



## Technical Report

# Experimental and optimizing flexural strength of epoxy-based nanocomposite: Effect of using nano silica and nano clay by using response surface design methodology



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## ABSTRACT

The influence of weight percentage of nano silica and nano clay and also fiber orientation on the flexural strength of epoxy/glass fiber/nano SiO<sub>2</sub>/nanoclay hybrid laminate composite has been investigated in the current study. The response surface design methodology was employed for predicting flexural behavior of new mentioned hybrid nano composite and also to present a mathematical model as a function of physical factors in order to reach the optimum structure of new hybrid nano composite. Using Minitab software totally 20 experiments were generated with 6 replicates at center points and flexural tests were done on 20 prepared samples. Analysis was replicated after eliminating non effective terms and also optimizing was done using optimization option of Minitab software. The optimization results indicated that the best flexural strength obtained from the software was 17.77 MPa which occurred in 3.5 wt% of nano-silica, 4 wt% of nanoclay and 0° of fiber orientation and after preparing and testing five samples average value of flexural strength was obtained 17.2 MPa.

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## 1. Introduction

Composite materials have some advantages offered by polymer matrices like economic efficiency properties, environmentally friendly nature, and high chemical resistance with the high stiffness and strength of fiber. On the other hand, these superior mechanical properties are the major reason of using such materials in new-tech industries [1].

Recent studies done in the field of composite materials have noted on developing of their thermal and mechanical properties. This aim can be achieved by incorporating fiber and resin [2]. Fiber leads the stress to be distributed throughout the restoration and improves the structural properties of material by acting as crack stoppers [2]. Glass fiber is most commonly used material comparing with the other kinds of fibers, since it can improve the in-plane mechanical properties much better than the others. Glass fiber is one of the best choices for maximizing mechanical strength and toughness. Panthapulakkal and Sain [3] studied mechanical and thermal properties of hemp/glass fiber–polypropylene composite.

They reported that additional of glass fiber into hem–polypropylene composite improved thermal properties. Eronat et al. [4] studied Effects of glass fiber layering on the flexural strength of microfill and hybrid composites and they reported that Glass fiber layering of microfill and hybrid composites presented higher flexural strength, and veneering of hybrid composite with microfill composite increased the resistance of the restoration.

Bekyarova et al. [5] reported that using carbon fiber/epoxy reinforced with carbon nanotubes shows a great laminar strength (~50 MPa). Also Gojny et al. [6] obtained that adding nanoclay to the glass fiber reinforced composite improves the mechanical properties considerably.

Resin matrix protects fiber, does geometrical arrangement and also supports the reinforcement. As mentioned above, glass fiber reinforced composites have good in-plane mechanical properties while the out-of-plane properties of these composites are poor and additionally weak fiber–matrix interfacial adhesions may occur if polymer matrices impregnation with fiber is not fully applied [6,7]. Using two kinds of polymer matrices is commonly applied to overcome these issues, adding the first matrix improve mechanical and thermal properties, and using the second one improves the wetting property infiltration and solves fiber–matrix

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interfacial adhesions [8]. Epoxy resins are widely used as one of the most important classes of polymer matrix composite material because of their good wetting ability, low viscosity and high activity, excellent mechanical and thermal properties and also fracture toughness [4,9]. Xu and Hoa [10] showed that adding a low weight percentage of nanoclay to the fiber/epoxy composites improved its flexural strength by 38%. Gojny et al. [11] reported that using 0.3 wt% multi-walled carbon nanotube in glass fiber/epoxy significantly increases inter laminar shear strength of the glass fiber. The main goal of most of the researches in the field of polymer nanotechnology is improvement of the thermal, electrical, optical, and mechanical properties of these materials [12]. Many researchers had improved epoxy resin performance by incorporating thermoplastic fillers, rubber agents, diluents, and nanoparticles into the epoxy. Inorganic nanoparticles have the potential to be used as a reinforcing material due to their low cost, ease of fabrication, and environmentally friendly nature [13]. Becker et al. [14] showed that adding Nanomer I.30E nanoclay into diglycidyl ether of bisphenol A (DGEBA) type epoxy resin increased its fracture toughness and elastic modulus toughness through the incorporation of layered silicates. Mirmohseni and Zavareh [15] filled epoxy resin with 2.5 wt% organically modified clay and obtained that its tensile modulus and strength and also its impact strength were increased remarkably compared to those of the neat epoxy. Zheng et al. [16] reported that adding 3% SiO<sub>2</sub> into epoxy matrix increases tensile strength and impact strength 115% and 56% respectively. Shokrieh et al. [17] founded that adding 0.25 wt% of carbon nanotube increased tensile and flexural strength 23% and 10% respectively compared to neat resin. Akbari et al. [18] used liquid carboxyl-terminated butadiene acrylonitrile (CTBN) to toughen epoxy resin and 26% improvement in tensile strength was observed when 5 phr CTBN was added.

Researchers were persuaded to use hybrid nanocomposites in order to achieve higher mechanical properties and crack propagation resistant. If used two or more kind of nano or micro particle in matrix, the composite is named hybrid nanocomposite [19]. Rostamiyan et al. [20] filled epoxy resin with HIPS as thermoplastic phase and nanoclay as a nano reinforcement and reported synergistic effects on mechanical properties. Their results showed that the tensile, compressive, and impact strengths of new ternary nanocomposite were improved up to 60%, 64%, and 402%, respectively higher than those of the neat resin. Fereidoon et al. [21] showed that using multi-walled carbon nanotube in present of high impact polystyrene improved mechanical properties such as tensile, compression and impact. Mirmohseni et al. [22] reported that epoxy/ABS/nanoclay/TiO<sub>2</sub> hybrid nanocomposite has synergistic effect on impact strength.

Various factors may be affected significantly, in the preparation of hybrid nanocomposite samples, so control of these parameters and optimizing them seems to be necessary [23]. OVAT (one variation at time) is a method for analyzing and optimizing significant parameters, but this method can only optimize one variable at a time, while in the most of studies under review variables depends on each other and the interactions between them should be determined, in this situations OVAT cannot ensure us about finding the real optimum point. For example Mirmohseni and Zavareh [15] determined optimum amount of hardener according to maximum tensile and impact strength of the prepared epoxy samples. Leardi [24] claimed that 93% of the published papers in 2009 with general titles containing “optimization”, “development”, “improvement” or “effect of”, employed the OVAT model. In addition, prediction of nonlinear effects of each parameter is an important note needed at least three points as parameter levels that directly increase number of required experiments for model prediction and consequently increases the costs. Central Composite Design (CCD) is one of the most commonly used sequential second order

experimental designs which is able to decrease the number of experiments, predict the possible nonlinear effect of each parameter and also the effect of interactions between them [25].

In the current paper flexural testes were carried out to evaluate flexural strength of glass fiber/epoxy/nanoclay/nanosilica quadratic hybrid laminates nanocomposite. Central composite design was employed to present a mathematical model for predicting mechanical behavior of mentioned new nanocomposite. Weight percentage of nanoclay and nano silica and fiber orientation [26] considered as coefficient parameter on flexural strength. Morphological and structural characteristics of the hybrid mechanism were investigated using scanning electron microscopy (SEM).

## 2. Experimental details

### 2.1. Details of materials

Epoxy resin utilized in this study was an undiluted clear difunctional bisphenol A, Epon 828 provided by Shell Chemicals Co. With epoxide equivalent weight 185–192 g/eq. Epon 828 is basically DGEBA (Diglycidyl ether of bisphenol-A). The curing agent was a nominally cycloaliphatic polyamine, Aradur<sup>®</sup> 42 supplied by Huntsman Co. The spherical silica nanoparticles with average particle size 10–15 nm and SSA (specific surface area) 180–270 m<sup>2</sup>/g were supplied from TECNAN Ltd. The organoclay Cloisite 30B was purchased from Southern Clay Products (Gonzales, TX, USA). E-glass unidirectional unitex (UT) with the areal density of 250 g/m<sup>2</sup> obtained from Gurit Holding AG, Wattwil/Switzerland. The solvent used was Tetrahydrofuran (THF) with purity (GC) more than 99% provided from Merck Co (Germany).

### 2.2. Characterization

Flexural tests were conducted based on 3-point bending according to ASTM: D790. This test method covers the determination of the flexural properties of reinforced plastics, including high-modulus composites. The dimensions of the specimens were 127 by 12.7 by 3.2 mm with a support span-to-depth ratio of 16. Mechanical tests were conducted using an STM-150 universal testing machine from Santam Company (Tehran, Iran) with a load capacity of 150 KN.

### 2.3. Sample preparation

The laminate plates were prepared with 6 layers with 2 mm thickness and also with different fiber orientations based on CCD method. For preparing each sample E-glass (UT) was laid-up with the specific sequence. The whole procedures of reinforcing the resin were done in an appropriate solvent to prepare homogenous mixture. In this way comparable results can be obtained and unwanted various solvent effects will be prevented. Tetrahydrofuran was employed as the solvent for all of the mixture components, including epoxy resin, organo clay and nano silica. Liquid epoxy resin was poured into an adequate amount of THF solvent, so in this way comparable neat epoxy samples could be prepared. Mixing by using a magnetic stirrer last 30 min, after that the solvent was completely evaporated using a vacuum Erlenmeyer. In the current study, the mixture was homogenized by ultrasonication (Ultrasonic SONOPULS-HD3200, 50% amplitude, 20 kHz, and pulsation; ON for 10 s and off for 3 s) for 30 min. At this stage 23 per (per hundred resins) of cycloaliphatic polyamine was added as hardener based on stoichiometric ratio. The mixture degassed using the vacuum pump to remove air bubbles. All of the specimens were prepared with the handy lay-up method and all of them were cured at room temperature for 24 h and followed post-curing from

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