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OPTIMIZING THE FORMULATION OF E-GLASS FIBER AND COTTON SHELL PARTICLES HYBRID COMPOSITES FOR THEIR MECHANICAL BEHAVIOR BY MIXTURE DESIGN ANALYSIS

OPTIMIRANJE OBLIKOVANJA HIBRIDNEGA KOMPOZITA Z E-STEKLENIMI VLAKNI IN DELCI BOMBAŽNIH LUŠČIN – ANALIZA MEHANSKIH LASTNOSTI MEŠANIC

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The purpose of this work is to create and examine the mechanical properties of an eco-friendly, low-cost natural fiber filled with glass-fiber-reinforced polymer composite. Cotton shell (CS) particles are a less expensive natural fiber material that can be used as filler, integrated with glass fiber in epoxy-resin matrices. By keeping the optimized resin weight as a constant (75 % of mass fractions), six composites C1, C2, C3, C4, C5 and C6 of different weight percentages of glass fiber and the CS filler were prepared and the corresponding mechanical properties were examined as per ASTM standards. The glass fiber of 15 % of mass fractions and filler material of 10 % of mass fractions of the composite "C3" shows the best mechanical properties. The mixture design analysis is used to evaluate the results obtained mathematically, and the predicted values confirm that the 10 % of mass fractions of addition of CS particles with the glass fiber augment the mechanical properties of the hybrid composites. The chemical composition of the cotton shell particles was analyzed by using energy-dispersive spectroscopy (EDS). Also, the surface morphology of the raw CS particles and the fractured surfaces of the tested specimens were examined with a scanning electron microscope (SEM).

Keywords: E-glass fiber, cotton shell (CS) particles, epoxy resin, mechanical properties, mixture design analysis

Namen tega dela je bil oblikovati in preiskati mehanske lastnosti cenenega, ekološko prijaznega polimernega kompozita iz naravnih vlaken ter ojačanega s steklenimi vlakni. Delci bombažnih lupinic (angl. CS) so cenen vir naravnih vlaken, ki se lahko uporabi kot polnilo in s steklenimi vlakni integrira v epoksidno matrico. Avtorji članka so v tej raziskavi obdržali konstantni masni delež epoksidne smole (75 mas. %) v šestih mešanicah C1,C2,C3,C4,C5 in C6 z različnimi masnimi deleži steklenih vlaken in CS polnila. Pripravljene kompozite so mehansko okarakterizirali v skladu z ASTM standardom. Kompozit C3 s 15 mas. % steklenih vlaken in 10 mas. % CS polnila je imel najboljše mehanske lastnosti. Da bi matematično ovrednotili dobljene rezultate so avtorji uporabili t.i. MDA (angl.: Mixture Design Analysis) in napovedane vrednosti analize so potrdile, da dodatek 10 mas. % CS delcev k steklenim vlaknom, izboljša mehanske lastnosti hibridnih kompozitov. Kemijsko sestavo bombažnih lupinic so analizirali z energijskim dispezijskim spektrometrom (EDS). Prav tako so površinsko morfologijo CS delcev in prelomne površine preiskovanih vzorcev pregledali z vrstičnim elektronskim mikroskopom (SEM).

Ključne besede: E-steklena vlakna, delci bombažnih luščin, epoksidna smola, mehanske lastnosti, analiza oblikovanih mešanic

1 INTRODUCTION

Natural fibers demonstrate predominant mechanical properties, for example, adaptability, firmness and modulus, compared with glass filaments. Currently, the popular natural filaments sisal and jute fibers are supplanting the glass and carbon fibers owing to their simple accessibility and cost. Polymers and their composites are being more and more employed because of their superior strengths and low densities. On account of their outstanding properties, fiber-reinforced polymer composites are used chiefly in the automotive and aircraft industries, spacecraft and ships. The accumulation of a particular filler diminishes the polymerization shrinkage of the radically polymerizing resin, enhances the dimensional stability, amplifies the elastic modulus and strength to the level found in dental tissues, decreases the friction wear and produces a better X-ray opacity.[1] The composite industry always seeks for an alternative low-cost source that can reduce the overall manufacturing costs and boost the stiffness of the materials. Natural materials have noteworthy advantages, which give a reason for their use. Other than their environmental profit, the probable benefits of natural fibers are, the plentiful accessibility of the raw materials from renewable resources, rather than fossil sources, and their lower cost. Also, they have an elevated specific strength due to their lower density. In addition, it is possible to achieve an elevated loading of natural fibers in reinforced plastic composites than in traditional inorganic fillers, since the former is a softer nonabrasive material.[2]

The majority of developing countries are very rich in agricultural fiber and a big fraction of agricultural waste is being utilized as a fuel. Only India produces more than 400 million tones of agricultural waste yearly. Growing concern about global warming, mainly due to deforestation, has led to the growth of new materials to replace wood, which improves most favorable consumption of natural resources. Presently, natural fibers form a substitute for glass fiber, the most extensively applied fiber in composite technology. The positives of the natural fibers over synthetic fibers like aramid, carbon or glass fiber are low densities, non abrasive, non-toxic, high filling levels, possibly resulting in a high stiffness and specific properties, biodegradable, low cost, good thermal and acoustic properties, good calorific value and superior energy recuperation. The environmental impact is reduced as the natural fiber can be thermally recycled and fibers come from a renewable resource. The natural fibers also put forward the possibility in developing countries to make use of their own natural resources in their composite processing industries.[3] Utilizing the easily obtainable lower cost fillers, we can advance the properties and reduce the overall cost of components.[4]

Natural fibers having cellulose, hemi cellulose and lignin are being examined for the appropriateness of alternating synthetic fibers. The utilization of these natural fillers has been due to its monetary benefit during processing, high specific strength, comparatively low density and the bio-degradability, thereby reducing the environmental contamination.[5] The idea of adding particles of sub-micro- or nano-scale into polymers is one of the most fascinating subjects in the current decades.[6] One of the methods to augment the mechanical properties of fiber-reinforced polymer (FRP) composites is to progress the properties of the epoxy matrix by integrating second-phase modifiers into the resin. Owing to their high specific strength and stiffness, FRP composites are broadly used in ship hull, airframe, and wind-turbine structural applications. When polymerized, the epoxy becomes amorphous and it is an extremely cross connected material. This microstructure of the epoxy polymer leads to many valuable properties such as a high modulus and failure strength, low creep, etc., but also directs to an adverse property in that it is moderately brittle and has reasonably poor resistance to crack instigation and propagation.[7]

Figure 1: Cotton shell

The FRP composite material consists of a polymer that acts as a matrix and a reinforcing material, which is chosen as per preferred applications and properties. In a typical FRP composite, the fiber performs as a typical load-carrying aspirant, while the matrix transmits the load to the fiber and to guard it from any fault. Extensively used glass-fiber reinforcing material has its outstanding corrosion resistance, weight reduction, high impact, superior tensile strength and little cost. In general, it is used with polymer matrices such as epoxy resin, vinyl ester, isophthalic polyester resin and unsaturated polyester resin.[8] Polymers are reinforced with fibers and fillers to get better mechanical and tribological properties and to achieve a better strength-to-weight ratio. Various types of non-metallic bearing materials currently exist commercially. Polymeric bearing materials are used exclusively where fluid lubricants are ineffective due to the aggressive environmental conditions, complexity to afford maintenance also due to product and environmental pollutions.[9] Currently, unambiguous fillers/additives are supplementary to improve and amend the superiority of composites as these are found to impart a key role in formatting the physical properties and mechanical performance of the composites.[10]

In the past two decades, the lignocellulosic natural fibers are being commonly used as reinforcements and fillers in polymer applications.[11] The mechanical properties are mainly influenced by the particle size, shape and specific surface area of integrated particles/fillers in polymers.[12] Hybridization that is reinforced with more than one fiber, is one of the best ways to defeat the limitations of the mechanical properties of the polymer composites.[13] Because of these combined properties of high strength and stiffness-to-weight ratio, corrosion resistance, excellent damping character and resilience, the hybrid composites are broadly used in industrial applications.[14] For the making of interior and exterior components of automotives, natural fiber mats are widely used.[15] The essential applications of these composites

Figure 2: Cotton shell powder

are developing quickly in most of the engineering areas, particularly where the cost of the material is a major criterion and to make better the erosive environment.[16] Nano-sized or micro-sized particles reinforced composites ended in agglomeration, which causes stress concentration, which ultimately reduces the potency of closely packed samples.[17] Composite materials afford design engineers with advanced quality and extended life period. Superior strength, lower weight with fewer maintenance have directed to numerous engineering applications.[18]

The objective of this paper is to learn the perspectives of using ligno-cellulosic plant remainder as a filler for a thermoset polymer. These plant residues are a low-priced by-product, eco-friendly and practically sustainable raw materials. The size of the particles ranging from 125 μm to 149 μm, and shape of the CS particles progress the uniform distribution of the filler particles in the matrix materials of the FRP composites. The CS particles have less density and can be considered as suitable filler materials for the development of lightweight and high-strength composites.

Hence, in this present work, a first attempt is made to study the mechanical properties of CS particles integrated into glass-fiber reinforced polymer composites by varying the addition of particles content. Also, the mixture design analysis is carried out to identify the optimal formulation of glass fiber and CS particles.

2 EXPERIMENTAL PART

2.1 Materials

Cotton Shell (CS) was produced from a local village in Kovilpatti, Tamilnadu, India. The density is 1.2726 g/cm^3. Araldite LY 556 is medium viscosity, unmodified liquid epoxy resin based on Bisphenol –A and Aradur HY 951 is a low viscosity, unmodified, aliphatic polyamine, were supplied by Javanthee enterprises, Guindy, Chennai, India. The specific gravity of the resin is 1.15–1.20 g/cm^3 whereas the hardener is 0.97–0.99 g/cm^3. The Chopped strand mat (CSM) E-Glass fiber of 300 GSM was purchased from Goa Glass fibers Ltd., Goa, India, and its specific gravity is about 2.6 g/cm^3. The resin and the hardener are mixed with a ratio of 100:10 by weight, as recommended, and they are mixed uniformly until they form a homogeneous mixture.

Initially, the CS was dried in the sun for a week and then washed thoroughly with distilled water to remove the residual dirt and impurities and then dried again in the sun for one week. The dried CS was ground to fine powder using a ball milling machine and a set of standard sieves are arranged in descending order of fineness and shaken for 20 min and the size in the range of 125 μm to 149 μm particles are used for this research work. The CS and CS powders are seen in **Figure 1** and **2**, respectively.

2.2 Methods

The percentages of glass fiber and resin were optimized by mixture design analysis.[19] The resin/glass fiber/CS composites were developed by mixing optimized constant 75 % of mass fractions of epoxy resin with different % combinations of glass fiber and CS filler content. The formulation of the different composites is given in the **Table 1**.

A thin coating of hard wax was applied over the mould cavity for easy removal of prepared composite. Measured amounts of epoxy and hardener were mixed in a container and stirred well using a mechanical stirrer, then a measured quantity of CS filler is added and again stirred well for a particular time and then the mixture is poured in the mould cavity and the Chopped Strand Mat (CSM) E-Glass fiber of measured quantity is placed in between the resin and filler layers so that the required thickness is achieved. Finally, the composite laminates of size of 250 mm × 250 mm × 3 mm were fabricated by hand lay-up technique followed by compression moulding.

Six different composites of various glass fiber and filler contents by weight were prepared. After that, for better curing of the composite, it was kept under pressure of about 7 MPa for 4 h to 6 h in the hydraulic press, at room temperature. The suitable dimensions of specimen were prepared according to the size and shape as per ASTM Standards, using a diamond cutter. The sample tensile specimens are shown in **Figure 3**.

Tensile, compression and flexural tests were done using a universal testing machine manufactured by ABS Instrumentation Pvt. Ltd., with the capacity of 40 T and accuracy of 1 N with a cross head speed of 5 mm per minute, as per the standards ASTM D 638, ASTM D 695 and ASTM D 790, respectively.

The hardness test was conducted by using the Barcol hardness tester as per the standard ASTM D 2583 also the impact test is conducted as per ASTM D 256 standard, using impact testing machine manufactured by Fuel Instruments and Engineers (FIE) Pvt. Ltd., For all the tests, four samples were tested and the average value

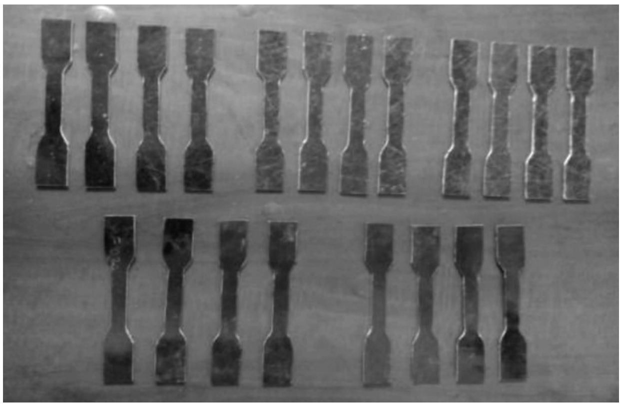

Figure 3: Tensile specimens for different composites

was taken into consideration. The values are exhibited in **Table 2**.

Table 1: Composition of cotton shell particles integrated fiber reinforced polymer

Sl. No:	Composite	Resin %	Glass fiber %	CS particles %
1	C1	75	25	00
2	C2	75	20	05
3	C3	75	15	10
4	C4	75	10	15
5	C5	75	05	20
6	C6	75	00	25

3 RESULT AND DISCUSSION

3.1 Mechanical properties

The tensile specimens of six different GFRP composites with filler were tested and the values were plotted on the Y axis against the various weight % of glass fiber and filler material in the X axis. The tensile strength values elevate from composite C1 to C3 and thereafter it starts descending in its values, as seen in the **Figure 4**. That is, up to addition 15 % of mass fractions of CS fillers, the tensile strength values increases and further addition of filler particles leads to decrement of tensile strength value. Hence, it is conclusive that the composite C3 has highest tensile strength value and is better when compared to the other combinations.

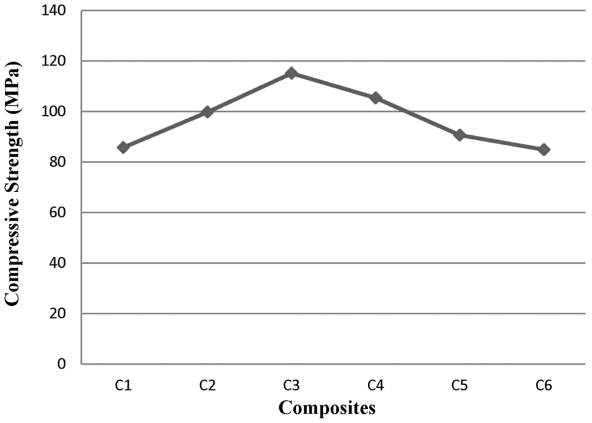

Figure 5: Compressive strength of FRP for different % of mass fractions of glass fiber and CS filler

Similarly, the corresponding other test values are plotted in the Y axis against the various weight % of glass fiber and filler material in the X axis. The respective graph **Figures 5** to **8**, reveals that the mechanical properties are increasing from C1 to C3 and falls down after crossing C3. From this, it is confirmed that the mechanical properties of C3 are dominative and found better with the composition of 15 % of mass fractions of glass fiber and 10 % of mass fractions of CS filler.

3.2 Scanning electron microscopy (SEM) analysis

Morphological studies using a scanning electron microscope (Carl Ziess EVO 18, Kalasalingam Univer-

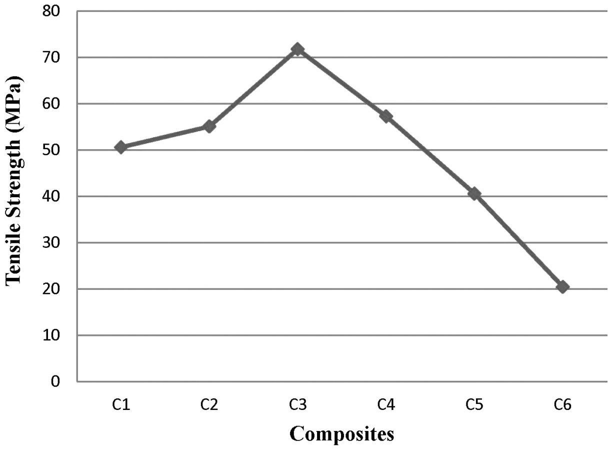

Figure 4: Tensile strength of FRP for different % of mass fractions of glass fiber and CS filler

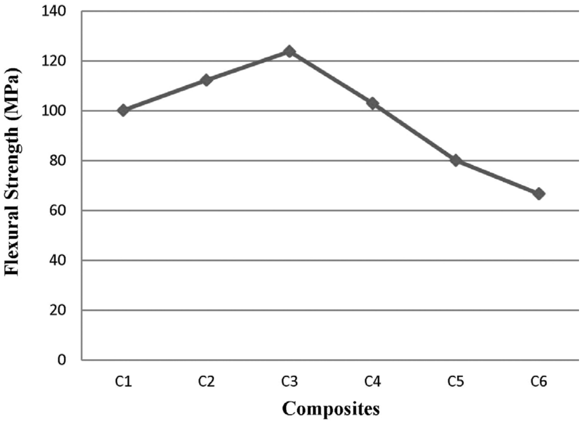

Figure 6: Flexural strength of FRP for different % of mass fractions of glass fiber and CS filler

Table 2: Mechanical properties of different composites

Sl. No:	Composite	Tensile strength (MPa)	Compressive strength (MPa)	Flexural strength (MPa)	Hardness (HB)	Impact energy (J)
1	C1	50.62	85.79	100.2	38	5
2	C2	55.1	99.83	112.3	40	6
3	C3	71.8	115.2	123.8	44	8
4	C4	57.3	105.4	103	41	6
5	C5	40.6	90.7	80.1	36	4
6	C6	20.45	84.9	66.7	34	3

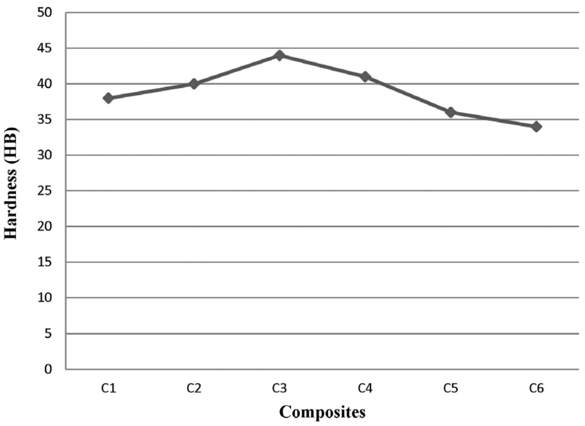

Figure 7: Hardness of composites for different % of mass fractions of glass fiber and CS filler

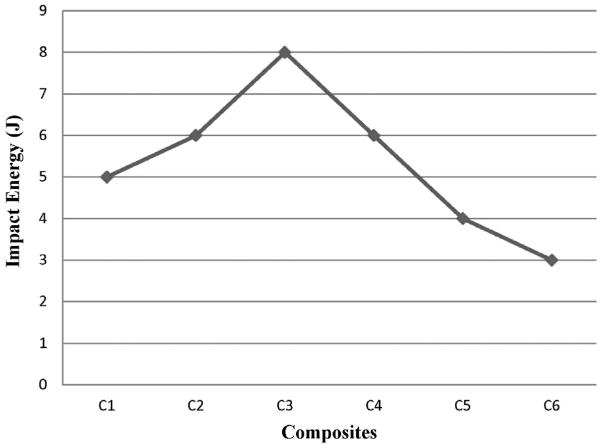

Figure 8: Impact energy of composites for different % of mass fractions of glass fiber and CS filler

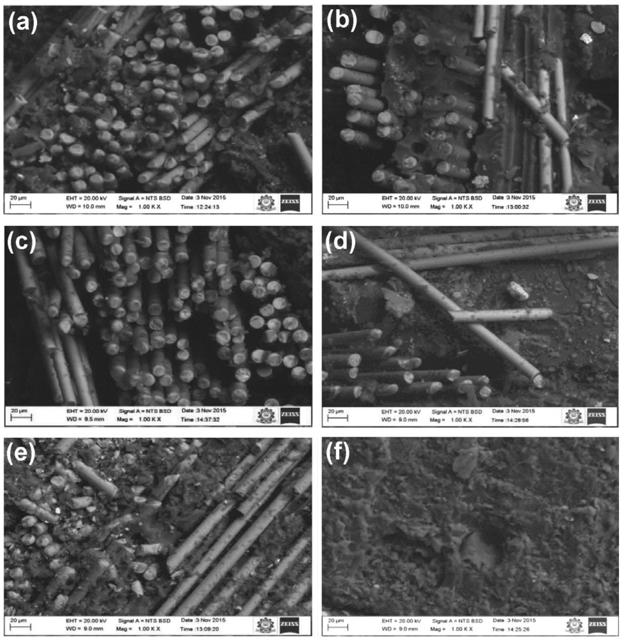

Figure 9: SEM: a) of glass fiber with 25 % of mass fractions, b) of 20 % of mass fractions glass fiber with 5 % of mass fractions of CS filler, c) of 15 % of mass fractions glass fiber with 10 % of mass fractions of CS filler, d) of 10 % of mass fractions glass fiber with 15 % of mass fractions of CS filler, e) of 5 % of mass fractions glass fiber with 20 % of mass fractions of CS filler, f) of 25 % of mass fractions of CS filler

sity, Krishnankoil, Tamilnadu, India), evidently illustrate the GFRP composites (**Figures 9a** to **9f**). The **Figure 9a** shows the morphology of the composite with only glass fiber of 25 % of mass fractions. Morphology of the GFRP composites with the filler addition starts from 5 % of mass fractions to 20 % of mass fractions are exhibited in the **Figures 9b** to **9e**. And micrograph in **Figure 9f** shows the composite with only CS fillers of 25 % of mass fractions.

The micrographs noticeably point out that while the CS particles were supplemented with GFR epoxy matrix, there is a change in the occurrence of the microstructure. The microstructure of the GFR epoxy matrix disclosed that there is a presence of amorphous structure with extended boundaries.

It was noticed that the glass fibers are protected by the layer of matrix and there is a fine diffusion of CS fillers with the matrix (**Figures 9b** to **9c**). The appropriate combination of the CS fillers – epoxy matrix has a significant role in the developed GFRP composites. Sturdy filler – epoxy matrix of interlinked bonding with glass fiber is important for better mechanical properties of the composites. When the CS particles contribution exceeds 10 % of mass fractions there is a non-uniform distribution of the particles in the matrix and poor interfacial bonding between the epoxy, glass fiber and CS fillers, which leads to the formation of delamination and pull out of the fiber and fillers, which leads to the descending values of the mechanical properties.

3.3 Density

The density of a composite depends on the relative proportion of the matrix and reinforcing materials and this is the most significant factor influencing the properties of the composites. The theoretical and measured densities along with the respective volume fraction of voids were exhibited in **Table 3**. The composite density values calculated theoretically from weight fractions are not in concurrence with the experimentally found values. The disparity between these two is a measure of voids and pores present in the prepared composites. The volume fraction of voids kept on changing as the filler content addition changes for different composites.

The presence of voids considerably affects the mechanical properties and performance of the composites in the position of utilization. More voids generally means less fatigue resistance and more susceptibility to water penetration. The awareness of void content is advantageous in assessing the excellence of the composites. It is logical that a superior composite must have fewer voids. However, the existence of a void is obvious to some extent in composite preparation.

Table 3: Theoretical and measured density of the composites with volume fraction of voids

Sl. No:	Composite	Theoretical density g/cm^3	Measured density g/cm^3	Volume fraction of voids (%)
1	C1	1.362	1.34	1.62
2	C2	1.325	1.30	1.89
3	C3	1.291	1.26	2.4
4	C4	1.254	1.21	3.51
5	C5	1.228	1.17	4.72
6	C6	1.198	1.12	6.51

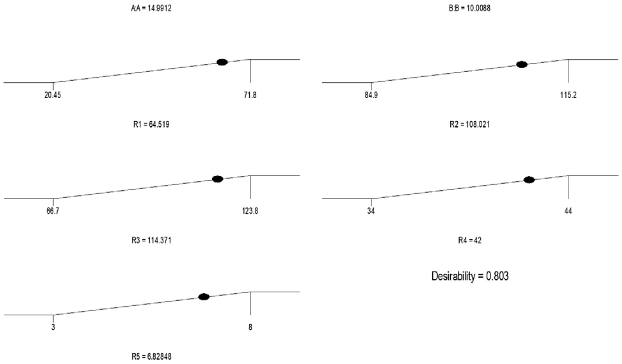

Figure 10: Ramps report of numerical optimization of glass fiber and filler particles

4 OPTIMIZATION BY GLASS FIBER AND CS FILLERS BY MIXTURE DESIGN ANALYSIS

In order to forecast the output response corresponding to the input parameters, here the mathematical modeling tool Mixture Design Analysis is used. The response is the function of the properties of the various components in the mixture. The mechanical properties of the CS fillers integrated GFRP composite specimens such as tensile strength (TS), compressive strength (CS), flexural strength (FS), hardness (HS) and impact energy (IE) are assigned as response functions. The glass fiber and CS filler are the components in the mixture. With the help of the arrived numerical expressions for the responses, the magnitude of the response can be established for any value of the input parameter. Then the predicted responses can be compared with the experimental values. The degree of the nearness of the predicted and experimental response value will exhibit the accuracy of the model for the specific experiment. Design Expert 9 software is used for the simple lattice mixture modeling. The design methodology can be extended for the mixture with 2-30 constituents. A simple lattice mixture design of degree m consists of m + 1 points of evenly spaced values between 0 and 1 for each component. For example, if m = 2 then the possible fractions are 0, 1/3, 1 and if m = 3 then the probable values are 0, 1/3, 2/3, 1.

To obtain the optimal formulation of the GFRP composites by forecasting the best possible composition of glass fiber and CS filler. The experimental results were analyzed by using mixture design analysis in order to attain an empirical model for the outstanding response. The modeling tool is arrived as linear, quadratic and cubic form of equations for the response functions. Using the mathematical equations represented as coded factors are in the **Table 4**, the mechanical properties TS, CS, FS, HS and IE for the various combinations of glass fiber and CS filler can be designed precisely.

The design expert works out the numerical optimization on an objective function designated as Desirability. The altogether desirability (D) is the geometric mean of all individual desirabilities (d_i) that ranges from zero to one, as given in Equation (1):

$$D = (d_1 \times d_2 \times d_3 \times . \times d_n)^{1/n} \qquad (1)$$

where n is the total number of responses. If any one of the responses beyond its desirability range, the overall function will become zero. For betterment of result, by assigning the highest response as the optimization parameter, the numerical optimization is worked out for the response functions.

By putting together the individual desirability values in to a whole single number and then look for the maximum overall desirability. After the analysis, here it is the overall desirability is 0.803 which is nearer to 1, which is the ideal value.

In **Figure 10**, the ramp report is displayed in which the individual response graph is given for a better understanding. The red dot corresponds to the component level in the specific mixture. The blue dot corresponds to the response level for the specific component levels in the mixture. The dot on every ramp represents the response prediction for that solution. The altitude of the dot indicates how desirable it is.

A bar graph showing the individual responses desirability and overall desirability is also obtained from the analysis. The overall desirability function D evolved as 0.803498, in which d_i varies between zero and one as per the proximity of response to its ideal value. The desirability values that are 1 and nearer to 1 are stated as excellent. In this the red bar represents the different

Table 4: Mathematical model for mechanical properties of glass fiber with filler

Response Sl. No	Response	Model	Numeric equation
R1	TS	Cubic	$TS = 48.95 \times A + 19.11 \times B + 114.62 \times A \times B$
R2	CS	Cubic	$CS = 86.88 \times A + 80.93 \times B + 97.98 \times A \times B$
R3	FS	Cubic	$FS = 102.25 \times A + 61.55 \times B + 118.35 \times A \times B$
R4	HS	Cubic	$HS = 38 \times A + 33 \times B + 25 \times A \times B$
R5	IE	Cubic	$IS = 5.07 \times A + 2.50 \times B + 11.61 \times A \times B$

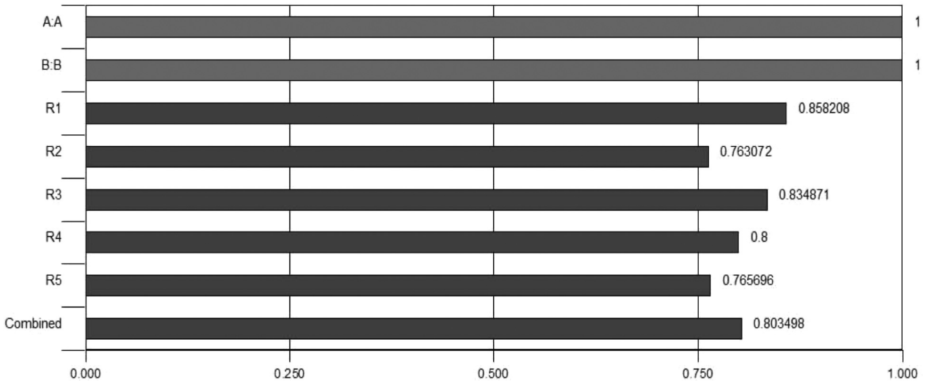

Figure 11: Desirability bar graph for the optimized glass fiber and filler particles

components in the mixture and the blue bar represents the responses of GFRP with CS filler. The desirability of all the five responses lie between zero and one, which indicates that all the responses are well within the agreeable limits, which is shown in the **Figure 11**.

In the **Table 5**, the end results of the analysis of variance (ANOVA) for the mechanical properties are exhibited, which clearly specify that the predictability of the model is at 95 percent confidence interval.

The predicted response fits well with those of the experimentally obtained responses. And also it is confirmed from the **Table 6**, that all the predicted values of the responses lie in the range between 95 % PI low and 95 % PI high.

Table 5: Response prediction for glass fiber with filler

Two sided component	Name	Confidence = 95 %		*n* = 1 Coding	
		Level	Low level	High level	
A	Glass fiber	14.99	0.00	25	Actual
B	Filler	10.01	0.00	25	Actual

Total = 25

Table 6: Summary of the prediction for the responses

Response	Prediction	Std. dev	SE (n=1)	95 % PI low	95 % PI high
TS	64.519	5.93736	6.95	42.39	86.65
CS	108.021	6.41165	7.51	84.12	131.92
FS	114.371	8.23169	9.64	83.69	145.05
HS	42	1.82574	2.14	35.20	48.80
IE	6.82848	0.941124	1.10	3.32	10.34

5 CONCLUSIONS

Based on the investigations, for developing new polymer composites, the cotton shell particles could be recognized as potential low-cost natural fibers filled with glass fiber.

As per the test results, the mechanical properties are showing their better values for the composite "C3" that is glass fiber of 15 % of mass fractions and CS fillers of 10 % of mass fractions, when compared to the other combinations. It is evident that after the 10 % of mass fractions, further addition of CS filler leads to a decrease in mechanical properties.

The surface morphology study of the fractured surfaces is carried out by SEM, which discloses that the CS fillers have appreciable interaction with the matrices up to 10 % of mass fractions addition, but further if the addition of filler exceeds there is poor bonding between them and the formation of cracks and voids occur. From the study, thus concluded that CS filler integrated GFRP composites with 15 % of mass fractions of glass fibers and 10 % of mass fractions of filler have better mechanical properties.

The mixture design analysis is implemented to reveal the predicted values for various responses. On the whole, the desirability for GFRP with CS filler is 0.803. This shows that the desirability value is nearer to 1 and good. From the numerical optimization it is established that 10 % of mass fractions CS filler in GFRP exhibits the best results.

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