FABRICATION OF ARECA NUT FIBRE-RUBBER COMPOSITES USING TAGUCHI OPTIMIZATION

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Abstract

In the present work, Taguchi method; a Design of Experiment (DOE) technique was used to optimize the process parameters to prepare the areca nut fibre - natural rubber composite possessing desired qualities, using minimum of time and resources. The tools and techniques such as, orthogonal array, signal-to-noise ratio (S/N), and the optimum condition were employed in Taguchi method to study the process parameters of the areca nut fibre - natural rubber composite. Three factors, namely fibre treatment type, fibre length and fibre loading (that is, loading of fiber) were considered as the process parameters. Three levels for each parameter were used. Accordingly, a suitable orthogonal array L_9 (3³) was selected and experiments were conducted. After conducting the experiments the mechanical properties (hardness, and tear strength) of the prepared areca nut fibre - natural rubber composites were measured and (S/N) ratios were calculated. With the help of graphs, optimum parameter values were obtained and the confirmation experiments were carried out, and satisfactory agreement was obtained.

Keywords; Taguchi method, orthogonal array, signal-to-noise ratio, optimum condition

Introduction

A composite material system is composed of two or more physically distinct phases whose combination produces aggregate properties that are different from those of its constituents. Composites can be very important because of its strong and stiff, yet very light in weight, so ratios of strength to weight and stiffness to weight are several times stronger than steel or aluminum and also possible to achieve combinations of properties not attainable with metals, ceramics, or polymers alone.

Natural rubber (NR) is one of the main elastomers and widely used to prepare many rubber compounding products. NR is frequently reinforced by assimilation of the filler to improve its mechanical properties like; tensile strength, modulus, tear strength, elongation at break, hardness, rebound resilience and abrasion resistance (Khalil *et al.*, 2014). For this purpose, fillers are commonly used for rubber. Effectiveness of the reinforcing filler depends on numerous factors such as particle size, surface area and shape of filler. Nowadays, there has been growing interest in the use of industrial and agriculture waste such as areca nut fibre as fillers for rubber and their blends. The benefits of these fillers include low cost, easy availability and protection to our environment.

In the recent years, Taguchi method is a statistical method developed by Genichi Taguchi. Initially it was developed for improving the quality of goods manufactured (manufacturing process development), later its application was expanded to many other fields in Engineering, such as Biotechnology etc. Professional statisticians have acknowledged Taguchi's efforts especially in the development of designs for studying variation. Success in achieving the desired results involves a careful selection of process parameters and bifurcating them into control and noise factors. Selection of control factors must be made such that it nullifies the effect of noise factors. Taguchi method involves identification of proper control factors to obtain the optimum results of the process (Srinivas and Venkatesh, 2012). Orthogonal Arrays (OA) are used to conduct a set of

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experiments. Results of these experiments are used to analyze the data and predict the quality of components produced (Rahul *et al.*, 2014).

Process Optimization

Process optimization can be defined as the method of finding the conditions that will give the maximum or minimum value desired of a response.

Taguchi method minimizes the effect of uncontrollable or noise factors. It determines the optimum combination of controllable factors that will give the best value of the desired response. It is a multi-response optimization where a balance is to be achieved between a number of desired responses. Taguchi experimental design is one of the most commonly used techniques of process optimization. Taguchi method is a robust design method, because it reduces the variation of the quality of product by making the process less sensitive to the noise. The fundamental principle of robust design is to improve the quality of a product by minimizing the effect of the causes of variation without eliminating the causes.

Approach to Product/Process Development

Many methods have been developed and implemented over the years to optimize the manufacturing processes. Some of the widely used approaches are as given below:

- 1. Best-guess" experiments
- 2. One-factor-at-a-time (OFAT) experiments
- 3. Statistically designed experiments (Taguchi Method belongs to this method.)

In the present work, Taguchi method was used where the design parameters change simultaneously. It uses orthogonal array (OA) to reduce the number of experiments to run. Calculation of the signal-to- noise ratio (S/N) is done to predict the optimum values of input parameters to achieve the target quality of the product.

Orthogonal Array

There are different orthogonal arrays, *i.e.*, the OA shown on the right is called $L_9(3^3)$ OA. The advantage of using OA is that it reduces the total number of experiments to be carried out: *e.g.*, for the 3 factors & 3 levels case (*i.e.*, each factor can haves three values), all possible combinations of factor levels requires $3^3 = 27$ experiments; but using $L_9(3^3)$ OA requires only 9 experiments. Columns of the array are mutually *orthogonal*. Its means that for any pair of columns, all combinations of factor levels occur and they occur equal number of times. This is called the balancing property and it implies orthogonality. The number of *columns* of an array represents the maximum *number* of *factors* that can be studied using that array. Each number under a column is a level of the factor represented by the column. The number of *rows* of an orthogonal array represents the *number of experiments*.

Experiment		Column	
Number	1	2	3
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 1. L₉ (3³) Orthogonal Array

Steps Involved in Taguchi Method

The use of Taguchi's parameter design involves the following steps;

- 1. Deciding the important process *parameters* and their *levels*, response parameter and its characteristics.
- 2. Selecting the appropriate OA and assigning the parameters to its various columns.
- 3. Conducting experiments for the levels given in each row, random order, and noting down the value of the response parameters. Each experiment is run three times.
- 4. Studying factors effects and finding out the optimum combination of the parameters. Calculating to predict the best value of the response characteristic.
- 5. Calculating the range within which the experimental value should lie and conducting confirmation experiment
- 6. Performing analysis of variance (ANOVA) to find out the significance of the various factors and their relative contribution.

Materials and Methods

The Taguchi method is well known by simplification of experimental plan and feasibility of study of interaction between various parameters. In this method a less number of experiments are carried out, hence time and cost are reduced considerably. Main effect analysis is performed based on the average output of the quality characteristic at each parameter. Using the main effect and S/N ratio a prediction of the best combination of optimum parameters can be calculated (YathiAjay *et al.*, 2015).

Design of Experiment (Taguchi Methodology)

The important process *parameters* (*factors*) and their *levels*, response parameter (hardness and tear strength in our case) (see below) and its characteristics was decided. Then an appropriate orthogonal array (OA) was chosen and the selected parameters and their levels were assigned to it. And then, each experiment was done according to the set of parameter levels in each row of the OA, in random order of rows. Each experiment was repeated three times and the values of the response parameters were recorded. The effect of factors on each level was calculated and the optimum combination of the parameters was chosen graphically. To do this, the chosen type of signal-to-noise ratio was calculated (the larger the better on our case) (see below). The best value of the predicted response characteristic was calculated and validated by running the experiment with the calculated optimum combination of input parameters.

Response Names ; Hardness (IRHD) and Tear Strength (kN/m)

Response Types ; Larger-the-better , SN_L= -10log [$\frac{1}{n}\sum_{i=1}^{n}\frac{1}{\nu_i^2}$]

Selecting of processing parameters

Three processing parameters (or) factors, each with three levels were chosen. (The nonlinear behavior of the response characteristic can only be studied if more than two levels of a parameter were used.)

Factor one is the type of fibre (its three levels are: untreated, alkali treatment and potassium permanganate treatment). Choosing these processing types was based on the more reactive groups on the areca fiber surface, and effective areca fiber surface area for good adhesion with the natural rubber matrix. Furthermore, chemically treated areca fiber surface became more hydrophobic and there is improvement in surface characteristics such as wetting, adhesion and porosity of areca fibers, which improve interfacial adhesion between the treated areca fiber surface and the natural rubber matrix.

Factor two is the fibre length. It was also one of the parameters chosen due to its influence on the strength of the composite (20 mm is reported to be the upper limit (Rameez *et al.*, 2016)). The three levels are: 5, 10 and 15 mm fibre lengths.

Finally, factor three is the fibre loading. It also has effects on the strength of a composite (Rameez *et al.*, 2016). The three levels are: 5, 10 and 15 % by loading. Thus three levels are selected for each parameter (Table 2). Nine experiments have been done with different combinations of levels of parameters according to L₉ orthogonal array was carried out (Table 3).

Table 2	The Parameters for	• Three Levels of Selected I	Factors

Factors	Level -1	Level-2	Level-3
Fibre treatment type	Untreated	Alkali treated	KMnO ₄ treated
Fibre length (mm)	5	10	15
Fibre loading (%)	5	10	15

Table 3 The Combination of Different Level	s of Parameters According to L9 Orthogonal
Arrav	

Experiment		Column	
No.	Fibre treatment type	Fibre length (mm)	Fibre loading (%)
1	Untreated	5	5
2	Untreated	10	10
3	Untreated	15	15
4	NaOH	5	10
5	NaOH	10	15
6	NaOH	15	5
7	$KMnO_4$	5	15
8	KMnO ₄	10	5
9	KMnO ₄	15	10

Synthesis of Natural Rubber Composites Reinforced by Areca Nut Fibre

Natural rubber smoked sheets for the experiments were procured from Myanmar Gone Yee Rubber Plantation, Bago Region. The areca nut fibre-rubber composites were prepared and their mechanical characteristics were recorded at Rubber Research and Development Centre in Yangon.



Figure 1 Natural rubber

Materials

Natural rubber smoked sheet grade 1, zinc oxide, stearic acid, mecrapto benzothiazole, oil, antioxidant, sulphur, untreated, alkali treated and potassium permanganate treated areca nut fibre.

Procedure

Natural rubber smoked sheet Figure 1was first rolled at 60°C for 5 minutes on a Two Roll Mill to break out the fibrous bond of rubber polymer chain. This step is called mastication. Then mercapto benzothiazole (MBT) was mixed on rolling. After ½ minute zinc oxide and stearic acid were added simultaneously and rolled for 2 minutes. And then petroleum oil was added and rolled for one minute. Sulphur was added and rolled about 3-4 minutes until thick sheet was obtained. Vulcanized rubber was obtained. Finally, the vulcanized rubber was mixed separately with untreated and treated fibres (various ratios according to Table 4) on rolling. The fibre loadings based on 100 g of rubber were in weight. Total mixing duration was 10 to 15 minutes. During mixing whenever the roller becomes too hot, water was sprayed on the roller (The temperature must be maintained at 60°C). The matrix material thus obtained for composite preparation was being allowed to age for 24 hours. Procedure for preparation of natural rubber–areca nut matrix and the composite was shown in Figure 2.



Figure 2 Flow diagram for the preparation of rubber-areca nut fibre composite

Making composites

The molded plates of aging matrix were compressed for shaping by Hand Press Machine). Firstly, the plates were hot pressed at 153-160 °C (Hardness for 8 minutes and Tear Strength for 6 minutes) under 1000 lb in⁻² loading. By mixing reinforcing agents nine samples were prepared and compositions of these samples were illustrated in Table 4.

Table 4 Composition of Prepared Natural Rubber Composites Reinforced by Areca Nut
Fibre

Sr. no.	Ingredient (g)	natural rubber (NR)	Composite								
1	Natural rubber	100	100	100	100	100	100	100	100	100	100
2	Zinc oxide	5	5	5	5	5	5	5	5	5	5
3	Stearic acid	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
4	Petroleum oil	3	3	3	3	3	3	3	3	3	3
5	MBT	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8	1.8
6	Antioxidant	1	1	1	1	1	1	1	1	1	1
7	Sulphur	2	2	2	2	2	2	2	2	2	2
8	Areca nut	-	U	U	U	S	S	S	Р	Р	Р
	fibre		(5, 5)	(10,10)	(15,15)	(5,10)	(10,15)	(15,5)	(5,15)	(10,5)	(15,10)

NR = Natural rubber

U(5, 5) =Composites with untreated areca nut fibre (5 mm in length and 5 % in loading of fibre to rubber)

U (10, 10) = Composites with untreated areca nut fibre (10 mm in length and 10 % in loading of fibre to rubber)

U (15, 15) = Composites with untreated areca nut fibre (15 mm in length and 15 % in loading of fibre to rubber)

S (5, 10) = Composites with alkali treated areca nut fibre (5 mm in length and 10 % in loading of fibre to rubber)

S (10, 15) = Composites with alkali treated areca nut fibre (10 mm in length and 15 % in loading of fibre to rubber)

S (15, 5) = Composites with alkali treated areca nut fibre (15 mm in length and 5 % in loading of fibre to rubber)

P (5, 15) = Composites with permanganate treated areca nut fibre (5 mm in length and 15 % in loading of fibre to rubber)

P (10, 5) = Composites with permanganate treated areca nut fibre (10 mm in length and 5 % in loading of fibre to rubber)

P (15, 10) = Composites with permanganate treated areca nut fibre (15 mm in length and 10 % in loading of fibre to rubber)

Determination of the Mechanical Properties of the Areca Nut Fibre-Natural Rubber Composites

Reinforced by areca nut fibre

Comparative determination of mechanical properties of natural rubber composites reinforced by areca nut fibre such as hardness and tear strength were carried at the Rubber Research and Development Centre in Yangon.

Determination of hardness

The hardness of a composite also depends on the distribution of the fibre into the matrix. Better dispersion of the fibre into the matrix with minimization of voids between the matrix and the fibre enhanced this hardness. Hardness is a measurement in degree and based on the penetration into the rubber of a definite indentor under a set load. Three scales are commonly used. IRHD (International Rubber Hardness Degree), Shore A, and Shore D. For hard materials over 90, Shore A scale is used. IRHD is preferred for most specifications, but Shore A is also in widespread use.

Materials

Natural rubber sheet, Rubber composite

Apparatus

Wallace Rubber Hardness Tester Figure 4

Procedure

Hardness is a measure of an elastomer's response to a small surface stress. The test was based on the measurement of the indentation of a rigid ball into the rubber Figure 3 under specified conditions. The specified test piece was placed in a Wallace rubber hardness tester Figure 4 and the vibrator was switched on. Hardness is a measurement in degree based on the penetration into the rubber of a definite indentor used. After 30 seconds, the hardness was read directly in IRHD on the micrometer gauge.





Figure 4 Wallace rubber hardness tester

Figure 3 Prepared samples to determine hardness

Determination of tear strength

Materials

Natural rubber sheet, Rubber-Fibre composite

Apparatus

H-5000E Tensile Testing Machine Figure 5

Procedure

The determinations were carried out at standard laboratory temperature in the H-5000E Tensile Testing Machine Figure 5. The test pieces were cut 100 mm length and the machine was started and the change in the test piece monitored continuously. Tear strength is a resistance to the growth of a cut or nick in a vulcanized fiber-rubber specimen when tension was applied. Tear strength is an important consideration, both as the finished article was being removed from the mold and as it performs in actual service.



Figure 5 Determination of tear strength

Results and Discussion

Since hardness and tear strength are the criteria that were chosen to study the optimum parameters, then in determination of S/N ratio, the larger- the- better quality characteristic has been selected.

After performing experimental design and tabulating performance data, appropriate signal-to-noise (S/N) ratio was calculated.

S/N ratio for "larger is better" : SN_L= -10 log [
$$\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}$$
]

Where, *n* is number of repetitions of each experiment and

 y_i is the measured result for i^{th} repetition of each experiment

Table 5 Results for Determination of Signal-to-	Noise Ratios for Hardness and Tear Strength				
of the Composites Prepared According to L9 Orthogonal Array Design					

Expt –	Hard	ness (IR	HD)	S/N	Tea	r Strength ((kN/m)	S/N
No.	R1	R2	R3	Ratio (dB)	R1	R2	R3	Ratio (dB)
1	38	36	36	31.277	22.3	27.5	27.5	28.092
2	45	40	42	32.503	23.0	21.9	22.2	26.986
3	51	45	47	33.529	18.2	14.7	17.3	24.362
4	40	47	45	32.808	16.0	22.6	20.4	25.598
5	43	48	48	33.282	20.7	24.0	23.6	27.088
6	40	42	40	32.178	20.9	25.5	24.1	27.329
7	44	41	43	32.590	19.4	18.9	19.0	25.619
8	37	38	38	31.517	23.0	28.1	27.1	28.222
9	42	40	40	32.178	19.3	28.6	26.9	27.537
Total	380	377	379	291.864	182.8	211.8	208.1	240.833
Over	all mean o = 42.0		ess	Mean, m=32.429	Overall mean of tear strength $= 22.322$			Mean, m= 26.759

Then the mean S/N ratios at each level for various factors have to be calculated. The factor levels corresponding to the highest average S/N ratio will give the optimized condition of maximum efficiency. The S/N ratio for the individual control factors are calculated as given below;

Effects of factor A at Level 1, $m_{A1} = (n_1 + n_2 + n_3)/3$

where n_1 is the signal-to-noise ratio of the first row

where n_2 is the signal-to-noise ratio of the second row, etc.

Effect of factor A at level 2,
$$m_{A2} = (n_4 + n_5 + n_6)/3$$

- Effect of factor A at level 3, $m_{A3} = (n_7 + n_8 + n_9)/3$
- Effect of factor B at level 1, $m_{B1} = (n_1 + n_4 + n_7)/3$
- Effect of factor B at level 2, $m_{B2} = (\eta_2 + \eta_5 + \eta_8)/3$
- Effect of factor B at level 3, $m_{B3} = (\eta_3 + \eta_6 + \eta_9)/3$
- Effect of factor C at level 1. $m_{C1} = (\eta_1 + \eta_6 + \eta_8)/3$
- Effect of factor C at level 2, $m_{C2} = (\eta_2 + \eta_4 + \eta_9)/3$

Effect of factor C at level 3, $m_{C3} = (n_3 + n_5 + n_7)/3$ (YathiAjay *et al.*, 2015)

Factor	actor Fibre treatment type			ength (mm)	Fibre loading (%		
Levels	Raw data	S/N ratio	Raw data	S/N ratio	Raw data	S/N ratio	
L1	42	32.437	41	32.225	38	31.657	
L2	44	32.756	42	32.434	42	32.496	
L3	40	32.095	43	32.628	46	33.134	

Table 6 The Effects of Factor on Individual Levels for Hardness

Factor	Fibre tre	atment type	Fibre le	ngth (mm)	Fibre loading (%)		
levels	Raw data	S/N ratio	Raw data	S/N ratio	Raw data	S/N ratio	
L1	21.6	26.480	21.5	26.436	25.1	27.881	
L2	22.0	26.672	23.7	27.432	22.3	26.707	
L3	23.4	27.126	21.7	26.410	19.5	25.690	

The optimum factor levels can be easily identified from the graphs showing the variation of S/N ratio (Y-axis) with the Levels (X-axis) for different factors. The graphs for hardness and tear strength are shown below.



Figure 5 Effect of the largest fibre type, fibre length and fibre loading on hardness and tear strength

Hardness and Tear Strength Test Result

From the graphs, the optimum combination input parameters for hardness can be observed as: the fibre treatment type NaOH treatment, fibre length 15 mm and fibre loading 15 % weight. Similarly, for tear strength the optimum combination input parameters are: fibre treatment type KMnO₄ treatment, fibre length 10 mm and fibre loading 5 % weight.

Table 8 Optimum Combinations of Values of Factor Levels for each Property Measured

Property		Factor levels				
measured	Fibre treatment type Fibre length (mm) Fibre loading (
Hardness	NaOH	15	15			
Tear strength	KMnO ₄	10	5			

Calculation of Optimum S/N ratio and the Prediction of the Corresponding Optimum Value of the Property

The optimum condition of the three process parameters for hardness is A₂B₃C₃, (A= fibre type, B= fibre length, C= fibre loading) then the theoretical value of ⁿ under the optimum conditions, denoted by n_{opt} is given by:

 $n_{opt} = m + (m_{Ai} - m) + (m_{Bj} - m) + (m_{Ck} - m)$

Where i, j, k are the best levels, respectively, for factors A, B and C.

The corresponding optimum value of larger-the-better type of response characteristic is given by

$$y_{opt}^2 = 1 / 10^{-\eta_{opt/10}}$$

Table 9 Example Calculated for Hardness of Optimum S/N Ratio

Factor	Fibre treatment type			Fibre leng	th (mm)	Fibre loading (%)	
levels	Raw data	S/N ra	tio	Raw data	S/N ratio	Raw data	S/N ratio
L1	42	32.43	7	41	32.225	38	31.657
L2	44	32.75	6	42	32.434	42	32.496
L3	40	32.09	5	43	32.628	46	33.134
Optimum Value - m _{Ai} , m _{Bj} , m _{Ck}							
		m _{A2}	m _{B3}	m _{C3}	m	n opt	y _{opt}
Hardn	less	32.756	32.628	33.134	32.429	33.660	48.20

Comparison of Optimum Condition and Experimental results for Mechanical Properties

It can be clearly shown from this table for theoretical optimum conditions and experimental results data of hardness and tear strength are nearly the same. So, NaOH, 15 mm, 15 % is more significant than other fibre treatment and filler content for hardness. The highest tear strength is $KMnO_4$, 10 mm, 5 % more significant. Therefore, Taguchi's Method of parameter design can be performed with lesser number of experiments as compared to that of other analyses. Taguchi's method can be applied for analyzing any other kind of problems as described in this paper. It is

found that the parameter design of the Taguchi method provides a simple, systematic, and efficient methodology for optimizing the process parameters.

	m _{Ai}	m _{Bj}	m _{Ck}	m	n opt	y _{opt}
Hardness	32.756	32.628	33.134	32.429	33.660	48.20
Tear Strength	27.126	27.432	27.881	26.759	28.920	27.93

Table 10 Optimum Values

Table 11 Experimental Results for Mechanical Properties

	Hardness (IRHD)	Tear Strength (kN/m)
NaOH 15, 15	58.6	19.8
KMnO ₄ 10, 5	50.6	26.1
Rubber only	30.0	24.1

Conclusion

Instead of the conventional one-factor-at-a-time (OFAT) method, the present work manipulates multiple variables simultaneously using statistical technique (design of experiments (DOE)) known as Taguchi method. This method has successfully provided the optimum values of the selected process parameters to be used to prepare two samples of areca nut fibre-natural rubber composites with preferential qualities, hardness for the one and tear strength for the other. Experiments were done according to a L₉ orthogonal array and the results were analysed using the conceptual signal-to-noise (S/N) ratio approach to get the optimum parameter values to effect the desired quality of each composite sample:

For the highest hardness	-	NaOH treated fibre, with fibre length (15 mm), and fibre
		loading (15 %) should be used.
For the highest tear strength	-	KMnO ₄ treated fibre, with fibre length (10 mm), and fibre
		loading (5 %) should be used.

The difficulty to tear is due to the high interfacial locking between the fibre and matrix. Both samples also were found to possess higher values of the respective selected qualities than the rubber without fibre.

These results clearly evidenced that chemical treatments are very effective in surface modification of the areca fibers and improving the mechanical properties of areca fiber reinforced natural rubber composite. So, these chemically treated areca fiber reinforced natural rubber composites are suitable for applications where hardness or tear strength are required, while at the same time reducing environmental issues caused by the unused betel nut shells. All these results have been realized with economy of time and resources by using Taguchi design of experiment.

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