Statistical analysis of mechanical properties of wood dust filled Jute fiber based hybrid composites under cryogenic atmosphere using Grey-Taguchi method

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Abstract

At present, composites are being utilized on account of simplicity in manufacturing and their characteristics are low weight, non-abrasive, non-flammable and non-harmful. This article attempts to achieve good mechanical properties of the wood and woven jute based natural fiber-reinforced plastics. The required composite materials were fabricated by using a hand layup method. The fabricated laminates were immersed in liquid nitrogen at 77 K for cryogenic treatments. The following parameters are (i) wood density, (ii) wood weight ratio, (iii) woven jute type, (iv) number of jute layers, (v) cryogenic treatment durations and (vi) alkaline treatment durations, each at three different levels were picked for composite development by utilizing the Gray-Taguchi method. Mechanical testing of fabricated laminates was done as per ASTM standards to estimate compressive, double shear and hardness properties. Based on the L27(3^6) orthogonal array of the Grey Taguchi technique, 27 investigational trials were conducted. According to the Gray relation analysis, 300 kg m^{-3} of wood density with a weight ratio of 12%, three layers of 350 gsm woven jute, 60 min of cryogenic treatment and 4 h of alkaline treatments resulted in good mechanical strength of the composites. Based on the Analysis of Variance, wood density was identified as the most influencing parameter that contribute up to 35.65% to the improvement of the mechanical strength of hybrid composites. The morphological expositions of NaOH treated hybrid composites and degree of fibers’ dispersion into the matrix were discussed based on Optical and SEM images.

1. Introduction

Natural fiber composites were extensively recycled in a large number of industries such as aerospace, automobile, and many more. The usage of natural fibers in reinforced composites has increased owing to the severe government rules and rising ecological consciousness. The additional benefits of bio-based fibers except their ecological welfare are its easy availability with low cost and low density. Due to its low density, these fibers have high specific strength. Compared to inorganic fillers, natural fibers can be used more in plastic/fiber composites because of the softer and non-abrasive properties [1]. In accordance with basic investigation and suggestions, wood fibers have fascinated huge attention because of the properties which are comparable to those of polypropylene/glass fiber composites [2]. Due to its inexpensive, aesthetically attractive quality and great physical force, structural wood is the maximum preferred structural material. However, its main drawback is the weakening of physical and mechanical properties due to environmental variations that lead to bond failure [3].

Composites having two or more fillers enclosed in the same matrix are called hybrid composites. The common drawbacks of individual composites can be improved by hybridization and it results in better mechanical properties of natural fiber reinforced plastic composites [4]. The tensile strength is proven to increase by hybridization of short and long fibers because the short fibers minimize the voids in the composite.
Hybridization is claimed to play a vital role in improving the mechanical properties of glass/palm fiber waste sandwich composites [5].

Amongst the variety of natural fibers, jute is inexpensive, easily available and has higher strength and modulus than plastic. Gon et al found that the jute fiber is the best substitute for wood. Since, it is an important bast fiber comprised of bundled ultimate cells, each containing spirally oriented micro fibrils bound together [6]. The natural fibers have different properties because they have different chemical compositions of cellulose, hemicelluloses and lignin [7, 8]. It is very much essential that the fiber and matrix should have compatibility and affinity for better properties of the composites. Most of the natural fiber-reinforced plastic composites are thermally stable up to 240 °C and beyond this temperature, the fiber starts to degrade [9].

Lignocellulosic fibers like jute and sisal consist of –OH groups that can easily absorb moisture and quickly deteriorate the behavior of natural composites, particularly dimensional stability. Due to this polar group, the natural fibers do not efficiently adhere to non-polar matrices [10]. To overcome this complexity, natural fibers are chemically pre-treated. Chemically pre-treated surfaces reduce moisture absorption and increase tensile strength, and improve thermal stability by reducing the hemicelluloses, lignin and wax contents on the surface of fibers [11–13].

Among the various chemicals, sodium hydroxide (NaOH) is broadly used to alter the boundary between the dissimilar materials, for instance, raw fibers and thermoplastic or thermosetting polymer. NaOH eliminates the regular wax and shines from the cellulose fiber exterior and consequently activates the hydroxyl groups. Similarly, NaOH responds with neighboring –OH bonds of fibers and separates the cellulose, water and impurities [14].

Further improvement of the mechanical properties of fiber-reinforced composites is possible through cryogenic treatments. For instance, the materials used for aircraft structures should sustain its mechanical properties to extreme temperatures up to −200 °C [15]. Cryogenically treated plastics and composites become stronger, durable and have improved hardness and wear resistance [16]. Thus, the cryogenic treatment of composites has become an integral part of continuous research and development to improve the material behaviors in natural composites. Among the base materials, the most commonly used resins are unsaturated polyester (USP) because it is very economical, easy to use and has a virtuous blend of electrical, chemical and mechanical attributes. Mishra et al [17] stated that natural fiber reinforced polyester composites were used in the infrastructure area, mostly in the construction of low-cost houses. Also, they claimed that the polyester resin performed well in a cryogenic environment because it developed a very low residual interfacial strain after cryogenic treatment.

The Grey based Taguchi method has been used for several multi-response complications in the fabrication process and their parameters were effectively optimized [18, 19]. Taguchi parameter design is an efficient method to optimize the manufacturing processes and helps in achieving high-quality targets without increasing the cost. To study a number of variables in fewer experiments, an orthogonal array (OA) is used in this method [20] and the signal to noise ratio (S/N) is used to measure the effect of noise factors on the target characteristics [21]. The three general categories of the performance characteristics in the analysis of the S/N ratio are lower-the-better, higher-the-better, and nominal-the-better. Depending on the type of character being evaluated, the S/N ratio compacts the multiple data points within a trial [22].

Among the various kinds of literature, the authors did not see a study on the hybrid composites with so many processing parameters like the density of wood dust, gsm value of Woven jute, number of jute layers and alkaline and cryogenic treatment durations. The main objective of this research work is to fabricate, evaluate and optimize the mechanical properties of the hybrid natural composites with the parameters mentioned above. To accomplish this objective, the wood dust and jute fiber-based hybrid polyester composites were fabricated through a simple hand lay-up technique. The natural fibers such as wood dust and the jute fibers were treated in NaOH solution to improve its adhesion and reduce the moisture uptake. Later, the fabricated composites were immersed at 77 K in liquid nitrogen for various lengths of time and their mechanical properties were experimentally measured and optimized by using the Grey-Taguchi method.

2. Experimental works

2.1. Materials

Sawdust of Teak wood was collected from the wood industry and the woven jute mats were collected from the jute service center at the southern region of Tamilnadu, India. Both the reinforcements were sun-dried for 2 days to remove the moisture in it. The collected wood dust mass was an aggregate of mixed particles of different sizes and orderly sorted into three types based on the density ((large size) 200, (medium size) 250 and (fine size) 300 kg m−3). Whereas, the jute fibers were separated based on their gsm values ((low thick) 250, (medium thick) 300 and (high thick) 350 gram per square meter). Unsaturated polyester resin, a catalyst of methyl ethyl ketone
peroxide and accelerator of cobalt naphthenate were procured from GVR Enterprises, Madurai, Tamilnadu, India.

The raw wood dust and jute fibers were washed individually with 1 to 2% detergent solvents at 60 to 70 °C for 1 h to remove any impurities present in it, followed by rinsing with distilled water and finally dried in a vacuum oven at 70 °C for 1 h 30 min as per the procedure elaborated in [23]. The dried fibers were designated as untreated fibers. The fibers were first de-waxed by soaking in 2:1 mixtures of benzene and ethanol for 70 to 72 h at 50 °C, and then the fibers were thoroughly washed with distilled water and dried out under the Sun for 24 h. The de-waxed yarns were then plunged in a 5% NaOH solution for 2, 4 and 6 h at 30 °C. Finally, the fibers were thoroughly washed with distilled water and dried in air to obtain 5% alkali-treated fibers.

2.2. Fabrication of hybrid composites

First, a mold of size 300 × 300 × 3 mm made up of stainless steel was polished. The unsaturated polyester resin was added with 1 wt% of cobalt naphthenate and 1 wt% of methyl ethyl ketone peroxide and then it was thoroughly mixed. The treated wood dust along with the woven jute fibers were taken for composite fabrication via hand lay-up technique. Wood dust of different weight % was first dispersed in the prepared polyester resin by intense hand stirring using a glass rod. This matrix combination was sprayed onto the layers of fibers located in the mold. When the fiber mats were entirely moist through matrix combinations, the mold was bolted and dried in the open air at least for a day.

According to Taguchi design for 6 parameters each with 3 levels, L27 orthogonal array was selected and subsequently, 27 composite plates were fabricated for further investigation. The fabricated hybrid composite samples were kept in the desiccators to ensure no more absorption of moisture. The fabricated samples were then immersed in liquid nitrogen at 77 K for cryogenic treatment for different durations as per the design. After the cryogenic treatment, the composites were taken out and kept at room temperature. Tables 1 and 2 list the parameters along with their levels and L27 orthogonal array (OA) respectively.

3. Testing and techniques

3.1. Testing

The specimens were cut into the sizes, according to ASTM D-5379 (width of 20 mm and length of 76 mm), ASTM D-3410 (25.4 mm wide and 120 mm length) and ASTM E-10 (20 mm length and 20 mm width) to quantify the double shear, compressive and Brinell hardness properties of the composites. Equations (1) and (2) were used to calculate the values of double shear and hardness of the hybrid composites. The responses for the investigational sets are presented, as shown in table 3.

\[
\text{Double Shear} = \frac{P}{b \times t}
\]

(1)

Where P is applied load at the fracture point, b is the width of the specimen, t is the thickness of the specimen.

\[
\text{Hardness} = \frac{2P}{\pi D(D - \sqrt{D^2 - d^2})}
\]

(2)

Where P is applied load, D is Steel ball diameter and d is a depression diameter of the specimen.
3.2. Investigation of experimental outcomes by grey relational method

The Taguchi method of design of the experiment is an efficient method for methodical demonstration, investigation and optimization of various process parameters to achieve the best possible outcome. Using this technique, the investigational outcomes are reformed into signal-to-noise (S/N) ratio to identify the dominant parameters. Based on the conclusive factor for the prominent feature to be enhanced, the S/N ratio features were separated into the following phases: (i) larger-the-better, (ii) nominal-the-better and (iii) smaller-the-better.

3.2.1. Signals-to-noise ratio

Despite the category of the performance feature, the larger S/N ratio represents the better performance characteristic. Hence, the level with the highest S/N ratio is the optimal level of the parameter. Throughout the mechanical testing of natural fiber wood dust and jute composites, compressive, double shear and hardness strengths were measured by the phase of larger-the-better. Subsequently, signal to noise ratios of the eminence features (larger-is-better) are explicit as

\[ \text{Signal to noise ratio} = -10 \log \left( \frac{1}{k} \sum_{i=1}^{k} \frac{1}{X_{ab}} \right) \]

Where \( k \) is the replication number, \( X_{ab} \) is the experiential reaction and \( a = 1, 2 \ldots k \) & \( b = 1, 2 \ldots k \)

3.2.2. Normalized signal-to-noise ratio

A transformation execution on single data input to share out the data evenly and scale it into an acceptable range for further analysis is said to be Normalization. \( X_{ab} \) is standardized as \( Y_{ij} \) (0 \( \leq Y_{ij} \leq 1 \)) using equation (4) to set the right outcome of assumption for several parameters and to reduce the inconsistency. The eminent features were selected from this investigation based on larger-the-better compressive strength, double shear strength and hardness. The adoption of S/N ratio and normalized S/N ratio for the above characteristics are well suited.

\[ Y_{ij} = \frac{x_{ab} - \min(x_{ab} | a = 1, 2 \ldots k)}{\max(x_{ab} | a = 1, 2 \ldots k) - \min(x_{ab} | a = 1, 2 \ldots k)} \]

Table 2. L27 Combinatorial design (orthogonal array).

| Experiment no | Stages of parameters A | Stages of parameters B | Stages of parameters C | Stages of parameters D | Stages of parameters E | Stages of parameters F |
|---------------|------------------------|------------------------|------------------------|------------------------|------------------------|------------------------|
| 1             | 1                      | 1                      | 1                      | 1                      | 1                      | 1                      |
| 2             | 1                      | 1                      | 1                      | 2                      | 2                      | 2                      |
| 3             | 1                      | 1                      | 1                      | 3                      | 3                      | 3                      |
| 4             | 1                      | 2                      | 2                      | 2                      | 1                      | 1                      |
| 5             | 1                      | 2                      | 2                      | 2                      | 2                      | 2                      |
| 6             | 1                      | 2                      | 2                      | 3                      | 3                      | 3                      |
| 7             | 1                      | 3                      | 3                      | 3                      | 1                      | 1                      |
| 8             | 1                      | 3                      | 3                      | 3                      | 2                      | 2                      |
| 9             | 1                      | 3                      | 3                      | 3                      | 3                      | 3                      |
| 10            | 2                      | 1                      | 2                      | 3                      | 1                      | 2                      |
| 11            | 2                      | 1                      | 2                      | 3                      | 2                      | 3                      |
| 12            | 2                      | 1                      | 2                      | 3                      | 3                      | 1                      |
| 13            | 2                      | 2                      | 3                      | 1                      | 1                      | 2                      |
| 14            | 2                      | 2                      | 3                      | 1                      | 2                      | 3                      |
| 15            | 2                      | 2                      | 3                      | 1                      | 3                      | 1                      |
| 16            | 2                      | 3                      | 1                      | 2                      | 1                      | 2                      |
| 17            | 2                      | 3                      | 1                      | 2                      | 2                      | 3                      |
| 18            | 2                      | 3                      | 1                      | 2                      | 3                      | 1                      |
| 19            | 3                      | 1                      | 3                      | 2                      | 1                      | 3                      |
| 20            | 3                      | 1                      | 3                      | 2                      | 2                      | 1                      |
| 21            | 3                      | 1                      | 3                      | 2                      | 3                      | 2                      |
| 22            | 3                      | 2                      | 1                      | 3                      | 1                      | 3                      |
| 23            | 3                      | 2                      | 1                      | 3                      | 2                      | 1                      |
| 24            | 3                      | 2                      | 1                      | 3                      | 3                      | 2                      |
| 25            | 3                      | 3                      | 2                      | 1                      | 1                      | 3                      |
| 26            | 3                      | 3                      | 2                      | 1                      | 2                      | 1                      |
| 27            | 3                      | 3                      | 2                      | 1                      | 3                      | 2                      |

4
### 3.2.3. Grey relational coefficients

The Grey relational coefficient was computed using the normalized S/N ratio values and is given by equation (5).

\[
\gamma(x_a(y), x_b(y)) = \frac{\Delta_{\text{min}} + \zeta \Delta_{\text{max}}}{\Delta_{ab}(y) + \zeta \Delta_{\text{max}}}
\]

Where \( b = 1, 2, \ldots; y = 1, 2, \ldots, m; y \) is the investigational statistics numbers and \( m \) is the quantity of retorts; \( x_a(y) \) is the orientation series \( \{ x_a(y) = 1, y = 1, 2, \ldots, m \} \); \( x_b(y) \) is the exact relationship series; \( \Delta_{ab} = \{ \Delta x_a(y) - x_b(y) \} \) is the entire difference values between \( x_a(y) \) and \( x_b(y) \); \( \Delta_{\text{min}} = \min \{ x_a(y) - x_b(y) \} \) is the least value of \( x_b(y) \); \( \Delta_{\text{max}} = \max \{ x_a(y) - x_b(y) \} \) is the maximum value of \( x_b(y) \); and \( \zeta \) is the discriminate constant set in between the range of zero to one. In the current research, the process parameters have an equivalent weight and the discriminate constant was fixed by 0.5.

### 3.2.4. Grey relational grades

Every response of the Grey relation coefficients is transformed into the Grey relation grade using the equation (6), which is used to estimate the optimal level for each controllable factor. The results are shown in table 5.

\[
\tau_j = \frac{1}{k} \sum_{i=1}^{m} \gamma_{ij}
\]

Where \( \tau_j \) the Grey relation grade of jth experiment and \( k \) is a number of performance characteristics.

Subsequently, for all L_{27} (3^5) orthogonal experiments, the Grey relation coefficient and Grey relation grade were computed. Utilizing the relationship of the Taguchi techniques and Grey relational analysis, the multi-response optimization problem was changed to a single-response objective function. The overall Grey relation grade was obtained by the Grey-based Taguchi method and that is the only performance feature used subsequently.

| Trial number | Compressive strength (MPa) | Double shear strength (MPa) | Hardness (BHN) |
|--------------|-----------------------------|----------------------------|---------------|
| 1            | 34.60                       | 19.33                      | 21.13         |
| 2            | 39.50                       | 31.66                      | 30.94         |
| 3            | 32.72                       | 18.33                      | 20.76         |
| 4            | 39.52                       | 34.56                      | 30.94         |
| 5            | 41.72                       | 43.67                      | 39.89         |
| 6            | 39.12                       | 29.98                      | 30.94         |
| 7            | 39.62                       | 33.33                      | 33.45         |
| 8            | 41.61                       | 41.66                      | 31.34         |
| 9            | 38.93                       | 27.33                      | 35.78         |
| 10           | 41.61                       | 46.78                      | 37.89         |
| 11           | 40.62                       | 35.67                      | 32.75         |
| 12           | 36.82                       | 28.89                      | 25.78         |
| 13           | 42.69                       | 50.00                      | 37.23         |
| 14           | 41.58                       | 41.66                      | 32.76         |
| 15           | 38.39                       | 25.00                      | 26.52         |
| 16           | 40.44                       | 33.33                      | 31.82         |
| 17           | 40.61                       | 33.33                      | 31.82         |
| 18           | 36.88                       | 23.33                      | 25.91         |
| 19           | 41.70                       | 41.66                      | 36.12         |
| 20           | 41.72                       | 43.33                      | 37.23         |
| 21           | 41.94                       | 45.00                      | 34.81         |
| 22           | 43.15                       | 51.66                      | 35.93         |
| 23           | 43.69                       | 53.33                      | 48.12         |
| 24           | 44.15                       | 55.00                      | 50.19         |
| 25           | 40.83                       | 36.78                      | 32.75         |
| 26           | 40.07                       | 35.67                      | 35.89         |
| 27           | 41.02                       | 36.66                      | 32.76         |
4. Results and discussion

The optimum parameters such as compressive, double shear and hardness strength of wood and jute based hybrid polyester composites were examined by the Taguchi method and Grey relation investigation. Grey relation investigation and ANOVA reduced the optimal constraint sets and the maximum significant feature in the natural composite. The calculated S/N ratios and normalized S/N ratios for each eminent feature are given in Table 4. The Grey Relational Coefficients and Grey Relational Grades for 27 experiments are listed in Table 5.

From the response table of the Taguchi method, the average Grey relation grade was determined for each factor level. For instance, to calculate the Grey relation score of the wood density, the average of grey relational coefficient of level 1 (trials 1 to 9), level 2 (trials 10 to 18) and level 3 (trials 19 to 27) of column A was calculated. Similar calculations were carried out for the other levels of wood weight ratio, woven jute type, number of jute layer, cryogenic treatment minute and alkaline treatment hours and the values are presented in Table 6. The response of the Grey relation grade at various levels of the composites are shown in Figures 1(a)–(f). The figures demonstrate the optimum process control level produced by the maximum Grey relation grade.

4.1. Mechanical properties

The levels A3, B2, C3, D3, E2 and F2 have the maximum Grey relational grade value for the parameters such as wood density, wood weight ratio, woven jute type, number of jute layer, cryogenic treatment minute and alkaline treatment duration respectively. From the above investigation, the optimal values of the hybrid composites were wood density of 300 kg m$^{-3}$, wood weight ratio of 12%, woven jute type of 350 gsm, number of jute layers of 3, cryogenic treatment of 60 min and alkaline treatment of 4 h. It shows that the higher density of wood dust and thicker woven jute would increase the strength of composites. This is because the effects of voids are offset by the occurrence of thicker and high-density fiber materials and this finding is in accordance with the reports of author ku [24].

The strength of natural composites increased with an increase in the wood weight ratio up to 12%. Further increase in wood weight ratio resulted in poor bonding between the matrix and fiber. Facca and Andrzej achieved the same trends [25, 26]. The composites exhibited a positive result when parameters C and D were

| Table 4. Signal to Noise Ratio and Normalized Signal to Noise ratio values. |
|--------------------------|--------------------------|--------------------------|--------------------------|
| Exp. no | Compressive strength (MPa) | Double shear strength (MPa) | Hardness (BHN) | Compressive strength (MPa) | Double shear strength (MPa) | Hardness (BHN) |
| 1    | 30.78 | 25.72 | 26.50 | 0.185 | 0.049 | 0.021 |
| 2    | 31.93 | 30.01 | 29.81 | 0.628 | 0.497 | 0.452 |
| 3    | 30.30 | 25.26 | 26.34 | −0.001 | 0.000 | 0.001 |
| 4    | 31.94 | 30.77 | 29.81 | 0.630 | 0.577 | 0.452 |
| 5    | 32.41 | 32.80 | 32.02 | 0.811 | 0.790 | 0.740 |
| 6    | 31.85 | 29.54 | 29.81 | 0.596 | 0.448 | 0.452 |
| 7    | 31.96 | 30.46 | 30.49 | 0.638 | 0.544 | 0.541 |
| 8    | 32.38 | 32.39 | 29.92 | 0.802 | 0.747 | 0.467 |
| 9    | 31.81 | 28.73 | 31.07 | 0.579 | 0.364 | 0.617 |
| 10   | 32.38 | 33.40 | 31.57 | 0.802 | 0.852 | 0.682 |
| 11   | 32.18 | 31.05 | 30.30 | 0.721 | 0.606 | 0.517 |
| 12   | 31.32 | 29.22 | 28.23 | 0.393 | 0.414 | 0.246 |
| 13   | 32.61 | 33.98 | 31.42 | 0.887 | 0.913 | 0.662 |
| 14   | 32.38 | 32.39 | 30.31 | 0.799 | 0.747 | 0.517 |
| 15   | 31.69 | 27.96 | 28.47 | 0.533 | 0.283 | 0.278 |
| 16   | 32.14 | 30.46 | 30.05 | 0.706 | 0.544 | 0.484 |
| 17   | 32.17 | 30.46 | 30.05 | 0.721 | 0.544 | 0.484 |
| 18   | 31.34 | 27.36 | 28.27 | 0.399 | 0.220 | 0.252 |
| 19   | 32.40 | 32.39 | 31.16 | 0.809 | 0.747 | 0.628 |
| 20   | 32.41 | 32.74 | 31.42 | 0.811 | 0.783 | 0.662 |
| 21   | 32.45 | 33.06 | 30.83 | 0.828 | 0.817 | 0.586 |
| 22   | 32.70 | 34.26 | 31.11 | 0.923 | 0.943 | 0.622 |
| 23   | 32.81 | 34.54 | 33.65 | 0.965 | 0.972 | 0.953 |
| 24   | 32.90 | 34.81 | 34.01 | 1.000 | 1.000 | 1.000 |
| 25   | 32.22 | 34.31 | 30.30 | 0.739 | 0.634 | 0.517 |
| 26   | 32.06 | 31.03 | 31.10 | 0.676 | 0.606 | 0.621 |
| 27   | 32.26 | 31.28 | 30.31 | 0.754 | 0.631 | 0.517 |
increased for the entire chosen level due to increase in the contact area between the fiber and matrix as fiber content increased. Because of this reason, higher energy is needed to break the bonding between the interlaced fiber bundles [27].

4.1.1. Effect of cryogenic treatment
The parameter E2 (60 min) showed better results as compared to E1 and E3. Under cryogenic treatment, the residual stresses developed at the interface were compressive in nature and it helped to strengthen the adhesion between matrix and fiber [28, 29]. Beyond this duration, the strength of the composites started to decrease due to the development of very high thermal stress between the fiber and resin. It also led to high curing shrinkage of polyester resin due to the debonding phenomenon that promoted laminate failure [30].

4.1.2. Effect of NaOH treatment
According to the grey relational analysis, from the parameter level F1, F2 and F3, the highest mechanical strength was obtained by F2 (4 h). When the fiber was treated with NaOH solution, the natural and artificial impurities were removed and led to rough surface topography. It helps to improve the compatibility and adhesion of natural fibers to the polymer matrix. Hence, the degree of crystallinity and the degree of polymerization was increased because of the reduction in porosity content. It is visible in optical and SEM images and that
characteristics increased the mechanical and morphological properties of the hybrid composite significantly [31]. When the fiber was treated for more than 4 h, the concentration of NaOH was increased and it lowered the crystalline index of fiber. Thus, reduce the effectiveness of stress transfer at the fiber interface which leads to the decline of mechanical properties [32, 33].

4.2. Analysis of variance

The average Grey relational grade value can be used to formulate the ANOVA. The result of ANOVA is used to measure the influencing process parameters of wood and jute based hybrid composites. This is completed by separating the total variability of the Grey relational grades, which is measured by the sum of the squared deviations from the total mean of the Grey relational grade, into the contributions by each process parameter and the error. The importance of the process parameter change on the performance characteristics can be evaluated by the percentage contribution through the total sum of the squared deviations [34, 35]. The following equations (7), (8) and (9) can be used to calculate the percentage contributions of various parameters.

Total sum of the square deviations \( (SS_d) = \frac{F_1^2 + F_2^2 + F_3^2}{3} - \frac{1}{m} \sum_{i=1}^{m} y_i^2 \)  \hspace{1cm} (7)

Sum of square deviations \( (SS_T) = \sum_{i=1}^{m} y_i^2 - \frac{1}{m} \left[ \sum_{i=1}^{m} y_i \right]^2 \)  \hspace{1cm} (8)

Percentage of contribution \( (\rho) = \frac{SS_d}{SS_T} \)  \hspace{1cm} (9)

Where F denotes the process factor, and 1, 2, 3 are the corresponding levels, m is the number of experiments in the orthogonal array and \( y_i \) is the mean Grey relation grade.

The influence of every process parameter is specified in the ANOVA table (table 7) and it can be seen that wood density and wood weight ratio contributed higher by 35.65% and 23.22% respectively. On the other hand, woven jute type and cryogenic treatment have contributed by a meagre 1.07% and 7.34% respectively. However,
the contribution by alkaline treatment and the number of jute layers are 15.50% and 17.13% respectively and are the mid-range parameters. This recommends that the wood density and its weight ratios are key parameters in the wood dust and jute based natural fiber hybrid reinforced polyester composites.

### 4.3. Morphological analysis

#### 4.3.1. Optical microscopy

The degree of fiber scattering was investigated through optical microscopy (Olympus Gx71) of the cleaned surface of the composites. The microstructures were taken arbitrarily at 10 different areas covering the whole region of each of the composites. Figures 2(a) and (b) show the microstructures taken at two different areas in which the dispersing of wood and jute is seen as uniform. The photos show that the wood fibers are consistently spread to the woven jute mate and the two strands were particularly dissipated in the grid. These microstructures do not show any huge agglomeration of the filaments and brought about low porosity [38]. It was also confirmed through a Scanning Electron Microscope (SEM) Analysis.

#### 4.3.2. Scanning electron microscope

The microstructures of the composites at the fractured surface were further evaluated through SEM analysis (Carl Zeiss, Supra55). The SEM image strengthens the previous findings of low porosity content in the composites because of smooth fiber dispersion in the matrix. The openings in the surface that can be found in figure 3(a) is a result of pullouts of single fibers and fiber packs. Figure 3(b) shows a single fiber that has been pulled out during the fracture. The SEM analysis demonstrated that the compatibility among matrix and cellulosic fibers are good [36]; for example, the fibers are all around wetted by the matrix. Also, it demonstrated that the fibers are dispersed in the matrix with neither significant matrix rich regions nor accumulation of fibers [37].

### Table 7. ANOVA for the grey relational grade.

| Factors       | Parameters                        | Degree of freedom | Sum of squares | Mean Sum of squares | % of contributions |
|---------------|-----------------------------------|-------------------|---------------|--------------------|--------------------|
| A             | Wood density (kg m⁻³)             | 2                 | 0.005 68      | 0.002 84           | 35.65              |
| B             | Wood weight ratio (%)             | 2                 | 0.003 71      | 0.001 85           | 23.22              |
| C             | Woven jute-type (gsm)             | 2                 | 0.000 172     | 0.000 086          | 1.07               |
| D             | Number of jute layer’s (no)       | 2                 | 0.002 73      | 0.001 365          | 17.13              |
| E             | Cryogenic treatment (min)         | 2                 | 0.001 17      | 0.000 585          | 7.34               |
| F             | Alkaline treatment (h)            | 2                 | 0.002 47      | 0.001 235          | 15.50              |
| Error         |                                   | 0                 | 0             | 0                  | 0                  |
| Total         |                                   | 12                | 0.015 93      |                    | 100                |

**Figure 2.** Optical microscopy images (a) and (b) represents the cross-section of the fractured sample in two locations.
5. Conclusion

In this research, the mechanical properties of the wood dust and woven jute based polyester hybrid composites have been investigated and the process parameters were optimized by using the Taguchi method and the Grey relational analysis. The following conclusions are made:

- The recommended level of the controllable process parameters of wood dust and jute based hybrid composites are 300 kg m\(^{-3}\) of wood density with 12% weight ratios, 350 gsm of jute with 3 layers, 60 min of cryogenic and 4 h of alkaline treatments. This combination of various parameters resulted in hybrid composites with improved mechanical properties.
- Compared to 200 and 250 kg m\(^{-3}\), the 300 kg m\(^{-3}\) of wood density resulted in high strength of hybrid composites, due to the effect of voids offset by the presence of thicker and high-density fiber materials. The strength of natural composites increased with an increase in the weight ratio of wood up to 12%.
- The hybrid composites displayed an optimistic outcome when parameter C (gsm value of jute) and D (the number of jute layers) increased. Since the contact area between the fiber and matrix was improved with an increase in the fiber content. For this reason, more energy is needed to break the coupling between the interlaced fiber bundles.
- Under the cryogenic treatment, the residual stresses developed at the interface became compressive in nature and helped to better adhesion between matrix and fiber, but only up to 60 min of treatment duration.
- Up to 4 h of NaOH treatment helped to increase the mechanical strength through improving the linkage and miscibility of natural fibers to the polymer background and hence improving the degree of polymerization and the degree of crystallinity of hybrid composites.
- According to the result of statistical analysis (ANOVA), it was found that wood density is the most significant parameter that contributed 35.65% and the woven jute type was the least contributing factor only with 1.07%.
- The images of optical microscopy and SEM exhibited lower porous contents. This might result in higher degree of dispersion of wood particles with jute fiber as well as the matrix.

Declaration of interest

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