Mechanical and Thermal Characterization of Short Salago Fiber (*Wikstroemia spp.*) Reinforced Epoxy Resin Composites

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Abstract. Natural fibers are used as alternative reinforcements in polymer composites due to their biodegradability, low density and cost. Applications of natural fiber reinforced polymers include aerospace industry, automotive parts and marine vessels. In the Philippines, one source of natural fiber is salago, which is a type of shrub with tough bark used to produce high quality paper products. In this research work, short salago fibers were reinforced in epoxy resin in random orientation. The fibers were treated with 5 wt.% NaOH solution. The fiber loading considered in the study are 5 wt.%, 10 wt.%, 15 wt.% and 20 wt.%. Mechanical properties of neat epoxy and salago fiber composites were characterized through tensile, flexural and impact tests. Thermogravimetric analysis was used to evaluate the thermal stability of the specimens. Results for 20 wt. % fiber loading show significant improvements on tensile modulus (83.5 %) and impact strength (26.4 %) as compared to neat epoxy. However, decrease on tensile and flexural strengths were observed as the fiber content increases. Nonetheless, noticeable improvement on thermal stability was obtained for the composite with 10 wt.% fiber content.

1. Introduction

In recent years, the applications of natural fiber reinforced composites (NFRCs) in industries have increased drastically due to the availability of natural fibers at lower costs as compared to synthetic materials [1]. Some applications of natural fibers include automotive parts, aerospace components, electronics cases, sports equipment, packaging and textiles [2]. NFRCs have properties that are comparable to synthetic materials like mechanical strength, thermal properties and tribological characteristics [3]. Mechanical properties of these composites depend on various parameters such as interaction between fiber and matrix, aspect ratio, fiber loading and orientation, manufacturing techniques, etc. [4]. Chemical treatment such as alkaline and silane can also be applied to natural fibers to enhance adhesion with the matrix which can further improve the properties of composites [5]. The commonly used natural fibers in polymers are coir, sisal, jute, banana and flax [6]. However, due to the increase in demand for natural fibers, there is a need to explore other sources of fibers and study their possible use in different matrices. In the Philippines, one of the sources of natural fiber is salago. It is a type of shrub in which the extracted bast fiber can be used to produce high quality paper products. In a study, it was found out that salago fiber has cellulose content of 79.05 % and tensile strength of 1187 MPa [7]. Salago fiber has also been studied as filler material in high-density polyethylene where the properties of the composites were characterized [8, 9]. Apparently, only few research works about the use of salago fiber as reinforcement or filler in composites were conducted.
2. Methods

2.1 Materials
Salago barks were obtained from a paper factory in Cavite, Philippines. Both epoxy resin and hardener were purchased from J. Spencer Technologies Philippines Corporation in Muntinlupa, Philippines. Sodium hydroxide and acetic acid were procured from a local supplier.

2.2 Fiber Separation and Treatment
Salago barks were cut into 10 cm long and washed with tap water to remove dirt. The barks were then subjected to water retting for one week. The fibers were separated manually and air dried for two weeks. The fibers were then soaked in 5 % NaOH solution for 30 minutes using a liquor to fiber weight ratio of 20:1. In order to neutralize the effect of NaOH, the fibers were immersed in 2 % acetic acid solution for 1 hour [10]. These were subsequently rinsed three times in distilled water. The alkali treated fibers were sun dried for 3 days and oven dried at 70 °C for 4 hours to remove excess moisture. The salago barks and their separated fibers are shown in figure 1 and figure 2, respectively.

![Figure 1. Salago barks.](image1)
![Figure 2. Separated fibers.](image2)

2.3 Specimen Preparation
An open molding process was used to fabricate the composites through silicone molds with shapes based on the dimensions specified in ASTM Standards D638 (Type I) and D790. The treated salago fibers were cut into 5 to 7 mm long and laid on the silicone molds in 2-D random orientation. The epoxy resin and hardener were mixed manually using the recommended weight ratio of 3:2 for 5 minutes before pouring the mixture into the silicone molds. A metal rod was used to distribute the resin to the fibers. The specimens were cured for 72 hours at room temperature. Varying fiber loading of 5 wt.%, 10 wt.%, 15 wt.% and 20 wt.% including neat epoxy were prepared. Figure 3 shows the salago fiber composites for tensile test.

![Figure 3. Salago fiber composites (a) 5%, (b) 10%, (c) 15% and (d) 20%](image3)

2.4 Mechanical Testing
The specimens for mechanical testing were brought to the Standards and Testing Division of the Industrial Technology Development Institute of the Department of Science and Technology (DOST-ITDI) in Metro Manila, Philippines. Tensile test was performed based on ASTM Standard D638 using Instron UTM (Model:5585 H) with 5 mm/min speed of testing. On the other hand, flexural test was conducted based on ASTM Standard D790 using Shimadzu UTM (AGS-50kNXD) with a support span to depth ratio of 16:1. For Izod impact test, it was performed based on ASTM Standard D256 Method A using Zwick/Roell 5.5P Izod Pendulum Tester. Five trials were considered for each mechanical test. The average values and standard deviations were then calculated.
2.5 Thermogravimetric Analysis (TGA)

The specimens for TGA were brought to the Advanced Device and Materials Testing Laboratory (ADMATEL) of DOST-ITDI. TGA was performed using Perkin Elmer STA 6000. The temperature program used was from 30 °C to 950 °C with heating rate of 10 °C/min. The atmosphere considered was nitrogen at 20 mL/min. The initial weight of samples ranges from 10.4 mg to 11 mg.

3. Results and Discussion

3.1 Tensile Properties

Figure 4(a) shows that the tensile strength of the composites decreases as the fiber content increases. The average tensile strength obtained for neat epoxy is 49.10 MPa while 26.80 MPa for 20 wt.% fiber loading. The decrease in tensile strength is due to the considered length of salago fiber (5–7 mm) which could be less than the critical fiber length. This resulted to inefficient stress transfer between the fibers and matrix at higher tensile loads [4, 11]. On the other hand, the tensile modulus of epoxy (3.10 GPa) increases as the fiber content increases to 20 wt.% (5.69 GPa) as presented in figure 4(b). The improvement on the tensile modulus indicates good adhesion between the fibers and matrix since the composites with higher fiber contents resisted extension better than neat epoxy.

![Figure 4. Tensile properties of salago fiber composites: (a) tensile strength and (b) tensile modulus.](image)

3.2 Flexural Properties

Figure 5(a) shows that the addition of short salago fibers in epoxy resin decreases the flexural strength. The average flexural strength obtained for neat epoxy is 63.5 MPa while 32.3 MPa for 20 wt.% fiber loading. One factor that contributed to a lower strength is the 2-D random orientation of the short fibers. The composites became vulnerable to bending since the load direction is perpendicular with respect to the fiber length. For the flexural modulus of epoxy (2.11 GPa), there is a slight improvement at 10 wt.% (2.24 GPa) as shown in figure 5(b). This is because of the good fiber-matrix interaction. However, the decrease at 15 wt.% (1.54 GPa) is due to the effect caused by the 2-D fiber orientation.

![Figure 5. Flexural properties of salago fiber composites: (a) flexural strength and (b) flexural modulus.](image)
3.3 Impact Strength

Figure 6 shows that the average Izod impact strength of neat epoxy (48.10 J/m) decreases at 5 wt.% fiber loading (31.7 J/m) but it increases at 10 wt.% (55.50 J/m) and 20 wt.% (60.8 J/m). The decrease in impact strength at 5 wt.% fiber content could be due to the tendency of the fibers to hinder deformation that resulted to a decrease in absorbed energy [12]. On the other hand, the improvement of Izod impact strength of the composites at 10 wt.%, 15 wt.% and 20 wt.% fiber loading is due to the good adhesion between the treated salago fibers and epoxy resin.

3.4 TGA Results

Results for TGA are presented in table 1 and figure 7. The initial weight loss observed in the samples is due to the removal of moisture and other present volatile components [13]. At the 1st degradation stage, there is an increase in peak temperature from 5 wt.% to 10 wt.% but it decreases as the fiber loading further increases from 15 wt.% to 20 wt.. The 10 wt. % fiber loading has the highest initial peak temperature (207.69 °C) with a corresponding weight loss of 16.41 % as compared to the peak temperature obtained for neat epoxy (189.05 °C). This is because of the good interaction between the alkali treated fibers and epoxy resin which is also supported by the results obtained in the tensile modulus and impact strength of the composites. The addition of salago fibers at 10 wt.% in the epoxy matrix significantly increased the initial peak temperature by about 18.64 °C. This indicates improvement on the thermal stability of the composite. However, at the 2nd degradation stage, the peak temperature of epoxy (369.39 °C) decreases slightly as the salago fiber content increases from 5 wt.% (367.27 °C) to 20 wt% (364.23 °C). The corresponding weight loss at this stage is associated with the decomposition of cellulose and hemicellulose of the fibers [14]. At the final temperature of 950 °C, the amount of residue left ranges from 4.10 % to 5.95 %.

| Sample        | 1st stage | 2nd stage | Residue at 950 °C |
|---------------|-----------|-----------|-------------------|
|               | Peak temperature (°C) | Weight loss (%) | Peak temperature (°C) | Weight loss (%) | (%)          |
| Neat Epoxy    | 189.05    | 16.24     | 369.39           | 78.09           | 5.67         |
| 5 wt% fiber   | 195.28    | 16.87     | 367.27           | 78.97           | 4.17         |
| 10 wt% fiber  | 207.69    | 16.41     | 366.14           | 78.73           | 4.86         |
| 15 wt% fiber  | 199.11    | 16.74     | 365.98           | 79.16           | 4.10         |
| 20 wt% fiber  | 177.25    | 15.43     | 364.23           | 78.62           | 5.95         |

Figure 6. Impact strength of composites.

Figure 7. TGA curves of composites.
4. Conclusion

The mechanical and thermal properties of short salago fiber reinforced epoxy resin composites were evaluated in this study. Based from the results of mechanical testing, there have been improvements on the tensile modulus and impact strength of the composites. As compared to neat epoxy, the tensile modulus and the impact strength obtained for 20 wt.% salago fiber loading increased by about 83.5% and 26.4%, respectively. However, noticeable decrease in tensile and flexural strengths were obtained as the fiber content increases which may be due to the short fiber length and 2-D random orientation of fibers. Lastly, the initial peak temperature obtained from TGA increases from 5 wt.% to 10 wt.% fiber loading which indicates improvement on the thermal stability.

5. References

[1] Alzebdeh K I, Nassar M M A and Arunachalam R 2019 Effect of fabrication parameters on strength of natural fiber polypropylene composites: statistical assessment Measurement 146 195–207
[2] Sanjay M R, Siengchin S, Parameswaranpillai J, Jawaid M, Pruncu C I and Khan A 2019 A comprehensive review for natural fibers as reinforcement in composites: preparation, processing and characterization Carbohyd. Polym. 207 108–121
[3] Sanjay M R, Madhu P, Jawaid M, Senthamaikannan S, Senthil S and Pradeep S 2018 Characterization and properties of natural fiber polymer composites: a comprehensive review J. Clean. Prod. 172 566–581
[4] Balla V K, Kate K H, Satyavolu J, Singh P and Tadimeti J G D 2019 Additive manufacturing of natural fiber reinforced polymer composites: processing and prospects Compos. B. Eng. 174 106956
[5] Kabir M M, Wang H, Lau K T and Cardona F 2012 Chemical treatments on plant-based natural fibre reinforced polymer composites: an overview Compos. B. Eng. 43 2883–92
[6] Jeyapragash R, Srinivasan V and Sathiyamurthy S 2020 Mechanical properties of natural fiber/particulate reinforced epoxy composites – a review of the literature Mater. Today Proc. 22 1223–7
[7] Pouriman M, Caparanga A R, Ebrahim M and Dahresobh A 2017 Characterization of untreated and alkaline-treated salago fiber (genus wikstroemia spp.) J. Nat. Fibers 15 296–307
[8] Pouriman M, Dahresobh A, Caparanga A R, Moradipour M and Mehrpooya M 2018 Morphological and physicomechanical analysis of high-density polyethylene filled with salago fiber J. Appl. Polym. Sci. 135 46479
[9] Pouriman M, Dahresobh A, Moradipour M and Caparanga A R 2019 Thermal and nondestructive analysis of high density polyethylene filled with milled salago fiber J. Appl. Polym. Sci. 136 47873
[10] Oduosote J K and Oyewo A T 2016 Mechanical properties of pineapple leaf fibre reinforced polymer composites for application as prosthetic socket Journal of Engineering and Technology 7 125–139
[11] Kerni L, Singh S, Patnaik A and Kumar N 2020 A review on natural fiber reinforced composites Mater. Today Proc.
[12] Eng C C, Ibrahim N A, Zainuddin N, Arrifin H and Yunus W M Z W 2014 Impact strength and flexural properties enhancement of methacrylate silane treated oil palm mesocarp fiber reinforced biodegradable hybrid composites Sci. World J. 2014 1–8
[13] Saw S K, Purwar R, Nandy S, Ghose J and Sarkhel G 2013 Fabrication, characterization and evaluation of luffa cylindrica fiber reinforced epoxy composites Bioresources 8 4805–25
[14] Chin S C, Tee K F, Tong F S, Ong H R and Gimbin J 2020 Thermal and mechanical properties of bamboo fiber reinforced composites Mater. Today Commun. 23 100876