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Mechanical properties, fracture morphology and thermal analysis of untreated and alkaline treated salago fiber epoxy laminated composites

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1. Introduction

Polymer composites with synthetic fiber reinforcements (e.g., glass and carbon fibers) became popular choices in some industries like aviation and automotive due to their superior properties such as excellent strength, high moduli, good fatigue resistance, low weight, etc [1]. However, the main problems encountered in the utilization of these synthetic materials are their harmful effects to the environment as well as the serious health concerns associated with their manufacturing processes [2]. Due to these challenges, there is a need to explore natural materials that have high potential on becoming alternatives to synthetic fibers. Particularly, natural fibers as reinforcing materials in polymer composites can offer some remarkable benefits over synthetic fibers. This is because, natural fibers are environmentally friendly, economical, lightweight, abundant and relatively safe [3]. Aside from these advantages, reinforcing natural fibers in polymer composites can enhance the mechanical properties such as tensile, flexural and impact strengths [2, 4]. The addition of natural fibers such as jute, coir, kenaf and sisal in polymer matrices can also improve the tribological performance which can make these composites suitable for components where good wear resistance is a requirement [5]. In terms of thermal properties, some natural fibers (e.g., sugarcane bagasse and oil palm empty fruit bunch) can improve the thermal insulation of composites through the possible reduction on the thermal conductivity [6]. Due to the characteristics of natural fiber composites, they have gained acceptance in several applications which include...
interior parts for automobiles or vehicles, components for renewable energy technologies, construction materials, electronic coverings, medical implants, furniture, sports equipment, etc. [7].

However, some drawbacks of natural fibers include weak adhesion to some polymer matrices, lower strength than synthetic fibers, poor thermal behavior, low moisture resistance and possible complications on the manufacturing process of composites [8]. Some studies reported that reinforcing natural fibers in polymer composites can increase the water absorption which might be related to the hydrophilic nature of fibers [9–11]. Also, composites with natural fibers have high flammability which could limit their usage in situations that involve exposure to high temperatures [12]. Nevertheless, the performance of natural fiber composites can still be enhanced further. One of the techniques is by subjecting the natural fibers to chemical treatment. A common method is by immersing the natural fibers in an alkaline or sodium hydroxide solution which can remove portion of hemicellulose and lignin that may result to a more uniform fiber surface and better adhesion to the matrix material [13]. In a study about Ensete stem fibers that were reinforced in unsaturated polyester, the alkaline treatment improves the flexural strength and tensile modulus as compared to the untreated fiber composite [14]. Likewise, the effect of alkaline treatment on bamboo fiber composites showed better tensile and flexural properties than the untreated fiber epoxy composites [15]. Similarly, the chemical treatment of Typha fiber using 5% NaOH solution enhanced the mechanical properties, water contact angle and thermal stability of epoxy composites [16]. However, longer hours and higher concentration of alkaline treatment can possibly cause damage on the fiber surface [17].

As for the matrix materials used for natural fibers, some of the common thermoset polymers are epoxy, polyester, vinyl ester and phenolic resins [18]. In the case of epoxy resin, it is used in composites because of its excellent characteristics. It has high mechanical strength, great chemical stability, low shrinkage rate, good electrical insulation, incredible corrosion resistance and strong adhesion ability to various surfaces [19]. Some of the less common natural fibers that were already reinforced in epoxy include Arundo donax, Sansevieria cylindrica, Manicaria saccifera, isora and napier grass fibers [20]. In the Philippines, a natural fiber that still needs further investigation in its potential use in composites is salago (genus Wikstroemia spp.). This bast fiber is mostly utilized in paper production. In a research work, it was reported that salago has a high cellulose content of about 79%. Also, the same study obtained a tensile strength of 1187 MPa for the untreated salago fiber where the alkaline treatment caused about 10.5% improvement [21]. Notably, this tensile strength of salago fiber is higher than the reported tensile strengths of some common natural fibers (e.g., flax, kenaf, sisal, banana, jute, cotton and coir) [22]. Likewise, the cellulose content of salago is comparable with some natural fibers [4]. In terms of polymer composites, a related study used milled salago fiber as filler in high-density polyethylene (HDPE) where better mechanical properties were achieved for the alkaline treated fiber composites than the untreated fiber composites. Although, the tensile and impact strengths of the treated salago composites were lower than neat HDPE, improvements on flexural properties were still attained [23]. Similarly, the HDPE composites with treated salago fibers achieved higher coefficient of linear thermal expansion and better thermal stability than the untreated fiber HDPE composites [24]. In a research work, the effect of reinforcing randomly oriented salago fiber (5 to 7 mm long) in epoxy resin were examined. It was revealed that the stiffness and impact strength of the composites improved due to the salago fiber reinforcement but significant decrease on tensile and flexural strengths were obtained which may be related to the shorter length considered [25]. Nonetheless, the enhancements on moduli and impact strength could make these salago fiber epoxy composites as candidate materials in drones or robots. In drones, it can probably be used as an airframe which serves as the main support that holds the electronic components together. Some characteristics of materials used in drone airframes have great strength to weight ratio and high modulus of elasticity [26]. A natural fiber like bagasse has already been studied for its use as a reinforcement in composites for the frame of drones where the flexural strength and impact toughness have been evaluated [27]. Similar to drone airframes, salago fiber composites can also be considered as an alternative material for the chassis of mobile robots.

The high tensile strength and cellulose content of salago fiber as well as the enhancements on properties that can be achieved by reinforcing this fiber in polymer matrices as discussed above made it more significant to be studied further in composites. Also, if the salago fiber has been reinforced in epoxy resin in a different orientation or condition aside from the randomly arranged short fibers, some possible improvements can still be achieved especially in terms of the mechanical properties. This could also make the salago fiber composites more suitable in the mentioned applications and even in other fields that require higher mechanical strengths. Hence, the researchers of this study reinforced salago in the form of fiber sheets, where the effects of fiber loading and alkaline treatment on the properties of epoxy resin composites were investigated.
2. Methods

2.1. Materials
Salago barks were given by a local paper manufacturer in Cavite, Philippines. West System 105 Epoxy Resin and 206 Hardener were used as the matrix for the composite. Sodium hydroxide was bought in a medical supply store in Manila, Philippines.

2.2. Fiber Sheet Preparation
Salago barks were first dipped in tap water for 5 min at room temperature to partially soften them. The wet salago barks were then struck with a mallet repeatedly to separate the fiber bundles until they became wider and thinner. The procedure is slightly similar to the typical preparation of bark cloths [28]. A wood rolling pin was then used to flatten the salago sheets. These were then cleaned in distilled water to remove dirt before subjecting the fiber sheets to air drying for 3 days. Figure 1 shows the photos of the salago barks and the fiber sheets.

2.3. Chemical treatment
The fiber sheets were immersed in a 5 wt. % sodium hydroxide solution for 1 h. The weight ratio of the fiber to the sodium hydroxide solution was 1:20 [29]. The alkaline treated salago fiber sheets were then washed in distilled water three times. Both the treated fiber and the untreated fiber sheets were sundried for 5 days. Figure 2 shows the immersion of the salago fiber sheets in 5 wt. % NaOH solution and the set-up for the Sun drying of the fibers.

2.4. Composite preparation
The process applied for the preparation of the composites was an open molding method through the use of silicone molds. Two shapes of silicone molds were used. One is for the specimens for tensile test while the other is for the specimens of flexural and impact tests [25]. The sundried salago fiber sheets were cut based on the dimensions required for mechanical testing. On the other hand, West System 105 epoxy resin was mixed manually with 206 slow hardener for 5 min using the mix ratio of 5:1 by weight. A portion of the mixture was first poured into the silicone molds to partially fill them. The salago fiber sheets were then placed on the silicone molds carefully. A plastic spoon was used not only to push the fiber sheets in the silicone molds but also to distribute the resin. After the last layer of salago fiber sheet was laid, the remaining mixed resin was poured to fill the molds. The composites were cured for 2 days at room temperature. The cured specimens were then removed from the silicone molds. For both alkaline treated and untreated fiber composites, samples with varying number
of sheets from 1 to 3 were prepared. Table 1 presents the details of the composites as well as their corresponding approximate fiber loading by weight. Figure 3 shows the cut salago fiber sheets and the molding of the composites. On the other hand, figure 4 presents the prepared specimens for mechanical testing.

2.5. Fourier transform infrared spectroscopy (FTIR)

The untreated and alkaline treated fiber sheets were subjected to FTIR Analysis. The equipment used was Perkin Elmer FT-IR Spectrometer Frontier while the technique applied was attenuated total reflectance (ATR) accessory.

| Specimen Label | Condition            | No. of Fiber Sheets | Fiber Loading (wt.%) |
|----------------|----------------------|---------------------|----------------------|
| E0             | Neat Epoxy           | 0                   | 0                    |
| U1             | Untreated            | 1                   | 5.4                  |
| U2             | Untreated            | 2                   | 10.8                 |
| U3             | Untreated            | 3                   | 16.2                 |
| A1             | Alkaline Treated     | 1                   | 4.6                  |
| A2             | Alkaline Treated     | 2                   | 9.3                  |
| A3             | Alkaline Treated     | 3                   | 13.9                 |
2.6. Optical microscopy of fiber sheets
The surfaces of untreated and alkaline treated salago fiber sheets were subjected to optical microscopy. The equipment used was Carl Zeiss High Power Microscope.

2.7. Tensile test
The test method applied for tensile test was ASTM D638 using Instron UTM Model 5585H. The speed of testing was 5 mm min\(^{-1}\). Five specimens per sample type were tested. For the data, the average and standard deviation of at least 3 specimens per sample type were considered since the trials where the instrument did not give reading during yielding were excluded in the calculation.

2.8. Flexural test
The test method applied for flexural test was ASTM D790 using Shimadzu UTM AGS-50kNXD. The ratio of the support span to the depth was 16:1. For each sample type, the average and standard deviation of the flexural strength and modulus of 5 specimens were calculated.

2.9. Izod impact test
The test method applied for impact test was ASTM D256 – 06a using Zwick/Roell 5.5P Izod Pendulum Tester with 1.0 J Capacity. For each sample type, the average and standard deviation of the Izod impact strength of 5 notched specimens were calculated.

2.10. Fracture analysis
The untreated and alkaline treated fiber composites with the highest tensile properties obtained were subjected to field emission scanning electron microscopy (FESEM) for fracture analysis. The instrument used was Dual Beam Helios Nanolab 600i. The accelerating voltage was 5 kV while the beam current was 0.17 nA.

2.11. Thermal analysis
Specimens E0, U3 and A3 were subjected to thermogravimetric-differential thermal analysis (TG-DTA) using Perkin Elmer STA 6000. The sample holder used was a ceramic crucible. The temperature range considered was from 30 °C to 950 °C at a heating rate of 10 °C min\(^{-1}\). From 30 °C to 600 °C, the atmosphere used was nitrogen at 20 ml min\(^{-1}\) which was switched to oxygen from 600 °C to 950 °C.

3. Results and discussion

3.1. FTIR analysis
The FTIR spectra of the raw and alkaline treated salago fibers are presented in figure 5. The peak that can be observed at 3339 cm\(^{-1}\) corresponds to the O-H stretching in the fiber components while the peak at 2916 cm\(^{-1}\) is related to the C-H stretching vibration \([21]\). The rise in the spectral intensity of the alkaline treated fiber at around 3339 cm\(^{-1}\) could be due to the increase in hydroxyl group caused by the removal of some fiber constituents affected by the chemical treatment \([30]\). Comparing the alkaline treated fiber to the raw salago fiber,
there has been a significant decrease or disappearance of peaks at around 1731 cm$^{-1}$ and 1243 cm$^{-1}$. The peak at 1731 cm$^{-1}$ corresponds to the C=O stretching of acetyl group of hemicellulose while the peak at around 1243 cm$^{-1}$ is associated to the C-O stretching of acetyl group of lignin [29]. Thus, the decrease at these peak intensities is due to the reduction of the hemicellulose and lignin content of the fibers caused by the alkaline treatment. There is also a peak at around 1627 cm$^{-1}$ which might be related to the water content present in the fibers [31]. Moreover, the peaks that can be noticed at around 1424 cm$^{-1}$ and 1316 cm$^{-1}$ could be related to the vibration of

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**Figure 6.** Microphotographs of the fiber surface (a) x50 magnification of raw salago fiber (b) x50 magnification of alkaline treated fiber (c) x200 magnification of raw salago fiber (d) x200 magnification of alkaline treated fiber.

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**Figure 7.** Results of Tensile Test of Composites (a) Tensile Strength (b) Tensile Modulus.
carboxylic acid group in pectin and the wagging vibration of methylene group in hemicellulose and cellulose, respectively [32]. On the other hand, the peak at around 1026 cm \(^{-1}\) is associated to the C-O stretching of the cellulose and hemicellulose of the fibers [33].

3.2. Microstructure of fiber sheets

Figure 6 shows the microphotographs of the surface of untreated and alkaline treated salago fiber sheets.

It can be noticed in figures 6(a) and (c) that the presence of the other fiber constituents is more evident in the untreated fiber sheet as compared to the alkaline treated fiber sheet. This is because NaOH treatment can clean the fiber surface while removing other components such as oils, waxes and some impurities [34]. However, as seen in figures 6(b) and (d), the alkaline treated salago fiber sheet have noticeable and larger holes as compared to the untreated salago fiber sheet. The development of the holes was initially caused by the repeated pounding of the fiber sheets during their preparation. These holes were broadened further by the alkaline treatment which could be related to the partial removal of hemicellulose and lignin. Apparently, NaOH treatment may also lead to fibrillation of fiber bundles due to the elimination of major binding fiber constituents specifically when used at high concentrations [35].
3.3. Tensile properties

The tensile strength and modulus of the composites are shown in figure 7. All of the composites obtained better tensile properties than the neat epoxy. The tensile strength and modulus of E0 are 53.4 MPa and 2.7 GPa, respectively. U3 has the highest tensile strength (81.6 MPa) followed by A3 (76.1 MPa). On the other hand, A3 has the best tensile modulus (5 GPa) followed by U3 (4.91 GPa). In the case of U3, the improvements obtained for the tensile strength and modulus as compared to E0 are about 52.8% and 81.9%, respectively. Based on the pattern observed, the tensile strength and modulus increase as the number of fiber sheets in the epoxy resin increases. A related study also reported an increasing tensile strength in epoxy resin composites as the number of layers of abaca increases from 1 to 3 fiber mats [36]. Remarkably, the untreated salago fiber composites have better tensile strengths than their alkaline treated fiber composite counterparts. This could be due to the presence of holes in the alkaline treated fiber sheet as observed in its microstructure as presented in figure 6. Another factor that could have affected the tensile strength is the fiber loading. As seen in table 1, the alkaline treated fiber composites have lower fiber content by weight as compared to their corresponding untreated fiber composites. This could be due to the dissolution of some non-cellulosic components in the NaOH solution as also reported in a research work where the fiber mass of different types of bamboo fibers has decreased after alkaline treatment [37]. Notably, a higher fiber content can lead to a higher tensile strength similar to the results obtained in a related study about sisal fiber reinforced epoxy resin composites [38]. On the other hand, in terms of tensile modulus, it appears that the composites with treated fiber sheets have slightly higher values than their untreated fiber composite counterparts. This could be due to the effect of the alkaline treatment on the fiber surface that improved the adhesion to the matrix [15, 35].

3.4. Tensile fracture morphology

The FESEM images of the fractured surface of U3 and A3 after tensile test are shown in figure 8. It can be noticed in figure 8(a) that the pulled fibers of U3 is more unbound than the fiber ends of A3 which were still held together by the epoxy as seen in figure 8(b). Moreover, it can be observed that the fiber pullouts are more evident in U3 than A3 as shown in figures 8(c) and (d). Also, there are holes on the fractured surface of U3 which might be due to the fiber pullout as seen in figure 8(e). In the case of A3, some fiber breakage occurred with very slight fiber
Figure 10. Results of Izod Impact Test of the Composites.

Figure 11. Thermal Analysis of Composites (a) TG Curve (b) DTG Curve (c) DTA Curve.
pullout as presented in figure 8(f). These remarks were quite similar to the observations of a related study on the cryo-fractured surface of epoxy composites reinforced with untreated and treated Borassus fibers [39]. The mentioned observations on the fractured surface of the composites indicate that the alkaline treatment enhanced the adhesion of the salago fiber sheet to the epoxy resin.

### 3.5. Flexural properties

Figure 9 shows the flexural strength and modulus of the composites. All composites obtained better flexural properties than the neat epoxy. The flexural strength and modulus of E0 are 71.3 MPa and 2.25 GPa, respectively. U3 obtained the highest flexural strength (98.1 MPa) and modulus (3.5 GPa). As compared to E0, the flexural strength of U3 is 37.6% higher while the modulus improved by about 55.6%. Similar to the tensile properties, the flexural strength increases as the number of salago fiber sheets in the composites increases. The pattern obtained for flexural strength is almost the same with the flexural modulus. In a research work, the flexural properties of epoxy resin composites reinforced with jute fibers enhance as the fiber content increases [40]. It can be seen that the flexural strength and modulus of treated fiber composites (i.e., A1 and A2) are slightly higher than their untreated fiber counterparts namely U1 and U2. This could be due to the improved adhesion of the salago fiber sheets to the epoxy resin caused by the alkaline treatment. In a related study, the epoxy resin composites with alkaline treated abaca fiber mats obtained better flexural strength than the untreated fiber composites [41]. Conversely, both the flexural strength and modulus of U3 are higher than A3 by about 8.9% and 19%, respectively. This could be associated to the higher fiber loading of U3 than A3. Also, the presence of holes in the alkaline treated fiber sheets as observed in the microphotographs could have affected the flexural strength of composite A3.

### 3.6. Izod impact strength

The Izod impact strengths of the composites are shown in figure 10. All of the composites have higher impact strength than the neat epoxy. The average Izod impact strength of E0 is 13.1 J m\(^{-1}\). Outstandingly, U3 obtained the highest impact strength (81.8 J m\(^{-1}\)) which is more than 6 times as E0.

Generally, the untreated fiber composites have greater impact strengths than the alkaline treated fiber composites. The lower impact strength of the treated fiber composites could be related to the presence of holes in the salago fiber sheets which affected the overall stress transfer. In a research work, the composites with alkaline treated sugar palm fibers, which were soaked in NaOH solution for 1 h, obtained lower Izod impact strengths than the untreated fiber epoxy composites. This is associated to the weakening of the region between the fibrils caused by the removal of some fiber constituents which led to poor dissipation of energy [42].

### 3.7. Thermal analysis

Figure 11 shows the thermogravimetric (TG), derivative thermogravimetric (DTG) and differential thermal analysis (DTA) curves of E0, U3 and A3. The TG-DTG data of the specimens are presented in table 2 while the DTA results are shown in table 3. The initial peak temperature obtained for E0 is 197.54 °C which could be due to dehydration or the volatilization of moisture present in epoxy resin [43].

| Sample | Peak Temperature (°C) | Heat Flow |
|--------|---------------------|-----------|
| E0     | 363.06              | Exothermic|
| U3     | 345.29              | Exothermic|
| A3     | 348.79              | Exothermic|

| | Temperature (°C) | Weight Loss (%) | Weight at 600 °C (%) |
|---|-----------------|----------------|-------------------|
| Onset | Peak | Range | E0 | U3 | A3 |
| — | 197.54 | 30.21–286.21 | 10.523 | 9.885 |
| 345 | 367.81 | 286.21–600 | 79.592 |
| — | — | 30.07–270 | 13.092 |
| 337 | 366.95 | 270–600 | 73.364 | 13.544 |
| — | 151.52 | 30.04–270.64 | 12.363 |
| 347.6 | 371.63 | 270.64–600 | 76.132 | 11.505 |

Table 2. TG-DTG Results of Composites E0, U3 and A3.

Table 3. DTA Results of Composites E0, U3 and A3.
From the TG curves, it can be noticed that the weight losses of U3 and A3 from 30 °C to 270 °C are greater than E0. The additional weight loss on U3 and A3 below 200 °C could be due to the evaporation of the water present in the fibers [44]. The maximum weight loss of the samples, which is greater than 70%, occurred in the temperature range from 270 °C to 600 °C. At this temperature range, the onset decomposition temperatures are 345 °C for E0, 337 °C for U3 and 347.6 °C for A3. Correspondingly, the DTG peak temperatures for E0, U3 and A3 are 367.81 °C, 366.95 °C and 371.63 °C, respectively. These peak temperatures are associated to the decomposition of the epoxy resin and the hemicellulose and cellulose of salago fibers [25]. Also, at this degradation stage, A3 has the highest onset and peak temperatures while U3 obtained the lowest onset and peak temperatures. These higher onset and peak temperatures obtained for A3 indicate improvement on thermal stability. This enhancement could be related to the partial removal of amorphous fiber components (e.g., hemicellulose and lignin) caused by the chemical treatment [16]. Similarly, a related study obtained higher onset and peak temperatures for the epoxy composite with treated luffa fibers as compared to the untreated fiber composite [45]. This could be due to the better adhesion of the treated fibers to epoxy resin than the untreated fibers.

After the major weight loss, which is above 70%, the residual weight at 600 °C for the composites E0, U3 and A3 are 9.885%, 13.544% and 11.505%, respectively. A3 obtained a lower residual weight at 600 °C than U3. This could be related to the lesser lignin content of the treated fibers as compared to the untreated fibers reinforced in U3. Apparently, lignin can add to charring as discussed in a research work where the treated pineapple leaf fibers exhibited lower residue than the untreated fiber [46]. On the other hand, the DTA peak temperatures obtained for E0, U3 and A3 are 363.06 °C, 345.29 °C and 348.79 °C, respectively. In a related study, exothermic peaks above 330 °C were obtained for sago fiber epoxy composites which could be linked to the decomposition of hemicellulose and cellulose [47].

4. Conclusion

In this study, the mechanical, morphological and thermal properties of epoxy resin composites reinforced with untreated and alkaline treated salago fiber sheets were characterized. Generally, the mechanical properties improved as the salago fiber sheets in epoxy resin increases from one to three layers. The composite with three sheets of untreated salago fibers obtained the highest tensile strength (81.6 MPa), flexural strength (98.1 MPa) and impact strength (81.8 J m \(^{-1}\)) with respective improvements of 52.8%, 37.6% and more than 6 times as compared to neat epoxy. However, alkaline treated fiber composites obtained lower strengths than their untreated counterparts which could be related to the presence of holes on the fiber surface as viewed through optical microscopy. Nevertheless, some improvements on tensile and flexural moduli were observed on the alkaline treated fiber composites which could be due to the good adhesion of the fibers to epoxy as observed on the FESEM images of the tensile fractured specimens. Similarly, TG-DTA revealed slight improvement on thermal stability of the alkaline treated salago fiber composite with onset temperature of 347.6 °C and peak temperature of 371.63 °C. This enhancement is associated to the partial removal of amorphous fiber components as supported by the FTIR analysis. Furthermore, the composite with untreated salago fiber sheets can be used in applications that require high impact strength and stiffness such as airframe for drones, chassis for robots and components for sporting goods.

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Data availability statement

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

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