# REGRESSION ANALYSIS OF MECHANICAL PROPERTIES OF CARBON FABRIC POLYMER HYBRID NANOCOMPOSITE

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## ABSTRACT

The development of nanoparticle reinforced composites is presently one of the most explored areas in materials science and engineering. Multiscale composites can be produced with superior properties by combining nanoparticles with traditional reinforcement materials. This paper focuses on the development of such composites, through the use of carbon nanofibers (CNFs), carbon fibers and modified epoxy resin for structural and impact applications. Flexural and tensile behavior of composites has been analysed to investigate the effect of modification of epoxy matrix by adding CNFs on the mechanical properties of composites. Morphological and fracture analysis of composites were performed by SEM. In the present study, Mechanical properties prediction model using regression analysis and ANOVA is developed to investigate the effects of wt% of CNF, ETBN during processing of carbon fabric-polymer hybrid nanocomposite.

**Key words:** carbon nanofibers, ETBN, Carbon fabric, polymer hybrid nanocomposites, regression, ANOVO.

## I. INTRODUCTION

The development of nanoparticle reinforced composites is presently one of the most explored areas in materials science and engineering. The exceptional properties of nanoparticles have made them a focus of widespread research. Carbon nanotubes (CNTs) carbon nanofibers (CNFs) and other nanoparticles have the potential to greatly enhance the properties of composites when combined with traditional reinforcements such as carbon-, glass-, and aramid-fibers [1].

Using ETBN to toughen epoxy resin and then the mechanical property of the toughening epoxy resin casting molding system is studied by Yuan-bo,li et al. It shows that with the increasing content of ETBN, the flexural strength, tensile strength and tensile modulus decreased, while the impact strength and elongation at break distinctly increased. This demonstrates that the toughness of the epoxy resin system could be improved effectively by adding ETBN [2].

Epoxy resins are widely used in a variety of applications because of their high chemical and corrosion resistance and good mechanical properties. Carbon nano fibres have been dispersed uniformly into the epoxy resin at a very low concentration (0.07 vol. %). Improvement of 98% in Young modulus, 24% in breaking stress and 144% in work of rupture was achieved in the best sample. The emphasis is on achieving uniform dispersion of carbon nanofibers into epoxy resin using a combination of techniques such as ultra sonication, use of solvent and surfactants [3].

Good dispersion of CNFs leads to an enhancement in both strength and modulus of nanocomposites [4, 5 – 8]. Choi et al. [4] found that CNF/epoxy nanocomposites had a maximum tensile strength and a large Young's modulus with 5wt% CNFs, and a reduced fracture strain with increasing filler content.

Based on the five key parameters (ferrite, cementite, modularity and content of carbon and silicon) linear regression equations were created for calculation of thermal conductivity and hardness. From these it is possible to predict the combination of parameters which gives a particular combination of hardness and thermal conductivity in compacted graphite iron [9].

Thomas et al reported an experimental program and an analytical assessment of the influence of addition of fibers on mechanical properties of concrete. Models derived based on the regression analysis of 60 test data for various mechanical properties of steel fiber-reinforced concrete have been presented [10].

The software for prediction of the high temperature plasticity development based on physical metallurgical and regression analyses was elaborated for the cast state of low carbon steels. The program was verified by using experimentally estimated values [11].

The analysis of variance (ANOVA) is widely used to consider effects of factors on responses. An Aminollah et al experimental investigation, ANOVA is often employed prior to other statistical analysis. Then regression analysis which establishes a relation between independent variables and dependent variables is widely applied [12].

*A. Materials Used*

Carbon nanofibers (M/s Chemapal Industries, Mumbai) have been used to prepare the nanocomposites. CNFs are having the diameter approximately in the range of 100-200 nm and length is in the order of 20-30 $\mu$ m.

Carbon fabric (T-300, PAN based carbon fabric, Toray, Japan) has been used to make composites.

Epoxy, Diglycidyl Ether of Bisphenol A (DGEBA) and curing agent HY5200 (both supplied by M/s ECMAS Pvt. Ltd, Hyderabad) have been used to process the multi phase nanocomposite.

Epoxy terminated butadiene-acrylonitrile copolymer (ETBN, M/s Fine Finish Organics, Mumbai) has been used to enhance the matrix properties through modification of epoxy by adding ETBN.

*B. Modification of epoxy matrix*

Epoxy resin is being modified to improve elongation capabilities. Toughness and other mechanical properties of epoxy resins can be significantly improved by adding rubber, in which Epoxy terminated butadiene-acrylonitrile copolymer (ETBN) is conventionally used. In this system, both epoxy groups on ETBN and epoxy groups on epoxy resin to form one network. Reactive liquid rubber dissolved in liquid resin, as curing proceeds, rubber precipitates from epoxy and forms fine dispersion of rubber particles with diameter of few micrometers or less. Two compositions were prepared using 2.5% and 5% ETBN to modify the epoxy resin. Epoxy was mixed and Di Ethyl Aniline acts as curing agent. Curing was performed at 120°C / 1h, continued to 150°C / 1h and then at 210°C / 3h. The same procedure is indented for another system to achieve the matrix modification. The morphology and distribution of second phase were carefully observed using optical microscopy and SEM.

*C. Composites Preparation and Testing*

Carbon nanofibers were used to understand the effect of nanophase additions on the properties of C-modified epoxy composites. C-modified epoxy and C-modified epoxy/CNFs multiphase nanocomposites have been prepared at 1wt% CNFs loading. Initially, CNFs were added directly to the modified epoxy system and then preheated at 60°C to reduce the viscosity. This mixture was sonicated with the help of probe type sonicator for 30 min at below 80°C. Curing agent was added to the mixture in the ratio of 4:1 by weight to improve the dispersion. Further, this was followed by ball milling at the speed of 150 rpm for 30 min by adding silane coupling agent to the CNF-resin mixture to enhance the degree of dispersion. This mixture was brushed on to the surface of the individual C-fabric layers. Curing was carried out at 120°C for 2 h, followed by 180°C for 4 h. Thus, Composites were synthesized using modified epoxy resin at 2.5 and 5% loading of ETBN rubber to modify the epoxy resin and with 1wt%, 2wt% and 3wt% of CNFs reinforcement.

Three point bend tests were carried out to measure the flexural strength of the polymer hybrid nanocomposites according to ASTM D790. The fracture analysis of failed composites has been analyzed by ESEM to know the role of CNFs on the strength of nanophased composites. Tensile test was carried out as per ASTM D3039 using Universal testing machine (Dak system INC model UTM machine).

## II. CHARACTERIZATION OF MATERIALS

Typical SEM image of carbon nanofibers at lower magnification shows presence of agglomerates in the order of few hundred microns as shown in Figure 1.

Optical images of cured Epoxy reinforced with 2.5% ETBN shows that the dispersion of ETBN (rubbery phase) in epoxy is not uniform (marked by arrows) at lower magnification, as shown in Figure-2.

At lower magnification, bundles of carbon fibers are observed, as shown in Figure-3. It can be observed that the nanofibers are formed clusters at most of the regions in Fig-3. From Figure-4, carbon nanofibers are

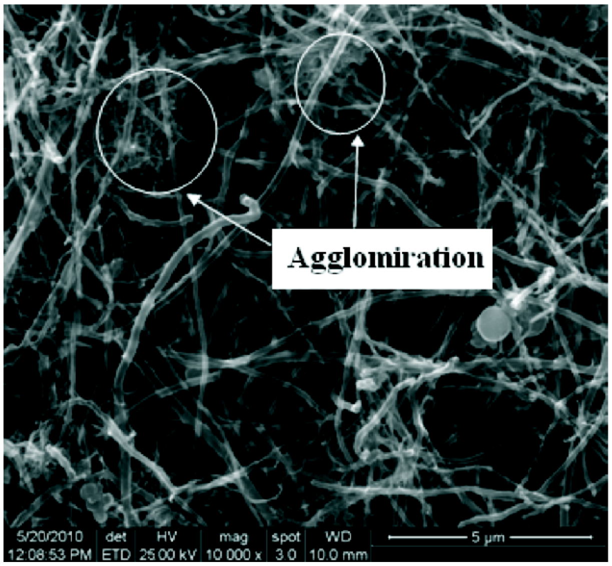


Fig. 1. SEM image of carbon nanofibers showing agglomerates.

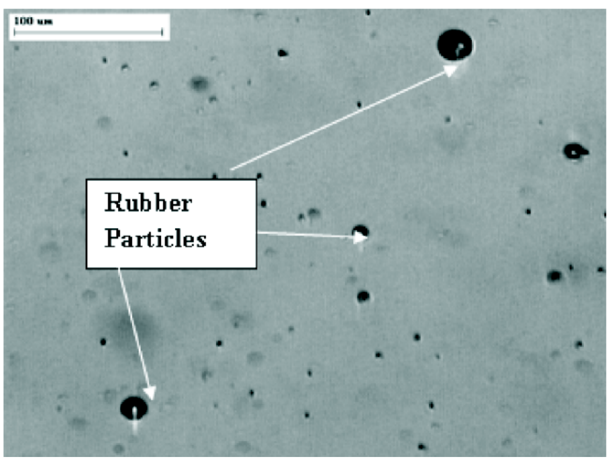


Fig. 2. Optical image of 2.5% ETBN -Epoxy composite showing dispersion of ETBN

entangled more in nature at higher magnification. Variation of size of nanofibers is very high. No alignment of nanofibers is observed at any region in the sample. The distribution of nanofibers shows that the diameter varied significantly. The diameter of nanofibers is in the range of 100-200 nm. However, non uniform distribution of carbon nanofibers is observed from the composite.

From the above observations, it can be concluded that the dispersion of ETBN particles in the epoxy matrix is not uniform. Homogeneous distribution of ETBN phase as well as dispersion of CNFs in the regular epoxy matrix decrease with increasing percentage of ETBN.

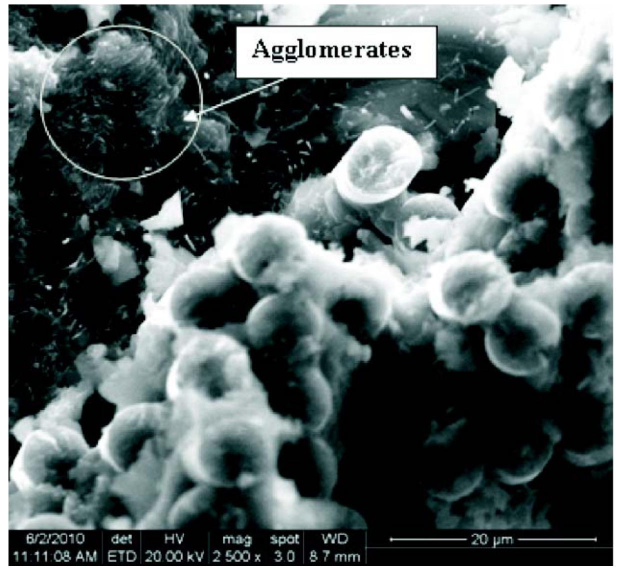


Fig. 3. SEM image of 1wt% CNF and 2.5wt% ETBN composite showing agglomerates of CNFs in the matrix

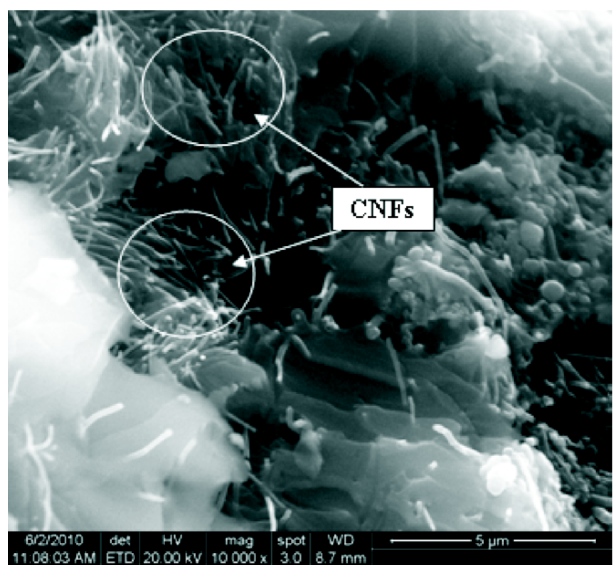


Fig. 4. SEM image of 1wt% CNF and 2.5wt% ETBN composite showing dispersion of CNFs in the matrix

The network of ETBN particles in the matrix materials should aid in the transfer of load between the resin and fibers resulting in multiscale reinforcing composite. Good interfacial bonding and dispersion enhances the resin dominated properties of the composites, while addition of large quantity of ETBN particles decreases the flexural properties. Formation of strong interfacial bonding with fibers leads to brittle failure. Addition of ETBN phase changes the failure mode of composites too ductile mode. Bent resin stripes are typical evidence of a change in failure mode. Decrease in flexural strength is due to non-uniform dispersion of excess ETBN.

Mechanical strengths like Tensile strength and Flexural strength are reported for the developed hybrid nanocomposites by varying weight percentage of carbon nanofibers and weight percentages of ETBN. Almost in all the cases both tensile and flexural strengths are improved with CNF reinforcement and ETBN modification. More than 100% improvement was noted in Tensile strength by reinforcing the CNFs in polymer composites. A constant improvement is noted between 1-2wt% of CNF and 2.5wt% of ETBN. Maximum 173 percent of improvement in tensile strength is noted at 1wt% of CNF and 5wt% of ETBN. Results conclude that 1wt% of CNF and 2.5wt% of ETBN shows better tensile and flexural strengths.

## III. RESULTS AND DISCUSSION

### *A. Regression Analysis*

Regression is the process of fitting models to data. The process depends on the model. If a model is parametric, regression estimates the parameters from the data. If a model is linear in the parameters, estimation is based on methods from linear algebra that minimize the norm of a residual vector. If a model is nonlinear in the parameters, estimation is based on search methods from optimization that minimize the norm of a residual vector. Categorical predictors are the subject of Analysis of Variance. Mathematical regression models have been built to estimate mechanical properties on line Statistical Package for the Social Sciences (SPSS, a registered product of SPSS Inc., Chicago).

Several influencing measurable parameters wt% of ETBN, wt% of CNF considered for the regression analysis and are used as the independent factors whereas tensile strength and flexural strengths are considered dependent factors. The general model to predict the Tensile strength over the experimental region can be expressed in the equation-1.

$$\text{Tensile Strength} = ((449.038*x) + (0*x^2) - (10.13*x^3) + (433.886*y) - (187.589*y^2) + (20.404*y^3) (14.041*xy) + (359.864)) \quad ...[1]$$

The model to predict the Flexural strength over the experimental region can be expressed in the equation-2.

$$\text{Flexural Strength} = ((2.972*x) + (0*x^2) - (0.064*x^3) + (5.818*y) - (2.121*y^2) + (0.12*y^3) - (0.141*xy) + (42.388)) \quad ...[2]$$

Where x = wt% of ETBN and y = wt% of CNF

Multiple linear regression, carried out for the linearized terms using SPSS, postulates a functional dependence between the independent and dependent variables minimizing the modeling error.

A linear function problem reduces to finding the coefficients V1, V2, V3, V4, V5, V6, V7 and constant. In the present analysis, these constants were found to be 449.038, 0, – 10.130, 433.886, – 187.589, 20.404, – 14.041 and 359.864 respectively. Experimental data was obtained to build the model and validation with 0, 1, 2 and 3wt% of CNF and 0, 2.5 and 5wt% of ETBN.

### *B. Analysis of variance (ANOVA)*

Analysis of Variance (ANOVA) is a powerful statistical technique used to confirm the effect of several simultaneously applied factors on the response variable [13]. A null hypothesis, postulating 'no dependence' of the applied factors & response variables are considered and is checked for its validity. Degrees of Freedom (DF) and sum of the squares (SS) are computed for the considered data. F-statistic (variance ratio) is computed as the ratio of sums of squares denoting influence of factors and their interdependence. The computed value of variance ratio (F) is compared with the standard ANOVA table and the hypothesis is accepted or rejected at a particular (1% or 5%) confidence level. If the hypothesis is rejected at 1% confidence level, it also stands rejected at 5% confidence level.

In the present work, the degrees of freedom were found to be 6 and 31; F-statistic was obtained as 481.069 from SPSS. The tabulated critical value of F distribution for the obtained degrees of freedom at 1% significance level was 4.10. Hence the proposed null hypothesis advocating no dependence of wear rate on the taken parameters was rejected at 1% significance level. Hence the choice of parameters is justified.

Table-1 summarizes the results from regression analysis. The first column gives the values of constant and coefficients of the variables in the formulated model. Standard Error Coefficients (SE Coef) obtained in the model give the significance of each independent variable. T-value gives the ratio of the parameter estimate and its standard error. P value denotes the probability of null hypothesis to be wrongly rejected.

**Table 1. Prediction model from regression analysis (hardness)**

| Predictor const | Coef | SE Coef | T | P |
|---|---|---|---|---|
| Constant | 359.864 | | 9.431 | 0.000 |
| V1 | 449.038 | 2.190 | 37.368 | 0.000 |
| V3 | − 10.130 | − 1.360 | − 29.163 | 0.000 |
| V4 | 433.886 | 0.955 | 5.356 | 0.000 |
| V5 | − 187.589 | − 1.311 | − 3.120 | 0.004 |
| V6 | 20.404 | 0.421 | 1.581 | 0.124 |
| V7 | − 14.041 | − 0.146 | − 3.273 | 0.003 |
| $R^2$ = 0.989, $R^2$ (adjusted) = 0.987 | | | | |

| ANOVA | | | | | |
|---|---|---|---|---|---|
| **Source** | **SS** | **DF** | **Mean Square** | **F** | **P** |
| Regression | 6494407.878 | 6 | 1082401.313 | 481.069 | 0.000 |
| Residual Error | 69749.698 | 31 | 2249.990 | | |
| Total | 6564157.576 | 37 | | | |

Both the high adjusted $-R^2$ and P values are close to zero, in the analysis of variance (ANOVA), showing that the postulated model has satisfactory goodness of fit.

Error from regression analysis is shown in Figure-5. Experimental and regression analysis of Tensile strength and Flexural strength along with wt% of CNF and wt% of ETBN is shown in Figures: 6-9. Regression analysis gives good agreement with experimental data and shows error less than 12% in all the conditions.

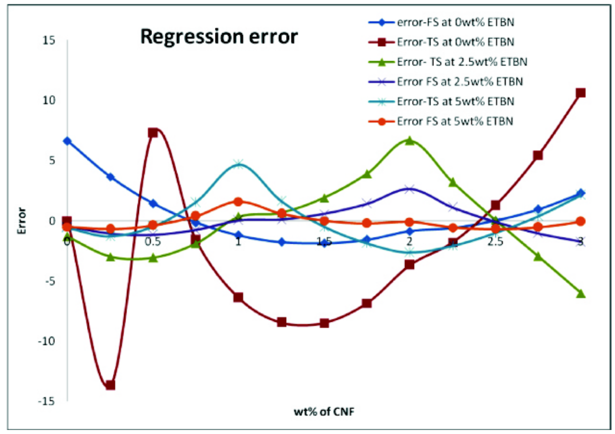


Fig. 5. Regression error for tensile strength and Flexural strength.

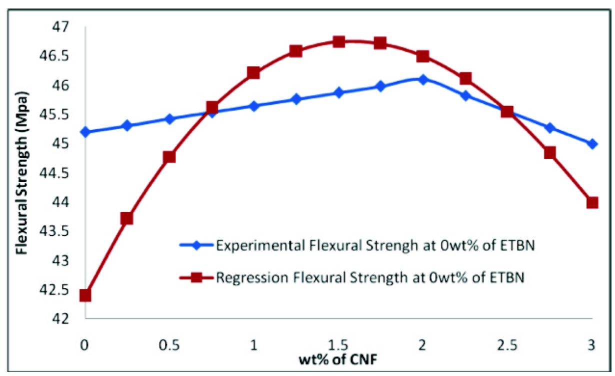


Fig. 6. Flexural strength at 0wt% of ETBN

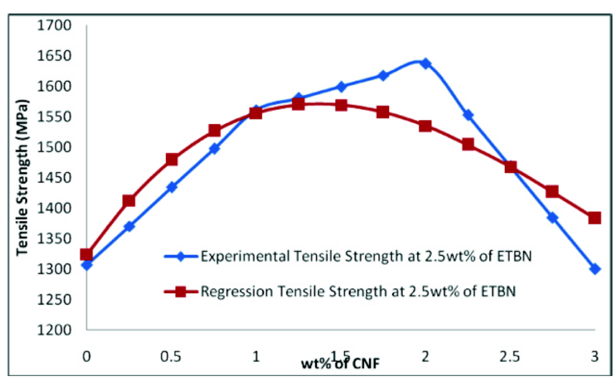


Fig. 7. Tensile strength at 2.5wt% of ETBN

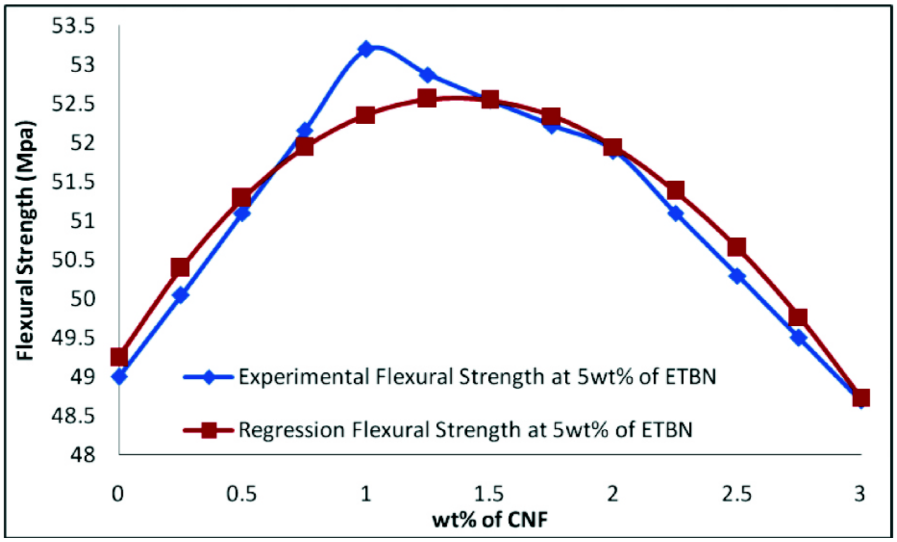


Fig. 8. Flexural strength at 5wt% of ETBN

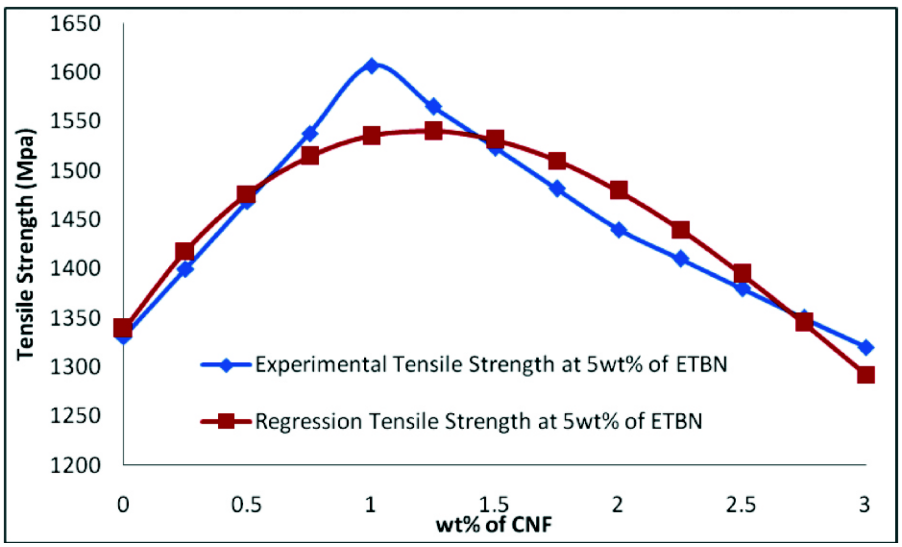


Fig. 9. Tensile strength at 5wt% of ETBN

## IV. CONCLUSION

It is concluded that the modification of epoxy matrix by adding ETBN rubbery phase is achieved as seen by optical and SEM analysis.

From the above observations, it can be concluded that the dispersion of ETBN particles in the epoxy matrix is not uniform at 5wt%. Homogeneous distribution of ETBN phase as well as dispersion of CNFs in the regular epoxy matrix decreases with increasing percentage of ETBN. It can be concluded that the presence of ETBN phase leads to good interfacial bonding between the matrix and fibers.

The presence of ETBN in the epoxy increases the mechanical properties of the hybrid nanocomposite up to 2.5% and later it starts decreasing due to the increase of resistance of epoxy matrix. Agglomeration and non-uniform dispersion of CNFs rather than that of uniform dispersion of CNFs and non-uniform growth of rubbery particles of ETBN act as obstacles for the composite failure. Modification of epoxy resin matrix by adding ETBN at higher loading affects the dispersion of nanofibers, which decreases the properties and increases the hardness of epoxy matrix.

It can be suggested that the optimization of ETBN additions can give better enhancement of the mechanical properties of regular epoxy. Lowering the percentage of ETBN below 2.5 wt% may gives better dispersion as well as better properties.

Regression analysis gives good agreement with experimental data and shows an error less than 12% in all the conditions.

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