

Optimisation of mechanical behaviour of *Calotropis gigantea* and *Prosopis juliflora* natural fibre-based hybrid composites by using Taguchi-Grey relational analysis

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ABSTRACT

The properties of organic fibre-based hybrid materials are influenced by a variety of factors and even minor changes in these variables can outcome in substantial discrepancies in strength. In this regard, the current study aims to optimise various influencing parameters such as weight percentage, alkaline treatment concentration, and fabrication parameters (compression moulding pressure, and temperature), with the goal of enhancing the overall strength of the composite. *Calotropis gigantea*-stem and *Prosopis juliflora*-bark fibres were used in varying weight percentages to create epoxy-based hybrid composites. After fabrication the mechanical characterisation like tensile, flexural, and impact properties of the composites were tested. Taguchi experimental design was applied, and the results were analysed using a hybrid Taguchi-grey relational investigation method. It was observed that a combination of 20 wt.% *Calotropis gigantea*/20 wt.% *Prosopis juliflora*/6 % NaOH pretreatment/100 °C temperature with 14 MPa pressure and had the most desirable mechanical properties in the fabricated composites. *Calotropis gigantea* ranks first in enhancing the composite strength, followed by *Prosopis Juliflora*, NaOH pretreatment%, compression moulding temperature and pressure. This work highlights the significant role of *Calotropis gigantea* and *Prosopis Juliflora* fibres in enhancing composite strength and provides valuable insights for future research in the field of hybrid composite development.

Introduction

Polymeric based lightweight materials are utilised in a numerous engineering applications owing to its superior properties over the conventional counterparts [1–3]. However, the majority of applications employ environmentally hazardous synthetic fibre-based composites. The utilisation of organic fibre-based composites was found to be an efficient method for reducing the use of non-sustainable artificial fibre composites [4,5]. The best way to reduce pollution and other detrimental environmental effects is to use bio-based materials, such as natural fibres like banana, kenaf, pineapple, cotton, jute, and hemp [6, 7]. The fibres made from natural materials have numerous benefits,

including being lightweight, less expensive, recyclable, good weight to strength ratio, and good acoustic properties [8,9]. However, natural fibres are hydrophilic, which may adversely affect composite properties [10,11]. To overcome this, various effective solutions (fibre treatment, hybridisation, modification in fabrication method, etc.) have been implemented and studied [1,12]. The properties of organic fibre-based composites vary depending on chemical modification of fibre, hybridisation, and fabrication methods [13–16]. Hence, it is critical to optimise the factors that will result in the production of high-strength materials. Furthermore, fibre selection is necessary since the strength of natural fibres varies from species to species [17–19].

Calotropis gigantea (CG) is an Asian country-based shrub plant native

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to India and China. These fibres are widely available in African countries as well [20]. CG stem is a source of natural cellulosic bast fibrous material with attractive properties wherein the amount of cellulose percentage, strength of the fibrous material, and its thickness swelling fall somewhere in between linen and cotton [21]. CG fibres (CGF) are heavier per square metre than cotton fibres and have a higher tensile and abrasive strength [22]. CG is a rapidly growing shrub throughout much of India, hence, using it as a resource of high strength cellulosic fibrous material could have significant economic implications. This fibre has the ability to substitute the other natural fibres as a reinforcing agent [23, 24]. Ashori and Bahreini [25] investigated CG fibres reinforced natural composites and concluded that CG fibre is a promising raw material for natural composites. The maximum tensile characteristics of the CG fibre-based material was reported to be 381 MPa, breaking strain was 2.1 %, and the elasticity of the material was 9.7 GPa. The mechanical characterisation of organic fibre-based composites is primarily determined by crystallinity orientation [26]. CGF has 42.5 % crystallinity and an 85.4 % crystallinity orientation index. Natural fibres can also be extracted from plant bark. One such plant whose bark can be used to extract fibre is *Prosopis juliflora* (PJ). PJ belongs to the Leguminosae genus *Prosopis*, which has 40 varieties classified into five branches and six groups [27]. PJ fibre (PJF) is made up of wax (0.72 %), cellulose (60.23 %), lignin (18.25 %) hemicellulose (17.25 %), and other ingredients (5.12 %). The PJ fibre density (0.49 g/cm^3) is lower than that of manmade fibres such as polyacrylonitrile (1.36 g/cm^3), Kevlar (1.49 g/cm^3), mineral wool (1.62 g/cm^3), and fibreglass (2.52 g/cm^3) [28].

Natural fibre composites readily absorb moisture from their surroundings, resulting in lower strength due to decreased fibre matrix bonding [29–31]. Fibre and matrix compatibility and adhesion must be strong for composites to have improved properties. Surface modification is a common approach for natural fibres, which aids in the enhanced interfacial bonding with the matrix [32–34]. To alter the surface properties of the natural filaments, a variety of pre-treatments such as alkaline, benzoylation, silane and permanganate treatment are used [35]. Due to its effectiveness and low cost, alkaline treatment was found to be the most effective method in natural fibre treatment [35]. A common solvent used in the treatment of natural fibres is sodium hydroxide (NaOH). NaOH cleans the fibre structure of dirt and unwanted chemical substances while also creating hydroxyl functionality in the fibre surface [36,37]. The concentration of NaOH is critical in changing the mechanical strength of natural fibres. For instance, when coir fibre was pre-processed with alkaline solution at 10 wt.% over a 4-h period, the flexural and tensile modulus enhanced by 25 % and 28 %, respectively, when compared to a composite with untreated fibre [38]. Khan et al. [39] fabricated NaOH treated banana fibre reinforced epoxy laminates. The NaOH concentration was varied at four different levels such as 0 %, 2.5 %, 4.5 %, and 6.5 %. It was reported that compared to untreated fibre composite, composite laminate with 4.5 % NaOH treated fibre showed ca. 24 % and ca. 35 % improvements in tensile and compressive strength, respectively. Furthermore, it has been reported that weight percentages of fibre loading and manufacturing parameters, such as moulding temperature and pressure, are critical in determining composite's mechanical properties. The mechanical characterisation of polypropylene/jute-based composites was investigated by Rana et al. [40]. The authors, the mechanical properties of the composite varied when adjusting the fibre content such as 30, 40, 50, and 60 wt.%. Notably, maximum tensile properties of 73 MPa were noted at 60 wt.% fibre loading. By varying the compression moulding parameters, Rangaswamy et al. [41] fabricated glass/Kevlar/epoxy composites through a compression moulding method. Composites were fabricated by varying compression pressures, temperatures and times. The moulding parameters were found to have a substantial result on the mechanical properties of the lightweight material produced. The composite developed at a mould temperature of 80°C , pressure of 20 MPa, and a time of 15 min had maximum tensile and flexural strength of approximately 181 and 143 MPa, respectively. Thus, it was found that

Table 1

Influencing process constraints and its stages.

Sl.No.	Processing Factors	Symbols	Levels			
			L1	L2	L3	L4
1	CG fibre (wt.%)	A	0	10	15	20
2	PJ fibre (wt.%)	B	0	10	15	20
3	NaOH Treatment (%)	C	0	2	4	6
4	Compression Pressure (MPa)	D	10	12	14	16
5	Temperature ($^\circ\text{C}$)	E	100	120	-	-

all chemical treatment concentration, fibre loading, compression moulding temperature and pressure were critical in influencing the strength of the lightweight materials. As a result, an effort has been made in this current study to optimise the chemical treatment concentration, fibre loading, compression moulding temperature, and pressure.

The main goal of this research is to fabricate a hybrid composite with CG and PJ fibre reinforcement and to optimise the processing parameters. To meet this goal, four loading amounts of CGF and PJF (0, 10, 15, and 20 wt.%) were chosen. The compression moulding method was used to create epoxy-based hybrid composites with varying pressures (10, 12, 14, and 16 MPa) and temperatures (100 and 120°C). Both the natural fibres were treated with NaOH at various chemical concentrations (0, 2, 4, and 6%). The process parameters were optimised using a mixture of Taguchi and Grey relational examination. A scanning electron microscope (SEM) was used to observe the fractured surfaces of the specimens. It is envisaged that this research would help researchers to know the impact of the various processing parameters commonly used for hybrid composite fabrication.

Experimentation

Materials

Plant stems of CG and bark from PJ were collected from agricultural areas near Theni, Tamil Nadu, India. The traditional water retting method was used to extract the fibres from their stems and barks. Stems of CG and barks of PJ were thoroughly dried for one week before being soaked in water for 66 h, after which the fibres were separated. The extracted fibres were cleaned carefully with fresh water accompanied by deionised water, and then dried in the sunlight up to 7 days, and finally deposited in plastic containers. Epoxy with a grade of LY556 and curing agent with a grade of HY951 were employed as the matrix material purchased from Jewanthi Enterprises in Chennai, Tamil Nadu, India.

Fibre treatment and composite fabrication

The CG and PJ fibres were pre-treated with NaOH to eliminate the unwanted surplus content, hemicellulose, and wax from the fibre, increasing its crystalline feature and interaction with matrix materials. The fibres were soaked in NaOH solutions prepared at 2, 4 and 6% NaOH concentrations for 4 h during alkaline treatment. The alkali-treated fibres were cleaned, and sun dried before being placed in a 70°C oven for 180 min. Finally, the fibres were manually cut into lengths of 5 mm.

The composites were fabricated through a compression moulding technique. The fibres were initially arranged in a steel mould with a size of $300 \times 300 \times 3 \text{ mm}^3$. A 10:1 mixture of epoxy and hardener was used to create the matrix mixture (prescribed by supplier). The matrix solution was then poured at top side of the fibre in the matrix. Finally, the steel mould was then closed and compressed for 24 h at four different pressures (10, 12, 14, and 16 MPa) and two temperatures (100 and 120°C). The pressure and temperature were regulated using the control system of the compression moulding machine (Make – Flowmech & Pillar-type (Down-Stroke) – 20 Ton press).

A Taguchi-based grey relationship analysis (GRA) was employed. Using Taguchi method, L16 orthogonal array of mixed levels was

Table 2L₁₆ Orthogonal array for experimentations.

Experiment No.	CGF (wt. %)	PJF (wt. %)	NaOH (%)	Compression Pressure (MPa)	Temperature (°C)
1	0	0	0	10	100
2	0	10	2	12	100
3	0	15	4	14	120
4	0	20	6	16	120
5	10	0	2	14	120
6	10	10	0	16	120
7	10	15	6	10	100
8	10	20	4	12	100
9	15	0	4	16	100
10	15	10	6	14	100
11	15	15	0	12	120
12	15	20	2	10	120
13	20	0	6	12	120
14	20	10	4	10	120
15	20	15	2	16	100
16	20	20	0	14	100

created for five parameters and 16 composite plates were created. Table 1 shows the parameters at various levels, and Table 2 shows the designed L16 orthogonal array (OA).

Testing

Tensile tests on the composite samples were carried out as per the ASTM D3039 (25 × 250 mm) using a universal testing machine at a load rate of 2 mm/min. Flexural tests in accordance with ASTM D790–10 (12.7 × 125 mm) were performed on the same equipment. Furthermore, the composite specimens were subjected to impact testing using an impact tester in accordance with ASTM D256–10 (64 × 12.7 mm). All the experiments were conducted in replicates of three, and the average results were reported.

To better understand the failure mechanism, micrographic images of composite sample after mechanical testing (tensile, flexural and impact test) were taken using SEM. SEM micrographs were taken through secondary electron mode, and the samples were sputtered coated with a very thin layer of gold. With respect to the dimension of the specimen, particularly in its shape, the working distance varied from 10 to 14 mm, and the voltage used was 15 kV.

Optimisation of results by GRA

Taguchi experimental design was employed to examine the impact of the control factors on product responses. The basic objective of the Taguchi approach is to generate excellent outcomes quickly and inexpensively. Taguchi's orthogonal array can be used to accomplish this. When using the Taguchi factorial experimental design, the total number of experimental runs is reduced. Taguchi experimentation technique can be used for systematic evaluation, analysis, and optimisation of numerous process factors in order to attain the best results. The product results are transformed into the signal-to-noise (S/N) ratio, which is used in this approach to determine the dominant variables. The S/N ratios were classified into the following levels based on the importance of the main feature that needed to be improved: (i) nominal is better (ii) smaller is better, and (iii) larger is better [42,43].

Signal to noise (S/N) ratio

The higher the S/N ratio characteristic was selected for the current research. The tensile, flexural, and impact strength of CGF and PJF based natural composites were evaluated using the larger-the-better phase throughout material characterisation. The S/N ratios of the selected method can be calculated using the following equation,

Table 3

Mechanical test results of CCF and PJF based hybrid composites.

Experiment No.	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (kJ/m ²)
1	14.53 ± 0.72	55.75 ± 2.78	6.11 ± 0.31
2	21.2 ± 1.06	62.42 ± 3.12	12.78 ± 0.64
3	25.2 ± 1.26	66.42 ± 3.32	16.78 ± 0.83
4	23.86 ± 1.19	65.08 ± 3.25	15.44 ± 0.77
5	22.53 ± 1.12	63.75 ± 3.18	14.11 ± 0.71
6	25.84 ± 1.29	66.59 ± 3.32	16.79 ± 0.84
7	30.53 ± 1.52	71.75 ± 3.58	22.11 ± 1.1
8	31.86 ± 1.59	73.08 ± 3.65	23.44 ± 1.17
9	23.86 ± 1.19	69.08 ± 3.45	15.44 ± 0.77
10	37.87 ± 1.89	75.75 ± 3.78	26.11 ± 1.3
11	22.53 ± 1.12	63.75 ± 3.18	14.11 ± 0.7
12	30.53 ± 1.52	66.89 ± 3.34	22.11 ± 1.1
13	25.42 ± 1.27	66.63 ± 3.33	16.81 ± 0.84
14	33.2 ± 1.66	74.42 ± 3.72	24.78 ± 1.24
15	34.98 ± 1.75	79.45 ± 3.97	29.12 ± 1.45
16	36.09 ± 1.8	78.46 ± 3.92	28.96 ± 1.45

$$\frac{S}{N} \text{ratio} = -10 \log_{10} \frac{1}{y} \sum_{a=1}^y \frac{1}{Z_{ab}^2} \quad (1)$$

Where y represents the number of replications, Z_{ab} is the product outcome and $a = 1, 2 \dots y$ & $b = 1, 2 \dots y$

Normalised s/n ratio

Normalisation or standardisation is a change made to a single data input to evenly scatter and level the data so that it can be further analysed. X_{ij} is standardised as Z_{ij} ($0 \leq Z_{ij} \leq 1$) by Eq. (2) to balance the impact of using multiple units and reduction unpredictability. Table 3 displays the results of the experiments. The S/N and normalised S/N ratios of the hybrid composites were represented in Table 4.

$$\text{Normalization } (X_{ij}) = \frac{x_{ab} - \min(x_{ab} = 1, 2, \dots, k)}{\max(x_{ab} = 1, 2, \dots, k) - \min(x_{ab} = 1, 2, \dots, k)} \quad (2)$$

Grey relational coefficients and its grades

Utilising normalised S/N ratio information, Eq. (3) produced the Grey relationship factor. The Grey relationship grade can be determined by using the Grey relationship factors, which is represented by Eq. (4).

$$\gamma(xa(y), xb(y)) = \frac{\Delta \min + \zeta \Delta \max}{\Delta_{ab}(y) + \zeta \Delta \max} \quad (3)$$

$$\bar{\gamma}_j = \frac{1}{k} \sum_{i=1}^m \gamma_{ij} \quad (4)$$

where $\bar{\gamma}_j$ is the GRG of j th experiment and k is a performance number. The Grey relational coefficient (GRC) and Grey relational grade (GRG) were then computed for all the experiments in the L16 orthogonal and are shown in Table 5. Integrating Taguchi techniques and Grey relationship analysis allowed the multi-response optimisation problem to be simplified to a single-response function problem.

Result and discussions

Tensile strength of the hybrid composites

The composite treated with 6 % NaOH having 15 wt.% CGF and 10 wt.% PJF fabricated at 4 MPa compression pressure and 100 °C temperature had the highest tensile strength of 37.85 MPa (10th trial). Tensile properties of CGF/PJF combinations improved significantly with CGF, PJF, and NaOH treatment, but decreased with compression pressure and temperature. Fig. 1 shows the tensile behaviour of CGF/PJF-based hybrid composites.

Table 4

S/N Ratio and Normalised S/N Ratio of the hybrid composites' mechanical strength (tensile, flexural and impact).

Experiment No.	S/N Ratio			Normalised S/N Ratio		
	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (KJ/m ²)	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (KJ/m ²)
1	23.25	34.93	15.72	0.000	0.000	0.000
2	26.53	35.91	22.13	0.415	0.330	0.474
3	28.03	36.45	24.50	0.605	0.512	0.649
4	27.56	36.27	23.77	0.545	0.452	0.596
5	27.06	36.09	22.99	0.482	0.392	0.538
6	28.25	36.47	24.50	0.633	0.520	0.650
7	29.70	37.12	26.89	0.816	0.739	0.826
8	30.07	37.28	27.40	0.863	0.793	0.864
9	27.56	36.79	23.77	0.545	0.627	0.596
10	31.57	37.59	28.34	1.000	0.898	0.933
11	27.06	36.09	22.99	0.482	0.392	0.538
12	29.70	36.51	26.89	0.816	0.533	0.826
13	28.11	36.47	24.51	0.615	0.522	0.650
14	30.42	37.43	27.88	0.908	0.846	0.900
15	30.88	38.00	29.28	0.966	1.000	1.000
16	31.15	37.89	29.24	0.999	0.987	0.977

Table 5

Grey relational coefficient and Grey relational grade of hybrid composites' mechanical strength (tensile, flexural and impact).

Experiment No.	Grey Relational Coefficients (GRC)			Grey Relational Grade (GRG)
	Tensile strength (MPa)	Flexural strength (MPa)	Impact strength (KJ/m ²)	
1	0.33	0.33	0.33	0.200
2	0.46	0.43	0.49	0.275
3	0.56	0.51	0.59	0.331
4	0.52	0.48	0.55	0.311
5	0.49	0.45	0.52	0.292
6	0.58	0.51	0.59	0.335
7	0.73	0.66	0.74	0.426
8	0.78	0.71	0.79	0.456
9	0.52	0.57	0.55	0.330
10	1.12	0.83	0.88	0.566
11	0.49	0.45	0.52	0.292
12	0.73	0.52	0.74	0.398
13	0.56	0.51	0.59	0.333
14	0.84	0.76	0.83	0.488
15	0.94	1.08	1.01	0.605
16	1.00	1.00	1.00	0.600

The tensile characteristics of the hybrid materials were greatly enhanced through the inclusion of the fibre and the NaOH processing. The combination of CGF 20 wt.%, PJF 20 wt.%, NaOH 6 %, pressure 14 MPa, and temperature 100 °C resulted in the best tensile strength (S/N ratio). The increased strength of the composites after chemical treatment was attributed to increased surface contact, which resulted in an improved interfacial bonding. The NaOH treatment removed the fibre's unwanted wax, oil-coating materials, and pectin, resulting in a clean surface [44,45]. This treatment also eliminated cavities and promoted surface homogeneity, which increased the stress-transfer potential of composite fibres. Furthermore, it was noted that the fibre impregnation occurs at higher alkaline concentration during chemical treatment, which damages and lowers the properties of the fibres.

Flexural strength of the hybrid composites

Fig. 2 shows the flexural characteristics of the CGF/PJF hybrids. As the proportion of fibre weight and the chemical process concentrations rose, the composites' flexural strength improved. The flexural properties of the composites decreased as the temperature increased. CGF and PJF reinforcement at 10 wt.% increased the flexural strength of CG/PJ composites. When GG fibre was added to the composite, the S/N ratio improved (36.80–37.58). Similarly, S/N ratio improved with PJ fibre addition (36.80 to 36.98), NaOH treatment (36.40 to 36.98), and

compression pressure (36.80 to 37.01). The best flexural results were obtained with a 20 wt.% CGF / 15 wt.% PJF / 4 % NaOH / 16 MPa compression pressure and 100 °C temperature combination. The flexural strength of hybrid composites decreased when reinforcement percentage increased to 15 wt.%, and this is mainly because of the uneven distribution of fibres or their agglomeration (or any additive) at a higher weight percentage [46,47]. Furthermore, as the weight percentage of fibre increases, the amount of resin material available for producing bonding decreases (i.e., lowered wetting), resulting in poor strength [19]. The addition of CJF was primarily responsible for the increased strength when compared to other parameters. In addition to fibre reinforcement, alkali treatment has a significant impact on composite flexural strength. Alkali treatment improved the length to diameter aspect ratio by decreasing the diameter of the fibre [48]. This improved the fibre and matrix interaction while increasing the effective contact area. However, higher concentrations damage and weaken the fibre surfaces, resulting in poor mechanical properties [48].

Impact strength of the hybrid composites

Addition of CGF and PJF and increment in NaOH concentration, compression pressure, and temperature improved the impact strength (S/N Ratio), which is portrayed in Fig. 3. The addition of CGF and PJF improved fibre/matrix interaction, which improved the impact characteristics of the hybrid composites. At 10 wt.%, CGF and PJF showed the greatest improvement in impact characteristics. It is primarily due to the improvement in the stress transfer ability of fibres. The addition of fibre reinforcement increased the load-carrying capacity of the matrix, enabling it to withstand deformation caused by impact loads and minimising the risk of cracking or failure. With the addition of CGF, the impact characteristics (S/N ratio) increased from 25.70 to 27.97 and the PJF increased from 25.94 to 26.24. The increase in strength due to fibre addition can be deduced from these findings. After a 2 % alkali treatment, the impact characteristics (S/N ratio) significantly improved (21.46–25.79).

The optimum impact properties were observed in a composite fabricated with 20 wt.% CGF / 15 wt.% PJF / 4 % NaOH / 16 MPa compressive pressure and 120 °C temperature. CGF had the greatest influence on impact characteristics, followed by PJF, NaOH, compression pressure, and temperature. By allowing more resin to flow through the fibres, compression pressure contributed to the improvement of fibre-matrix adhesion. Temperature increased the curing of composites during manufacturing; however, higher temperature increased the composite brittleness leading to poor strength [49,50]. Another important factor is the degradation of fibre at high temperature. Prolonged exposure might cause decomposition, below the usual lignocellulose

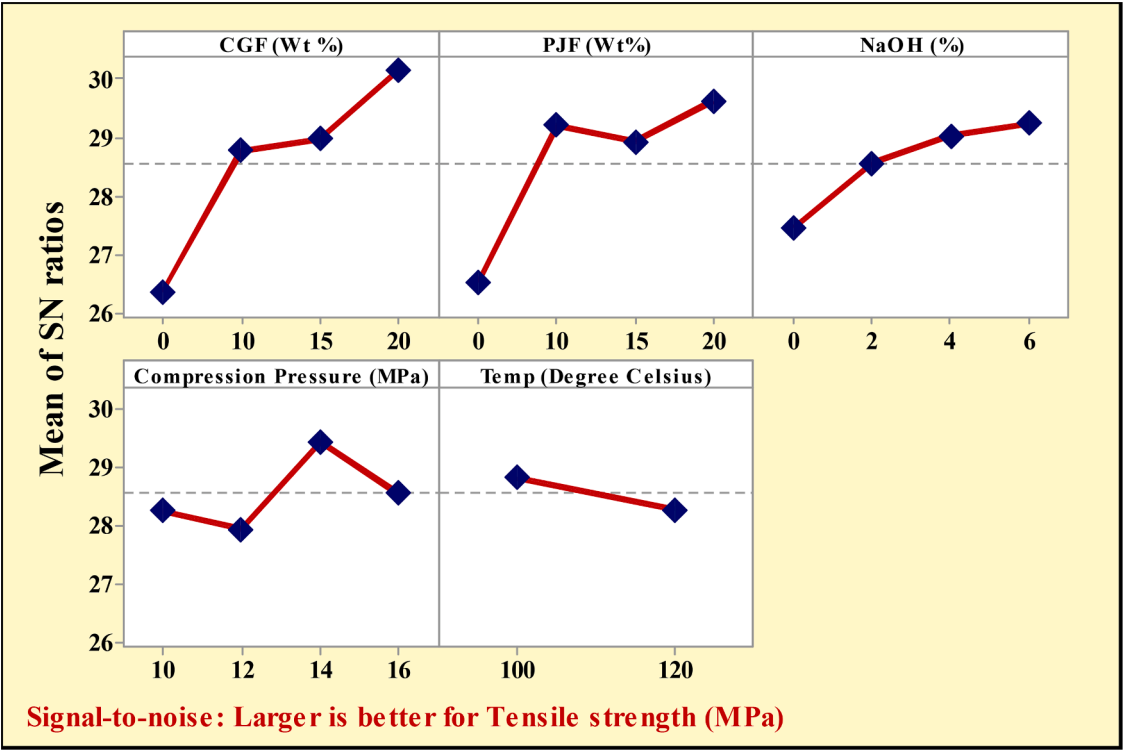


Fig. 1. Tensile strength of CGF/PJF hybrid composite with various influencing parameters.

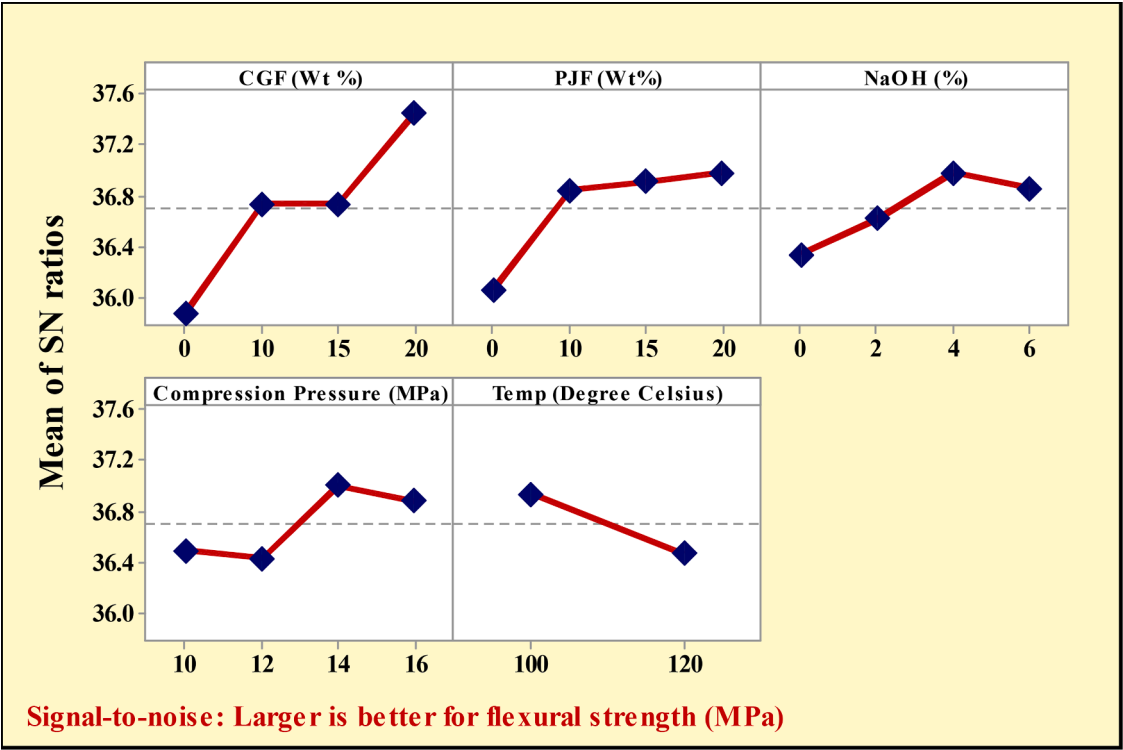


Fig. 2. Flexural strength of CGF/PJF hybrid composite with various influencing parameters.

decomposition temp of 200 °C [51].

Response of CGF and PJF- based hybrid composites

The levels A4, B4, C4, D3, and E1 have the highest Grey relational

grade value for factors such as CGF wt.%, PJF wt.%, NaOH concentration wt.%, compression moulding pressure, and temperature. This is demonstrated in Fig. 4. According to the study’s findings, the optimum properties for hybrid composites can be obtained at 20 wt.% CGF, 20 wt.% PJF, 6 % NaOH, 14 MPa pressure, and 100 °C temperature. The figure

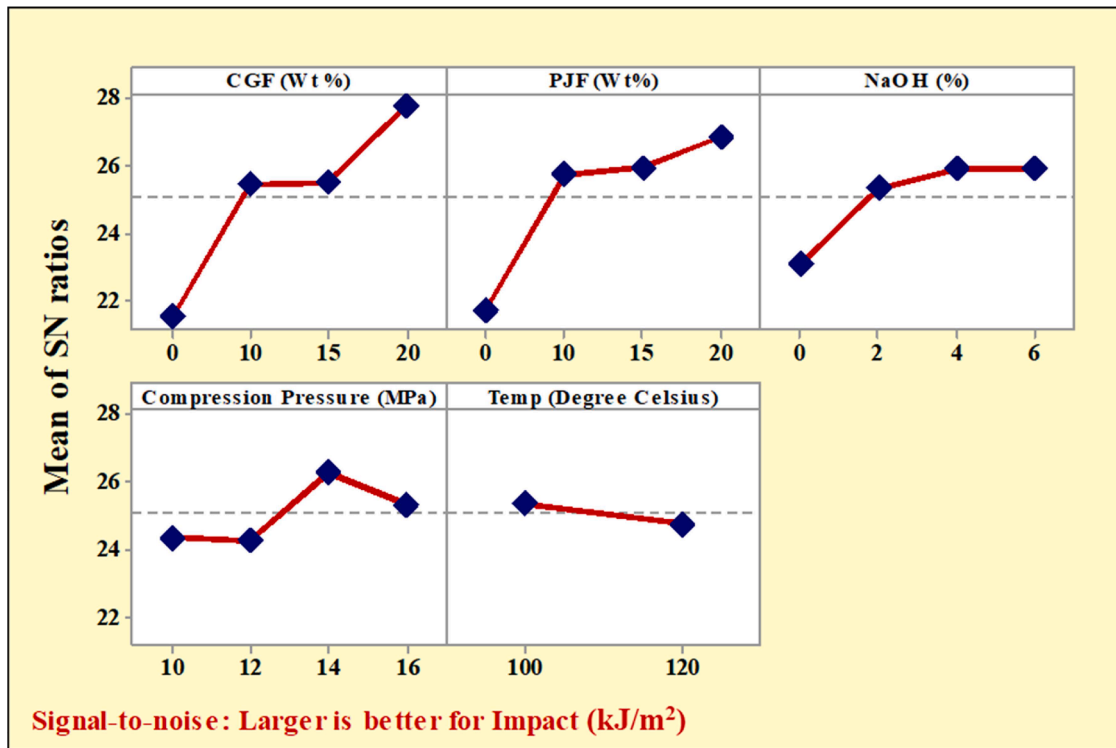


Fig. 3. Impact strength of CGF/PJF hybrid composite with various influencing parameters.

depicts the optimisation response of the CGF and PJF hybrid composites. The weight percentage of CGF and PJF showed positive effects on the composites at lower values. This demonstrates a strong interfacial connection between the fibre and matrix, providing efficient stress transmission. Tensile strength of a composite is primarily determined by the interfacial adhesion of the fibre and matrix. If the interfacial bonding was good, adding high strength fibres to a low strength matrix should result in a stronger composite. A 6 % alkali concentration yields the best results. The findings of this study showed that 6 % NaOH concentration improves surface adhesion by removing organic and inorganic impurities, resulting in a rough topographic surface [52].

The results clearly demonstrate that as the concentration of NaOH increases, its impact on fibres becomes more pronounced. Higher NaOH concentrations efficiently removed impurities such as waxes and oils from the fibres, thereby enhancing fibre purity. Conversely, when the NaOH concentration is low, the impurity removal process was inefficient, leading to suboptimal fibre-to-matrix interaction. It is crucial to note that elevated NaOH concentrations pose the risk of causing fibre damage and cellulose degradation. Thus, despite the potential for improved mechanical properties, optimising NaOH concentration requires a delicate balance between achieving desired modifications and mitigating the risk of fibre damage. In the current study, NaOH concentration was varied between 2 %, 4 %, and 6 %, with optimal results observed at 6 %. Further increments beyond this concentration are anticipated to potentially induce fibre damage and deterioration.

Another important factor influencing fibre adherence and endurance is compression temperature. This improves mechanical and morphological structure by increasing the polymerisation levels and the fibre crystalline structure [41]. The optimised temperature of 100 °C is significant in terms of thermally activating the matrix material and promoting intermolecular interactions. The cross-linking of the matrix was facilitated at this temperature, resulting in the development of a more robust and interconnected network within the composite. The compression temperature in the compression moulding process of natural fibre composites holds pivotal significance, with both high and low temperatures exerting distinct influences on the composite properties.

Elevated compression temperatures facilitate improved interaction between natural fibres and the polymer matrix by enhancing polymer flow and penetration into the fibre structure. However, excessively high temperatures may lead to thermal degradation of fibres, consequently diminishing their reinforcing capabilities. The experimental findings unveiled that with an increase in heating temperature (120 °C), there was a notable decrease in mechanical properties and the most desirable properties were observed at 100 °C. This observation led to the understanding that beyond 100 °C, the tested fibres in the current experienced damage, resulting in a diminished capacity to carry stress.

An optimum compression pressure to attain the enhanced strength was found to be 14 MPa. This pressure aided in the reduction of void spaces, the enhancement of fibre-matrix contact, and the removal of entrapped air and volatiles. The resulting densification improves mechanical properties. High compression pressures enhance fibre consolidation within the polymer matrix, leading to increased mechanical strength and reduced porosity. However, there is a risk of potential fibre damage at excessively high pressures. In the present study, when the fibre was subjected to high compressive pressure, the rate of the mechanical property degradation was quite noticeable (16 MPa). On the contrary, low compression pressures may result in incomplete fibre consolidation, compromising mechanical properties and fostering increased porosity. The optimisation of compression pressure is paramount for achieving a balance between enhanced fibre consolidation and preventing damage, ensuring optimal mechanical performance. This optimisation is crucial for controlling density, porosity, and promoting uniform distribution of fibres, contributing to the production of high-quality composites.

Furthermore, the combination of temperature and pressure is critical for achieving the desired physical and mechanical properties. As discussed, higher temperatures generally accelerate chemical reactions and increase the mobility of molecules, whereas pressure aids in the formation of denser and more compact structures. In the present investigation, interaction between temperature and pressure played a crucial role in developing the strength of composite materials. When temperature increases, polymer flow is enhanced, facilitating improved

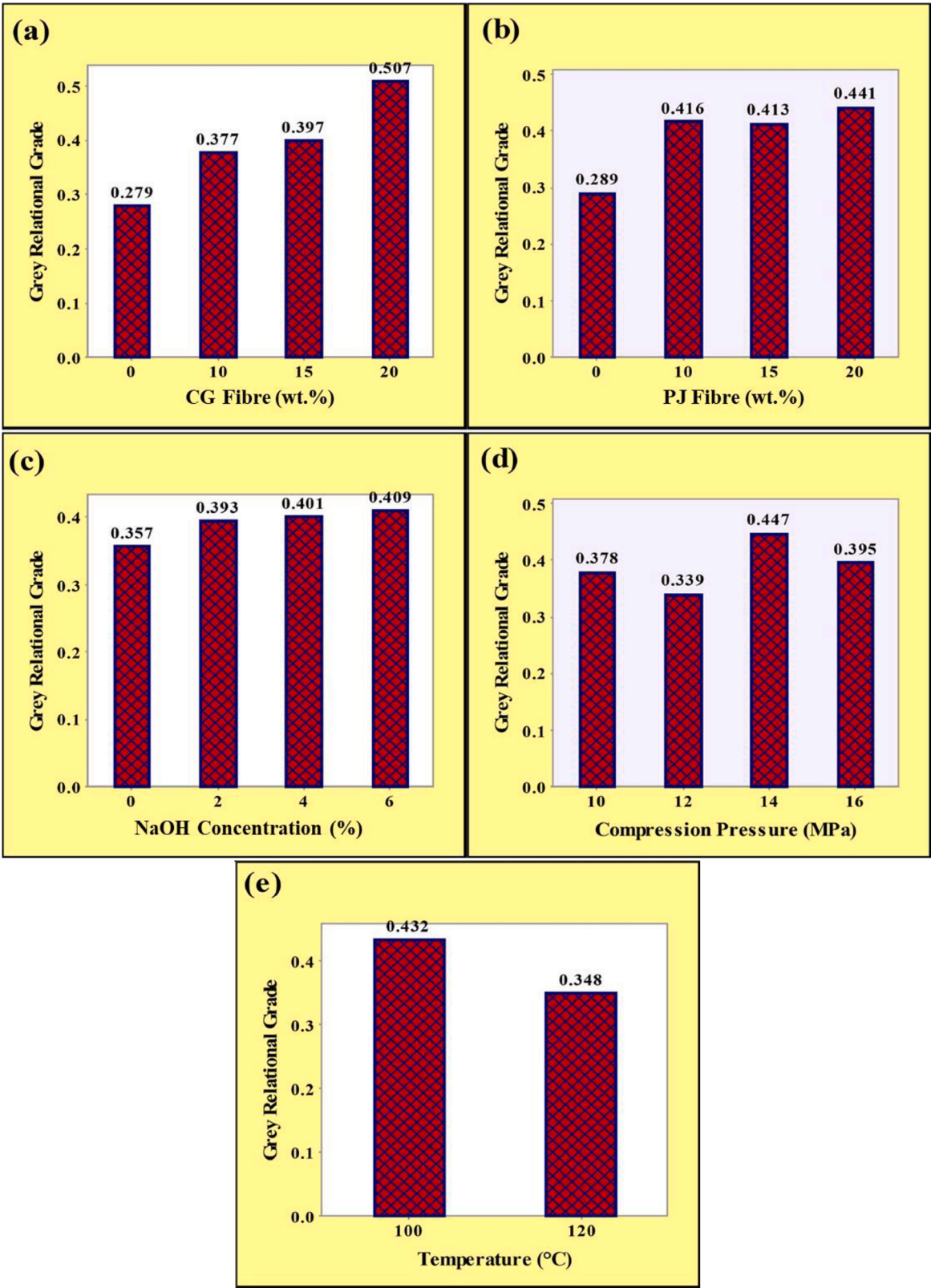


Fig. 4. The Grey relational grade value of CGF/PJF composites with varying influencing parameters.

impregnation and bonding between fibres and the polymer matrix. Simultaneously, pressure aided in compaction, ensuring close contact between fibres and the matrix. This combined effect contributed to the densification of the composite, reducing void spaces and promoting improved mechanical properties, including increased strength and load

carrying ability. Moreover, finding the optimal compression pressure and temperature not only influence mechanical properties but also play a vital role in energy efficiency, sustainability, and the overall consistency of the manufacturing process.

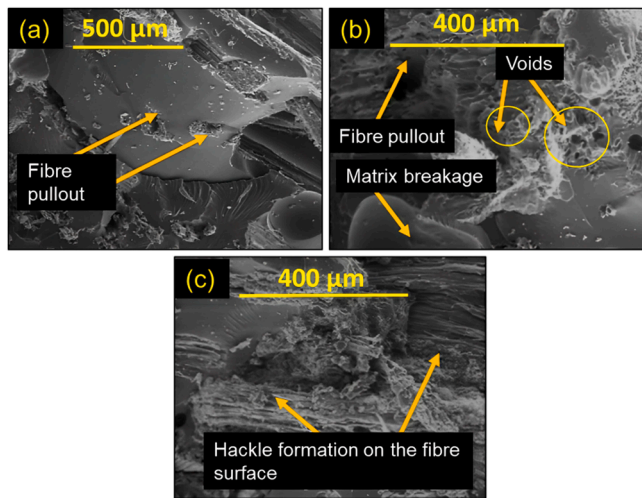


Fig. 5. Scanning electron micrographs of fractured CGF/PJF hybrid composites.

Microstructural analysis of CGF/PJF composites

The microstructures of the composites at the fractured surface were examined using SEM analysis. Fibre pull-out was observed on the tensile tested composites 10 wt.% CGF/10 wt.% PJF/2 % NaOH/14 MPa compression pressure/120 °C temperature (Fig. 5a). The combination of compression pressure and 120 °C temperature caused matrix fracture, fibre pull-out and void gaps in the composites (Fig. 5b). This developed lower fibre/matrix adherence due to the non-homogeneous mixing of fibres in the matrix. Hackle development in the fibre/matrix interface was seen in the composite with 15 wt.% CGF/10 wt.% PJF/6 % NaOH/14 MPa compression pressure and 100 °C temperature (Fig. 5c), indicating a ductile failure. The ductile failure proves the effectiveness of load carrying ability developed in the composite.

Conclusions

This investigation delved into the mechanical properties of epoxy hybrid composites made from the natural fibres extracted from *Calotropis gigantea* (CG) stem and *Prosopis juliflora* (PJ) bark. The optimisation of process parameters involved in composite fabrication was achieved through the Taguchi technique and Grey relational analysis. The identified optimal levels for CG and PJ-based hybrid composites included 20 wt.% CGF, 20 wt.% PJF, 6 % NaOH, 14 MPa pressure, and 100 °C temperature ('F' refers to fibres). The positive influence of the weight percentage of CG and PJ fibres highlighted a robust interfacial interaction between the fibres and the matrix, resulting in enhanced strength. The synergistic combination of *Calotropis gigantea* and *Prosopis juliflora* fibres underscored the value of these hybrid composites, particularly in applications where achieving optimal stress distribution and superior mechanical properties is paramount. The utilisation of a 6 % alkali (NaOH) concentration proved effective in improving surface adhesion by removing organic and inorganic contaminants. The optimised temperature of 100 °C played a significant role in matrix activation and cross-linking, contributing to the development of a more resilient composite network. Furthermore, an optimum pressure of 14 MPa was identified as striking the right balance between enhanced consolidation and preventing potential fibre damage. This investigation not only provided insights into the optimal process parameters for CG and PJ-based hybrid composites but also highlighted the critical role of hybridisation in achieving superior mechanical properties. The findings contribute valuable knowledge to the composite research field, guiding the development of hybrid composites for applications that demand high strength, stress distribution, and overall good mechanical

performance.

CRediT authorship contribution statement

Velmurugan Ganesan: Conceptualization, Formal analysis, Investigation, Visualization, Writing – original draft, Writing – review & editing. **Vigneshwaran Shanmugam:** Conceptualization, Formal analysis, Investigation, Visualization, Writing – review & editing. **Vasudevan Alagumalai:** Writing – review & editing. **Babu Kaliya-moorthy:** Writing – review & editing. **Oisik Das:** Formal analysis, Visualization, Writing – review & editing, Supervision. **Manjusri Misra:** Formal analysis, Visualization, Writing – review & editing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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