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World Scientific News

An International Scientific Journal

WSN 124(2) (2019) 155-170 EISSN 2392-2192

Optimization of Mechanical Characteristics of Rice Husk Particle Reinforced Polymer Composites Using Taguchi Experimental Technique

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ABSTRACT

In this study a successful fabrication of rice husk filled polymer composites with different filler content was possible by hand lay-up technique. An experimental approach was used to determine the tensile strength & flexural rigidity of rice husk filled polymer composites using Instron Model 1122 testing machine (Instron Corp. Canton, MA). At last, Optimization of mechanical properties of rice husk filled polymer composites was done using Taguchi Technique. The analysis of means (ANOM) was done to find out the optimal parameter level & analysis of variance (ANOVA) was employed to identify the level of importance on the parameters on each of the properties. In the present work the ANOM results showed that the combination of rice husk particle size of 150 μm with volume fraction of filler material (15%) with Epoxy as the matrix material is beneficial for maximizing the mechanical properties of rice husk particles reinforced polymer composite materials. From ANOVA results, it was found out that the polymer resin has major influence (80.344% & 84.17%) on maximizing tensile and flexural strength. Finally, the results revealed that using rice husk particles as reinforcement for polymer matrix could successfully develop beneficial composites and can be used for the mechanical applications.

***Keywords*:** Rice husk particles, Polymer resin, Taguchi design, Analysis of means (ANOM), Analysis of Variance (ANOVA), Signal to noise (S/N) ratio

(Received 26 February 2019; Accepted 13 March 2019; Date of Publication 14 March 2019)

1. INTRODUCTION

Natural fiber as a replacement to synthetic fibre in polymer matrix is the focus of many scientists and engineers. The reason for focus on natural fibre reinforced polymer matrix is because of its low cost, eco-friendly, low energy consumption, non abrasive nature, and good insulator of heat and sound. In recent years, major industries such as automotive, construction and packaging industries have shown enormous interest in the development of new bio composite materials and are currently engaged in searching for new and alternate products to synthetic fibre reinforced composites. For example, jute is a common reinforcement for composites in India. Jute fibers with polyester resins are used in buildings, elevators, pipes, and panels. Natural fiber composites can also be very cost effective material for application in building and construction areas (e.g. walls, ceiling, partition, window and door frames), storage devices (e.g. bio-gas container, post boxes, etc.), furniture (e.g. chair, table, tools, etc.), electronic devices (outer casting of mobile phones) and other miscellaneous applications (helmets, suitcases). Many authors have reviewed the properties of thermoset and thermoplastic composites with the application of natural fibers such as kenaf [1, 2], jute [3, 4], sisal [5, 6], bagasse [7], bamboo [8], pineapple [9] and groundnut shell [10] etc. The various mechanical properties such as tensile strength and flexural rigidity of natural fibre reinforced polymer composites were investigated by various authors like Sreekala et al. [11] found the significant decrease in the flexural strength was observed at the highest empty fruit bunch (EFB) fibre volume fraction of 100% which was due to the increased fibre – to – fibre interactions and dispersion problem which results in low mechanical properties of composite.

Nomenclatures	
F_{max}	Maximum (peak) load (N)
A_s	Cross sectional area (m^2)
P	Maximum load (N)
b	Width of the specimen (m)
t	Thickness of the specimen (m
L_s	Span length of the specimen (m)
$N_{Taguchi}$	Number of experiments to be conducted
NV	Number of parameters
L	Number of levels
S/N	Signal to Noise ratio
A	Particle Size
B	Rice husk
C	Polymer resin
N	Number of experiments
y	Response
n	Number of replications for each trial i.
SS_T	Total sum of square
SS_A	Sum of square for Particle size of rice husk
SS_B	Sum of square for Volume fraction of rice husk
SS_C	Sum of square for Polymer resin(C)
SS_e	Sum of square for error
V_e	Variance of error
N	Total no. of trial in orthogonal array
Greek Symbols	
σ_t	Tensile strength (N/m^2)
σ_f	Flexural strength (N/m^2)

η	Response value of S/N ratio
η_A	Response value of *S/N* ratio of factor A
η_1, η_2, η_3	Response value of *S/N* ratio of No.1, No.2, and No.3
η_i	Experimental result for i_{th} experiment
η_m	Over all mean of S/N ratio
η_{opt}	Predicted optimum response value of S/N ratio
η_{ijmax}	S/N ratio of optimum level *i*of factor *j*
k_1	Number of main design parameter
η_{obs}	Observed value of S/N ratio
υ	Degrees of freedom of *k1* factors
η_{ver}	Confirmatory test trial number
η_{eff}	Effective value of S/N ratio
Abbreviations	
ANOM	Analysis Of Means
ANOVA	Analysis Of Variance
OA	Orthogonal Array
RHPC	Rice Husk Polymer Composite
UTM	Universal Testing Machine
ASTM	American Society for Testing & materials
DOF	Degree Of Freedom
CI	Confidence Interval

Premlal et al. [12] used rice husk as organic filler in polypropylene and observed that these composites exhibit relatively lower yield strength, Young's modulus, flexural modulus and higher elongation at break as compared to those of talc filled composites. Yamamoto et al. [13] reported that the structure and shape of silica particles have significant effects on the mechanical properties such as fatigue resistance, tensile and fracture properties. Ibrahim [14] investigated the effects of reinforcing polymer with glass and graphite particles on enhancing their flexural properties. Shoemaker and Kackar [15] and Phadke and Dehnad [16] have subsequently applied the Taguchi method to design the products and process parameters. This inexpensive and easy-to operate experimental strategy based on Taguchi's parameter design has been adopted to study effect of various parameters and their interactions in a number of engineering processes

As per author's information, no investigation has been discussed in the literature on optimization of mechanical properties of rice husk particles reinforced polymer composite materials. Hence, an attempt has been made in this paper to optimize the mechanical properties of rice husk particles reinforced polymer composite materials using Taguchi technique.

2. MATERIALS & METHODS

2. 1. Polymer resins

In the present study three different polymer resins, namely, epoxy, vinyl ester and polyester were used as matrix materials. The epoxy of grade LY554 and hardener HY951 was used with the volume ratio of 10:1 supplied by Hindustan Ciba Geigy (India) Ltd. to prepare the composite specimens. The vinyl ester of grade GR 200-60 was used with hardener, catalyst and accelerator with 1.5 vol. % to prepare the composite specimens. The polyester of grade

PxGp 002 and the catalyst benzoyl peroxide with prescribed proportion was used to develop the composite specimens.

2. 2. Rice husk

In this work, rice husk, a by- products is chosen as the filler material mostly for its ecofriendly, available at low cost, non-toxic and basically it is considered as a waste product This can be utilized in many useful applications such as light weight concrete, an insulating material, fillers in plastics, building materials, panel boards, and activated carbon , electricity generation, husk-fueled steam engines etc. Rice husk can be used as an alternative biomass energy source against fossil fuels. The husk is collected from Shiva Shakti Rice Mill, Dhenkanal, Odisha, India, is used as a filler material in the polymer matrix composite.

2. 3. Composite Preparation

The low temperature curing epoxy resin and corresponding hardener were mixed in a ratio of 10:1 by volume as recommended. Rice husk particles with average size of 150 μm, 200 μm and 250 μm were reinforced in epoxy resin (density 1.1 gm/cc), vinyl ester and polyester separately with different volume fraction (15%, 30% and 45%) to prepare the composites. The Rice husk is dried before manufacturing in a vacuum oven for 24 hour at 80 ºC in order to remove moisture. Hand-lay-up technique was used for preparation of the specimen (sample) with different volume fractions. Silicon spray was used to facilitate easy removal of the composite from the mould after curing.

The cast of each composite was cured under a load of about 50 kg for 24 hours before it was removed from the mould. Then this cast was post cured in air for another 24 hours. The specimens were prepared having dimension of 165 mm × 19 mm with thickness of 3.2 mm for tensile test and 55 mm × 10 mm with thickness of 4 mm for flexural test.

2. 4. Determination of Tensile Strength

In this experiment the tensile test was determined according to ASTM D638-97 standard test method using Universal Testing Machine (UTM) i.e. Instron Model 1122 testing machine (Instron Corp., Conton,MA). The cross head speed for the test is maintained at 5mm/min and the test is repeated five times for each sample to get the mean value of the tensile strength. It was calculated according to the following equation:

$$\sigma_t = F_{\max} / A_s \qquad (1)$$

where: σ_t = Tensile strength (N/m^2), F_{max} is the maximum (peak) load (N), A_s is the cross sectional area (m^2).

Specimen dimension

Width of narrow section – 13 mm, Width overall – 19 mm, Gage length – 50 mm

Length of narrow section – 57 mm, Length overall – 165 mm, Radius of fillet – 76 mm

Distance between grips – 115 mm, Thickness – (3.2±0.4) mm

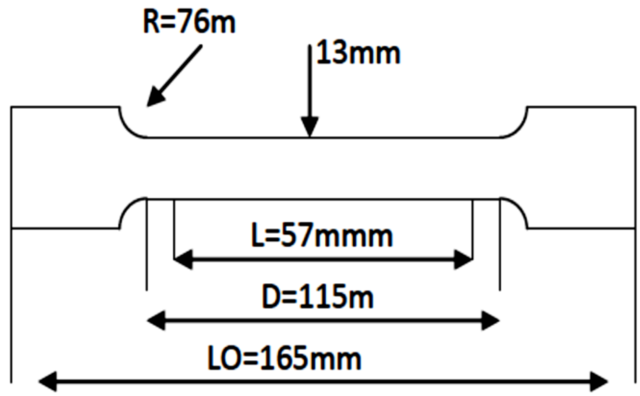

Figure 1. Standard Tensile Test Specimen

2. 5. Determination of Flexural strength

The three point bend testing method was used to determine the flexural strength according to ASTM D 790 – 97 using the same UTM machine i.e. Instron Model 1122 testing machine (Instron Corp. Canton, MA).The cross head speed for the test is maintained at 5mm/min and the test is repeated five times for each sample to get the mean value of the flexural strength. The test specimen for composite sample had nominal dimensions of 55×10×4 mm. The data recorded during the 3 – point bend test was used to evaluate the flexural strength (F.S) using the following equation

$$\sigma_f = 3PL_s / 2bt^2 \qquad (2)$$

where: σ_f is the flexural strength, P is the maximum load, b the width of the specimen, t is the thickness of the specimen and L is the span length of the specimen.

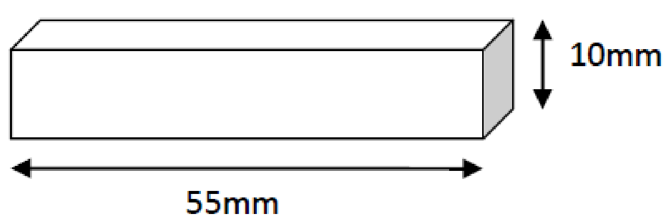

Figure 2. Standard flexural strength specimen

2. 6. Design of experiments via Taguchi method

Taguchi developed a method for designing experiments to investigate how different parameters affect the mean and variance of a process performance characteristic that defines how well the process is functioning. The experimental design proposed by Taguchi involves using orthogonal arrays to organize the parameters affecting the process and the levels at which they should be varied; it allows for the collection of the necessary data to determine which factors most affect product quality with a minimum amount of experimentation, thus saving time and resources. Analysis of variance (ANOVA) on the collected data from the Taguchi design of experiments can be used to select new parameter values to optimize the performance characteristic.

2. 7. Orthogonal Array Selection

Orthogonal arrays are special standard experimental design that requires only a small number of experimental trials to find the main factors effects on output. The following standard orthogonal arrays are commonly used to design experiments:

2-Level Arrays: L4, L8, L12, L16, L32
3-Level Arrays: L9, L18, L27
4-Level Arrays: L16, L32

Before selecting an orthogonal array, the minimum number of experiments to be conducted is to be fixed based on the formula below

$$N_{Taguchi} = 1 + NV(L-1) \qquad (3)$$

$N_{Taguchi}$ = Number of experiments to be conducted
NV = Number of parameters
L = Number of levels

In the present study, particle size, volume fraction of reinforcement material and matrix material are selected as the process parameters, which affect the mechanical properties, namely, tensile strength & flexural rigidity of rice husk particles reinforced polymer composite materials. There are three numbers of parameters. Each parameter was examined at three levels to study the non-linearity effect of the process parameters. Considering the equation- (3) the minimum no. of experiments is 7. But for 3-Level arrays L9 is the minimum one. Hence, minimum 9 experiments were required. It would require a total of 27 experiments to optimize the parameters. Taguchi experimental design of experiments suggests L9 orthogonal array, where 9 experiments are sufficient to optimize the parameters. In the present study, the selected process parameters and their levels are given in Table 1 and the three parameters at three levels each, *L9* (3^4) orthogonal array (OA) was used and accordingly nine rice husk polymer composites (RHPC) specimens were prepared as per the experimental layout plan (Table 2).

Table 1. Process parameters and their levels selected for the preparation of RHPC specimens.

Code	Parameters	Levels		
		1	2	3
A	Particle size (μm)	150	200	250
B	Rice husk (Vol, fraction,%)	15	30	45
C	Polymer resin	Epoxy	Vinyl ester	Polyester

Table 2. Experimental layout plan

Trial No.	Levels of parameter settings		
	Particle size (A)	Rice husk (B)	Polymer resin (C)
1	1	1	1
2	1	2	2
3	1	3	3
4	2	1	2
5	2	2	3
6	2	3	1
7	3	1	3
8	3	2	1
9	3	3	2

2. 8. S/N ratio

In Taguchi's design method the design parameters (factors that can be controlled by designers) and noise factors (factors that cannot be controlled by designers, such as environmental factors) are considered influential on the product quality. The Signal to Noise (S/N) ratio is used in this analysis which takes both the mean and the variability of the experimental result into account.

The S/N ratio depends on the quality characteristics of the product/process to be optimized. Usually, there are three categories of the performance characteristics in the analysis of the S/N ratio; that is, the lower-the-better, the higher-the-better, and the nominal-the-better. The S/N ratio for each response is computed differently based on the category of the performance characteristics and hence regardless of the category the larger S/N ratio corresponds to a better performance characteristic. In our present investigation, to obtain the optimal operating parameters, larger the better type category is used for tensile as well as flexural strength.

S/N ratio for larger-the-better type category is

$$\eta = -10\log_{10}\left[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^{2}}\right] \qquad (4)$$

where: y is the response and n is the number of replications for each trial i.

3. RESULTS & DISCUSSIONS

3. 1. Taguchi Experimental Calculation

Table 3 summarizes the experimental results of tensile strength & flexural strength of rice husk particles reinforced polymer composite materials. It is observed that the RHPC materials have tensile strength and flexural strength in the range 2.692 to 32.096 MPa and0.35 to 2.007MPa respectively. The specimens having epoxy matrix with particle size of 150µm and lowest volume fraction (15%) of filler material increases tensile as well as flexural strength. However, the specimens having polyester matrix composites with 150 µm particle size and highest volume fraction (45%) of particle shows increased tensile as well as flexural strength. On the other hand, vinyl ester matrix with highest range of particle size (250µm) and 45% filler material exhibit increased tensile strength where as flexural strength increased with medium particle size (200µm) and lowest volume fraction (15%).

Table 3. Experimental values of mechanical properties

Trial No.	Tensile strength (MPa)	Flexural strength(Mpa)
1	32.096	2.007
2	2.692	0.425
3	5.387	1.510
4	3.807	0.754
5	3.274	1.012
6	13.187	1.546
7	4.846	1.113
8	13.284	1.480
9	6.270	0.350

The computed values of S/N ratio (η) for each trial of L_9 OA for each of the thermal properties are demonstrated in Table 4.

Table 4. Computed values of S/N ratios for mechanical properties.

Trial No.	S/N ratio (dB) for mechanical properties	
	Tensile strength	Flexural strength
1	30.129	6.050
2	8.601	-7.430

3	14.622	3.580
4	11.612	-2.450
5	10.301	0.104
6	22.403	3.780
7	13.707	0.930
8	22.467	3.405
9	15.945	-9.120

3. 2. Analysis of mean value

The *S/N* ratio in the experiment can be calculated, which is then used to create a response table and plot a response diagram. The influence of the factors on the system can be calculated using the data in Table 6 to create the response table and plot the response diagram of the factors. The response value of the *S/N* ratio of each factor is calculated in Equation (5), and the calculated data are shown in Table 7.

$$\eta_{A_1} = \frac{1}{3}[\eta_1 + \eta_2 + \eta_3] \quad (5)$$

where: η_{A1} is the response value of *S/N* ratio of 1 level of factor A, η_1, η_2 and η_3 are the response value of *S/N* ratio of No. 1, No. 2, and No. 3 respectively.

The Table 5 and Table 6 represent the mean S/N ratio for tensile strength and flexural strength respectively.

Table 5. Mean S/N ratio for tensile strength.

Level	Particle Size (A)	Volume fraction (B)	Polymer resin (C)
1	17.785	18.482	25
2	14.770	13.789	12.052
3	17.373	17.658	12.880
Effect	3.015	4.693	12.948
Rank	3	2	1

Table 6. Mean S/N ratio for flexural strength

Level	Particle Size (A)	Volume fraction (B)	Polymer resin (C)
1	0.733	1.510	4.411
2	0.478	-1.307	-6.333
3	-1.595	-0.586	1.538
Effect	2.328	2.817	10.744
Rank	3	2	1

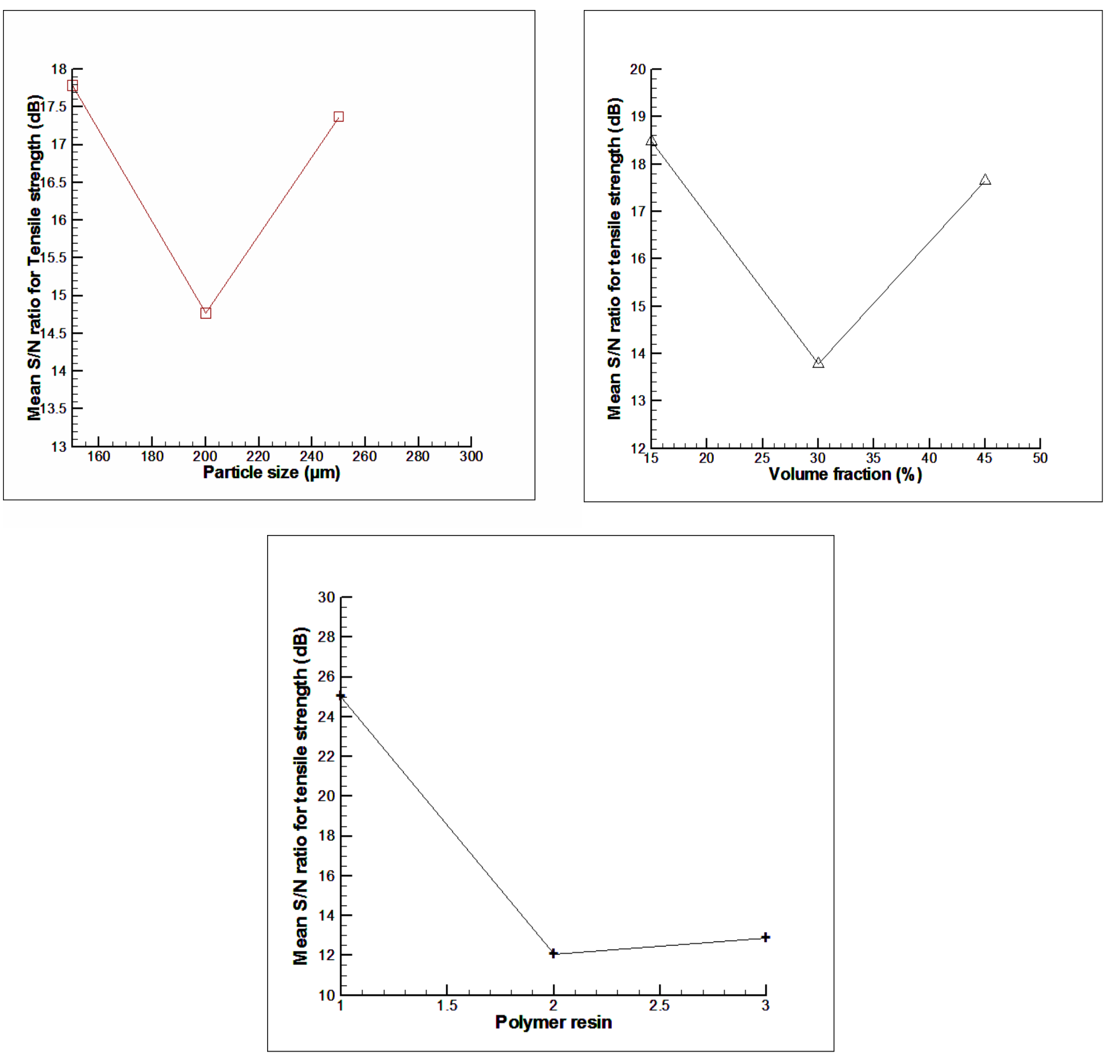

Figure 3. Response graph of Mean S/N ratio for tensile strength

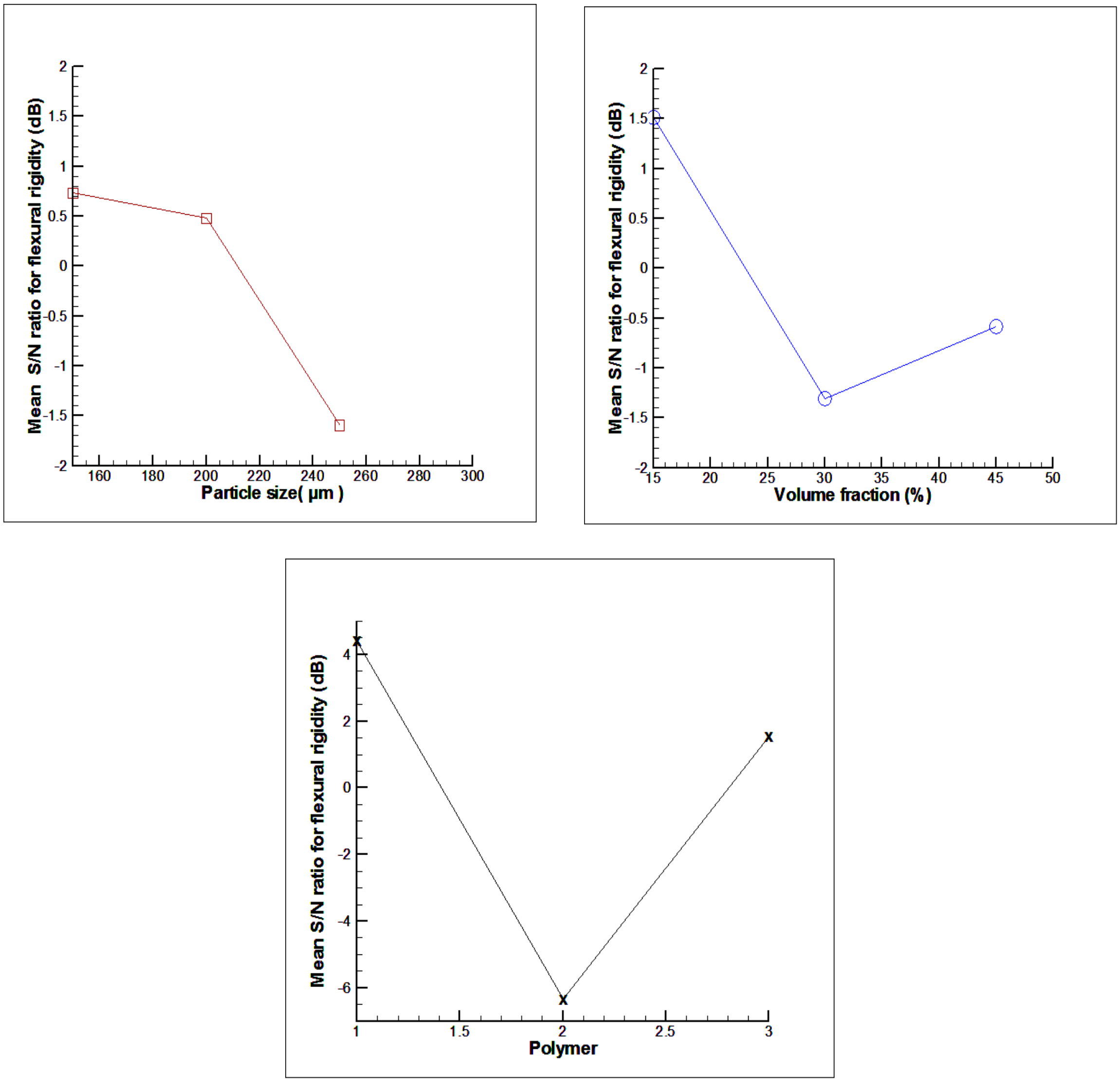

Figure 4. Response graph of Mean S/N ratio for flexural rigidity

The results of ANOM are represented in response graphs (Figures 5 & 6). The level of a process parameter with highest signal to noise (S/N) ratio value is the optimum level. As seen in Figure 5 & 6, the optimal combination of process parameter settings for maximizing the tensile and flexural strength of rice husk particles reinforced polymer composite is A_1, B_1 and C_1 i.e. the specimen having particle size of 150 μm with 15% volume fraction of rice husk particles using epoxy as the matrix material.

It has also presented the results of the experiments conducted to evaluate tensile as well as flexural strength of the polymer composites under study. Based on the response in Table 5 and 6, the degree of contribution of the parameters to the system can be calculated. The calculation method is to subtract the *S/N* maximum level of each factor from the *S/N* ratio of the minimum level of each factor. The contribution degree of parameters is higher for higher value of subtraction results. In the Table 5 and 6, C has highest subtraction value, B has medium value and A has lowest value. Hence the contribution parameters is C > B > A.

3. 3. Analysis of Variance

ANOVA ascertains the comparative importance of the parameters in terms of % contribution to the response. ANOVA is also required for determining the error variance for the effects and variance of prediction error. This is to be accomplished by separating the total variability of S/N ratio, which is measured by sum of squared deviations from the total mean S/N ratio, into contributions by each of the design parameters and the error. The % contribution designates the relative power of a parameter to diminish variation. For a parameter with a high % contribution with a small variation has a huge control on the response. The total sum of squares can be calculated using the relation

$$SS_T = \sum_{i=1}^{N} [\eta_i - \eta_m]^2 \quad \text{.........................(14)}$$

where:

N – Number of experiments

η_i – Experimental result for i_{th} experiment

η_m = Over all mean of S/N ratio

In the present experiment the overall mean for tensile as well as flexural strength are 16.643dB and -0.128 respectively.

The calculated data of ANOVA on S/N ratio for tensile and flexural strength are shown in Table 7 & 8.

Table 7. Summary of ANOVA on S/N ratio for tensile strength.

Source	Degrees of freedom (DOF)	Sum of Squares	Mean Square	% contribution
Particle size of Rice husk (A)	2	15.866	7.933	4.044
Volume fraction of Rice husk (B)	2	37.672	18.836	9.600
Polymer resin (C)	2	315.23	157.61	80.344
Error	2	23.588	11.794	6.012
Total	8	392.35	49.044	100

Table 8. Summary of ANOVA on S/N ratio for flexural strength.

Source	Degrees of freedom (DOF)	Sum of Squares	Mean Square	% contribution
Particle size of Rice husk (A)	2	9.782	4.891	4.440
Volume fraction of Rice husk (B)	2	12.850	6.425	5.832
Polymer resin (C)	2	185.522	92.761	84.171
Error	2	12.250	6.125	5.557
Total	8	220.411	27.551	100

From Table 7 & 8, it can be seen that the polymer resin has major influence (80.344% & 84.17%) on maximizing tensile and flexural strength. The volume fraction of rice husk has less effect (9.6 % for tensile strength and 5.832% for flexural strength), whereas the particle size does not have significant effect in maximizing tensile and flexural strength.

3. 4. Confirmation experiment

The confirmation experiment is the final test in the design of experiment process. The purpose of the confirmation experiment is to validate the conclusions drawn during the analysis phase. Once the optimal levels of the process parameters have been identified for each of the mechanical properties, the next step is to predict and verify the performance characteristic using the optimal level of design parameters. The predicted optimum value of S/N ratio (η_{opt}) of the response is determined by the formula

$$\eta_{opt} = \eta_m + \sum_{j=1}^{k_l}\left[(\eta_{ij})_{\max} - \eta_m\right] \qquad (15)$$

where: η_m is the overall mean of S/N ratio; η_{ijmax} is the S/N ratio of optimum level *i* of factor *j* and k_l is the number of main design parameter that affect the response.

In the present work, η_m = 16.643 dB for tensile strength and -0.128dB for flexural strength.

$\eta_{opt} = \eta_m + \sum_{j=1}^{k_l}\left[(\eta_{ij})_{\max} - \eta_m\right]$ = $A_1 + B_1 + C_1 - 2\eta_m$ = 27.981 dB for tensile strength and 6.910 dB for flexural strength and η_{obs} = 30.129 dB for tensile strength and 6.050 dB for flexural strength. $\eta_{obs} - \eta_{opt}$ = 2.148 dB & 0.860 dB for tensile and flexural strength respectively. In order to see the closeness of observed value of S/N ratio (η_{obs}) with that of the predicted value (η_{opt}), the confidence interval (*CI*) value of η_{opt} for the optimum parameter level combination at 95% confidence level is calculated.

$$CI = \sqrt{\left[F_{1,v_e} V_e \left(\frac{1}{\eta_{eff}} + \frac{1}{\eta_{ver}}\right)\right]} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (16)$$

In the present work,$F_{1,ve}$is the F value for 95% confidence interval,v_e is the degrees of freedom for error=2 ,$V_e$is the variance of error, $\eta_{eff} = \frac{N}{1+\upsilon}$

N = Total no. of trial in orthogonal array, υ = degrees of freedom of *k1* factors and η_{ver} = confirmatory test trial number Substituting all the values in equation (16), the results for CI= ±13.027 & ±9.387 for tensile and flexural strength respectively. Since, the error of prediction i.e. (η_{opt}–η_{obs}) are 2.148 dB & 0.860 dB for tensile and flexural strength respectively& which are within *CI* value, hence the optimum process parameter level combination and additive model for the variable effects are valid. The results of conformity tests are presented in Table 9. It is observed that the calculated values of prediction error of the mechanical properties are within the confidence limit, thus clearly indicating the adequacy of the additive of mechanical property models. The best combinations of process parameters for achieving maximum mechanical properties along with the corresponding optimal values of mechanical properties are exhibited in Table 9 & 10 respectively.

Table 9. Results of the confirmation test.

Performance measure	Tensile strength	Flexural strength
Levels(A,B,C)	1,1,1	1,1,1
S/N predicted (η_{opt}),dB	27.981	6.910
S/N observed (η_{obs}),dB	30.129	6.050
Prediction error, dB	2.148	0.860
Confidence interval (CI), dB	±13.027	±9.387

Table 10. Best combination values of the process parameters and the corresponding optimal values of thermal conductivity.

Mechanical property	Optimal process parameter settings			Optimal value (MPa)
	Particle size (µm)	Volume fraction (%)	Polymer resin	
Tensile strength	150	15	Epoxy	32.096
Flexural strength	150	15	Epoxy	2.007

4. CONCLUSION

This experimental study investigates the mechanical characteristics of rice husk filled polymer composites. An environmental waste like rice husk can be gainfully utilized for preparation of composites. Taguchi experimental method used here finds the way to optimize the mechanical properties of rice husk filled polymer composites. The ANOM results point out that the combination of lower particle size (150 μm) with lower volume fraction of filler material (15%) with Epoxy as the matrix material is beneficial for maximizing the mechanical properties of rice husk particles reinforced polymer composite materials. The ANOVA results revealed that the polymer resin has major contribution in maximizing the mechanical properties. The confirmation results indicate that the additive models are adequate for determining the optimum mechanical properties at 95% confidence interval.

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