut. Two types of coir fibres available, one is white fibre, and the other is brown fibre. White fibre is flexible compared to brown fibre, as it is obtained from the immature coconuts and has low lignin content than brown fibre. The length if the coir fibre is around 1 mm, and the width varies from 10 to 20 µm [29].
- Rice Hulls: Rice belongs to the Oryza Sativa family, which can be utilized to develop stem and hull fibres. The rice hulls are brittle and can be utilized to reinforce the thermoplastics to develop the particleboards [29].

## 3. Mercerization/Alkali Surface Treatment: An Effective Way for Natural Fibre Surface Treatment

The surface treatment of NFs to enhance adhesion between the NF and the polymer matrix underpins an important concept in the stress transfer mechanism [34]. The presence of strong adhesion can generate stresses at the interface by the process of ‘shear lag’, and this enables elastic stress transfer to occur between the NF and the matrix [35,36]. The presence of weak adhesion would generate stresses at the interface by the process of ‘shear sliding’, giving rise to plastic stress transfer between the NF and the matrix [36,37].

The Alkali/Mercerization treatment of the NF surface is considered an effective treatment amongst the other available chemical treatments. A sodium hydroxide (NaOH) solution is utilized in this treatment, which reacts with the available OH groups in the cellulose and partially removes the lignin, pectin and hemicellulose, and waxy and oils from the surface of the fibre and provides enhanced adhesion strength. The treatment of the natural fibres by Sodium hydroxide (NaOH) causes advances in the ionization of the OH groups to the alkoxide [1]. The chemical equation (Equation (1)) can be understood as follows:

$$\text{Fibre} - \text{OH} + \text{NaOH} \rightarrow \text{Fibre} - \text{O} - \text{Na} + \text{H}_2\text{O} \quad (1)$$

Mishra and co-workers perform the alkali treatment on the jute fibres and the sisal fibres by utilizing 5 wt. % NaOH solution up to 72 h. Researchers noticed that mercerization enhances the amorphous cellulose content by decreasing the crystalline cellulose [38]. Researchers also concluded that the effect of the mercerization or alkali treatment results in the improving the roughness of the fibre leading to the excellent adhesion ability with the matrix and the improvement of the cellulose content "on the fibre surface [39]". Budtova and co-workers [25] reported that alkali perforates into the amorphous region resulting in the swelling of the cellulose fibres. The polymer chain then diffuses the alkali in the lateral direction and forms an alkali complex. After that, the alkali perforates into the crystalline region and develops an antiparallel crystalline soda complex known as Na-Cell II. Alkali treatment ultimately improves the adhesion strength between the fibre and the hydrophobic matrix leading to the improvement in the various mechanical properties, specifically tensile strength. Jayabal and co-workers [40] reported that by performing the 5 wt. % alkali treatment on the coir fibres increases tensile strength values (approx. 31%). Much more research reported that the chemical treatment ultimately enhanced the composite's mechanical properties.

## 4. Various Fabrication Methods of the Polymer Composite Development

Fabrication method selection for the development of the polymer composite is the major task. It depends on the various parameters such as type of the matrix used, types of the reinforcement, i.e., short or long fibres, particulate type, woven mat, and non-woven mat, etc. A brief description of some of the specific processing techniques of polymer composite development is described as follows:

### *4.1. Hand Lay-Up*

This technique comes under the "open moulding technique" for the fabrication of composites. In this technique, woven form or knitted form fibres are placed in a mould, and the matrix is applied over these fibres using the brush. After that, a roller is utilized to improve the interactivity between the fibres or and the matrix, provide uniformity of the resin, maintain the composite's thickness, and maintain the composite's thickness.

### *4.2. Vacuum Infusion Technique*

This is the extended version of the hand lay-up technique in which the pressure is put in to improve the unification. This can be done by providing a plastic sheet over the reinforcement. In this technique, a vacuum pump is provided, which applied a 1 atm pressure to the reinforcement for the unification. The most suitable resin for this technique is the epoxy and the phenolic resin.

### *4.3. Resin Transfer Moulding*

This is a technique for the development of the parts made by fibre plastics composites. A reaction resin is applied onto the dry, in-process fibre components and is accordingly soaked by applying pressure within a closed vessel.

### *4.4. Compression Moulding*

This technique is one of the best techniques for developing composites due to its reliability and less cycle time. The two techniques come under this are compression and flow compression technique. This technique comprises compressing material consisting of temperature-instigated catalyst in a heated die that utilizes press (either cold press or hot press) [26].

### 4.5. Filament Winding

This technique is specifically used for the development of cylindrical composite's tubes. This technique consists of a filament winding machine, fibre creel unit, an impregnating drum which is placed in between the winding machine and the drum. A roller is also provided at the top of the drum to press the yarn [41].

## 5. Mechanical Properties of the Mercerized Natural Fibre Composites

Zizumbo et al. 2011 [42] used alkalized (12 wt. %) bagasse fibres and observed that the young's modulus of composites had become 1800 MPa, which is greater than the untreated bagasse fibre composites. This is due to the high surface area available after eliminating hemicellulose and lignin, which enhances the composites' mechanical properties.

Sarkar et al. 2001 [43] performed an alkali treatment of 5 wt. % on jute fibres for different hours such as 2, 4, 6, and 8 h at 30 °C. They observed that the mechanical properties of the composite are increase for the period of 4 h at most of the fibre contents. It has also been noticed that increasing the fibre content also affects the mechanical properties of the composite. At 4 h period, the composite showed good mechanical properties, as the fibre has become rigid and brittle leading to the evolution of the crystallinity, which provides greater strength.

Thamae et al. 2007 [44] performed the alkali treatment on Agave Americana fibres and showed the fibre tensile properties using the Weibull statistics. Weibull distribution showed fibre strength distributions. Additionally, when these fibres are treated with NaOH, then it shows an improvement in characteristic strength by 30%. This is because of the removal of the impurities from the fibre surfaces.

Goud et al. 2011 [45] performed alkali treatment on the Roystonea regia fibre (royal palm) at 5 wt. % for 2 h. The authors concluded that an increase in all of these properties is noticed by increasing the fibre loadings. Authors also reported that alkali-treated fibre composite shows approximately an improvement in tensile properties by 8%, 8.2% improvement in tensile modulus and about 2.6% increase in young's modulus at 20 wt. % fibre content.

L. Yan 2012 [46] performed the alkali treatment (at 5 wt. %) on flax and linen fabric and evaluated the various mechanical properties of the composite. Their research finding deduced that in treated flax and linen composites showed good compressive properties than treated composites. The alkali-modified flax and linen composites enhance compression properties such as compressive strength and compressive modulus by 3 and 4.6%, respectively. The enhancement in the compressive properties is just because of the alkali treatment, i.e., alkali treatment ultimately enhances the adhesion strength of the fibre with the matrix and removes the impurities from the fibre surface such as impurities from the surface of the fibre.

Karthikeyan et al. 2012 [47] show that the alkalization increases the impact strength of the coir fibre composites. They performed alkalization at different concentrations such as 2, 4, 6, 8, and 10% for 10 days. The alkalization/mercerization makes the fibre surface rough. It provides the better interlocking between the fibre and the matrix as the greater surface area is available leading to the greater mechanical strength, specifically impact strength.

Kumar et al. 2013 [48] utilized alkali treatment (5 wt. %) to modify the bamboo strip mats. It reinforced it along with nano-clay into the polylactic acid resin. Authors with the help of Figure 2 showed an improvement in the mechanical properties such as tensile values and impact values. The increase in the mechanical properties was due to the good adhesion strength at the interfaces, which is only achieved by the chemical treatment of the bamboo fibre mats.

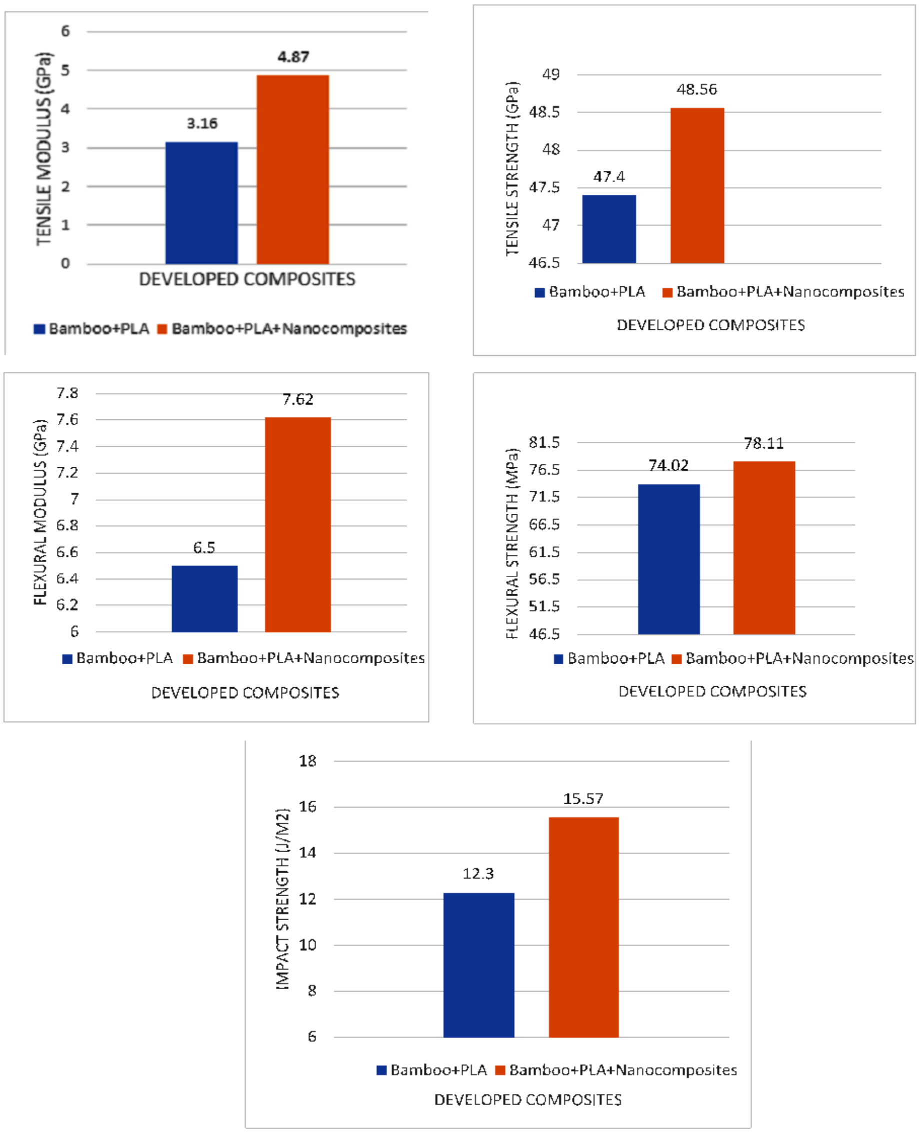



**Figure 2.** Tensile strength, Flexural strength, and Impact strength of the bamboo + PLA, and Bamboo + PLA + Nanocomposites [48].

Panneerdhass and co-workers [49] used alkali treatment (2 wt. %) for 30 min on the luffa fibre and the groundnut shell and reinforced the treated fibres in the epoxy matrix. They evaluated different mechanical properties such as compression, tensile, bending/flexural, and impact strength. Figure 3 shows the impact strength of the composites. From the figure, it is observed that the impact strength increases till the 30 wt. % of the fibre fraction, i.e., after that, a reduction in the impact values was observed. The maximum values of the impact strength are observed to vary from 0.6 to 1.3 joules. Similarly, the tensile strength varies from 26.66 MPa to 52.22 MPa, and flexural strength was also found to be optimum for the 30 wt. % of the fibre content. The results are shown in Figures 4 and 5 for both of the mechanical properties.

Rwawiire et al. 2015 [50] used biodegradable bark cloth and chemically treated it by alkalization (5 wt. %) process and reinforced it in the epoxy matrix for the automotive application. The authors noticed that alkalization/mercerization ultimately enhances the adhesion strength of bark cloth with the matrix material. The excellent adhesion ability exhibits good mechanical properties. A good enhancement in the different mechanical properties was observed and shown in Table 1.

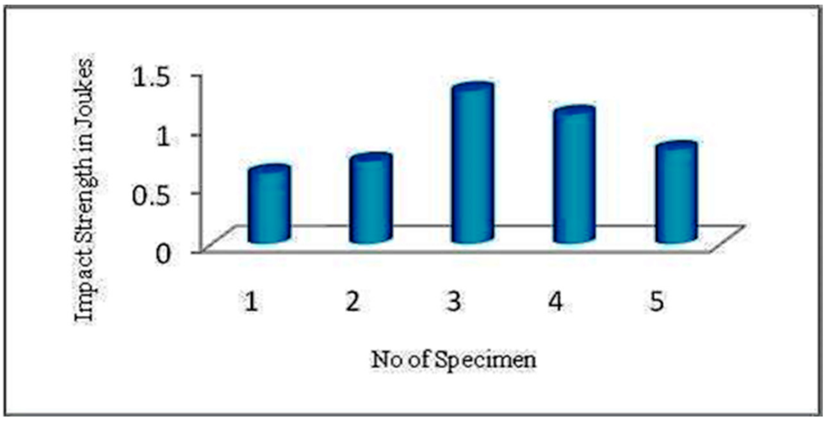



**Figure 3.** Impact Strength (reproduced with permission form ref. [49]).

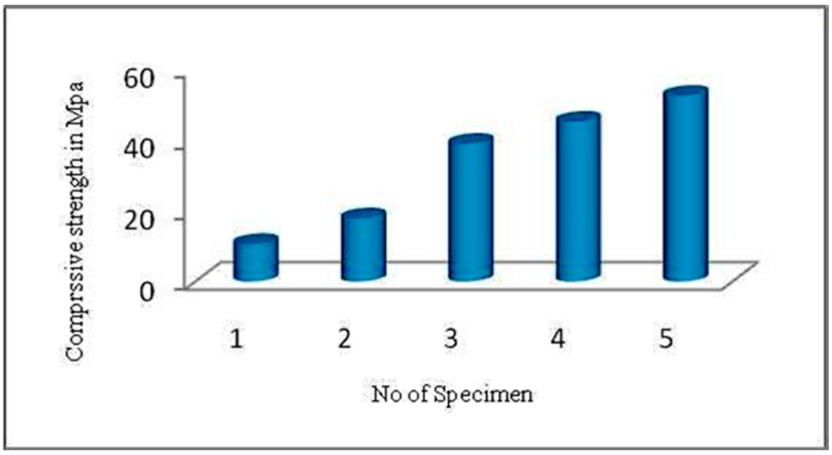



**Figure 4.** Compressive Strength (reproduced with permission form ref. [49]).

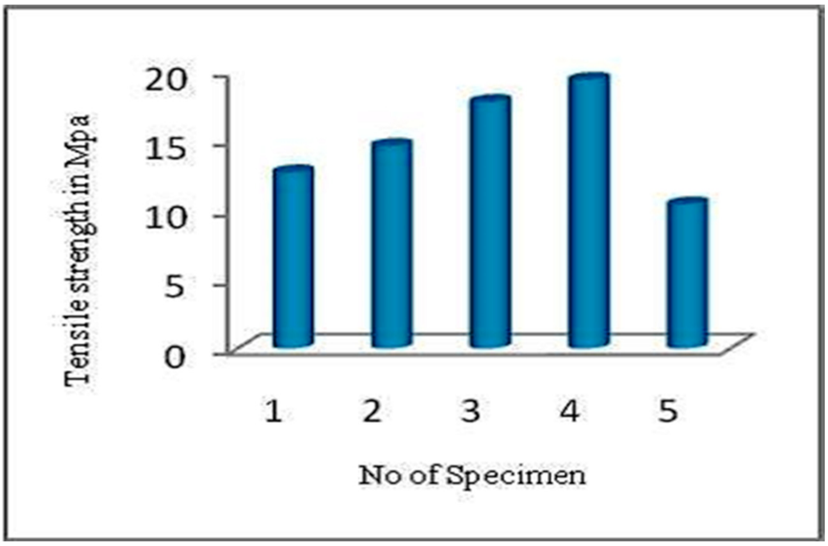



**Figure 5.** Tensile strength (reproduced with permission form ref. [49]).

**Table 1.** Biocomposites different mechanical properties. (Obtained from Elsevier (reproduced with permission form ref. [50])).

| Composites | Polymer Matrix | Tensile Strength (MPa)" | Tensile Modulus (GPa) | Fracture Strain (MPa) | Flexural Strength (MPa) | Flexural Modulus (GPa) | Impact Strength (MJ) |
|---|---|---|---|---|---|---|---|
| Bark Cloth Biocomposites | Green Epoxy | 33 | 3 | 2.1 | 207 | 1.4 | 5.73 |
| Bark Cloth Composites | Synthetic Epoxy | 30 | 4.1 | 1.8 | 153 | 3.1 | 9.62 |

Takagi et al. 2016 [51] treated bamboo fibre by alkali treatment, reinforced it into a PLA matrix, and developed a bamboo/PLA composite, and evaluated their mechanical properties. The alkali treatment makes the fibre surface rough and enhances the adhesion strength of the fibre with the matrix. Figure 6 shows the tensile strength of the PLA and steam-exploded bamboo PLA cross-ply laminate composite by utilizing the hot press method. The results show that the tensile strength and elastic modulus of the laminates are observed to be 223 MPa and 10.3 GPa by reinforcing 68 wt. % of bamboo fibres.

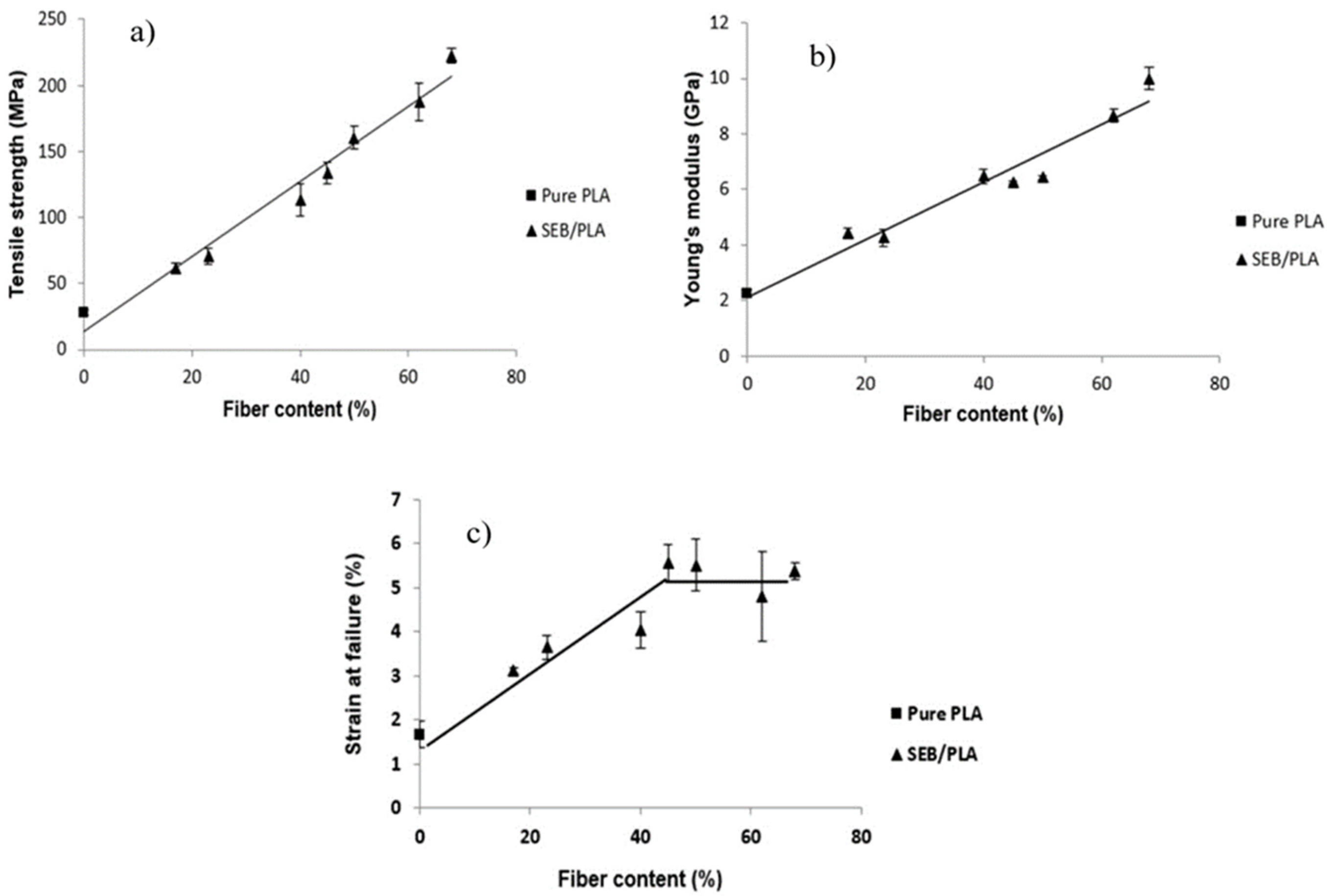



**Figure 6.** Mechanical Properties of laminates (**a**) Tensile strength (**b**) Modulus of Elasticity and (**c**) Failure strain (reproduced with permission form ref. [51]).

Balakrishnan et al. 2017 [52] performed the alkali treatment (2 wt. %) on the pineapple leaf fibre and reinforced it to the potato thermoplastic starch for the development of the nanocomposites. The morphology of the fibre can be understood by Figure 7a,b. From the figures, it can be observed that the fibre surface becomes smooth, which signifies the removal of the waxy substances and impurities from the fibre surface.

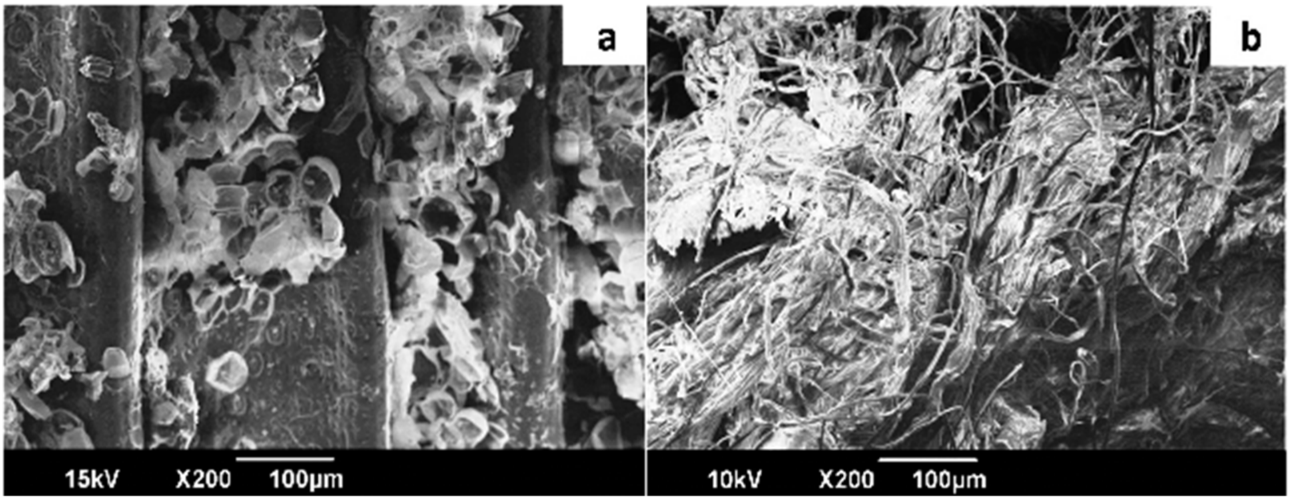
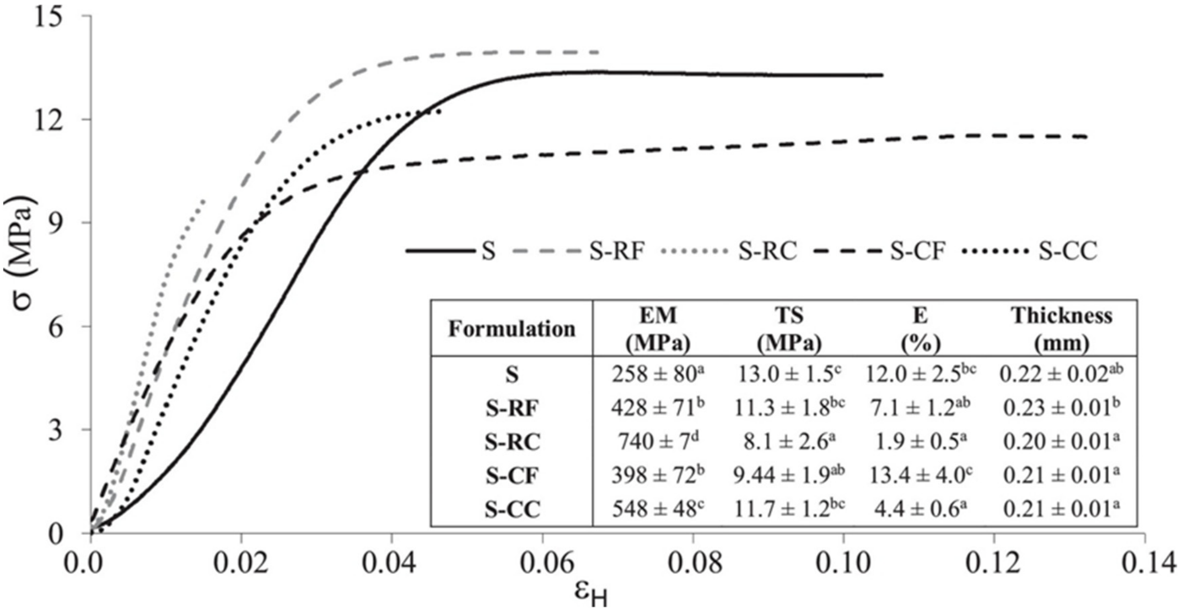



**Figure 7.** Scanning Electron Microscopy images of the PALF after various treatments. (**a**) Raw PALF (**b**) steam exploded. (reproduced with permission form ref. [52]).

Bigliardi et al. 2018 [53] utilized cellulose nanocrystals from the coffee husk and performed the alkali treatment on these nano-crystals. They utilized these nano-crystals into thermoplastic starch and developed a biocomposite. The authors observed that by keeping the aspect ratio to more than 10, there is 121% enhancement was observed in the elastic modulus of the composite. Figure 8 shows the tensile behaviour of the corn starch biocomposites films with and without cellulose fillers (1 wt. %). The tensile behaviour shows the different tensile properties of the bio-composites, such as tensile strength, modulus of elasticity and percentage deformation, etc. Figure 8 shows that the nano-crystals enhance the modulus of elasticity of the biocomposites film than the fibres.




| Formulation | EM (MPa) | TS (MPa) | E (%) | Thickness (mm) |
|---|---|---|---|---|
| S | $258 \pm 80^{a}$ | $13.0 \pm 1.5^{c}$ | $12.0 \pm 2.5^{bc}$ | $0.22 \pm 0.02^{ab}$ |
| S-RF | $428 \pm 71^{b}$ | $11.3 \pm 1.8^{bc}$ | $7.1 \pm 1.2^{ab}$ | $0.23 \pm 0.01^{b}$ |
| S-RC | $740 \pm 7^{d}$ | $8.1 \pm 2.6^{a}$ | $1.9 \pm 0.5^{a}$ | $0.20 \pm 0.01^{a}$ |
| S-CF | $398 \pm 72^{b}$ | $9.44 \pm 1.9^{ab}$ | $13.4 \pm 4.0^{c}$ | $0.21 \pm 0.01^{a}$ |
| S-CC | $548 \pm 48^{c}$ | $11.7 \pm 1.2^{bc}$ | $4.4 \pm 0.6^{a}$ | $0.21 \pm 0.01^{a}$ |

**Figure 8.** Tensile properties of corn starch films without (S) and with 1 wt % cellulose fillers (S-CF: with coffee husk fibres, S-RF: with rice husk fibres) and CNCs (reproduced with permission form ref. [53]).

Prasad et al. 2018 [54], used alkali-treated flax fibres and reinforced it into the epoxy matrix and TiO2. The authors used three fibre volume fractions of the flax fibres, such as 38%, 42%, and 46%. They observed that the fibre fraction of 42% is the optimum one and gives better mechanical properties such as tensile values and modulus of elasticity.

This is only because of the chemical treatment by NaOH at 5 wt. % which makes the good interlocking leading to the improved properties.

Sahai et al. 2019 [55] performed the four chemical treatments such as Silane, maleic anhydride treatment, and alkalization on the wheat straw and reinforced it with the polystyrene matrix material. They have observed that there is an improvement in the tensile, impact strength, and water absorption behaviour of the composite. The optimum chemical treatment is found to be 20 wt. % NaOH and the 1 wt. % Silane treatment. The treatment ultimately removes the impurities present at the surface of the fibre and enhances the mechanical interlocking between the fibre and the matrix which ultimately improves the mechanical properties of the composites. The water absorption capacity of NFs generally results in a diminution of fracture strength and stiffness of the NF as demonstrated in several studies on different types of NF, evaluated by the single fibre tensile test method [56–58]. One key question arising from these findings is, does the diminution of the mechanical properties of NF compromise the overall composite material? To date this is not clearly understood yet and could be a subject for further study. Table 2 shows the summary of the research reported in the current article.

**Table 2.** Summary of the research in Alkalization of natural fibres with improved mechanical properties.

| Alkalization/Mercerization Treatment | Type of Natural Fibre | Mechanical Properties | References |
|---|---|---|---|
| Alkalization at 12 wt. % | Bagasse fibres | Improved young's modulus by 1800 MPa | [42] |
| Alkalization at 5 wt. % | Jute Fibres | Improved flexural strength and flexural modulus | [43] |
| Alkalization at 5 wt. % | Agave Fibres | An improvement in characteristic strength of 30% was observed | [44] |
| Alkalization at 5 wt. % | Roystonea regia fibre (royal palm) | An improvement in tensile properties was observed | [45] |
| Alkalization at 5 wt. % | Flax and Linen Fabric | Improvement in compressive properties was observed | [46] |
| Alkalization at 2, 4, 6, 8, and 10 wt. % | Coir fibres | Provides better interlocking between matrix and fibre leading to improvement in mechanical properties | [47] |
| Alkalization at 5 wt. % | Bamboo strip mats | Improvement in tensile properties and impact properties was observed | [48] |
| Alkalization at 2 wt. % | Luffa fibres | Improvement in impact strength and flexural strength was observed | [49] |
| Alkalization at 5 wt. % | Bark cloth | Improvement in the tensile properties and flexural properties was observed | [50] |
| Alkalization at 5 wt. % | Bamboo fibre | Improvement in tensile properties and young's modulus was observed | [51] |
| Alkalization at 5 wt. % | Coffee husk nano crystals | Excellent improvement in tensile properties was observed | [53] |
| Alkalization at 5 wt. % | Flax fibres | Improved tensile properties | [54] |
| Alkalization at 20 wt. % | Wheat straw fibres | Improvement in tensile strength, impact strength, and water absorption | [55] |
| Alkalization at 1M NaOH | Bagasse fibres | Improved flexural properties | [27] |

Oladele and co-workers [27] mercerized the bagasse fibre (1M NaOH at the temperature of 50 °C for 4 h) along with the $CaCO_3$ into the polypropylene matrix and developed a composite by using the compression moulding technique. The flexural properties of both the chemically modified and unmodified composite are exhibited in Figure 9. It is observed from the figure that the flexural properties enhance by increasing the $CaCO_3$ proportion from 3 wt. % to 15 wt. % for both of the composites (i.e., both treated and untreated). Additionally, from the figure, it is noticed that the sample E of mercerized bagasse fibre composites shows higher flexural strength, i.e., 21.75 MPa, as compared to the unmercerized sample E, which is 17.51 MPa. Of note, Oladele and co-workers have termed their composites as 'hybrid fibre reinforced composites' to emphasize the addition of an inorganic compound to lend further support to the reinforcement efficiency of the NF in the composite. Alternatively, one could also synthesize natural fibres by blending compounds derived from NF, e.g., lignin, into the biopolymer, e.g., chitosan, as reported elsewhere [59]. Such hybrid natural fibres have fracture strengths as high as 150 MPa and stiffness as high as 3.5 GPa based on a single fibre testing approach and, thus, have great potential for providing reinforcement to polymer composites.

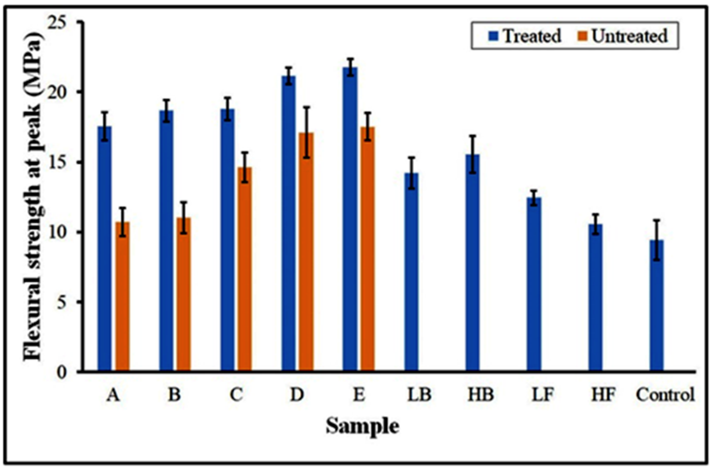



**Figure 9.** Sample's Flexural strength graph (reproduced with permission form ref. [27]).

Kassab and co-workers [60] identified a Juncus plant and performed the alkali treatment (4 wt. % NaOH at a temperature of 80 °C for 2 h). It has been observed that as compared to the PVA films, the PVA/CNF composites show an improvement in modulus of elasticity, tensile properties, and the fracture strain by 37%, 60%, and 90%, respectively. The enhancement in the mechanical properties is just because of the CNF, their optimized aspect ratio, and their functionalized surface, which are specifically responsible for good dispersion and adhesion.

## 6. Conclusions

Natural fibre (NF) and its reinforcement are attractive alternatives to synthetic fibre for the development of polymer-based composites. The main benefits of utilizing the NFs for composite materials are their biodegradability and good mechanical properties. NF composites are lighter and show excellent mechanical properties. However, the main problem of utilization of the natural fibres into the polymer matrix is their hydrophilicity, less moisture resistance, which ultimately affects the adhesion strength of the NF with the hydrophobic matrix and cannot be suggested for the various industrial applications. To make the NFs hydrophobic, they must undergo various treatments, such as physical and chemical treatments. Chemical treatment of the NF is an effective option through which the fibre can acquire hydrophobic properties. There are so many chemical treatments available out of which the alkali/mercerization treatment has shown to be effective, and economical one. Alkalization/mercerization ultimately enhances the interfacial strength/bonding between the fibre and the matrix material. The better interlocking of fibres with the matrix results in improved mechanical properties, which is the primary objective of polymer-based green composites. This article has discussed the alkali treatment/mercerization on the natural fibre and their utilization in the various polymer matrix for developing composite material. The past research evidenced that alkalization or mercerization ultimately enhances the adhesion ability of the fibres with the matrix materials. If fibre compatibility with the matrix is good, it will improve mechanical properties as desired for distinct applications.

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