Effects of fiber content and its chemical treatment on the mechanical properties of screw pine fiber reinforced vinyl ester composite

S Venkatarajan 1, C Subbu 2, A Athijayamani 3, and S M Sivagami 4
1 Department of Physics, Alagappa Chettiar Government College of Engineering and Technology, Karaikudi-630003, Tamil Nadu, India
2 Department of Physics, Alagappa Government Arts College Affiliated to Alagappa University, Karaikudi-630003, Tamil Nadu, India
3 Department of Mechanical Engineering, Alagappa Chettiar Government College of Engineering and Technology, Karaikudi-630003, Tamil Nadu, India
4 E-mail: subbucphysics@gmail.com

Keywords: screw pine fiber, vinyl ester, fiber content, alkali treatment, mechanical properties, scanning electron microscopy

Abstract

Natural fiber-reinforced polymer composites have several advantages over traditional composites. The chemical modification of natural fibers helps to develop polymer composites with better mechanical properties. In the present work, mechanical properties such as tensile, flexural, and impact strength of chopped Screw pine fiber reinforced vinyl ester composites have been evaluated under-treatment conditions based on the volume fractions of Screw pine fibers. The fibers have been treated with 5% of NaOH solution for 1 h at room temperature. The hand lay-up method has been used to prepare composite plates at room temperature. The results revealed that mechanical properties of composites increased with the increase of the fiber content up to 35.57 vol% at both the untreated and treated conditions and then dropped. However, the modulus values have been increased continuously from the fiber content of 8.43 to 45.3 vol%. It was identified that the critical or optimum fiber content for better mechanical properties is 35.57 vol% for both the untreated and treated conditions. The percentage of improvement at every combination was obtained by comparing the composites prepared with the untreated and treated fibers. The fractured surface of the treated fiber composites was examined by scanning electron microscopy. Moreover, the tensile properties are predicted using the Hirsch and Modified Bowyer and Bader model and compared with experimental values. The predicted results revealed that the Modified Bowyer and Bader model shows better conformity.

1. Introduction

The production and export of natural fibers play a vital role in the economies of many developing countries and the livelihoods of low-wage workers and small-scale farmers in the Southern Region of India. Natural fiber-reinforced polymer composites are a system of composite material which consists of a polymer matrix entrenched with natural fibers, like plant and vegetable fibers, animal fiber, and mineral fibers. The strength of a natural fiber-reinforced polymer composite is strong-willed by the fiber-matrix interfacial bonding [1–3]. The fiber-matrix interface acts as a binder and transfers the applied load among the resin matrix and the reinforcing fibers [4, 5]. Good Interfacial adhesion is the better compatibility between the fiber and matrix and the formation of a chemical bond and mechanical interlocking between the reinforcing fiber and the resin matrix. Natural fiber polymer composites with good environmental performance and mechanical properties can be developed by imparting hydrophobicity to the fibers by suitable chemical treatments [6]. Among various chemical treatments, alkali treatment is the easiest and, most commonly used treatment technique for natural cellulose fibers. Therefore, modifying the fiber surface and better compatibility with resin matrices is necessary to prepare a quality natural fiber composite. Generally, the reinforcing efficiency of natural cellulose fibers (hydrophilic) with the polymer resin matrix (hydrophobic) is reduced due to the higher moisture absorption

© 2022 The Author(s). Published by IOP Publishing Ltd
tendency of the natural cellulose fibers. This problem can be resolved by modifying the structure of natural cellulose fibers using suitable chemical treatment methods. Moreover, the interfacial adhesion between natural cellulose fibers and the matrixes can also be improved by the treatment of a suitable chemical solution [7, 8]. Several authors report the influence of alkali treatment on the properties (physical and mechanical) of natural fiber-reinforced polymer composites. The moisture absorption tendency of natural fibers can be decreased by suitable treatment methods. It may also be improved the properties (physical and mechanical) of natural fiber polymer composites [9]. The properties of natural fiber reinforced polymer composites can be enhanced by suitable physical and chemical treatment methods. The moisture sensitivity and biological decay of natural cellulose fibers can be reduced and optimized the properties of the interface between the fiber and the matrix [10].

The mechanical properties of jute-phenolic resin composites were investigated at alkali (5% of NaOH) treated conditions and found that the treated fiber composites exhibit the improved mechanical properties, as well as the water uptake behavior, which is the lowest [11, 12]. Chemical treatment was also increasing the surface roughness on the surface of the fibers. The interfacial adhesion between the sisal fiber and the matrix can be enhanced by chemical and thermal treatments like NaOH, H2SO4, conjoint H2SO4, and acetylated treatment [13]. The mechanical behavior of Roselle fiber and sisal fiber reinforced polyester hybrid composite was evaluated under the treated condition and compared with the untreated fiber composites. It was observed that the alkali solution of 10% can be used to improve the properties with high toughness [14]. The mechanical properties of oil palm fiber reinforced epoxy composite were studied based on the fiber concentration and alkali treatment. It was identified that the treated oil palm fiber reinforced epoxy composites show a higher level of mechanical properties than that of untreated fiber composites. The optimum mechanical properties were obtained at the fiber content of 20 wt% [15, 16]. The different methods of surface treatments were used for the surface modification of natural fibers to improve the resistance to water absorption [17].

The surface morphology, structure, and interfacial adhesion characteristics of sisal fiber-reinforced polypropylene composites were studied by the influence of chemical treatment processes (acetylation and alkali). It was clearly observed that the impurities presented on the surface of the fibers are removed with the increase of surface roughness. Moreover, the FTIR analysis confirmed the reduction of chemical compositions like hemicellulosic and lignin, which are unfavorable to the bonding strength of resulted composite [18]. Based on the above works of literature, in this work, the influence of NaOH treatment and the fiber content on the mechanical properties of Screw Pine Fiber (SPF) reinforced vinyl ester composite was evaluated. The SPFs were immersed at 5% of NaOH solution for 1 h. Scanning Electron Microscopy (SEM) is used for the fractographic study to be carried out on the fracture surface of the treated fiber composites. Tensile properties are predicted using the Hirsch and modified Bowyer and Bader (MBB) models and also compared with experimental tensile property values.

2. Experimental procedures

2.1. Materials
The SPFs are extracted from the leaves of well-grown Screw pine plants. For the production of fiber, the leaves were collected at the bud stage. The leaves were tied into bundles and retted in water for 2–4 days. The retted leaves were cleaned with running water. Then, the outer surface of the leaves was detached manually using comb-like components, and the fibers are dried in sunlight. The vinyl ester resin (Trade name Satyen Polymer Pvt. Ltd, Bangalore, India) was used in resin matrix with Methyl Ethyl Ketone Peroxide (accelerator), cobalt 6% naphthenate (Catalyst), and N–N dimethyl aniline (promoter). All the chemical agents were purchased from the GVR Enterprise, Madurai, Tamilnadu, India.

2.2. Treatment of SPFs
Generally, the treatment of natural cellulose fibers with the alkali solution is a prioritization process over any chemical treatment. The sodium hydroxide opens up the structure of fiber allowing the hydroxyl groups to react with the resin matrix. A significant reaction was taking place between the hydroxyl groups of cellulose and NaOH. During washing with NaOH, some chemical components such as part of cellulose, lignin, and hemicellulose, the wax, and the cuticle layer, are detached partially in a particular amount. The partially removed chemical components enhance the fiber-matrix interface and ensure a good bond between the fibers and matrix. If the alkali treatment parameters are not optimized, it can cause fiber defibrillation, fiber embrittlement, and pore formation [19]. The reaction between the fiber and NaOH is shown by the following equation and the corresponding chemical structure of the possible reaction is given in figure 1.
Fiber OH NaOH Fiber O Na H O

NaOH treatment not only increases the surface roughness of the natural fibers resulting in good mechanical interlocking but also increases the amount of cellulose content exposed on the surface of the fiber, thus increasing the number of possible reaction sites on the fiber surface. The surface treatment with alkali solution changes the crystallographic structure and surface topography of the used fibers. Generally, the use of alkali with NaOH in natural fiber can provide an increase in cellulose content, mechanical properties, and also water resistance properties of the fibers [20]. In the present work, the SPFs were soaked at 10% of the alkali solution for 1 h at room temperature, and then they have rinsed with running water and also allowed to dry for 48 h at room temperature.

2.3. Preparation of composites
A mould with the size of 150 \( \times \) 150 \( \times \) 3 mm was used to prepare the composite plates by hand lay-up technique. The SPFs were evenly spread with the help of a mechanical roller inside the mould. The fiber length was fixed at 3 mm. Then, the promoters and accelerators were mixed with resin to start the chemical reaction and to speed up the curing reaction. The air bubbles were removed by degassing, which process is applied to the removal of dissolved gases from liquids. The Catalyst, which is used to get faster a chemical reaction by lowering the amount of energy, was added and then poured into the mould box. Finally, the filled mould box was closed and kept within the room under atmospheric pressure and also temperature for 48 h. After removing the composite plates from the mould box, they were extra cured by a post-curing for 80 °C in an oven.

2.4. Testing
Mechanical properties such as tensile, flexural, and impact were determined by characterizing the composite specimens based on the ASTM standards. According to ASTM D638:2014 [21], the tensile tests were performed on composite specimens at a computerized FIE universal testing machine at a crosshead speed of 2 mm/min. The flexural tests at the three-point bending method were carried out according to ASTM D790:2017 [22] on the FIE universal testing machine. The impact tests were conducted on the Izod impact tester as per ISO 180:2019 (en) [23] to measure the impact strength. For each combination totally, five composite specimens were tested and their average values were recorded for statistical purposes.

The fractured composite specimens after the tensile, flexural, and impact tests were examined by the SEM (HITACHI S-3000N) for a detailed study. SEM images were obtained at a voltage of 30 kV.

3. Results and discussion
The impurities (natural and synthetic) from the fiber’s surface may be efficiently removed by increasing the duration of chemical treatment or by increasing the % of chemical solution. During the alkaline treatment, natural fibers are separated from one another, which increases the effective surface area for wetting by the resin matrix. Moreover, the alkaline treatment modifies the unit cell structure, fiber orientation, and crystallinity [24, 25]. In this work, 5% of NaOH solution at 1 h of treatment duration was taken to observe the effect of NaOH treatment of fibers on the mechanical properties of composite materials.

3.1. Influence of fiber content on mechanical properties of the untreated fiber composites
The fiber content plays a significant role in the performance study of fiber-reinforced polymer composites. Figures 2(a)–(c) illustrated the influence of the fiber content of SPFs on the tensile properties of the untreated SPF/vinyl ester composites. From figure 2(a), it is identified that the composite specimen reached the tensile properties of the cured neat resin sample at 17.15 vol%. Moreover, composite shows an increasing trend for the tensile strength from 8.43 to 35.57 vol% and then, it is dropped. Therefore, composite with 35.57 vol% of the SPFs fiber content showed the maximum tensile strength, which is 45% higher when compared with the cured un-reinforced resin sample. Furthermore, the tensile strength of 45.3 vol% composite was lower than that of SPFs fiber content. However, it is concluded that the composite specimen reached the tensile properties of the cured neat resin sample at 17.15 vol%.

\[
\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H}_2\text{O}
\]
26.19 vol% composite. The modulus of composite also increases linearly with fiber content. The 45.3 vol% composite shows the highest value of tensile modulus, as shown in figure 2(a).

The flexural strength of the vinyl ester composite increases with the addition of SPF content, as given in figure 2(b). It is observed that composite with 35.57 vol% of fiber content shows 52.2% of the improvement in flexural strength when compared with the cured neat resin sample. Composite having a fiber content of 30.84 vol% demonstrates a higher flexural strength than that of 45.3 vol% of the composite (58.4 MPa). The same trend observed in the tensile modulus was also identified in the flexural modulus. The maximum flexural modulus (1681.3 MPa) was attained at composite with 45.3 vol% of fiber content and showed 30% of the improvement when compared with the neat resin sample.

The impact strength values of composites after impact tests based on the fiber content were presented in figure 2(c). It is observed that composites with 17.15 vol% reach the impact strength value of the un-reinforced cured resin sample. The impact strength shows the increasing trend up to 35.57 vol%, and then, it is decreased. Composite with 35.57 vol% of fiber content attained 45.3% of the improvement when compared with the neat resin sample.

It is clear that the mechanical properties of composites are increased up to 35.57 vol% of fiber content and then dropped. The addition of fiber content beyond 35.57 vol% leads to the poor compatibility of the fiber with the resin matrix. The poor compatibility results in loss of the mechanical properties of composites. Moreover,
the poor dispersion of fibers results in the aggregation of fibers due to the higher amount of fiber; therefore, the resin matrix could not properly wet the fibers during the preparation. The fracture surface of untreated fiber composite shows the fiber pullout and weak interfacial bonding, which lowers the mechanical interlocking between the fiber and resin matrix. The lower mechanical interlocking results in loss of mechanical properties.

3.2. The effect of NaOH treated SPFs on mechanical properties of composites
The stress-strain diagram of treated test samples (8.43, 17.15, 26.19, 35.57, and 45.3 vol%) subjected to tensile test was given in figure 3. Composite with 8.43 vol% showed a maximum strain value of 1.9. It was observed that the addition of SPFs increased the strain. At the highest volume fraction of 45.3 vol%, the lowest strain was observed compared to 35.57 vol.%. Composite having 35.57 vol% of SPFs showed considerably higher strength compared to other composite specimens. A significant increase in tensile properties was observed owing to the increase of fiber content at the treated condition, as shown in figure 4. Composite with all combinations at the treated condition showed the maximum mechanical properties compared to composites in the untreated condition. Composite with 12.75 vol% of the treated fiber content reaches the tensile strength of the neat resin sample. Composite having 35.57 vol% of the treated fiber content exhibit a high tensile strength value [26] and it is 63% higher than that of the un-reinforced resin sample. A linearly increasing trend was observed at the tensile modulus from 8.43 to 45.3 vol%. The maximum tensile modulus (1469.6 MPa) was attained at composite with 35.57 vol% of fiber content. When compared with the neat resin sample, it shows 26% of the improvement.

The addition of the treated fibers with vinyl ester resin matrix increases the flexural properties of composites, as shown in figure 5. An increase of flexural strength continued up to 35.57 vol% and beyond which, the addition of the treated fibers decreases the flexural strength. The same flexural values were observed at composites with the fiber content of 30.84 and 40.39 vol%. Composite with 35.57 vol% shows the highest value of flexural strength as compared to 45.3 vol% composite (80.6 MPa), which is 16% higher than 45.3 vol% of composites [27].
The impact strength of composite increased with the addition of the treated fiber content, as illustrated in figure 6. Composite with 12.75 vol% of the treated fiber content reaches the impact strength of the un-reinforced resin specimen. The maximum impact strength was attained at 35.57 vol% composite, and an improvement of 56.14% was observed when compared with the un-reinforced resin sample. The impact strength \( (1.73 \text{ KJ m}^{-2}) \) of 45.3 vol% composite was lower than 35.57 vol% composite, which is by a percentage of 13. From the results, it was identified that the critical or the optimum content of fiber is 35.57 vol%, in which the best combination of mechanical properties can be obtained. The critical or optimum content may vary based on the nature of the fiber and degree of fiber-matrix adhesion, etc.

The less fiber pullout and brittle fracture (figure 7(a)) observed on the fracture surface of composite prove the above explanation after the tensile test. After the flexural test, a less number of fiber pullouts with brittle fractures were identified, as shown in the SEM image (figure 7(b)). The fracture surface of the composite after the impact test was also examined by SEM, and it is illustrated in figure 7(c). From figure 7(c), it is clear that a good bonding (mechanical interlocking) strength between the matrix and fiber is observed.

### 3.3. The percentage of improvement in the mechanical properties due to the alkali treatment

The percentage of improvement in mechanical properties of Screw pine/vinyl ester composites was obtained by the calculation of percentage improvement. Composite with 35.57 vol% of the treated fibers shows better improvement in all mechanical properties at all combinations, as shown in figure 8. The determination of the percentage of improvement had obtained from the initial addition of the fiber. Composite having the fiber content of 35.57 vol% shows a gain of 48% in tensile strength compared to the untreated fiber composite. Almost the same level of improvement was attained at 40.4 and 45.3 vol% composites. From figure 6, it was observed that composite with 35.57 vol% of the fiber content shows the highest level of improvement when compared with the untreated composite at flexural strength. Moreover, it was identified that composite having 35.57 vol% of the fiber content shows better improvement in the impact strength. Furthermore, 17.15 and 21.63 vol% of composites prepared with the treated fibers indicate almost the same level of increment. Finally, it was
observed that the mechanical properties of composites have increased with the addition of alkali-treated SPF. It may be due to the removal of impurities (natural and artificial) from the surface of SPF during alkali treatment. Therefore, the level of interfacial adhesion increases between the fiber and the matrix, resulting in improved mechanical properties.

3.4. Prediction and comparison of tensile properties using theoretical models
Several theoretical models may be used to predict the tensile property of the natural fiber reinforced polymer composites and compare them with the experimental tensile properties. In the present study, the Hirsch and modified Bowyer and Bader models are used to predict the tensile strength and modulus of Screw pine fiber/vinyl ester composite. Hirsch and MBB models were shown good agreement with experimental results of natural fiber-reinforced polymer composite materials [29, 30]. The following equations (1) and (2) of the Hirsch model are used to predict the tensile properties (strength and modulus):

Figure 7. SEM images of fracture surfaces of composite (35.57 vol%): (a) tensile, (b) flexural, and (c) impact after alkali treatment.
where F, C, and M are the characteristic strength property of fiber, composite, and matrix, respectively. \( e_F \), \( e_C \), and \( e_M \) are the modulus value of fiber, composite, and matrix. \( \sigma_F \), \( \sigma_C \), and \( \sigma_M \) are the tensile strength of the fiber, matrix, and composite respectively. \( V_F \) and \( V_M \) is the volume fraction of fiber and matrix. X is a parameter that depends on the type of fiber, resin matrix, and fiber-matrix interaction and also determines the stress transfer between the fiber and the matrix. The value of X varies between 0 and 1, taken as 0.512 in the present study.
which gives nearby results to the experimental strength values. Moreover, the tensile properties (strength and modulus) of the fiber-reinforced polymer composite can also be estimated by the modified MBB model (equations (3) and (4)) as follows:

\[
\epsilon_C = \alpha_V \times \epsilon_f \times V_f + \epsilon_M \times V_M
\]

\[
\sigma_C = \alpha_V \times \sigma_f \times V_f + \sigma_M \times V_M
\]

where \(\alpha_V\) is the overall reinforcing factor. The value of \(\alpha_V\) is taken as 0.0578 in equations (3) and (4). The \(\alpha_V\) expresses that at what amount the modulus of elasticity of the fiber contributes to composite. From figures 9(a)–(b), it was clear that the tensile properties of the composites increased with the increase of the weight percentage of Screw pine fibers. It was found that there is a good agreement with the MBB model for the tensile properties of the composite compared to other models. For the prediction of tensile properties of the Screw pine fiber/vinyl ester composites, it was observed that the Hirsch model could also be used.

4. Conclusion

Mechanical properties of alkali-treated Screw pine fiber reinforced vinyl ester composites were evaluated for varying fiber content and compared with the untreated fiber composites. Mechanical properties of vinyl ester were enhanced by the incorporation of both the untreated and treated Screw pine fibers. However, the level of improvement by the treated fiber composites was higher than the untreated fiber composites. Moreover, the mechanical properties of both the untreated and treated fiber composites increased continuously up to 35.57 vol% of fiber content and then, dropped. From the obtained results, it was identified that the optimum fiber content to get a better level of mechanical properties is 35.57 vol% for both the untreated and treated fiber composites. When compared with the Hirsh model, the MBB model has predicted the tensile property values very close to experimental tensile property values. However, the Hirsch model can also be used to predict the tensile property values of composites. Finally, it was concluded that the chemical treatment of Screw pine fibers with a particular concentration of alkali solution and duration has a significant impact on the mechanical properties of composites.

Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

ORCID iDs

S Venkatarajan https://orcid.org/0000-0002-3873-3627
C Subbu https://orcid.org/0000-0002-0900-6041
A Athijayamani https://orcid.org/0000-0001-6118-1106

References

[1] Satyanarayana K G, Guimaraes J L and Wypych F 2007 Composites Part A: Applied Science and Manufacturing 38 1694–709
[2] Livingston T, Athijayamani A and Alavudeen A 2020 Mater. Res. Express 8 1–9
[3] Vijaya Ramnath B, Arvind R, Dinesh I and Hari prasad M 2018 IOP Conf. Series: Materials Science and Engineering 390 1–7
[4] Saba N, Aloethman O Y, Almutairi Z, Jawaid M and Ghori W 2019 Journal of Materials Research and Technology 8 3959–69
[5] Nijandhan K, Muralikannan R and Venkatakalam S 2018 Mater. Res. Express 5 1–9
[6] Hamidon M H, Mohamed, Sultan M T H, Ariffin A H and Shah A U M 2019 Journal of Materials Research and Technology 8 3327–37
[7] Kusmono H H and Jamasri 2020 Journal of Materials Research and Technology 9 4110–20
[8] Martins M A and Joekes I 2003 J. Appl. Polym. Sci. 89 2507–15
[9] Bisanda E T N and Ansel M P 1991 Compos. Sci. Technol. 41 165–78
[10] Feng D, Caulfield D F and Sanadi A R 2001 Polym. Compos. 22 506–17
[11] Razera I A T and Frollini E 2004 J. Appl. Polym. Sci. 91 1077–85
[12] Kumar S, Shamprasad M S and Varadarajan Y S 2021 IOP Conf. Series: Materials Science and Engineering 1065 1–12
[13] Li Y, Mai Y and Ye L 2000 Compos. Sci. Technol. 60 2037–55
[14] Athijayamani A, Thiruchirambalam M, Natarajan U and Prakash A B 2010 Polym. Compos. 31 723–31
[15] Obasi H C, Iheutra N C, Onuoha F N, Chike-Onyegbula C O, Akanbi M N and Ezeh V O 2014 American Journal of Engineering Research 3 117–23
[16] Abhisheen A V, Abhisheen Dharmanarath T and Narendiranath Babu T 2021 IOP Conf. Series: Materials Science and Engineering 1123 1–11
[17] Kumar R, Obrai S and Sharma A 2011 Der Chemica Sinica 2 219–28
[18] Mokaloba N and Batane R 2014 International Journal on Engineering, Science and Technology 6 833–97
[19] Sari N H and Padang Y A 2019 IOP Conf. Ser.: Mater. Sci. Eng. 539 1–6
[20] Zin M H, Abdan K, Mazlan N, Zainudin E S and Liew K E 2018 IOP Conf. Ser.: Mater. Sci. Eng. 368 1–10
[21] ASTM D638–14 2014 Standard test method for tensile properties of plastics Annual Book of ASTM Standards 1–17
[22] ASTM D 790–17 2017 Standard test methods for flexural properties of un-reinforced and reinforced plastics and electrical insulating materials. Annual Book of ASTM Standards 1–12

[23] ISO 180: 19 2019 Plastics—determination of Izod impact strength. Third edition (Switzerland: ISO Central Secretariat) 1–13

[24] Ray D, Sarkar B K, Rana A K and Bose N R 2001 Composites Part A: Applied Science and Manufacturing 32 119–27

[25] Colom X and Carrillo F 2002 Eur. Polym. J. 38 2225–30

[26] Alexandre G, Takanori M, Koichi G and Junji O 2007 Composites Part A: Applied Science and Manufacturing 38 1811–20

[27] Ngo W L, Pang M M, Yong I C and Tshai K Y 2014 Advances in Environmental Biology 8 2742–7

[28] Roni K, Raharjo W W, Ariawan D, Ubaidillah and Ariffin Z 2021 IOP Conf. Ser.: Mater. Sci. Eng. 1096 1–9

[29] Kalaprasad G, Joseph K, Thomas S and Pavithran C 1997 J. Mater. Sci. 32 4361–7

[30] Yashwant SM and Ravindra B I 2015 Procedia Technology 19 320–6