

EXPLORING COMPRESSIVE AND TENSILE PROPERTIES OF HYBRID CARBON BLACK–SILICA NANOFILLER-REINFORCED EPOXY NANOCOMPOSITE

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ABSTRACT

Carbon black (CB) has a relatively large surface area which leads to large interaction sites leading to efficient load transfer between the reinforcing CB nanoparticles and the epoxy matrix. This expected performance has not been obtained due to the difficulty of dispersing CB nanoparticles in epoxy matrix. The proper dispersion of CB in epoxy is crucial to achieving a good epoxy-CB nanocomposite; Lack of proper dispersion can result in reduced matrix-filler interaction and lack of dominant interfacial surrounding around the nanoparticle which must be overcome to produce an epoxy nanocomposite sensor with acceptable mechanical strength. This study investigates the compressive and tensile properties of epoxy nanocomposites reinforced with hybrid carbon black (CB) and silica nanofillers (Si). By systematically exploring the effects of CB weight fraction, silica weight fraction, mixing speed, mixing time, and the state of CB nanoparticles (as received, thermally treated, and thermally and chemically treated), this research aims to enhance the mechanical performance of these advanced materials. The nanocomposites were fabricated using Epox Amite 100 epoxy resin, VULCAN® XC72 carbon black, and silica nanoparticles with diameters of 10-25 nm. The optimal processing parameters were determined using response surface methodology (RSM) with a Box-Behnken design, encompassing 87 experimental runs. Statistical analysis revealed that the compressive strength of the nanocomposites improved significantly with the thermal-chemical treatment of CB nanoparticles, achieving a maximum compressive strength of 118 MPa at 5% CB-M and 20% silica content. Similarly, the tensile strength showed a marked enhancement, with the highest tensile strength recorded at 83.51MPa for the same hybrid filler combination. The results demonstrated that the interaction between the CB and silica nanofillers, along with the appropriate treatment of CB nanoparticles, led to superior mechanical properties. The quadratic regression models for compressive and tensile strengths exhibited high R-square values of 0.90 and 0.86, respectively, indicating good fits with the experimental data. These findings underscore the potential of hybrid CB-silica nanofillers in developing high-performance epoxy nanocomposites, suitable for applications in automotive, aerospace, civil engineering, and electronics. The study contributes valuable insights into the design and optimization of nanocomposite materials, emphasizing the importance of nanofiller treatment and synergistic effects in achieving enhanced mechanical performance.

Keywords: Epoxy Resin, Carbon Black, Silica, Nanocomposites, Hybrid, Compressive Strength, Tensile Strength

1 INTRODUCTION

Composites are planned materials containing two or more constituents with diverse qualities, and their blend leads to such materials having desired properties [1] Furthermore, a composite can also be classified as a nanocomposite if at least one of the reinforcement dimensions is in the nanometer range (10–200 nm) [2]. Nanocomposite shows many great promises in terms of higher mechanical performances and improved thermal, electrical [3], optical, and other properties [4]. Nanocomposites have

emerged as a promising solution across various engineering applications in the quest for advanced materials with superior mechanical properties [1, 5, 6], for example analysis shows a significant improvement both in static as well as fatigue properties when pristine boehmite nanoparticles were dispersed in epoxy [7], similarly noticeable effect on the elastic strength, toughness, ductility, and hardness performance of the epoxy nanocomposites was observed when hybrid reinforcement of Al_2O_3 nanoparticles [8] and graphite was used on the epoxy nanocomposites [9], epoxy-based nanocomposites reinforced with nanofillers have garnered significant attention due to their enhanced mechanical, thermal, and electrical properties. Polymers which are generally classified as thermoplastic or thermoset are essential matrix materials for fabricating advanced composites [10], During the past few decades, thermosets have gained widespread utilization as essential structural and engineering materials [6, 11, 12], Epoxy resins as a thermoset are known for their excellent adhesion, chemical resistance, and thermal stability [3, 13], form the matrix in which nanofillers are embedded to improve overall performance. Nanoparticles are the most essential components in the fabrication of nanocomposites, with sizes in nanometer ($1 \text{ nm} = 10^{-9} \text{ m}$) range [4], Nanoparticles continue to receive generous approval for their potential to reinforce polymers. Their high aspect ratio and large surface area improve the mechanical and thermal properties of polymer nanocomposites [6, 14] in comparison to conventional composites [15] at considerably low loading fractions owing to their greater interaction with the matrix [4]. The choice and combination of nanofillers or nanoparticle play a crucial role in determining the properties of the resulting nanocomposite [4]. Carbon black and silica nanoparticles are two widely studied nanofillers [16], each contributing unique benefits. Carbon black, known for its electrical conductivity and reinforcing capabilities [17, 18], enhances composites' structural integrity and durability [19, 20]. On the other hand, silica nanoparticles offer excellent thermal stability and mechanical strength due to their rigid and chemically stable nature [16, 21]. Combining these two nanofillers to create hybrid nanocomposites can potentially harness both advantages, leading to superior mechanical properties [22, 23]. The treatment of nanofillers is a critical factor that can significantly influence the performance of the nanocomposites [24]. Untreated carbon black nanoparticles may exhibit poor dispersion within the epoxy matrix due to their tendency to agglomerate [14], resulting in suboptimal mechanical properties. Thermal treatment of carbon black nanoparticles can enhance their dispersion by reducing surface energy and removing volatile organic compounds. Further chemical treatment can introduce functional groups on the nanoparticle surfaces, promoting better interaction with the epoxy matrix [25]. These treatments are hypothesized to improve the interfacial bonding between the nanofillers and the matrix, thereby enhancing the mechanical properties of the nanocomposites [26]. Compressive and tensile strengths are critical parameters in evaluating the mechanical performance of materials. Compressive strength measures a material's ability to withstand compressive forces without failure, which is vital for applications subjected to high pressures and load-bearing conditions. Tensile strength, which assesses the material's resistance to breaking under tension, is crucial for applications requiring high durability and flexibility [8]. Enhancing these properties in epoxy nanocomposites through the strategic incorporation and treatment of hybrid nanofillers can significantly broaden their application range, from automotive and aerospace to civil engineering and electronics. Despite the potential benefits, optimizing the mechanical properties of epoxy nanocomposites using hybrid nanofillers presents several challenges [27]. Factors such as the dispersion of nanofillers within the epoxy matrix, the interaction between different types of nanofillers, and the processing conditions significantly influence the final properties. Previous studies have often focused on single types of nanofillers, providing limited insights into the synergistic effects of hybrid fillers and the role of nanofiller treatment. This study aims to systematically investigate the compressive and tensile strengths of epoxy nanocomposites reinforced with hybrid carbon black and silica nanofillers. By employing a response surface methodology (RSM) with a Box-Behnken design, we seek to understand the individual and interactive effects of key processing parameters—mixing speed, mixing time, carbon black weight fraction, silica weight fraction, and the state of carbon black nanoparticles (as received, thermally treated, and thermally and chemically treated)—on these mechanical properties. We hypothesize that optimal combinations of these parameters and the appropriate treatment of nanofillers will significantly enhance both compressive and tensile strengths, leveraging the synergistic effects of the hybrid nanofillers. The findings from this study will provide

valuable insights into the design and optimization of high-performance epoxy nanocomposites. Understanding the interplay between processing conditions, nanofiller characteristics, and treatment methods will guide the development of materials with tailored mechanical properties, meeting the demands of various advanced engineering applications. Furthermore, this research will contribute to the broader field of nanocomposite materials by demonstrating the potential of hybrid nanofiller systems and the importance of nanofiller treatment in achieving superior mechanical performance. In summary, this study explores the compressive and tensile properties of epoxy nanocomposites reinforced with hybrid carbon black and silica nanofillers. By systematically investigating the effects of key processing parameters and nanofiller treatments using a robust experimental design, we aim to unlock the full potential of these advanced materials, paving the way for their application in diverse high-performance environments.

2 METHODOLOGY

2.1 Materials

The epoxy resin used was supplied by Smooth-On with the commercial name of Epox Amite 100 Resin and 102 medium hardeners. The 102 medium hardener consists of triethylenetetramine (TETA) with propylene oxide (50-55%), polyoxopropylene triamine (POPTA) (20-25 %) and TETA (15-20%). The CB nanoparticle was obtained from CABOT under the commercial name of VULCAN[®] XC72 with a volume resistivity of 6 Ω -cm at 23°C. VULCAN[®] XC72 has an iodine number of 253 mg/g density of 1.7-1.9 g/cm³ and OAN of 174 cc/100g, The SiO₂ nanoparticle with 10-25 nm diameter was obtained from Zhengzhou Dongshen Petro Chemical Technology Co, Ltd. The analytical grade chemicals used for surface treatments of the nanoparticles include tetrasodium pyrophosphate (TSPP), formic acid (FA), 3 Isocyanatopropyltriethoxysilane (IPTES), 3-Aminopropyltrimethoxysilane (APTES) and isophorone diisocyanate (IPDI).

2.2 Production of Epoxy Resin/ Carbon Black Nanocomposite

Three variations of CB which include as received CB, thermally treated CB and chemically modified thermally treated CB are used as one of hybrid nanofillers. 170g of epoxy was poured into beaker and preheated for 30 minutes at 40°C to improve viscosity. 20% nano silica with respect to CB%wt was measured and added to the beaker containing 170g of epoxy. 10 ml of IPDI was measured using a syringe and placed into the epoxy containing beaker, also added is 1.5ml of IPTES. The mixture was stirred manually until a homogenous mixture was achieved. 12.5%wt of CB with respect to epoxy resin content was placed into another beaker containing 250 ml of acetone, 4.6% of APTES with respect to %wt of CB was also added. 5%wt of TSPP with respect to CB %wt was added to the acetone containing beaker, FA was added gradually using a syringe until the PH value of the mixture is 4. The content of the beaker is manually mixed before the sonication process. Sonication was done with an ultrasonic cleaner model 008 with an ultrasonic power of 50w and a frequency of 40 KHz. The sonication process proceeded for 15 minutes at a time and a break period of 2 minutes between intervals. After 60 minutes of sonication, the content of the beaker containing acetone was mixed with the content of epoxy-containing beaker in a new beaker, the content is stirred manually until a homogenous mixture was achieved as shown in Figure 1. The beaker was then placed on a magnetic stirrer, the temperature was set to 60°C and the speed was adjusted to 950 rpm. The magnetic stirring was made for 60 minutes while maintaining the temperature at 60°C to ensure proper dispersion. Further stirring was done by the use a mechanical mixer at 600 rpm for 30 minutes; the mechanical mixer use was a pensonic PM-112 hand mixer with an electrical power of 120 w and a 5-speed selection. After the mixing process, acetone was evaporated using a hot air blower and by increasing the temperature of the magnetic stirrer to 75°C to guarantee thorough evaporation of the solvent. The sample was then placed in the degassing chamber; the vacuum pump was operated for 30 minutes to remove air bubbles trapped within the sample. The resin-hardener ratio of 100:29 as advised by the manufacturer was adopted; 49.3g of hardener was added to the degassed sample and blended manually till a homogenous mixture was attained. The nanocomposite was then transferred into the molds, the samples were post cured for 60 minutes at 100°C in an oven to complete the curing action the mold was designed

according to ASTM D638 for the tensile strength samples and ASTM D695 for the compressive strength sample. Figure1 shows the Production of Epoxy-Carbon Black-Silica Nanocomposite.

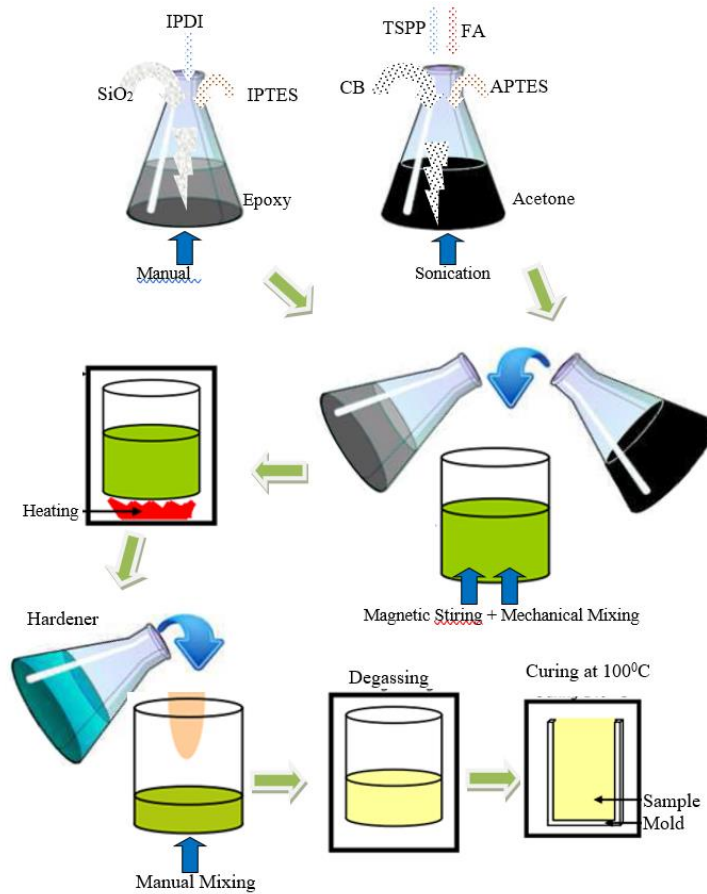


Figure 1. Production of Epoxy-Carbon Black-Silica Nanocomposite

2.3 Experimental Design

Response surface methodology was adopted to optimize the production of the nanocomposite; the empirical model was obtained using Box Behnken experimental design. Table 1 shows the Experimental Factors and their Levels.

Table 1. Experimental Factors and their Levels

Levels of Variables	Input Variables						State of CB Nanoparticle
	Magnetic Stir Speed (rpm)	Magnetic Stir Time (Hours)	CB Weight Fraction (%wt)	Silica Weight Fraction (%wt)			
1	700	1	5	0		As Received [1 0]	
0	950	2.5	12.5	10		Thermally Treated [0 1]	
1	1200	4	20	20		Thermally & Chemically treated [-1 -1]	

One (1) categorical and four (4) numeric experimental factors (state of CB nanoparticle, magnetic stir Speed, magnetic stir time, CB weight fraction and silica weight fraction) were used in the research as shown in Table 1. Each response was measured according to the standards specified in Table 2. A total of 87 runs in a single block were used with 5 central points for each categorical factor as is typical with Box-Behnken experimental design. Some of objectives of this research are to determine which factors are most significant on the response variables, and to determine where to set the dominant controllable factor so that

the response is almost near the desired optimal value. The Design-Expert 10 software was used in the generation of the experimental design and the analysis of the interactions of the factors.

Table 2. Response Variables

S\No.	Response	Unit	Standard
1	Tensile Strength	MPa	ASTM D638
2	Compressive Strength	MPa	ASTM D695

3 RESULTS AND DISCUSSION

3.1 Statistical Analysis of The Compressive Strength Response Data

Table 3 shows the statistical summary of the model with the R-square values, the coefficient of determination or R-square is the ratio of the explained variation to the total variation; it measures the degree of fit. A good model should possess an R-square ≥ 0.8 [28].

Table 3. Summary Statistics of Compressive Strength Model

Source	Std. Dev.	R-Squared	Adjusted R-Squared	R-Predicted R-Squared	PRESS	
Linear	6.62	0.8449	0.8333	0.8158	4165.53	
2FI	6.95	0.8591	0.8164	0.7452	5762.09	
Quadratic	5.89	0.9049	0.8681	0.8029	4457	Suggested
Cubic	5.69	0.9513	0.8769	0.5953	9154	Aliased

The evaluated R-square shows a high R-square value of 0.95 for the cubic model; however, it is aliased which means that the effects of each variable responsible for the signal difference are impossible to differentiate. A quadratic model with an R-square of 0.90 was suggestive of indicating a good fit between predicted values and the experimental data points. The R-square value indicates that 90% of the variation in compressive strength of the nanocomposite sample can be explained by the independent variables of the model equations. The predicted R-square of 0.80 is in logical agreement with the adjusted R-square since their difference is less than 0.2. The regression model equations are the following:

$$Y_{\text{comp(CB)}} = +57.65 + 5.19 * A + 0.11 * B - 3.81 * C - 0.40 * D + 5.11E^{-003} * AB + 0.21 * AC + 0.07 * AD + 2.67E^{-004} * BC + 9.33E^{-004} * BD - 0.02 * CD - 2.42 * A^2 - 7.52E^{-005} * B^2 + 0.02 * C^2 - 1.61E^{-003} * D^2 \quad (1)$$

$$Y_{\text{comp(CB600)}} = +65.56 + 5.08 * A + 0.11 * B - 4.06 * C - 0.31 * D + 5.11E^{-003} * AB + 0.21 * AC + 0.07 * AD + 2.67E^{-004} * BC + 9.33E^{-004} * BD - 0.02 * CD - 2.42 * A^2 - 7.52E^{-005} * B^2 + 0.02 * C^2 - 1.61E^{-003} * D^2 \quad (2)$$

$$Y_{\text{comp(CB-M)}} = +69.01 + 5.13 * A + 0.11 * B - 4.26 * C - 0.19 * D + 5.11E^{-003} * AB + 0.21 * AC + 0.07 * AD + 2.67E^{-004} * BC + 9.33E^{-004} * BD - 0.02 * CD - 2.42 * A^2 - 7.52E^{-005} * B^2 + 0.02 * C^2 - 1.61E^{-003} * D^2 \quad (3)$$

Where $Y_{\text{comp(CB)}}$ is Compressive Strength of Epoxy-CB Nanocomposite sample, $Y_{\text{comp(CB600)}}$ is Compressive Strength of Epoxy-CB600 Nanocomposite sample, $Y_{\text{comp(CB-M)}}$ is Compressive Strength of Epoxy-CB-M Nanocomposite sample, A is Magnetic Stir Time, B is Magnetic Stir Speed, C is CB %wt, D is SiO₂ %wt. The positive sign (+) associated with the term indicates synergistic effect which increases the compressive strength and the negative sign (-) indicates antagonistic reducing effect.

3.2 Analysis of Variance (ANOVA) for the Compressive Strength Data

Table 4 shows the ANOVA for the regression model, Based on 95% confidence interval the model F-value of 24.59 is indicative of a significant model and an F-value of 0.0001 means that there is 0.01% possibility that this F-value could occur due to noise [29]. The p-value of less than 0.05 indicate that the terms C, D, E, A² and B² are significant as observed by Sawyer [29].

Table 4. ANOVA for the Regression Model of Equation 3.1-3.3, Compressive Strength

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	20466.78	24	852.78	24.59	< 0.0001
A-Magnetic. Stir Time	132.25	1	132.25	3.81	0.0554
B-Magnetic Stir speed	51.36	1	51.36	1.48	0.2282
C-CB content	17512.11	1	17512.11	504.94	< 0.0001
D-Si Content	802.78	1	802.78	23.15	< 0.0001
E-Modification	610.85	2	305.43	8.81	0.0004
AB	44.08	1	44.08	1.27	0.2639
AC	70.08	1	70.08	2.02	0.1602
AD	14.08	1	14.08	0.41	0.5263
AE	0.17	2	0.083	2.40E-03	0.9976
BC	3	1	3	0.087	0.7697
BD	65.33	1	65.33	1.88	0.1748
BE	1.56	2	0.78	0.022	0.9778
CD	30.08	1	30.08	0.87	0.3553
CE	66.89	2	33.44	0.96	0.3869
DE	26.39	2	13.19	0.38	0.6851
A2	578.58	1	578.58	16.68	0.0001
B2	430.37	1	430.37	12.41	0.0008
C2	26.74	1	26.74	0.77	0.3833
D2	0.51	1	0.51	0.015	0.9043
Residual	2150.28	62	34.68		
Lack of Fit	1861.88	50	37.24	1.55	0.2068
Pure Error	288.4	12	24.03		
Cor Total	22617.06	86			

The adequacy of the model can be seen from Figure 2 the predicted vs measured plots show design points that are evenly scattered around the regression line, indicating that the quadratic model equations provides an accurate description of the experimental data and captures the correlation between the independent variables. The lack of fit value of 0.2068 of the model implies that there is a 20.68% possibility that the lack of fit value of this magnitude may occur due to noise, it indicate a good model as explained by Sawyer [29].

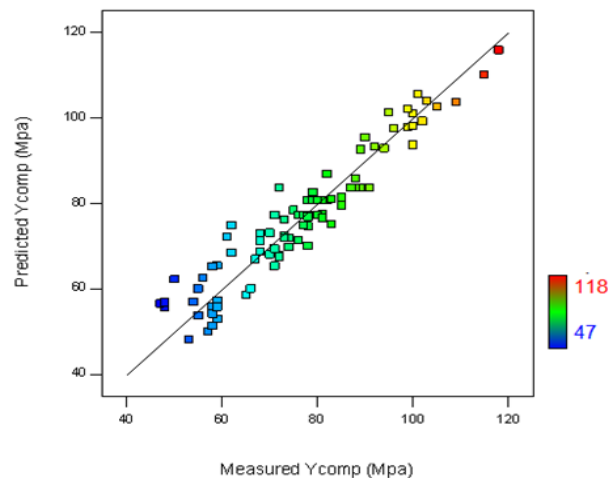


Figure 2. Plot of Predicted vs. Measured Compressive Strength

3.3 Response Surface Plots of Compressive Strength and Effects of Parameters

The CB-M nanoparticles has the best effects owing to better dispersion and interaction at the particle-resin interface, hence it is shown in Figures 3 to 5. Figure 3 shows the effect of CB-M loading and magnetic stir time on the compressive strength of the nanocomposite. CB-M loading has a significant effect with a high F-value of 504.94; however the magnetic stir time is slightly significant with a p-value of 0.0554 as explained by Sawyer [29]. Their interaction has the effect of improving the compressive strength to about 103 Mpa at a time of 4 hours and a 5% wt of CB-M nanoparticle.

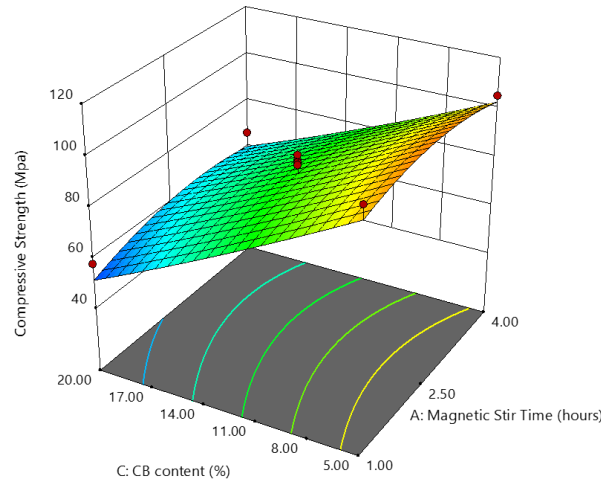


Figure 3. Response Surface Plots for the Compressive Strength of Epoxy/CB-M Nanocomposite at Magnetic Stir Speed of 950 rpm and 10% wt of SiO₂; Magnetic Stir Time vs Carbon Black

Figure 4 shows the effect of SiO₂ % wt and Magnetic stir time on the compressive strength of the nanocomposite. The SiO₂ content with F-value of 23.15 and a p-value of 0.0001 is very significant and explained the improved compressive strength of the nanocomposite with increase in SiO₂ % wt as indicated by Sawyer [29]. The interaction between the two factors resulted in an improvement of the compressive strength of the nanocomposite only to about 84 Mpa as shown in Figure 4. Figure 5 shows the effect of CB-M content and SiO₂ content on the compressive strength of the nanocomposite, both parameters are individually significant. The high compressive strength of 118 MPa achieved at 5% wt loading of CB-M and 20% wt of SiO₂ is due to the dispersion-synergistic effect of SiO₂ addition and thermal chemical treatment of the CB nanoparticle.

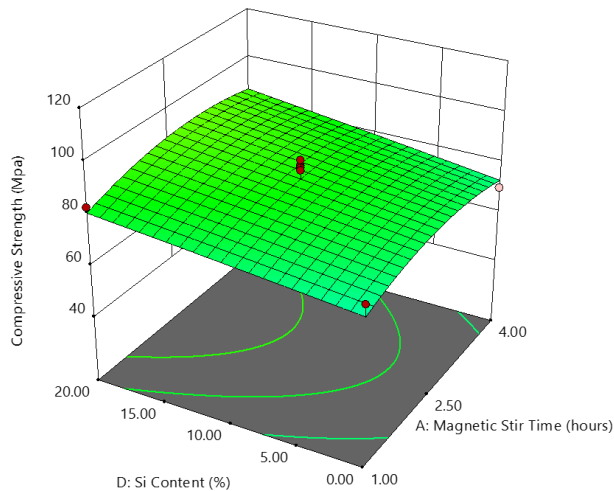


Figure 4. Response Surface Plots for the Compressive Strength of Epoxy/CB-M Nanocomposite at Magnetic Stir Speed of 950 rpm and 10% wt of CB-M; magnetic Stir Time vs Silica content

The result agrees with established facts that the addition of small amount of nanofiller (< 4-5% wt) to a polymer matrix can increase mechanical properties without compromising the processibility of the composite [30], however for conducting polymer nanocomposite, electrical conductivity is achieved at the expense of mechanical properties at a relatively high nanoparticle %wt loading. Figure 5 show a decline in the compression strength of the epoxy / CB nanocomposite as the CB nanofiller content gets higher, the compressive strength of nanocomposite decreases gradually as the nanofiller content goes further than 5 %wt. It is obviously due to lower degree of nanoparticle/epoxy matrix interaction as the CB nanofiller contents increases. Higher CB nanofiller loading will also cause interfacial de-bonding or sliding during compression test, thus reducing the compressive strength of the nanocomposite, this trend is confirmed by Lin [31].

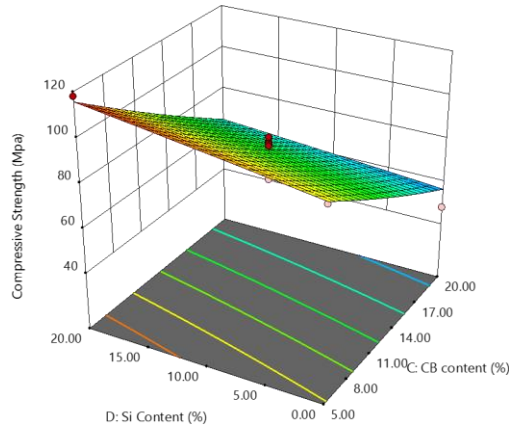


Figure 5. Response Surface Plots for the Compressive Strength of Epoxy/CB-M nanocomposite at Magnetic Stir Time of 2.50 Hours and Speed of 950 rpm

Figure 6 shows the 2-factor interaction between the modification of the nanoparticle and the SiO₂ content, E1, E2 and E3 represent the as received, thermal treated and the thermal-chemical treated CB nanoparticles. The modifications of the CB nanoparticle improved the compressive strength of the nanocomposite at all the design points; the maximum effect is obtained with the thermal-chemical treated CB nanoparticle.

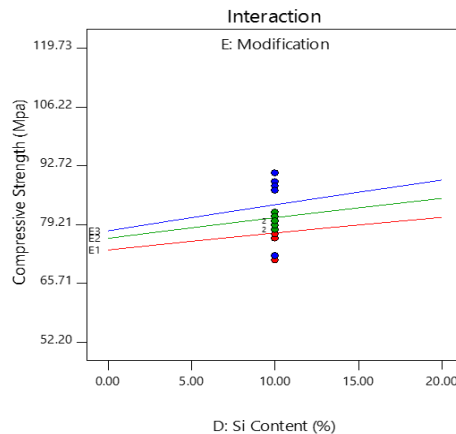


Figure 6. Interaction Effect Plot of SiO₂ Content and Modification of CB Nanoparticle on Compressive Strength

Figure 7 show the interaction between the CB content and the modification of the CB nanoparticle, similarly the thermal-chemical treated CB nanoparticle gives the best improvement. The effect of the modification of the CB nanoparticle is more felt at lower CB %wt due to better CB nanoparticle/epoxy matrix interactions. Chemical modification provides functional polymeric molecules to

the hydroxyl groups at the surface of nanoparticles in order to overcome the incompatibility between inorganic fillers and epoxy resin matrix [32].

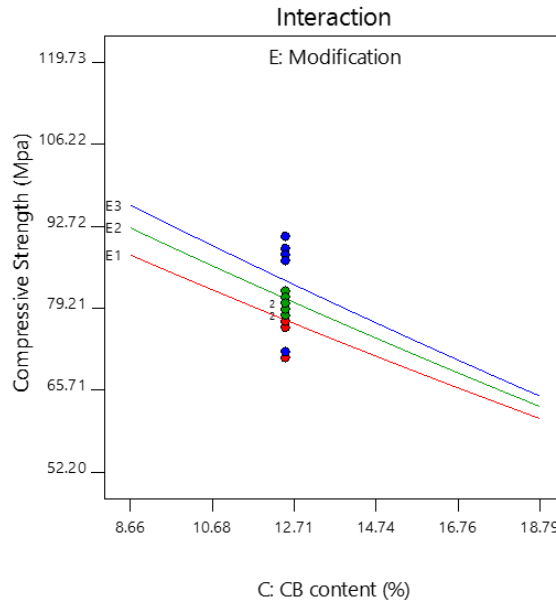


Figure 7. Interaction Effect Plot of CB Content and Modification of CB Nanoparticle on Compressive Strength

3.4 Statistical Analysis of The Tensile Strength Response Data

From Table 5 the Predicted Residual Sum of Squares (PRESS) for the quadratic model is 6854.13; the model has an R-square of 0.86, the cubic model has a higher R-square of 0.9616 but is aliased; the predicted R-square of the quadratic model of 0.7084 is in logical agreement with the adjusted R-square since their difference is less than 0.2. as interpreted by Sawyer [29]. The polynomial model equations for the Tensile strength are as stated below in equations.

Table 5. Summary Statistics of Tensile Strength Model

Source	Std. Dev.	R-Squared	Adjusted R-Squared	Predicted R-Squared	PRESS	
Linear	8.69	0.6784	0.653	0.6162	6854.13	
2FI	8.69	0.7378	0.6532	0.5308	8379	
Quadratic	6.47	0.8639	0.8075	0.7084	5206.72	Suggested
Cubic	4.78	0.9616	0.8951	0.6978	5396.26	Aliased

$$Y_{ten(CB)} = +77.06 + 5.99 * A - 0.02 * B - 2.54 * C - 1.41 * D + 1.11E^{-003} * AB + 1.22 * AC + 0.17 * AD + 7.56E^{-003} * BC + 1.43E^{-003} * BD + 7.78E^{-003} * CD - 3.81 * A^2 - 1.54E^{-006} * B^2 - 0.17 * C^2 - 9.29E^{-003} * D^2 \quad (4)$$

$$Y_{ten(CB600)} = +80.29 + 6.08 * A - 0.02 * B - 2.38 * C - 1.49 * D + 1.11E^{-003} * AB + 1.22 * AC + 0.17 * AD + 7.56E^{-004} * BC + 1.43E^{-003} * BD + 7.78E^{-003} * CD - 3.81 * A^2 - 1.54E^{-006} * B^2 - 0.17 * C^2 - 9.29E^{-003} * D^2 \quad (5)$$

$$Y_{ten(CB-M)} = +83.51 + 4.45 * A - 0.02 * B - 2.51 * C - 1.20 * D + 1.11E^{-003} * AB + 1.21 * AC + 0.17 * AD + 7.56E^{-004} * BC + 1.43E^{-003} * BD + 7.78E^{-003} * CD - 3.81 * A^2 - 1.54E^{-006} * B^2 - 0.17 * C^2 - 9.29E^{-003} * D^2 \quad (6)$$

Where $Y_{ten(CB)}$ is: Tensile Strength of Epoxy-CB Nanocomposite sample, $Y_{ten(CB600)}$ is: Tensile Strength of Epoxy-CB600 Nanocomposite sample, $Y_{ten(CB-M)}$ is Tensile Strength of Epoxy-CB-M Nanocomposite sample. A, B, C and D are as previously defined.

3.5 Analysis of Variance (ANOVA) for the Compressive Strength Data

Based on 95% confidence interval the model F-value of 15.33 is indicative of a significant model and an F-value of 0.0001 means that there is 0.01% possibility that this F-value could occur due to noise. The p-value of less than 0.05 indicate that the terms A, C, D, AC, A² and D² are significant (Table 6).

Table 6. ANOVA for the Regression Model of Equations 4 to 6, Tensile Strength

Source	Sum of Squares	Df	Mean Square	F Value	p-value Prob > F
Model	15426.07	24	642.75	15.33	< 0.0001
A-Magnetic. Stir Time	665.21	1	665.21	15.87	0.0002
B-Magnetic Stir speed	23.36	1	23.36	0.56	0.4584
C-CB content	11554.46	1	11554.46	275.66	< 0.0001
D-Si Content	406.69	1	406.69	9.7	0.0029
E-Modification	130.78	2	65.39	1.56	0.2188
AB	2.08	1	2.08	0.05	0.8244
AC	1411.1	1	1411.1	33.67	<0.0001
AD	80.08	1	80.08	1.91	0.1722
AE	41	2	20.5	0.49	0.6157
BC	24.08	1	24.08	0.57	0.4515
BD	154.08	1	154.08	3.68	0.0601
BE	4.22	2	2.11	0.05	0.9509
CD	4.08	1	4.08	0.097	0.7561
CE	7.55	2	3.78	0.09	0.914
DE	51.72	2	25.86	0.62	0.5431
A ²	1265.43	1	1265.43	30.19	<0.0001
B ²	0.17	1	0.17	4.07E-03	0.9493
C ²	1523.19	1	1523.19	36.34	<0.0001
D ²	15.99	1	15.99	0.38	0.5392
Residual	2431.11	58	41.92		
Lack of Fit	2100.91	47	44.7	1.49	0.2424
Pure Error		330.2	11	30.02	
Cor Total	17857.18	82			

ANOVA is a statistical decision making tool used for detecting any differences in average performances of tested parameters [33]. It employs sum of squares and F statistics to find out relative importance of the analyzed processing parameters, measurement errors and uncontrolled parameters. ANOVA was used to check the adequacy of the model for the responses in the experimentation. The F-value of the quadratic model is 15.33 as shown in Table 6 There is only a 0.01% chance that an F-value this large could occur due to noise. The significance of each term was determined by p-value (Prob> F), which is listed in Table 6, the terms A, C, D, AC, A² and C², were significant, with very small p-values (p< 0.05). Figure 8 shows measured values versus the predicted values of tensile strength of the nanocomposite using the second order model equation developed. The regression model equation provided a very accurate description of the experimental data, revealing that it was successful in capturing the correlation between the five process parameters to the tensile strength of the nanocomposite. The coefficient of determination (R-square) of 0.86 as explained by Sawyer [29] means that the variability in the tensile strength of 86 % could be explained by the polynomial equations 4 to 6 and only 14 % of the total tensile strength variations were not explained by the independent variables. Adeq Precision value is 15.20, it measures the signal to noise ratio. A ratio greater than 4 is desirable hence this model can be used to navigate the design space. The lack of fit F-value of 1.49 implies the lack of fit is not significant relative to the pure error. There is a

24.24% chance that a lack of fit F-value this large could occur due to noise [29]. Figure 8 shows the plot of predicted versus measured tensile strength.

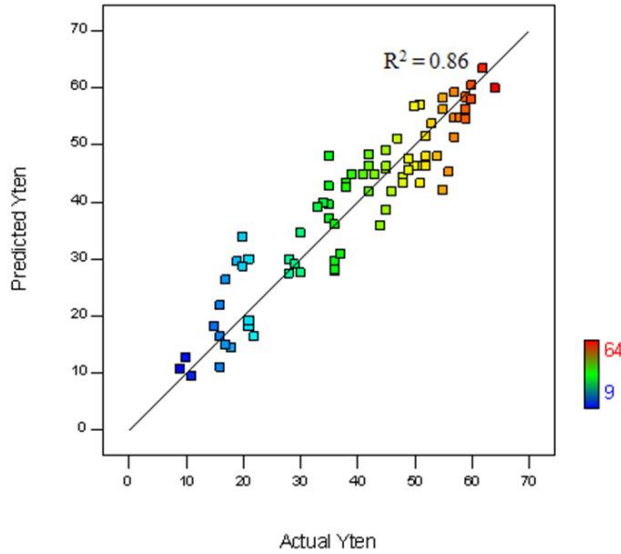


Figure 8. Plot of Predicted versus Measured Tensile Strength

3.6 Response Surface Plots of the Tensile Strength and Effects of Parameters

Figure 9 shows the effect of interaction between CB content and the magnetic stir time on the tensile strength of the nanocomposite, this interaction is significant with a high F-value of 33.67 and a low p-value of <0.0001 .

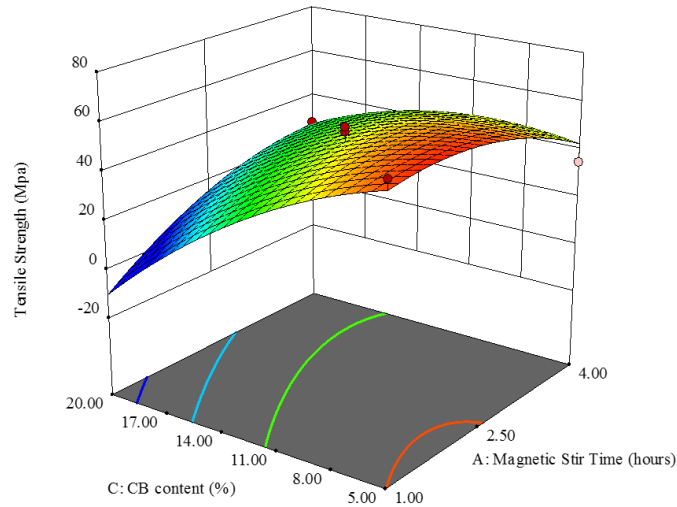


Figure 9. Response Surface Plots for the Tensile Strength of Epoxy/CB-M Nanocomposite at Magnetic Stir Speed of 950 rpm and 10 % wt of SiO_2

As the CB-M%wt loading increases the tensile strength of the nanocomposite reduces, crazing is the phenomenon that produces a network of fine crack on the surface of polymer nanocomposite [34], as agglomerated nanoparticle populate the craze/bulk interface they serve as separators between polymer chains and weaken them, hence increasing the probability of debonding. Furthermore, the viscosity of the mixture during processing directly increases with increased %wt of CB-M, that lead to decrease in CB nanoparticle wettability (by the epoxy resin) with subsequent decrease in mechanical properties of the nanocomposite. Increasing the stirring time, increases the tensile strength of the nanocomposites for the

20%wt CB-M. Figure 10 shows the response of tensile strength to SiO₂%wt addition and magnetic stir time, adding SiO₂ increases the tensile strength of the nanocomposite as noted by [21]. It was shown that addition of SiO₂ improves dispersion by creating a disperse network of obstacle within nanocomposite, thereby forcing the CB-M nanoparticle to occupy the available space. Synergistic effect of increasing the SiO₂ content and the magnetic stir time is an improved nanocomposite with an improved tensile strength at the midpoint of the design space.

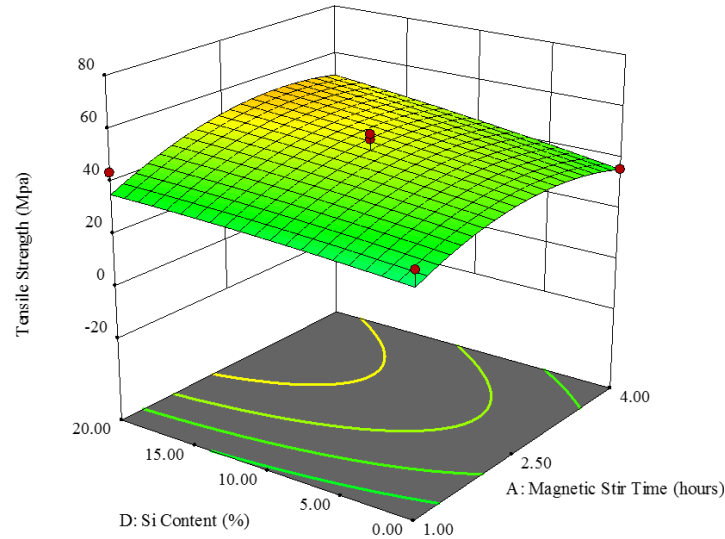


Figure 10. Response Surface Plots for the Tensile Strength of Epoxy/CB-M Nanocomposite at Magnetic Stir Speed of 950 rpm and 12.5%wt of CB-M

With increasing content of silica, CB nanoparticles are optimally dispersed as noted by [35] as reflected in the mapping image of Figure 11 contributing to the generation of a dispersed network CB-M nanoparticles, creating a homogeneously dispersed system of nanoparticles with increased tensile strength.

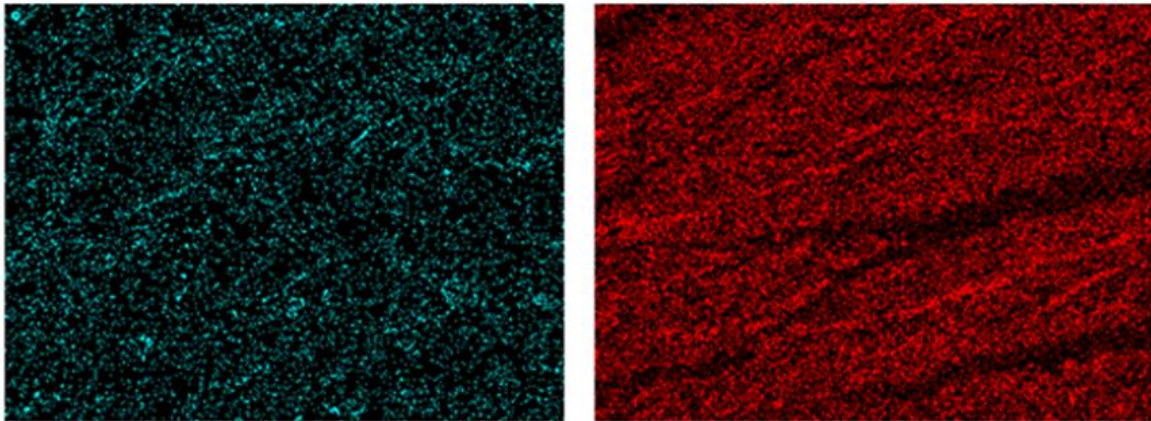


Figure 11. (a) SiO₂ Nano,article and (b) CB-M Nanoparticle Mapping in Epoxy Resin-Carbon Black-Silica Nanocomposite

Figure 12 shows the tensile strength response to magnetic stir speed and SiO₂ content; the figure shows slight improvement of the tensile strength with increasing SiO₂ content. The synergistic effect of increasing the magnetic stir speed and the SiO₂ content is an increased tensile strength of 58 Mpa at 20% wt of SiO₂ and magnetic stir speed of 1200 rpm.

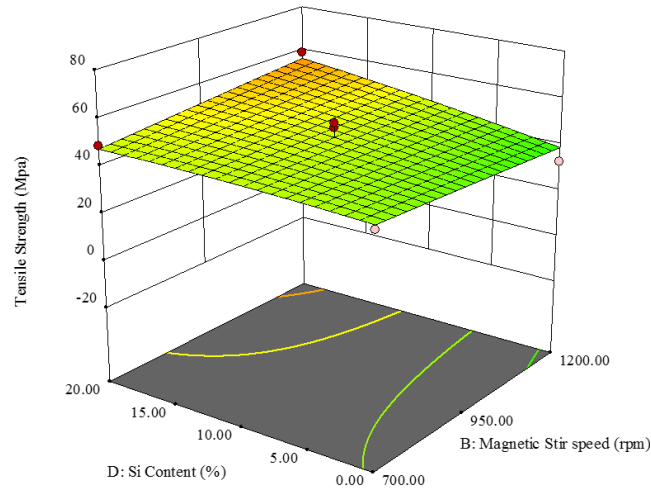


Figure 12. Response Surface Plots for the Tensile Strength of Epoxy/CB-M Nanocomposite at Magnetic Stir Time of 2.50 hours and 12.5% wt of CB-M

Figures 13 and 14 show the existence of carboxylic functional groups on the thermal and the thermal-chemical treated CB nanoparticle, while additional amine functional group exist on the thermal-chemical modified CB nanoparticle. As observed by [36] establish that the interfacial thermal conductance and normalized interaction energy increase between the functionalized nanoparticle and epoxy matrix, which may be responsible for the enhanced mechanical properties in the functionalized CB/SiO₂/epoxy nanocomposites. Figure 15 shows the 2-factor interaction of SiO₂ content and modification of CB nanoparticle on tensile strength. It is obvious that the tensile strength of the nanocomposites increases with modification of the CB nanoparticles. This could be attributed to the existence of the functional group at the surface of the CB nanoparticles.

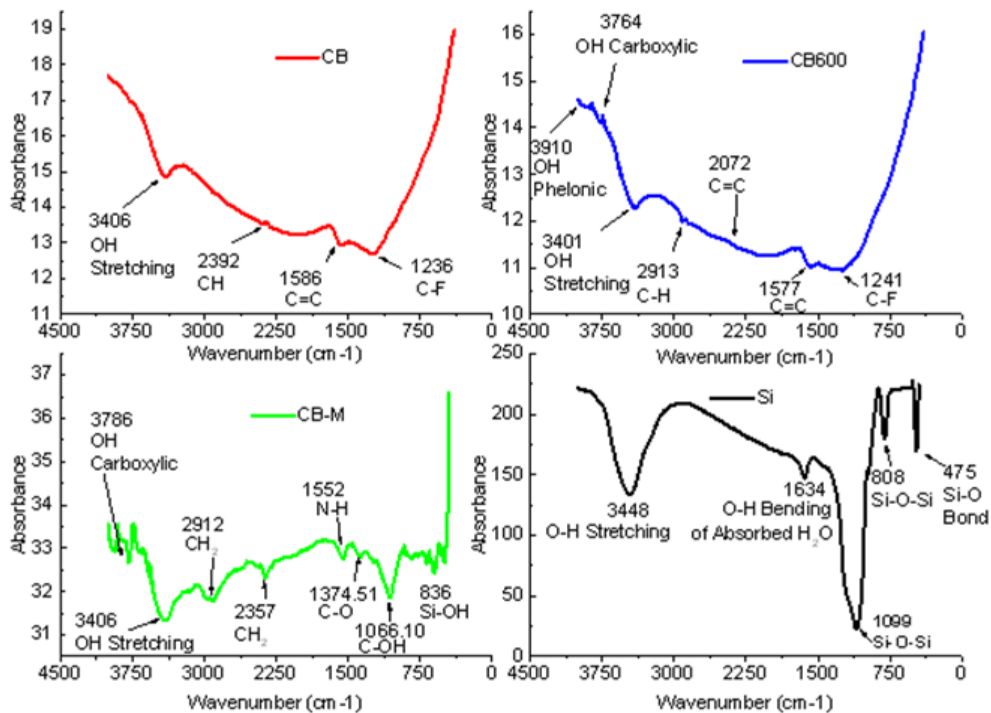


Figure 13. FTIR of the Nanoparticles (a) as Received CB, (b) Thermally Treated CB at 600°C, (c) Thermally Treated CB at 600°C Followed by Chemical Modification, (d) Silica

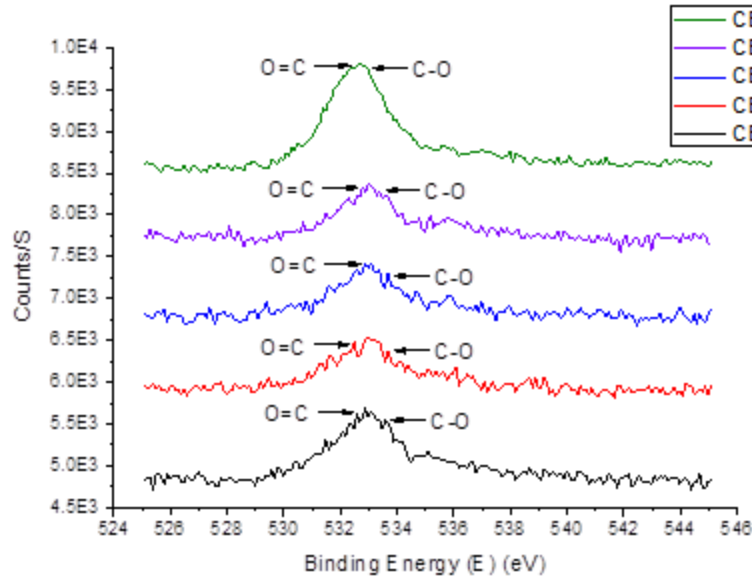


Figure 14. O1s Scan of the CB Nanoparticles

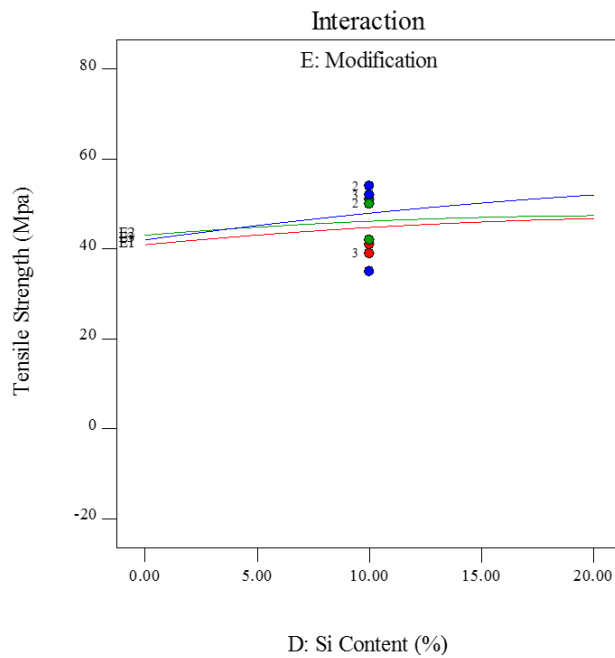


Figure 15. Interaction Effect Plot of SiO₂ Content and Modification of CB Nanoparticle on Tensile Strength

Figure 16 shows the 2-factor interaction between the CB content and the modification of the CB nanoparticle, while the tensile strength increases with decrease in the CB-M % wt the modification of the CB nanoparticle is observed to increase it. The untreated CB nanoparticle tends to formed large agglomerate within the matrix, creating inhomogeneous island of nanoparticle within the matrix with resultant reduced tensile strength.

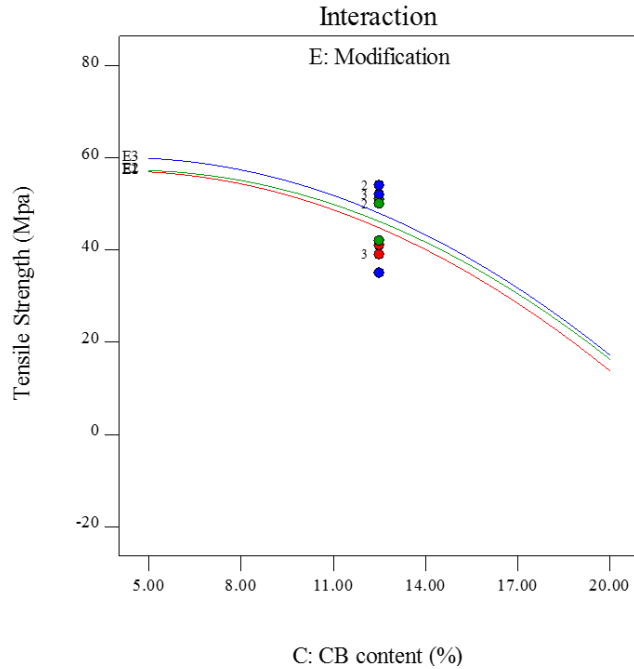


Figure 16. Interaction Effect Plot of CB-M Content and Modification of CB Nanoparticle on Tensile Strength

The ANOVA shows that the interaction represented in Figure 17 of the magnetic stir time and the CB-M%wt significantly affected the tensile strength of the nanocomposite. The CB-M loading is most noticeable at lower magnetic stir time than the higher stir time of 4 hours. At higher CB-M loading the increase in the magnetic stir time record more tensile strength of the nanocomposite. The interaction result suggests lower CB-M%wt at lower magnetic stir time to produce an epoxy nanocomposite with an improved tensile strength.

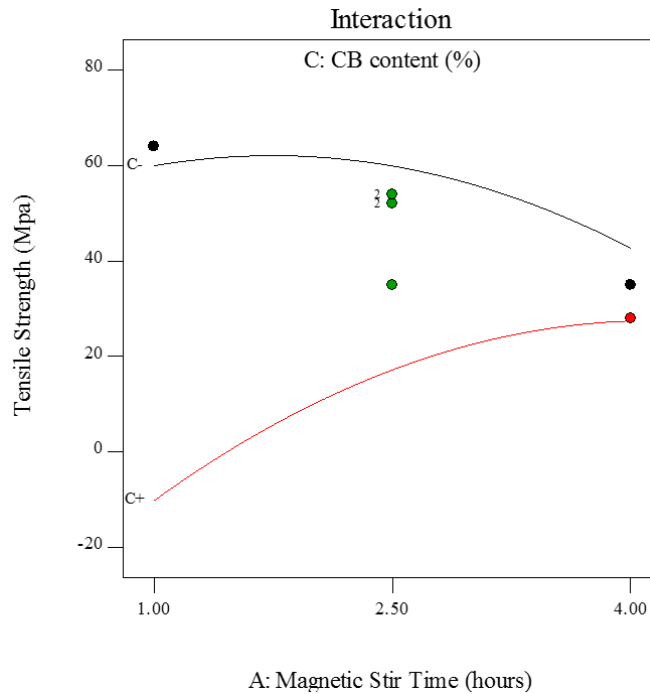


Figure 17. Interaction Effect Plot of Magnetic Stir Time and CB-M Content on Tensile Strength

4 CONCLUSIONS

The improved compressive and tensile properties of the nanocomposite with the modification of the carbon black at specific nanoparticle %wt loading proved the existence of a better interaction between the carbon black nanoparticles and epoxy matrix. The evidence of the synergistic effect of mixing speed, mixing time, carbon black weight fraction, silica weight fraction, and the state of carbon black nanoparticles (as received, thermally treated, and thermally and chemically treated) was shown by the result obtained. The use of Box Behnken response surface method described the significant processing factors and the extent of their effects on the properties of the nanocomposites. The incorporation of hybrid carbon black and silica nanofillers into the epoxy matrix resulted in substantial improvements in mechanical properties. Optimal concentrations of nanofillers were identified, demonstrating that a synergistic effect between carbon black and silica can be achieved to maximize the mechanical performance of the epoxy nanocomposites. The developed models provided accurate predictions of the mechanical behavior, facilitating the identification of optimal processing conditions to achieve the desired mechanical properties. Surface treatments of the nanofillers played a crucial role in enhancing their dispersion within the epoxy matrix and improving the interfacial bonding between the nanofillers and the epoxy resin. The findings of this study offer valuable insights for the design and optimization of high-performance epoxy nanocomposites tailored for advanced engineering applications, such as aerospace, automotive, and construction industries. By fine-tuning the type and concentration of nanofillers, as well as the processing conditions, it is possible to develop epoxy nanocomposites with customized properties for specific application requirements. In conclusion, our research demonstrates the promising potential of hybrid carbon black and silica nanofillers in enhancing the mechanical properties of epoxy nanocomposites. The application of response surface methodology provided a robust framework for optimizing the processing parameters and understanding the complex interactions within the composite system. These findings pave the way for future advancements in the development of high-performance epoxy nanocomposites, offering significant benefits for a wide range of engineering applications.

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