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Investigation of Contribution of Control Factors on Mechanical Properties of Randomly Oriented *Helicteres isora* Short Fiber Reinforced Epoxy Composites

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Abstract – *Novel plant fiber composites are acquiring much popularity in recent days due to increased global demand for sustainable materials. The present investigation describes the fabrication and mechanical characterization of randomly oriented Helicteres isora reinforced epoxy composites. Hand layup procedure is used to fabricate short Helicteres isora reinforced epoxy composites at room temperature varying three control parameters i.e. the length of the fiber, the weight fraction of the fiber and the Alkali (NaOH) treatment percentage. Mechanical characterization of the composites were carried out by varying the length of the fiber, fiber weight fraction and NaOH percentage for the fiber treatment. 5 mm, 10 mm and 15 mm fiber lengths, 5%, 10% and 15% of fiber weight fractions and 0% (untreated fiber), 3% and 6% NaOH concentrations were used for fiber treatment. Taguchi's approach was used to optimize the experiments and parameters for increasing the tensile and flexural properties. The influence of various factors on the tensile and flexural properties of the composite materials were analyzed. The statistical study revealed that the length of the fiber significantly influenced the tensile modulus of the composite. Fiber weight fraction was found to have a considerable effect on tensile strength. NaOH treatment of fiber improved the tensile strength and flexural properties of the composites. The result has shown that the optimum composition among chosen factors for the composite's overall enhanced mechanical characteristics is 15% fiber weight fraction, 15 mm fiber length and 6% NaOH solution treatment.*

Keywords: *Helicteres isora*, Epoxy resin, Alkali treatment, Taguchi method, ANOVA.

1. INTRODUCTION

In recent times, engineering materials places have been taken by natural fiber-reinforced polymeric composite materials in certain applications due to their advantageous properties [1]. Hence the researchers are concentrating more on natural fiber reinforced polymer composites to improve their usability and desired properties. An emerging field in composite technology is natural fiber reinforced composite. The development of eco-friendly materials has been driven by stringent environmental guidelines and growing interest in the sensible use of sustainable resources. Natural fibers show good mechanical properties, have low density in comparison to synthetic fibers, are abundantly available, and can be disposed of without any hassles. When compared the specific strength of synthetic fibers, natural fibers make an affordable alternative [2].

Researchers have investigated a wide range of natural lignocellulose fibers using different polymeric matrix materials which are being used in many applications like automotive interiors, food packaging and storage. Many naturally available fibers in the country's rural places were used by village people to make household items and other useful articles to store and handle goods [3]. The same fibers can be improved and utilized for engineering and similar uses by converting them into advanced materials [4]. Thus it helps in protecting the environment from non-degradable synthetic fibers. *Helicteres isora* is a plant which produces a significant volume of bast fibers and has a cellulose percentage of 75% [5]. *Helicteres isora* bast fibers can be employed in polymeric composites as reinforcement.

Epoxy resin being inexpensive, has superior properties and is extensively used as a matrix material for natural and synthetic fiber-reinforced composites. *Helicteres isora* is a bast fiber extracted from the plant's bark by retting in water [6]. Isora plants grow in many locations of South India, majorly in Kerala and Karnataka. The fiber extracted from the plant has good mechanical strength, better durability and an appealing appearance [7]. *Helicteres isora* fiber has rich cellulose content and comparatively low lignin content, which enhances its performance as a reinforcement [8]. The effect of chemical surface modification of natural fiber is significant on the properties of polymeric composites [9].

In the current investigation, the raw *Helicteres isora* fiber was treated with aqueous Sodium Hydroxide (NaOH) solutions to remove the impurities and modify the surface elements in the fiber, which enhances the bonding ability of the fiber with the matrix. A parametric study of the *Helicteres isora*-reinforced epoxy composite is carried out to analyze the influence of various control factors on the mechanical properties of the composites. Three control factors, namely fiber weight fraction, fiber length, and NaOH percentage for treatment, were considered for the study. Three levels of fiber weight percentages (5%, 10% and 15%), fiber length (5 mm, 10 mm and 15 mm), and NaOH concentrations (0%, 3% and 6%) were considered for the study [5, 10, 11]. Taguchi's approach was used to optimize the experiments and parameters for increasing the tensile and flexural properties. The influence of various factors on the tensile and flexural properties of the composite material was analyzed. The optimum composition of fiber weight %, fiber length and NaOH treatment were also analyzed by a statistical approach.

2. MATERIALS AND METHODS

2.1 Fiber preparation

Helicteres isora fibers were obtained from the bark of the plant. The bark was retted in water for 15 days. The fibers manually extracted from the retted bark were washed with distilled-water and dried to remove moisture present in the washed fiber. Figure 1 illustrates the process of getting fibers from bast. Table 1 shows the comparison of compositions and the properties of *Helicteres isora* and other commonly used natural fibers.

Table 1: Comparison of compositions and properties of commonly used plant fibers [5, 12–14]

Fiber	Cellulose %	lignin %	Tensile strength (MPa)	Density (g/cm^3)
Isora	75	23	270-500	1.25-1.39
Flax	71	5	343-1035	1.38
Coir	43	45	130-170	1.15
Sisal	67	12	400-600	1.45
Oil palm	65	19	200-250	1.45
Jute	63	12	430-530	1.45
Banana	65	5	500-700	1.35

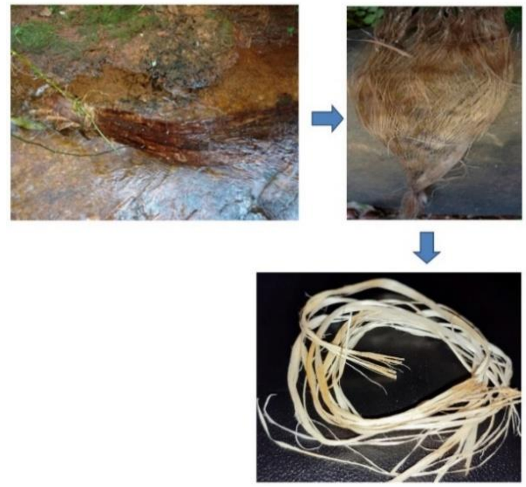

Figure 1: Process of preparation of Helicteres isora fiber

2.2 Matrix Material

The epoxy polymeric resin used in this investigation is Araldite CY 230-1 and hardener HY 951 in the ratio 10:1 [15]. The mechanical and physical characteristics of the resin are shown in Table 2.

Table 2: Properties of Araldite CY 230-1

Properties (unit)	Values
Density (g/cm^3)	1.19
Glass transition temp. (°C)	67
Tensile Strength (MPa)	52
Tensile Modulus (GPa)	2.8
Elongation at break (%)	11

2.3 Chemical treatment of *Helicteres isora* fiber

The required amount of *Helicteres isora* fiber was taken and immersed in the 3% and 6% w/v aqueous NaOH solution for 120 minutes [10]. Treated fibers were then washed and dried to eliminate moisture trapped in the fiber. Untreated, 3% and 6% NaOH-treated fibers were then cut into 5 mm, 10 mm and 15 mm lengths and stored in an air-tight container to avoid moisture uptake. Fiber loadings considered for the study were 5%, 10% and 15% by weight.

2.4 Taguchi method

The mechanical characteristics of composites were subject to a variety of control parameters that have varying degrees of influence. The Taguchi approach is an economic design technique used to select the most suitable and effective control factor combinations with a minimum number of investigations [16]. Typically, a full factorial experimental design is necessary to ascertain the optimal control factor combinations to obtain optimum mechanical properties, which requires significant time and cost. Fiber length, fiber weight fraction and NaOH percentage for treatment of the fiber were chosen as the three significant control factors in the current study.

A study was conducted on the influence of selected control factors, each at three levels. Table 3 displays the chosen control parameters and their respective levels.

Fiber length, fiber weight and alkali treatment are the major parameter influencing the properties of the composite [12–14]. To study the effect of these three parameters Taguchi method-based design of experiment was used. In this study Orthogonal array L9 (3^3) experimental design was selected to determine the plan of the experiment. Table 4 shows the orthogonal array selected for the study. The control parameters fiber length, fiber weight ratio and NaOH treatment were set as levels as decided in the previous section and shown in Table 3. Taking tensile and flexural strengths and moduli as response variables, based on these variables effect of these parameters was studied. To calculate S/N ratio "Larger the better" criterion was selected in all cases because higher tensile strength and flexural strength indicates better performance. Equation 1 shows the theoretical formula to calculate the S/N ratios based on "Higher the better" criterion [20]. The Taguchi approach was used to determine the plan of experiments, which can effectively establish the percentage contribution of individual factors on the responses.

$$\mathrm{S/N} = 10 * \log\left[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{{Y_i}^2}\right] \text{ .... (1)}$$

Where,

n - number of experiments in the orthogonal array

Y_i - i^{th} value measured.

Table 3: Control parameters and levels

Control Factors	Level I	Level II	Level III	Unit
Fiber fraction	5	10	15	% (w/w)
Fiber length	5	10	15	mm
NaOH concentration	0	3	6	% (w/v)

2.5 Composite preparation

The composites were fabricated using hand layup process. Mould for the hand layup process was made from wooden material. Figure 2 shows the wooden mould setup and composites prepared. Mould was prepared with a releasing agent to ensure that the part would not adhere to the mould. Initially, the mould was cleaned and dried before the Epoxy could be laid upon the mould. Composites of different compositions were made according to Taguchi Orthogonal Array. Both treated and untreated fibers were mixed thoroughly in Epoxy resin and hardener mixture as per the array. Then the mixture of resin and fiber was transferred into the mould carefully and flattened appropriately by using the roller. The mould was kept at ambient temperature for curing. After 24 hours, the composites were separated from the mould cured and cut as per the respective ASTM standards. This process was repeated and composites were prepared according to the Taguchi orthogonal array.

Table 4: Taguchi Orthogonal array [20]

Sl. No.	Weight %	Length (mm)	NaOH %
1	3	5	0
2	3	10	3
3	3	15	6
4	6	5	3
5	6	10	6
6	6	15	0
7	9	5	6
8	9	10	0
9	9	15	3

Figure 2: Wooden mould and sample prepared

2.6 Test Specimen

Tensile test samples were prepared as per ASTM 638 standard [21]. The tensile test was conducted using Zwick Roell universal testing machine at a test speed of 1 mm/min, and the same speed was maintained throughout the experiment. Figure 3 (a) shows the tensile test sample as per the standard.

According to the ASTM D 790 standard, three-point flexural tests were conducted [22]. The speed of the test was set to 1 mm/min and maintained throughout for flexural tests. Figure 3 (b) shows the flexural test specimen as per the standard.

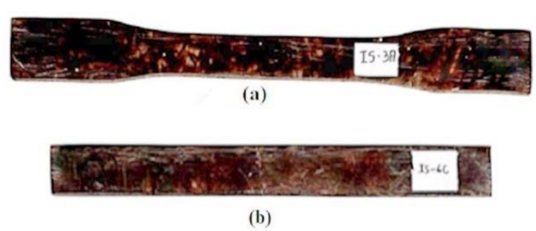

Figure 3: (a) Tensile test specimen, (b) Flexural test specimen of *Helicteres isora* reinforced epoxy composite

3. RESULTS AND DISCUSSIONS

3.1 Tensile Properties

Tensile test results obtained from the tests are listed in Table 5. To find the significance of the selected control factors on the tensile strength, nine different compositions were made as per Taguchi orthogonal array L9 [20]. Analysis of the investigational data is processed using MINITAB software which is used for the design of experiment applications. Deviation of quality attributes from the target value is measured by S/N ratio. If S/N value is high, it means that the signal is much higher than the noise factor. To get the better composition, the composite having higher S/N value was selected.

Table 5: Experimental data for tensile test

Fiber weight %	Fiber Length mm	NaOH %	Tensile Modulus (MPa)	Tensile Strength (MPa)	S/N ratio for T. Modulus	S/N ratio for T. Strength
5	5	0	510	15.7	51.87	23.92
5	10	3	384	14.8	50.29	23.41
5	15	6	337	23.8	54.15	27.53
10	5	3	602	18.8	51.69	25.48
10	10	6	556	17.3	51.25	24.76
10	15	0	365	20	54.57	26.02
15	5	6	535	29.3	54.90	29.34
15	10	0	392	20.6	52.34	26.28
15	15	3	414	20.1	55.59	26.06

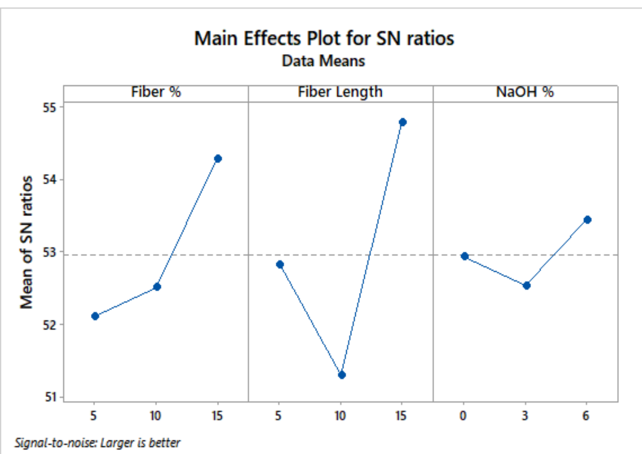

Figure 4: Main effect plot for S/N ratio for tensile modulus

Figure 4 illustrates the S/N ratios graphs, the effect three parameters on the tensile modulus of the composite can be observed here. Each graph's maximum numerical value of the point displays the optimum value for that particular parameter. Graph shows that 15% fiber weight fraction, 15 mm fiber length and 6% NaOH treatment has maximum value. This has more effect on the tensile modulus of the composite. Composites with 10 mm fiber length have shown lower properties than 5 mm and 15 mm fiber length composites. The decrease in the modulus is because of discontinued fibers and lower fiber matrix bonding [11].

From Table 6, the response data of S/N ratios of tensile modulus shows that the contribution of fiber length is more. This shows that the fiber length has more influence on the tensile modulus of the composite and is highly significant, which means that, higher the fiber length higher the tensile modulus.

Table 6: Response table for S/N ratios of tensile modulus

Level	Fiber %	Fiber Length	NaOH %
1	52.1	52.82	52.92
2	52.5	51.29	52.52
3	54.28	54.77	53.43
Delta	2.18	3.48	0.91
Rank	2	1	3

From Figure 5, it is observed that level 3 of all the factors has more influence on tensile strength. As observed in the response tables for S/N ratios of tensile strength listed in Table 7, fiber weight % has a significant influence on tensile strength. As the fiber loading % increased, the tensile strength also increased. NaOH % has a little more significant effect on the tensile strength than the fiber length. An increase in the tensile strength after NaOH treatment is because NaOH treatment has increased the surface bonding ability of the fiber by modifying the surface characteristics of the fiber. The graph shows that 15% weight, 15 mm fiber length and 6% NaOH treatment have the maximum value. This has more significance on the tensile strength of the composite.

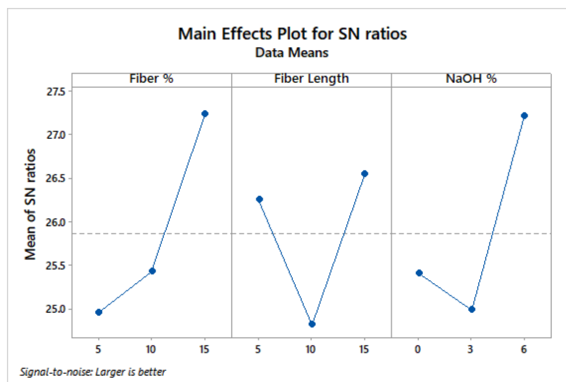

Figure 5: Main effect plot for S/N ratio for tensile strength

From the statistical study of response data listed in Table 6 and Table 7, it is observed that the 15% fiber weight fraction, 15 mm fiber length and 6% NaOH % for treatment give the optimum tensile properties for the composite.

Table 7: Response table for S/N ratios of tensile strength

Level	Fiber %	Fiber Length	NaOH %
1	24.95	26.25	25.41
2	25.42	24.81	24.98
3	27.23	26.54	27.21
Delta	2.27	1.72	2.23
Rank	1	3	2

3.2 Flexural properties

Flexural test results are listed in Table 8 as per the Taguchi's orthogonal array. As in the tensile tests, to get a better composition higher S/N value is selected since higher flexural strength and modulus are desirable.

Table 8: Experimental data for flexural test

Fiber weight %	Fiber length mm	NaOH %	Flexural modulus MPa	Flexura l strength MPa	S/N ratio for F. Modulus	S/N ratio for F. Strength
5	5	0	2170	55.4	66.73	34.87
5	10	3	2980	48.8	69.48	33.76
5	15	6	2840	61.8	69.07	35.81
10	5	3	2520	49.1	68.03	33.82
10	10	6	2740	54.8	68.76	34.77
10	15	0	3080	52.5	69.77	34.4
15	5	6	3570	62.1	71.05	35.86
15	10	0	3160	66.9	69.99	36.5
15	15	3	2190	46.4	66.81	33.33

Figure 6 shows that 15 % weight, 10 mm fiber length and 6% NaOH treatment have maximum values. This has more effect on the flexural modulus of the composite. It can be observed that the fiber weight and fiber length have less influence on the flexural modulus, and the treatment of fiber has a greater effect on the flexural modulus because of the increased bonding ability of fiber with the matrix after chemical modification. From Table 9, it is observed that the contribution of NaOH treatment is more significant in increasing flexural modulus. Comparatively, fiber % and length of the fiber have very low significance in deciding the flexural modulus of the composites. The optimum levels of factors for flexural modulus are 15 % fiber weight fraction, 15 mm fiber length and 6% NaOH % for treatment.

Table 9: Response table for S/N ratios of flexural modulus

Level	Fiber %	Fiber Length	NaOH %
1	68.42	68.6	68.6
2	68.86	69.41	67.86
3	69.29	68.55	70.1
Delta	0.87	0.86	2.24
Rank	2	3	1

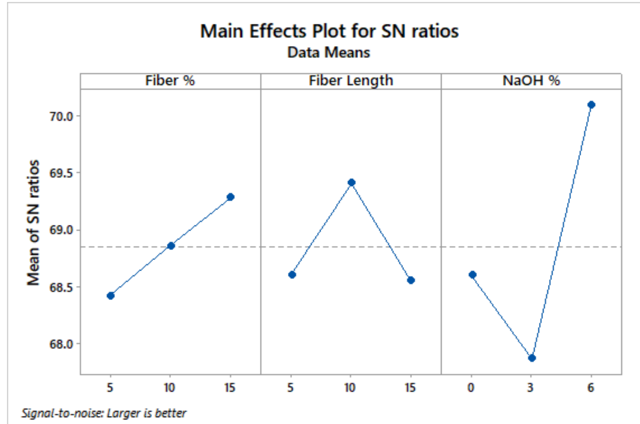

Figure 6: Main effect plot for S/N ratio for flexural modulus

Figure 7 shows that 15% weight, 10 mm fiber length and 6% NaOH treatment have maximum values. This has more effect on the flexural strength of the composite. S/N ratio was maximum for the combination of factors with levels 15% fiber weight, 10 mm length of the fiber and 0% NaOH treatment (untreated).

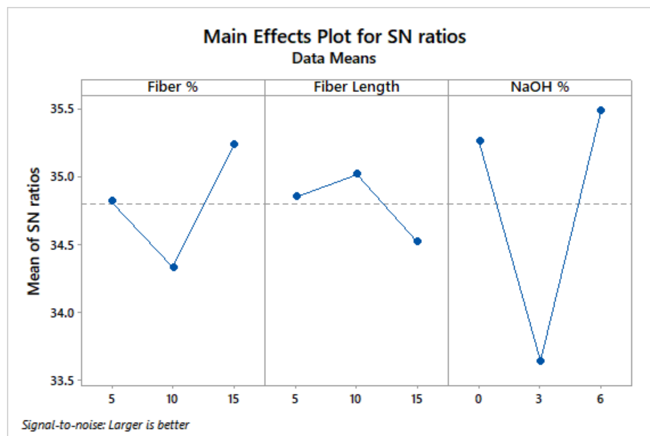

Figure 7: Main effect plot for S/N ratio for flexural strength

From the statistical data of S/N ratios of flexural strength, as listed in Table 10, it is observed that the contribution of fiber length and fiber weight fraction have very less influence on the flexural properties. At the same time, NaOH treatment has a significant effect on the flexural strength of the composite. The optimum levels of factors for flexural strength are 15 % fiber weight fraction, 10 mm fiber length and 6% NaOH % for treatment. Increase in fiber length

Table 10: Response table for S/N ratios of flexural strength

Level	Fiber %	Fiber Length	NaOH %
1	34.82	34.85	35.26
2	34.33	35.02	33.64
3	35.23	34.52	35.49
Delta	0.9	0.5	1.85
Rank	2	3	1

4. CONCLUSIONS

- The Taguchi method's design of experiments technique made it possible to analyze the tensile and flexural characteristics of composites, with fiber weight fraction, fiber length, NaOH treatment % as test variables.
- Experiment results were shown that fiber length has more effect on the tensile modulus of the composite 15 mm length of fiber has shown better tensile modulus, fiber weight % and NaOH treatments have a greater influence on the tensile strength. 15% fiber weight fraction and 6% NaOH solution treatment has given better tensile strength. NaOH treatment significantly affects the flexural properties and the optimum results were obtained at 6% NaOH treatment.
- Based on the analysis of S/N ratios of tensile and flexural properties, the optimum composition among chosen factors for the overall optimum characteristic of the composite is 15% fiber weight fraction, 15 mm fiber length and 6% NaOH solution treatment.

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