Original Article

Effects of thermal aging and functionalized epoxy matrix with graphene nanoplates in fique fabric-reinforced composites

Michelle Souza Oliveira [1]*
Fernanda Santos da Luz [1]
Andreza Menezes Lima [2]
Foluke Salgado de Assis [3]
Artur Camposo Pereira [1]
Fábio de Oliveira Braga [4]
Sérgio Neves Monteiro [1]
André Ben-Hur da Silva Figueiredo [1]

Abstract

Currently, the demand for high quality and reliable components and materials is increasing, so bending testing has become a vital test method, both in research and manufacturing and development process, to explain in detail about the material's ability to withstand deformation under load. This research investigated flexural properties of polymeric composites reinforced with natural fiber, in particular the fique fabric, with addition of graphene nanoplates (GNP) (0.1%; 0.5% and 0.9%) and degradation at high temperature (0, 5 and 10 days), as it was never reported. Using design of experiments (DoE), ie, 3-full factorial design with two replications, aiming to analyze the effects of important parameters, which are exposure time and GNP addition percentage. The output response measurement was identified as deflection at fracture, modulus of rupture and elasticity values. Randomized experiments were conducted based on table generated via Minitab 19 software. Scanning electron microscopy analysis confirmed the main influences on flexural analysis responses.

Keywords: Natural fiber; Thermal aging; Graphene nanoplates; Polymer matrix.

Efeitos do envelhecimento térmico e da matriz epóxi funcionalizada com nanoplacas de grafeno em compósitos reforçados com tecido fique

Resumo

Atualmente, a demanda por componentes e materiais de alta qualidade e confiabilidade está aumentando, de modo que os testes de flexão se tornaram um método de teste vital tanto na pesquisa quanto no processo de fabricação e desenvolvimento para explicar em detalhes sobre a capacidade do material de suportar deformação sob carga. Esta pesquisa investigou as propriedades de flexão de compósitos poliméricos de reforço de fibra natural, em especial o tecido de fique, com adição de nanoplacas de grafeno (NPG) (0,1%; 0,5% e 0,9%) e degradação em alta temperatura (0, 5 e 10 dias), uma vez que nunca foi relatado. Usando o planejamento de experimento fatorial (PEF), ou seja, fatorial completo com dois fatores e três níveis com duas repetições, objetivando a análise dos efeitos de parâmetros importantes que são o tempo de exposição e a porcentagem de adição de NPG. A medição da resposta de saída foi identificada como valores de deflexão, resistência à flexão e rigidez. Os experimentos randomizados foram conduzidos com base na tabela gerada via software Minitab 19. Análise de microscopia eletrônica de varredura ratificou as principais influencias nas respostas da análise de flexão.

Palavras-chave: Fibra natural; Envelhecimento térmico; Nanoplacas de grafeno; Matriz polimérica.

[1]*Laboratório de Fibras Naturais e Compósitos, Instituto Militar de Engenharia, Rio de Janeiro, RJ, Brasil.*
[2]*Instituto Militar de Engenharia, Laboratório de Biomateriais, Rio de Janeiro, RJ, Brasil.*
[3]*Grupo de Tecnologia de Materiais, Instituto de Pesquisa da Marinha do Brasil, Rio de Janeiro, RJ, Brasil.*
[4]*Departamento de Engenharia Civil, Universidade Federal Fluminense, Niterói, Rio de Janeiro, Brasil.*

**Corresponding author: oliveirasmichelle@ime.eb.br*

1 Introduction

Recently, a worldwide trend in search of natural resources that will benefit humanity, not only in creation of new products, but also in opportunity to create jobs and generate income through development of new technologies [1]. There is great interest in search for natural fibers that can adequately replace synthetic fibers such as, for example, aramid [2]. Natural lignocellulose fibers, usually defined only as natural fibers, are currently widely used as reinforcements in polymeric composites due to their low cost, being biodegradable, which is in line with current appeal to environmental preservation and use of renewable material. The main applications of these composites are in civil construction, furniture, packaging, automotive and ballistic protection components, generally used as functional material or for light and medium-sized loads [3]. An important characteristic of polymeric composites is the synergistic effect observed in system, where final properties are improved from individual properties of its components [4]. As plant fibers have comparatively good specific mechanical properties (strength/weight, elastic modulus/weight), this makes them a viable component for application in composites [3,4].

Depending on the application of composite, the study of natural degradation can be of fundamental importance, especially when degradation is combined with heat, solar radiation, pollution and static or fatigue loads; conditions that lead to material' aging with decreases in strength and stiffness properties [5]. Degradation causes an irreversible change in properties of polymeric materials, being evidenced by progressive deterioration of these properties, including visual appearance [6]. In some cases, degradation reactions may be desirable, for example, for non-recyclable polymeric waste. However, in general, degradation reactions are undesirable. Accelerated aging tests have advantage of speed, providing data on likely material performance over its lifetime [5]. In these tests, material' exposure conditions are simulated, but with high intensities, to accelerate the degradation process. Despite advantage of speed, it can provide inaccurate results, due to parallel mechanisms occurring in materials. In accelerated tests, processes normally studied are thermooxidation, continuous use at high temperatures, and photooxidation, exposure to ultraviolet radiation [7]. Thermal aging presents major challenge for high temperature applications of natural fiber composites [6]. Degradation processes can be monitored by changes in material' physical state and mechanical properties.

Composites based on natural fibers have extensive requirements in today's competitive framework of manufacturing and industrial applications [8]. An example of application is as intermediate layer in multilayer armor systems (MAS). MAS with frontal alumina ceramic followed by composite reinforced with curaua non-woven fabric and supported by Aluminum alloy were investigated in terms of thickness ratio optimized by Braga et al. [9]. The authors used the Box-Behnken Design (BBD) and multiple regression analysis (MRA) model to assess the contribution of each layer of MAS to its overall trauma absorption performance. As the early mentioned study based on the systematic DoE method, one can choose a process configuration that works more consistently in operating environment and reduces process variability. A considerable amount of literature has been published and applying design of experiments (DoE) in bending test [10-16].

The demand for high quality and reliable components and materials is increasing, so bending testing has become a vital testing method both in research and in manufacturing and development process to explain in detail about the material's ability to withstand deformation under load [17]. Recently, there is a lack of research studies on the effect of degradation and addition of graphene nanoplates (GNP) in bending condition by the DoE approach method.

Therefore, the objective of present work is to apply statistical tools to quantify the influence of exposure to high temperature and addition of GNP on flexural performance of fique fabric-reinforced composite. Statistical tools such as 3^k full factorial and MRA were used.

2 Materials and methods

Fique fabric commercially available in Colombia was supplied by Compañia de Empaques, Antioquia, Colombia. The matrix was diglycidyl ether of bisphenol A (DGEBA) epoxy resin mixed with triethylenetetramine (TETA) hardener in stoichiometric proportion phr 13. Both DGEBA and TETA were supplied by Epoxyfiber, Rio de Janeiro, Brazil. The 40 vol% fique fabric–epoxy matrix composite was fabricated by placing previously dried fabric piece as ply layers inside a steel mold, and DGEBA-TETA resin with GNP was also poured in. Laminated plates of 150 × 120 × 3 mm were made by the compression molding process and cured for 24 h at room temperature, inside the mold, under a 5-ton load. The method of incorporating graphene nanoplates into the epoxy was like described by Şükür e Önal [18]. The GNP amounts being first added, with proportions of 0.1%, 0.5% and 0.9% by epoxy weight.

The importance of using GNP is due to Brazil has the second largest reserves and is the third largest producer of graphite in the world and is necessary to consider the use of this material and its derivatives to optimize compatibility between matrix and composites reinforcement. Minas Gerais Development Company (CODEMGE), together with Nuclear Technology Development Center (CDTN) and Federal University of Minas Gerais (UFMG), established MGgrafeno Project and implemented the first pilot plant in Brazil for graphene production from natural graphite. MGgrafeno has developed liquid phase exfoliation process that produces high quality nanomaterials, each suitable for a variety of applications. This work was developed in partnership with MGgrafeno.

2.1 High temperature aging

The samples were exposed to high temperature (170 °C) in Nova Instruments drying and sterilization oven. Three exposure times were considered, corresponding to 0, 5 and 10 days. Elevated temperature, selected to accelerate degradation process, was relatively low compared to epoxy matrix glass transition temperature as seen in previous study [6]. The thermogravimetric characteristics of plain epoxy and unaged fique fabric-composite were investigated in a previous work. It was revealed that 170 °C might be considered the maximum temperature before a sudden decrease in mass loss associated with effective thermal degradation of the material. These TGA results justify the choice of 170 ºC for the basic temperature for different aging times selected in the present work.

2.2 Flexural test

The three-point bending test was performed on computer-controlled universal testing equipment EMIC, model DL 10000. Samples were made from composite plates in dimensions 127 mm × 13 mm × 3 mm, according with ASTM D790-17, deformation speed was 2 mm/min and distance between the supports was 96 mm. Modulus of rupture (σ), Equation 1, and modulus of elasticity (E), Equation 2, were calculated, where Q_m is the maximum load, L is the distance between the supports, b and d are width and thickness, respectively, and Δy is the level of composite deflection at maximum flexural strength.

$$\sigma = \frac{3LQ_m}{2bd^2} \tag{1}$$

$$E = \frac{Q_m L^3}{4bd^3 \Delta y} \tag{2}$$

2.3 Design of Experiments (DoE): three-level full factorial designs

Design of experiments (DoE) provides powerful means to achieve breakthrough improvements in product quality and process efficiency. From the point of view of manufacturing fields, this can reduce the number of experiments needed by considering main factors that affect experimental results. DoE is a systematic method for determining the relationship among factors that affect processes and its outputs. In other words, it is used to find cause and effect relationships. This information is needed to manage process inputs to optimize output. DoE can show how to run fewest number of experiments while keeping most important information.

2.3.1 3^k factorial design

Three-level design is written as a 3^k factorial design. This means that k factors were considered, each at 3 levels, generally referred to as low (-1), intermediate (0) and high (+1) levels, as presented in Figure 1. The reason three-level designs were proposed is to model possible curvature in response function and deal with case of nominal factors at 3 levels. A third level for a continuous factor facilitates the investigation of a quadratic relationship between the response and each of the factors.

DoE performed was a simple method to understand the influence of exposure time and percentage of GNP addition on natural-based composite bending performance. The factorial method was chosen because it is appropriate method when several factors must be studied at two or more levels and interactions between these factors may be important. As for its structure, in each complete repetition of experiment, all possible combinations of factor levels (treatments) are studied.

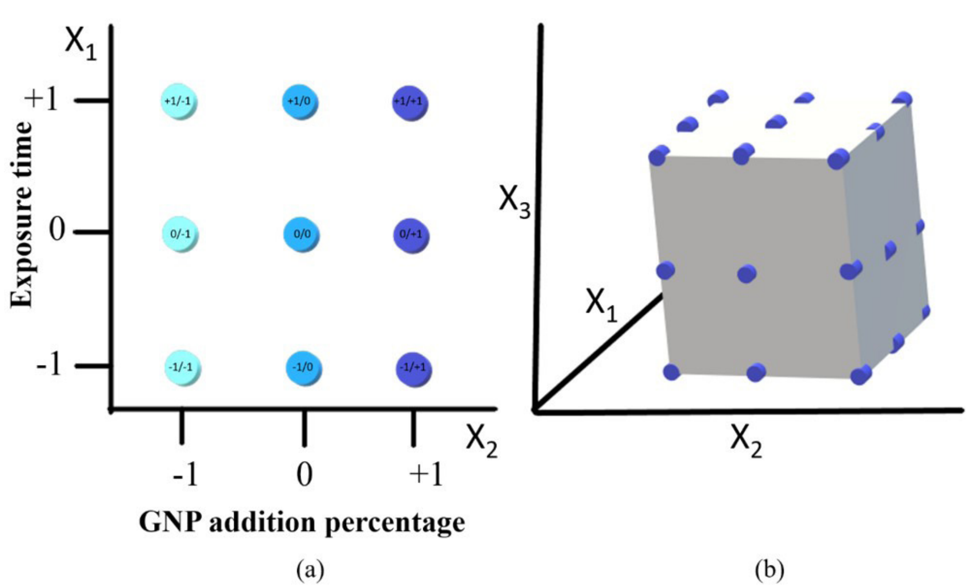


Figure 1. 3² design schematic considered (a) flat and (b) 3D perspectives.

The experimental allocation units to treatments and order in which tests are performed are done randomly. To improve statistical significance of factorial 3^2, factorial points were tested in duplicate.

The response was analyzed using multiple regression analysis (MRA) statistics. To apply factorial 3^2, it is necessary to code variables using the Equation 3.

$$c_i = \frac{t_i - t_m}{\delta} \tag{3}$$

Where t_i is the exposure time i (i=-1 for 0 days; i=0 for 5 days and i=+1 for 10 days); c_i is the coded factor value assumed by t_i; t_m is the average value of exposure time and δ is the interval $|t_i - t_m|$. The same is valid for percentage of GNP addition i (i=-1 for a 0.1%; i=0 for a 0.5% and i=+1 for a 0.9%). The MRA procedure consists of finding polynomial equation that best describes the phenomenon studied. In this work, a 2nd order equation was considered [1]. The model fitted with coded variables is shown in Equation 4.

$$\Delta\gamma;\sigma;E\left[ET,GNP\right]: a + b_1c_1 + b_2c_2 + b_3c_1c_2 + b_4c_1^2 + b_5c_2^2 + \varepsilon' \tag{4}$$

Where c_i is the value of coded factor assumed by t_i; a is the linear regression coefficient, b_i is the regression slope and $\dot{\varepsilon}$ is the error. The interaction effect is failure of one variable to produce the same effect on response at different levels of another variable.

2.4 Scanning Electron Microscopy (SEM) analysis

Microstructural properties, damage mechanisms, and degradation were monitored using scanning electron microscopy (SEM) images of fique fabric-composite in aged state. A model Quanta FEG250 FEI microscope Thermofisher Scientific, Hillsboro, OR, USA, operating with secondary electrons at 20 kV was used. Samples were gold-sputtered for electron conduction.

3 Results and discussion

3.1 Flexural parameters

Composite without GNP addition and without exposure, in other words, the control group showed deflection at fracture of 6.75 ± 2.12 mm, flexural modulus of rupture of 77.97 ± 27.08 MPa and flexural modulus of elasticity of 5.22 ± 2.29 GPa. As compared with control group, it can be observed that as GNP content increases in composite, greater its flexural modulus of rupture. Young's modulus showed a high decrease, and deflection at fracture was proportional increased with incorporation of GNP content.

Table 1 also shows that, from the first exposure to high temperature, flexural modulus of rupture decreases, but there is an increase in flexural modulus of elasticity (101%; 170%; 200%). With increase of GNP content, flexural modulus of rupture and modulus of elasticity increased. With continued exposure to high temperature, flexural modulus of rupture significantly decreased (-15%; -17%; -9%), while flexural modulus of elasticity tended to increase (136%; 168%; 218%).

3.2 Normality test

Normality testing is usually performed for a dataset to ensure that sample data represents the population of data. The normality test was performed using Ryan-Joiner test, like as Shapiro-Wilk test, and it showed that P value is >0.10 which is greater than 0.05. Thus, the data is considered normal, once p-value is greater than 0.05. Figure 2 shows normal probability plot for flexural deflection at fracture, modulus of rupture, and elasticity.

The null hypothesis of Ryan-Joiner test is that population is normally distributed. Thus, if p-value is less than chosen α level (α=0.05), then null hypothesis is rejected and there is evidence that tested data are not normally distributed. On the other hand, if p-value is greater than chosen α level, then null hypothesis cannot be rejected.

Table 1. Arrangement of the 3-level full factorial experimental design

3^2 FULL FACTORIAL EXPERIMENTAL NUMBER	PROCESS PARAMETERS					RESPONSE VARIABLES		
	A	B	AB	A^2	B^2	Deflection at Fracture (mm)[y]	MOR (MPa) [σ]	MOE (GPa) [E]
1	-1	-1	1	1	1	16.15 ± 1.04	70.16 ± 7.67	0.77 ± 0.06
2	0	0	0	0	0	6.85 ± 2.16	75.75 ± 1.75	2.08 ± 0.70
3	1	0	0	1	0	5.14 ± 0.15	58.35 ± 5.72	2.07 ± 0.24
4	-1	0	0	1	0	13.22 ± 0.33	79.34 ± 17.27	0.91 ± 0.24
5	0	-1	0	0	1	8.11 ± 0.92	65.32 ± 7.21	1.55 ± 0.10
6	0	1	0	0	1	7.37 ± 0.17	92.14 ± 1.62	2.31 ± 0.03
7	1	-1	-1	1	1	6.25 ± 1.27	59.71 ± 8.42	1.82 ± 0.13
8	1	1	1	1	1	5.02 ± 0.5	63.92 ± 3.41	2.45 ± 0.21
9	-1	1	-1	1	1	11.72 ± 0.31	81.34 ± 14.58	1.03 ± 0.32

A: Exposure time; B: GNP addiction percentage; GPa: Gigapascal; MOR: Modulus of Rupture; MOE: Modulus of Elasticity; MPa: Megapascal.

3.3 Analysis of Variance (ANOVA) and Multiple Regression Analysis (MRA)

The results were obtained with Minitab 19 edition software. In addition, a step-by-step method was used for potential terms (A, B, AB, A^2, B^2), with rule of "α to insert: 0.15 and α to remove: 0.15".

3.3.1 Deflection at fracture

The highest deflection at fracture was found for the configuration (-1) (-1), ie, without exposure to high temperature and with GNP addition of 0.1%. It is noteworthy that in all conditions without exposure obtained high values of deflection level, and deflection at fracture had its potential reduced with the increase of GNP percentage. The smallest deflection at fracture was observed for (1) (1) configuration, as expected, since material embrittlement occurs after exposure to high temperature and graphene nanoplates can serve as stress concentrators, which may be a fracture preferred path. Table 2 presents full factor regression data for deflection at fracture.

It can be seen from Table 2 that there is good fit of the regression data to experimental data, through S "ANOVA parameter" definition. Another point is the correlation fit, which was very satisfactory. Equation 5, based on predictor

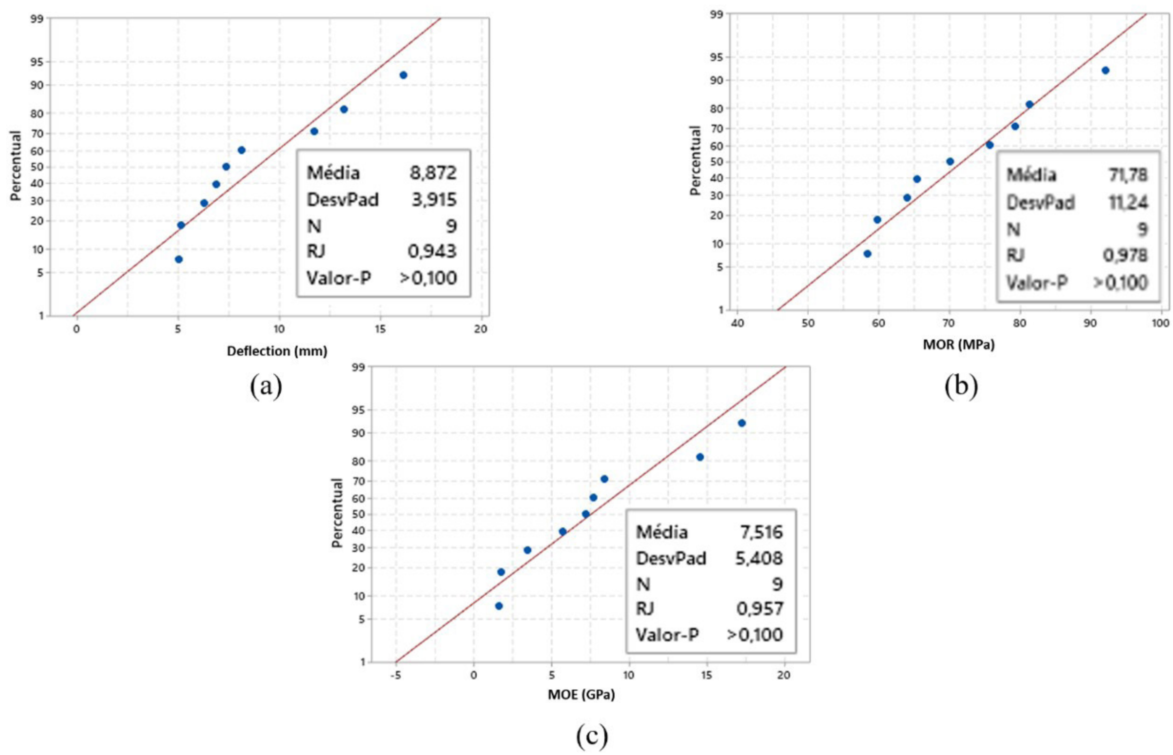


Figure 2. Normal probability plot for flexural data: (a) deflection at fracture, (b) modulus of rupture and (c) modulus of elasticity.

Table 2. Analysis of variance with CI of 95% for model and factors (deflection at fracture)

ANOVA						
Source	**Degrees of freedom**	**Sum of squares**	**Mean square**	**F**	**P-value**	**Significance**
Model	4	120.1	30.0	48.23	0.001	Significant
A	1	101.5	101.5	163.1	0.000	
B	1	6.81	6.81	10.9	0.030	
AB	1	2.56	2.56	4.12	0.112	-
A^2	1	9.16	9.16	14.7	0.019	
Residue	4	2.49	0.62			
Total	8	122.6				

$$\Delta\tilde{\mathbf{a}} = (7.445 \pm 0.456) - (4.115 \pm 0.322)\mathbf{A} - (1.066 \pm 0.322)\mathbf{B} + (0.801 \pm 0.395)\mathbf{AB} + (2.141 \pm 0.558)\mathbf{A}^2 \quad (5)$$

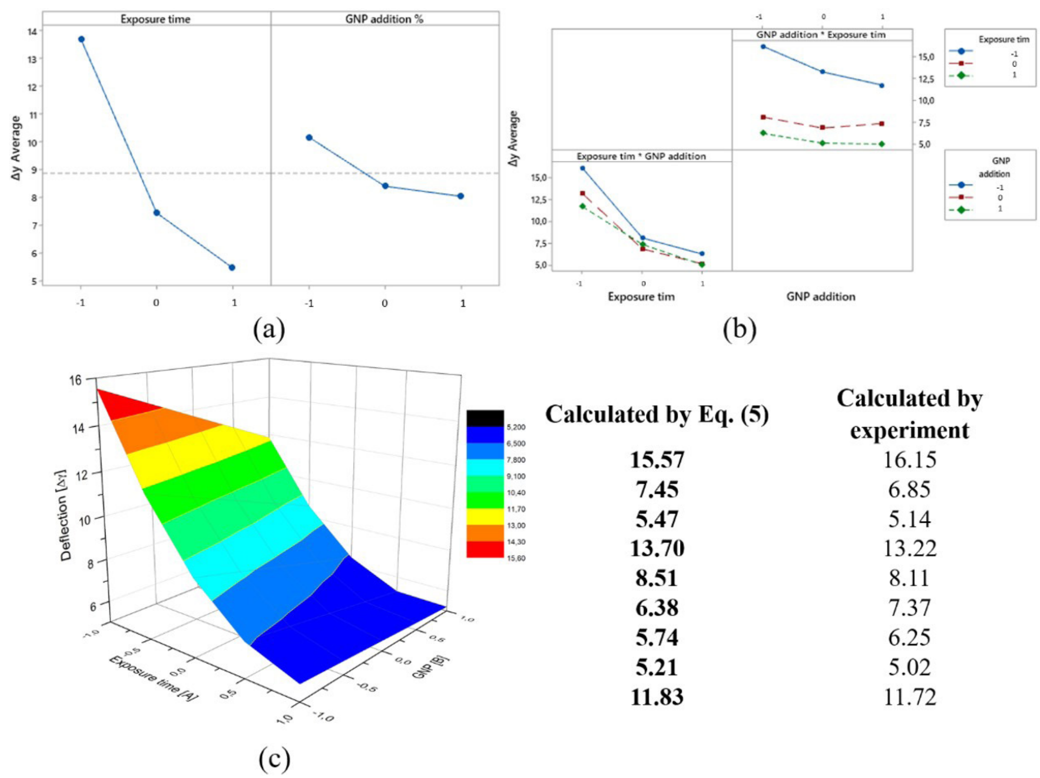
Model summary			
S (mm)	R^2	R^2(adj)	R^2(pred)
0.789	97.97%	95.94%	86.47%

adj: adjusted; F: F-test is used for comparing the factors of the total deviation; P-value: Computer method calculates the probability (p-value) of a value of F greater than or equal to the observed value; A, B: Process parameters presented; S: ANOVA parameter; pred: predictor.

coefficient, has good prediction relationship for this property, and Figure 3 presents the response surface of deflection at fracture data.

3.3.2 Modulus of rupture

The matrix configuration that generated better performance in terms of flexural modulus of rupture was (-1) for exposure time, i.e., after 0 hours under high temperature, and (0) for GNP percentage, i.e., 0.5%. However, it was the one with highest standard deviation. Another group that stood out was (0) (1), with 120 hours at high temperature and GNP addition of 0.9% in epoxy matrix. In contrast, the worst performance was for configuration (1) (-1), considering exposure time of 240 hours and with lowest GNP addition, i.e., 0.1%. Another group that stood out negatively was (1) (0), it is suggested that there is great influence of thermal degradation on composite behavior. Table 3 shows what was previously mentioned, greater influence of exposure time on composite flexural performance. Note, from Eq. 6, that factor loadings of both exposure time and GNP addition are negative, therefore, the best results of flexural modulus of rupture will occur in conditions without exposure to degradation (-1) and with addition of up to 0.1% GNP (-1).



Calculated by Eq. (5)	Calculated by experiment
15.57	16.15
7.45	6.85
5.47	5.14
13.70	13.22
8.51	8.11
6.38	7.37
5.74	6.25
5.21	5.02
11.83	11.72

Figure 3. (a) Main effect plot for Exposure time and GNP addition%, (b) interaction plot for deflection at fracture and (c) 3D plot of deflection at fracture as function of exposure time and GNP content.

Table 3. Analysis of variance with CI of 95% for model and factors (flexural strength)

Source	Degrees of freedom	Sum of squares	Mean square	F	P-value	Significance
Model	3	854.3	284.7	9.10	0.018	Significant
A	1	397.8	397.7	12.7	0.016	
B	1	296.9	296.9	9.49	0.027	
A^2	1	159.6	159.6	5.10	0.074	-
Residue	5	156.5	31.30			
Total	8	1010.8				

$$\sigma = (77.74 \pm 3.23) - (8.14 \pm 2.28)\mathbf{A} - (7.03 \pm 2.28)\mathbf{B} - (8.93 \pm 3.96)\mathbf{A}^2 \quad (6)$$

Model summary

S (MPa)	R^2	R^2(adj)	R^2(pred)
5.594	84.52%	75.23%	40.67%

adj: adjusted; F: F-test is used for comparing the factors of the total deviation; P-value: Computer method calculates the probability (p-value) of a value of F greater than or equal to the observed value; A, B: Process parameters presented; S: ANOVA parameter; MPa: Megapascal; pred: predictor.

Multiple linear regression model fit was found to be satisfactory, however, the model does not predict with high quality the response regarding modulus of rupture. The S parameter, which represents standard deviation of distance between data values and fitted values, was high, indicating that the model does not accurately describe modulus of rupture. The model seems promising ($R^2 \approx 85\%$), however, predicted R^2 is only 41%, which indicates that the model may be overfit. Figure 4 shows a more parabolic shape when compared to the response surface plot generated for deflection at fracture.

3.3.3 Modulus of elasticity

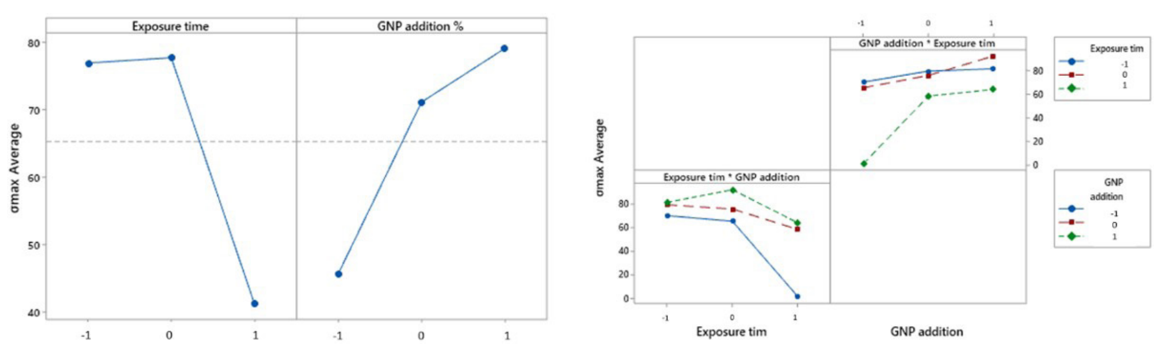
MOE is intrinsically correlated with elastic deformation of materials, determining whether the material is susceptible to this deformation type. Therefore, modulus of elasticity is strongly associated with material stiffness and can be defined as material resistance to elastic deformation, as well as greater this modulus, more rigid the material and smaller elastic deformation resulting from a stress.

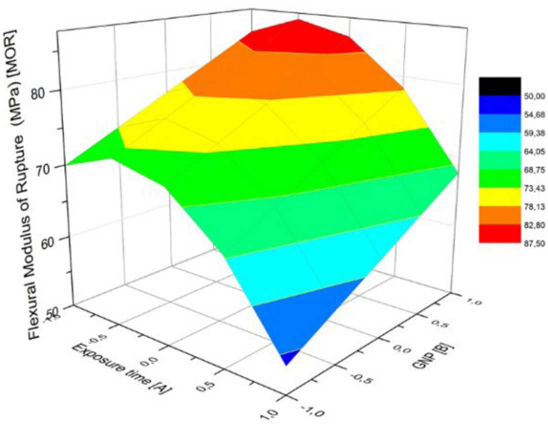
GNP incorporated in epoxy matrix affected the material performance, since its rigidity was reduced when compared to group without the GNP addition. However, increasing GNP content, an increase in material stiffness can be observed in Figure 5. The highest value obtained for modulus of elasticity is related to the condition (1) (1), i.e., with 0.9% of GNP and exposure for 240 hours at high temperature. In contrast, the material that obtained lowest modulus of elasticity was with configuration (-1) (-1), i.e., with addition of only 0.1% of GNP and without exposure to high temperature. Table 4 presents the data obtained by ANOVA of flexural modulus of elasticity.

The data variation is well explained from the model summary, once the correlation coefficient (R^2) is high. Another relevant factor is predicted R^2, which was satisfactory.

3.4 Morphological analysis

Figure 6 shows micrograph of epoxy matrix with the GNP addition of 0.1% (a), 0.5% (b) and 0.9% (c). It can be observed that there is a concentration of graphene nanoplates in sample with the GNP addition of 0.1%, this can be an indicator of stress concentration, once with greater additions of graphene nanoplates this phenomenon was not observed, showing good homogenization with epoxy matrix. Figure 6(d) to 6(f) show fracture after composite bending test without addition of nanoplates, without and with aging of 5 days, and 10 days, respectively.





Calculated by Eq. (6)	Calculated by experiment
83.98	70.16
77.74	75.75
60.67	58.35
76.95	79.34
84.77	65.32
70.71	92.14
67.70	59.71
53.64	63.92
69.92	81.34

(c)

Figure 4. (a) Main effect plot for Exposure time and GNP addition%, (b) interaction plot for MOR and (c)3D plot of MOR as function of exposure time and GNP content.

Figure 6(g) shows sample with 0.1% GNP addition without exposure to high temperature, while Figure 6(h) and 6(i) show samples with addition of 0.5% GNP with 5 days under thermal aging and 0.9% GNP with 10 days under thermal aging, respectively.

Due to wide variety of factors, it is extremely difficult to predict where and how damage forms and even its propagation in composite material. The main types of damage found in these composites were matrix cracking, fiber rupture, fiber/matrix debonding, delamination.

Cracking in matrix, fiber breakage and fiber matrix detachment are types of damage that can occur in any fibrous composite material, but delamination occurs in laminated composites. It is important to note that the increase in composite damage impairs the mechanical properties of laminate so that its elastic constants decrease.

Table 4. Analysis of variance with CI of 95% for model and factors (flexural stiffness)

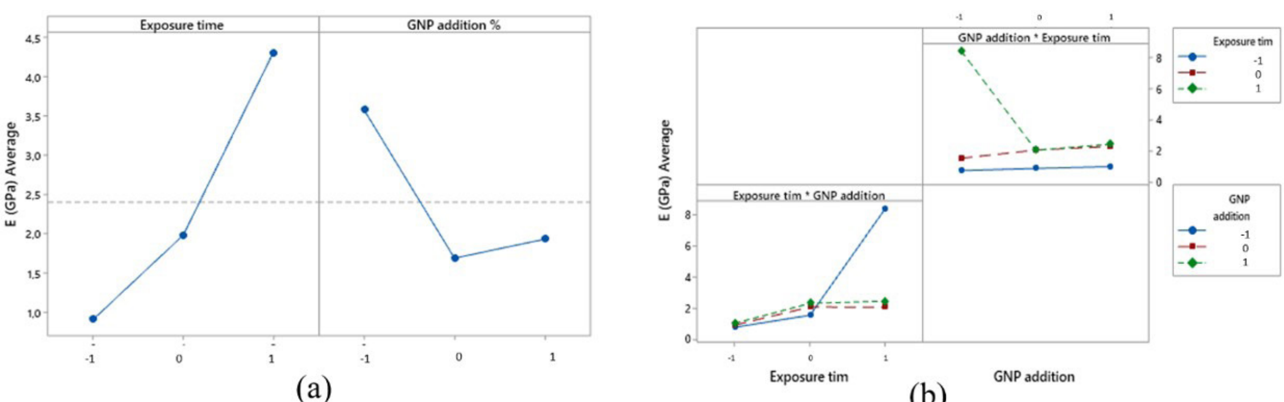
Source	Degrees of freedom	Sum of squares	Mean square	F	P-value	Significance
Model	3	3.08	1.02	59.18	0.000	Significant
A	1	2.18	2.18	125.7	0.000	
B	1	0.45	0.45	26.13	0.004	
A^2	1	0.44	0.44	25.63	0.004	
Residue	5	0.08	0.01			
Total	8	3.17				

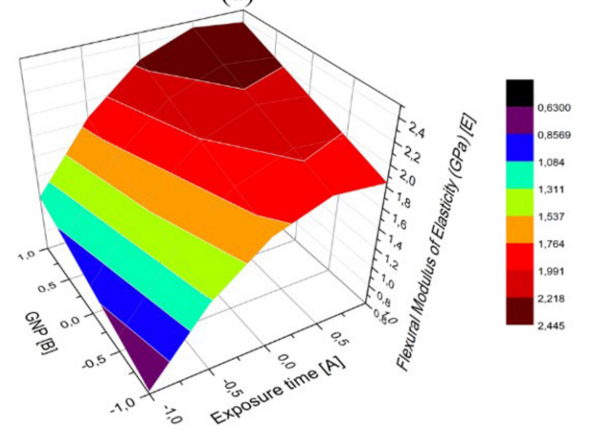
$$\mathbf{E} = (1.982 \pm 0.076) + (0.603 \pm 0.053)\mathbf{A} + (0.275 \pm 0.053)\mathbf{B} - (0.472 \pm 0.093)\mathbf{A}^2 \quad (7)$$

Model summary

S (GPa)	R^2	R^2(adj)	R^2(pred)
0.132	97.26%	95.62%	89.71%

adj: adjusted; F: F-test is used for comparing the factors of the total deviation; P-value: Computer method calculates the probability (p-value) of a value of F greater than or equal to the observed value; A, B: Process parameters presented; S: ANOVA parameter; GPa: Gigapascal; pred: predictor.





Calculated by Eq. (7)	Calculated by experiment
0.63	0.77
1.98	2.08
2.11	2.07
0.91	0.91
1.71	1.55
2.26	2.31
1.84	1.82
2.39	2.45
1.18	1.03

(c)

Figure 5. (a) Main effect plot for Exposure time and GNP addition%, (b) interaction plot for MOE and (c) 3D plot of MOE as function of exposure time and GNP content.

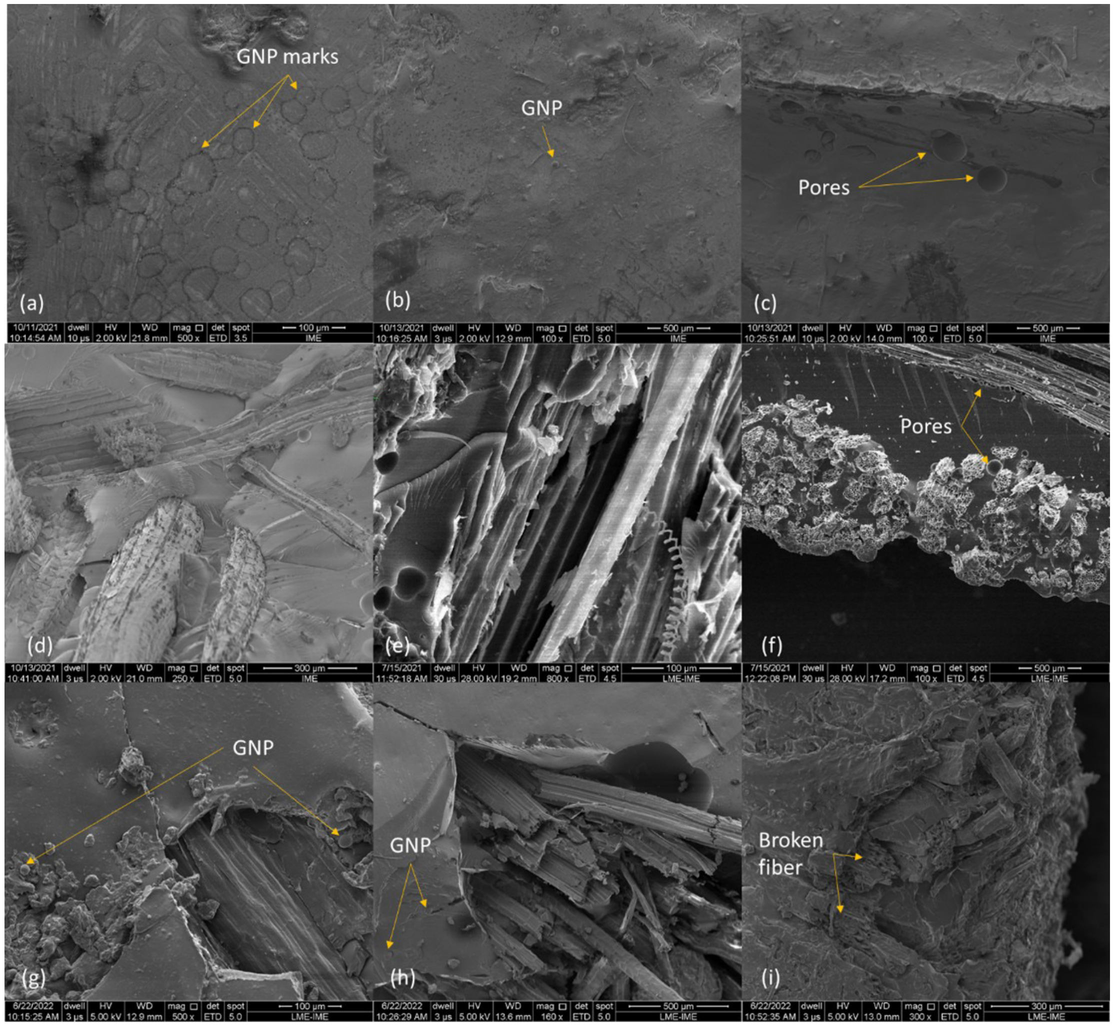


Figure 6. Micrographs of epoxy samples with GNP addition of 0.1% (a), 0.5% (b) and 0.9% (c) without thermal aging; composite without GNP addition and without aging (d), with aging of 5 days (e) and 10 days (f); of composite configurations (-1) (-1) (g), (0) (0) (h) and (1) (1) (i), respectively.

4 Conclusions

Bending test is one of the main mechanical tests used to guarantee quality control and performance evaluation of materials under this type of request. The results showed that two factors have a significant impact on flexural properties. Based on results, it can be concluded that experimental design methodology associated with multiple regression and response surface is a tool that allows evaluation of flexural behavior of aged and no-aged composite reinforced with fique fabric with addition, or not, of the graphene nanoplates. Reducing sample space and estimating more accurately material behavior.

Acknowledgements

The authors would like to thank the Brazilian agencies CNPq (process E-151276/2022-6) and FAPERJ (process E-26/202.045/2020) for their support, as well as MGgrafeno for the material provided and support in the research.

References

1 Thyavihalli Girijappa YG, Mavinkere Rangappa S, Parameswaranpillai J, Siengchin S. Natural fibers as sustainable and renewable resource for development of eco-friendly composites: a comprehensive review. Frontiers in Materials. 2019;6:226.

2 Luz FSD, Garcia FC Fo, Oliveira MS, Nascimento LFC, Monteiro SN. Composites with natural fibers and conventional materials applied in a hard armor: a comparison. Polymers. 2020;12(9):1920.

3 Karimah A, Ridho MR, Munawar SS, Adi DS, Ismadi, Damayanti R, et al. A review on natural fibers for development of eco-friendly bio-composite: characteristics, and utilizations. Journal of Materials Research and Technology. 2021;13:2442-2458.

4 Monteiro SN, Assis FS, Ferreira CL, Simonassi NT, Weber RP, Oliveira MS, et al. Fique fabric: a promising reinforcement for polymer composites. Polymers. 2018;10(3):246.

5 Wang X, Petrů M. Degradation of bending properties of flax fiber reinforced polymer after natural aging and accelerated aging. Construction & Building Materials. 2020;240:117909.

6 Oliveira MS, Luz FS, Costa Garcia F Fo, Pereira AC, Oliveira Aguiar V, Lopera HAC, et al. Dynamic mechanical analysis of thermally aged fique fabric-reinforced epoxy composites. Polymers. 2021;13(22):4037.

7 Kuram E. UV and thermal weathering of green composites: comparing the effect of different agricultural waste as fillers. Journal of Composite Materials. 2020;54(24):3683-3697.

8 Sanjay MR, Arpitha GR, Naik LL, Gopalakrishna K, Yogesha B. Applications of natural fibers and its composites: an overview. Natural Resources. 2016;7(3):108-114.

9 Braga FDO, Lopes PHLM, Oliveira MS, Monteiro SN, Lima ÉP Jr. Thickness assessment and statistical optimization of a 3-layered armor system with ceramic front and curaua fabric composite/aluminum alloy backing. Composites. Part B, Engineering. 2019;166:48-55.

10 Koronis G, Silva A, Foong S. Predicting the flexural performance of woven flax reinforced epoxy composites using design of experiments. Materials Today. Communications. 2017;13:317-324.

11 Kumar R, Singh RP, Kataria R. Study on flexural performance of fabricated natural fiber hybrid polypropylene composite: an experimental investigation through designed experiments. World Journal of Engineering. 2019;16(3):389-400.

12 Liu F, Shi Y, Najjar L. Application of design of experiment method for sports results. Procedia Computer Science. 2017;122:720-726.

13 Nurulhuda A, Hafizzal Y, Izzuddin MZM, Sulawati MRN, Rafidah A, Suhaila Y, et al. Analysis on flexural strength of A36 mild steel by Design of Experiment (DOE). In: IOP Conference Series: Materials Science and Engineering. Bristol: IOP Publishing.

14 Badizi RM, Parizad A, Askari-Paykani M, Shahverdi HR. Optimization of mechanical properties using D-optimal factorial design of experiment: Electromagnetic stir casting process of A357−SiC nanocomposite. Transactions of Nonferrous Metals Society of China. 2020;30(5):1183-1194.

15 Silva BJ, Menezes R, Santana L, Melo L, Neves GA, Ferreira HC. Use of statistical techniques to model the flexural strength of ceramic bodies containing granite waste. Matéria (Rio de Janeiro). 2012;17(1)

16 Rostamiyan Y, Fereidoon A, Rezaeiashtiyani M, Hamed Mashhadzadeh A, Salmankhani A. Experimental and optimizing flexural strength of epoxy-based nanocomposite: Effect of using nano silica and nano clay by using response surface design methodology. Materials & Design. 2015;69:96-104.

17 Rua J, Buchely MF, Monteiro SN, Echeverri GI, Colorado HA. Impact behavior of laminated composites built with fique fibers and epoxy resin: a mechanical analysis using impact and flexural behavior. Journal of Materials Research and Technology. 2021;14:428-438.

18 Şükür EF, Önal G. Graphene nanoplatelet modified basalt/epoxy multi-scale composites with improved tribological performance. Wear. 2020;460:203481.

Received: 27 June 2022
Accepted: 17 May 2023