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Experimental Investigation Mechanical Behaviour of Composite Made by Carbon Fibre Mixed with Zro2 and Phenolic Resin

E. Latha Devi [1]*, N. Kishore Nath [2], P. Ramesh Babu [3]

Abstract

Composites are one of the major research criteria in aerospace technologies to fulfil the present needs. Addition of materials to carbon fibers without increasing of weight of the parts Nano and micro fillers have its own criteria in improving mechanical properties. ZrO2 particles added to carbon fibers can improve the strength with a light add up in density. Present study focused on the mechanical behavior of zirconium oxide addition to carbon fibers coated with Phenolic resin. It could contribute introduce a new class of CFRP composites that perform well enough to be used in applications with high strain rates without experiencing major failures during use. The cooling temperature for curing playing a crucial role the present laminates checked at different levels as 30°, 50°, 70°, 90°C.The amount of filler addition varies from 0.5%,1%.1.5% to 2% with different pressure rates for the preparation of carbon fiberous layers using vacuum impinging method. Design of experiments with Taguchi varying the parameters in 4-levels with L16 orthogonal array. 1.5mm thick carbon fibre layer with 300mm square mould taken as dimensions for the test pieces. A pressure input of 10 bar, 15 bar,20bar and 25 bar used for mixing of fillers and further tested the sample with a Split- Hopkinson pressure bar (SHPB), operated at momentum's of 15 kg m/s and 28 kg m/s for impact testing. 1.5% addition with 15 bar given better results in mechanical properties compared with other samples.

Keywords: CFRP, ZrO2, phenolic resin, mechanical properties, SEM, vacuum impinging

INTRODUCTION

Carbon fiber/phenolic composites enhanced with nanomaterials have recently piqued the curiosity of many in the realm of cutting-edge, high-performance building materials and constructions [1]. They find particular usage in aircraft applications, where the majority of ceramic and metallic materials are deemed undesirable. Adsorbing the heat and preventing its transfer to the internal parts, ablative materials safeguard the spacecraft from the enormous quantity of heat created by atmospheric friction. Phenolic resin is an appealing ablative material candidate because of its low flammability, high char yield, excellent thermal insulation, and outstanding thermal stability. It is the first synthetic thermoset polymer. [2][3]. This produced char is essential to the ablation process because it slows down the pyrolysis rate by re-emitting a lot of radiation into the gas phase. Nevertheless, in conditions of high pressure and velocity, shear force can mechanically remove a char layer, even when fiber reinforcement is present. In an effort to address these issues, carbon fiber/phenolic composites have been tested with various nanosized reinforcements [4]. Researchers have looked at the manufacturing and thermal properties of phenolic resin/silica composites made using a high shear rate-mixing

***Author for Correspondence**
E. Latha Devi

[1]Assistant Professor and Research Scholar, Department of Mechanical engineering, Stanley college of engineering and technology, Hyderabad, Telangana, India
[2] Project Director at Advanced Systems Laboratory, Defence Research and Development Laboratory, Kanchanbagh Hyderabad-500058
[3]Professor, University College of engineering, Osmania University, Hyderabad, Telangana, India

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technique, as well as the use of various nanoparticles like carbon nanofibers, montmorillonite organoclays, nano silica, and carbon nanotubes (CNTs) to enhance the thermal behavior of polymer composites [5][6]. Researchers looked at how adding nano silica to carbon fiber/phenolic composites affected its ablation and thermal properties, and they found that all of the resulting composites were more thermally stable than the pure matrix. High tensile strength and modulus: Due to the carbon fibers' high mechanical properties and the phenolic resin's ability to bind them together [7][8][9]. Improved hardness and wear resistance with the addition of ZrO2 particles, which would enhance the composite's surface hardness and resistance to abrasion . Phenolic resin's high thermal stability would help maintain the composite's mechanical properties atelevated temperatures [10]. The ZrO2 particles and phenolic resin would contribute to the composite's resistance to corrosive environments. The fabrication process has been shown in Figure 1.

Significance of Work

ZrO2 helps to improve the performance of composites by providing an additional layer of protection against wear, making it more resistant to shock and impact, and by preventing it from warping and deforming at high temperatures. Zirconium oxide is an excellent filler for composite materials because it has a low coefficient of thermal expansion, which reduces the material's tendency to crack and deform at high temperatures. Additionally, its high hardness and abrasion resistance make it an ideal material for improving wear resistance, toughness, and stability. ZrO2 has a strong ability to form bonds with fiber surfaces, which helps to create a strong bond between the matrix and reinforcement fibers. This helps to increase the stiffness and strength of the composite. ZrO2, being an amorphous phase, reduces the number of interfaces between the matrix and the reinforcement fibers, allowing for better bonding between the two can check in micro-structures. This helps to reduce the amount of stress in the composite, which leads to improved mechanical properties.

The curing temperature, pressure, and resin content all affect the curing process of the composite, which in turn affects the final mechanical properties of the composite. The curing temperature affects the rate and extent of curing, the pressure affects the uniformity of the curing process, and the resin content affects the strength of the material. The curing temperature and pressure affect the resin's ability to form strong bonds between the fibers. The resin content affects the overall strength of the composite, as well as the stiffness and toughness of the material. Additionally, the curing process itself affects the composite's mechanical properties. ZrO2 helps to increase the interfacial shear strength between the matrix and the reinforcement fibers, resulting in a stronger bond between the two. It also helps to reduce the tendency of the matrix to crack, which helps to prevent delamination. The mechanical properties of the composite are determined by the properties of the constituent materials, such as their strength, stiffness, and toughness. These properties affect the fracture toughness and crack propagation behavior of the composite, because it determines the amount of energy required to propagate cracks. The addition of ZrO2 and phenolic resin to the composite material increases the thermal stability, as ZrO2 is an excellent thermal insulator and phenolic resin provides a strong bonding between the ZrO2 particles. As a result, the composite material is better able to withstand higher operating temperatures without degrading.

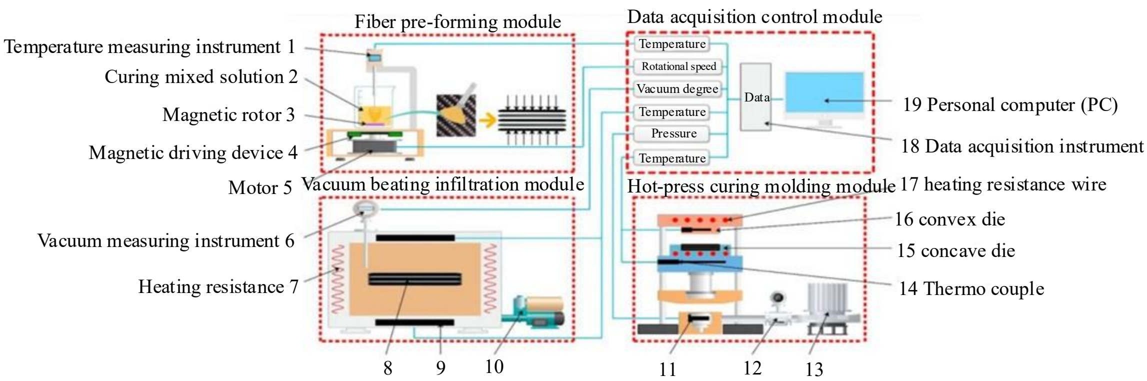

Figure 1. Schematic diagram of the vacuum infiltration hot-presses forming experimental system.
1—Temperature measuring instrument; 2—curing mixed solution; 3—Magnetic rotor; 4—magnetic driving device; 5—Motor; 6—vacuum measuring instrument; 7—heating resistance; 8—Composite material; 9—temperature sensor; 10—vacuum pump; 11—pressure sensor; 12—solenoid valve; 13—hydraulic cylinder; 14—Thermo couple; 15—concave die; 16—convex die; 17— heating resistance wire; 18— data acquisition instrument; 19—personal computer (PC).

FABRICATION PROCESS AND MATERIALS

Fabrication of composites started with surface modification of the resin and filler. A solution of silane coupling agent and acetic acid was made by mixing in 1:5 weight ratios respectively. Later ZrO_2 is mixed in prepared solution in 1:1 proportion by weight using ultrasonication process for 30 min. After that, ZrO2 particles were sieved and dried at 110 ℃ for 3 h. Figure 2 illustrates surface treatment of ZrO2 particles. Final formulation based on previous literature and fabrication of mono and hybrid composites Mixing ratio of epoxy and the hardener is 100:25 by weight[11,12,13]. After fixing the reinforcement, filler and epoxy wt.%, hand lay-up process is implemented for stacking the carbon fabric with proper phenol coating. A 300x300X1.5mm mould has been used to prepare the laminates with carbon fibre added with ZrO_2 fillers[14,18,19].Taguchi method of DOE with 4 levels and three parameters with L16 orthogonal array considered for experimentation and optimized the parameters using Mini-Tab software. SEM analysis carried out for the best samples to check the filling of ZrO_2 affect on mechanical properties of Tensile and Flexural strengths.

Materials

Carbon fibre, phemolic resin and Nano ZrO2 particles used as fabricated materials in the present work the properties of the materials were briefly discussed.

Carbon fiber

Carbon fibers have a low density, a high melting point, and great tensile and compressive strength [15,16,17]. As seen in table1, the carbon fiber's characteristics.

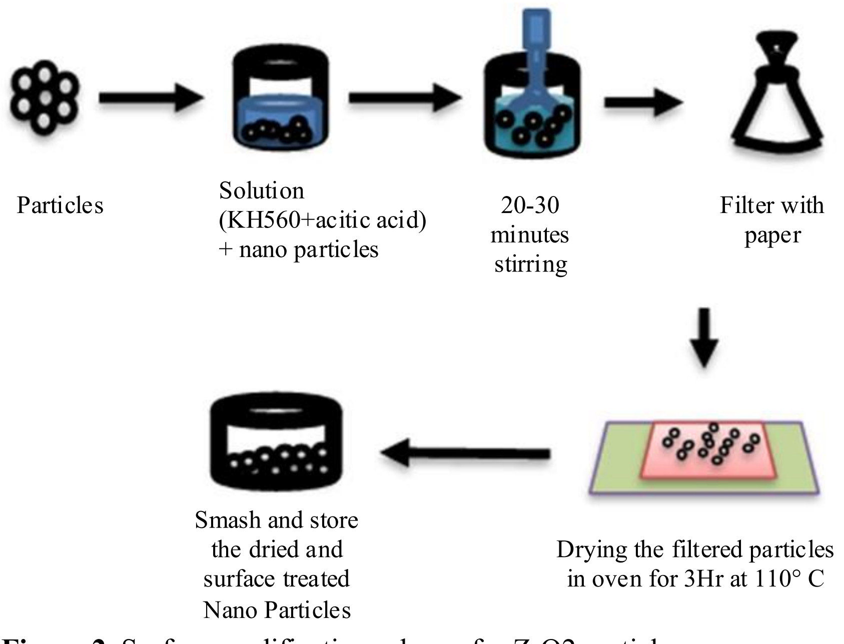

Figure 2. Surface modification scheme for ZrO2 particles.

Table 1. The characteristics of the carbon fiber

Type of fiber	Density (g/cm^3)	Coefficient of thermal expansion (x10-6 /°C)	Thermal conductivity (W/m.K)	Tensile strength (GPa)	Young modulus (GPa)

carbon fiber (rayon-based	1.8-1.9	0.4-0.75	20-80	3.4-6.2	220-450

Phenolic resin

The phenolic resin was chosen as the matrix phase in this investigation because, in comparison to other polymeric matrixes and resin qualities shown in table 2, it has excellent ablation resistance, heat resistance, and a high char yield.

Zirconium dioxide

Zirconium dioxide is one of the UHTC materials that has been the subject of the most research. Zirconia, which has a monoclinic phase structure at ambient temperature, can undergo phase transitions to tetragonal and cubic structures when heated to certain temperatures. The ZrO2 characteristics shown in Table 3.

TAGUCHI DESIGN OF EXPERIMENTS FOR VACUUM BAG INFUSION METHOD

Taguchi orthogonal design creates the experimental plan using a specific set of predetermined arrays known as orthogonal arrays (OAs). By using these standard arrays, we can get complete data on every variable that has an effect on the process's efficiency. Consideration of the experimental parameters and their levels guides the selection of the appropriate OA from a pool of predetermined possibilities. Table 4 shows the three-level factors utilized in the current experiment, and Table 5 shows the matching orthogonal array, L16. Using analysis of variance (ANOVA), we may find out which process parameters had a major impact on the quality parameter.

Table 2. Properties of as-received phenolic resin.

Type of resin	Density (g/ml)	Thermal conductivity (w/mK)	Viscosity (cp) @300 °C
Phenolic Resin	1.32	0.25	150-350

Table 3. Properties of synthesized ZrO_2.

Filler	Density (g/m^3)	Thermal conductivity (w/mK)	Melting temperature (°C)	size (µm)
Phenolic Resin	5.65	1.7	2700	5

Table 4. Parameters and levels for experimentation.

Parameter	Level-1	Level-2	Level-3	Level-4
Filler content (w%)	0.5	1	1.5	2
Pressure (bar)	10	15	20	25
Curing temperatures	30	50	70	90

Table 5. DOE for experimentation.

	C1	C2	C3	Filler content (w%)	Pressur e (bar)	Curing temperatures (^{0}C)
	A	*B*	*C*			
1	1	1	1	0.5	10	30
2	1	2	2	0.5	15	50
3	1	3	3	0.5	20	70
4	1	4	4	0.5	25	90
5	2	1	2	1	10	50
6	2	2	1	1	15	30
7	2	3	4	1	20	90
8	2	4	3	1	25	70
9	3	1	3	1.5	10	70
10	3	2	4	1.5	15	90
11	3	3	1	1.5	20	30
12	3	4	2	1.5	25	50
13	4	1	4	2	10	90
14	4	2	3	2	15	70

15	4	3	2	2	20	50
16	4	4	1	2	25	30

Experiment Samples After Fabrication

The vacuum bagging method is used to heat and cure the carbon fibre phenolic components, allowing them to become strong and rigid. The vacuum bagging method also helps to reduce the formation of air bubbles, which can cause the components to become weak and unstable, the synthesized samples after curing shown in the Figure 3a and the coated laminates shown in the Figure 3b.

The phenol coating process on laminates shown in the Figure 4, This technique produces components with superior mechanical properties due to the uniform distribution of pressure and temperature. In addition to that, it helps in making the production process more efficient and affordable. Samples for testing after coating for tensile and flexural as per ASTM D3039 and ASTM D790 respectively shown in the Figure 5a and 5b.

In the flexural test, the materials are subjected to three-point bending to measures the flexural strength and modulus. The three-point bending test is more reliable to measure the flexural properties at a specific location of the specimen. During testing, the specimen subjected to all three types of stresses such as tensile, compressive and shear which produce net resultant of all three stresses. The resultant flexural strength, defined as the material resistance to deforming, represents the maximum stress attained by reinforcement on either tensile or compression side testing sample.

The X-ray diffraction pattern of the ZrO2 nanoparticles is used to identify the distribution of the nanoparticles in the phenolic coating on the CFRP laminates. This helps to determine the effectiveness of the phenolic coating in protecting the CFRP laminates from corrosion as shown in the fig6. It can be found that the characteristic peak of the prepared CF-ZrO2 hybrid and the single phase of ZrO2 correspond to each other, and the intensity of the diffraction peaks is high and sharp. Besides, the weak characteristic peak at $2\theta = 26^0$ is caused by the typical diffraction of amorphous carbon in the CF, and the intensity of the (002) crystal plane is very low, suggesting that the synthesized ZrO2 nanoparticles are decorated on the surface of CF.

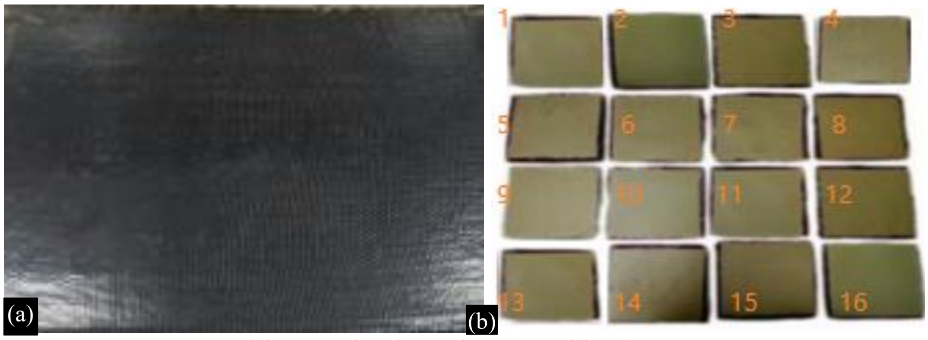

Figure 3. a)CFRP with ZrO_2 laminate b) Coated laminates.

Figure 4. Phenol coating progress for CFRP laminates.

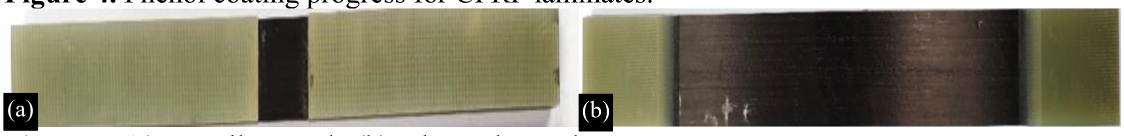

Figure 5. (a). Tensile sample (b). Flexural sample.

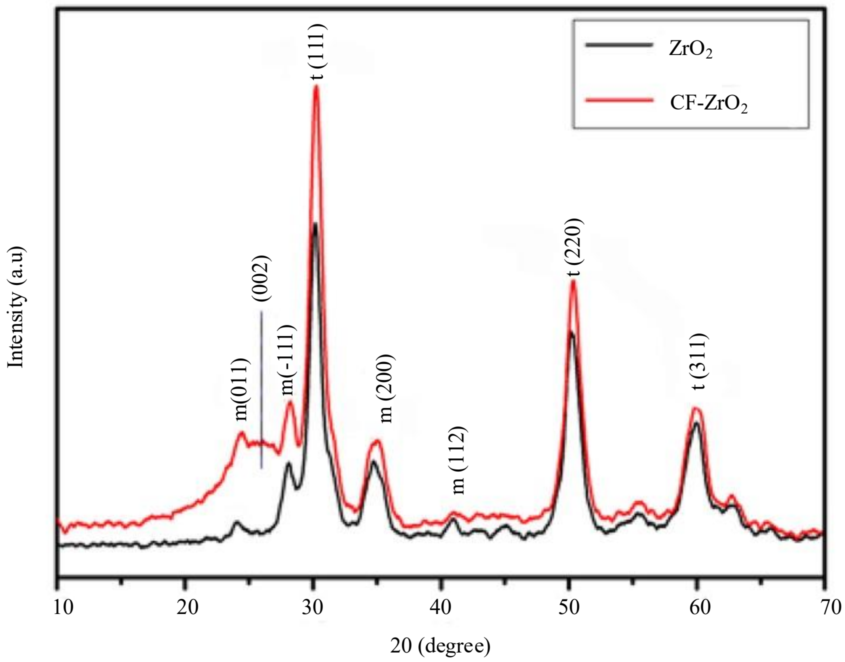

Figure 6. Typical X-ray diffraction pattern of ZrO2 nanoparticles and CF-ZrO2 hybrid.

RESULTS AND DISCUSSIONS

Experimental study results for laminated composites with phenol coated samples and the optimization results are given in the Table 6 for tensile strength and flexural strength as out put result for Analysis of variance using Mini-Tab Taguchi optimal prediction .

Table 6. Experiment results of tested samples with 1.5mm thickness.

S no	Filler content (w%)	Pressure (bar)	Curing temperatures (^{0}C)	Tensile strength (Mpa)	Flexural strength (Mpa)
1	0.5	10	30	150.6	91.1
2	0.5	15	50	161.1	93.4
3	0.5	20	70	169.6	94.9
4	0.5	25	90	174.2	96.6

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5	1	10	50	179.9	97.1
6	1	15	30	180.3	101.8
7	1	20	90	182.4	102.6
8	1	25	70	184.4	105.4
9	1.5	10	70	186.7	109.2
10	1.5	15	90	189.2	118.3
11	1.5	20	30	188.7	117.9
12	1.5	25	50	188.1	116.0
13	2	10	90	187.4	114.6
14	2	15	70	185.0	110.8
15	2	20	50	184.6	109.7
16	2	25	30	180.5	108.9

Taguchi Optimization With MINI-TAB

Taguchi methods achieve this through a two-stage optimization procedure.Step one is all about reducing variability, whereas step two is all about hitting the mark. Obtaining a maximum signal-to-noise ratio requires first adjusting all variables that significantly impact it to their maximum values. Mini-tab software used for the optimized output result combinations. The estimated model coefficients given in the Table7.

Pressure and curing temperatures are thesignificant parameters followed by the filler content as the results shown in Table 7, with the high F value 52.55 of variance in Table 8 the model accuracy is up to 91.49%.

The analysis of variance by means of results obtained in tensile and flexural there is a significant affect of curing temperature with an F value of 2.64 followed by the filler content given in the Table9.

Response table of signal to noise ratio and the means in Table 10 filler content always have its high significance while injected pressure and curing temperatures after fabrication giving equal impact on present laminates.

Table 7. Estimated Model Coefficients for SN ratios.

Term	Coef	SE Coef	T	P
Constant	***42.1588***	***0.04973***	***847.768***	***0.000***
A 1	-0.9280	0.08613	-10.774	0.000
A 2	-0.1871	0.08613	-2.173	0.073
A 3	0.7019	0.08613	8.149	0.000
B 1	-0.2083	0.08613	-2.418	0.052
B 2	0.0235	0.08613	0.273	0.794
B 3	0.0718	0.08613	0.834	0.436
C 1	-0.1033	0.08613	-1.199	0.276
C 2	-0.1078	0.08613	-1.252	0.257
C 3	0.0109	0.08613	0.127	0.903

Table 8. ANOVA for the S/N ratio

Analysis of Variance for SN ratios							**Model Summary**		
Source	***DF***	***Seq SS***	***Adj SS***	***Adj MS***	***F***	***P***	***S***	***R-Sq***	***R-Sq(adj)***
A	3	6.2381	6.2381	2.07937	52.55	0.000	0.1989	96.60%	91.49%
B	3	0.2475	0.2475	0.08249	2.08	0.204			

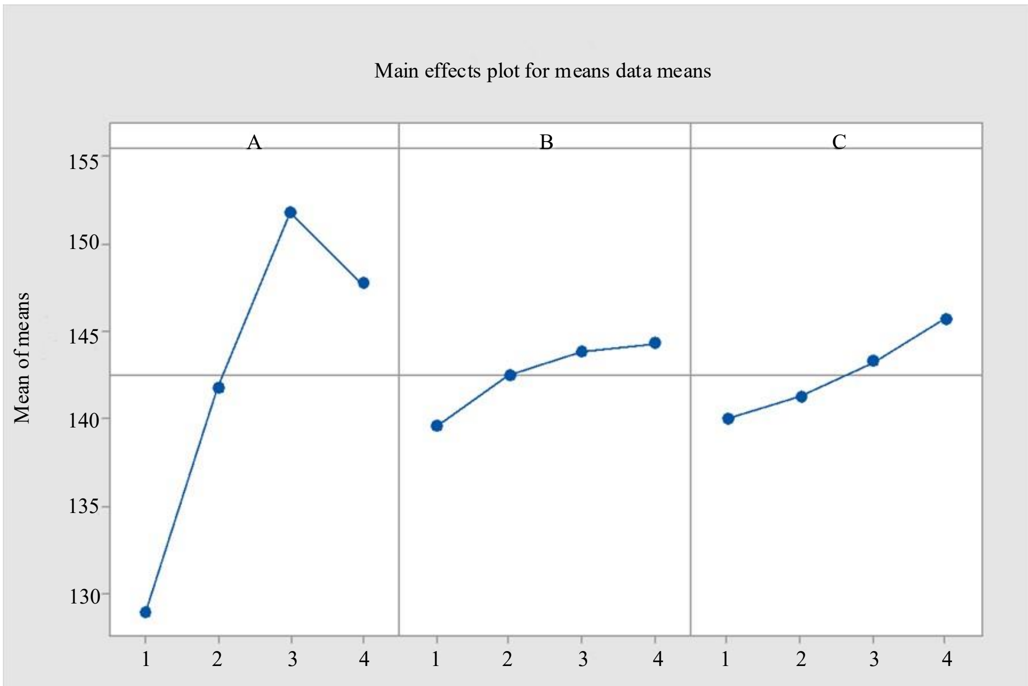
C	3	0.2500	0.2500	0.08332	2.11	0.201			
Residual error	6	0.2374	0.2374	0.03957					
Total	15	6.9729							

Table 9. Analysis of Variance for Means.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
A	3	1188.89	1188.89	396.297	42.32	0.000
B	3	53.39	53.39	17.798	1.90	0.231
C	3	74.12	74.12	24.706	2.64	0.144
Residual error	6	56.18	56.18	9.364		
Total	15	1372.58				

Table 10. Responses for S/N and Mean.

Response table for signal to noise ratios larger is better				Response table for means		
Level	***A***	***B***	***C***	***A***	***B***	***C***
1	41.23	41.95	42.06	128.9	139.6	140.0
2	41.97	42.18	42.05	141.7	142.5	141.2
3	42.86	42.23	42.17	151.8	143.8	143.3
4	42.57	42.27	42.36	147.7	144.3	145.7
Delta	1.63	0.32	0.31	22.8	4.7	5.7
Rank	1	2	3	1	3	2



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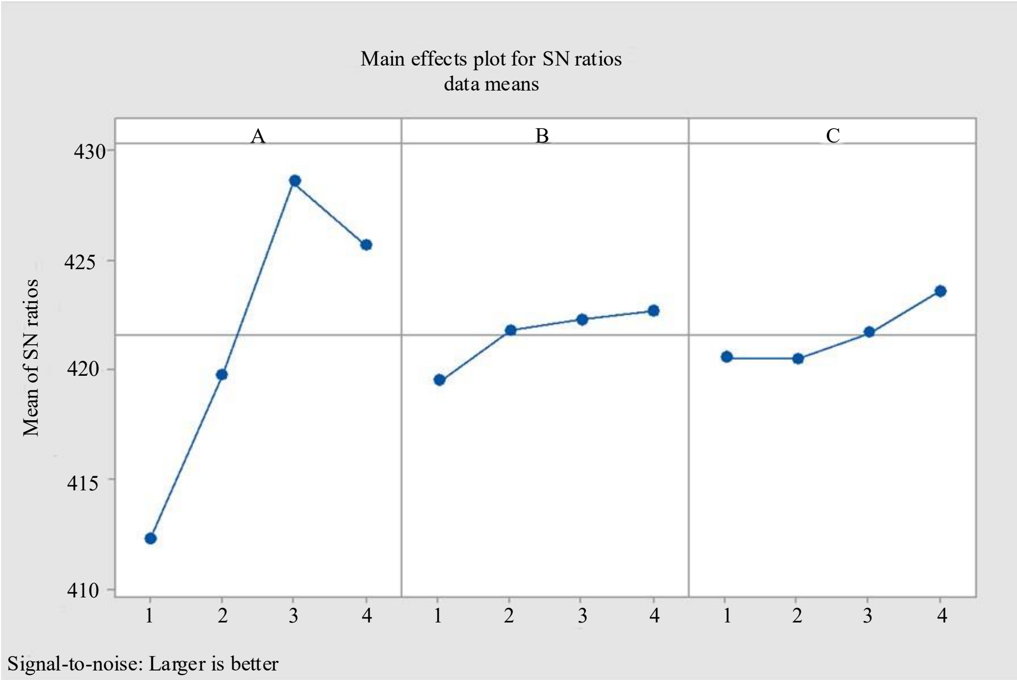

Figure 7. Main effects plot for Mean & S/N Ratio.

The main affective plots of Mean and S/N ratios in Figure 7 the affective composition is A2(1%) B2(15bar) and C3 (70℃)are the best parameters for the fabrication of fibre phenol composite. The factors can be deviated for the results so the standard deviation affects with estimated coefficients given in Table 11.

There is a least deviation found with filler increment, the modeled filler parameters are better , much variance found in the curing temperature as per the results in Table 11, the standard deviation variance observed in Table 12. The deviation in another two factors with a summary of 70.72 accuracy with pressure given most variation in vaccum bagging method . Low residuals in Standard deviations found in Table 13, the deviation in incremental pressure is very low of 1.5 of its means and much varied result is again filler composition with 7.18.

The standard deviation plot in Figure 8 also showing that the variaton in results from filler parameter A1 to A2 is more and decreased gradually and the pressure is more affective between B2 to B3 and curing temperatures are good between C2 to C3.

Table 11. Estimated Model Coefficients for StDevs.

Term	Coef	SE Coef	T	P
Constant	***52.3436***	***0.8407***	***62.260***	***0.000***
A 1	-2.9345	1.4562	-2.015	0.091
A 2	4.2426	1.4562	2.914	0.027
A 3	-0.8485	1.4562	-0.583	0.581
B 1	-0.6187	1.4562	-0.425	0.686
B 2	-0.8485	1.4562	-0.583	0.581
B 3	0.7248	1.4562	0.498	0.636
C 1	-2.7754	1.4562	-1.906	0.105
C 2	0.2475	1.4562	0.170	0.871
C 3	1.6440	1.4562	1.129	0.302

Table 12. ANOVA for St. dev.

Analysis of Variance for Standard deviation							**Model summary**		
Source	***DF***	***Seq SS***	***Adj SS***	***Adj MS***	***F***	***P***	***S***	***R-Sq***	***R-Sq(adj)***
A	3	110.170	110.170	36.723	3.25	0.102	3.3629	70.72%	26.80%
B	3	8.717	8.717	2.906	0.26	0.854			
C	3	44.993	44.993	14.998	1.33	0.351			
Residual Error	6	67.855	67.855	11.309					
Total	15	231.735							

Table 13. Response Table for Standard Deviations.

Level	**A**	**B**	**C**
1	49.41	51.72	49.57
2	56.59	51.50	52.59
3	51.50	53.07	53.99
4	51.88	53.09	53.23
Delta	7.18	1.59	4.42
Rank	1	3	2

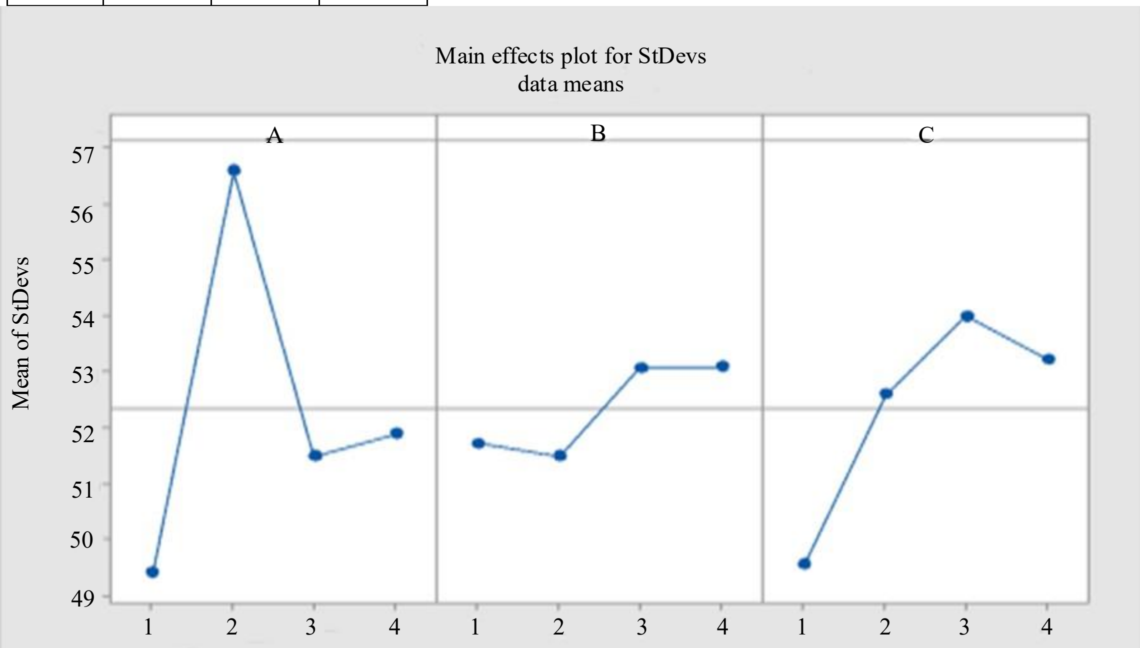

Figure 8. Effects plot for St Devs.

The prediction Table given in the Table 14 for better selected combination in the present modeled work, the experiment sample 10 given better predicted values compare to others.

Table 14. Taguchi prediction.

	Parameters			**Taguchi prediction**			
	A	***B***	***C***	***S/N Ratio***	***Mean***	***St Dev***	***Ln (St Dev)***
1	1	1	1	40.9192	123.425	46.0150	3.82184
2	1	2	2	41.1465	127.6	48.8080	3.88471

3	1	3	3	41.3135	130.925	51.7779	3.94398
4	1	4	4	41.5440	133.8	51.0354	3.92929
5	2	1	2	41.6555	137.488	56.2150	4.02386
6	2	2	1	41.8918	139.138	52.9623	3.96389
7	2	3	4	42.2436	146.138	58.1949	4.06960
8	2	4	3	42.0956	144.188	58.9727	4.08486
9	3	1	3	42.6633	149.525	52.5204	3.95766
10	3	2	4	43.0843	154.85	51.5304	3.94414
11	3	3	1	42.8292	150.475	49.4444	3.89983
12	3	4	2	42.8659	152.2	52.4850	3.96154
13	4	1	4	42.5638	147.862	52.1491	3.95075
14	4	2	3	42.6063	148.363	52.6795	3.96718
15	4	3	2	42.5359	147.663	52.8562	3.96932
16	4	4	1	42.5817	146.862	49.8510	3.90818

CONCLUSION

The effects of hybridization and the stacking sequence of hybrid composite materials under pressure based impacts were investigated. Work focused on tensile and mechanical properties were optimized. The mechanical properties of a composite material are determined by the properties of the fibers and the resin used to bind them together. The strength and stiffness of the fibers and the strength and stiffness of the resin determine how much energy is needed to cause a crack to propagate through the material, which in turn affects its fracture toughness and crack propagation behavior.The better samples need to test for SEM structures for the better clarity. 1.5% addition with 15 bar and curing temperature of 90°C given better results in mechanical properties compared with other samples. The samples were subjected to a visual inspection to check for the presence of voids and defects. The samples were then tested to measure their strength and stiffness. Finally, the samples were need to test further for their ability to withstand extreme thermal conditions.

Nomenclature

Density	**Coefficient of thermal, expansion (x10-6 /°C)**	**Thermal conductivity (W/m.K)**	**Tensile strength (GPa)**	**Young modulus (GPa)**
Viscosity (cp) @300 °C				
150-350				

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