CHARACTERIZATION AND TREATMENT OF WATER HYACINTH FIBERS FOR NFRP COMPOSITES

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Keywords: NFRP, biocomposite, water hyacinth, natural fiber, fiber treatment

1 Introduction
The widespread application of carbon, aramid, glass and other high-modulus and high-strength fibers as reinforcement for plastic materials has pose problem in handling its final disposal due to resistance to degradation of the plastic composite. In addition, these fibers are derived from non-renewable resources; hence, the future demand for the use of these fibers in high strength applications will push prices higher as their supply dwindles.

To address this issue, many research investigations have been undertaken on the use of natural fibers as plastic reinforcement to substitute for the synthetic fibers in medium-strength industrial applications. Natural fibers have a major advantage over synthetic ones such as they are environmentally friendly, cheaper, more lightweight, and have high specific mechanical and thermal properties. The presence of the biodegradable fibers improves the overall mechanical properties of the polymeric composites as well as contributes to its capacity for carbon sequestration.

Natural fiber-reinforced plastic (NFRP) composites have been developed for various applications where inexpensive, lightweight, high specific strength and modulus, and eco-friendly materials are needed. Mostly in the form of wood/plastic composites, the current market for NFRP’s is relatively large and is expected to increase further with the introduction of more variants and higher quality NFRP products to the global market.

Agricultural fibers such as abaca (Manila hemp), jute, flax and hemp have shown increasing potential application as fiber reinforcements for fabricating green composites. However, there is an underlying concern regarding the increase of their production, as an increased demand for these fibers may potentially compete with other agricultural land use such as food production; this may be addressed by focusing on natural fibers derived from agricultural by-products or any non-agricultural plants such as water hyacinths. Water hyacinth (Eichhornia crassipes) is an invasive and resistant plant that has infested the water bodies of most tropical and subtropical countries, including the Philippines. In addition, this plant has high growth rate, which makes it a potential renewable source of fibers.

In this study, fibers extracted from water hyacinth found locally in Philippine waters are used to reinforce orthophthalic-type unsaturated polyester (ortho-UP) resin, and the composite’s mechanical properties and carbon storage potential are evaluated. The high moisture absorption and poor wettability of the natural fibers is remedied by grafting functional moieties using coupling agents as fiber treatment [1].

2 Experimental
2.1 Materials
Water hyacinth fibers (shown in Fig. 1) were obtained from stalks harvested along the shores of Laguna Lake in the Philippines (shown in Fig. 2). The decortications of the fibers were done at the Philippine Textile Research Institute (PTRI). The ortho-UP matrix resin (R10-103) was purchased from Polymer Products Philippines, Inc. All other chemicals used in this study are reagent grade chemicals.

2.2 Fiber preparation
The water hyacinth stalks were retted for 7 days to remove the lignin, waxes, hemicelluloses, and any adhering dirt. The retted fibers were dried in an oven at 40°C and cooled at room temperature. The fibers were then cut into 5 cm length before subjecting to fiber treatments.
The decorticated fibers were then retted for another week to further remove adhering dirt as well as waxes and other foreign materials from the fiber. The retted fibers had then undergone washing and air-drying at room temperature followed by oven-drying at 50°C for 1-2 hours. Before chemical treatment, the dried water hyacinth fibers were first cut into 12 cm length (flexural test specimen) and 17 cm (tensile test specimen). Then the mass of water hyacinth required to fabricate 6 samples of the composite was calculated based on the dimension of the mold used for the production of the NFRP composites.

2.3 Fiber treatment

In this study, the fibers were subjected to two types of chemical treatments: alkali treatment and enzyme treatment. Sodium hydroxide was used for the alkali treatment while xylanase was used for the enzyme fiber treatment.

2.3.1 Alkali treatment

In this study, there are three concentrations of NaOH that were used namely: 2.5%, 5% and 10% NaOH (by weight). A previous study considered the 15% NaOH concentration by weight, but the fibers were damaged caused by high alkali concentration [2]. The samples were treated with NaOH for 2, 4, 8, 12 and 24 hours. Full factorial design methodology was used in this study, which determined that there must be 15 runs for the tensile specimen and 15 runs for the flexural specimen production, for a total of 30 runs for the alkali-treated composites. Each combination of parameters (time and concentration) had at least 5 specimens.

2.3.2 Enzyme treatment

There are four enzyme concentration employed in the optimization studies: 1%, 2%, 4% and 8% xylanase by volume. These concentrations were based on literature survey conducted wherein on the study by John and Anandjiwala in 2009 [3] on the chemical modification of non-woven flax fiber-reinforced polypropylene composites using 2% zein solution and 2 hours of fiber immersion.

In this study, the water hyacinth fibers were soaked in xylanase solution for 1, 2, 4 and 8 hours. There were a total of 30 runs conducted for the xylanase-treated fibers- the tensile and flexural tests each having 15 runs- with 5 specimens in each run.

2.4 Fiber mat fabrication

After treatment, the water hyacinth fibers were flushed with copious amount of water. An equimolar solution of HCl was prepared beforehand for the neutralization of the samples. The pH was noted each time. The neutralized samples were then washed with water before air-drying at ordinary temperature prior to the production of fiber mats. The neutralized samples were lightly pressed to remove excess water, air-dried overnight and then sandwiched between aluminum sheets before placing in a Carver hydraulic press. The samples were pressed at about 1.5 MPa pressure for about one hour. Afterwards, the fiber mats were slightly pressed with tissue paper, especially at the edges, to absorb excess moisture and the water left in the fibers after pressing. The fiber mats were then put in a convection oven for final drying. The temperature was set at about 60°C for 2 hours. The dried fiber mat was then weighed and cut into proper dimension for composite production.

2.5 NFRP lamination

Flat bars and aluminum sheets were used as mold to produced water hyacinth fiber reinforced unsaturated polyester laminates. The flat bars were designed to follow ASTM D638 for tensile specimen and ASTM D790 for flexural specimen production.

MEKP was mixed with R-10-103 resin in a 3% by weight formulation to accelerate polymerization of the resin. A small amount of Durawax® from Polymer Products Philippines, Inc. was first applied on the surface of the mold to serve as a release agent after curing of the composite. Then after drying of the release agent hand lay-up technique of composite production was applied. A small quantity of the resin pre-mix was initially poured into the mold, followed by the hand layup of the prepared water hyacinth fiber mats. The mats were first soaked in the resin bath to completely wet the mats before the actual lamination process. Afterwards, another layer of resin was poured on the water hyacinth fiber mat. This procedure was continued until all the fiber mats were completely covered by the resin to produce the water hyacinth fiber-reinforced composites.

The resin mixture was then placed inside a vacuum desiccator to remove extra bubbles that formed during the lay-up and mixing of the resin. The composite was then allowed to stand for three hours.
at ambient temperature in a fume hood. After 3 hours, the composite was taken out of the mold by removing the flat bars. The composite was cured in a convection oven for 3 hours at a temperature of 150°C. Then the cured composite was cooled at ambient temperature for 24 hours before subjecting the sample to mechanical testing.

2.6 Analytical and testing methods

The water hyacinth fibers and the resulting NFRP composites were subjected to the following analysis for characterization and property measurements:

1. The water hyacinth fibers were sent to the Fiber Industry Development Authority (FIDA) of the Philippines for chemical composition analysis. The constituents of the fiber analyzed for were α-cellulose, holocellulose, hemicellulose, ash and lignin.
2. The fibers were subjected to scanning electron microscopy using Hitachi S-3400. The voltage was set at 1 kV, current of 26 μA at a 200-300× magnification. SEM studies were also conducted to study the fracture surface of the NFRP specimens after mechanical strength testing.
3. FTIR studies are conducted to determine the functional groups present in the chemical structure of materials under investigation.
4. Shimadzu AGS-X Universal Testing Machine was used to characterize the tensile and flexural properties of water hyacinth NFRP.

3 Results and Discussion

3.1 Fiber characterization

It is necessary to determine the changes that occur in the water hyacinth fibers after treatment with alkali and enzyme. A detailed study on the properties and characteristics of untreated and treated fiber will yield sufficient data as to how the fiber will behave as reinforcement to a polymer. In this study on fiber characterization, only chemical analysis, tensile property evaluation of treated and untreated fiber, FTIR studies and SEM studies were conducted due to time constraint. However, these studies are fundamental to evaluating the fiber as reinforcement.

3.1.1 Chemical analysis of water hyacinth fiber

The analysis of the decorticated fiber chemical composition, specifically the *E. crassipes* variety of water hyacinth, found along the shores of Laguna de Bay shows a high content of cellulose evidenced by high values of holocellulose and alpha cellulose in the fibers. The chemical composition of the decorticated water hyacinth fiber reveals a high amount of holocellulose, 83.94%, and alpha cellulose, 61.63%. A complete tabulation of chemical composition is shown in Table 1. The results also reveal a very low percentage of lignin (3.87%) and some hemicellulose (16.26%). During the process of decorticsations, large quantities of non-fiber constituents are removed from the water hyacinth stalk. The amount of water hyacinth fiber that can be recovered from 100 kg of water hyacinth stalks harvested from the source is only about 2-3 kg (2-3% recovery). These large quantities of wasted material consist of waxes, pectin, other foreign material, some hemicellulose and sizable amounts of lignin removed from the retted fibers. Decortication of the fibers is succeeded by further retting, which degrades some other organic materials attached to the decorticated fibers as well as other hemicellulose and lignin left during the previous treatment through biological action.

3.1.2 Single fiber tensile test

The tensile properties of single untreated and treated water hyacinth fibers were measured based on ASTM D3379-75. At least 20 specimens were tested for each parameter combination, hence there are at least 300 samples for alkali-treated fibers and 330 samples for enzyme-treated fibers, for a total of 630 samples.

A paper frame with thickness 220 GSM was used as support where fiber specimens were attached using glue stick or epoxy. The frame has a dimension of 100 mm by 20 mm as depicted in Fig. 3. A rectangular hole at the middle measures 25 mm by 13 mm were the fiber will be attached. During testing the sides of the hole will be cut so that only water hyacinth fiber strength will be tested using Shimadzu AGS-X. The fiber gage length is 25 mm and the testing cross head speed is 0.5 mm/min. Tensile testing results show a general decrease in the strength of the fiber, an improvement in the fiber elongation and a decrease in the modulus of elasticity or the rigidity of the fiber. This observation is in agreement with other previously published papers on the characteristic mechanical properties of natural fibers when subjected to chemical treatment.
Chemical treatment accompanied the removal of some lignin and hemicellulose backbone that cement cellulose and lignin together. Several studies also suggest that alkali treatment leads to an increase in the amount of amorphous cellulose at the expense of crystalline cellulose.

Fig. 4 is a graphical representation of tensile strength of alkali-treated fiber as a function of immersion time at different concentrations. In general, the graph shows a decrease in tensile strength as a function of immersion time except for the 24-hour treatment where the strength rebounds to a higher value compared with 12-hour treatment. Treatment duration plays a significant role in the resulting strength of alkali-treated fiber. Longer treatment time removes further the lignin and hemicellulose backbone of the fiber as well as exposing the microfibrils that make up the individual water hyacinth fiber. As more microfibrils are exposed the resulting strength of the fiber as a bundle or collection of smaller fibers called microfibril is similarly reduced.

Alkali fiber treatment also affects fiber elongation to a certain extent, as shown in Fig. 5. It can be seen that the elongation is increased for all the treated samples at different concentrations and treatment periods.

The tensile strengths of xylanase-treated fibers (shown in Fig. 6) show an overall of decreasing trend with the increase in immersion time and enzyme concentration. This may be attributed to fiber damage caused by enzymatic reaction. This damage may be compared to the degrading effect of alkali treatment that causes a change in chemical structure such as that of cellulose in the fibers partially changing from crystalline cellulose I into amorphous cellulose II [4].

3.1.3 FTIR fiber analysis

FTIR results suggest an increase in the number of hydroxyl units exposed to the surface due to the elimination of some lignin and hemicellulose after treatment with 5% NaOH and 10% NaOH solutions. However, for the 2.5% NaOH concentration the newly exposed –OH units are deactivated by the alkali- thus, a slight decrease in –OH peak can be observed.

FTIR-ATR spectra for untreated water hyacinth fibers show a broad and intense band in the range of 3100-3800 cm⁻¹ due to the –OH vibration of cellulose structure as shown in Figure 7. It can be observed also that the presence of –OH group is significantly reduced as a result of treatment using 2.5% NaOH concentration. The largest change is about 94% reduction in absorbance observed for the 12-hour immersion time.

Fig. 8 shows the FTIR fiber analysis at different NaOH concentrations. The results show that the 5% NaOH concentration is the most effective in the deactivation of –OH units present along the fiber surface. On the other hand, the 2.5% NaOH concentration has the effect of exposing more –OH units as more lignin and hemicellulose are removed from the fibers.

The increase in peaks as observed in from the FTIR spectra for 5% and 10% NaOH treated fibers confirms the earlier analysis regarding the removal of some hemicellulose and lignin structure along the fiber surface. Hemicellulose structure is branched and networked unlike cellulose that is mostly a linear polymer. This network structure does not provide enough –OH groups to be exposed to the surface of the fibers. Lignin, on the other hand, is believed to be polycyclic and aromatic in nature with some degree of branching that contain even less –OH groups.

From Fig. 9, it can be observed from the FTIR spectra of xylanase-treated fibers that an increase in concentration and immersion time increases the number of exposed –OH units. This can be attributed to the hemicellulose and lignin that cover the surface before are being removed by the enzyme. Xylanase specifically catalyzes the hydrolysis of xylosidic linkages of xylan hemicellulose polymer. Since hemicellulose is the material that acts as a “bridge” connecting lignin and cellulose together, the cleavage of the bonds that link hemicellulose molecules together will eventually lead to the separation of hemicellulose and lignin from the cellulose backbone of the fiber. The removal of hemicellulose and lignin will expose more cellulose molecules to the surface of the fibers, which translates to increase in –OH absorbance. Since xylanase only catalyzes the cleavage of hemicellulose molecules it will not be consumed in the reaction; it is hypothesized that the unconsumed xylanase may still react with the exposed cellulose molecules: the hydrophilic part of xylanase enzyme will react to the hydrophilic fiber and the hydrophobic end of the enzyme will link with the
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3.1.4 Scanning electron microscopy

Fig. 10 shows the roughening effect of the alkali treatment on the surface of the water hyacinth fibers. The increased roughness promotes better mechanical interlocking between the fibers and the matrix resin [5]. However, excessive alkali treatment can cause severe fiber damage.

Fig. 11 shows the effect of the xylanase treatment to the fiber surface. The SEM micrographs of xylanase-treated fibers show partial removal of the waxy material covering the surface of untreated water hyacinth fibers.

These SEM images of treated and untreated water hyacinth fiber showing the surface morphology confirm the previously shown results of FTIR studies regarding the removal of some of the constituent materials from the fiber surface. These materials include waxes, pectins, hemicellulose and lignin providing more exposure to cellulose microfibril. The SEM analysis shows the removal of waxy surface films/structures covering the fiber confirming earlier result.

3.2 Characterization of water hyacinth NFRP

3.2.1 Mechanical strength and modulus

Mechanical properties are tested to evaluate the effect of alkali treatment on the surface property of fiber and matrix. The effectiveness of modifying the fiber surface can be assessed by comparing the resulting mechanical strengths obtained at different concentrations and treatment time.

Flexural strength of treated water hyacinth NFRP composite was measured under three point bending test. The results for alkali treatment at different concentrations and immersion time are presented in Fig. 12, while the ones for the xylanase treatment are shown in Fig. 13. For the tensile strength, the effects of treatment concentration and immersion time using NaOH and xylanase on the composite strength are shown in Fig. 14 and Fig. 15, respectively.

A summary of measured mechanical property values is given in Table 2 for comparative analysis. Comparing the flexural strengths of alkali-treated, xylanase-treated and untreated water hyacinth NFRP composites, the alkali-treated composite has the best tensile strength (194.884MPa). This is 51.65% higher than the tensile strength of untreated fiber reinforced composite and 6.71% higher than the xylanase-treated fiber-reinforced composite. In contrast, the flexural moduli of alkali-treated fiber reinforced composite are significantly lower than the xylanase-treated and untreated fiber reinforced composite. The flexural modulus of the alkali-treated fiber-reinforced composite is 8.77% and 54.13% lower than those of the untreated and xylanase-treated NFRP composites, respectively.

In summary, the alkali-treated fiber reinforced composite has the best tensile and flexural strength compared with xylanase-treated and untreated fiber reinforced composite. However, the tensile and flexural moduli of xylanase-treated fiber reinforced composite are superior to either the alkali-treated or untreated fiber-reinforced composite.

The chemical treatment of water hyacinth fibers significantly affected the resulting mechanical properties of the water hyacinth fiber-reinforced composite. This is due to the improvement of interfacial adhesion between the natural fibers and the polymer matrix. The changes in the surface structural properties as evidenced by the FTIR and SEM studies are congruent with these results.

It can be observed from Table 2 that the flexural strength of alkali-treated FRP and xylanase-treated FRP have no significant difference, but the flexural moduli for the two different treatments show otherwise. One possible explanation for this observation is that the stress developed in a NFRP specimen subjected to a fixed load is independent of the material properties, while the deflection depends on the material’s modulus. Hence, the stress from a given load is the same for both alkali-treated NFRP or xylanase-treated NFRP’s, but the strain may be different even if the dimensions are the same. In other words, flexural modulus is a property that is intrinsic to a material while flexural strength can be affected by defects in the material.

3.3.2 SEM at the fracture surface

Shown in Fig. 16 is the SEM image of the fracture surface for the untreated fiber reinforced composite. The micrograph shows gaping holes and fiber pull out as a result of poor interfacial adhesion between polymer and matrix.

Fig. 17 and Fig. 18 are SEM micrographs of alkali- and xylanase-treated water hyacinth fiber-reinforced...
composites, showing short fiber protrusion from the fractured sample as well as little fiber pull-out. These SEM micrographs show better interfacial interaction between the alkali- and xylanase-treated fibers and the polymer matrix. The use of chemical modification on natural fibers improves the ability of the fibers to have a better adhesion with the polymer matrix through improve mechanical interlocking with matrix in the case of the alkali treatment. Xylanase, on the other hand, can also serve as a coupling agent to bind the natural fibers and the polymer matrix together. The improved interfacial adhesion is evident on the results of the mechanical testing showing significant difference between the tensile and flexural properties of the alkali- and enzyme-treated NFRP’s against those of the untreated NFRP. Chemical modification of the fibers also prevents the formation of hydrogen bonds and improves the fiber surface property, which, in turn, improves the fiber’s interaction with the polymer matrix.

3.2.3 Water absorption

The effect of moisture on the mechanical strength of water hyacinth NFRP composite has been tested at different immersion periods. Fig. 19 illustrates the change in tensile properties after a 24-hour water immersion. This resultant lowering of the strength capabilities of the water-soaked composite may be due to the breakdown of the natural fibers within the composite as a result of leaching of the water-soluble materials present. Another consequence of water immersion may be the decomposition of these materials into lower molecular weight lignin, hemicelluloses, and other degradation products. The introduction of water into the composite also causes disruption in the bonding between the fibers and the matrix after prolonged exposure to water molecules, ultimately resulting to disintegration, delamination, cracking and/or swelling, and a significant decline in mechanical properties [6].

4 Conclusions

Water hyacinth fiber was treated with alkali and xylanase before laminating into an ortho-UP composite. The resulting green composite was assessed based on improvement in the mechanical properties. The mechanical property changes were supported by FTIR and SEM studies of chemically treated water hyacinth fiber as well as fractographic studies of tensile specimen. The following conclusions were made based on the results of this study:

1. Alkali and xylanase treatment reduce the tensile strength of water hyacinth fibers while the corresponding tensile strain is significantly improved.

2. The most effective alkali concentration that yielded the best tensile strength is 10% NaOH w/w treated for 4 hours. The best flexural strength for alkali-treated fiber-reinforced composite is achieved through treatment with 5% NaOH w/w for 8 hours.

3. The best treatment condition for xylanase treated fiber reinforced composite to yield the highest enhancement in tensile strength is 2% xylanase w/w for 1 hour, but 4% w/w xylanase-treated fiber-reinforced composite soaked for 1 hour yields superior flexural strength.

4. Alkali treatment yields slightly higher flexural and tensile properties compared with xylanase treatment. Tensile and flexural strength of alkali treated fiber reinforced composite are 10.25% and 6.71% higher compared with xylanase-treated fiber-reinforced composite.

5. Alkali treatment yields superior tensile and flexural properties compared with untreated fiber reinforced composite. Alkali treatment yields 51.65% and 74.66% improvement in flexural and tensile strength respectively.

References


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Fig. 1. Water hyacinth stalk fibers.

Fig. 2. Water hyacinth stalk obtained along the shores of Laguna de Bay, Philippines.

Fig. 3. Single fiber specimen for tensile testing.

Fig. 4. Tensile strength of alkali-treated water hyacinth fibers at different immersion times and at different alkali concentrations.
Fig. 5. Tensile strain of alkali-treated water hyacinth fibers at different immersion times and at different alkali concentration.

Fig. 6. FTIR spectra of water hyacinth fiber treated with 2.5% NaOH at different immersion times.

Fig. 7. FTIR spectra of water hyacinth fibers immersed for 2 hours at three different NaOH concentrations: 2.5%, 5% and 10%.

Fig. 8. FTIR spectra of water hyacinth fibers treated with 1% xylanase for 1 h, 1% xylanase for 4 h and 8% xylanase for 1 h.
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Fig. 10. SEM images of untreated water hyacinth fibers and those of fibers subjected to 6 hours of alkali treatment at different concentrations.

Fig. 11. SEM micrograph of water hyacinth fiber treated with xylanase.

Fig. 12. Effect of NaOH concentration and immersion time to the flexural strength of water hyacinth NFRP composites.

Fig. 13. Effect of xylanase concentration and immersion time to the flexural strength of water hyacinth NFRP composites.
Fig. 14. Effects of NaOH concentration and immersion time to the tensile strength of water hyacinth NFRP composites.

Fig. 15. Effects of xylanase concentration and immersion time to the tensile strength of water hyacinth NFRP composites.

Fig. 16. SEM image of the fracture surface of untreated water hyacinth NFRP composite.

Fig. 17. SEM image of the fracture surface of the alkali-treated water hyacinth NFRP composite.
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Table 1. Chemical composition of decorticated water hyacinth fiber.

<table>
<thead>
<tr>
<th>Component</th>
<th>%</th>
</tr>
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<tbody>
<tr>
<td>Ash</td>
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<tr>
<td>Lignin</td>
<td>3.78</td>
</tr>
<tr>
<td>Holocellulose</td>
<td>83.94</td>
</tr>
<tr>
<td>α-Cellulose</td>
<td>61.63</td>
</tr>
<tr>
<td>Hemicellulose</td>
<td>16.26</td>
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</table>

Table 2. Flexural and tensile properties of alkali- and xylanase-treated water hyacinth fiber-reinforced composites and untreated fiber reinforced composite.

<table>
<thead>
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<th></th>
<th>Untreated</th>
<th>NaOH</th>
<th>Xylanase</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flexural Strength</td>
<td>128.5</td>
<td>194.9</td>
<td>182.6</td>
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<tr>
<td>Flexural Modulus</td>
<td>5.920</td>
<td>5.401</td>
<td>11.77</td>
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<td>Tensile Strength</td>
<td>30.76</td>
<td>53.73</td>
<td>48.73</td>
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<tr>
<td>Tensile Modulus</td>
<td>1.107</td>
<td>1.141</td>
<td>1.441</td>
</tr>
</tbody>
</table>

Fig. 18. SEM image of the fracture surface of the xylanase-treated water hyacinth NFRP composite.

Fig. 19. Effect of water absorption on the tensile properties of the water hyacinth NFRP composites.

Table 1. Chemical composition of decorticated water hyacinth fiber.

Table 2. Flexural and tensile properties of alkali- and xylanase-treated water hyacinth fiber-reinforced composites and untreated fiber reinforced composite.