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The strength of glass fiber composite materials by inclusion of $CaCO_3$ and SiO_2 nanoparticles into resin

$CaCO_3$ ve SiO_2 nano parçacıkların reçineye ilavesi ile cam elyaf kompozit malzemelerin mukavemeti

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Abstract

In this study, the effects of $CaCO_3$ and SiO_2 nanoparticles on tensile strength of E-Glass fiber composite materials were investigated. Composite structures were produced by mixing $CaCO_3$ nanoparticles into 3%, 5% and 10% by weight, SiO_2 nanoparticles into 1%, 3% and 5% by weight epoxy resin. High nanoparticle rates are due to the large diameter of the powders and the desire to form a darker resin. In addition, different fiber orientations were used to emphasize inclusion of nano-additive materials into the resin in case of a decrease in the number of unidirectional fibers. Samples prepared according to the ASTM D3039 Standards were tested by uniaxial tensile machine, Instron 8801. Scanning electron microscopy analysis were performed and failure was mainly caused by debonding and clumping of fibers and fiber/matrix failure. An inclusion of $CaCO_3$ and SiO_2 nanoparticles improved the strength of the glass fiber composite sandwich structures. The optimum amount of nano-particles supplemented with different ratios were determined as 3% for $CaCO_3$ and 1% for SiO_2. Sufficient enough nanoparticles inclusion increased the quality of the adhesion between fiber and matrices. The inclusion of nanoparticle additives in E-Glass fiber composite materials had a significant effect and positively affected the adhesion properties of the matrix.

Keywords: Glass fiber, $CaCO_3$, SiO_2, Epoxy resin, Tensile strength.

Öz

Bu çalışmada, $CaCO_3$ ve SiO_2 nano parçacıkların E-Cam elyaf kompozit malzemelerin çekme dayanımı üzerindeki etkileri araştırılmıştır. Kompozit plakalar, $CaCO_3$ nano parçacıkların ağırlıkça %3, %5 ve%10, SiO_2 nano parçacıkların ağırlıkça %1, %3 ve%5 oranında epoksi reçineye karıştırılmasıyla üretilmiştir. Yüksek nano parçacık oranları, tozların geniş çapından ve daha koyu bir reçine oluşturma arzusundan kaynaklanmaktadır. Ek olarak, tek yönlü elyaf sayısında azalma olması durumunda nano katkı maddelerinin reçineye dahil edilmesini vurgulamak için farklı elyaf oryantasyonları kullanılmıştır. ASTM D3039 Standartlarına göre hazırlanan numuneler tek eksenli çekme test cihazı, Instron 8801 ile test edilmiştir. Taramalı elektron mikroskobu analizler yapılarak hasarların liflerin bağlarının ayrılması ve kümelenmesi ve elyaf/matris bozulması olarak gerçekleştiği görülmüştür. $CaCO_3$ ve SiO_2 nano parçacıkların dahil edilmesi, cam elyaf kompozit sandviç yapıların dayanımını arttırmıştır. Farklı oranlarda takviye edilmiş optimum nano parçacık miktarı $CaCO_3$ için%3 ve SiO_2 için%1 olarak belirlenmiştir. Yeterli nano parçacık ilavesi, fiber ve matrisler arasındaki yapışma kalitesini geliştirmiştir. Nano parçacık katkı maddelerinin E-Cam elyaf kompozit malzemelere dahil edilmesi önemli bir etkiye sahipti ve matrisin yapışma özelliklerini olumlu yönde etkilemiştir.

Anahtar kelimeler: Cam elyaf, $CaCO_3$, SiO_2, Epoksi reçine, Çekme dayanımı.

1 Introduction

Composite materials, a new generation plastic material with high performance, have become the preferred materials because of their high strength properties. In particular, polymer matrix composites, which are frequently used in aerospace, marine, automotive and defense industries, are produced by combining epoxy, phenolic or polyester matrix elements and reinforcing them with fibers in different orientations and axes [1]. Today, the biggest advantages of composite materials are; It has become a popular material because of its easy production, environmental friendliness, the less affected impact from water and moisture than metals, corrosion resistance and economical. For this reason, current application areas of composite materials are increasing in many engineering applications. It is especially preferred because of its corrosion resistance and low costs [2],[3]. The use of epoxy resin as a matrix material puts composite materials into an important class due to its superior mechanical properties and better adhesion [4],[6]. For this reason, it is known that the interface layers in epoxy resin composite materials used for bonding cause chemical and structural differences to a great extent affecting mechanical properties [7],[10]. Many researchers have now carried out studies to increase strength by adding various diluents, chopped fibers, nanoparticles to increase mechanical, thermal and electrical properties [11],[16]. Nowadays, it is aimed to

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increase the mechanical and electrical properties of glass fiber composite materials by adding nanoparticles [16]. Recent studies have concentrated on the use of nanoparticles as fillers or additives to enhance the adhesion of the epoxy resin. In this context, they observed that the mixing of hard spherical nanoparticles into epoxy significantly increased the bond density and was a promising approach [17, 20]. Thanks to these additives, it is possible to produce composite materials with superior properties. For this reason, it is important to choose the matrix material that can adapt to glass fiber composite materials and provide suitable environment conditions [21]. Fiber orientation is especially important in tensile testing of polymer matrix composite materials. The fibers, which are oriented parallel or angled to the draw axis, cause an increase in strength in the longitudinal direction. Fibers perpendicular to the drafting axis are weak and may suffer damage even at low tensile forces. Fiber length and fiber density are another factors influence the mechanical properties of material structure [22]. The effects of fiber orientation on tensile strength in composite materials reinforced with short fibers were investigated by Fu et al. [23], they observed an increase in strength by decreasing the fiber orientation angle in glass fiber composite materials. The effects of nanoparticles additive in composite materials were focused by many scientists. Christy et al. [24] mixed ultrasonically SiO_2 nanoparticles with up to 3% epoxy resin as matrix material. They improved impact properties by using the optimum values of nanoparticles supplemented with different ratios.[22] Pedrazelli et al. [25] Polypropylene/glass composite materials with the addition of 7% by weight of silica nanoparticles were investigated on the shear properties and elastic modulus of the material. Consequently, they observed that the addition of nanoparticle additive in hybrid composite materials had a positively significant effect on the adhesion properties of the matrix surface. Shafiur Rahman et al. [26] produced the natural nanoparticles product obtained by mixing the natural calcite structure with polyester resin. In their studies, they synthesized eggshell in the form of natural calcite, analyzed by XRD method and examined the tensile strength, elongation at break, bending strength and impact properties. The best mechanical values were observed under the presence of 10% filler in polyester based films. Kiehl et al. [27] studied the rheological behavior of unsaturated resin and calcite compounds. They used 0-60% nanoparticles as fillers in their studies. They investigated the critical ratio, the effect of waiting time and shear rate on joint properties. Arezoo et al. [28] studied the basalt fiber (BF)/epoxy composites by mixing silanized $CaCO_3$ particles with aminopropyltrimethoxysilane coupling agents at 0%, 1%, 3% and 5% by weight in polymer matrix composites. They observed that strength values increased by 28%, 35%, 20%, and 30%, respectively. Zhidan et al. [29] used three types of nano-SiO_2 powders with Portland fill and ultra-fine fly ash (FA). Spherical SiO_2 powders with an average particle size of 20 nm and a purity of 99.5% were added to the medium. The mechanical properties improved by adding SiO_2 up to a 3%. The microstructure showed that SiO_2 particles dispersed homogeneously and densely distributed in the bonding surface. Paliwal and Sachin [30] produced E-glass/epoxy resin-based composite material, by adding 5%, 10% and 20% $CaCO_3$ particles as fillers. The maximum tensile strength was reached at 141 MPa with 5% filler. Borkar et al. [31] studied the mechanical properties of woven glass fiber composite by adding cost reducing SiO_2 and $CaCO_3$ fillers. The tensile strength and the impact of resistance decreased by 3-15% and 2-12%, respectively. However, the flexural strength increased by 44-58% using filler contents. Baskaran et al. [32] synthesized and characterized nano-sized $CaCO_3$ particles using in-situ deposition technique. Uniformly distributed $CaCO_3$ particles (size of about 50-60 nm) improved the thermal properties and impact strengths.

Our study has focused on the use of SiO_2 and $CaCO_3$ nanoparticle additives in order to improve the quality of the adhesion of the epoxy resin [33]. In the literature the presence of SiO_2 and $CaCO_3$ nanoparticles improved the corrosion resistance/the mechanical properties and toughness of the epoxy, respectively. Optimum amount of addition of nanoparticles used in the epoxy was investigated in the current study.

2 Materials and experimental procedure

E-Glass Fiber and epoxy resin were used in the production of E-Glass Fiber composite materials. The epoxy resin used was a standard Diglycidyl Ether of Bisphenol A and Araldite LY 1564, available from Huntsman, Switzerland. The fiberglass fabric was unidirectional 0° fiber from Huntsman, Switzerland, with a field weight of 330 g/m^2 and 1200 TEX. The SiO_2 powder particle size was average 15 nm and > 99% purity. $CaCO_3$ powders were preferred as white powders having a particle size of 50-80 nm and a purity of > 99%. SiO_2 and $CaCO_3$ powders were purchased from Ege Nanotek Chemical Industry Ltd. Izmir/Turkey.

The epoxy resin and curing agent, which had previously been kept in the oven at about 80 °C temperature, were mixed in a 1,0: 0,8 ratio to form the matrix element. Then, nanoparticles were added to the resin + hardener mixture and mixed with Hielscher UP400S ultrasonic stirrer Figure 1(a) until a homogenous dispersion was achieved (~30 min.). In order to prevent heating of the stirrer probe during the mixture and to prevent hardening, a controlled mixture was made to ensure that the temperature does not exceed 45 °C using Labo CH750 cooler Figure 1(b). SiO_2 powders were mixed at a rate of 1%, 3%, 5% according to the total weight of the resin and hardener. $CaCO_3$ powders were mixed in rate of 3%, 5% and 10%.

Fiberglass composite structures were produced using the hand lay-up procedures shown in Figure 2. The fiber orientation of 90°, 45° and 0° were used to construct the sandwich composite structures with 400 mm x 500 mm dimensions.

The fiber angles 45° and 90° were preferred to construct more stable structures. The prepared layers were left to harden in hot press at 6 bar pressure, 135 °C for 2 hours. Composite sandwich structures were cut by water jet to prepare 25 mm x 250 mm samples according to ASTM D3039/D/D3039M standard [34].

Figure 1(a): Hielscher UP400S ultrasonic stirrer.
(b): Labo CH750 cooler.

Two angle-oriented types of sandwich structures such as Type A, (0° / + 45° / -45° / 90° / 0° / 90° / -45° / + 45°/0° angles) and Type B (90° / + 45° / -45° / 0° / 90° / 0° / -45° / + 45°/90° angles) were prepared. Minimum three specimens were prepared and tested for each configuration. The thickness of the samples was 2.5 ± 0.2 mm in accordance with ASTM standard. The samples were tested by tensile machine which is Instron 8801 with 50 kN load capacity according to ASTM D3039 international standard, shown in Figure 3. The test specimens were placed on the instrument in a fixed manner from the upper and lower jaws of the test device and tests were carried out quasistatic loading at a rate of 0.5 mm/min.

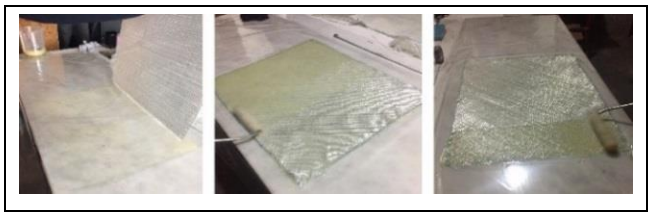


Figure 2. Hand lay-up of composite structures by tilting method.

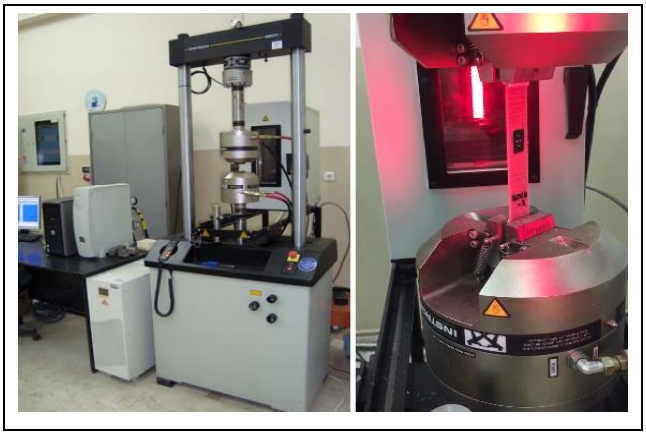
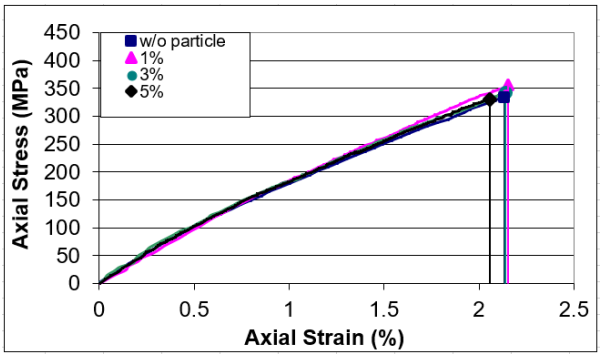


Figure 3. INSTRON 8801 universal test machine.

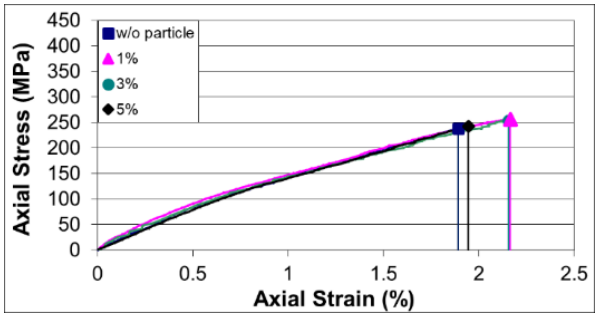
3 Results and discussion

E-Glass/Epoxy composite structures produced using 0%, 1%, 3%, 5% nano silica and 0%, 3%, 5%, 10% nano $CaCO_3$ powders were determined by tensile test for Type A and Type B.

The use of 1% silica admixture increased the tensile strength by 8% for Type A and 10% for Type B relative to the pure composite sample that does not contain any additives, presented in Figures 4 and 5. The optimum value was reached with a contribution rate of 1% and it was observed that the strength of the contribution rate was decreased proportionally in the subsequent contribution rates.



(a)



(b)

Figure 4. SiO_2 Axial stress-axial strain curves of tests samples
(a): Type A 0°/+45°/-45°/90°/0°/90°/-45°/+45°/0°.
(b): Type B, 90°/+45°/-45°/0°/90°/0°/ -45°/+45°/90°.

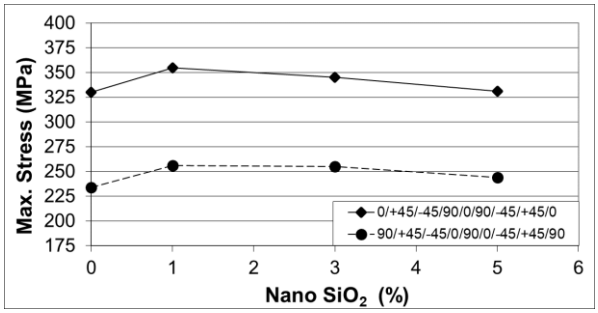
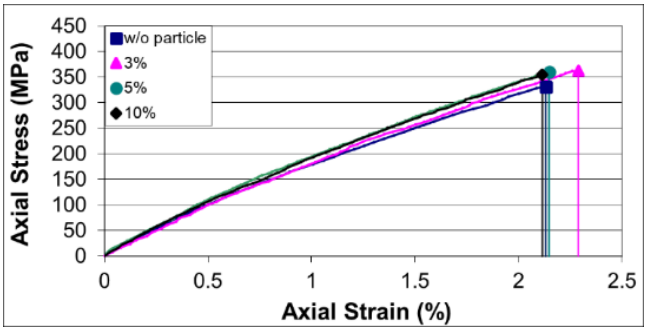
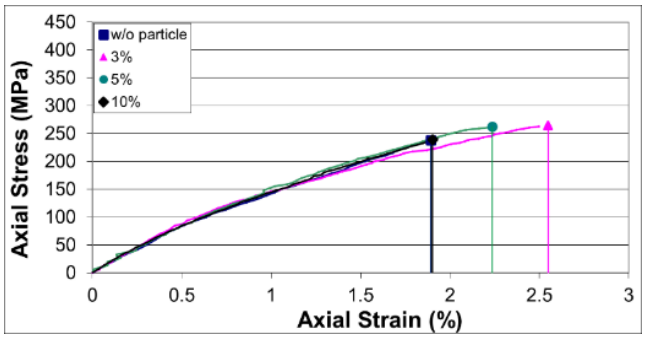


Figure 5. Effect of Nano SiO_2 contribution by weight on the tensile strength.

The values given in the table indicate that the use of silica nanoparticles above 5% silica content will continue at a level lower than the tensile strength of the pure glass fiber composite samples. As shown in Figures 6 and 7, the maximum strength value of the calcite nanoparticles reached with 3% additive inclusion. Tensile strength increased by 10% for Type A and 13% for Type B. Although it generally acts as a filler, the use of low amounts as an additive has led to an increase in tensile strength.



(a)



(b)

Figure 6. $CaCO_3$ Axial stress-axial strain curves of tests samples. (a): Type A 0°/+45°/-45°/90°/0°/90°/-45°/+45°/0°. (b): Type B, 90°/+45°/-45°/0°/90°/0°/-45°/+45°/90°.

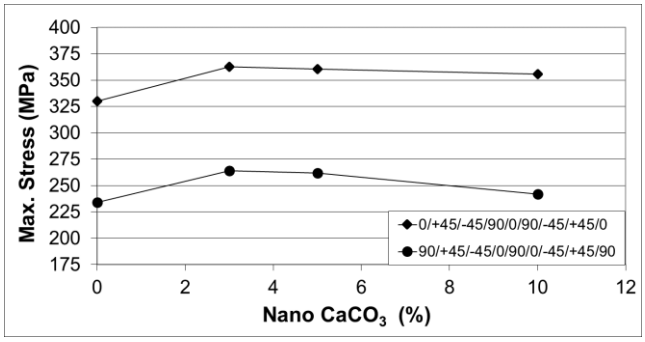
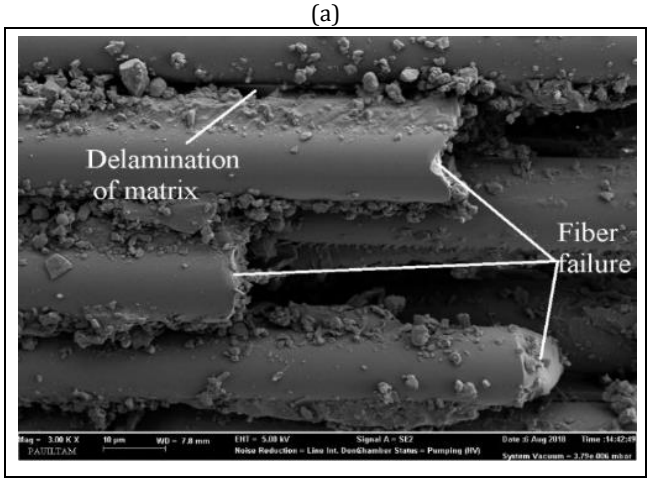


Figure 7. Effect of $CaCO_3$ contribution by weight on the tensile strength.

The morphology of the damaged samples was viewed by Scanning Electron Microscope (SEM). The delamination of matrix appeared in the absence of fibers and in the absence of nanoparticles and are shown in the figures. In the fracture zones, damage has occurred due to fiber breakdown and interface separation. A small amount of resin adhering to the fibers that separated from the matrix element due to poor adhesion was observed in the fracture zones. The development of the microstructure is directly related to the interaction of epoxy resin and nanoparticles. Optimum amount of nanoparticles well mixtured with the matrix provides better adhesion to the fiber. Excessive use of the nanoparticle showed the clumping and caused such as notch and hole effect on the matrix. SEM images of pure glass fiber composite materials that do not have any inclusion of additives are shown in Figure 8. Since there is no nanoparticle that binds the matrix element and the fibers in the composite materials, gaps adversely affect tensile strength are formed between the fibers. The fibers are collected in the form of a ball of yarn, causing material to reduce of strength.

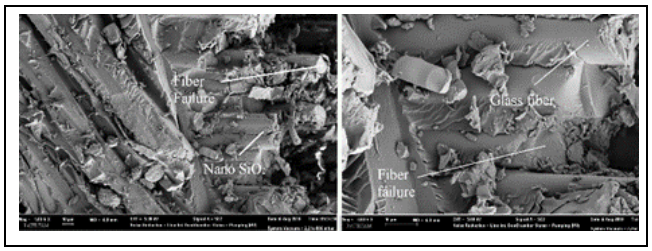
(a)



(b)

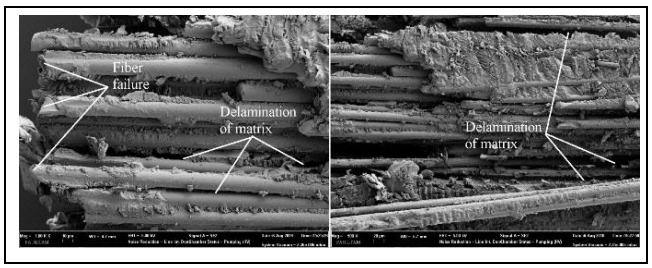
Figure 8. SEM images of pure glass fiber composite material. (a): 100x, (b): 3000x.

The appropriate addition of nano SiO_2 and $CaCO_3$ materials into the polymer matrix showed a positive contribution on sandwich composite structure. Figure 9 shows the SEM image of the 1% SiO_2 inclusion produces the highest strength value with low numbers of delamination of matrix. Excessive amount of SiO_2 content affected the adhesion quality of the epoxy led to an increase of number of delamination and decrease in the strength, shown in Figure 10. The adhesion strength between the matrix and the fibers was increased with the addition of 3% nano $CaCO_3$ additive. It was observed that the adhesion quality between the fiber and the matrix was increased and the fibers maintained the linearity, shown in Figure 11. When the optimum $CaCO_3$ nano addition was exceeded, it was seen that the number of delamination and deterioration in matrix were increased in many regions. The inclusion of excessive amounts of nano $CaCO_3$ material caused a clumping and gap in the matrix and resulted in a decrease in the strength of the composite materials, Figure 12.



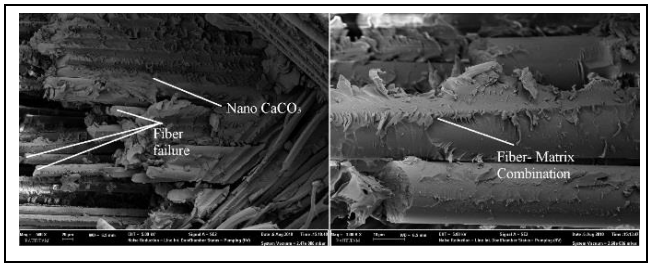
(a) (b)

Figure 9. SEM images at the fracture of samples damaged with the use of 1% SiO_2. (a): 1000x, (b): 3000x.



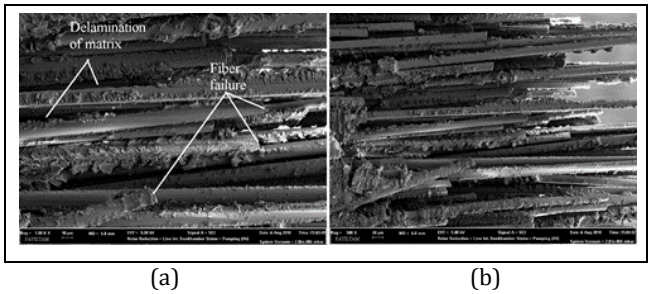
(a) (b)

Figure 10. SEM images at the fracture of samples damaged with the use of 5% SiO_2, (a): 1000x, (b): 500x.



(a) (b)

Figure 11. SEM images at the fracture of samples damaged with the use of 3% $CaCO_3$. (a): 500x, (b): 3000x.



(a) (b)

Figure 12. SEM images at the fracture of samples damaged with the use of 10% $CaCO_3$. (a): 1000x, (b): 500x.

4 Conclusions

The sufficient inclusion of nano SiO_2 and $CaCO_3$ materials into the polymer matrix showed a positive contribution on sandwich composite structure and improved the strength of the composite materials. Well mixture of nanoparticles and epoxy resin has increased the mechanical properties and adhesion quality of the matrix. Optimum amount of SiO_2 and $CaCO_3$ nanoparticle inclusion were determined as 1% and 3% by weight of resin, respectively. The use of SiO_2 nanoparticles increased the strength of composites by 8% for Type A and 10% for Type B by applying 1% by weight. The inclusion of $CaCO_3$ nanoparticles increased the strength by 10% for Type A and 13% for Type B when applied at a rate of 3%.

As indicated in the SEM images, when the nanoparticle additive exceeds the optimum amount, it has been concluded that the number of delamination and deterioration in matrix were increased in many regions. Therefore, clumping and gap in the matrix resulted in a decrease in the strength of the composite materials.

In this study, besides the nanoparticle contribution, the effects of fiber orientation on the strength were also investigated and the increase in the amount of fiber in the direction parallel to the drawing axis indirectly increases the strength. It has been observed that the use of nano-additive materials is important in case of a decrease in the number of unidirectional fibers. When the number of fibers decreased, it was found that the matrix had to be reinforced by adding nanoparticles.

5 Author contribution statements

Can TUNCER took part in the study's experimental study process and writing phase and provided control and supervision. Olcay Ersel CANYURT took part in the literature research, construction, and writing of the experiments. This study was supported by the Unit of Scientific Research Projects (USRP) of Pamukkale University. Project number is 2017FEBE065. The authors would express their thanks to Pamukkale University for their kind supports.

6 Ethics committee approval and conflict of interest statement

There is no need to obtain permission from the ethics committee for the article prepared.

There is no conflict of interest with any person / institution in the article prepared.

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