Rajmohan B. ✉
Arunachalam K.

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MECHANICAL PROPERTIES OF BANANA/BAMBOO/COCONUT FIBRE BASED PHENOLIC HYBRID COMPOSITES MADE BY USING AUTOCLAVE MOULDING TECHNIQUE

Summary

Nowadays, composite materials are greatly preferred, and they started replacing the traditional materials such as metal and wood due to their high strength-to-weight ratio. In this study, three different fibres, coconut, banana, and bamboo, are hybridized with seashell powder and phenolic resin to form polymer matrix composites. To prepare the test specimens, an autoclave moulding technique was used. The experimental design was developed as an orthogonal array utilizing the design of the experimental technique. Accordingly, experiments were conducted with respect to four factors and three levels. The factors include varying temperature, time, percentage by weight of resin, and fibre. The mechanical properties such as hardness, tensile strength, and flexural strength were experimentally measured. Also, the grey relational analysis was carried out and it can be observed that specimen 2 with a tensile strength of 20.71 MPa, the flexural strength of 41.87 MPa, and hardness 36.846 BHN (Percentage by weight of resin and reinforcement was 60 wt. % and 36 wt. %, respectively. Temperature was 70˚C and time was 15 min) was the most superior specimen to all other specimens. SEM micrographs were obtained, and surface topography was investigated. The chemical composition of the composite was determined by using the EDX analysis. The findings revealed the optimal mass percentage for superior strength, hardness, and surface morphological properties.

Key words: *natural fibres, mechanical properties, NaOH treatment, SEM analysis, EDX analysis*

1. Introduction

In recent years, the interest in using natural fibres has increased globally. The automotive industry is an example that shows that industries can change and become more environmentally friendly by making small steps towards a sustainable future. Natural fibres have a significant role in achieving the goals of sustainability and environmentally friendly products produced by industry. As technology progresses and the processes for developing natural fibre products become more refined, it becomes easier and more cost-effective to replace the current less environmentally friendly products with natural fibre based products [1]. Fibre-reinforced plastics that are currently used on spacecraft result in approximately 40% of weight savings which in turn results in fuel cost savings too [2]. The application of natural fibre composites has increased tremendously with load bearing and outdoor applications such

as automotive exterior, underfloor panelling, sports equipment, and marine structures [3]. Grey relational analysis was conducted to determine the creation of a unique response factor in multi-response factors due to different weights of individual responses [4]. However, alkaline treatment is the most widely used technique for enhancing mechanical, thermal, and water absorption properties of natural fibre based epoxy composites due to its economy and high efficiency. This method has been widely employed to improve the incompatibility and poor interfacial adhesion between the filler and the matrix [5]. The kenaf fibres of both untreated and sodium hydroxide (NaOH) treated versions can be deployed as a reinforcing agent of epoxy resin. Moreover, the alkaline treatment improved mechanical properties of the composites due to the improvement in fibre/matrix compatibility [6]. Alkaline treatment improved the tensile and flexural modulus and coconut/epoxy composite strength. Scanning electron microscope (SEM) studies confirmed enhanced fibre/matrix interfaces when the alkaline treatment was used [7]. SEM is an excellent tool available to determine the internal structure of the coconut fibre. With the help of 3D information and three orthogonal virtual slides, the fibres can be scanned and the fibre porosity and dimensions of lumen, lacuna, and elementary fibres can be observed. According to the published studies [8], coconut fibre has high porosity. In comparison, the mechanical properties of plain woven hybrid composites are superior to those of randomly-oriented composites. The result shows that the minimum stress was developed due to the distribution of load along the fibre direction. Furthermore, the treatment with alkaline sodium hydroxide (NaOH) and sodium lauryl sulfate (SLS) had a positive impact, i.e., it enhanced the mechanical strength through improved interfacial bonding. SEM was used to study the morphological structure of the fractured samples to understand the de-bonding of fibre/matrix adhesion [9]. Fibre direction and fibre volume fraction are significant due to the orthotropic behaviour in the stress condition [10]. Grey relational analysis was used to determine the best formation parameters in terms of the output response. The studies with optimized parameters reveal a minimal difference in grade value of 0.15 percent from the projected grade, which is acceptable [11].

The alkaline treatment of natural fibre improves the tensile, flexural, and compressive strength of the fibre. However, the fibre direction and fibre volume fraction affect the strength and crack path in crack propagation of the composite. Therefore, in this present study, the suitability of 5% sodium hydroxide (NaOH) alkaline treated fibres such as banana, bamboo, and coconut fibre for the application of building an automotive body was studied. It was done by fabricating the specimens and conducting the test to check mechanical properties such as hardness, tensile strength, flexural strength, surface topography and the chemical composition of the composite specimens.

2. Materials

In automobile industries, natural fibres are widely used due to their excellent mechanical features. Figure 1(a)-(d) shows the photographic images of banana fibre, bamboo fibre, coconut fibre, and seashell powder, respectively. The banana fibre is extracted from leaves of banana trees, and it is available as a raw banana fibrous material. Coconut is nothing but the fibre plucked from the outer husk of coconut, which consists of coarse fibres. Bamboo fibres are extracted from the stem of natural bamboo by cutting the bamboo stem into small thick strips and pulling the fibre out of these strips. These three fibres were purchased from Go Green Products, Chennai. Table 1 shows physical, chemical, and mechanical properties of the natural fibres [12]. Alkaline treatment of coconut fibre increased the fibre surface roughness leading to the increase in mechanical interlocking between the fibre and the matrix in the composites. Coconut fibre was treated with 5% NaOH for 72 hours and dried in the sunlight [13]. However, a notable disadvantage of lignocellulosic fibres is their polarity, which makes it incompatible with the matrix. This incompatibility results in poor interfacial bonding between the fibres and the matrix. This defect can be remedied by the chemical

modification of fibres [14]. Seashells are collected from the seashore and powdered by using a ball milling machine. Table 2 shows the chemical composition of seashell powder [15]. Phenolic resin is used as a matrix material because its coefficient of thermal expansion is similar to that of aluminium. The phenolic resin was purchased from Vasavibala Resins, Chennai. The properties of the phenolic resin are presented in Table 3 [12].

(a) Banana fibre (b) Bamboo fibre (c) Coconut fibre (d) Seashell powder

Fig. 1 Fibre and powder

Table 1 Physical, chemical, and mechanical properties of natural fibres [12]

Cellulosic fibres	Banana	Bamboo	Coconut
Chemical properties of natural fibres			
Cellulose (%)	62.5	34.5	46
Hemi-cellulose (%)	12.5	20.5	0.3
Pectin (%)	4	-	4
Ligin (%)	7.5	26	45
Physical properties of natural fibres			
Diameter (μm)	12-30	12-30	10-460
Length (mm)	2.9	2	1.3
Density (kg/m^3)	1325	1500	1250
Moisture gain (%)	-	-	13
Micro-fibril angle (degree)	11	-	44
Mechanical properties of natural fibres			
Relative density (g/m^3)	0.6-1.1	1.35	1.15-1.46
Tensile strength (MPa)	140-800	500	95-230
Elastic modulus (GPa)	11-32	12	2.8-6
Specific modulus (GPa.cm^3/g)	25	9	4
Elongation at failure (%)	2.5-3.7	1.5-9	15-51.4

Table 2 Chemical composition of seashell powder [15]

Oxide	Percentage (%)
SiO_2	1.60
Al_2O_3	0.92
CaO	51.56
MgO	1.43
Na_2O	0.08
K_2O	0.06
H_2O	0.31
LOI	41.84

Table 3 Properties of phenolic resin [12]

Property	Phenolic
Density (g/cm^3)	1.29
Elastic modulus (GPa)	2.8-4.8
Tensile strength (MPa)	35-62
Compressive strength (MPa)	210-360
Elongation (%)	1.5-2
Water absorption (24 h at 20 ˚C)	0.1-0.36
Cure temperature (˚C)	25-200

3. Alkaline treatment

To perform the alkaline treatment, the alkaline solution was prepared using a 5% sodium hydroxide (NaOH) solution. The chosen fibres, coconut, banana, and bamboo, were soaked for about 72 hours in this solution. After having been taken out of the solution, the fibres were thoroughly washed with water followed by distilled water. Then the fibres were dried in the sunlight to remove the moisture content and were used to fabricate the composite. The primary advantage of this treatment is the increase in strengths of both fibre and composite. This is because crystalline cellulose forms the major component in the natural fibre which consists of waxy substances, lignin, and hemicelluloses. Over time, both lignin and hemicelluloses tend to degrade. So, the sodium hydroxide (NaOH) alkaline fibre treatments are proved to improve the adhesion properties of natural fibres through the removal of lignin, pectin, and hemicelluloses, thus providing the fibre surface with a rough texture.

4. Fabrication methodology

In this study, composite specimens were fabricated using the autoclave moulding process. The specimens were made at a vacuum pressure of 1 bar. A closed chamber with a hollow cylindrical heater was utilised, with the chamber insulated with glass wool to prevent heat emission from the heater. The heater had a 1,000 W rating and could heat up to 200 ˚C. An external temperature controller controlled the temperature of the chamber. The long fibres were sliced into 100 mm and then used in the mould (100 × 100 mm). The hand layup method was initially used to prepare layers of fibres and fibres stacked upon the mould surface, followed by coating the phenolic resin on each layer. The mould was then placed inside the vacuum chamber to fabricate the specimen.

Hardness, tensile, and flexural strength were tested with one trial for each specimen and the data are tabulated in Table 1. The design of variable levels and parameters is presented in Table 4. Table 5 shows the L9 orthogonal array matrix and the test results.

Table 4 Design of variable levels and parameters

Parameters	Units	Levels		
		1	2	3
Fibre and powder	gram	20+3	30+6	40+9
Resin	gram	60	90	120
Time	min	10	15	20
Temperature	^{0}C	60	70	80

Table 5 L9 orthogonal array matrix and test results

Specimen No	Fibre (g)	Resin (g)	Time (min)	Temp (˚C)	Tensile strength (MPa)	Flexural strength (MPa)	Hardness value (BHN)
Specimen 1	23	60	10	60	22.50	32.03	29.353
Specimen 2	36	60	15	70	20.71	41.87	36.846
Specimen 3	49	60	20	80	10.84	25.46	30.364
Specimen 4	23	90	15	60	11.71	29.05	33.507
Specimen 5	36	90	20	70	15.86	25.66	35.710
Specimen 6	49	90	10	80	14.27	31.81	36.617
Specimen 7	23	120	20	60	18.32	30.70	32.485
Specimen 8	36	120	10	70	20.29	44.75	31.509
Specimen 9	49	120	15	80	16.98	42.09	35.388

5. Results and discussion

5.1 Hardness

Hardness test was conducted using a Brinell hardness tester TKB – 3000 SR.NO: 2010/671. In the Brinell hardness tester, 5000Nwas applied for 10 s, and 100 ball diameter was used. The output hardness values are shown in Figure 2. The reinforcement percentage exerted a significant effect on the mechanical behaviour of the composite. This study obtained the highest hardness value in specimen 2 (36.846 BHN). The mass of the resin was 60 g, the mass of the reinforcement was 36 g, temperature was 70 ˚C, and fabrication time was 15 min. The values obtained for each specimen are as follows: specimen 5, specimen 6 and specimen 9 show the hardness values of 35.710 BHN, 36.617 BHN, and 35.388BHN, respectively. The hardness of these specimens is next to the highest hardness.

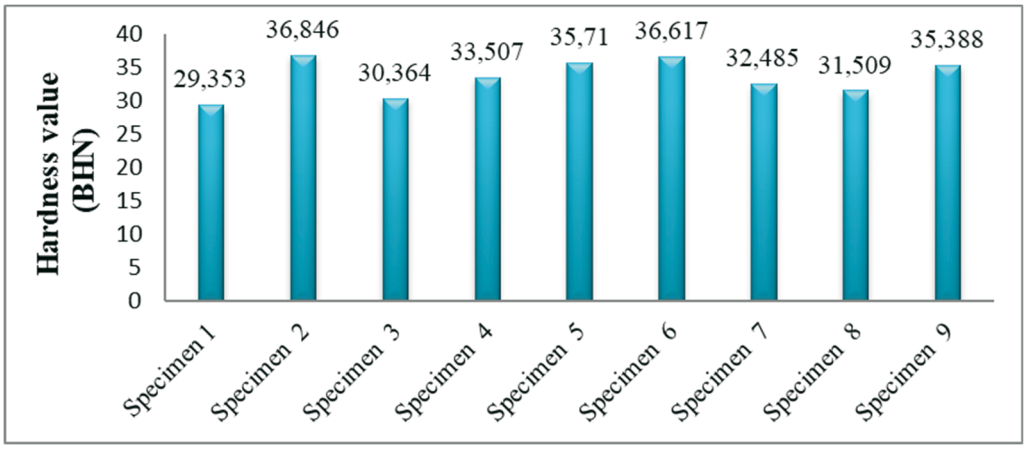


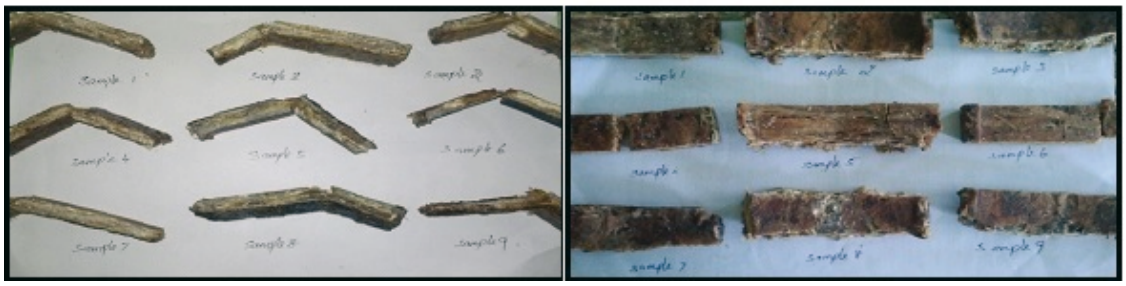
Fig. 2 Hardness values (BHN) of specimens

Both specimens 2 and 5 had medium level reinforcement, whereas specimens 6 and 9 had high-level reinforcement. It is observed that the percentage of the reinforcement has influence on the hardness of the specimen. The hardness values of specimen 1 and specimen 3 were 29.353 BHN and 30.364 BHN, respectively. These values were found to be very low compared to specimens 2, 5, 6, and 9. Both the matrix material composition and the weight percentage of the reinforcement material had an impact on the hardness value. This is the reason for the very low hardness value in specimens 1 and 3. Further, poor hardness was observed in specimens 1, 4, and 7. In these specimens, the reinforcement weight was the least (23 g). Figure 2 shows that the weight percentage of reinforcement is a major influencing factor on the value of hardness.

5.2 Tensile strength

Tensile strength is a measure that can be defined as a material's ability to withstand longitudinal stress. A tensile test was carried out as per the ASTM E8M standard. In composite materials, fibre treatment and interfacial bonding phenomena increase mechanical strength. Further, both temperature and time parameters also influence the tensile strength.

Figure 3 shows photographic images of composite specimens subjected to tensile strength (3a) and flexural strength (3b) testing. The values of tensile strength of the composites are shown in Figure 4. It can be seen that the highest tensile strength (22.50 MPa) was observed in specimen 1. Specimens 2 (20.71 MPa) and 8 (20.29 MPa) had tensile strength values closer to specimen 1. This is because the adhesion between reinforcement (fibre and powder) and matrix resin in these three specimens was good. In addition to that, it was a very close and tight bond with superior interface bonding that resulted from the application of temperature and time factors.



(a) **(b)**

Fig. 3 Composite specimens subjected to tensile strength (a) and flexural strength (b) testing

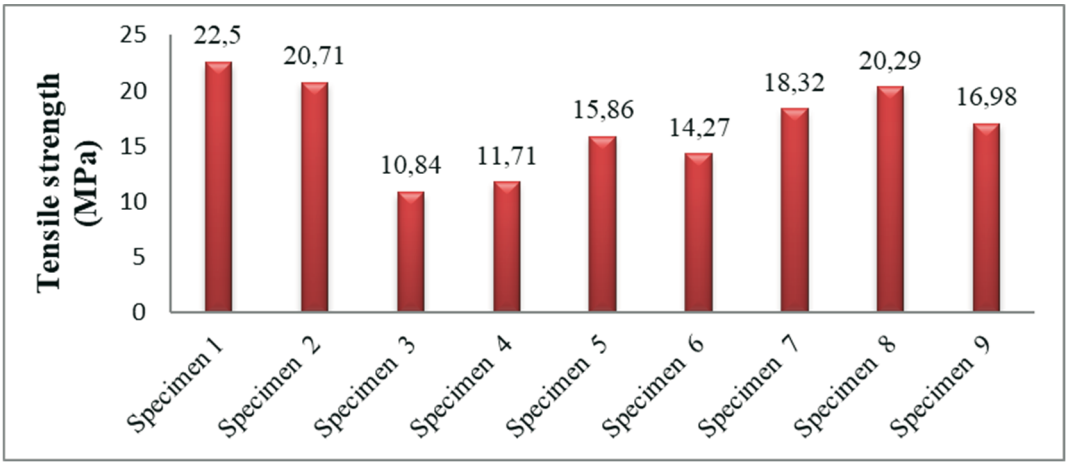


Fig. 4 Tensile strength (MPa) of specimens

Figure 4 shows that specimens 1, 2, and 8 exhibited the highest values of tensile strength based on the interfacial bonding in composite specimens. The values of specimens 3, 4, 5, and 6 were as low as 10.84 MPa, 11.71 MPa, 15.86 MPa, and 14.27 MPa, respectively. The interfacial bonding strength was reduced because of the high heat applied to the composite specimens that were exposed to a higher temperature and for a longer period of time, i.e.80 ˚C and 20 minutes. This infers that both temperatures and time are significant factors in creating an impact on the tensile strength of the composite. Meanwhile, matrix resin and reinforcement weight percentage are denoted as minor factors that reduce tensile strength. Therefore, it can be concluded that higher tensile strength can be obtained if the interfacial bonding in specimens is stronger.

5.3 Flexural strength

Flexural strength is also known as bending strength. The flexural strength is stress at failure in bending. A flexural test was carried out involving bending (three-point) using UTM as per the ASTM D790 standard. Figure 5 shows the results of the investigated bending strength. The flexural strength of specimens 2, 8, and 9 was41.87 MPa, 44.75 MPa, and 42.09 MPa, respectively. There was a higher flexural strength observed in specimen 8 compared to other composite specimens. Higher flexural strength is based on the percentage of reinforcement and resin matrix composition with low and medium levels of the temperature range. Low flexural strength was obtained in specimens 3 (25.46 MPa), 4 (29.05 MPa), and 5 (25.66 MPa). The reduced flexural strength might be attributed to the low or high level of resin or reinforcement. The flexural strength of specimens 3 and 5 was low because of the excess time applied at a high temperature level (20 min). The high temperature (80˚C) used for a longer period of time (20 min) made the specimen brittle due to overheating. This is also the reason for decreased flexural and tensile strength values, but at the same time, hardness of these specimens was good due to high toughness.

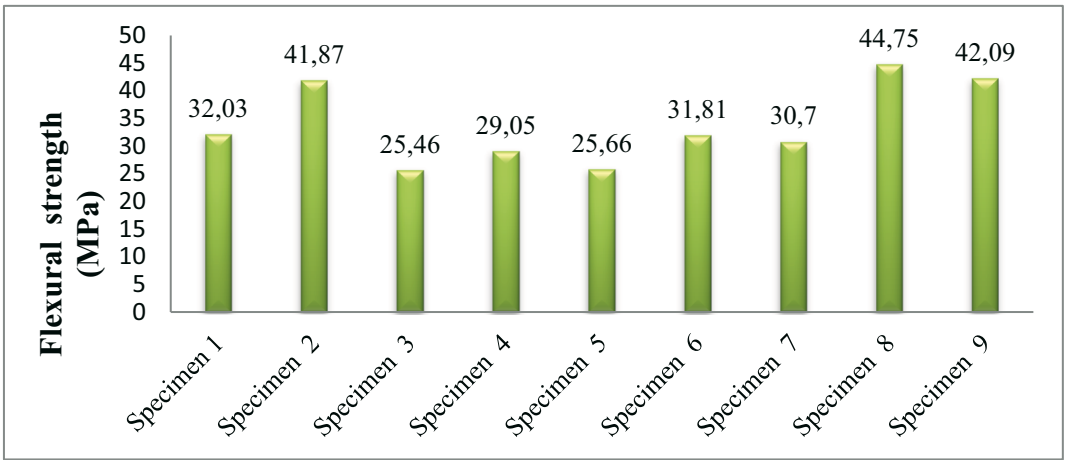


Fig. 5 Flexural strength (MPa) of specimens

6. Grey Taguchi method

In this experiment, three levels and four input parameters were used (see Table 4). The three levels are low, medium, and high and the four parameters are: fibres and powder, resin, temperature, and time. An L_9 orthogonal array was developed by variable parameters and created as orthogonal design. The input composition of matrix and reinforcement were in a 3:1 ratio. Specimens were fabricated as per the Taguchi L9 orthogonal array. Table 5 shows the experimental runs and their results.

6.1 Normalization

Normalization is a tool used for pre-processing experimental data. Normalization has three characteristic formulas such as ‘larger the better’, ‘smaller the better, and ‘nominal the better.’ Based on the application and usage, the user chooses a characteristic formula. For example, the values of mechanical properties should be higher for automobile body building applications. So ‘larger the better’ was chosen for all the three output results in this study. Table 6 clearly illustrates the normalization, grey relational coefficient, and grey grade values. The formula used for ‘larger the better’ is as follows:

$$Xi(K)=\{Xi(K) - \min Xi(K)\} / \{\max Xi(K) - \min Xi(K)\} \quad (1)$$

Table 6 Normalization, grey relational coefficient and grey grade

Specimen No	Normalization			Grey relational coefficients			Grey grade
	Tensile strength (MPa)	Flexural strength (MPa)	Hardness value (BHN)	Tensile strength (MPa)	Flexural strength (MPa)	Hardness value (BHN)	
Specimen 1	1	0.3394	0	1	0.4309	0.3333	0.6292
Specimen 2	0.8482	0.8495	1	0.7669	0.7687	1	0.8374
Specimen 3	0	0	0.1346	0.3333	0.3333	0.3661	0.3431
Specimen 4	0.0745	0.1862	0.5544	0.3507	0.3804	0.5287	0.4130
Specimen 5	0.4323	0.0104	0.8487	0.4682	0.3355	0.7656	0.5176
Specimen 6	0.2957	0.3200	0.9693	0.4152	0.4267	0.9424	0.5767
Specimen 7	0.6405	0.2707	0.4182	0.5818	0.4068	0.4622	0.4933
Specimen 8	0.8124	1	0.2874	0.7269	1	0.4123	0.7144
Specimen 9	0.5266	0.8611	0.8057	0.5136	0.7824	0.7201	0.6561

6.2 Grey relational coefficient

After the normalization process, the grey relational coefficient value (Table 6) was determined to find the correlation between ideal and normalized output results. Formula used to determine the grey relational coefficient value is the following.

$$\varepsilon i(K) = \{\Delta \min + \varsigma \Delta max\} / \{\Delta oi(K) + \varsigma.\Delta max\} \quad (2)$$

where $\varsigma = 0.5$, $\Delta_{oi}(k) = X_0(k) - Xi(k)$, $X_0(k) = 1$

6.3 Grey grade

Grey grade was identified based on the weightage allotted to the output parameters gathered from the grey relational coefficient. Tensile strength is essential among the three outputs compared to the other two output parameters. So, 0.4 weightage was given to tensile strength, and the other two parameters i.e., hardness and flexural strength, were given 0.3 weightage. The grey relational coefficient values were multiplied by the weighted levels (0.4, 0.3 and 0.3), and they were added to each other, after which the grey grade was calculated. The specimen in grey grade obtained the highest value and this specimen is superior to other eight specimens.

In Table 6, it can be observed that specimen 2 (resin 60 g, 36 g reinforcement, temperature 70˚C, time 15 min) was superior to all other specimens. The input parameters of this specimen, such as reinforcement (fibre and seashell powder), applied temperature, and time duration were at the medium level. On the other hand, the input parameter matrix resin was at the low level. Therefore, both temperature and time as input parameters have an impact on the adhesion and bonding of the composite specimen. Due to this influence, atoms and molecules in the matrix (resin) and reinforcement (fibre and powder) combine and form a tight interfacial bond between them. Good interfacial bond enhances the mechanical strength of the composite material.

7. Scanning electron microscope

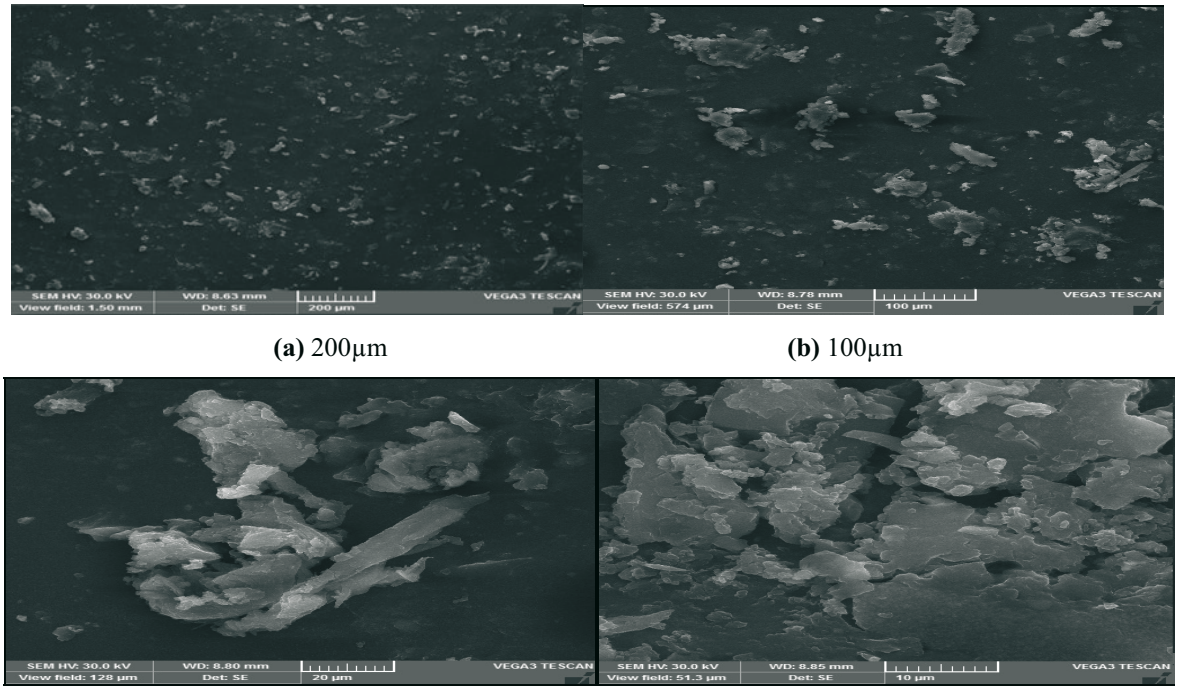
A VEGA3 TESCAN scanning electron microscope was used to take scanning electron microscope (SEM) micrographs. Two different specimens were selected, and their surface textures were examined. The surface microstructure was investigated for matrix resin and reinforcement by changing the magnification of images and the viewfield in specimens 2 and 8. Specimens 2 and 8 were chosen for SEM and energy dispersive X-ray (EDX) analyses based on the GRA optimization results.

Nature of specimen: Dry condition

Coating: Prior to the SEM imaging process, no special coating was applied to the specimen

Specimen size: 1 cubic centimetre and a thin film of 1×1 centimetre

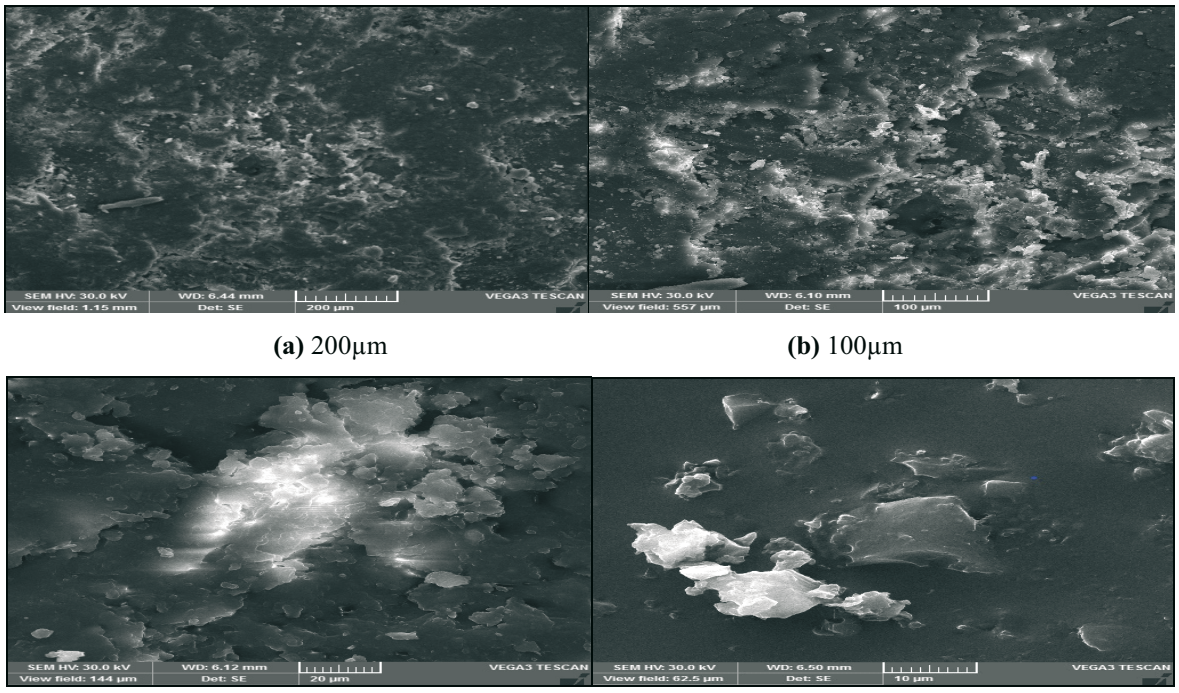
Figure 6(a)-(d) shows the SEM micrographs of specimen 2 with scale bars of 200µm,100µm, 20µm, and 10µm. Figure 7(a)-(d) illustrates the SEM micrographs of specimen 8 with scale bars of 200µm,100µm, 20µm, and 10µm.



(a) 200µm **(b)** 100µm

(c) 20µm **(d)** 10µm

Fig. 6 SEM images of specimen 2



(a) 200µm **(b)** 100µm

(c) 20µm **(d)** 10µm

Fig. 7 SEM images of specimen 8

From Figures 6 and 7, it can be concluded that there is a tight package of fibre and matrix in the micrograph. Thus, the adhesion between resin and fibre was good. Figure 7(a) shows the fibre end position in a line segment manner. Figure 6(a) reveals that the resin was well distributed throughout specimen 2. The percentage by mass of fibre reinforcement was 37.5 % and the percentage by mass of resin was 62.5% for specimen 2. In the medium level specimen, the resin and the reinforcement were equally combined with each other.

In Figure 7(a) the presence of a large amount of matrix resin can be observed in specimen 8. The percentage by mass of fibre reinforcement was 23.07 % and the percentage by mass of resin was 76.92%. When viewed at 200µm, it is understood from the images that there are no cracks and blowholes on the top surface and in the inner layers. The images show the fibre mixture, whereas the matrix resin is uniform throughout the specimen. From Figures 6 and 7, it is inferred that the interfacial interaction between fibre and resin is better since the former aids in enhancing the mechanical strength of the composite specimens.

8. Energy dispersive X-ray analysis

The specimens that exhibited higher strength were analyzed for their chemical composition using the EDX analysis. The scanning electron microscope paired with a Bruker EDX instrument was used to perform the EDX analysis.The chemical composition of specimens 2 and 8 is shown in EDX graphs, Figures 8 and 9.

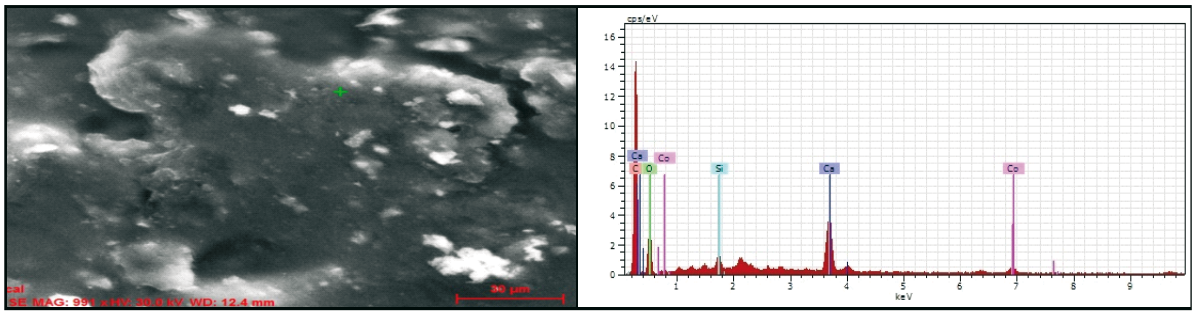


Fig. 8 EDX chemical composition graph of specimen 2

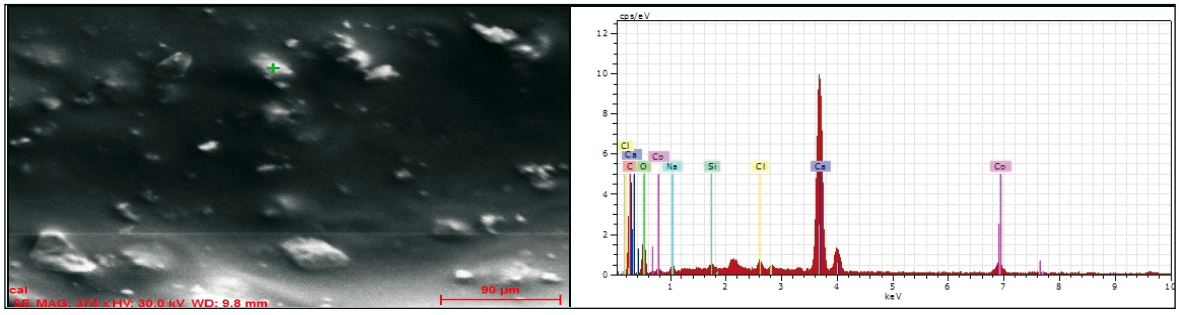


Fig. 9 EDX chemical composition graph of specimen 8

The weight percentage values of the chemical compositionof specimen 2 and specimen 8 are presented in Tables 7 and 8. In the EDX analysis, the graphs show that both specimens 2 and 8 reach their calcium (Ca) peaks due to the presence of seashell powder. This phenomenon is appreciated since it contributes to the improvement of mechanical properties of the specimen. Figure 8 shows that the low intensity of carbon (C), cobalt (Co), oxygen (O), and silicon (Si) peaks indicates a lower percentage of the elemental content in specimen 2. Figure 9 shows that the low intensity of carbon (C), cobalt (Co), oxygen (O), chlorine (Cl), sodium (Na), and silicon (Si) peak indicates the lower percentage of the elemental content in specimen 8. In the results of the EDX analysis of the composites, the presence of an oxygen peak indicates that oxide formation has occurred in specimens 2 and 8.

Table 7 EDX chemical composition of specimen 2

Sl.No	El	AN	Series	Unn. C (wt.%)	Norm. C (wt.%)	Atom.C (at.%)	Error (1 sigma)
1	C	6	K-series	62.47	62.47	70.67	8.84
2	O	8	K-series	32.62	32.62	27.68	5.98
3	Ca	20	K-series	3.51	3.51	1.18	0.15
4	Si	14	K-series	0.54	0.54	0.25	0.06
5	Co	27	K-series	0.88	0.88	0.21	0.08

Table 8 EDX chemical composition of specimen 8

Sl.No	El	AN	Series	Unn. C (wt.%)	Norm. C (wt.%)	Atom.C (at.%)	Error (1 sigma)
1	C	6	K-series	33.26	45.58	59.09	5.51
2	O	8	K-series	24.58	33.66	32.75	5.01
3	Ca	20	K-series	12.14	16.63	6.46	0.42
4	Na	11	K-series	0.91	1.24	0.84	0.13
5	Co	27	K-series	1.71	2.32	0.63	0.08

9. Conclusion

The study conducted on the hybrid composite made of natural fibres such as coconut, banana, and bamboo fibres showed excellent mechanical properties of the composite. The maximum hardness value was obtained from specimen 2 (36.846 BHN). The specimen contained 60 g of resin, 36 g of reinforcement and it was fabricated at a temperature of 70˚C during 15min. This high hardness value resulted from the mixture of the reinforcement and the matrix. Specimens 1 and 8 exhibited higher tensile strength, 22.50 MPa, and flexural strength, 44.75 MPa. Better interfacial bonding exerted influence on these two output parameters. The 5% NaOH alkaline treatment of the three natural fibres improved the mechanical strength of the composite, and it also eliminated dust and other impurities from the fibre. It was very helpful to achieve interfacial adhesive bonding between matrix and reinforcement. The matrix and reinforcement parameters highly influenced the mechanical properties and had an important role in fabricating the composite specimens. The mechanical properties were also improved by variations in other minor factors such as temperature and time. The grey relational analysis was selected to determine the optimum composition of matrix and reinforcement. Thus, the grey relational analysis proved (specimen 2) the suitability of the specimen exhibiting higher stress and hardness for automobile body building applications. The microstructural analysis results revealed better adhesion and uniform distribution between the resin and the fibre.

10. Future work

Instead of bamboo, banana, and coconut fibres, other natural fibres, such as jute, sisal, kenaf, abaca, and hemp fibres, can be used for reinforcement. Also, different resins, fabrication techniques, and mechanical testing and optimization techniques should be used to study the material.

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Rajmohan B.*, Research Scholar
Dr. Arunachalam K., Professor
MIT Campus, Anna University, Chennai, Tamilnadu, India
*Corresponding author:
rajmohanbose1990@gmail.com