Studying Mechanical, Thermal and Absorption, Characteristics of Water Hyacinth (Eichhornia crassipes) Plant Fibre Reinforced Polymer Composites

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Abstract

The natural fibre extracted from water hyacinth waste could be used for making natural fibre polymer composites. The main intent of this manuscript is to develop polymer composite materials reinforced by aquatic wastewater hyacinth natural fibre having varying lengths. The water hyacinth fibres were extracted using a mechanical drum extractor followed by a drying process at a speed of 320 rpm. Mechanical testing of the composites was conducted as per the relevant ASTM standard and subsequently, thermo gravimetric analysis was also conducted to assess the thermal characteristics of the composites. Fourier-transform infrared spectroscopy (FTIR) and X-Ray diffraction (XRD) techniques were employed to characterize the elemental and microstructural properties of the composites. A 20 mm fibre length with a 30% fibre content resulted the best mechanical properties. Fractured surfaces from the composite samples

are evaluated by using a scanning electron microscope (SEM). Brittle fracture, fibre pulled out, fibre clusters are identified as the general failure characteristics of the composites. This study demonstrated that the hyacinth fibre reinforced epoxy resin composite could be useful for developing particleboard products, as well as other lightweight products.

Keywords: Natural fibre, Water Hyacinth Fibre Composite, Mechanical Strength, Absorption Behaviour, TGA, SEM.

Introduction

Nowadays, synthetic fibres do cause hazardous impacts on the environment in a number of ways. Researchers are therefore utilizing natural fibres in developing polymer composite materials instead of the synthetic ones. A variety of synthetic fibres including carbon, glass, and aramid were employed previously for developing fibre reinforced composites (FRC). As the synthetic fibres poses negative effects on the ecosystem, their use has decreased and gradually being replaced by the natural fibres as an alternative. Compared to synthetic fibres, natural fibres possess a number of advantageous properties, including low density, high modulus, low cost, simplicity, availability, low cost, and environmental friendliness. As a result of their lightweight and strong characteristics, FRCs find a lot of application in the automotive, mechanical, aerospace, and construction industries (Abral et al. 2013c). Natural fibre-based composites have been suggested by many researchers as an effective substitute for synthetic counterparts for retaining their strength. With modern polymer matrix composites that use natural fibre as a primary phase, engineering materials become more affordable and accessible. (Abral et al. 2014; and Jirawattanasomkul et al. 2021).

Applications of water hyacinth (WH) fibre reinforced composites are increased in the last few years in many fields for instance construction industries, production of commercial items such as particle boards, packaging, and mainly lightweight materials in the automotive

applications (Flores Ramirez et al. 2015). Generally, the hyacinth plants show very fastest-growing characteristics. Water hyacinth plants reproduces the daughter plants from sexual and asexual manner. The seeds of hyacinths are spread by humans, birds, and other animals (Ajithram et al. 2021). Hydrogen potential, dissolved oxygen, dissolved solids, and salinity are just a few of the important factors in water content that are reduced by plants such as hyacinths. This plant provided small-scale paper manufacturers with raw materials for making paper. There are a number of countries around the world that use hyacinths as organic feed for animals. Hyacinth roots absorb heavy amounts of mercury and other pollutants. The plants remove nitrogen more effectively from water in the surrounding area (Gajalakshmi et al. 2002, Chukwuka et al. 2008; and Kumar et al. 2009). Many nations are cultivating water hyacinths in waterbodies to reduce the amount of nuisance (Istirokhatun et al. 2015).

By varying the length of the fibre, this research seeks to characterise the structural, mechanical, thermal stability, absorption, and fracture characteristics of the hyacinth fibers extracted from the parent plant in a novel mechanical way of fibre extraction. In the conclusion of this study, it is clearly explained that composites reinforced with hyacinth fibres have achieved higher mechanical strengths and have a good thermal stability.

Materials and Methods

WH Fibre extraction

The hyacinth fibre was extracted from the parent plant stem by utilizing a novel mechanical way of extraction. In this method, a 0.5 HP electric motor (bare motor), mono block bearings, two alternative shafts, one permanent shaft with fully threaded and 50 cm diameter of rounded plants, 55 cm length of flat rods were used to fabricate the mechanical way of extraction machine. This machine's speed was set to 350 rpm. One kilogram of hyacinth plant was used for extracting the fibres using five different techniques and data are presented in Table

1. Figure 1 shows images of fibres extracted by different methods. Figure 2 shows the mechanical extraction process and machine. By utilizing this mechanical way of extraction machine, higher fibre quantity was obtained, and the wastage was reduced to a maximum of 80%. Mechanically extracted hyacinth fibres have contain 16.8% cellulose, 43.52 hemi cellulose, 6.2% of lignin and 33.48% other contents. This chemical composition analysis is conducted by using AOAC25 method. The quality of the fibre extracted by the mechanical technique was also appeared much better than that obtained by the other techniques. However, in the mechanical way of extraction process, the original length of the hyacinth fibres are derived from the parent plant stem.

Fig. 1. Different methods of extracted fibres; (a) manual extraction, (b) hot water boiling (c) chemical extraction (d) retting Process (e) mechanical way of extraction method

Fig. 2. Mechanical Way of Fibre Extraction Machine

Table 1. Water hyacinth fibre different extraction methods final outcome

Composite Preparation

Once the fibres are properly dried in a hot air oven, they are chopped into different lengths such as 10 mm, 15 mm, 20 mm, and 25 mm. Each length of the hyacinth fibres was mixed with epoxy matrix material with different weight ratios (20 wt.%, 25 wt.%, 30 wt.%, and 35%). The epoxy matrix material and hardener (LY556 and HY951 grades) were purchased from Seenu and company suppliers, Coimbatore, Tamil Nadu, India. The mixing ratio of the epoxy and hardener was 10:1. Figure 3 clearly explains the fabrication process of the WH fibre reinforced polymer composite samples.

Fig. 3. Fabrication process of water hyacinth polymer composite; (a) water hyacinth extracted fibres, (b) dried hyacinth fibre, (c) compression moulding (d) hyacinth fibre composite sample

The manually mixed materials were poured into a hot compression moulding machine where the upper and lower plate temperatures were set to 120°C and 100°C respectively for a quick curing. Then, the pressure was set to 1.03×10^7 Pascal. The viscosities of the epoxy matrix material and hardener at 25°C are 10,000 mPa.S, and 12 mPa.S, 10 mPa. On the other hand, the densities of the matrix and hardener are 1.15gm/cc and 0.97g/cc.

Mechanical strength tests

The mechanical strength of the water hyacinth reinforced composite was determined by using a universal testing machine and Charpy impact test machine. Initially, the samples are cut to the dimensions of 200 mm × 25 mm ×3 mm for tensile testing according to ASTM D3039 standard, 125 mm × 13 mm × 3 mm for flexural testing according to ASTM D 790 standard, 65 mm × 13 mm × 3 mm for impact testing according to ASTM D256 standard. From each composite, three samples are tested and the average mechanical strength was reported. The crosshead speeds were maintained at 2 mm/min and 1.5 mm/min during the tensile and flexural tests respectively.

Absorption studies

For the usage of lightweight natural fibre reinforced composites in commercial applications, the investigations on water and chemical (NaOH) absorption studies are important. Initially, the WH composite samples were cut to a dimension of 50 mm × 20 mm × 3 mm for both the water and chemical absorption tests according to the ASTM D570 and ASTM C413-18 standards respectively. At the earlier stage, initial weight of the composite test sample was measured by using a digital weight balancing machine (0.002g linearity, 0.001g

readability, 10-40C operating temperature, ISO 9001:2015 standard) and the sample was placed into 100 ml of water and chemical (NaOH) solution.

$$\Delta W = \frac{Wt - Wd}{Wd}$$

ΔW= Weight percentage, W_t and W_d are the specimen weight before and after immersion.

Thermal analysis of composites

Thermogravimetric analysis (TGA) was conducted by a thermal analyser (ceramic crucible) to determine the weight loss of the water hyacinth composite samples between a temperature range of 100-800°C and a heating rate 10°C/min in the presence of nitrogen inert gas environment at a flow rate of 20 ml/min. TGA gives a continuous measurement of the sample weight with respect to the temperature.

Microstructural study

To investigate the crystallinity index and crystal structure of the composite samples the X-ray diffraction technique was used. This process was carried out in a BRUKER D8 advance machine at 25°C room temperature, scanning angles ranging from 10° to 80° and with step size of 0.02.

Fourier Transform Infrared Spectroscopy Study

The composite sample structures were analysed in both quantitative and qualitative methods with Fourier-transform infrared spectroscopy (FTIR). The SHIMADZU instrument with 16cm^{-1} resolution, 1 scan accumulation, 600 total scans, 50 msec interval time was used to evaluate the WH-epoxy composite sample within a frequency range of 4000-400 cm^{-1} by maintaining a resolution of 2 cm^{-1}.

Fractured surface observation procedure

The fractured surfaces of the composite samples from the mechanical testing were observed in a VEGA3 TESCAN scanning electron microscope with 15 kV accelerating voltage, 9.92 mm working distance (distance of the sample and lower end of the pole) and at different magnifications (×10 to ×1000). The composite samples were coated with a thin gold palladium alloy for SEM imaging.

Results and Discussions

Mechanical Properties

The mechanical strength (tensile, flexural, and impact) of the WH-epoxy composites with respect to different fibre lengths and loadings are presented in Figure 4(a-c). Table 2 explains the statistical data of mechanical strength of hyacinth fibre composite samples. The tensile strength of the composites varied from 18.16 MPa to 25.124 MPa with 10 mm WH fibre, 22.42 MPa to 36.16 MPa with 15 mm WH fibre, 34.782 MPa to 42.612 MPa with 20 mm WH fibre, and 32.986 MPa to 36.141 MPa with 25mm WH fibre. The flexural strength of the composites varied from 38.64 MPa to 46.614 MPa, 36.17 MPa to 44.81 MPa, 48.82 MPa to 58 MPa, 44.264 MPa to 48.87MPa for the fibre lengths of 10 mm, 15 mm, 20 mm, 25 mm in the composites.

According to the results obtained, the strength of the fibre composite increased significantly with increasing the fibre length up to 20 mm, but decreased when the fibre length was increased further (25 mm), possibly due to the agglomeration effect from the primary fibre reinforcements. The results of the mechanical strength test (tensile, flexural, and impact) can be interpreted as showing that the fibre length of 20 mm reinforced with 30 wt.% fibres achieved a higher mechanical strength content than the remaining fibre lengths (Promdee et al. 2012; and Muchanyereyi et al. 2016).

Fig. 4. Mechanical strength of WH composites fabricated with different fibre lengths and loadings (wt.%); a) Tensile strength, b) Flexural strength, c) Impact strength, (d) water absorption and (e) chemical absorption

Table 2. Water hyacinth fibre composite mechanical strength statistical data

Absorption Properties

Figure 3 (d and e) represents the water and chemical absorption behaviour of water hyacinth composite samples. The final results clearly demonstrated that the WH composites was not significantly affected by the water and chemical solution as the weight percentage of the samples increased only by approximately 5% before reaching the saturation level after the 8th hour. The hydrophobic nature of the WH fibre in the composites could be attributed to this low water or chemical intake. This WH based composites showed minor impact on both water and chemical solution when compared to other natural fibres such as coir composites (Sanjay et al. 2018; and Saputra et al. 2015). Previous research has found that coir-based composites need 28 hours to reach a saturation level and the saturation weight percentage for a composite based on coir is 8.60%. Similar to the sisal composite sample, it took 36 hours for the sample to reach the saturation point, and the final weight percentage was recorded as 7.20%. The hyacinth fibre-based composites on the other hand quickly reached saturation point (within 10 hours) and absorbed a significantly smaller quantity of water (5.40%).

Microstructural characteristics

The X-ray diffraction patterns of the WH composites are shown in Figure 5a. Normally, the lower intensity peaks contain the amorphous phase of the reinforcement (fibre) material. In general, the crystallinity index (CI) is measured in four ways like XRD peak height method, XRD deconvolution method, amorphous subtraction method, and NMRC4 peak separation method. The second method deconvolution crystallinity index measurement was used in this

work with the help of Equation 1. Table 3 clearly denotes the area of crystalline peaks, and area of all peaks (crystalline and amorphous) of WH fibre reinforced composites (Senthil Kumar et al. 2016; and Siengchin et al. 2018).

$$CI = \frac{Area\ of\ crystalline\ peaks}{Area\ of\ all\ peaks} \times 100 \quad (1)$$

The term CI means the crystallinity index, A_c means area of crystalline peaks, A_a means area of all peaks.

Table 3. Crystalline Peak, Lower Intensity Peak, Crystallinity Index of WH Polymer Composite

The crystallinity index of WH composites with the 10 mm, 15 mm, 20 mm, and 25 mm of fibre lengths and 30 wt.% fibre content were 48.41%, 51.12%, 60.68%, and 56.27% respectively (Silva et al. 2015). The results showed that the 20 mm fibre length composite had the highest crystallinity index due to its excellent fibre and epoxy matrix bonding, which increased the composite's mechanical properties. However, when fibre length exceeded 20 mm, CI decreased, indicating poor interfacial bonding and reduced mechanical strength.

Fig. 5. (a) XRD spectra of WH composite (30 wt.%) and (b) FITR Characterization of WH Composites

Fourier transforms analysis technique was used to investigate the chemical functional groups within the WH composites. Figure 5b explains the chemical functional groups of water hyacinth natural fibre reinforced polymer composite. Table 4 clearly explains the peak bands present in the composites. The 10 mm WH composite band peak 3481.857cm^{-1} identified the oxygen-hydrogen (O-H) stretching of the cellulose and hemicellulose contents. In general, the peaks occurred within the range of 3360-3420 cm^{-1}. For the remaining composites with 15 mm,

20 mm, and 25 mm long fibres, the peaks occurred at 3419.07 cm^{-1}, 3425.480cm^{-1}, 3421.480cm^{-1} respectively.

Table 4. Water Hyacinth Polymer Composite Peak Band Alignments

The peaks of the WH composites at 1517.62cm^{-1}, 1524.635cm^{-1}, 1562.327cm^{-1}, 1532.486cm^{-1} represented the C-C aromatic group and the lignin contents from the reinforcement area was indicated by the OCH3 groups. The carbon, hydrogen symmetrical determination and carbon-oxygen (C=O) double bond peaks were identified at 1236.51 cm^{-1}, 1237.962cm^{-1}, 1212.953cm^{-1}, 1235.147cm^{-1}. Carbon-oxygen single bond (C-O), carbon-carbon single bond (C-C), and (C-O)/(C-C) stage of peaks represented the hyacinth composites at 1030.562 cm^{-1},1236.55 cm^{-1}, 1045.72 cm^{-1}, 1053.926 cm^{-1}. In the WH fibre phase, the O-H bonding has resulted in a decrease in lignin and hemicellulose contents due to the proper drying and removal of moisture. A peak at 1652.354 cm^{-1} in the 20mm composite indicates that there is an increase in the cellulose content of the particular composite as a result of the proper removal of moisture. The other peaks of the graph also occur for different lengths of fibre composites, as well as the raw peaks of these composites on their own.

Thermal characteristics

The thermo gravimetric, first-order derivative, derivative thermo gravimetric curves of the WH composite are shown in Figure 6. Table 5 clearly explains the initial decomposition (T_i), final decomposition (T_f), and maximum decomposition temperatures (T_{max}). The 10 mm, 15 mm, 20mm, and 25mm WH composites started to decompose at 292°C, 342°C, 266°C, and 286°C respectively.

Table 5. TGA analysis of WH polymer composite

Before 100°C the decreasing trend in the curves of the TG graph (Figure 5) shown in the moisture content of the hyacinth fibre composite. The hyacinth composite samples major peaks occurred at 312°C, 364°C, 322°C, and 288°C for 10 mm, 15 mm, 20 mm and 25 mm fibre composites. Compared to the different hyacinth composite samples, the 15 mm length samples attained maximum decomposition temperature. Normally, high mechanical strength of the sample attained maximum peaks and withstanding higher temperature. However, in this work, the 15mm composite sample attained the maximum decomposition temperature peak over the 20 mm length hyacinth composite. This could be reasoned due to the intermolecular effect of the polymer matrix with reinforcement material at high temperatures (Monteiro et al. 2008).

Fig. 6. TGA of the WH composites a) TG, b) DTG, c)1st Order Derivative

Fractured surface characteristics

The fractured surfaces of the WH fibre reinforced composites are placed in a hot air oven (64°C) for 24 hr to remove any moisture. Through this process, the wax substance and surface impurities were highly reduced. Figure 7 shows the primary reinforcement and secondary phase epoxy adhered well leading to good mechanical properties (Nair et al. 1996). Figure 6(a) shows the presence of minute impurities in the fibre phase in the WH composite surface. It was likely that this impurity resulted from the mechanical extraction process. Some minute dust particles were also present on the upper surfaces of the fibres possibly attached during the fibre processing. The high amount of fibre content created the agglomeration effect within the matrix phase. Figure 7(b) clearly demonstrated the fibre clusters leading to the poor interfacial bonding with the epoxy matrix, which eventually degraded the mechanical properties of the composite (Norizan et al. 2017). Figure 7(c) shows that the failure of the composite sample under the impact load and fibres were pulled out from the matrix phase.

Fig. 7. Fractured surface of the WH composites a) tensile failure b) flexural failure c) impact failure (20 mm fibre length and 30 wt.%)

Conclusion

Aquatic water hyacinth (WH) fibre reinforced epoxy composites were developed with varying fibre content and fibre lengths and their microstructural, mechanical, absorption, thermal and fracture surface characteristics were evaluated. The following conclusions can be drawn based on the findings in this investigation.

- The hyacinth fibre was extracted from the parent plant stem using a new novel mechanical way with better quality and reduced wastage.

- The optimum fibre and epoxy matrix combination was determined as 30:70 and the optimum fibre length was found as 20 mm for obtaining the best mechanical properties (tensile, flexural and impact). Adding fibres above 30 wt.% and 20 mm led to a decrease in the mechanical properties.

- The WH reinforced epoxy composites achieved higher strength (42.64MPa tensile, 58MPa flexural) compared to the other natural fibre composites like coir and bamboo reported in the literature.

- Based on the absorption results, the water and chemical solutions did not affect the composites possibly due to a change from hydrophilic to hydrophobic nature when the fibre was mixed with the epoxy.

- In terms of thermal stability, the WH fibre-based composite with a length of 20 mm showed higher thermal stability than the composites with other lengths. As a result of

the characterization studies, the 20mm long hyacinth fibre composite with a 30% content has gained more essential functional groups than the other samples.

- The WH composites could be a light-weight alternative to the synthetic fibre based composites for commercial applications.

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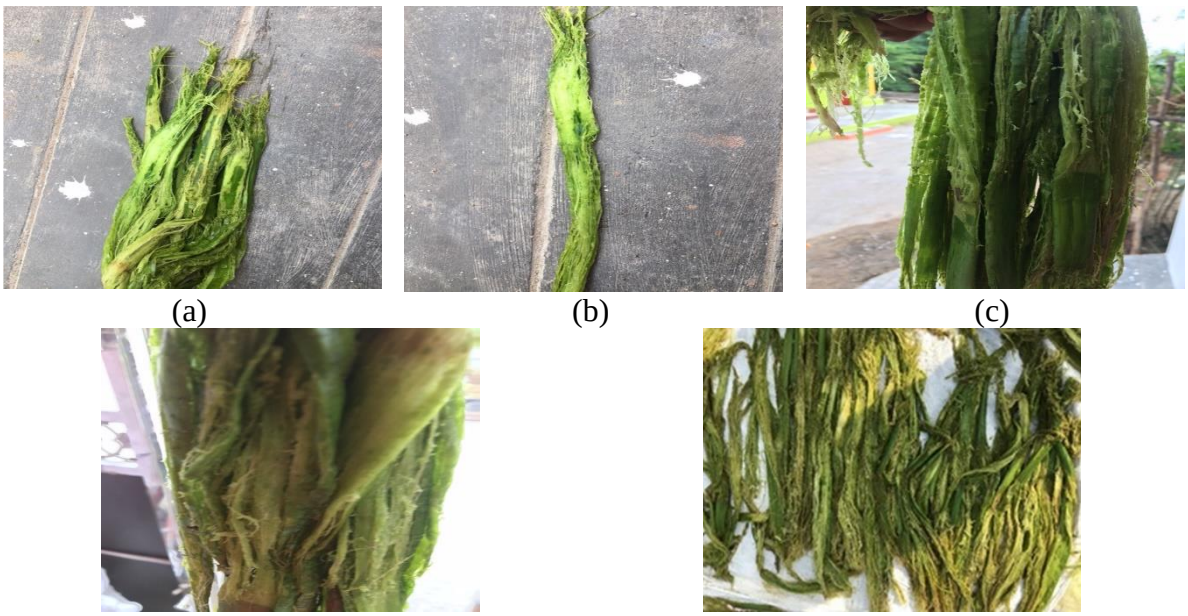
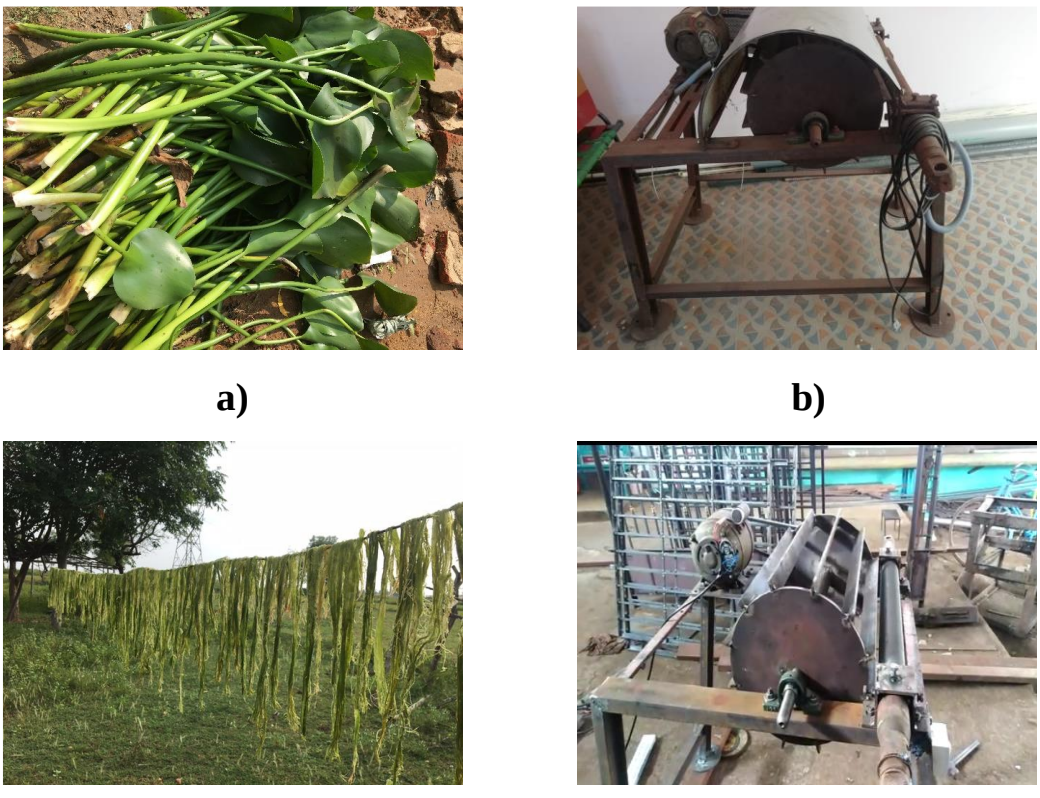


(a) (b) (c)

(d) (e)

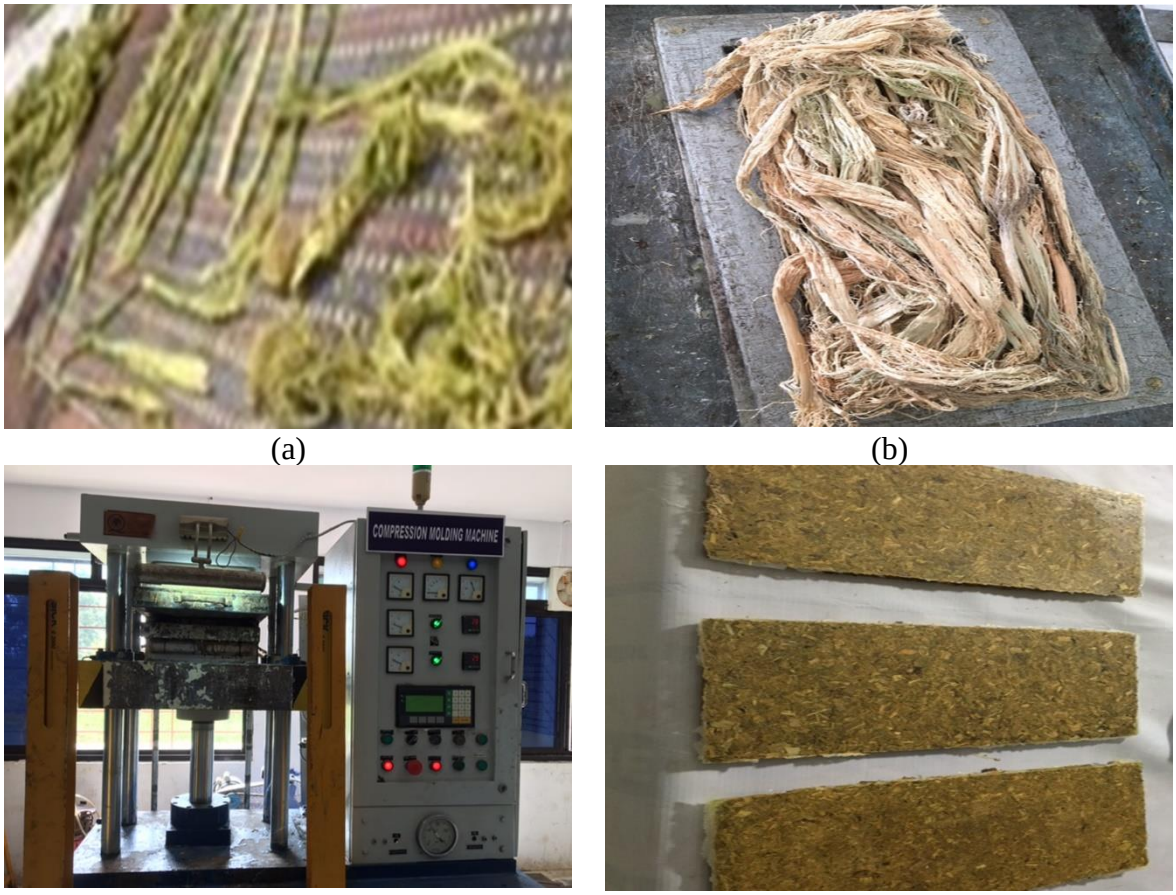
Fig. 1. Different methods of extracted fibres; (a) manual extraction, (b) hot water boiling (c) chemical extraction (d) retting Process (e) mechanical way of extraction method



a) **b)**

d) **c)**

Fig. 2. Mechanical Way of Fibre Extraction Machine



(a) (b)

(c) (d)

Fig. 3. Fabrication process of water hyacinth polymer composite; (a) water hyacinth extracted fibres, (b) dried hyacinth fibre, (c) compression moulding (d) hyacinth fibre composite sample

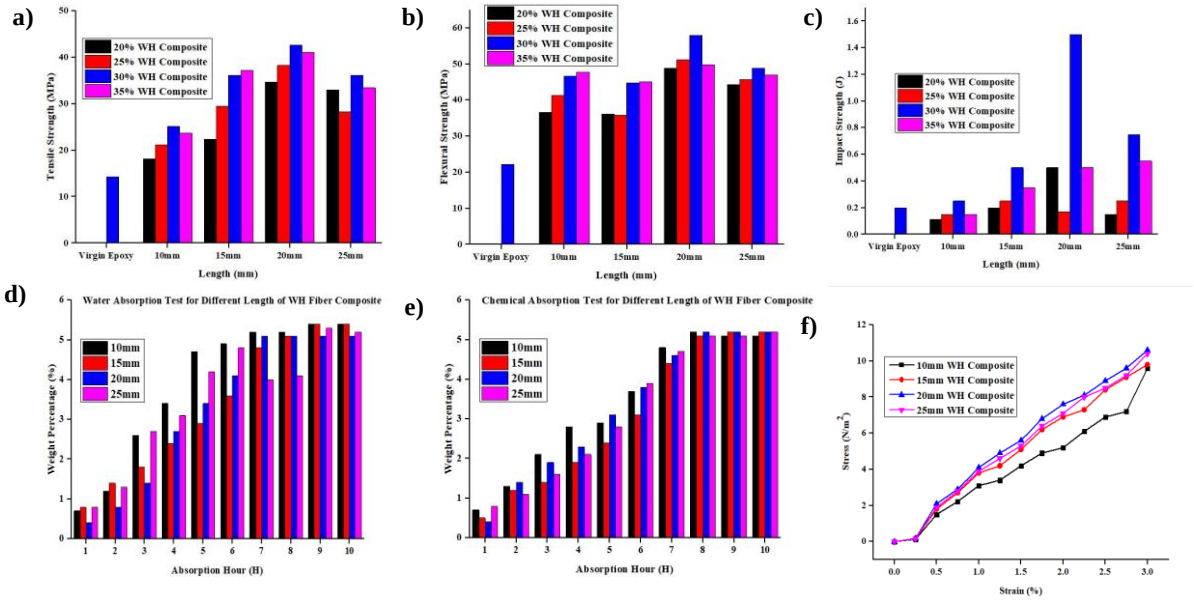
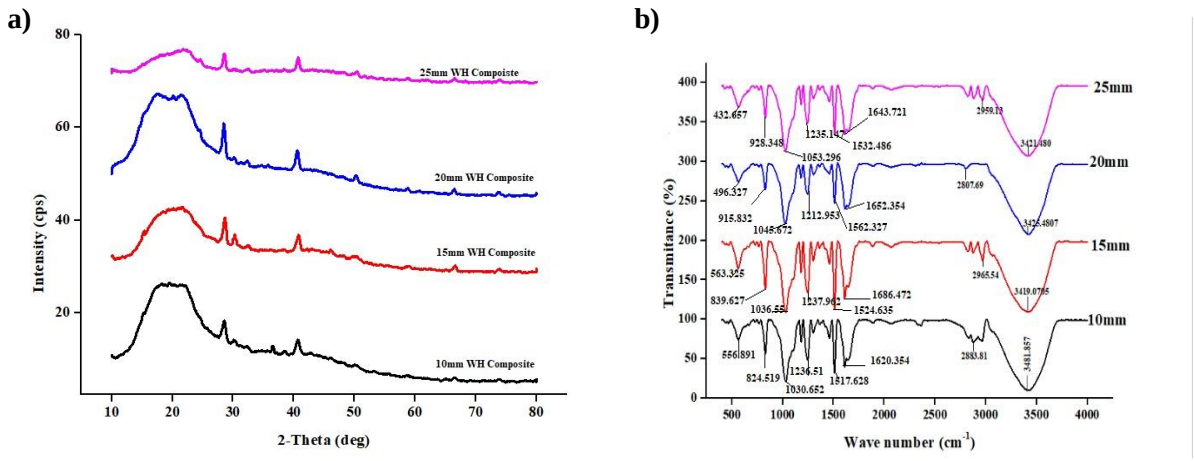


Fig. 4. Mechanical strength of WH composites fabricated with different fibre lengths and loadings (wt.%); a) Tensile strength, b) Flexural strength, c) Impact strength, (d) water absorption and (e) chemical absorption (f) stress-strain curve



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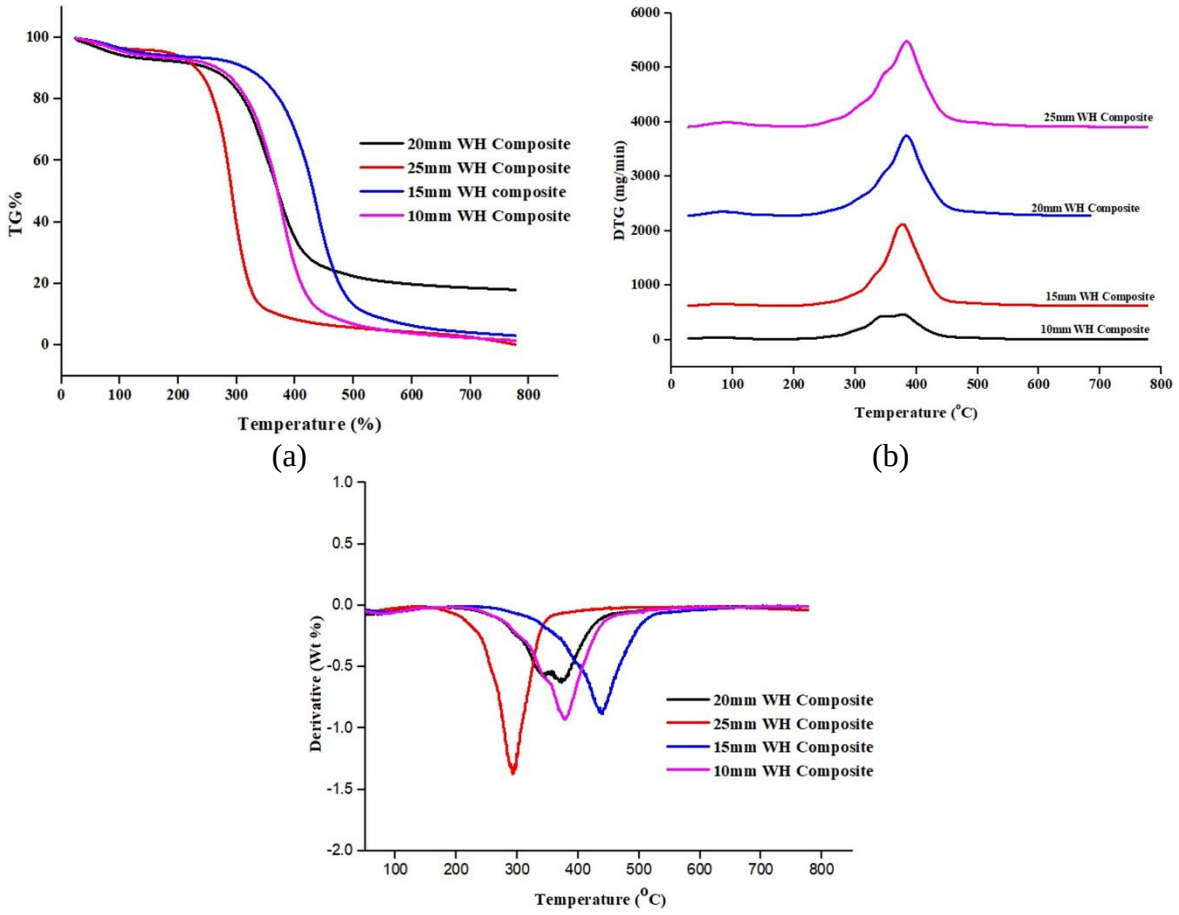

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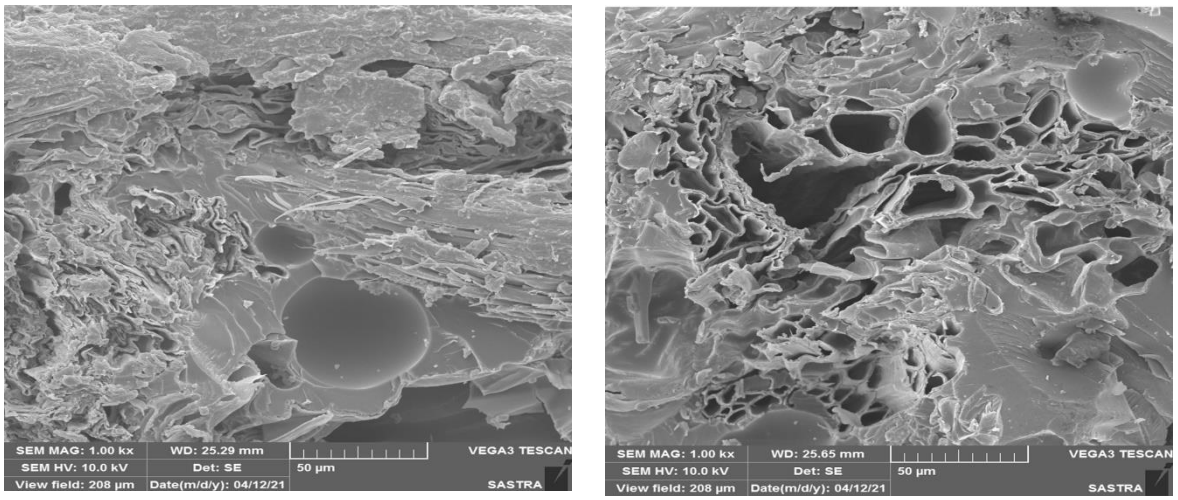

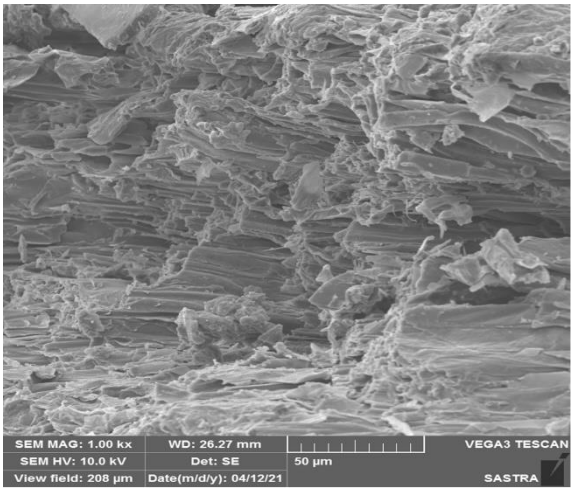

(**c**)

Fig. 7. Fractured surface of the WH composites a) tensile failure b) flexural failure c) impact failure (20 mm fibre length and 30 wt.%)

Table 1. Water hyacinth fibre different extraction methods final outcome

Serial No.	Extraction Method	Original plant quantity(gram)	Final fibre quantity (gram)
1	Manual way of extraction	1000	200
2	Hot water boiling method	1000	85
3	Chemical way of extraction	1000	30
4	Retting process (Most used method)	1000	450
5	Mechanical way of extraction method	1000	780

Table 2. Water hyacinth fibre composite mechanical strength statistical data

Water Hyacinth Fibre Composite Tensile Test Statistical Data				
Sample Length (mm) with 30% Reinforcement	Tensile Strength Mean (MPa)	Tensile Modulus (MPa)	Tensile Strength Standard Deviation	Tensile Strength Co-efficient of Variation
Virgin Epoxy	14.32	2542	2.12	14.22
10	25.124	4658	4.18	26.458
15	36.16	4482	5.14	27.214
20	42.612	4296	3.86	20.14
25	36.41	4284	6.24	28.24
Water Hyacinth Fibre Composite Flexural Test Statistical Data				
Sample Length (mm) with 30% Reinforcement	Flexural Strength Mean (MPa)	Flexural Modulus (MPa)	Flexural Strength Standard Deviation	Flexural Strength Co-efficient of Variation
Virgin Epoxy	22.14	2456.82	6.32	28
10	46.614	3386.42	15.13	46
15	44.81	2620.38	17.24	18.15
20	58.02	3145.21	8.42	24
25	48.87	2865.42	2014.86	42

Table 3. Crystalline Peak, Lower Intensity Peak, Crystallinity Index of WH Polymer Composite

Serial No.	**Area of Crystalline Peaks (A_c)**	**Area of All Peaks (A_a)**	**Crystallinity Index, CI (%)**
1	435.12	899.76	48.41
2	467.14	913.76	51.12

3	492.76	812.2	60.68
4	455.80	801.27	56.27

Table 4. Water Hyacinth Polymer Composite Peak Band Alignments

Serial number	**Wave number (cm^{-1})**	**Peak band**	**Reference**
1	3360-3430	O-H stretching	[1, 5]
2	2800-2920	C-H stretching (Cellulose, Hemi cellulose)	[3]
3	1730-1760	C=O stretching (hemi cellulose)	[8, 14]
4	1610-1690	C=O	[9, 11]
5	1580-1600	Lignin OCH3 group	[15]
6	1520-1540,1410-1450	C-C stretching, aromatic group	[16]
7	1210-1260	C=O (lignin, hemi cellulose)	[21]
8	1020-1090	C-O / C-C (stretching vibration)	[22]
9	520-640	C-OH (cellulose)	[21, 23]

Table 5. TGA analysis of WH polymer composite

WH Composite Sample	**Initial decomposition temperature (℃) (T_i)**	**Final decomposition temperature (℃) (T_f)**	**Maximum decomposition temperature (℃) (T_{max})**
10 mm	292	442	312
15 mm	342	512	364
20 mm	266	418	322
25 mm	286	342	288