

# RESEARCH ON EXPERIMENTAL INVESTIGATION AND PREDICTIVE LEARNING OF (TiO<sub>2</sub>) PARTICULATE FILLED POLYESTER BASED GFRP COMPOSITE

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**ABSTRACT:** The new set of polyester based polymer composites consisting of e-glass fiber reinforcement, unsaturated polyester resin matrix and titanium oxide (TiO<sub>2</sub>) particulate fillers were fabricated by hand layup method. The particulate filled e-glass fiber reinforced composites were prepared with three different fiber lengths (3 cm, 5 cm and 7 cm), fiber content (20 wt%, 35 wt% and 50 wt %) and particulate content (4 wt%, 6 wt% and 8 wt %). The experimental investigations were conducted to examine their tensile strength and hardness. The fiber length, fiber content and particulate content were chosen as 3 factors in 3 different levels with L<sub>9</sub> orthogonal array and the mathematical model was done using Taguchi method. The influences of these parameters were determined for tensile strength and hardness using Analysis of variance (ANOVA) table and S/N ratio. An expert system was produced using artificial neural network (ANN) which helps in predicting the properties of particulate filled e-glass fiber composition within the above used parameters. The predicted values were experimentally ensured with the maximum coincidence.

**KEY WORDS:** Polymer composite, Experimental investigations, Analysis of variance (ANOVA), Artificial neural network (ANN)

## 1 INTRODUCTION

Composites that form heterogeneous structures and which meet the requirements of specific design and function, imbued with desired properties, limit the scope for classification. Fiber Reinforced Composites are composed of fiber embedded in matrix material. The fibers are principal load-carrying members and the embedded matrix retains its desired position and orientation in the matrix. It acts like a load transfer medium between the matrix and protect the fiber from the damages. Various mechanical properties are improved by adding particles with the composite (Shao - Yun Fu, 2008). The properties of the composites can further be modified by increasing fiber content gradually with respect to the particulate filler content. The increased fiber content reinforcement with particulate reinforcement determines the properties of the composites (S. Srinivasa Moorthy, K.Manonmani, 2013).

The fiber-matrix interface polymer composites are used in automobile, marine and industrial applications.

It may use for even some house hold applications (Herrera-Franco P.J. and Valadez-Gonzalez A., 2005).

The addition of the nano particles increase the bonding strength of the composites and improve the strength (Reynaud E, Jouen T, Gauthier C, Vigier G, Varlet J, 2001; Sabeel Ahmed, K., Vijayaranga, S. and Rajput, C, 2006; Venkata Reddy, G., Venkata Naidu, S., Shobha Rani, T. and Subha, M.C.S, 2009; Panthapulakkal, S. and Sain, M, 2007). Taguchi method is widely used for optimization of various parameters (Tsaoa.C.C and Hocheng.H, 2004; Bala Murugan Gopalsamy, Biswanath Mondal, Sukumal Ghosh, 2009; Dobrzanski.L.A, Domagala.J, Silva. J.F, 2007). In this work, Taguchi L<sub>9</sub> orthogonal array was used with three factors like fiber length, fiber content and particulate loading with three different levels of these variables. Expert system is the efficient tool to predict various process parameters (Chang Chiun Huang and Tsann Tay Tang, 2006, Rajendraboopathy.S, SasiKumar.T, Usha.K.M, Vasudev. E.S, 2008). The main objective of this work is the development and mechanical characterization of a new set of polymer composites consisting of glass fiber reinforcement, unsaturated polyester resin and TiO<sub>2</sub> particulate fillers. Composites are characterized and the predictive learning model was developed within the parameters by using artificial neural network.

## 2 MATERIALS AND METHODS

### 2.1 Materials

The important considerations are obligatory for

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a proper selection of material include fiber, fiber reinforcement form, fiber volume, matrix material, processing cost and safety factors for polymer composites. E-glass is a low alkali glass with a typical nominal composition of SiO<sub>2</sub>-54wt%, Al<sub>2</sub>O<sub>3</sub>-14wt%, CaO+MgO-22wt%, B<sub>2</sub>O<sub>3</sub>-10wt% and Na<sub>2</sub>O+K<sub>2</sub>O. E-glass fibers are commercially used because of its low cost, high productive rates, good chemical resistance and high density. Particle fillers are widely used to improve the properties of matrix materials such as to reduce friction, increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage. E-glass fiber was used as reinforcement and titanium oxide TiO<sub>2</sub> was the filler content.

The Unsaturated polyester resin was used as shown in table 1.

Table 1. Various composition of fiber reinforced polymer composites

Fiber Length (cm)	Particulate (wt %)	Fiber Content (wt %)
3 (A)	4 (U)	20 (X)
5 (B)	6 (V)	35 (Y)
7 (C)	8 (W)	50 (Z)

composite matrix. Accelerants are used to alter a chemical bond, speed up a chemical process, or bring organisms back to homeostasis. Butanone, also known as methyl ethyl ketone / MEK, [CH<sub>3</sub>C(O)CH<sub>2</sub>CH<sub>3</sub>] organic compound was used as accelerant. Catalysis is the process in which the rate of a chemical reaction is changed by a substance known as a catalyst. Here cobalt (ii) naphthenate was used catalyst.

**2.2 Specimen Preparation**

Before the Unsaturated Polyester Resin was laid up on the mould, it was kept well cleaned and dried. A release agent, wax polish was laid up on the mould. E-glass fiber was laid into the fiber mould. The Unsaturated Polyester Resin was mixed with accelerator (1.5%wt) and catalyst (1.5%wt). The titanium oxide particulate was added with this mixture and stirred well. Using a special brush, Unsaturated Polyester resin mixture was laid up uniformly on to the mould

Composites of 27 different compositions were prepared with the same manner. The mould was kept closed and pressed uniformly about 24 hours to cure.

The process of specimen preparations is given in figure 1.



Figure 1. Specimen preparation

After the composites were completely dried, the specimens were cut with standard size. Different compositions of the hybrid composites are

**2.3 Mechanical Characterization**

Tensile test was performed on a Shimadzu AG-IS 50 KN Autograph Universal testing machine according to the guidelines of ASTM D638. The hardness of the composites was measured using Brinell hardness tester.

**2.4 Design of Experiments**

Design of experiments is a powerful analysis tool for modeling and analyzing the influence of process variables over some specific variable, which is an unknown function of these process variables. The most important stage in the design of experiment lies in the selection of the control factors. Many process variables should be included in the design of experiment, so that it would be possible to identify the most significant variables at the earliest opportunity.

**2.5 Taguchi Method**

Taguchi method uses a special design of orthogonal arrays to study the entire parameter space with a small number of experiments. The experimental results are then transformed into a signal-to-noise (S/N) ratio. Taguchi recommends the use of the S/N ratio to measure the characteristics deviating from the desired values.

The S/N ratio for each level of process parameters is computed based on the S/N analysis. Regardless of the category of the quality characteristic, a greater S/N ratio corresponds to better quality characteristics. Therefore, the optimal level of the process parameters is the level with the greatest S/N ratio.

Table 2. L<sub>9</sub> orthogonal array for three variables and three levels

S.No.	Fiber length (cm)	Fiber content (wt %)	Particulate content (wt %)
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

2.5.1 Orthogonal Array

The total number of factor chosen for the experiment was three. The three factors are fiber length (cm), fiber content (wt %), particulate content (wt %) and the number of levels was chosen as three. A L<sub>9</sub> orthogonal matrix array was selected for the experiment. The L<sub>9</sub> orthogonal array is given in table 2.

2.5.2 Analysis of variance (ANOVA)

The purpose of the analysis of variance (ANOVA) is to investigate the design parameters that significantly affect the characteristic of the process. This is accomplished by separating the total variability of the S/N ratios, which is measured by the sum of the squared deviations from the total mean S/N ratio, into contributions by each of the design parameters.

ANOVA can be useful for determining influence of any given input parameter from a series of experimental results by design of experiments for machining process and it can be used to interpret experimental data. Statistically, there is a tool called an F test named after Fisher to see which design parameters has a significant effect on the quality characteristic. In performing the F test, the mean of squared deviations due to each design parameter needs to be calculated. Then, the F value for each design parameter is simply the ratio of the mean of squared deviations to the mean of squared error.

2.6 Artificial Neural Network

Neural networks, which are simplified models of biological neuron system, is a massively parallel distributed processing system made up of highly interconnected neural computing elements that have the ability to learn and acquire knowledge and make it available for use. The neural network is trained so that the application of set of input produces the desired output. Training is accomplished by

sequentially applying the input vectors and adjusting the network weights according to the predetermined algorithm. Training is being done until the network weights gradually converge to values such that each input vector produces the desired output vectors.

2.6.1 Neural Network Analysis

As the study involves many a neural network model which works on past data a back propagation neural network has been chosen. This was used to predict output parameter for given input set. Neural network model was generated using MATLAB 7.5, 2007. Before training a feed forward neural network, the weights and biases must be initialized. This function takes a network object as input and returns a network object with all weights and biases initialized.

2.6.2 Training

Training phase is to learn the relationship between process inputs and targets. Feed the training pair of input and target vectors to the back propagation neural network. The network will attempt to learn the mapping, and if the normalized system error is reached before the end of available iterations, the error value is presented and training is stopped. If the network is unable to reach the error value at the end of iteration, then error value is presented for further training. If the error value is not reduced, increase the number of nodes in the hidden layer or number of hidden layers and go to second step. Else go to third Step. The network has learned the mapping between input and target. Go to testing phase.

2.6.3 Testing

Once the training completed the weights been set for the optimum value and new value feed into the network and output of the network is predicted. The predicted value compared with an experimental value if the error value is less than desired value then the network deemed to have learnt the mapping.

3 RESULTS AND DISCUSSION

3.1 Tensile Strength

The strength of the glass fiber composites depends on the type of fiber, matrix, fiber concentration and bonding between the fiber and matrix. The values of tensile strength of the composites are shown in figure 2. It shows that the tensile strength of the composite increases with increase in fiber and particulate loading. This increase in the tensile strength property may be due

to the fact that the chemical reaction at the interface between the filler particles and the matrix may be too strong to transfer the tensile stress.



Figure 2. Tensile strength values of different composites

### 3.2 Hardness

The hardness of the composite increased with increase in filler material. The filler material may form good bonding strength to resist the load applied. The hardness test results show that the Brinell hardness number increases as the aspect ratio increases. The highest value of 38 BHN and the minimum value of 17 BHN were obtained. The hardness values of the hybrid composites are shown in figure 3.



Figure 3. Hardness values of various composites

### 3.3 Taguchi Method

The measured values of tensile strength as a function of the parameters were shown and the corresponding S/N ratio was shown in the table 3. The S/N ratio is found to be highest value in experiment No.7 which is predicted to be the optimized result.

Table 3. S/N ratio response for tensile strength using larger the better

ExpNo.	Fiber length (cm)	Fiber content (wt%)	Particulate content (wt%)	Tensile strength (N)	S/N ratio
1	3	20	4	2800	70.8432
2	3	35	6	3400	70.6296
3	3	50	8	4700	73.4420
4	5	20	6	4400	72.8691
5	5	35	8	6000	75.5630
6	5	50	4	5300	74.4855
7	7	20	8	6600	76.3909
8	7	35	4	6400	76.1236
9	7	50	6	6400	76.1236

The Mini tab software was used to obtain the response table for the tensile strength. The mean S/N ratio of response for the tensile strength is given in table 4.

The main effects plot for S/N ratios and the main effect plot for means are shown in figure 4 and 5. It ensures that the fiber length and particulate content increases the tensile strength increases.

The response table shows the different parameter and levels S/N ratio. This was useful to find the significance of parameter and optimal condition for hardness. The response table for mean S/N ratio for hardness is shown in table 6.

The main effects plot for S/N ratio and the main effect plot for means are shown in figure 6 and 7. It ensures that the hardness increases when the particulate content increases with 35 wt% of the fiber content. It may be the good bond between the matrix with the fiber and particulate content. Then the hardness was decreased may due to the increase of fiber content may cause to weak the bond between the matrix and the fiber and particulate reinforcement.

Table 4. Mean S/N response for tensile strength using larger the better

Parameter	Mean S/N ratio				
	Level 1	Level 2	Level 3	Delta	Rank
Fiber length (cm)	71.638	74.305	76.213	4.575	1
Fiber content (wt. %)	73.367	74.105	74.634	0.529	3

Particulate content (wt. %)	73.817	73.207	75.132	1.315	2
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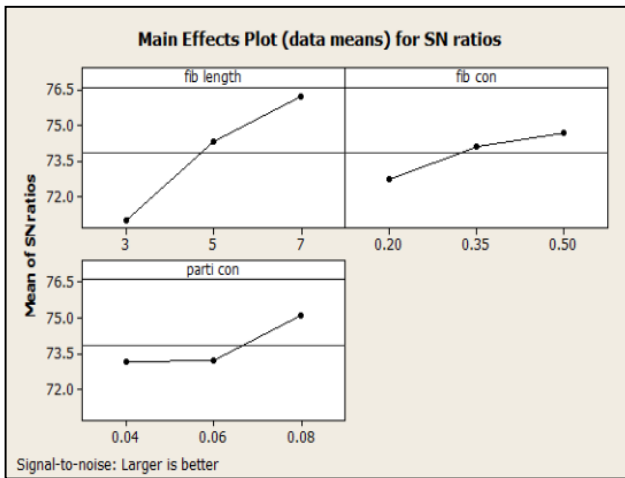


Figure 4. Main effect plot for S/N ratio for tensile strength

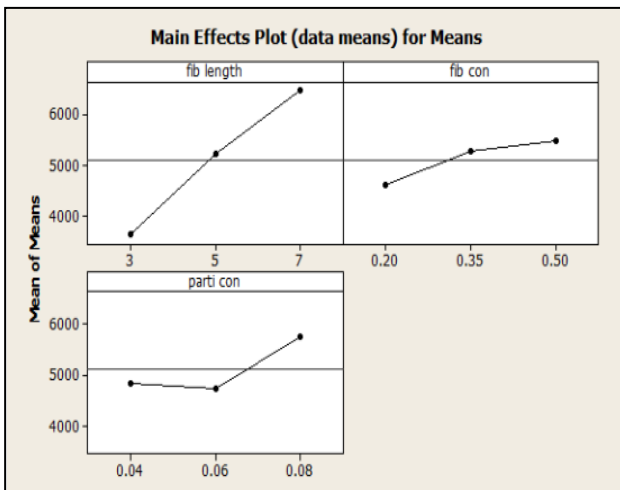


Figure 5. Main effect plot for means for tensile strength

Table 5. S/N ratio for hardness using larger the better

Ex p. No .	Fiber length (cm)	Fiber content (wt%)	Particulate content (wt%)	Hardness (BHN)	S/N Ratio
1	3	20	4	21	27.9439
2	3	35	6	24	27.604

					2
3	3	50	8	26	28.2995
4	5	20	6	28	28.9432
5	5	35	8	33	30.3703
6	5	50	4	26	28.2995
7	7	20	8	31	29.8272
8	7	35	4	26	28.2995
9	7	50	6	30	29.5424

Table 6. Response for mean S/N ratio for hardness

Parameter	Mean S/N ratio				
	Level 1	Level 2	Level 3	Delta	Rank
Fiber length (cm)	27.949	29.204	29.223	1.274	2
Fiber content (wt%)	28.905	28.758	28.714	0.191	3
Particulate content (wt%)	28.181	28.697	29.499	1.318	1

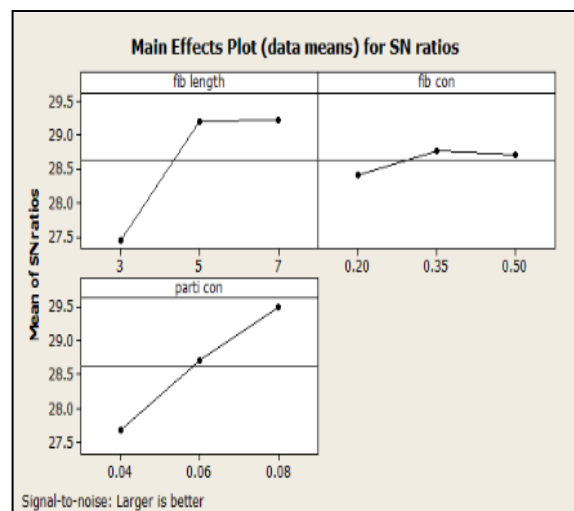


Fig. 6. Main effect plot for S/N ratio for hardness

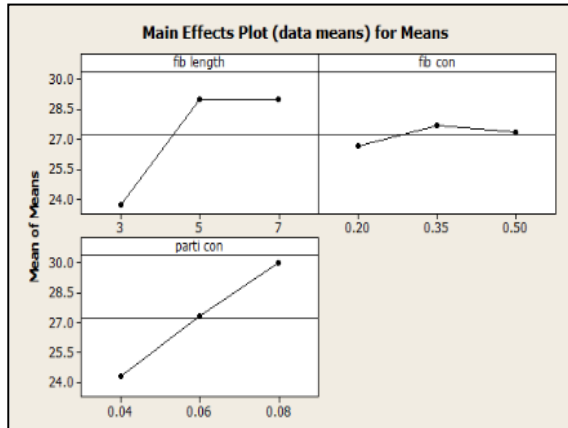


Fig. 7. Main effect plot for means for hardness

Analysis of Variance for tensile strength was carried for the tensile strength using Minitab software and the results are shown in table 7.

Table 7. ANOVA for tensile strength

Source	D F	seqS S	Adj SS	Adj MS	F $\alpha = 5\%$	P
Fiber length (cm)	2	3.4286	3.428	1.570	18.55	0.235
Fiber content (wt%)	2	0.8661	0.866	0.423	4.12	0.182
Particulate content (wt%)	2	0.7400	0.740	0.370	3.50	0.059
Error	2	0.2250	0.225	0.112	-	
Total	8	5.2597			-	

The analysis of Taguchi for delamination factor and the results for tensile strength and hardness are shown in table 7 and table 8.

### 3.4 Artificial Neural Network

#### 3.4.1 Training phase

The 20 sets of data were used for the training purpose. The training phase data are given in table 9. Once the network weights and biases have been initialized, the network is ready for training. During training the weights and biases of the network are iteratively adjusted to minimize the network performance function.

#### 3.4.2 Testing phase

The seven sets of data were used for the testing purpose. The test data were in normalized form. After the successful training, the test data was an input to the system. The network gives the predicted output parameters.

Table 10 and table 11 indicate that the error values were acceptable. The values obtained by Artificial Neural Network using Matlab were confirmed that it can be used as an Expert System.

Table 8. ANOVA table for hardness

Source	D F	SeqSS	adjSS	Adj MS	F $\alpha = 5\%$	P
Fiber length (cm)	2	130.667	130.667	65.333	15.11	0.10
Fiber content (wt%)	2	16.133	16.133	8.066	1.31	0.15
Particulate content (wt%)	2	64.00	64.00	32.00	6.60	0.18
Error	2	10.667	10.667	5.333	-	
Total	8	221.467			-	

Table 9. Training phase

S. NO	Fiber length (cm)	Particulate content (wt%)	Fiber content (wt%)	Tensile Strength (GPa)	Hardness test (BHN)
1	3	4	20	0.047	18
2	3	4	35	0.057	20
3	3	4	50	0.060	21
4	3	6	35	0.057	18
5	3	6	50	0.060	18
6	3	8	20	0.070	18
7	3	8	50	0.078	18
8	5	4	20	0.070	19
9	5	4	50	0.088	20
10	5	6	20	0.073	23
11	5	6	50	0.093	23
12	5	8	20	0.097	23
13	5	8	35	0.100	22
14	5	8	50	0.107	23
15	7	4	20	0.103	23
16	7	4	50	0.100	23
17	7	6	20	0.103	22
18	7	6	35	0.107	19
19	7	8	20	0.110	23
20	7	8	50	0.108	24

Table 10. Testing Phase of tensile test

R un s	Fib er leng th(cm)	Parti culat e (%)	Fiber cont ent (%)	Actual value	Predict ed value	Error

21	3	6	20	0.0584 61	0.0521 25	- 0.0633 7
22	3	8	35	0.0707 69	0.0641 52	- 0.0661 7
23	5	4	35	0.0707 69	0.0615 38	- 0.0923 1
24	5	6	35	0.0738 46	0.0763 41	0.0249 48
25	7	4	35	0.0984 61	0.0954 36	- 0.0302 6
26	7	6	50	0.0984 61	0.0960 03	- 0.0245 9
27	7	8	50	0.0953 84	0.0948 17	- 0.0056 8

Table 11. Testing phase of hardness test

R un s	Fib er length (cm )	Part iculate (%)	Fib er content (%)	Actu al value	Predict ed value	Error
21	3	6		0.07	0.0506 45	- 0.019 355
22	3	8	35	0.05 75	0.0781 65	0.020 665
23	5	4	35	0.07 25	0.0544 58	- 0.018 042
24	5	6	35	0.09 25	0.0787 72	- 0.013 728
25	7	4	35	0.06 5	0.0812 45	0.016 245
26	7	6	50	0.07 5	0.0922 15	0.017 215
27	7	8	50	0.09 5	0.0823 52	- 0.012 648

4 CONCLUSION

The polymer composites consist of titanium oxide filled, e glass fiber reinforced in the unsaturated polyester resin matrix were fabricated by hand layup method. The composites were made with three different process variables like fiber lengths, particulate loading and fiber content. 27 different combinations of these variables were

considered and the experiments were conducted on the composites to study the tensile strength and hardness. It is observed that the tensile strength and hardness were increased with increase in fiber content and particulate loading. The optimization was carried out by Taguchi’s method and the influencing parameters were determined. An Expert system was created using Artificial Neural Network to predict the properties of e-glass fiber composites within the above used parameters. Final experiments ensured with the maximum coincide from the values of expert system.

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