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Experimental investigation of tensile, flexural and hardness properties of polyester resin echinatus fiber reinforced composite material

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Nowadays, composites made of plant-based fibers may be used to polymer resin synthetic fiber reinforced composites since they are less expensive, renewable, abundant, less abrasive, and lightweight. Echinatus fiber obtained from the stem of the echinatus plant in an abundant amount. The main objective of this study was to develop echinatus polyester-resin fiber reinforced composite material and investigate its flexural strength, hardness and tensile strength. Echinatus plant was collected and echinatus fiber was extracted by the decortication process from the echinatus plants manually, and treated with 5% NaOH for the improvement of bond and interfacial shear strength. And then, the test specimens were manufactured using a mass fraction with 0^o, 45^o and 90^o orientations using technique of hand lay-up. The tensile strength, hardness, and flexural strength were investigated using samples that were prepared according to the ASTM standards. 70% echinatus fiber to 30% polyester composite material with 0^o orientation was found as the material with maximum tensile strength of 60.60 MPa, flexural strength of 96.8 MPa, and hardness values of 44.54 HRA. Considering the mechanical properties' experimental results, echinatus fiber-reinforced composite with 70% fiber at 0^o orientation can be good substitutes for synthetic materials.

1. Introduction

Nowadays, the world is challenged with waste materials that are not safe environmentally. Many studies have been conducted to produce materials that have good properties and they are safe to overcome environmental problems. Among these materials that have fibers called natural fibers are getting attention for different mechanical applications compared to synthetic fibers (Palani Kumar et al., 2021; V. Mastan, 2014). The sources of natural fibers are mostly plants and animals. For example, fiber can be obtained from the hair of animals and fibers can also be obtained from leaves or stems. Plants that have fibers on their stems like flax, bamboo, stinging nettle and others. By using single natural fiber or hybrid fibers, like natural and natural or natural and synthetic are very interesting areas to produce composite materials that are reinforced by polyester or epoxy for different application and they have been become good research areas (Rao, Patil, Ponkshe, Sahembekar, & Mali, 2018; Sanjay, Arpitha, Naik, Gopalakrishna, & Yogesha, 2016; Subbiah Jeeva. G, 2015). Natural fiber composite materials have low cost, no or less effect on health, good relative mechanical properties, like tensile strength, bending strength and impact strength, good biodegradable properties, less energy consumption, and low weight compared to synthetic composite materials. As results, natural fiber composite materials are now adapted for different applications, like construction, automotive industries, packaging industries, aerospace and others (Bongarde, Shinde, & Technology, 2014; Ku, Wang, Pattarachaiyakoop, & Trada, 2011), and these materials have two or more chemically distinct compositions, on a macro-scale, having a different interface separating them on a macro-scale level (Jauhari, Mishra, & Thakur, 2015). The fiber materials give strength as result called as reinforcement and the ground substance material is known as a matrix. In composite material,

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the stress is transferred between reinforced fibers and protected from mechanical failure by matrix (Kaushik, Jaivir, & Mittal, 2017). Composite materials are categorized as particle reinforced, fiber-reinforced and structural based on the constituents as shown on Fig. 1.

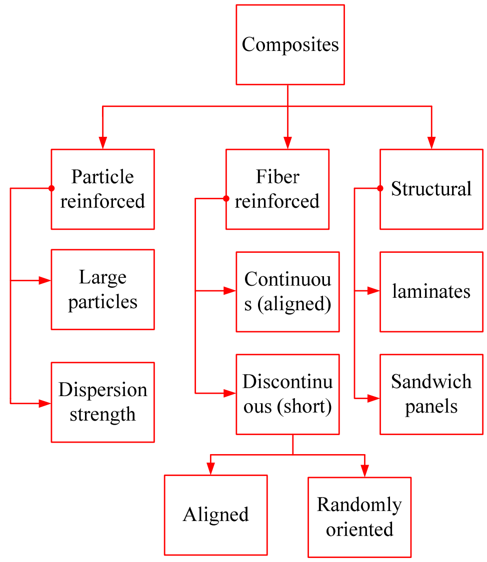


Fig. 1. Types of composite materials (Otani, Pereira, Melo, & Amico, 2014)

Over the past decades, research on the replacement of synthetic fibers with cellulose fiber and getting better materials by reinforcing cellulose fibers into polymer matrices have been carried out (Azman et al., 2021; Sood & Dwivedi, 2018). Fiber Orientations also have an impact on mechanical properties of composite material. Tensile test and bending test were performed and discussed for 0°, 30°, 45°, 60°, and 90° angles of fibers orientation, the test was performed based on the ASTM standard and the result attains at 0° fiber orientation was maximum (Abd-Ali & Madeh, 2016). Fiber can be treated using chemicals, the fiber treatment can be made by using sodium hydroxide (NaOH), acetylation treatment, H_2O_2 treatment, isocyanate treatment, silane treatment, surface impregnation with polyethylene dilute solution and benzoylation treatment (Rokbi, Osmani, Imad, & Benseddiq, 2011). Based on study for bast fibers (Antiohos, Tsimas, & research, 2004), it is better using NaOH because of its less cost, and does not affect the mechanical property. The influence of quicklime that was produced industrially was investigated on the strength enhancement and reaction rate of pozzolanic of different fly ash/cement (FC) systems. Experiment was carried out by using two high calcium fly ash diversified on their active silica and calcium oxide content and one with moderate calcium content. During this experiment, hydration evolution, strength enhancement and pozzolanic reaction rates of the quicklime fly ash cement were monitored and presented, so that the result shows that the activation increases the strength for both cases (Das et al., 2021; Pujari, Ramakrishna, Padal, & Technology, 2015). The 1% of matrix materials, 0.02% of hardener and 0.005% of accelerator in a weight ratio was prepared using polyester resin LY 556 and the angles were 0°, 15°, 30°, and 45°. Each layer of the fiber was pre-impregnated with matrix materials by placing one over the other in the mold, taken care of to maintain practically achievable tolerances on fiber alignment as suggested (Velmurugan et al., 2014). The species of echinatus plant is categorized under corchorus plant (Datta, 2009) and it has fiber on stem. The mechanical properties of echinatus fiber-reinforced polymer composite were not investigated. So, the objective of this research was to develop the echinatus fiber composite material that was reinforced by polyester resin and fabricated using continuous hand lay-up technique. Besides, the developed composite material was investigated on its mechanical properties, like tensile strength, flexural strength and hardness by varying the fiber concentration and orientation using the polyester as resin.

2. Materials and Methods

2.1 Materials

In addition to echinatus fiber, the materials such as polyester resin, hardener, fly ash, wax, distilled water and NaOH were used to produce composite materials. Polyester Resin that was used for this study was unsaturated polyester and it was a general-purpose polyester resin. Polyester resin was cured by adding a hardener and fly ash. Wax was used as a purpose of mold releaser due to its collapsibility after use. NaOH solution was used for fiber treatment.

2.2 Methods

Experimental sample was selected and 27 specimens for tensile strength test, 27 specimens for three point flexural test and 9 specimens for hardness test based on the parameters namely, angle orientation and weight concentration were prepared. The samples were prepared to analyze mechanical properties such as tensile strength, flexural strength and hardness of echinatus fiber reinforced polyester resin composite material. The experimental work consisted of the following 4 phases,

- Fiber extraction from the echinatus plant,
- Mold design and manufacture, as well as other setups,
- Composite material and specimen preparation, and
- Composite material mechanical testing

The extraction of the echinatus fiber was done by the way known as decortications. Echinatus plants which were mature 3-4 months old were collected. And, they had been immersed after pilling their cellulose from the stem of the plant in water for two weeks so that the unwanted materials, resinous materials, from the surface of the fiber were removed and fiber strands were obtained as shown in Fig. 2. The extracted fiber was then treated with 5% NaOH for 24 hours, and then it was washed with distilled water and dried in air with the help of sunlight for 48 hours as shown in Fig. 3.

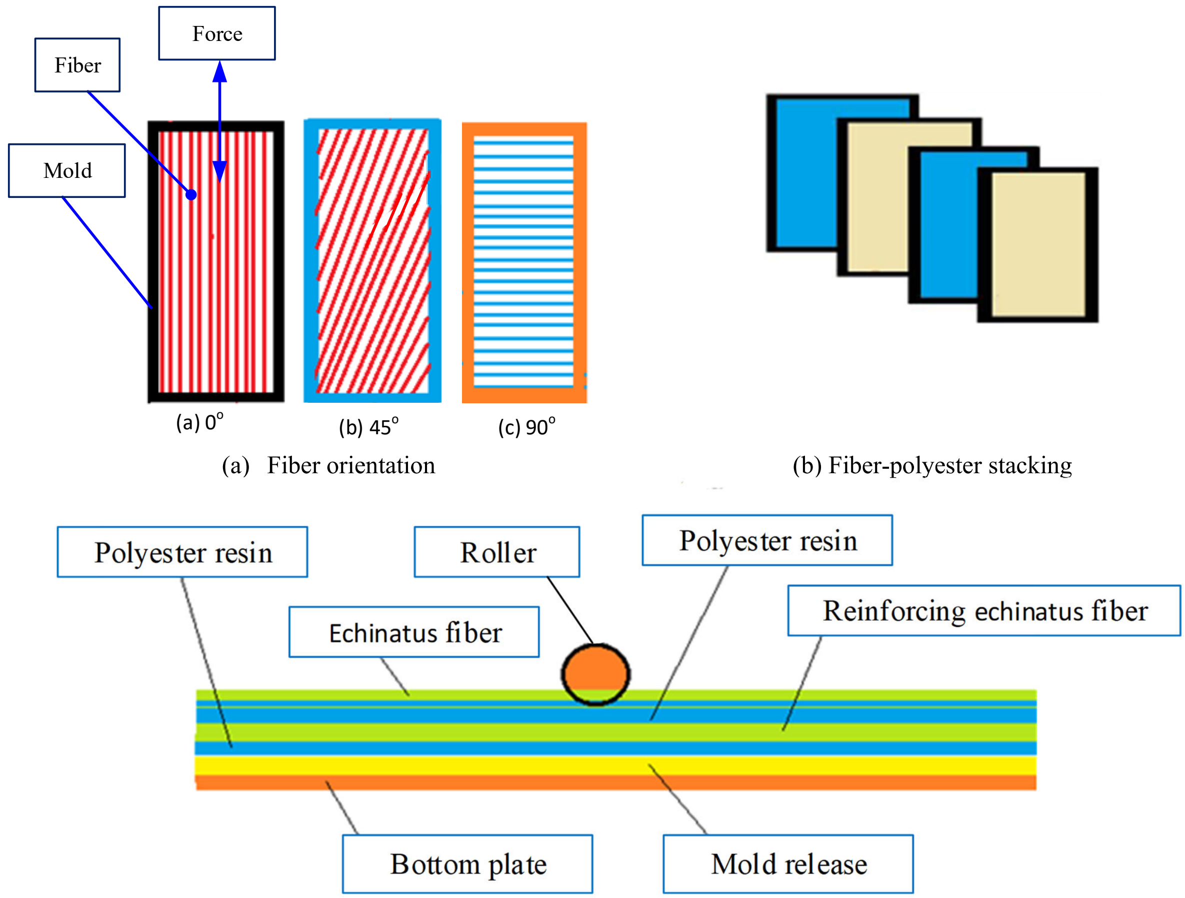
(a) Young echinatus plant (b) Decorticated fiber (c) Fiber in distilled water (d) Extracted fiber

Fig. 2. Echinatus plant and its fiber extraction

(a) Echinatus fiber treatment (b) Fiber washing after treatment (C) Fiber while drying

Fig. 3. Echinatus fiber treatment and drying process

Digital balance for measuring the weight ratio of fiber and polyester and fiber concentration, syringe for subtracting hardener to mix bucket for treatment solution preparation, brush laying of fiber, scissors for cutting the fiber to the required fiber length, table used as workbench, manual pressing mold die used for compressing the specimen matrix during mixing were tools and devices used in the fiber preparation. Polyester having density of 1.255 g/cm^3 at 25°C was used as the matrix to fabricate the specimen (Mohammed & Journal, 2016), and it was also mixed with hardener having density of 0.97 to 0.99 g/cm^3 in the weight ratio as per the recommendation of the supplier (Gavade, 2016). A general purpose polyester resin was mixed with hardener MEKP W 1900 HARDENER in the weight ratio of 3% to make the composite material. The carbonaceous material in fly ash is formed of angular particles, and fly ash boosts the strength and reduces the concentration of water in the fiber. Fly ash is frequently used as an accelerator in conjunction with hardeners. As a result, fly ash was also added to the mixture. The specimens were prepared based on varying fiber concentration and orientation. 10%, 30% and 70% fiber were used in 0^o , 45^o and 90^o fiber orientations relative to the applied force direction. It is thought that the angle of orientation of fiber in the fabrication may show an effect on the mechanical strengths, basically tensile strength, flexural strength and hardness. Thus, specimens were also prepared based on different angular orientations of fiber using a lay-up method as shown on Fig. 4, and investigation of mechanical properties of polyester resin echinatus fiber reinforced composite is shown in schematic flow diagram on Fig. 5.



(c) Composite fabrication

Fig. 4. Echinatus fiber reinforced polyester composite molding schematic view

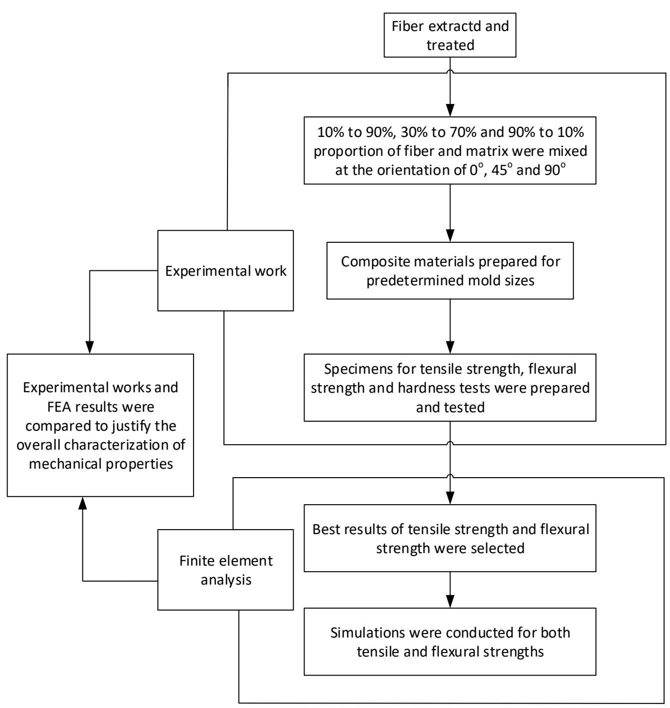


Fig. 5. Schematic flow diagram for the investigation of mechanical properties

3. Experimental work

While carrying out the investigation, the effect of mass fraction (mass concentration), orientation of fiber and sample size determination of the specimens was considered. Standards of ASTM D-3039, ASTM D790. ATSM D790 and ASTMD-785 were used for tensile strength, flexural strength and hardness tests, respectively (Somashekhara, 2018). When preparing the mold, the volume (V) was determined using the Eq. (1).

$$V = lengh \times width \times thickness \tag{1}$$

The mass of composite was calculated based on analysis of fractional volume of composite using Eq. (2).

$$C_v = F_v + M_v \tag{2}$$

where, C_V= composite volume, F_V = volume of echinatus fiber, and M_V = matrix volume. Mass fraction can also be given using Eq. (3).

$$C_m = F_m + M_m \tag{3}$$

where, C_m= mass of composite, F_m = mass of echinatus fiber, and M_m = mass of matrix. Density of the composite can be given and rewritten as the following

$$C_p = \frac{C_m}{C_v} \tag{4}$$

$$\frac{1}{C_p} = \frac{1}{F_p}\left(\frac{F_m}{C_m}\right) + \frac{1}{M_p}\left(\frac{M_m}{C_m}\right) \tag{5}$$

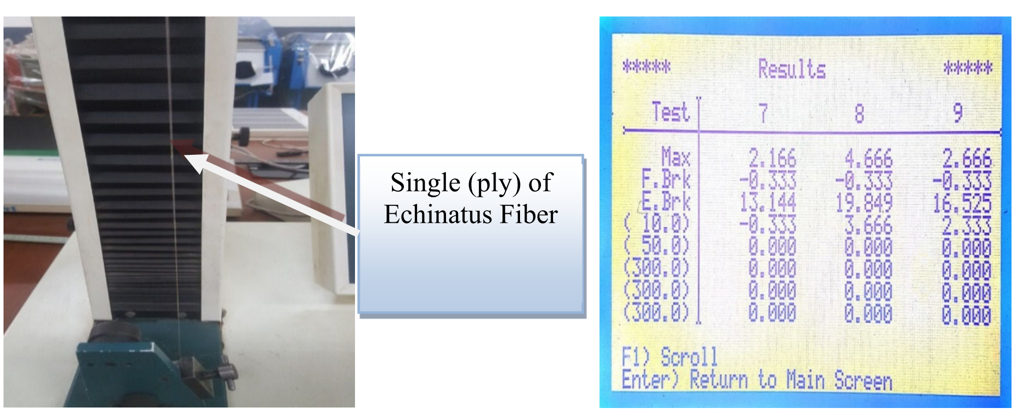
In this research, the density of echinus fiber was determined from the known volume of container and the given mass of fiber, and it was obtained 1.29 g/cm^3, and density of polyester matrix was taken as 1.255 g/cm^3. The volume of each mold for the preparations of tensile, flexural and hardness tests was 250 mm x 100 mm x 4 mm. The specimens' constituents in mass that were prepared for tensile, hardness and flexural tests were obtained using Equation 1, Equation 2, Equation 3, Eq. (4) and Eq. (5) are shown on Table 1.

Table 1. Tensile, flexural and hardness tests composition specimens by mass preparation

SC	M (%)	EF (%)	TM (g)	F (g)	TM (g)	P (g)	H (g)
S1	90	10	125.85	12.99	112.86	109.35	3.51
S2	70	30	127.56	38.91	87.65	84.93	2.72
S3	30	70	127.96	90.51	37.45	36.29	1.16

Note: SC = sample code, M = matrix EF = Echinatus fiber, C = composite, F = Fiber, TM = total matrix, P = Polyester, and H = Hardener.

The material characteristics of the individual constituents must be known before doing a finite element analysis. Experimentation was required to estimate the modulus of elasticity for echinatus fiber single strand. As results, 16 separate tensile tests were done to determine the modulus of elasticity for the echinatus fiber strand as shown Fig. 6. The fiber strand from fabric was cut into 40 cm pieces and fastened to the cardboard backing to perform the analysis. The cardboard backing was utilized to give support for the fiber strand at the clamps, and to assist avoid failure due to fiber pull out near the clamps. And, the experimental results are summarized in Table 2 and Table 3 after certain calculations (Mallick, 2007). The young's modulus and shear modulus were higher in the arrangement of 0° orientation and 70% fiber concentration.



(a) During testing (b) Test results

Fig. 6. Determination of tensile strength of a single strand of echinaus fiber

Table 2. Elastic properties of 0º ply laminate

Property	Young's modulus			Poisons ratio		
	E_{xx}(GPa)	E_{yy} (GPa)	E_{zz}(GPa)	v_{xy}	v_{xz}	v_{yz}
Values	15.4	4.51	4.51	0.20	0.11	0.233

Table 3. Elastic properties of 0º ply laminate (continued)

Property	Shear modulus			density
	$G_{xy}(GPa)$	$G_{xz}(GPa)$	$G_{yz}(GPa)$	$\rho(g/cm^3)$
Values	1.36	1.36	0.54	1.293

The treated echinatus fiber reinforced polyester resin for tensile, flexural and hardness tests were prepared. After fabricating the laminated composite materials, the specimens were cut into the desired dimensions using a hacksaw by slightly tight in order to reduce the residual stress development and they were based on their respective ASTM standards. Finally, 27 specimens for tensile test, 27 specimens for flexural test and 9 specimens for hardness test were used in this study as shown on Fig. 7, Fig. 8, and Fig. 9, respectively.

Fig. 7. Tensile test specimens

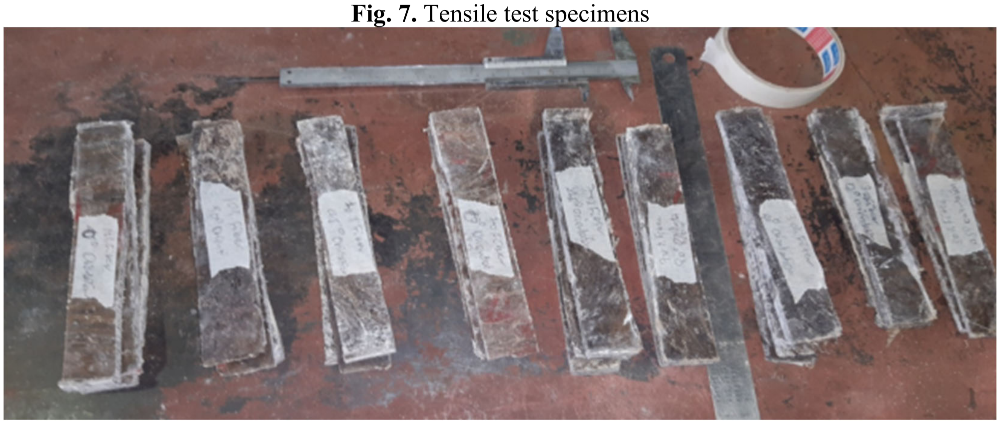


Fig. 8. Flexural test specimens

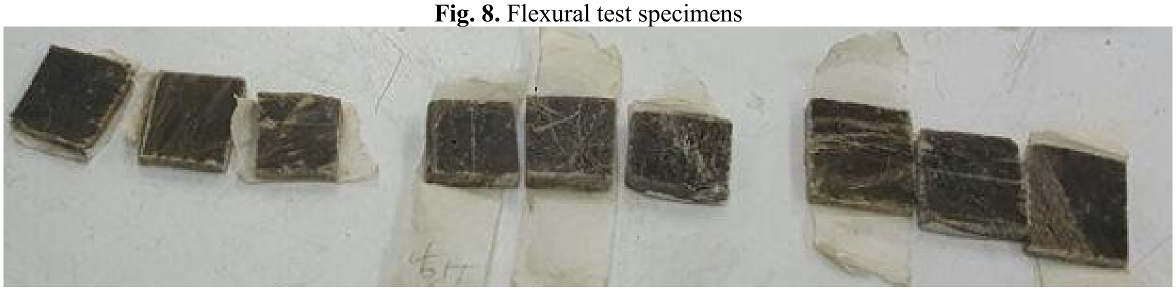
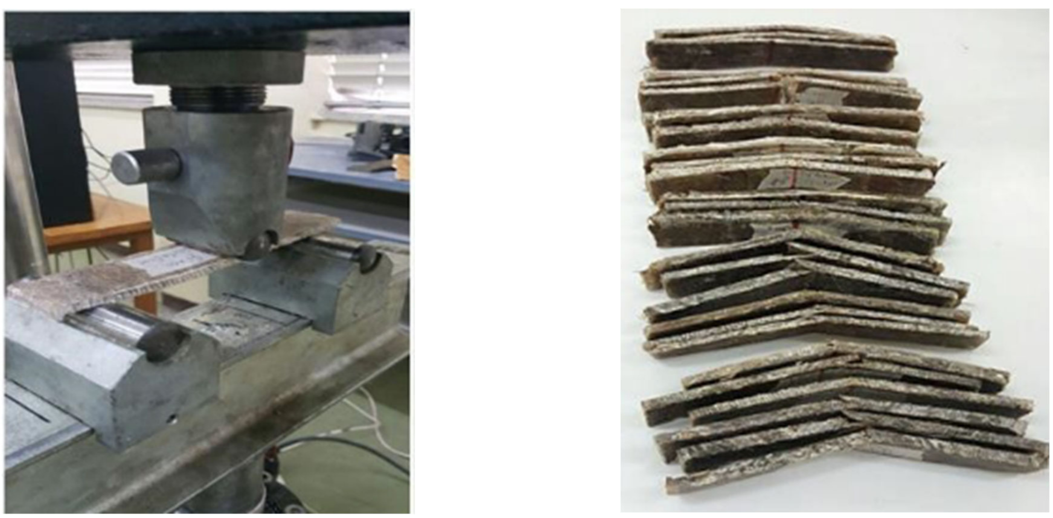


Fig. 9. Hardness test specimens

After the specimens were prepared, the composites were characterized for tensile strength as shown on Fig. 10, flexural strength as shown on Fig. 11, and hardness as shown on Fig. 12. And, for each variable combination (weight fraction and orientation), three replicate specimens were tested and the results were presented as an average of tested specimens. For each composite specimen, the thickness as per ASTM standard along the length of each specimen and the tests were conducted at room temperature and pressure.

(a) Tensile specimen under loading (b) Some specimens after loading

Fig. 10. Tensile strength testing using universal testing machine



(a) Flexural specimen under loading (b) Some specimens after loading

Fig. 11. Flexural strength testing using universal testing machine

Fig. 12: Specimen under hardness test

4. Results and Discussion

4.1 Tensile strength

The adhesion of echinatus fiber and polyester determines the tensile characteristics of composites, and a universal testing machine (UTM) was used for this tensile testing. Through both ends, a uniaxial load was applied. ASTM D3039 standards were used to create the tensile samples. 210 mm long, 4 mm thick and 25 mm width were the dimensions of the samples. With a strain rate of 2 mm/min, the samples were examined in UTM. The peak tensile property of the samples was obtained. The tensile behavior of the echinatus fiber reinforced polyester composite material (EFRPCM) is presented below using the average of its tensile strength tests' experimental results with varying weight fraction and reinforcement orientations.

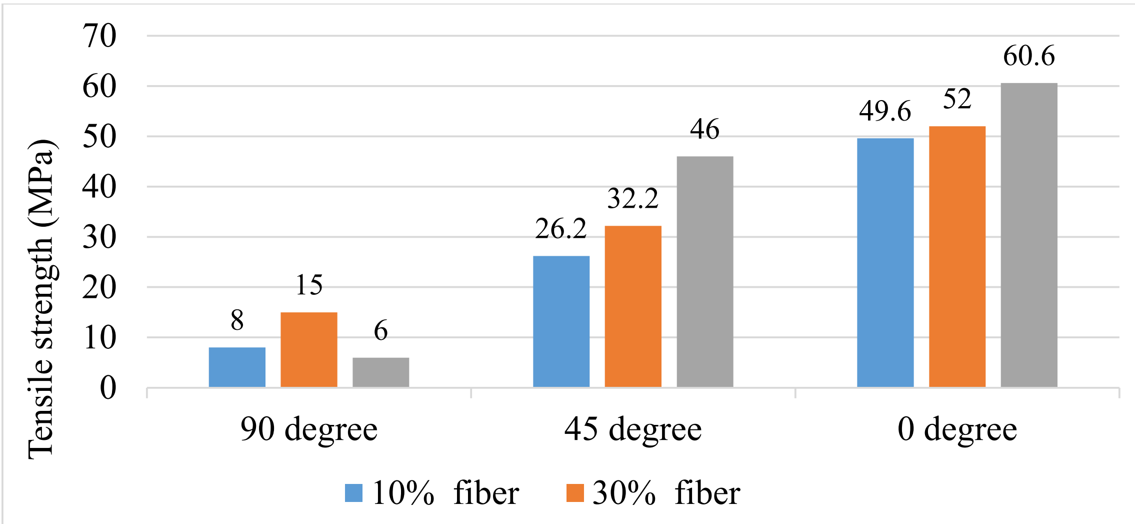


Fig. 13. Tensile strength at different fiber orientation and weight concentration

The results from Fig. 13 revealed that fiber orientation and matrix/fiber composition affected the tensile strength of polyester resin echinatus fiber reinforced composite.

1. Despite the fact that tensile strength rises as fiber content rises in the fiber orientation of 90°, 45° and 0° because it is the direction of the applied load so that the change of tensile strength almost reduces relatively. It indicates that the content of fiber in EFRPCM has gone to saturation state, or adding more fiber at least in EFRPCM more than 70% leads to a zero percent change in tensile strength between two consecutive measures. The maximum tensile strength appears at the maximum fiber concentration and 0° fiber orientations because the concentration of fiber is maximum and 0° fiber orientation means that the fiber is arranged to the direction of load applied, and Fig. 13 depicts the variance in tensile strength composites with variable fiber orientation and loading. The tensile strength of composites with fiber loading of 70% is clearly increased, as shown in Fig. 13. Tensile strength was determined to be highest when the fibers are oriented at 0° and loaded at 70%. This might be because of optimal resin-fiber mixing and good resin-fiber adhesion.
2. Based on the standard set for failure type, the tensile strength faces the LGM failure types which means that failure appears laterally through gauge length and middle points. Therefore, failure is acceptable.

4.2 Flexural strength

The force applied at the center point of the specimen was delivered at a speed of 0.5 mm/min in the 3-points flexural test with the 90 mm of span length to determine the composites' flexural strength using Eq. (6). The purpose of the test was to use the data obtained from the test to select elements that resist bending load without inflection.

$$\sigma_f = \frac{3LP}{2bd^2} \tag{6}$$

where; σ_f is maximum flexural strength in MPa, P is force at a given point in N, L is support span of the specimen in mm, b is width of the specimen tested in mm, and d is thickness of the specimen in mm.

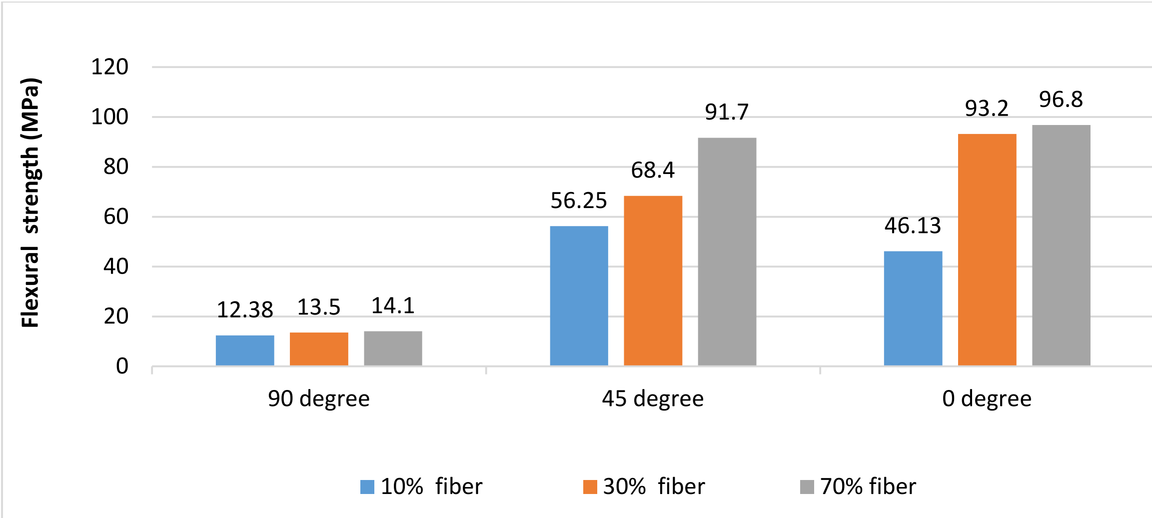


Fig. 14. Flexural Strength at different fiber orientation and weight concentration

Fig. 14 shows the comparison of flexural strength for the three fiber concentrations in weight percentage (10%/90%, 30%/70% and 70%/30% fiber and matrix concentration of echinatus fiber) in three orientations (0^o, 45^o and 90^o) and the findings are:

1. There is a clear phenomenon that the 10%/90% fiber-matrix concentration of echinatus fiber polyester composite is the lowest its flexural strength.

2. The flexural strength of echinatus fiber increased in corresponding with increasing fiber concentration with fiber orientation of 90^o, 45^o and 0^o, respectively. And as a tensile strength test, the maximum flexural strength was also shown when fiber - matrix concentration was 70%/30% and 0^o orientation was related to excellent fiber-to-polyester interfacial bonding.

3. As per the overall observation the composite's flexural strength, incorporation of fiber content in the polymer composite influences the composites' properties. The increasing fiber content in the composite increased the strength of the composite.

4.3 Hardness

Hardness tester quantifies the extent of indention in the material caused by a given force on a standardized presser foot. The final value of the hardness confides on the extent of penetration of the indenter after it has been imposed for 15 seconds on the material. The hardness properties of EFRPCM with different orientation and loading are shown in Fig. 15.

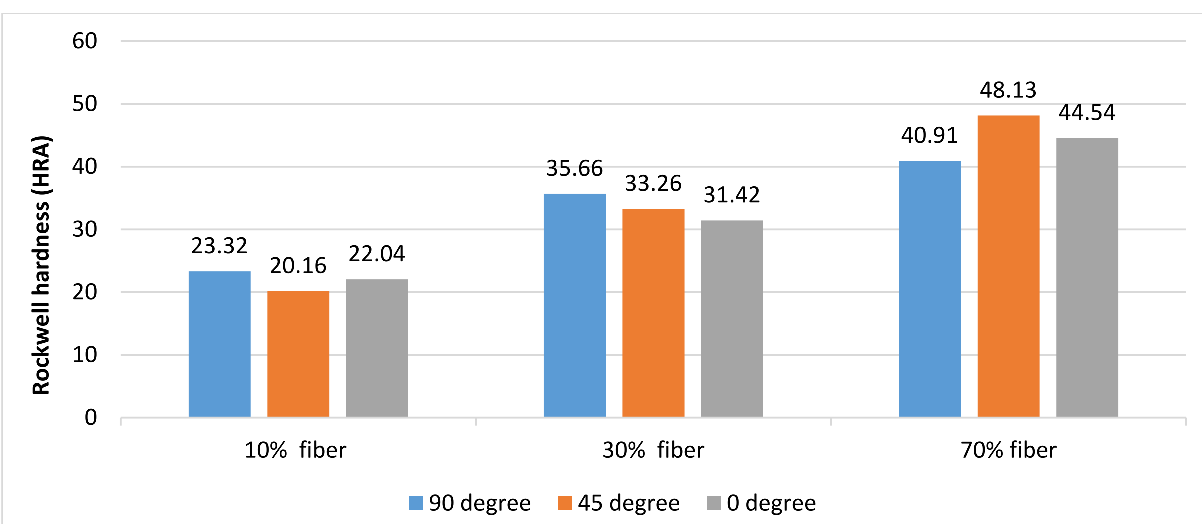


Fig. 15. Hardness value (HRA) of fiber orientation and weight concentration

One of the most important parameters governing the composites' erosion resistance is the hardness of the EFRPCM. Fig. 15 depicts the influence of orientation and weight concentration of fiber and loading of composite hardness. It shows that regarding fiber concentration, composites having 70% fiber loading have the highest hardness properties of the others. In terms of the impact of echinatus fiber composites with 0°, 45° and 90° fiber orientation didn't show an influencing effect on the hardness value of the composite. The part under the indentation test is representative of the whole microstructure.

4.4 Finite element model (FEM)

Tensile testing is a type of test in which a sample is forced to tension until it fails. The equivalent stress or von Mises stresses' values as shown in Fig. 16 was obtained following the modeling of solid-composite specimens and analyzed using ANSYS software. The tensile stress result for 70% fiber 0° tensile specimen of polyester resin echinatus fiber reinforced composite of both experimental and FEM results were compared. From experiments conducted, the maximum tensile test result was found from 70% fiber concentration and 0° orientation were 60.60 MPa and the result from FEM is 62.27 MPa, the error that appears is 2.7% to the experimental tensile strength result, and it is in the recommend tolerable zone which is less than 10%. Besides, both experimental results and simulation results showed that the result from the experimental test for mechanical properties of a single ply echinatus fiber is in the recommended error zone.

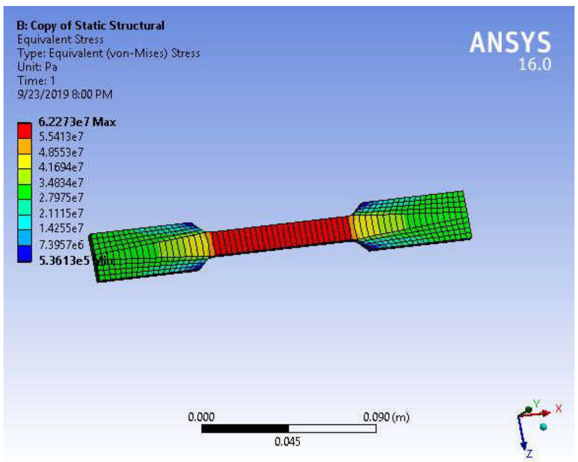


Fig. 16. Tensile stress simulation result

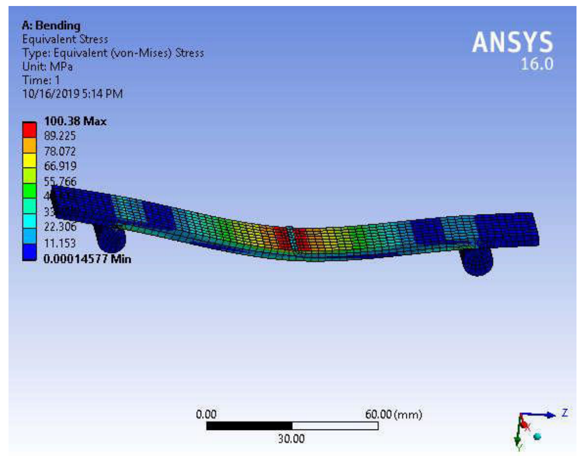


Fig.17. Flexural stress simulation result

ASTM D-790 standard was used to model the flexural test samples and the models were imported to ANSYS program. The FEM analysis for flexural test was also carried out on the samples 70% fiber 0° flexural specimen as shown in Fig. 17. Similar to the tensile test, the flexural strength result for 70% fiber 0° flexural specimens of polyester resin echinatus fiber reinforced composite of both experimental and FEM results were compared. From experiments conducted, the maximum flexural test result was obtained from 70% fiber concentration with 0° orientation was 96.8 MPa and the result from simulation is 100.38 MPa, the error that appears is 3.57% and it is in the recommended tolerable zone which is less than 10%. Besides, both experimental results and simulation results showed that the experimental result for a single ply fiber is in the required error.

5. Conclusion

The echinatus fiber was extracted manually from the Echinatus plant using the decortication process, and then the alkaline (NaOH) treatment was carried out. Next to that, EFRPCM was prepared for the appropriate fiber matrix concentration and orientation with the required dimension and its mechanical performance such as the tensile, flexural and hardness properties was determined in experiment as per ASTM standards. The EFRPCM was made with three distinct fiber orientations of 0°, 45° and 90°, as well as three different concentrations of fiber by weight of echinatus fibers and polyester resin in the ratio of 10%/90 %, 30%/70% and 70%/30%. The following findings have been drawn from the tensile, flexural, and hardness strength experiments:

- Based on the experimental results, composite samples having fiber orientation of 0° were performing the most of all the composite samples having 90° and 45°, and according to the fiber content echinatus fiber-reinforced polyester resin composite samples were yielding best results at 70% fiber concentration tested for all tensile, flexural and hardness.
- The maximum strengths had been observed by the 0° orientation of fiber and 70% fiber concentration, and echinatus reinforced polyester composite can hold the tensile strength of 60.6 MPa, flexural strength of 96.8 MPa, and hardness values of 44.54 HRA.

- The experimental results for all developed composites revealed that the tensile, flexural, and hardness properties are strongly dependent on the fiber orientation and concentration.
- This study shows that the durability of natural fiber composites may be improved by a sufficient ratio of natural fibers in order to achieve a good cost-performance balance while lowering the material's environmental effect.

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