



Research Article

Treatment of water hyacinth fibers to improve mechanical and microstructural properties of green composite materials

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ABSTRACT

In this study, the water hyacinth was used as a reinforcement of green composites. The chemicals for treatment of water hyacinth are solutions of alkaline, soap and water to determine water absorption. The chemical treated process of the water hyacinth fibers had important influences to the chemical structures and mechanical properties of the green composites. Micrographs analyses of scanning electron microscope (SEM) show obviously roughness of fiber surface after the chemical treatment as well as arrangement of structures among the water hyacinth fibers. Similarly, the results of analysis by Fourier transform infrared spectroscopy (FT-IR) showed that most of the intensity of the absorbed peaks was decreased markedly at the wavenumbers of 1375 cm^{-1} and 1542 cm^{-1} . These are related to lignin extraction with alkaline solution, effectively. In particular, the compressive strength of the water hyacinth/roving composite has high value approximately to that of fiberglass composites (unfriendly composites). The water hyacinth/roving composites have significantly improvements in flexural and compressive strengths and this proves that the environmentally friendly composite responded to high requirements for various applications.

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INTRODUCTION

The water hyacinth is an aquatic plant species native to South America, mainly in the Amazon basin. Since 1895, the water hyacinths were grown to Australia. At the beginning of the 20th century, the water hyacinth was also found in Asian countries such as India in 1902, Vietnam in 1905 and Malaysia in 1910.

The water hyacinths grow rapidly in humid environments, especially in the summer. They are able to grow at very high densities (more than 60 kg/m^2). Therefore, the water hyacinths can increase double on areas in a period of 5 to 15 days with mean density around 2 million trees per hectare and weight from 270 to 400 tons [1, 2]. One

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of the most important applied characteristics of the water hyacinth is absorption ability of heavy metals in water such as copper, zinc, nickel, cadmium, chromium, arsenic, mercury and lead [3]. In the paper industry, the water hyacinth is used as high quality raw material for pulp production. In addition, the water hyacinth is also braided to make rope or fibers board with low cost. The water hyacinth also has the potential as a convenient and low-cost raw material source for the recovery of antioxidant [4], anti-aging and cancer solutions [5]. In the agricultural fields, the water hyacinth is used with a small scale as green or organic fertilizer, poultry feed or as a buffer in fruit transport [6].

The composites based on natural fibers have lower mechanical properties than that of synthetic fibers, especially fiberglass [7-9], carbon fibers [10] and easy to absorb moisture related to contain hydroxyl groups. Some impurities related to natural fibers such as lignin, pectin, and others are surrounding the natural fibers which caused weak adhesion among the resin and fiber surfaces. The lignin is considered as the binder in fibrous cells, fills the gaps in the cell surfaces, and creates covalent bonds with hemicellulose. This leads to good bonding among cellulose fibers or among cellulose-hemicellulose. The important advantages of the natural fibers are low density, less abrasion, lower cost, recyclable and biodegradable which responded the requirements of environmentally friendly composites [11].

There have been several studies on natural fiber-reinforced composites such as coffee bean-reinforced composite [12], wood reinforced composite [13] and the water hyacinth-reinforced composite [14]. In this article, the water hyacinth fibers were treated in various chemical solutions and used as reinforcement for fabrication of the green composites.



Figure 1. The water hyacinth is grown at canals in Binh My commune, Cu Chi district, Ho Chi Minh City, Vietnam.

MATERIALS AND METHODOLOGY

The water hyacinth raw material was collected at canals in Binh My commune, Cu Chi district, Ho Chi Minh City, Vietnam. The chosen water hyacinths had well-developed and smooth bodies surfaces. The sizes of water hyacinth are uniform in diameter and length.

The treatment process of water hyacinth

The water hyacinth was washed for several times to clean from trash and impurities attached around the body. And then it was soaked in dilute salt and washed again. The water hyacinth was continuously carried out to dry to constant weight. Three solutions of NaOH, soap, and water were used to soak and determine the water absorbance of the water hyacinth. The process is to choose the suitable treatment solution. The next step, the water hyacinth was washed several times with warm water to reach neutral pH. After that, the water hyacinth was dried secondly to complete the treatment of the water hyacinth. Finally, the water hyacinth fibers were prepared into roving. The fabrication of water hyacinth roving is shown in Figure 2.

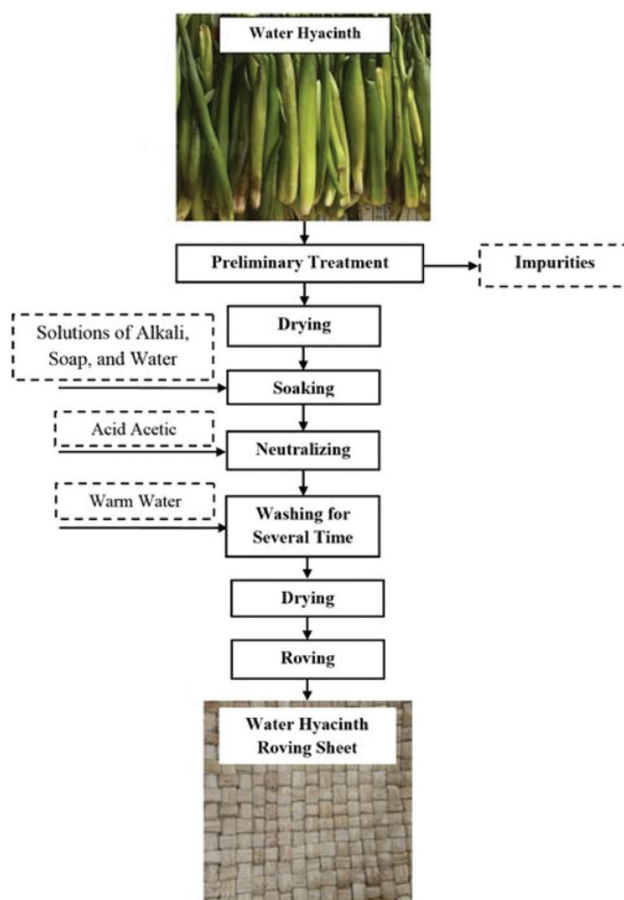


Figure 2. The processes of the water hyacinth treatment and fabrication for roving from the water hyacinth.

Methodology

This study used the water hyacinths to fabrication of the water hyacinth/roving composite materials with the experiments carried out as follows:

Determination of the water content in fresh water hyacinths: These experiments were carried out at 70°C for 24 hours to dry the water hyacinth in an oven with convection air to constant mass.

Determine the water absorption of the water hyacinth: After dried, the water hyacinth was soaked in the different solutions of NaOH, soap and water at room temperature for 3 hours. And then they were washed with water and soaked for one minute to determine for increase of the water hyacinth weight.

Determination of the treatment temperature: With the selected solution from alkaline, soap, and water, the water hyacinth was carried out at the treatment temperatures of 30, 40, 50, 60, and 70°C.

Analyses on morphologies of the water hyacinth using SEM micrographs: The materials were observed morphologies included the UWH (untreated water hyacinth) and TWH (treated water hyacinth). The experiments were analyzed at the Institute of Nanotechnology - Vietnam National University Ho Chi Minh City using the scanning electron microscope (SEM) JSM - 6480LV with magnification at 100X.

Method of Fourier transform infrared spectroscopy (FTIR): The FTIR spectrometer of Perkin Elmer was used to evaluate the changes in chemical bondings of the water hyacinth before and after treatment with alkaline solution. The experiments were conducted at the Institute of Nanotechnology - Vietnam National University Ho Chi Minh City.

Fabrication of the water hyacinth fiber reinforced composites by hand lay-up method: The unsaturated polyester (UPE) was used as a matrix, the MEKP (methyl ethyl ketone peroxide) amount was at 1% by weight of the UPE resin. The reinforcement material was the water hyacinth/roving. In which, the ratio of UPE resin and water hyacinth was at 7 and 3, respectively. The composite sheets were made by hand lay-up method with 2 layers of roving. The roving layers were prepared individually from the treated water hyacinth (TWH) and the untreated water hyacinth (UWH) with the sizes of fiber width at 2 mm and 4 mm.

Evaluation on mechanical strength of the water hyacinth fiber reinforced composites: The flexural strengths of the composite samples were tested according to ASTM D790 using TESTOMETRIC Machine with the moving speed of the clamps at 1 mm/min. The experiments were conducted at Polymer Laboratory, Ho Chi Minh City University of Technology and Education for the composite samples of TWH and UWH. The compressive strength of the water hyacinth fiber roving reinforced UPE composite samples were tested according to ASTM D695 using

INSTRON 5569 50K Machine at Mechanical Laboratory, Ho Chi Minh City University of Technology.

RESULTS AND DISCUSSION

Water content in the fresh water hyacinth

The drying experiments showed that the water content in the fresh water hyacinth has a very high proportion at 90%. The high value of water content is mainly related to porous structure in the cell walls of the water hyacinth. The experimental results are consistent with that of Akendo, (2008) at 91% and Saputra and Putri, (2017) at 90% [15, 16]. In comparison with some other plants, the water content of water hyacinth is much higher than sisal (10%) and coir (8.79%) because the water hyacinth belongs to the group of aquatic plants.

Water absorption of the water hyacinth

The water hyacinth samples were soaked in three solutions of water, sodium hydroxide (NaOH) and soap for 3 hours. Changes on outside colors and shapes of the samples before and after treatment are shown in Figure 3.

The results in Figure 4 show that the water absorption of the water hyacinth had the highest value at 1803.77% for treatment with NaOH solution. After treated with the soap solution, the water hyacinth had water absorption at 1622.73%. The lowest value of water absorption was at 672.92% for the water hyacinth soaked in water. Thus, treatment with NaOH solution had the ability to absorb water

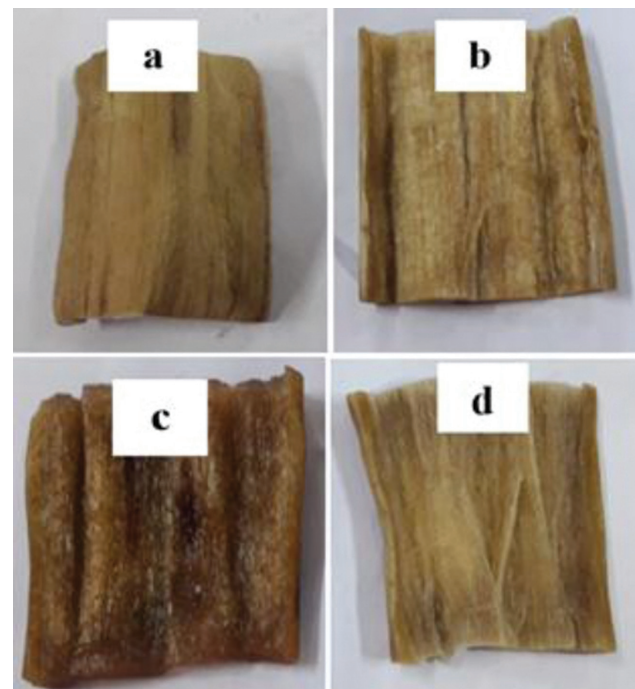
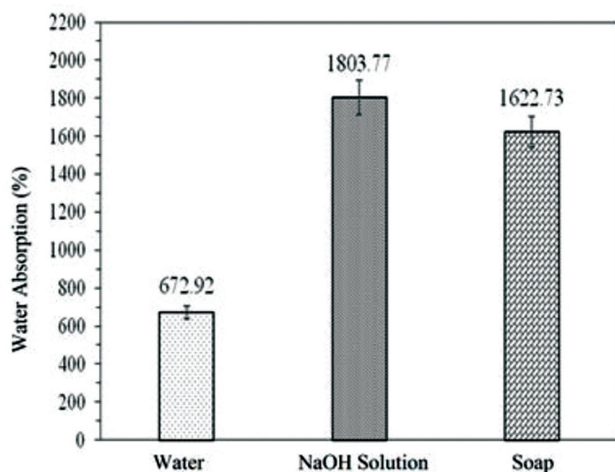


Figure 3. The water hyacinth before (a) and after treatment with water (b), NaOH solution (c) and soap (d).

Table 1. The various values of water absorption after treated in three solutions of the water hyacinth samples

The treatment solution	Water	NaOH	Soap
Water absorption (%)	672.92	1803.77	1622.73

**Figure 4.** The values of water absorption (%) of the water hyacinth in the various solutions.

2.68 times higher than water and 1.11 times higher than soap solution (Figure 4). The NaOH solution with strongly alkaline was able to hydrolyze a number of compounds in which NaOH molecules easily access into the capillaries in the water hyacinth fiber structures. Siroky et al. (2011) and Punyamurthy et al. (2012) used NaOH solution for treatment of several natural fibers [17, 18]. Sangeetha et al. (2015) also used NaOH solution for treatment of the water hyacinth fibers for 3 hours [19]. These experimental results are suitable with previous research and NaOH solution was chosen to carry out the next experiments.


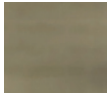



Effects of the extracted temperature to lignin content

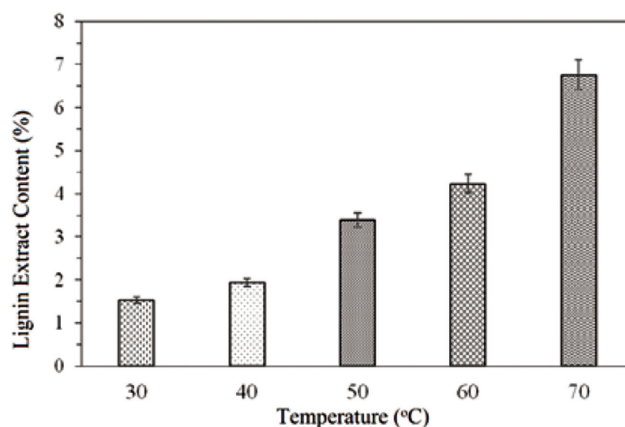
In this experiment, the water hyacinth samples were soaked in the solution of 10% NaOH with changes of temperature from 30°C to 70°C during 3 hours to obtain the extracted solutions. Then, the extracted solutions were filtered and determined the lignin content as shown in Table 2.

Table 2 shows that the lignin content increased slightly (around 26.67%) in range of the temperature from 30°C to 40°C. In contrast, from 40°C to 70°C, the lignin content increased dramatically (up to 250.78%). At the temperature of 70°C, the lignin extract has darker color than that of the extract under 60°C [20, 21]. Moreover, after treated at 70°C, the water hyacinth samples became brittle and easy to be broken.

At high temperatures (>70°C), the insoluble components such as lignin, pectin, and tannins were extracted and dissolved by NaOH solution. The extracted solution

Table 2. Changes of the extract color and the lignin content in the various extracted temperatures

Temperature (°C)	Color of Lignin extract	Lignin Mass (mg)	Lignin Content (%)
30		2.90	1.53
40		3.90	1.93
50		6.80	3.38
60		6.80	4.23
70		13.50	6.77

**Figure 5.** Changes of lignin content (%) in various range of treatment temperature.

changed into darker brown and the bonds among water hyacinth fibers became weak and easily destroyed. This is consistent with the case study of Nguyen (2021) [22].

Therefore, the temperature of 40°C was chosen for the treatment of the water hyacinth fibers.

Surface microstructures of the water hyacinth fibers using SEM

Two samples were used to analyze surface structures including the untreated water hyacinth (UWH) and the treated water hyacinth (TWH) in 10% NaOH solution at 40°C. Figure 6 shows that the surface of the UWH fiber is tightly bound together because some components such

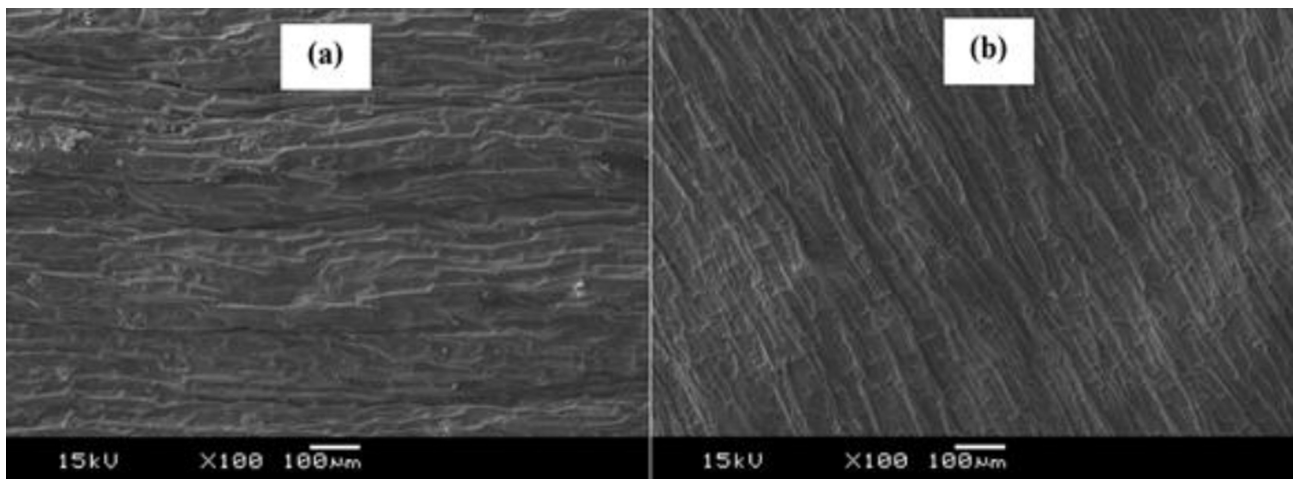


Figure 6. Surface structures of the water hyacinth fibers using SEM with morphologies of the UWH sample (a) and the TWH sample (b).

as lignin, pectin and wax were coated the water hyacinth fibers. In which, the lignin is considered as the binding component in the fibrous cells, filling the gaps in the cell walls [11]. Therefore, on the SEM micrographs, it is difficult to observe the surface of the UWH fibers as well as the arranged structures among the water hyacinth fibers (Figure 6a). After treatment in NaOH solution, the above components were partially dissolved depending on the concentration of NaOH solution. In addition, a portion of the hemicellulose in the water hyacinth was dissolved, resulting in a fractured and spongy water hyacinth fiber structure than the UWH sample. The hemicellulose-cellulose bonds were hydrolyzed as the extraction of lignin, exposing the water hyacinth fibers, leading to the surface of TWH became rougher than the UWH sample [20-22]. The morphology of treated water hyacinth fiber can be observed more clearly by the SEM micrographs (Figure 6b). In addition, the distance among the TWH fibers is larger due to the loss of mainly lignin and some insoluble components.

Characteristics of bonding vibrations in the UWH and TWH samples using FTIR analysis:

The UWH and TWH samples were used to characterize bonding vibrations using FTIR with the results as shown in Figure 7.

The wavenumber from 3380 to 3420 cm^{-1} is the strong oscillation zone of the O-H bond that characterizes cellulose and hemicellulose in the water hyacinth. The absorption peaks at wavenumber of 2855 cm^{-1} and 2923 cm^{-1} are the tensile oscillation regions of the C-H group in all the hydrocarbon components of cellulose, hemicellulose and lignin. The absorption peaks in the wavenumber range from 2445 to 2451 cm^{-1} are the tensile oscillation regions of the O-H group in the carboxyl function group. The wavenumber of 1641-1645 cm^{-1} represents the tensile

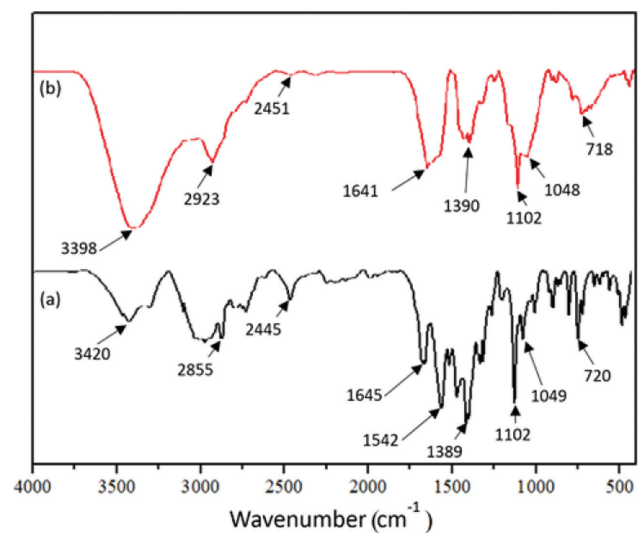


Figure 7. Surface structures of the water hyacinth fibers using SEM with morphologies of the UWH sample (a) and the TWH sample (b) .

oscillation zones of the C = O group and the absorbent water around the fibers [23]. The absorption peak at wavenumber of 1542 cm^{-1} exhibits a long oscillation of C-C bonding in the aromatic group of lignin. The wavenumber of 1389 or 1390 cm^{-1} is the vibration of the symmetrical C-H bond in the cellulose, the bending oscillation of the O-H and the tensile vibration of the C-O functional group [24, 25]. The peak at wavenumber of 1375 cm^{-1} is the oscillating effect of the C-O-C bond associated with the aromatic rings of the hemicellulose. The absorption peak at wavenumber of 1102 cm^{-1} is asymmetric tensile fluctuations of C-O-C related to cellulose and hemicellulose

structures. The wavenumber at 1048 and 1049 cm^{-1} is the tensile vibrations of C-O and O-H of the polysaccharide in cellulose [26, 27]. The peaks at wavenumber of 718 cm^{-1} and 720 cm^{-1} represent the presence of β -glycoside bonds in polysaccharide vessels.

Figure 7 shows that most of the intensity of the absorption peaks was reduced after the treatment process. However, the C-O-C groups bonded the aromatic rings in hemicellulose (absorption peak at wavenumber of 1375 cm^{-1}), the C-C bonds of lignin (absorption peak at wavenumber of 1542 cm^{-1}) were significantly reduced. The results show that the structures of water hyacinth have been altered due to hemicellulose and lignin being separated from the water hyacinth fibers treated using alkaline solution. In addition, a decrease of the absorbance intensity of the O-H bonds indicates the efficiency of the TWH surface treatment [28].

Effects of the sizes of Water Hyacinth/roving to mechanical properties of the green composites

Two samples of UWH/UPE and TWH/UPE composite are prepared with two sizes of 2 mm and 4 mm/roving. The TWH/UPE composites with two sizes of roving are shown in Figure 8. The differences on morphologies of both two samples of UWH/UPE and TWH/UPE composite are analyzed by SEM micrographs (Figure 6).

The composites materials were fabricated with four samples known as 2 mm-UWH/UPE; 4 mm-UWH/UPE; 2 mm-TWH/UPE; 4 mm-TWH/UPE and tested for

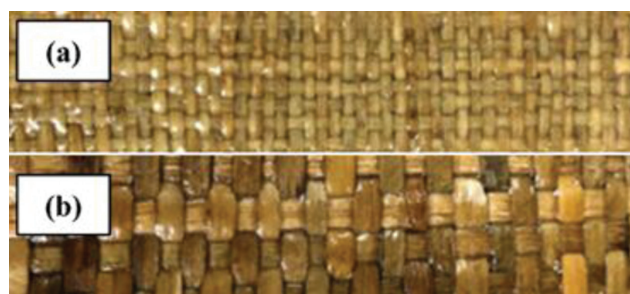


Figure 8. The TWH/UPE composite samples with two sizes of roving in 2 mm (a) and 4 mm (b).

mechanical strengths. The strengths of green composites are shown in Table 3.

Table 3 shows that the composite sheet of 2 mm-TWH/UPE has high flexural strength at 31.11 MPa. This value is 2.8 times higher than that of the 2 mm-UWH/UPE of 11.05 MPa. In addition, the flexural module of the sample of 2 mm-TWH/UPE composite was at 1304.54 MPa with 3.8 times higher than that of 2mm-UWH/UPE composite (344.16 MPa). It is noted that the samples of 4 mm-TWH/UPE and 4 mm-UWH/UPE composites have very low flexural strength at 7.16 MPa and 3.27 MPa, respectively. The water hyacinth surface without treatment in NaOH solution was covered with lignin and wax. They prevented the contact among water hyacinth surface and UPE resin caused low strength of the composite. In contrast, the water hyacinth treatment in NaOH solution removed the coating layers on the surface of water hyacinth mainly lignin, pectin, some minerals and others. Thus, the water hyacinth fibers were exposed leading to good bonded with UPE resin [20]. The experimental results demonstrate a significant improvement in the mechanical properties of the TWH/UPE based green composite. It is compared with flexural strength among water hyacinth reinforced composite with other natural fibers such as jute fiber, sisal fiber, banana fiber [29], pineapple fiber [30], coir [31]. The jute fiber/composite had the highest flexural strength at 76.53

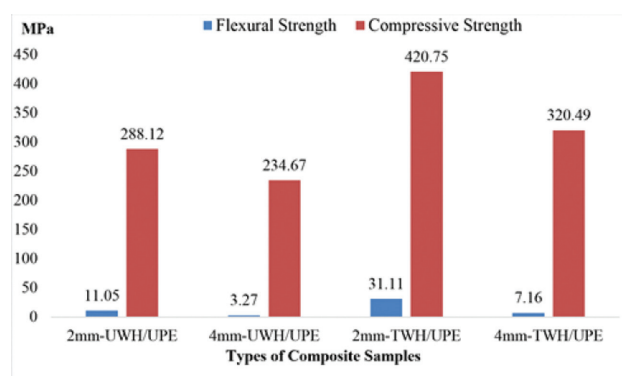


Figure 9. Mechanical strengths of four the water hyacinth reinforced green composites.

Table 3. Specifications on mechanical properties of water hyacinth/UPE composites materials based on the water hyacinth/UPE.

Mechanical Strength (MPa)	Composite Materials			
	2 mm-UWH/UPE	4 mm-UWH/UPE	2 mm-TWH/UPE	4 mm-TWH/UPE
Flexural Strength	11.05	3.27	31.11	7.16
Flexural Module	344.16	125.22	1304.54	550.27
Compressive Strength	288.12	234.67	420.75	320.49
Compressive Module	281.35	215.72	526.81	466.92

MPa and coconut fiber/composite had the lowest flexural strength at 30.00 MPa. The flexural strength of TWH/composite is close to that of the coir fiber/composite.

The green composites reinforced water hyacinth fibers were tested for compressive strength with the results as shown in Figure 9. The sample of 2 mm-TWH/UPE composite has the highest values of compressive strength at 420.75 MPa and compressive module at 526.81 MPa. The lowest values at 234.67 MPa for compressive strength and 215.72 MPa for compressive module are of the 4 mm-UWH/UPE composite. Specifically, the compressive strength of 2 mm-TWH/UPE is 1.31 times higher than that of 4 mm-TWH/UPE. This suggests that smaller width in 2 mm of roving fibers leads to higher fiber density resulting in higher mechanical properties than that of 4 mm. In addition, if the width of the roving fiber is large (4 mm), the interface among the matrix-reinforcement is low causing the poor compatibility leading to lower mechanical strength (Figure 9).

In comparison with the previous research on fiberglass and carbon fiber reinforced composites, the compressive strength of the water hyacinth fiber reinforced-composites is similar to that of fiberglass based-composite and it is 1.40 times lower than that of carbon fiber based-composite and others [8, 32-34]. The results showed that green composites reinforced the water hyacinth fibers have really high compressive strength reaching to the value of the composite reinforced by the traditional fiberglass. It is noted that the fiberglass based-composites are not environmentally friendly products. This proves the superiority of the water hyacinth reinforced composite with both highly mechanical strength and environmentally friendly material.

CONCLUSION

The water hyacinth fibers were treated with alkaline solution showed an important effect on the chemical structures and mechanical properties of the water hyacinth reinforced composites. The results of SEM micrographs and Fourier transform infrared spectroscopy analysis showed that the water hyacinth had markedly changed in chemical structures and morphologies. A change of the water hyacinth surface significantly increased the compatibility between UPE/matrix and the water hyacinth/reinforcement that greatly improves the mechanical properties of the green composites. Specifically, the compressive strength of the 2mm-TWH/UPE composite is similar to that of fiberglass based-composites. The success of the research creates a new direction by using water hyacinth as a potential material to produce green construction material with high mechanical strength and environmentally friendly materials.

AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

NOMENCLATURE

Subscripts

<i>ASTM</i>	Refers to American Society for Testing and Materials
<i>FTIR</i>	Refers to Fourier transform infrared spectroscopy
<i>MEKP</i>	Refers to methyl ethyl ketone peroxide
<i>SEM</i>	Refers to scanning electron microscope
<i>TWH</i>	Refers to treated water hyacinth
<i>UPE</i>	Refers to unsaturated polyester
<i>UWH</i>	Refers to untreated water hyacinth

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