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OPTIMIZATION OF PROCESS PARAMETERS FOR CARBON FIBRE PHENOLIC COMPOSITES WITH ADDED NANO ZrO_2 AND GRAPHENE OXIDE FILLERS

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Abstract: Carbon fibers are one of the high strength carbon laminates used in various aeronautical and industrial applications. Improvement in mechanical properties is always recognized research in materials, carbon fiber laminates also need to improve the properties in specific complex shapes. To maximize the extent of research Nano fillers of ZrO_2 and a substitution of graphene oxide added to the layers in vacuum impingement method. Previous researches stated that graphene filler with 1% given better results when mixed with carbon fiber, the research varied ZrO_2 filler only. Taguchi method of optimization with L9 experimentation conducted to optimize better parameters after graphene addition. All samples coated with phenol resin with 0.5mm thickness for stable results. Mini-tab used to optimize the Taguchi prediction and the results showing that 1.5 ZrO_2 at 20bar and 90 degrees curing temperature given good results compare to other experiments.
Key words: Composites, Carbon fiber, Phenol, ZrO_2 Nano, Graphene nano, Mechanical properties.

1. INTRODUCTION

Composites are engineered materials comprised of two or more elements with distinct qualities, and their combination leads to such materials having desired properties. These properties include high strength, excellent stiffness, no catastrophic failure, low thermal expansion, resistance to the chemical and environmental factors, which of them to be used in a myriad of applications such as electrical equipment, transportation, construction, sports, defence and aerospace industries Phenolic is a thermoset polymer that is widely used in polymer composite systems. Phenolic is ubiquitous in electrical applications, automobiles, aerospace, military weapons, and sports goods due to its superior mechanical strength, heat resistance, dimensional stability, and high chemical, acid, and water resistance Phenolic is also one of the essential ingredients used in friction material as it is a strong binder, and high-strength binders improve the overall friction materials' performance There is literature that discusses the improvement of phenolic's properties when reinforced with fillers such as alumina, calcium carbonate, silicon carbide, talc, copper, carbon black, graphite, and CNTs The use of a single filler in polymer matrix composites (PMCs) can no longer meet the demands of advanced PMC applications, which require multifunctional properties from a combination of two or more fillers and a hybrid system Attempts to combine several fillers or hybrid materials to form composites are not recent endeavours. For example, clay, iron, mica, and carbon were used as fillers in automotive brake pads in these applications, more than one filler component is usually used (hybrid). Carbon phenolic materials generally having low density and high ductility; Because of its ductile nature the process of carbon phenolic materials addition to any other metals / polymers become complicated. even though there is some evidence to improve mechanical properties of carbon phenolic material by literature review, no such evidences were found to overcome the actual problem in a proper method of preparation. There is some research about nano addition to carbon phenolics density may increase but there is a small hype in mechanical properties.

2. Literature Review

Adnan Amjad **[1]** This reviews the most recent development on the effect of different nanofiller addition and surface treatment on the mechanical, thermal, and wetting behaviour of NFRPCs. It concludes that the fibre surface treatment and nanofillers in natural fibre polymer composites positively affect mechanical, thermal and water absorption properties Abdulganiyu **[2]** The effects of microfiller addition on the flexural properties of carbon fiber reinforced phenolic (CFRP) matrix composites were investigated. The CFRP was produced using colloidal silica and silicon carbide (SiC) micro fillers, 2 D woven carbon fibers, and two variants of phenolic resole (HRJ-15881 and SP-6877). The resins have the same phenol and solid content but differ in their viscosities and HCHO (formaldehyde) content. Agnieszka Slosarczyk, **[3]** The article presents the synthesis of silica aerogel from a much cheaper precursor of water glass that was reinforced with short pitch carbon fiber by way of ambient pressure drying. Before being added to the silica gel, the carbon fibers were surface modified to increase adhesion at the interfacial border. Dr. N. Kishore Nath **[4]** A Composite Material is a macroscopic combination of two or more distinct materials, having a recognizable interface between them. Composites are used not only for their structural properties, but also for electrical, thermal, tribological, and environmental applications. Feng Xu et al **[5]** In this study, the surface-decorated ZrB_2/SIC and its modified carbon fabric reinforced phenolic composites have been successfully prepared. The self-modification mechanism of the surface-decorated ZrB_2/SIC particles was characterized. **Jiuqiang Song [6]** In their study to effects of ZrSi2 on the vulcanization, mechanical and ablation resistance properties of the composites were also investigated. The results showed that the introduction of ZrSi2 decreased the vulcanization time of silicone rubber. FTIR spectra showed that ZrSi2 did not participate in reactions of the functional groups of silicone rubber. K. Senthilkumar, et al **[7]** Phenolic resins are brittle at room temperature, hence to obtain suitable properties such as enhanced mechanical, thermal, etc., the Phenolic resins are preferred to incorporate with fiber and/or filler. The Phenolic based composites could be used to make complex geometries as they possess high strength and stiffness with high impact properties; hence, they would be the best alternative for metals. L. Paglia, et al **[8]** In this work, two different kinds of carbon-Phenolic ablators with a density of 0.3 g/cm^3 were manufactured and their mechanical and thermal properties were experimentally evaluated. The thermal protection performances of the developed ablators were assessed in a hypersonic plasma wind tunnel facility, setting representative enthalpy and heat flux conditions (6 and 13 MW/m^2), consistent with atmospheric reentry missions from high energy orbits. Ren, Yu; Lin et al **[9]** Boron-modified high-orthodox Phenolic resins (BPRs) were prepared under normal pressure by using Phenol and formaldehyde as raw materials, zinc acetate, and oxalic acid as

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catalysts, and boric acid as a modifier. Boron-modified Phenolic fibers (BPFs) were prepared by melt spinning and curing in a mixture of formaldehyde and hydrochloric acid, followed by a heat treatment under high temperature. Tingli Yang et al **[10]** Carbon fiber fabric-reinforced Phenolic resin composites are widely used as thermal protection materials for thermal protection systems in hypersonic vehicles and capsules. In this work, carbon fiber fabric-reinforced boron Phenolic resin composites modified with MoSi2 and B4C were prepared via a compression molding technique. The high-temperature performance of the composites as well as the oxidation behavior of the carbon fibers was studied. Umar Farooq et al **[11]** The influence of Nano diamonds (NDs) on the thermal and ablative performance of carbon fiber-reinforced–epoxy matrix composites were explored. The ablative response of the composites with 0.2 wt. % and 0.4 wt. % NDs was studied through pre-and post-burning morphologies of the composite surfaces by evaluation of temperature profiles, weight loss, and erosion rate. Yang T et al **[12]** Carbon fiber fabric-reinforced Phenolic resin composites are widely used as thermal protection materials for thermal protection systems in hypersonic vehicles and capsules. In this work, carbon fiber fabric-reinforced boron Phenolic resin composites modified with $MoSi_2$ and B_4C were prepared via a compression molding technique. The high-temperature performance of the composites as well as the oxidation behavior of the carbon fibers was studied. Wei Yang et al. **[13]**In this work, the above performances of the composites were characterized and analyzed. Results reveal that the addition of $MoSi_2$ decreases the graphitization temperature of glass carbon of BPR pyrolysis, promoting the formation of a more ordered structure of the glassy carbon during pyrolysis. Xu F, Zhu S et al. **[14]** In their study, the surface-decorated ZrB_2/SiC and its modified carbon fabric reinforced Phenolic composites have been successfully prepared. The self-modification mechanism of the surface-decorated ZrB_2/SiC particles was characterized. The mechanical performance and ablation behavior of the composites were investigated. Yang, Guangyuan et al.**[15]** Carbon fiber reinforced composite (CFRP) has been widely used in a lot of areas with its distinguished properties, especially mechanical properties. However, both carbon fibers and polymer substrate cannot resist high air temperature environment, limiting the application of CFRP, such as in the aerospace fields.

3. METHODOLOGY

When compared to the hand layup method, the hoover bag infusion has many advantages. A better fiber-to-resin ratio, reduced resin demand, consistent resin use, and limitless setup time are some of the benefits. Vacuum bagging improves the properties of composite materials while reducing drying time and voids.

Vacuum bags can remove surplus resin, but how much depends on factors including processing time, resin type, and reinforcing. of the vacuum bagged composite as seen using scanning electron microscopy. By combining polyester resin and hardener in the correct proportions, enough resin was produced. A gradual application of carbon fibre resin formed the composite. We made sure the air wouldn't get trapped and form blowholes. Vacuum bagging and the vacuum pump worked together to create a uniform, one-way vacuum. This ensured that the binders were properly distributed during the reinforcement. Thus, the composite produced had minimum defects. During manufacturing of the composite, no anomalies occurred due to error although nano-cracks were observed at specific places

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in the composite. Then, no-cracks occurred because of stress during the thermally induced expansion (exothermic reaction) of the binder during the setting of the adhesive

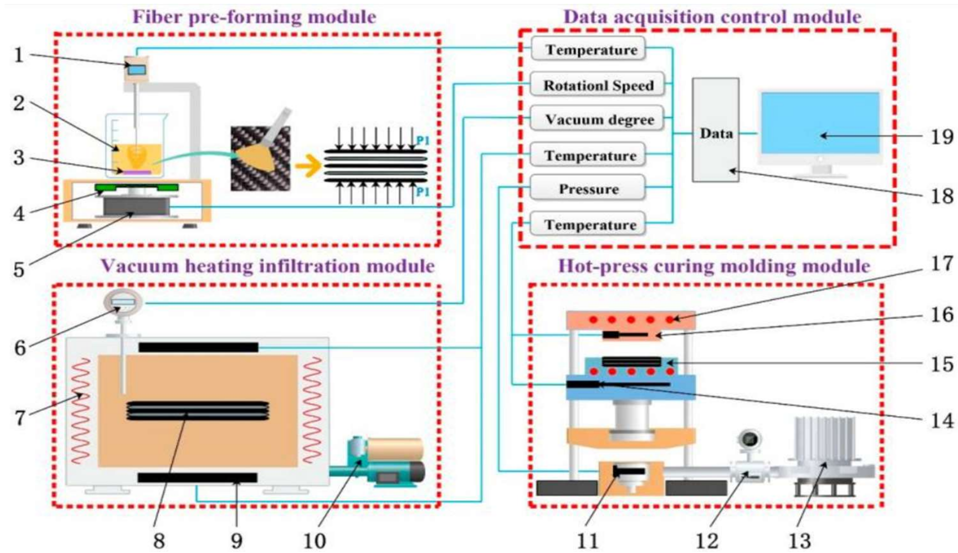

Figure:1 Schematic diagram of the vacuum infiltration hot-presses forming experimental system (VIHPS). 1—temperature measuring instrument; 2—curing mixed solution; 3—magnetic rotor; 4—magnetic driving device; 5—motor; 6—vacuum measuring instrument; 7—heating resistance; 8—composite material; 9—temperature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

3.1 Fabrication process

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 °C for 3 h. Figure 1 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. 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. Taguchi method of DOE with 3 levels and three parameters with L9 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.

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Figure 2: Surface modification scheme for ZrO2 particles.

Carbon Fiber:

Carbon fibers possess low density, excellent tensile and compressive strength, and high char strength due to the relatively high melting point.

Table:1 Properties of as-received carbon fiber as procured details

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:

In current study, the phenolic resin was used as a matrix phase due to its good ablation resistance, thermal resistance and high char yield compare to other polymeric matrix

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

Zirconium Dioxide:

Most studied ceramics among the UHTC materials is zirconium dioxide. At room temperature the zirconia, be -have as a monoclinic phase structure and transitions to tetragonal and cubic phase structure when treated to some high temperature

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Table 3: Properties of synthesized ZrO2

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

Graphene oxide:

It is used in nanocomposite materials, polymer composite materials, energy storage, biomedical applications, and catalysis, and as a surfactant with some overlaps between these fields.

Table 4: Properties of synthesized Graphene oxide

Filler	Density (g/m^3)	Thermal Conductivity (W/m·K)	Melting temperature (°C)	size (µm)
Graphene oxide	2.267	~5000	4510	4

For each samples a standard amount of 1% graphene oxide added in fabrication process

3.1.1 Taguchi analysis of Vacuum Bag Infusion Method parameters

In this process parameters can be investigated by ANOVA to verify the parameters that significantly affected the quality characteristic. We conducted the ANOVA analysis for a standard filler material and the properties were obtained as follows

Table 5: Parameters and levels for experimentation

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

3.2 Selection of orthogonal array

Taguchi orthogonal design uses a special set of predefined arrays called orthogonal arrays (OAs) to design the plan of experiment. These standard arrays stipulate the way of full information of all the factors that affects the process performance. The corresponding OA is selected from the set of predefined OAs according to the number of factors and their levels that will be used in the experiment. For the present experimental work, three factors with their three levels are used for which the corresponding orthogonal array is L9 which is shown in Table.

Table 6: DOE for experimentation

	C1	**C2**	**C3**
	A	**B**	**C**
1	1	1	1
2	1	2	2

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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

4. Results and Discussions

Experimental study results for laminated composites and phenol coated with graphene oxide samples, along with the optimization results, are presented in the table.

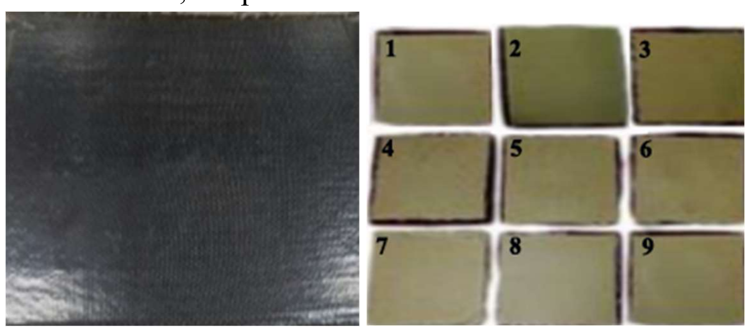

Figure 3: a) CFRP with ZrO_2 laminate b) Coated laminates

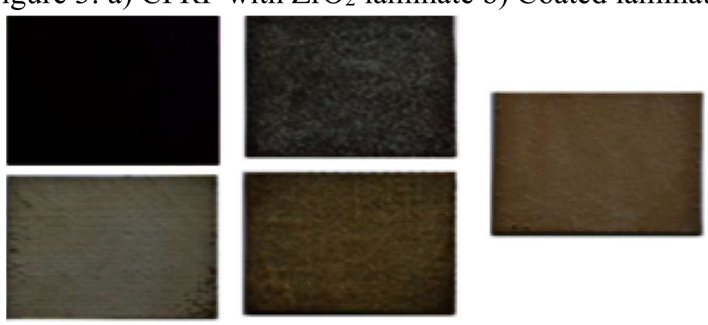

Figure 4: Phenol coating progress for CFRP laminates

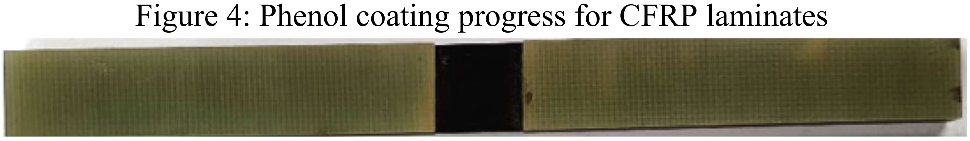

Figure 5: Tensile sample

Figure 6: Flexural sample

In the flexural test, Three-point bending is used to measure the modulus and flexural strength of the materials. For more accurate measurement of flexural properties at a specific specimen location, the three-point bending test is recommended. The specimen was tested under tensile, compressive, and shear stresses, which together produce a net resultant of all three. The maximum stress achieved by reinforcing the tensile or compression side of the testing sample is represented by the resultant

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flexural strength, which is the material's resistance to deformation.

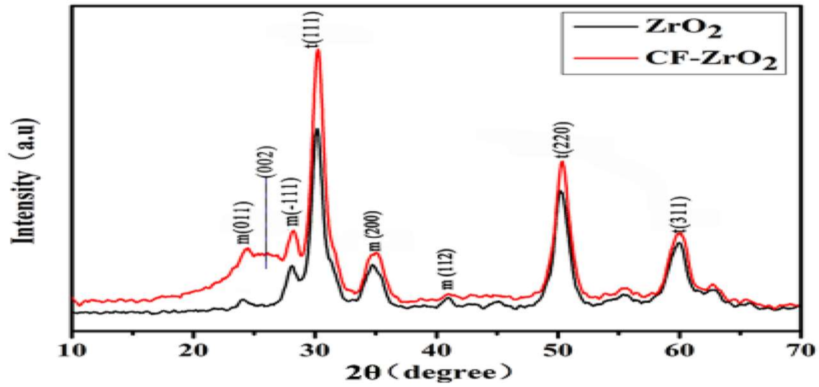

Figure 7: Typical X-ray diffraction pattern of ZrO2 nanoparticles and CF-ZrO2 hybrid

The prepared CF-ZrO2 hybrid and the single phase of ZrO2 show a correspondence in their distinctive peaks, and the diffraction peaks are crisp and intense. Additionally, the usual diffraction of amorphous carbon in the CF is responsible for the faint characteristic peak at 2θ = 260. The low intensity of the (002) crystal plane implies that the synthesised ZrO2 nanoparticles and graphene oxide are adorned on the surface of the CF.

Table 7: Experiment results of tested samples with 1.5 mm thickness

S.NO	Filler content (w%)	Pressure (bar)	Curing temperatures (^{0}C)	Tensile strength (Mpa)	Flexural strength (Mpa)
1	1	15	50	150.4	96.6
2	1	20	70	154.6	97.5
3	1	25	90	169.1	104.2
4	1.5	15	70	178.3	109.9
5	**1.5**	**20**	**90**	**183.6**	**119.4**
6	1.5	25	50	181.1	116.2
7	2	15	90	179.2	111.3
8	2	20	50	178.8	107.9
9	2	25	70	168.7	102.1

4.1 Taguchi optimization with Mini-tab

Taguchi methods do this by a two-step optimization process. The first step concentrates on minimizing variability, and the second focuses on hitting the target. First, set all factors that have a substantial effect on the signal-to-noise ratio at the level where the signal-to-noise is maximized. Mini-tab software used for the optimized output result combinations.

Table 8: Estimated Model Coefficients for SN ratios

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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 9: 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	6.2382	2.07937	52.55	0.000	0.1989	96.60%	91.49%
B	3	0.2475	0.2474	0.08249	2.08	0.204			
C	3	0.2500	0.2502	0.08332	2.11	0.202			
Residual Error	6	0.2374	0.2373	0.03957					
Total	15	6.9729							

Table 10: Analysis of Variance for Means

Source	DF	Seq SS	Adj SS	Adj MS	F	P
A	3	1189.88	1188.89	396.297	42.32	0.000
B	3	52.38	53.39	17.798	1.90	0.231
C	3	75.14	74.12	24.706	2.64	0.144
Residual Error	6	56.18	56.18	9.364		
Total	15	1372.58				

Table 11: Responses for S/n and mean

Response Table for Signal to Noise Ratios Larger is better	Response Table for Means

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Level	A	B	C	A	B	C
1	42.23	41.95	42.06	128.9	139.6	140.0
2	40.97	42.11	42.05	141.7	142.5	141.3
3	41.86	42.23	42.17	151.7	143.8	143.5
Delta	1.63	0.32	0.31	22.7	4.7	5.9
Rank	1	2	3	1	3	2

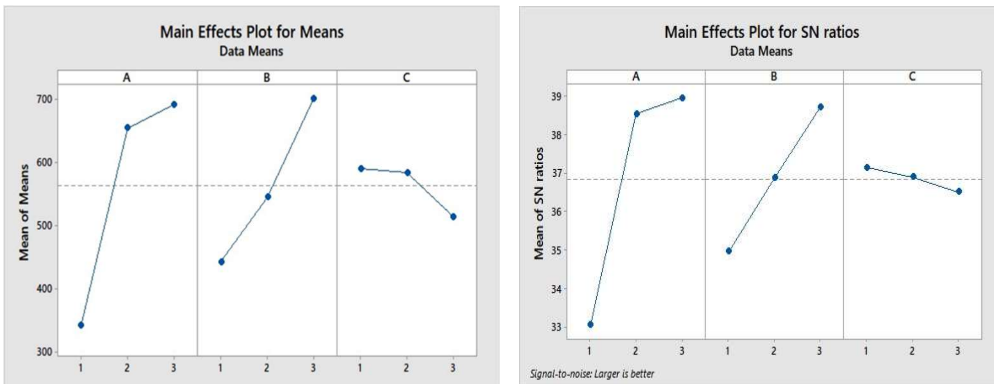

Figure:8 Main effects plot for Mean & **S/N Ratio**

Table 12: Estimated Model Coefficients for St Devs

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 13: ANOVA for St. dev

Analysis of Variance for Standard deviation	Model Summary

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Source	DF	Seq SS	Adj SS	Adj MS	F	P	S	R-Sq	R-Sq(adj)
A	3	111.170	110.170	36.723	3.25	0.103	3.3631	70.73%	26.81%
B	3	7.717	8.719	2.906	0.26	0.852			
C	3	43.993	44.901	14.998	1.33	0.352			
Residual Error	6	66.855	67.853	11.309					
Total	15	231.735							

Table14: Response Table for Standard Deviations

Level	A	B	C
1	49.40	51.73	49.34
2	56.58	51.51	52.56
3	51.52	53.02	53.98
Delta	7.13	1.52	4.42
Rank	1	3	2

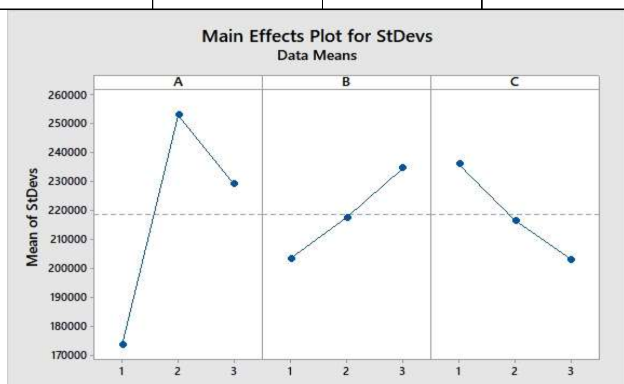

Figure 9: Main plots of standard deviation

Table 15: Taguchi prediction

	Parameters			Taguchi prediction			
	A	**B**	**C**	S/N Ratio	Mean	St Dev	Ln (St Dev)
1	1	1	1	41.8919	139.137	52.9622	3.96389
2	1	2	2	42.2437	146.138	58.1949	4.06960
3	1	3	3	42.0956	144.186	58.9727	4.08486
4	2	1	2	42.6633	149.525	52.5204	3.95766
5	**2**	**2**	**3**	43.0843	154.85	51.5304	3.94415
6	2	3	1	42.8292	150.475	49.4444	3.89982
7	3	1	3	42.8659	152.2	52.4850	3.96154
8	3	2	1	42.5638	147.862	52.1491	3.95075

9	3	3	2	42.6063	148.363	52.6795	3.96718

5. 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. Graphene oxide was added to improve the mechanical properties of the samples. By optimizing the strength and elasticity of the samples, the SEM structures would be clearly visible. For each samples a standard amount of 1% graphene oxide added in fabrication process to determine the better samples need to test for SEM structures for the better clarity. This optimization process was necessary to ensure that the graphene oxide was evenly distributed throughout the sample and that the sample was able to withstand the SEM process. 1.5% addition with 20 bar and curing temperature of 90℃ given better results in mechanical properties compared with other samples.

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