

Experimental Study on Optimization of Thermal Properties of Natural Fibre Reinforcement Polymer Composites

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Abstract

In the present work, an experimental approach was used to find out the thermal conductivity of rice husk filled polymer composites using guarded heat flow meter method in accordance with ASTME-1530 standard. The result shows that the incorporation of rice husk reduces the thermal conductivity of polymer resin and improves its insulation capability. At last, an attempt has been made to optimize the thermal properties of rice husk reinforced polymer composites (RHPC) materials using Taguchi technique. In this work, the ANOM results showed that the combination of rice husk particle size of 250 μ m with volume fraction of filler material (45%) with vinyl ester as the matrix material was beneficial for minimizing the thermal properties of rice husk particles reinforced polymer composites and the degree of contribution of the parameters to the system were volume fraction > particle size > polymer resin. From ANOVA results, it was found out that the % of filler material has major influence (79.588%) on minimizing thermal conductivity and the particle size has less effect (1.126). Finally, the results revealed that using rice husk particles as reinforcement for polymer matrix could successfully develop beneficial composites and can be used for thermal applications.

Subject Areas

Rice Husk Particles, Polymer Resin, Thermal Properties, Taguchi Technique, Guarded Heat Flow Meter, ASTME-1530

Keywords

Mechanical Engineering

1. Introduction

In recent year, natural fibres such as rice husk, hemp, kenaf, jute, sisal, banana,

flax, oil palm etc. have been considerable demand due to their eco-friendly & renewable nature. These fibres received considerable attention as potential reinforcement in polymer composites. The attention towards utilization of natural fibres as a reinforcement of polymer based composites is mainly due to their various advantages over synthetic fibres such as low density, low cost, light weight, high strength to weight ratio, biodegrability, acceptable specific properties, better thermal and insulating properties, less wear and tear in processing, lower energy requirement processing, wide availability and relative non-abrassiveness over traditional reinforcing fibres like glass & carbon. Natural fibre reinforced polymer composites have been increasingly utilized in quite widespread applications. They are used in transportation such as automobiles, railway coaches, aerospace etc., military applications, building and construction industries (ceiling, panelling, and partition boards), packaging, consumer products etc. Natural fibres include those made from plants, animal and mineral sources.

The improvement of the insulating properties of composites can be determined by measuring their thermal properties *i.e.* the values of thermal conductivity. Generally thermal conductivity is a property which has ability to conduct heat of materials. It plays an important role in determining their heat conduction/insulation capability. Some studies have investigated the thermal conductivity of wood based composites, but few have explored the thermal conductivity of natural fibre and thermoplastic composites. Agrawal et al. [1] studied the thermal conductivity and thermal diffusivity of oil-palm-fibre-reinforced untreated and differently treated composites using transient plane source technique at room temperature. All the silane and alkali treatments of the fibres increased the thermal conductivity and thermal diffusivity of the composites in comparison with the acetylated composite. Saxena et al. [2] studied the variation of thermal conductivity and thermal diffusivity of banana fibre reinforced polyester composite caused by addition of glass fibre. They observed that the thermal conductivity of composites increased when compared to neat matrix. However, the thermal conductivity of the composites with increased percentage of glass fibre decreases in comparison to composite of pure banana. Mangal et al. [3] studied the effect of volume fraction of pine apple leaf fibre on thermal properties of the composite using transient plane source technique. Increasing the fibre content in the matrix decreases the thermal conductivity and thermal diffusivity of the pine apple leaf fibre reinforced composite which means that it could not provide the conductive path to the heat energy in the composite material. Li et al. [4] determined the various thermal properties, namely, thermal conductivity, thermal diffusivity and specific heat of flax fibre-HDPE bicomposites around 170°C - 200°C temperature range. The thermal conductivity, thermal diffusivity and specific heat found to be decreased with increase in fibre content; however, there was no appreciable change in thermal conductivity as well as thermal diffusivity in the specified temperature range. Mounika et al. [5] investigated the thermal conductivity characterization experimentally by a guarded heat flow meter method. The results showed that the thermal conductivity of the composite decreased with increase in fibre content and quite opposite trend was observed with respect to temperature. Raju et al. [6] aimed to elucidate the optimization of thermal properties such as thermal conductivity, linear thermal expansion and specific heat of ground nut shell particles reinforced polymer composite materials. Using ground nut shell particles as reinforcement for polymer matrix could successfully develop beneficial composites and can be used for thermal applications. Dedeepya et al. [7] measured mechanical properties of composite such as tensile strength, tensile modulus using universal testing machine and thermal conductivity of natural fibre typha angustifolia reinforced composite using Guarded hot plate apparatus. It was observed that mechanical properties increased as fibre content increased and thermal conductivity decreased as fibre content increased. AL-Shabander [8] presented the flexural properties and thermal conductivity of composites made from wood dust filler particles and epoxy resin. Experimental results showed that the flexural strength of the composites decreased with the increase of the filler particle content and the thermal conductivity of the composite decreased with increasing weight percentage of wood dust. Qi et al. [9] investigated the thermal conductivity of composite panels hot-pressed with varying proportions of sweet sorghum and high density poly ethylene (HDPE). The results showed that the thermal conductivity increases in a linear manner with temperature and density in a non linear manner with HDPE content. Taguchi and Konishi [10] advocated the use of orthogonal arrays and Taguchi [11] devised a new experiment design that applied signal-to-noise ratio with orthogonal arrays to the robust design of products and processes. In this procedure, the effect of a factor is measured by average results and therefore, the experimental results can be reproducible. Phadke [12], Wu and Moore [13] and others [14] [15] 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.

No investigation has been discussed in the literature on optimization of thermal properties of rice husk particles reinforced polymer composite materials. Hence, an attempt has been made in this paper to optimize the thermal 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. Epoxy resin is a polymer containing two or more epoxy groups and has high mechanical properties due to its low shrinkage and relatively unstressed structures. 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. A vinyl ester resin has excellent physical and mechanical properties and is familiar for its versatility as a composite matrix. The vinyl ester of grade GR 200-60 was used with hardener, catalyst and accelerator with 1.5-vol.% to prepare the composite specimens. Polyester resin is durable, comparatively inexpensive, superior corrosion resistance and little weight. 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 the present work the rice husk has been considered as reinforced filler because of its low cost, easy availability in nature, insulating characteristics and the most important factor is that it is an eco-friendly material. 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 μ m, 200 μ m and 250 μ m 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°C 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 diameter of 110 mm with thickness of 5 mm as shown in **Figure 1** for thermal conductivity test.

2.4. Experimental Procedure

A guarded heat flow meter has been developed for thermal conductivity measurements. This is achieved by using a thermal conductivity testing system Unitherm Model 2022 in accordance with ASTM-E-1530 standard shown in **Figure 2**. The sample and a heat flux transducer (HFT) shown in (**Figure 2**) are sandwiched between two flat plates controlled at different temperatures to produce a heat flow through the stack. A cylindrical guard surrounds the test stack and is maintained at a uniform mean temperature of the two plates, in order to minimize the lateral leak of heat. In Unitherm 2022, a sample of the material is held under a uniform compressive load between two polished surfaces, each controlled at a different temperature. The lower surface is part of a calibrated heat flow transducer. The heat flows from the upper surface, through the sample, to



Figure 1. Mould to cast specimens.



Figure 2. The Unitherm 2022.

the lower surface, establishing an axial temperature gradient in the stack. After reaching thermal equilibrium, the temperature difference across the sample is measured (**Figure 3**). The heat flux transducer measures the Q value. Taking these values with known cross sectional area and the sample thickness, the thermal conductivity is calculated using Equation (1).

$$Q = KA \left[\frac{T_1 - T_2}{L} \right] \tag{1}$$



Figure 3. Schematic model showing the testing arrangement.

where,

Q = Rate of heat flow [W], K = Thermal conductivity [W/m·K], A = Area of cross section of the specimen [m²], $T_1 \& T_2$ are the inlet and out let temperature of the specimen [K], L = Thickness of the specimen [m].

2.5. Design of Experiments via Taguchi Method

Although a large number of researchers have reported on properties, performance and on thermal characteristics of composites, neither the optimization of thermal processes nor the influence of process parameters has adequately been studied yet. Selecting the correct operating conditions is always a major concern as traditional experiment design would require many experimental runs to achieve satisfactory result. In any process, the desired testing parameters are either determined based on experience or by use of a handbook. It, however, does not provide optimal testing parameters for a particular situation. Thus, several mathematical models based on statistical regression techniques have been constructed to select the proper testing conditions. The number of runs required for full factorial design increases geometrically whereas fractional factorial design is efficient and significantly reduced the time. This method is popular because of its simplicity, but this very simplicity has led to unreliable results and inadequate conclusions. The fractional design might not contain the best design point. Moreover, the traditional multi-factorial experimental design is the "change-one-factor-at-a-time" method. Under this method only one factor is varied, while all the other factors are kept fixed at a specific set of conditions. To overcome these problems, Taguchi advocated the use of orthogonal arrays and applied signal-to-noise ratio with orthogonal arrays to the robust design of products and processes. He 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.

2.5.1. 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

$$N_{\text{Taguchi}} = 1 + NV(L-1) \tag{2}$$

 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 thermal properties, namely, thermal conductivity 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 (2) 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 (3⁴) orthogonal array (OA) was used and accordingly nine rice husk polymer composites (RHPC) specimens were prepared as per the experimental layout plan (**Table 2**).

2.5.2. S/N Ratio

The Signal to Noise (S/N) ratio is used in this analysis which takes both the

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

Code	Parameters —	Levels		
		1	2	3
А	Particle size (µm)	150	200	250
В	Rice husk (Vol., fraction, %)	15	30	45
С	Polymer resin	Epoxy	Vinyl ester	Polyester

	Levels of parameter settings			
Trial No.	A-Particle size (µm)	B-Rice husk (Vol. fraction%)	C-Polymer resin	
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	

Table 2. Experimental layout plan.

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. In our present investigation, to obtain the optimal oper-ating parameters, smaller the better type category is used for thermal conductivity.

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

$$\eta = -10\log_{10}\left[\frac{1}{n}\sum_{i=1}^{n}y_{i}^{2}\right]$$
(3)

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

3. Results & Discussions

The thermal conductivity obtained from the experimental study for the particulate filled epoxy composite with varied proportion of rice husk are shown in **Figure 4**. This figure shows that the incorporation of rice husk results in reduction of thermal conductivity of epoxy resin and there by improves its insulation capability. This reduction might have been attributed due to the core of the filler is porous and also the thermal conductivity of rice husk is less than the thermal conductivity of epoxy resin. Therefore, increasing the percentage of rice husk decreases the thermal conductivity of the composite. The shape of rice husk assumed to be spherical while in actual practice they are irregular shaped. Though the distribution of rice husk in the matrix body is assumed to be in an arranged manner, but it is actually dispersed in the resin almost randomly. With addition of 10 Vol.% and 45 Vol.%, of rice husk, the thermal conductivity of epoxy resin dropped by 8.4% and 58% for 200 µm size (rice husk) respectively. The values of thermal conductivities of composite with two components *i.e.* epoxy and rice husk are given in **Table 3**.



Figure 4. Thermal conductivity of epoxy composites as a function of filler content (Rice husk).

im, Rice husk).				
Sample	Particulate content (Vol.%)	Experimental value of thermal conductivity (W/m-K)	% reduction of thermal conductivity with respect to neat epoxy	
1	0 (neat epoxy)	0.310	0	
2	10	0.284	8.4	

0.131

Table 3. Measured thermal conductivity values of composites of varied composition (200 μ m, Rice husk).

3.1. Taguchi Experimental Calculation

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3

Table 4 summarizes the experimental results of thermal conductivity and S/N ratio of rice husk particles reinforced polymer composite materials. It is observed that the RHPC materials have thermal conductivities in the range 0.104 to 0.251 W/m-K. The specimens having epoxy matrix with particle size of 200 μ m and highest volume fraction (45%) of filler material decreases the thermal conductivity. However, the specimens having vinyl ester matrix with 250 μ m particle size and highest volume fraction (45%) of particle shows decreased thermal conductivity. On the other hand, polyester matrix composites with medium ranges of particle size (200 μ m) and 30% filler material exhibit lower thermal conductivity. Decrease in thermal conductivity for higher filler loading is due to the lower thermal conductivity of rice husk particle filler material.

3.2. Analysis of Mean Value

The S/N ratio in the experiment can be calculated, which is then used to create a

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Trial No.	Thermal conductivity (W/m-K)	S/N ratio (dB) for thermal conductivity
1	0.197	14.112
2	0.143	16.904
3	0.155	16.198
4	0.223	12.006
5	0.121	18.356
6	0.131	17.645
7	0.251	13.036
8	0.152	16.364
9	0.104	19.666

Table 4. Experimental values of Thermal conductivity & computed values of S/N ratios for Thermal conductivity.

response table and plot a response diagram. The influence of the factors on the system can be calculated using the data in **Table 4** 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 (4), and the calculated data are shown in **Table 5**.

$$\eta_{A_1} = \frac{1}{3} [\eta_1 + \eta_2 + \eta_3] \tag{4}$$

where, η_{A_1} is the response value of *S*/*N* ratio of 1 level of factor A, η_1 , η_2 and η_3 is the response value of *S*/*N* ratio of No.1, No.2, No.3 respectively.

The results of ANOM are represented in response graphs (**Figure 5**). The level of a process parameter with highest signal to noise (S/N) ratio value is the optimum level. As seen in **Figure 5**, the optimal combination of process parameter settings for minimizing the thermal conductivity of rice husk particles reinforced polymer composite is A3, B3 and C2 *i.e.* the specimen having particle size of 250 μ m with 45% volume fraction of rice husk particles using vinyl ester as the matrix material.

This chapter has presented the results of the experiments conducted to evaluate thermal conductivity of the polymer composites under study. Based on the response in **Table 5**, 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 **Table 5**, B has highest subtraction value, A has medium value and C has lowest value. Hence the contribution parameters is B > A > C.

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

Level	Particle size (A)	Volume fraction (B)	Polymer resin (C)
1	15.738	13.051	16.040
2	16.002	17.208	16.192
3	16.355	17.836	15.863
Effect	0.617	5.785	0.329
Rank	2	1	3

Table 5. Integration results of response in Taguchi experiments.



Figure 5. Response graph of Mean S/N ratio with respect to [(a)-Particle size, (b)-volume fraction of rice husk, (c)-Polymer resins *i.e.* Epoxy, Vinyl ester & Polyester] for thermal conductivity.

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

$$SS_{T} = \sum_{i=1}^{N} [\eta_{i} - \eta_{m}]^{2}$$
(5)

where,

N-Number of experiments;

 η_i -Experimental result for *i*th experiment;

 η_m = Over all mean of the experimental results.

In the present experiment the overall mean is 16.032 dB.

The calculated data of ANOVA on S/N ratio for thermal conductivity are shown in **Table 6**.

From **Table 6**, it can be seen that the % of filler material has major influence (79.588%) on minimizing thermal conductivity and the particle size has less effect (1.126), whereas the vinyl ester matrix material does not have significant effect in minimizing thermal conductivity.

4. Conclusion

In this study, a successful fabrication of rice husk filled polymer composites with different filler contents was possible by hand lay-up technique. From the experiment, it is found that the insulation properties of the polymer composites increase with increase of volume fraction of rice husk. Taguchi experimental method is used to find out the optimization of thermal properties of rice husk filled polymer composites. The analysis of means (ANOM) was done to find out the optimal parameter level. The analysis of variance (ANOVA) was employed to identify the level of importance on the parameters on each of the properties. The specimen having particle size of 250 μ m with 45% volume fraction of rice husk particles using vinyl ester as the matrix material has the lowest thermal conductivity and hence it is considered as the best insulating composite.

Table 6. Summary of ANOVA on S/N ratio for thermal conductivity.

Source	Degrees of freedom (DOF)	Sum of squares	Mean square	% contribution
Particle size of rice husk (A)	2	0.574	0.287	1.126
Volume fraction of rice husk (B)	2	40.57	20.285	79.588
Polymer resin (C)	2	0.162	0.081	0.318
Error	2	9.169	4.5845	18.968
Total	8	50.975	6.372	100

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