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Preparation and Characterization of Water Hyacinth Stems (*Eichhornia Crassipes*) Impregnated with Modified Polystyrene

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ABSTRACT

Stems of water hyacinth contain cellulose, a natural polymer capable of interacting with modified polystyrene. This research aims to determine the physical and mechanical properties of water hyacinth stems and to trigger the modification of polystyrene with acrylic acid using the initiator benzoyl peroxide. The impregnation technique is used to modify water hyacinth stems with modified polystyrene, resulting in optimum conditions at an impregnation time of 3 h. The results of impregnation reveal a decrease in water content from 17.97% to 6.45%, a decrease in water absorption from 94.85% to 81.37%, and an increase in the modulus of elasticity (MoE) from 22.94 MPa to 191.18 MPa. Then FT-IR analysis was carried out to determine the success of the polystyrene modification, and the surface structure of the impregnated water hyacinth stems was examined using SEM. Therefore, from these data, it can be seen that modified polystyrene resin can improve the mechanical and physical properties of water hyacinth stems.

1. INTRODUCTION

Water hyacinth is a plant that has a high growth rate so this plant is considered a weed that can damage the aquatic environment [1]. It is feared that the growth of water hyacinth plants in the Porsea area, Tobasa Regency, North Sumatra Province will pollute the environment of Lake Toba, so it needs to be cleaned so that the beautiful and largest lake in Southeast Asia can be preserved [2].

One prospective effort to overcome water hyacinth weeds in lake waters is to use water hyacinth plants for crafts. Utilizing the potential of water hyacinth can increase the economic value of this material while contributing to global sustainability efforts. Sirisoda [3] has reported various products that can be made using water hyacinth woven products, such as packaging materials, textiles and construction materials. Water hyacinth crafts are in great demand in domestic and international markets. This is because crafts made from water hyacinth stems have a strong traditional ethnic charm. In fact, many water hyacinth craft items with certain models and qualities are exported to Europe and the United States, which are increasingly interested in goods produced from natural materials (back to nature). However, this water hyacinth craft is very susceptible to high humidity which causes it to have low strength and durability.

Efforts that are usually made to improve the quality of water hyacinth crafts are by providing finishing. The finishing that is often used is paint, and the durability of this craft can last for an average of 8 years. To increase water resistance, water hyacinth stems can be modified with synthetic polymers such as resin [4]. Traditionally, resin has been used as an adhesive and coloring agent [5]. One of the resins that is widely used is polystyrene. Polystyrene foam is widely known as styrofoam, styrofoam is waste that cannot be broken down by nature and if it is burned, the smoke produced by burning styrofoam can have bad consequences for health and the environment, causing global warming because the manufacture of styrofoam uses CFC gas as a blowing agent [6, 7]. If thrown away carelessly, Styrofoam waste will end up in the sea and damage marine ecosystems [8].

Because of its hydrophobic nature, polystyrene can be modified to reduce its hydrophobicity by adding a coupling agent with a polar group [9]. With the presence of acrylate groups, the polystyrene chains have polar groups and their hydrophobicity decreases, so they can interact with other polymers that are polar such as cellulose [10, 11].

Thus, drawing on previous studies and recognizing the considerable potential for manipulating water hyacinth stems and the significance of utilizing recycled polystyrene, the researcher aims to enhance the quality of water hyacinth stems treated with modified polystyrene. Furthermore, this investigation offers a novel approach to addressing the proliferation of water hyacinth in Lake Toba, along with the management of discarded polystyrene materials.

2. MATERIALS AND METHODS

2.1 Materials

The water hyacinth stem samples used as test samples in this study were taken from mature stems of water hyacinth plants in the Porsea area, Tobasa Regency, North Sumatra of the *E. crassipes* species, with an average height of 0.4-0.8 m.

Meanwhile, used polystyrene (PS) was obtained from the Medan city final disposal site. Acrylic acid, benzoyl peroxide (BPO), xylene, methanol, distilled water, and phenolphthalein were the compounds utilized in this study.

2.2 Water hyacinth stem preparation

The water hyacinth stem sample used was the *E. crassipes* type with an average height of 0.4-0.8 m. It was cleaned and then the specimen was cut to a length of 15 cm. After that, it is dried for 10 h at a temperature of 62-66℃. Good water hyacinth stems have the characteristics of being clean, perfectly dry, not growing mold and not rotting (blackening) [12].

2.3 Preparation of modified PS resin

The used PS was weighed at 9.2 g, put into a 250 mL bottom flask, dissolved in 100 mL of xylene on a hot plate stirrer until the used PS dissolved in the xylene. Then add 0.3 g of BPO then 1 mL of acrylic acid until mixed. Then observed at a temperature of 105℃ within 70 min. After mixing, the tool is turned off and the results are released. Afterwards 70 mL of methanol was dropped into the reflux product little by little and then the sediment and filtrate were separated. The resulting precipitate was then dried at a temperature of 120℃ [13].

2.4 Preparation of 0.05 N KOH solution

An amount of 0.28 g of KOH was dissolved in methanol. Then put it into a 100 mL measuring flask up to the flask mark, then 0.05 N KOH in methanol is obtained [14].

2.5 Calculating the percent of modified PS grafting

A total of 0.25 g of used PS that has been grafted and refluxed again with 25 mL of xylene until dissolved for 15 min. Add 3 drops of phenolphthalein indicator then titrate with 0.05 N KOH while hot. The titration is stopped when the color changes from clear to violet red and the volume of KOH used is recorded.

Percent Grafting Formula:

$$Percent\ Grafting = \frac{V\ KOH\ \times\ N\ KOH\ \times\ Mr\ Acrylic\ Acid}{M} \times\ 100\%$$

where:

V KOH = KOH used (mL)

N KOH = KOH normality (mek mL^{-1})

M = Mass (g)

Mr acrylic acid = Relative mass of acrylic acid (72.06 g mek^{-1})

2.6 Water hyacinth stem impregnation process using modified polystyrene resin

A total of 10 g of modified PS was refluxed for 15 min with 100 mL of xylene until it dissolved. Then, this treatment is carried out by impregnating the water hyacinth stems with varying times of 1, 2 and 3 h. In this study, the impregnation time was varied to determine the effect of extending the impregnation time on the physical and mechanical properties of the material.

2.7 Characterization and testing of impregnated water hyacinth stems

Characterization of impregnated water hyacinth stems includes physical and mechanical tests, namely water content, water absorption capacity, degradation, modulus of elasticity (MoE), Fourier Transform Infrared Spectroscopy (FT-IR) and surface analysis using Scanning Electron Microscope (SEM). Where all these tests are carried out before and after the water hyacinth stem samples are impregnated.

Determination of the elastic modulus involves computing the gradient of the stress–strain curve within the region of elastic deformation. The determination of water content involved the utilization of an oven to dry the samples at a temperature of 103 ± 2℃ for 12 h. the percentage of water content was derived by assessing the weight loss of the sample pre and post drying process. This procedure was iterated thrice to ensure consistent and reliable outcomes.

The water absorption capacity (%) is calculated using the formula as follows.

$$\text{Water absorption (\%)} = \frac{M2 - M1}{M1} \times 100$$

where, M1 represents the initial mass of the sample before immersion, and M2 signifies the mass of the sample subsequent to a 24-hours soaking period in distilled water [15].

3. RESULTS AND DISCUSSION

3.1 Characteristics of water hyacinth stems

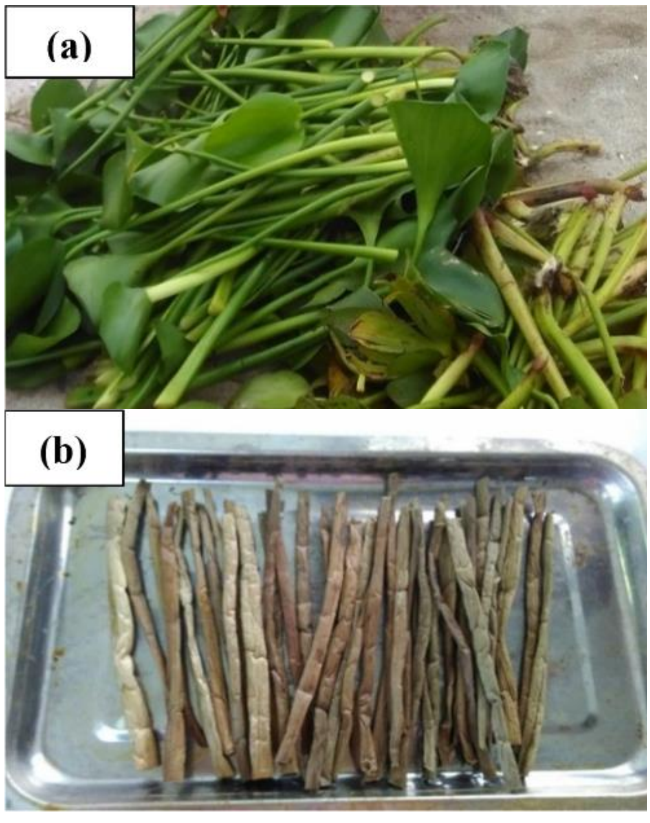

Figure 1. Water hyacinth stems (a) before preparation, and (b) after preparation

Table 1. Characteristics of dry water hyacinth stems

Characteristics	Quantity/Unit
Water Content	17.97%
Water Absorption Capacity	94.85%
MoE	22.94 Mpa

The water hyacinth stems used in this research were water hyacinth stems taken from the species *E. crassipes* which originates in the Porsea area, Tobasa Regency, North Sumatra, Indonesia, precisely at 2°26'24" N 99°9'25" E.

Initial preparations are made to make water hyacinth stems that are ready to be used as crafts (Figure 1). Generally, water hyacinth stems are dried in the open air for up to + 10 days. This treatment functions to clean and reduce the water content in the water hyacinth stems so that they are easier to create with crafts and last longer. The characteristics of dry water hyacinth stems can be seen in Table 1.

3.2 Impregnating resin modification

The used PS (Figure 2) will be modified first using the grafting method. Because PS is non-polar, while water hyacinth stems, the largest component of which is cellulose, are polar, these two materials cannot mix perfectly because of the difference in polarity. Therefore, the resin chain structure is modified to form polar groups, which will increase its interaction with the surface of the water hyacinth stem material.

Figure 2. Used polystyrene

In this study, the method used to modify the resin was by adding acrylic acid modifying material which functions as a coupling agent between PS and water hyacinth stems and benzoyl peroxide as an initiator (especially in the formation of free radicals). The addition of acrylic acid causes the polystyrene chains to have polar groups so that they are expected to interact with polar compounds. This modification process is carried out using the grafting method, where the result is the percentage of PS grafted which can be calculated using acid-base titration.

The grafting reaction that occurs by free radicals from monomers into hydrocarbons is a type of initiation through peroxide decomposition. Grafting of acrylic acid into PS occurs when the polymer becomes radical. The form of formation of acrylic acid grafting into PS can be in the form of cross-linking. The greater the amount of acrylic acid grafted onto the PS, the higher the grafting percentage. From experiments carried out using 0.3 mL of BPO and 1 mL of acrylic acid, the % grafting was obtained at 0.474%.

3.3 Water content and water absorption capacity of water hyacinth stems after impregnation

The graph of water content and water absorption capacity of water hyacinth stems after impregnation is shown in Figure 3. In this study, it was found that the initial weight of water hyacinth stems before impregnation was 17.97% and after impregnation for 1, 2, 3 h respectively was 14.92%; 10.77%; 6.45% where this treatment was carried out 3 times.

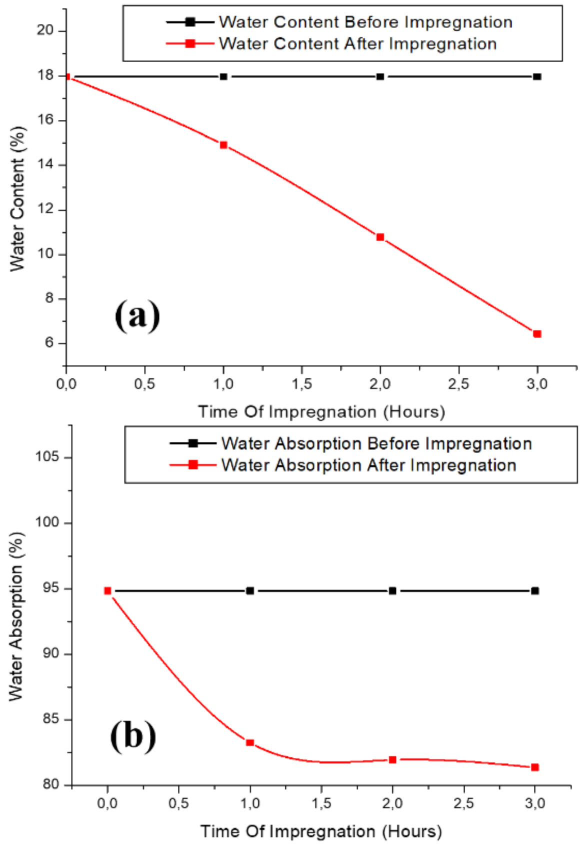

Figure 3. Graphs of (a) water content, and (b) water absorption capacity of water hyacinth stems

Hydrogen bonds that form between the OH groups of water hyacinth stems and water are what cause them to absorb water. The results of this research showed that the water absorption capacity before impregnation was 94.85%, and after impregnation with time variations of 1, 2, and 3 h, respectively, it was 83.26%, 81.94%, and 81.37%. Water hyacinth stems that have lower water content and water absorption capacity will have better quality in terms of resistance (degradation to fungi). So it can be concluded that optimum results were obtained at an impregnation time of 3 h.

This is in accordance with the report by Ishak et al. [15], who stated that because the polymer is hydrophobic, it encases and protects the fiber to absorb moisture. This is due to higher resin absorption with longer impregnation time, causing the fiber to have higher resistance to moisture.

3.4 MoE of water hyacinth stems after impregnation

Flexural strength testing is carried out to determine the resistance of a material to loading at the bending point and also to determine the elasticity of a material. The results of this research showed that before impregnation the MoE value was 22.94 MPa and after impregnation with time variations of 1, 2, 3 h respectively it was 27.94 MPa; 46.83 MPa; and 191.18 MPa (Figure 4). Water hyacinth stems that have a greater MoE value will be easier to weave into crafts and will not break easily. So the optimal results obtained are impregnation within 3 h.

According to prior investigations as documented by Poletto and Zattera [9], it is asserted that the utilization of a coupling agent results in an enhanced dispersion of cellulose fibers within the matrix, subsequently decreasing fiber-fiber interactions. This, in turn, enables the polymer matrix to effectively transmit the applied stress to the fibers, leading to an augmentation in deformation under flexural stress. Similarly, the enhancement in interfacial adhesion facilitates the transfer of stress from the polymer matrix to the more rigid cellulose fibers, consequently raising the flexural modulus. The incorporation of a coupling agent not only enhances the dispersion of cellulose fibers but also promotes a more uniform distribution of the applied stress. Consequently, a higher amount of energy is necessitated for debonding and fiber pullout, resulting in an escalation in impact strength [9].

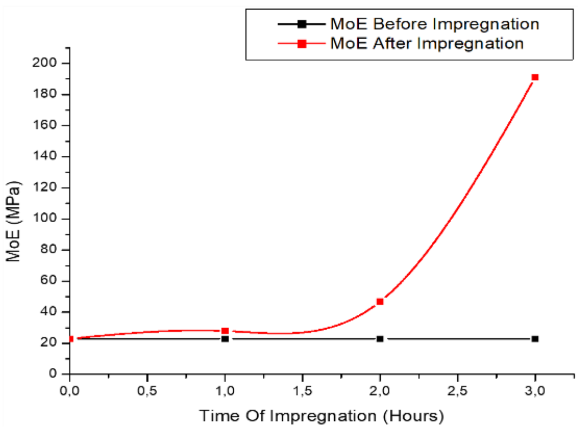

Figure 4. MoE graph of water hyacinth stems before and after impregnation

3.5 Degradation test

Degradation testing is conducted to determine the sample's durability, and biodegradation testing is performed with the aid of soil microorganisms. Tests were carried out by burying samples in compost soil for thirty days. Every ten days, samples are collected, cleaned with distilled water to remove soil, and soaked in alcohol for five minutes to eliminate pathogens. The results of biodegradation tests were characterized by the percentage of weight loss. Figure 5 displays the biodegradation test graph of water hyacinth samples before and after impregnation.

The results obtained were that in the initial water hyacinth stem samples, the percent weight loss value for 10, 20 and 30 days was 26.03%; 45.97%; 53.59%. Meanwhile, the sample after impregnation has a percent weight loss value of 10, 20, 30 days, namely 30.32%; 48.43%; 52.79%.

Based on the characterization that has been carried out to determine the optimum conditions for water hyacinth stems after impregnation, the results obtained for optimum conditions in the water content, water absorption capacity and MoE tests were obtained for optimum conditions at an impregnation time of 3 h. Where in the water content test it had a value of 6.45% which was originally 17.97%, the water absorption capacity had a value of 81.37% which was originally 94.85% and the MoE was 191.18 MPa which was originally 22.94 MPa.

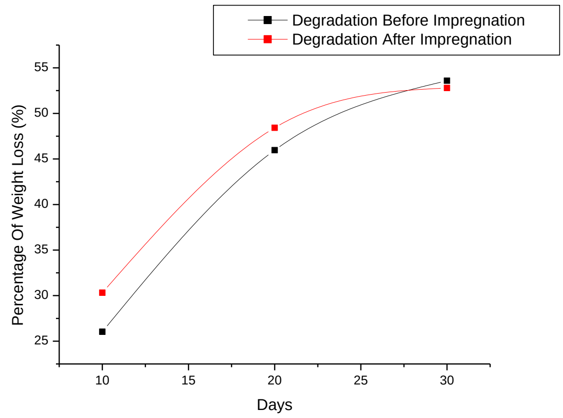

Figure 5. Degradation test graph before-after impregnation

3.6 Morphological analysis

Morphological analysis is carried out to determine the shape and surface changes of a material. In principle, if changes occur in a material, for example fractures, indentations and changes in the structure of the surface, then the material tends to experience changes in energy. This changing energy can be emitted, reflected, absorbed and transformed into other electromagnetic wave functions that can be captured and read in SEM images. SEM images of water hyacinth stems before and after impregnation are shown in Figure 6.

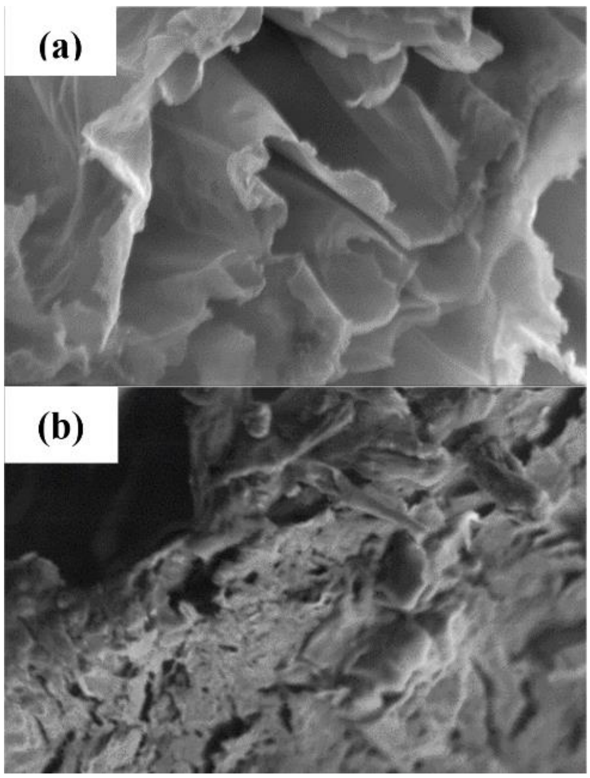

Figure 6. SEM images of water hyacinth stems (inside) before (a) and after (b) impregnation with 4000x magnification

Based on Figure 6a, the water hyacinth stem has fibers (fibrils) and vascular bundles (light parts) that surround parenchyma tissue (dark parts) and this parenchyma tissue has many and large porous cavities. Figure 6b shows that the cavities or pores of the water hyacinth stems have mostly been filled with impregnating resin. The pores of the water hyacinth stems have been covered and the impregnating resin is distributed evenly so that the surface is also flatter so that the pores of the water hyacinth stems are tighter.

3.7 Infrared spectroscopic analysis (FT-IR)

The application of infrared spectroscopy in this research is the qualitative aspect in the form of determining the structure by observing the typical frequencies of the functional group FTIR spectra obtained by comparing the spectra of pure water hyacinth stems with the spectra of a mixture of water hyacinth stems that have been impregnated (3 h) with modified polystyrene (Figures 7-10).

Based on the spectra in Figure 7, the modified polystyrene resin shows that there has been an interaction between polystyrene and acrylic acid. This is indicated by the presence of aromatic C-H groups at wavelengths between 3000-3100 cm^{-1}, namely 3060.93 cm^{-1}; 3025.84 cm^{-1} and the wave number in the region 2500-3000 cm^{-1} (absorption of carboxylic acids) is typical for -OH, namely 2965.03 cm^{-1}; 2928.71 cm^{-1}; 2871.65 cm^{-1} of acrylic acid which shows that grafting of acrylic acid has occurred on the used polystyrene with peroxide initiator. The C=C aromatic ring group at a wavelength of 1500 cm^{-1}, 1600 cm^{-1} is around 1605.46 cm^{-1}; 1493.98 cm^{-1} which is the absorption of the polystyrene group. In the FTIR results there are also spectra in the 768.42 cm^{-1} area; 742.34 cm^{-1}; 694.62 cm^{-1} where this spectrum shows that there is an aromatic ring C-H functional group at a wavelength of 675-870 cm^{-1}. These results are in line with previous studies [16, 17].

From Figure 8, water hyacinth stems before impregnation show the presence of -OH groups of alcohol hydrogen bonds in the area 3200-3600 cm^{-1}, namely in the absorption width of 3342.59 cm^{-1} which is cellulose from water hyacinth stems. The absorption in the area of 1607.39 cm^{-1} shows the presence of C=C cellulose, namely in the range of 1500, 1600 cm^{-1} and the typical C-O absorption in the range of 1080-1300 cm^{-1}, namely 1031.10 cm^{-1}, which is a ring form of cellulose [18, 19].

In Figure 9, the water hyacinth stem which has been impregnated for 3 h with modified polystyrene shows a change in the intensity of the -OH group of the water hyacinth stem from an absorption of 3342.59 cm^{-1} to 3322.26 cm^{-1}, this occurs because -H bonds between molecules continue to increase so that new bands appear and the intensity decreases. Likewise, the water hyacinth stems had interaction -H from polystyrene with the -OH groups of the water hyacinth stems during impregnation, resulting in a decrease in intensity. Then there was also a slight increase in intensity in the absorption, which was initially 1607.39 cm^{-1} to 1607.76 cm^{-1}, which was the absorption of the C-C group of cellulose. However, there was a decrease in the typical C-O absorption, namely from 1031.10 cm^{-1} to 1029.62 cm^{-1} which is the ring form of cellulose. There is a shift in the wave number from 742.34 cm^{-1}; 694.62 cm^{-1} (presence of aromatic C-H functional groups) to 758.05 cm^{-1}; 694.73 cm^{-1}. These data prove that the water hyacinth stems have been impregnated. A Comparison of FT-IR results of water hyacinth stems at the beginning and after impregnation (3 h) can be seen in Figure 10.

The empirical results reported herein should be considered in the light of some limitations. Other variables in the research design may have an influence on the quality of the material produced. Thus, it needs to be developed for subsequent studies. However, based on several analyzes of test results that have been carried out, this method shows quite good potential for improving the physical and mechanical properties of samples.

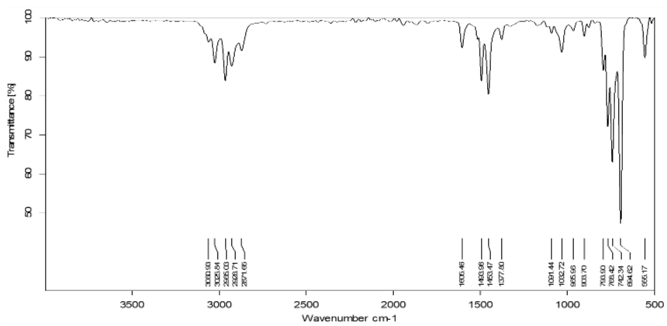

Figure 7. Spectra of modified polystyrene resin

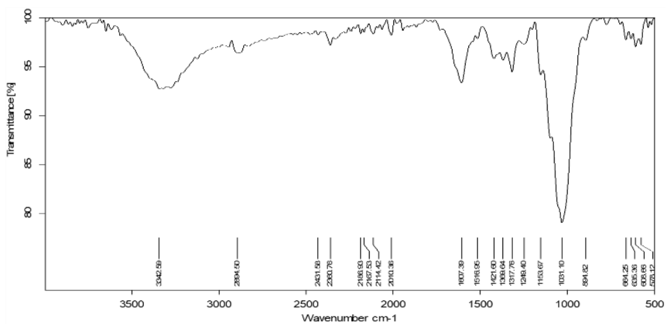

Figure 8. Spectra of water hyacinth stems

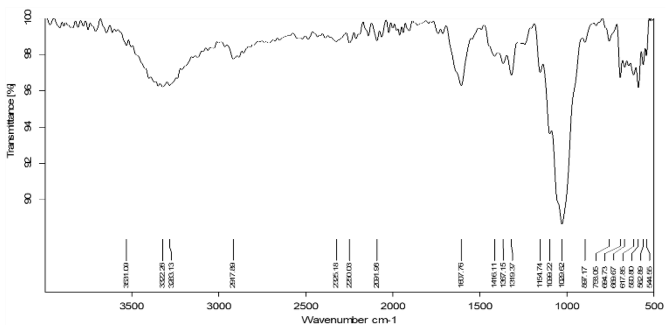

Figure 9. Spectra of water hyacinth stems impregnated (time 3 h) with modified polystyrene

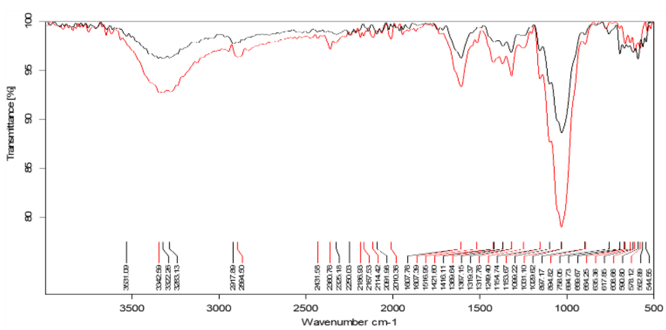

Figure 10. Comparative spectra of FT-IR results of water hyacinth stems and after impregnation (3 h)

4. CONCLUSIONS

Based on this research, it can be concluded that water hyacinth stems can be changed using acrylic acid-modified polystyrene resin and that the physical interaction of modified polystyrene with water hyacinth stem cellulose can improve the quality of water hyacinth stems. The optimum conditions for impregnation of resin into water hyacinth stems with a resin concentration of 10 $g.ml^{-1}$ are 3 h of impregnation time. Water hyacinth stems, after being impregnated at optimum conditions with an impregnation time of 3 h with modified polystyrene resin, increased in quality. The water content was 6.45% from 17.97%, the water absorption capacity was 81.37% from 94.85%, and the MoE increased to 191.18 MPa from 22.94 MPa. The expectation is that the outcomes of this study may offer a substitute approach for addressing the proliferation of water hyacinth in Lake Toba, in addition to repurposing discarded polystyrene materials. Furthermore, it has the potential to offer insights into enhancing the characteristics of water hyacinth stalks, which hold promise as a valuable resource moving forward.

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