

# The influence of nano-silica on the wear and mechanical performance of vinyl-ester/glass fiber nanocomposites

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**Abstract.** In the present article, silica nanoparticles (SNPs) were exploited to improve the tribological and mechanical properties of vinyl ester/glass fiber composites. To the best of our knowledge, there hasn't been any prior study on the wear properties of glass fiber reinforced vinyl ester SiO<sub>2</sub> nanocomposites. The wear resistance is a critical concern in many industries which needs to be managed effectively to reduce high costs. To examine the influence of SNPs on the mechanical properties, seven different weight percentages of vinyl ester/nano-silica composites were initially fabricated. Afterward, based on the tensile testing results of the silica nanocomposites, four wt% of SNPs were selected to fabricate a ternary composite composed of vinyl ester/glass fiber/nano-silica using vacuum-assisted resin transfer molding. At the next stage, the tensile, three-point flexural, Charpy impact, and pin-on-disk wear tests were performed on the ternary composites. The fractured surfaces were analyzed by scanning electron microscopy (SEM) images after conducting previous tests. The most important and interesting result of this study was the development of a nanocomposite that exhibited a 52.2% decrease in the mean coefficient of friction (COF) by augmenting the SNPs, which is beneficial for the fabrication/repair of composite/steel energy pipelines as well as hydraulic and pneumatic pipe systems conveying abrasive materials. Moreover, the weight loss due to wearing the ternary composite containing one wt% of SNPs was significantly reduced by 70%. Such enhanced property of the fabricated nanocomposite may also be an important design factor for marine structures, bridges, and transportation of wind turbine blades.

**Keywords:** coefficient of friction; energy applications; mechanical properties; SiO<sub>2</sub> vinyl ester nanocomposites; wear resistance; wind turbine blades

## 1. Introduction

The extensive applications of polymer composites in various industries highlight the necessity of developing polymer nanocomposites with multi-functional characteristics. Toward this end, considerable efforts have been devoted to elaborating such nanocomposites with improved mechanical, chemical, magnetic, and electrical properties. One of the effective methods to improve the properties of two-component composites is employing nanoparticles. This method has attracted the attention of many researchers recently. In order to improve the properties of polymer composites adding silica nanoparticles, carbon nanotubes, graphene and graphene-oxide nanoparticle are very common (Ebrahimi *et al.* 2019a, b, Safaei *et al.* 2019, Fattahi *et al.* 2021). Polymeric pipes are widely used in the petroleum and aerospace industries due to their excellent strength, corrosion and wear resistance (Rahmani *et al.* 2021a). On the other hand, the pipelines used to transmit water, oil, and gas or convey abrasive materials are usually made of steel and composite materials. They are susceptible

to wear and material damage (Nookala *et al.* 2021). In other words, the tribological properties like other mechanical properties should be considered as design parameters for above structures made of composite materials or for the case of advanced composites adopted for repair purposes. For instance, the type of observed wear is similar to abrasion at a low angle of impingement. All different forms of wear share some common features. The degradation of material over the service life is affected by various effects, including friction and corrosion. Thus, the current research is directed toward studying wear and mechanical properties in glass fiber/vinyl ester composites reinforced by SNPs.

In recent years, many studies have been conducted to investigate the effect of silica nanoparticles on the mechanical properties of neat thermoset resins. Among these works, we can refer to Chen *et al.* (2008) who investigated the mechanical properties of silica nanocomposite by adding silica nanoparticles to epoxy resin. They reported an improvement in the toughness and modulus of the nanocomposite. Hsieh *et al.* (2010) also reported that the toughness and Young's modulus of silica nanocomposites increased by increasing the nanoparticle content. Liu *et al.* (2011) determined improved mechanical properties by adding silica nanoparticles to the thermoset resin. Also, they evaluated the fracture mechanisms of silica nanocomposite using the images of scanning electron

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microscopy. He *et al.* (2016) examined the effect of silica nanoparticles on the thermoset epoxy resin. One of the most critical points in this study was to improve the mechanical properties of silica nanocomposite relative to the epoxy resin by increasing the volume fraction of nanoparticles. Constantinescu *et al.* (2018) investigated the effects of three different mass fractions of nanoparticles in silica/epoxy nanocomposites. They dispersed the silica nanoparticles directly inside the resin material, and the obtained results indicated no significant change in the mechanical properties of the nanocomposite. Asif *et al.* (2019) added silica nanoparticles to polymer and examined the mechanical properties of the resulted composition. They also reported an improvement in tensile properties by adding silica nanoparticles.

The wear properties of thermoset resins filled with SNPs have also been examined by several researchers. Antunes *et al.* (2014) studied the mechanical and wear properties of silica nanocomposites. Palraj *et al.* (2015) evaluated the wear resistance and corrosion of silica nanocomposite as a coating. This increase was achieved as long as no agglomeration of the silica nanoparticles was observed. Abenojar *et al.* (2017) studied the mechanical, thermal, and wear resistance properties of the epoxy nanocomposites reinforced by SNPs. They found a significant increase in the wear resistance happens for silica nanocomposite containing 3 wt% of nanoparticles. However, there are no exact quantities in the study that correspond to the percentage of wear resistance improvement. Based on the graphs, it would be found that the weight loss under the wear pin-on-disk test was reduced by about 34%. Imani *et al.* (2018) studied the wear resistance and COF of silica nanocomposites. According to this study, some inorganic particles can enhance the bonding between the resin and metallic counterpart which leads to improved wear resistance. However, some fillers can generate more discontinuities in the same material that result in opposite effects on the wear performance.

Meanwhile, studying the influence of the surface modification of silica nanoparticles and the agglomeration phenomenon on the mechanical properties of the resulted nanocomposite has been the topic of a number of papers. In fact, the proper distribution of nanoparticles through the polymer matrix may contribute to a further improvement of the mechanical properties. The silica nanoparticles are hydrophilic due to the presence of hydroxyl groups on the surface, so they need surface modification because the absorption of water by the polymer is a critical parameter. Different chemical methods can modify the surface of silica nanoparticles. Agents such as silane, and amino-silane increase the hydrophobicity of the silica nanoparticles and also allow better distribution of nanoparticles through the resin matrix (Yu *et al.* 2019, Karnati *et al.* 2020, Xiao *et al.* 2020). The surface modification of nanoparticles using silane modifiers can be used as an effective method for the prevention of nanoparticles agglomeration. Therefore, more appropriate distribution of nanoparticles in the resin matrix and also, preventing agglomeration of nanoparticles increases and improves the mechanical properties of nanocomposites (Ahangan and Navarchian 2020, Das *et al.* 2020, Yang *et al.* 2022). Furthermore, Agglomeration

phenomena have been considered by researchers due to their importance in the fabrication of nanocomposites. Various methods have been proposed to prevent this phenomenon, such as modifying the nanoparticle surface, increasing the nanoparticle distribution process time, and combining nanoparticle distribution processes using the mechanical mixer and ultrasonic probes (Megahed *et al.* 2018, Zhang *et al.* 2019, Ghahramani *et al.* 2022). Indeed, the agglomeration behaves like a defect. The stress concentration occurs in the region where the agglomeration exists, so that the mechanical properties of the nanocomposite are reduced. That is why some theoretical and experimental studies have been conducted to determine the effects of these phenomena (Moradi-Dastjerdi *et al.* 2017, Karnati *et al.* 2020, Moradi-Dastjerdi and Behdinin 2021a, b).

Moreover, some studies have dealt with the influence of adding silica nanoparticles on the mechanical and wear properties of fiber polymer composites. Such advanced materials are known as three-component composites that have many promising applications in different industries. In this regard, Uddin and Sun (2008) evaluated the mechanical properties of a unidirectional glass/epoxy composite containing the silica nanoparticle. They fabricated the three-component composites by using the hand layup method. In another study, Mahrholz *et al.* (2009) improved the stiffness, strength, and toughness of a three-component composite by adding the silica nanoparticles to glass fiber composites using liquid composite molding. Also, Tsai *et al.* (2010) conducted research work on glass fiber three-component composites filled with silica nanoparticles. They fabricated glass fiber/silica/epoxy composite by vacuum hand layup process. Manjunatha *et al.* (2010) studied the effect of silica nanoparticles on the tensile fatigue of glass fiber composites. In this study, they could enhance the tensile fatigue life of glass fiber composite by about three times after adding silica nanoparticles. They also manufactured nanoparticle-modified epoxy/glass fiber reinforced plastic composite laminates using the resin infusion. Tang *et al.* (2012) studied the fracture mechanism in a three-component composite containing silica nanoparticles. They reported an increase in the elastic modulus of the three-component composite. Sprenger (2015) reviewed recent studies on the improved mechanical properties of fiber composites modified by silica nanoparticles. Feli and Jalilian (2016) firstly examined the mechanical properties of silica nanocomposite. Then, they could improve the mechanical properties by adding the silica nanoparticles to the glass fiber composite. They used a hand layup technique to produce a three-component composite. Tian *et al.* (2017) investigated the interfacial properties of composites containing silica nanoparticles. They reported that the interfacial properties increased effectively by adding the silica nanoparticles. Islam *et al.* (2021) added the silica nanoparticles to a two-component composite. They used the SNPs to avoid micro-crack growth at very low temperatures. Alsaadi *et al.* (2018) investigated the effect of silica nanoparticles on tensile, flexural, and vibrational properties of carbon/Kevlar-reinforced epoxy composite. Shaker *et al.* (2019) studied the tensile, flexural, and impact properties of a three-

component composite produced by adding SNPs to vinyl ester/glass composite. They claimed that adding the silica nanoparticles to the composite causes an increase in the adhesion between the matrix and fibers. Rajaei *et al.* (2019) examined the effects of adding styrene-butadiene rubber and silica nanoparticles on the mechanical properties of a three-component composite made of glass fibers. They made a three-component polyester/silica/chopped glass fiber composite by using a bulk molding compound process. Su *et al.* (2020) examined the improvement of the mechanical properties of epoxy/fiber and vinyl ester/fiber composites by adding SNPs. One of their significant results was the increase in the toughness of the three-component composites. In other research, the enhanced mechanical properties of three-component composites by adding nanoparticles to glass fibers were reported (Setoodeh and Sokhandani 2017, Zhou *et al.* 2017).

Concerning the wear resistance properties of three-component composites reinforced by different nanoparticles, Osterle *et al.* (2016) examined the wear properties and COF of carbon fiber composites containing silica nanoparticles. Rahmani *et al.* (2021b) examined the effect of different fibers on the hardness and wear behavior of composites. In this study, the wear test results showed that the wear rate of the carbon fiber-reinforced composite is 26% less than the glass fiber-reinforced composite. Su and Zhang (2010) investigated the influence of adding 4 wt% of silica nanoparticles to glass/polytetrafluoroethylene composite to examine the wear resistance. They reduced the COF of the wear by an amount of 19% measured by the pin-on-disk test.

Researchers have rarely studied the wear properties of ternary vinyl ester composites by adding other nanoparticles, including TiO<sub>2</sub>, ZnO, and silicon carbide, to glass fiber and basalt fiber/vinyl ester composites. Singh *et al.* (2018) found that the presence of hard particles (TiO<sub>2</sub> and ZnO) in the matrix helps in absorbing a good fraction of the kinetic energy. Therefore, the energy available for the plastic deformation of the thermosetting vinyl ester was significantly declined. This in turn, delayed the initiation of the fiber exposure in comparison with the composite without any filler reinforcement. In another study, a three-component composite (basalt mat/vinyl ester matrix reinforced by a maximum of 3 wt% of silicon carbide nanoparticles) exhibited better pull-out resistance because of the fiber bonding characteristics which may overcome the agglomeration of nanomaterial in the fiber bundles (Thooyavan *et al.* 2022).

Vinyl ester resin as a thermoset polymer incorporates both advantages of polyester and epoxy and exhibits good mechanical and anti-corrosive properties. However, despite the widespread and increasing use of glass fiber reinforced vinyl ester composites, to the best of our knowledge, no study so far has investigated the influence of nano-silica on the wear resistance properties of such composites fabricated by either the hand layup or vacuum-assisted resin transfer molding (VARTM). Hence, this paper mainly aimed to comprehensively examine the improved mechanical properties and, in particular, wear resistance properties of a ternary composite made of vinyl ester/glass fiber/nano-silica in comparison with the silica binary composite.

Consequently, the mechanical properties such as the ultimate strength, Young's modulus, and toughness of silica nanocomposites were experimentally determined thoroughly. Then, the tested specimens were analyzed using SEM images by focusing on the role of the silica nanoparticles dispersed into the vinyl ester resin. The best results were used to fabricate the vinyl ester/glass fiber/nano-silica composites. Eventually, the three-component composite was subjected to the tensile, flexural, impact, and wear tests. The obtained results indicated improved mechanical and wear properties, which were more significant in the wear testing of the manufactured composite. Also, the SEM images appropriately interpreted the results obtained from the experimental tests and thus, successfully verified the proper functioning of the nanoparticles.

## 2. Experimental procedure

### 2.1 Materials

In the current research, silica nanoparticles (SiO<sub>2</sub>) of the trade name, Aeorsil RA 200 produced by Evonik Aerosil® (Germany), with an average particle size of about 12 nm was used as a reinforcement. Also, the vinyl ester resin was purchased from FARAPOL® (Iran) and used as the matrix material. The viscosity of vinyl ester resin at 25°C is 400-450 cP. Curing this resin was conducted for two hours at 80°C and one hour at 120°C. 3D Glass fiber mat was purchased from METYX® (Turkey). The approximate diameter of the fibers is 15 micrometers.

### 2.2 Fabrication method

Vinyl ester exhibits good resistance to corrosive materials, water and gases. Thus, it has been largely employed in pipeline systems, marine industry, and chemical processing equipment. Moreover, vinyl ester and glass fibers are widely used in the manufacture of GRP pipes as well as wind turbine blades. In addition, silica nanoparticles are economically justified for use in various industries due to their low price. Another advantage of SNPs is their hardness that makes them an ideal candidate for wear resistance application. On the other hand, the shape of these particles leads to increased sphericity, which has a significant effect on reducing the contact surface between two nanoparticles. This effect in turn, can contribute to weak the van der Waals force between the nanoparticles and allowing better distribution of nanoparticles through the polymer matrix. In this study, seven different weight percentages of silica nanoparticles (0, 0.25, 0.50, 0.75, 1.00, 1.50, 2.00) were dispersed in the vinyl ester matrix. Dispersing silica nanoparticles in the vinyl ester resin was accomplished by a mechanical mixer and an ultrasonic probe device. First, nanoparticles were placed inside the ultrasonic device with a solvent (Acetone), and vinyl ester resin was added to the solution after dispersing nanoparticles in the solvent. Then, the mechanical mixer was used to mix the solution for 30 minutes with a rotational speed of 1000 rpm. The shear force resulting from the mixer's blade rotation leads to a uniform

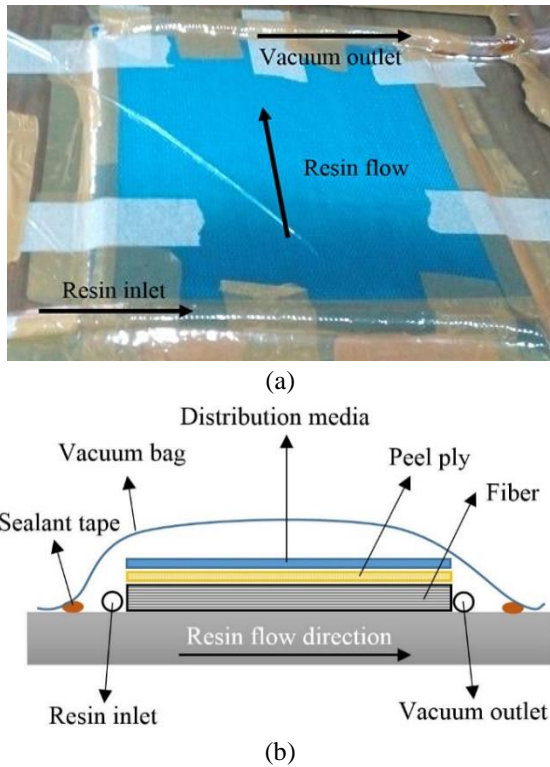


Fig. 1 VARTM process

Table 1 The glass fiber and void volume fractions in the ternary composite

Samples	Nano-silica (wt%)	Glass fiber volume fraction (vol%)	Void volume fractions (vol%)
vinyl ester/glass fiber	0	51.9± 1.2	1.11 ± 0.02
vinyl ester/glass fiber/nano-silica	0.25	51.2 ± 1.4	1.30 ± 0.03
vinyl ester/glass fiber/nano-silica	0.50	52.3 ± 1.1	1.12 ± 0.02
vinyl ester/glass fiber/nano-silica	1	51.5 ± 1.4	0.94 ± 0.03

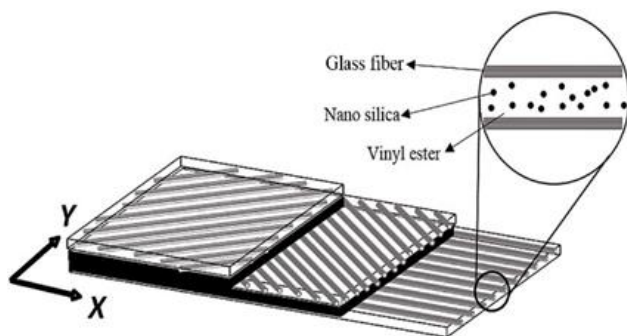


Fig. 2 Glass fiber orientation and schematic of vinyl ester/glass fiber/nano-silica composite

distribution of silica nanoparticles in the polymer matrix. In order to increase the nanocomposite uniformity, the vinyl ester resin with nanoparticles was then sonicated inside the ultrasonic device. After completing the dispersion steps, the resin was placed in an oven for five hours at 60 °C to remove the solvent material. Finally, the hardener material

was added to the resin, and eventually, bubbles were eliminated in the resin containing nanoparticles by creating a vacuum. This fabrication process caused the defects to be reduced in the final nanocomposite. Also, an appropriate distribution of nanoparticles was obtained, and afterward, the nanocomposite specimens were molded using prefabricated molds. The tensile tests were performed on the nanocomposite specimens in the next step.

The maximum strength (ultimate tensile strength) of silica/vinyl ester nanocomposites was determined by tensile strength testing. According to the silica/vinyl ester nanocomposites tensile test, four different weight percentages of silica nanoparticles with the maximum increase in strength were selected in order to add to the vinyl ester/glass fiber composite. The weight percentages of silica were equal to 0, 0.25, 0.50, 1.00. The method used for fabricating these three-component composite materials was VARTM. In the VARTM fabrication method, the mold is closed by a vacuum bag and sealant tape to avoid air leakages and the formation of air bubbles. The glass fiber mats were firstly cut and put inside the mold. Then, peel ply and distribution media were cut and placed on the glass fiber mats. The vacuum outlet and resin inlet were fixed and then the prepared vacuum bag was set over the laminate. The air was removed from the mold by using the compressor vacuum pump. It should be noted that any air leakage would cause defects in the final composite. The vinyl ester resin containing silica nanoparticles was prepared according to the procedure explained before. Eventually, the resin was injected into the mold by applying a vacuum. The duration of this process depends on the viscosity of the resin, the diameter of the inlet and outlet pipes, the density of the fibers as well as the vacuum pressure. The vacuum pressure applied to inject the resin into the mold in the VARTM process was -0.85 bar. Fig. 1 shows the VARTM process. This method reduces the composite defects and improves the mechanical properties by decreasing the number of bubbles formed in the final composite. The volume fraction of glass fiber and void in the ternary composite were measured according to the ASTM D792-13, ASTM D3171-15 and ASTM D2734-16. A pyrolysis treatment of 3 h at 600°C was used to fully degrade the vinyl ester matrix (Tüzemen *et al.* 2017, Fang *et al.* 2021). In this procedure, five replicates from each specimen were taken to get the mean values. Table 1 presents the volume fraction of glass fiber and void in the vinyl ester/glass fiber/nano-silica composite. According to the results, the volume fraction of void in all samples is less than 1.3%. Also, increasing the weight percentage of silica nanoparticles had no effect on the void volume fractions. The main reason for the low amount of void volume fractions is due to performing degas process in a vacuum chamber, before injecting the vinyl ester resin. 3D glass fibers were used for fabricating composite specimens, which were manufactured with seven layers. The fiber mass fraction for the glass fibers was found to be 0.68±0.02. Fig. 2 demonstrates the orientation of glass fibers and a schematic of vinyl ester/glass fiber/silica nanocomposite. After fabricating the three-component composite, specimens were prepared for tensile, flexural, impact, and wear testing according to related standards.



(a) Closed mold of VARTM used in this study



(b) Schematic of VARTM

Fig. 3 Wear testing machine and specimen under test

### 2.3 Tensile test

In order to specify the ultimate strength and elastic modulus of vinyl ester resin and silica/vinyl ester nanocomposites, the tensile test should be used according to polymer-related standards. The standard considered in this study for tensile testing of polymers is ASTM D638 (part 4). The dimensions of the polymer specimen for the tensile test were 115 mm in length and 6 mm in width. The thickness of the polymer and nanocomposite specimens was 3.2 mm and the load rating of the test was 5 mm/min. All the tensile and bending tests were conducted using the STM-50 tensile testing machine produced by SANTAM. Each test was performed five times for each specimen and the average values were reported.

In order to determine the ultimate strength of the two-component and three-component glass fiber composites, the tensile test was conducted on specimens. According to the utilized standard ASTM D3039 for composites with multi-directional fibers, the specimen dimensions were 250 mm in length and 25 mm in width. The speed of testing was 50 mm/min. This test was also carried out five times for each sample.

### 2.4 Three-point flexural test

The three-point flexural test was carried out on two-component and three-component composites. The standard applied in this study was ISO 178. According to this standard, specimens have a length of 80 mm and a width of 10 mm.

### 2.5 Impact test

The Charpy impact test specifies the amount of energy absorbed for breaking down a specimen. In-plane or out-of-plane impacts can be applied to the specimen. The impact test was conducted obeying the standard ISO 179. The specimens were cut with dimensions of 80 mm by 10 mm. The SIT-20E device produced by SANTAM was utilized for impact testing. To accurately perform this test, the amount of energy dissipated due to the air resistance was first measured. Then, this amount of energy was subtracted from the final value to obtain more accurate results. Finally, the obtained energy was divided by area to determine the normalized value of absorbed energy.

### 2.6 Wear test

The pin-on-disk wear test was applied to obtain the wear properties and surface resistance of the specimens. After conducting the wear test, the COF of materials was reported as an important design factor. The standard applied for wear testing is ASTM G99. Dimensions of specimens prepared from composites for wear testing is a square with sides of 20 mm. Then, all specimens were weighted and installed on the specific support while a pin was placed above them. The test was conducted by applying a 10 N force with a velocity of 0.05 m/s. After traversing 200 m distance, the test specimen was removed from the testing machine and weighted again. The weight difference before and after the test was calculated and presented as a variable. Fig. 3 shows the wear testing machine, which is WTC02 produced by Tajhiz Sanaat Nasr Co.

### 2.7 Microscopic evaluation

To clarify the role of both nano-silica particles and glass fiber on the deformation and fracture mechanism of vinyl ester polymer, the fracture surfaces of some samples were observed using a scanning electron microscope (TESCAN-Vega 3). Also, to avoid charge building up, the surface of all samples was coated with gold using Q150R-ES produced by Quorum Technology before testing.

## 3. Solution procedure

In this section, the results obtained from the test were examined. Also, all results were analyzed and compared using diagrams and SEM images. It is worth mentioning that the mechanical properties of the ternary composites were compared with the corresponding properties of the binary glass fiber reinforced vinyl ester composites, wherein the weight fraction of the nano-silica is identical to zero.

### 3.1 Tensile testing of silica/vinyl ester nanocomposite

First, the results obtained from tensile testing of silica/vinyl ester nanocomposite were evaluated in this study. The obtained results from these tests present the ultimate tensile

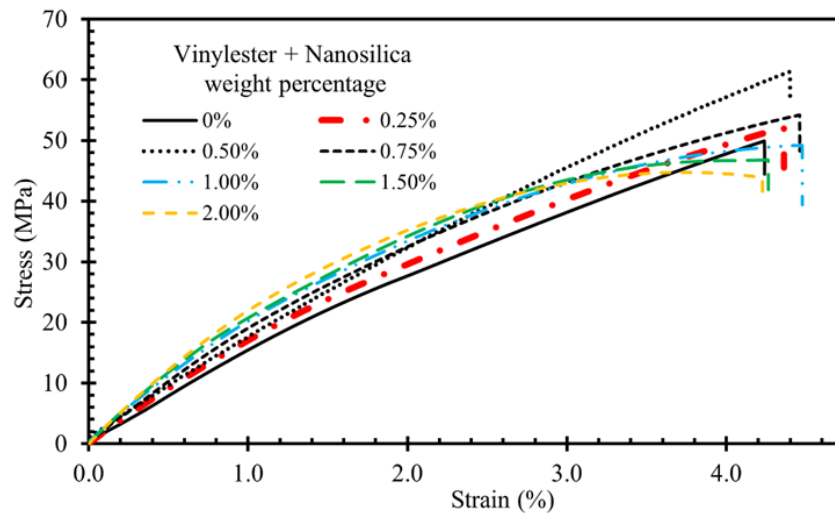


Fig. 4 Stress-strain curve of nano-silica/vinyl ester nanocomposite with different weight percentages

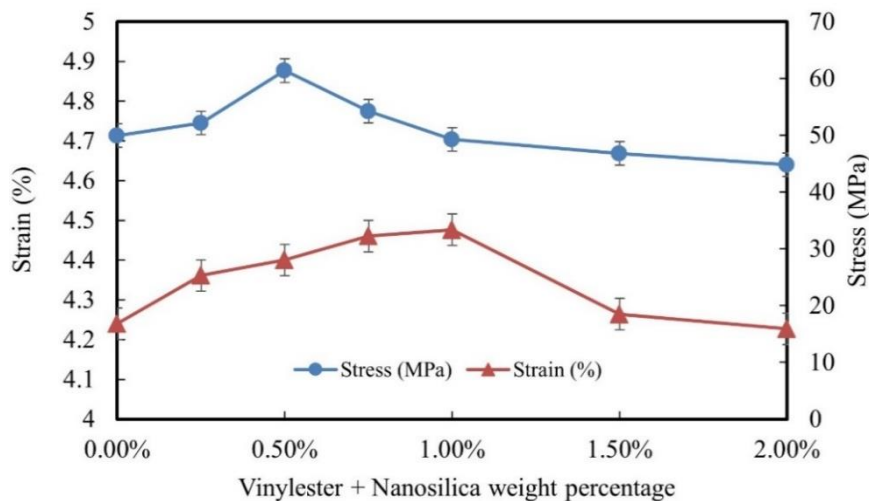


Fig. 5 Failure stress-failure strain curve of nano-silica/ vinyl ester nanocomposite with different weight percentages

strength, maximum strain, and toughness of the material. Also, the fracture mechanism was evaluated using SEM images. Fig. 4 indicates the stress-strain curve for seven different weight percentages of the nanocomposite.

According to the stress-strain curve resulting from the tensile test, it can be observed that adding 0.50 wt% nano-silica to vinyl ester resin increases the ultimate tensile stress by 21.8%. Also, the strain increases by 3.7%. In ordinary materials, the strain usually decreases by increasing the strength. However, adding nanoparticles to the resin of the nanocomposite increases the strain and strength. To better explain the results, the maximum stress-strain curve was plotted for each weight percentage of nanoparticles distributed in the nanocomposites, as shown in Fig. 5. It can be observed that the maximum strain obtained for the nanocomposite with one wt% of nano-silica. The percentage level of strain increment relative to the raw resin is 5.6%. Also, it is observed that the stress and strain of nanocomposite decreased by increasing the weight percentage of the silica nanoparticles to a particular value of one wt%.

This is due to the agglomerate phenomenon. Increasing

the weight percentage of silica nanoparticles increases the number of these particles in the vinyl ester resin to the extent that the van der Waals force between these nanoparticles prevents more dispersion. The nanoparticles are absorbed by each other due to the strong van der Waals force, and the agglomerate phenomenon occurs. Since the agglomerate phenomenon emerges like a defect in the nanocomposite, the ultimate strength and strain decrease by increasing the weight percentage of the nanocomposite. Then, the area below the stress-strain curve was calculated for each nanocomposite. It is worth considering that this quantity is representative of toughness.

According to Fig. 6, the maximum level of toughness was obtained for a nanocomposite with 0.5 wt% nano-silica. The variations of the results show that the elastic modulus of the nanocomposite increases by increasing the weight percentage of the silica nanoparticles. It can also be seen that the toughness decreases in a range of 9.5% after experiencing a maximum value. This variation can be attributed to the agglomerate phenomenon. The different tensile behavior of the binary composite in comparison to the ternary one can be observed from the SEM images of

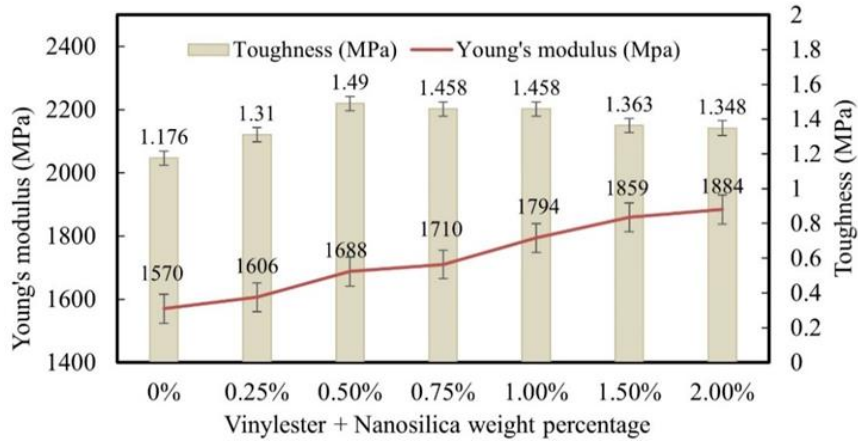
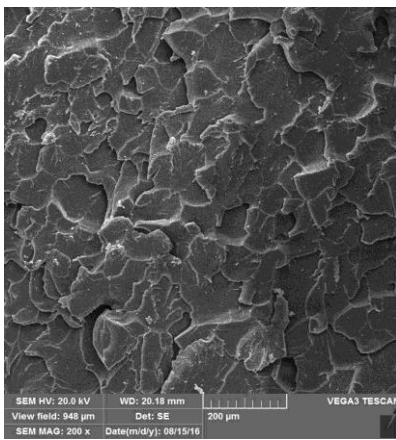
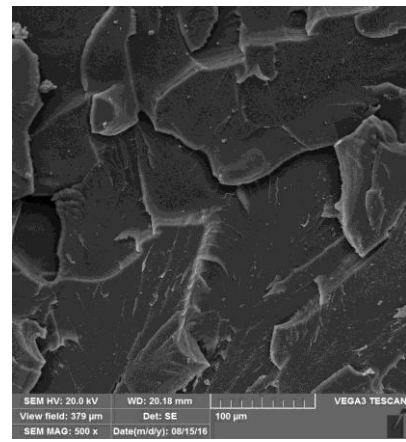


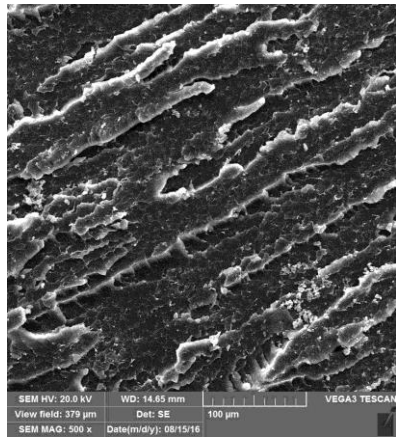
Fig. 6 Toughness and Young's modulus of silica/vinyl ester nanocomposite with different weight percentages



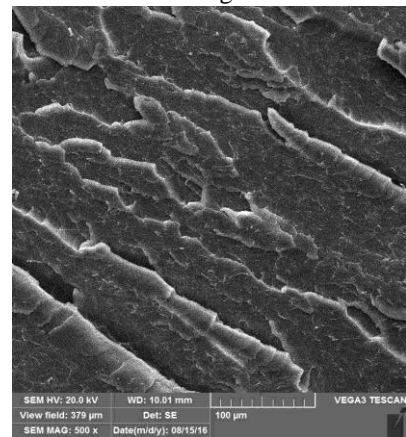
(a) Fracture surface of the vinyl ester resin



(b) Fracture surface of the vinyl ester resin with a different magnification factor



(c) Fracture surface of the nanocomposite with 0.5 wt% nano-silica



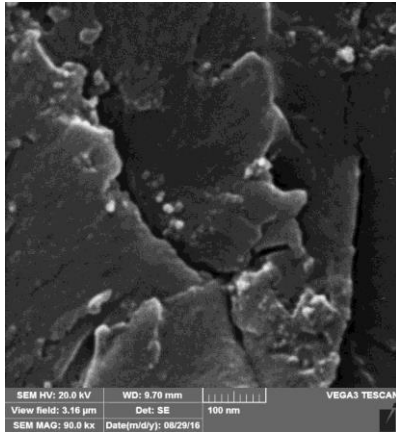
(d) Fracture surface of the nanocomposite with 1.5 wt% nano-silica.

Fig. 7 Fracture surface SEM micrographs

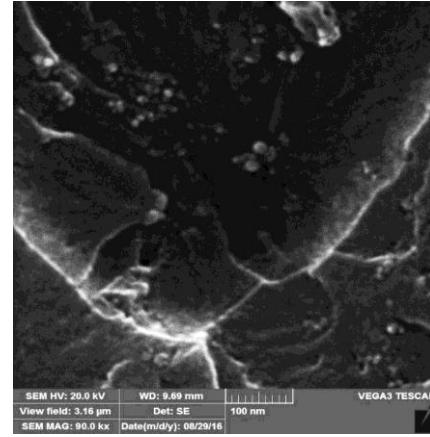
Fig. 7. It can be seen that the magnitude of crack length and the changes in the crack orientation are more significant in the nanocomposite relative to the vinyl ester resin. On the other hand, the encounter of the crack propagation path with the nanoparticles barrier was changed the interlaminar and intralaminar crack propagation paths, which clearly shown in Fig. 7 (Fang *et al.* 2020). So that, this mechanism resulted in an increase in the energy required for the crack

growth through the structure. Thus, it is rational for the toughness to be increased as well. The depicted SEM images can better express the behavior of the specimens.

Based on SEM images, it was found that the variations in the crack orientation for the nanocomposite with a 0.5 wt% nano-silica were much more compared with the raw resin as well as the nanocomposites with other weight percentages of nano-filler. This means that a more

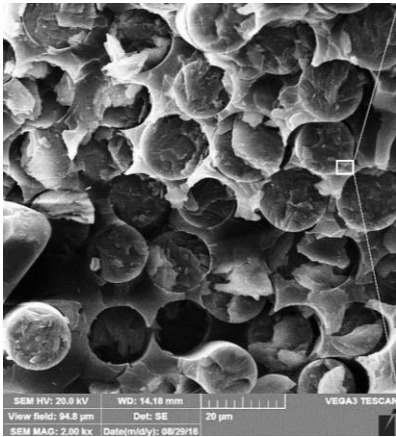


(a) Silica nanoparticles in the nanocomposite containing 0.5 wt%

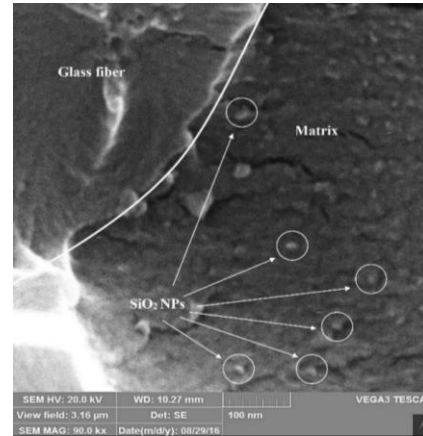


(b) Silica nanoparticles in the composite with 1.5 wt% nano-silica.

Fig. 8 SEM images of silica nanoparticles in the nanocomposite



(a) SEM image of vinyl ester/glass fiber/nano-silica composite with 0.5 wt% of nano-silica



(b) SEM image of vinyl ester/glass fiber/nano-silica composite containing 0.5 wt% of nano-silica with a different magnification factor

Fig. 9 SEM image of vinyl ester/glass fiber/nano-silica composite containing 0.5 wt% of SNPs

significant amount of energy is needed for an increase in the crack length and consequently, an enhancement in the strength and toughness is obtained. Also, the agglomerate phenomenon can be identified by increasing magnification, as shown in the SEM images in Fig. 8.

According to the SEM images, it is evident that increasing the weight percentage of silica nanoparticles leads to an increase in their tendency to create agglomerates. The silica nanocomposite with 0.5 wt% nano-silica shows the maximum tensile strength. This increase is recognized from SEM images wherein the nanoparticle dispersion in the vinyl ester resin is performed in an excellent manner.

According to the observations, agglomerations firstly reduce the ultimate tensile strength of the nanocomposites because the load applied to the nanocomposite is maximized when a failure occurs. Although, the agglomeration has a considerable effect on the stiffness of the composite, however, as Young's modulus of the nanocomposites was calculated in a region with a lower load (Range 0 to 1% strain in the stress-strain diagram), then, the agglomeration

rate exhibits a minor effect on the measured property. Moreover, good distribution of SNPs through the resin by combing two mixing processes leads to minimizing this effect. It is also seen that the growth rate of Young's modulus decreases with the increasing weight percentage of the nanoparticles due to the substantial increase in the agglomeration degree. This phenomenon was also observed in the research conducted by Yan *et al.* (2019).

### 3.2 Tensile testing of vinyl ester/glass fiber/silica nanocomposite

The silica weight percentage of 0, 0.25, 0.5, and 1.00 were selected according to the results obtained from the tensile testing of vinyl ester/silica nanocomposite. The glass fiber mat used in this research is three-dimensional with fiber orientations of [0/-45/+ 45]. The two-component and three-component composites with seven layers of this mat were made. Also, Fig. 2 schematically depicts a layer of the glass fiber mat. The tensile test was carried out on the resulting composite after injecting the vinyl ester resin

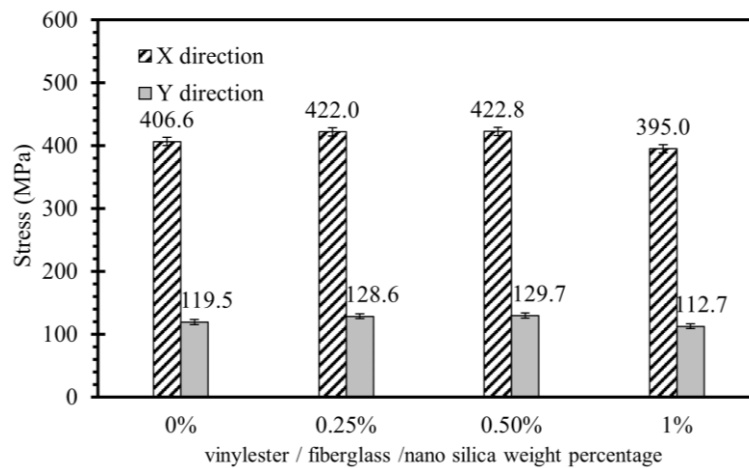


Fig. 10 Ultimate strength of vinyl ester/glass fiber/nano-silica composite in both X and Y directions

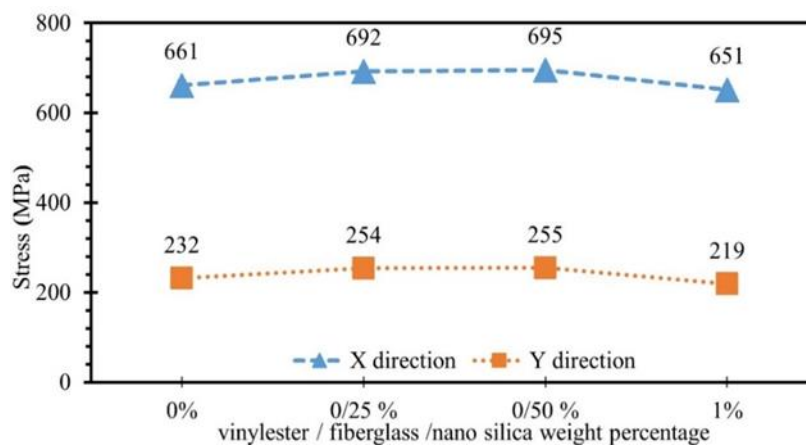


Fig. 11 Flexural stress curve in both X and Y directions for vinyl ester/glass fiber/nano-silica composite

containing nanoparticles into the glass fibers. In order to better recognize the effect of silica nanoparticles, the tensile test was conducted in two perpendicular directions, X and Y (see Fig. 2). Fig. 9 shows the SEM images of silica nanoparticles with a weight percentage of 0.5 distributed through the three-component matrix. According to the images, the nanoparticles were properly dispersed in the polymer matrix, which resulted in substantial enhancement in the mechanical properties of the three-component polymer. According to Fig. 10, it is observed that the three-component composite with a 0.50 wt% nano-silica exhibits the maximum values of the ultimate strength in both directions.

By adding 0.5 wt% nano-silica to the vinyl ester/glass fiber composite, the ultimate strength in the X direction increased by 16.2 MPa. Also, a 10.2 MPa improvement in the strength is observed for the Y direction. In fact, adding the nanoparticles to the matrix leads to a more uniform distribution of stress in the entire composite. Also, the force transmission from the composite matrix to the reinforcement phase (glass fiber) is well performed. Therefore, an increase in the ultimate stress value can be observed in this composite relative to the two-component counterpart. However, the drawback of agglomeration demonstrates its weakening effect when the density of the nano-silica

particles is enhanced from a critical value.

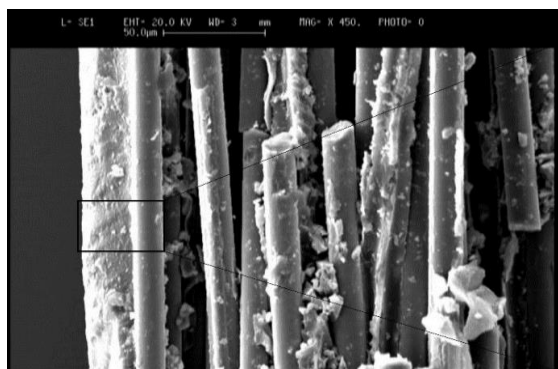
### 3.3 Three-point flexural test on vinyl ester/glass fiber/silica composite

In this section of the study, the bending flexural behavior of the two and three-component composites was examined under bending load. The flexural test was carried out in both X and Y directions. Fig. 11 depicts the results determined by performing three-point bending test for different specimens.

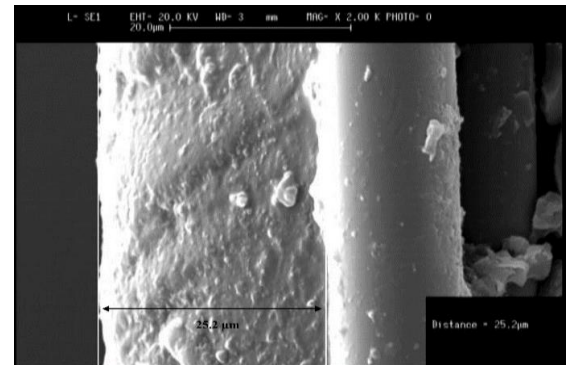
Similar to the tensile test, by examining the results obtained from the flexural test, the maximum ultimate stress tolerated by vinyl ester/glass fiber/nano-silica composite belongs to the composite containing 0.5 wt% nano-silica. The ultimate flexural strengths of the nanocomposite are obtained as 695 MPa in the X direction, and 255 MPa in the Y direction. By increasing the weight percentage of nano-silica in the three-component composite, the composite strength under bending load is reduced. The results illustrate the critical values of the nanoparticles wt% that can be dispersed in the resin. It is seen that the ultimate flexural strength of the three-component composite is decreased in both X and Y directions by adding one wt% of the nano-silica particles.

Table 2 Results of Charpy impact test for vinyl ester/glass fiber/nano-silica composite in both X and Y directions

Nano silica wt%	Impact direction	Area (mm <sup>2</sup> )	Total absorption energy (J)	Normalize energy (kJ/m <sup>2</sup> )
0	X	44.21 ± 1.20	14.14 ± 0.12	319.83
0.25	X	43.90 ± 1.02	14.93 ± 0.14	340.07
0.50	X	43.53 ± 0.80	14.97 ± 0.11	343.89
1.00	X	43.85 ± 1.13	13.91 ± 0.08	317.23
0	Y	44.221 ± 1.23	4.61 ± 0.03	104.27
0.25	Y	43.73 ± 0.94	4.72 ± 0.04	107.93
0.50	Y	43.44 ± 1.12	4.88 ± 0.02	112.33
1.00	Y	43.76 ± 1.41	4.42 ± 0.03	101.01



(a) SEM image of the thickness of the resin on the upper surface



(b) SEM image of the thickness of the resin on the upper surface with a different magnification factor

Fig. 12 SEM image of the thickness of the resin on the upper surface of the vinyl ester/glass fiber/nano-silica composite

### 3.4 Charpy impact testing of vinyl ester/glass fiber/silica composite

The Charpy impact test is applied to indicate the energy absorption level of the material. In this test, a swinging pendulum impacts the specimen to break the sample. Table 2 presents the results obtained from the Charpy impact test for vinyl ester/glass fiber/nano-silica composite in both X and Y directions.

Based on the results obtained from the Charpy impact test, the normalized energy absorbed by the three-component composite containing 0.50 wt% nano-silica was significantly increased in both X and Y directions. The addition of 0.50 wt% nano-silica to the vinyl ester/glass fiber composite yielded 7.5 % and 8 % increase in the normalized absorbed energy relative to the two-component composite in the X and Y directions, respectively. This test was carried out five times for each specimen, and the average value of the results was reported.

### 3.5 Pin-on-disk wear testing of vinyl ester/glass fiber/silica composite

The wear test was conducted by a pin and a disk such that the pin caused the wear of the rotating composite specimen by applying a 10 N force. With the aid of SEM images shown in Fig. 12, the thickness of the resin on the upper surface of the prepared laminates was measured.

According to the images the thickness of the resin matrix was measured as equal to  $25 \pm 1$  micrometer. The distance of 200 m was traversed to accurately measure the wear characteristics and related results. The test was performed at a velocity of 0.05 m/s.

The results obtained from this test were the curve of variations of COF, average friction, and weight loss of the specimens due to wear. First, the COF variation against the distance is presented in Fig. 13. Regarding the COF diagram of the two-component composite, the COF reads the values of 0.2 and 0.50 respectively after traversing 30 m and 50 m. The latter value was not varied significantly up to 200 m distance. It is seen that when the test is finished, the COF achieves a stable condition. Then, the wear test was repeated for the three-component composite containing 0.25 nano-silica.

Based on the above diagram, by adding 0.25 wt% nano-silica to the glass fiber composite, the COF respectively got the values of 0.2 and 0.49 after traversing 35 m and 60 m.

The wear process reached the stable state after traversing a 60 m distance. By increasing the nano-silica content to 0.5 wt%, the measured COF was varied from about 0.2 after 38 m to 0.43 after 56 m. After traversing 56 m, the increasing trend of COF stops, and fluctuates around 0.43 up to the end of the test. The COF reached a value of about 0.2 at a distance of 80 m by adding one wt% nano-silica to the glass fiber composite. Also, after 180 m of sliding distance, the COF trend showed a stable pattern. The

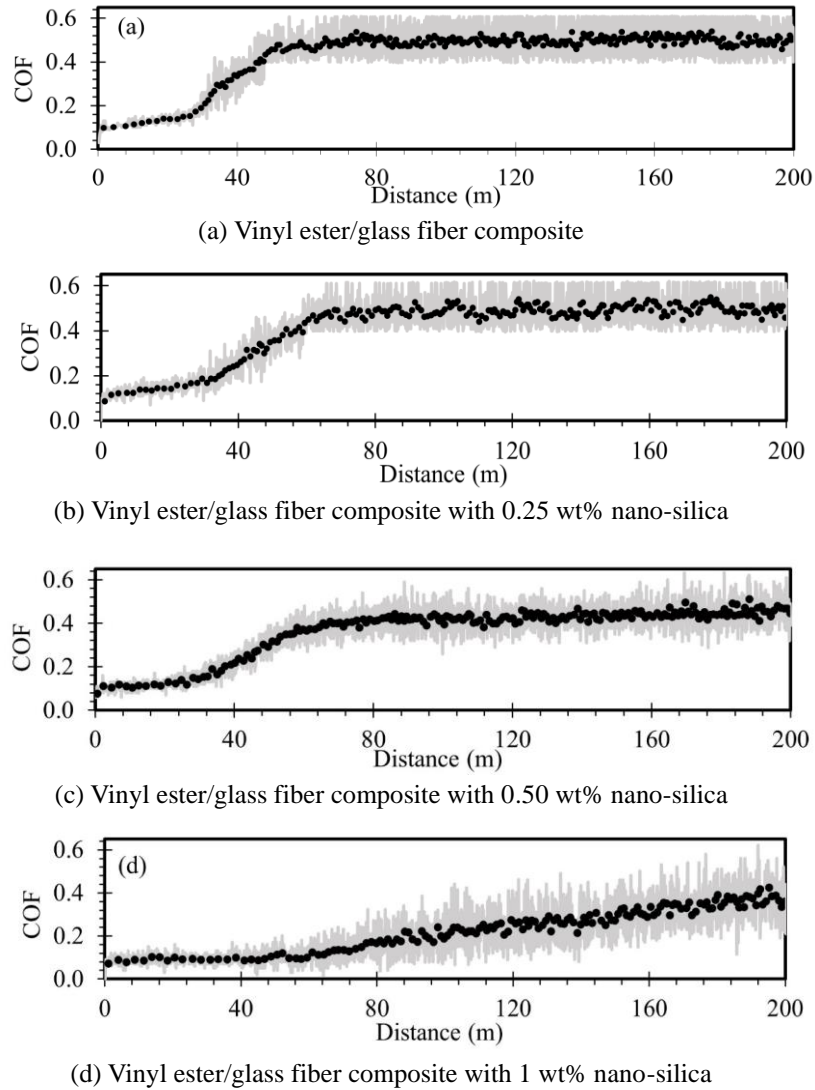


Fig. 13 The variation of COF for specimens

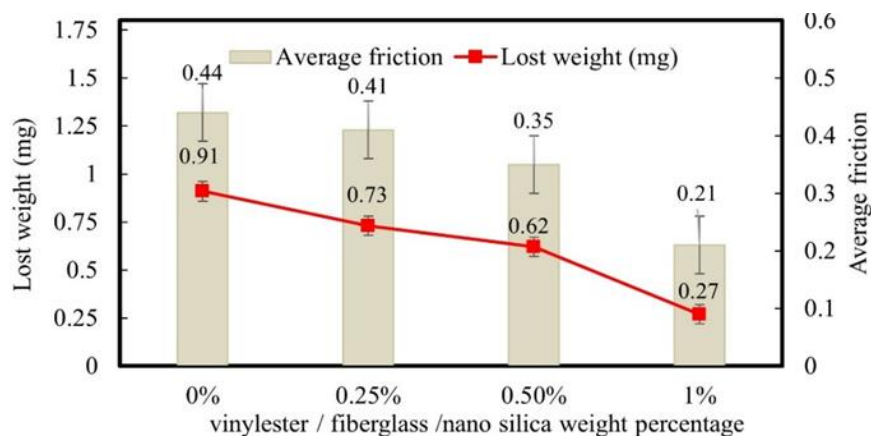
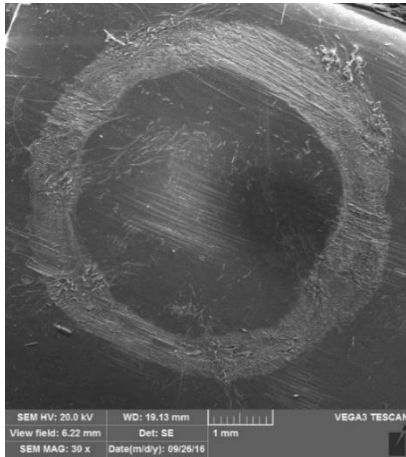


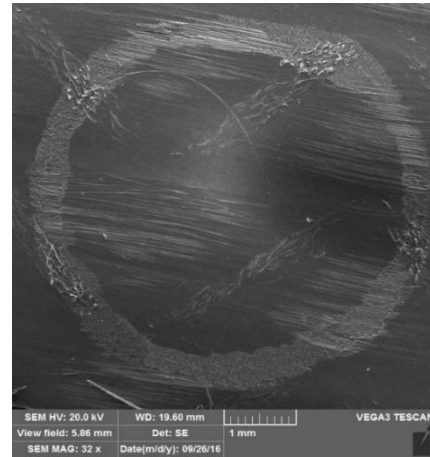
Fig. 14 Weight loss and the average value of COF for vinyl ester/glass fiber/nano-silica composite

growth of COF from 0.2 to 0.4 is due to eliminating the polymeric surface from the composite and the pin penetration to the fiber-contained region. Therefore, by considering the COF of 0.2 as a measure, it is evident that the traversed area for the pin to enter the fiber-contained

region is increased by increasing the weight percentage of the nano-silica. Also, after the pin penetrated the fiber-contained part of the composite and traversed some distance, the COF reached a stable value and oscillated around that. By increasing the content of the silica

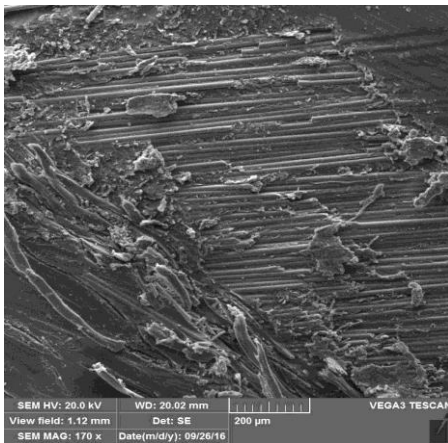


(a) Worn surface of vinyl ester/glass fiber composite

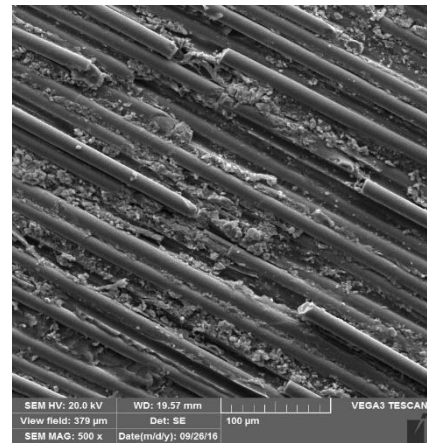


(b) Worn surface of vinyl ester/glass fiber/nano-silica composite containing 1 wt% nano-silica

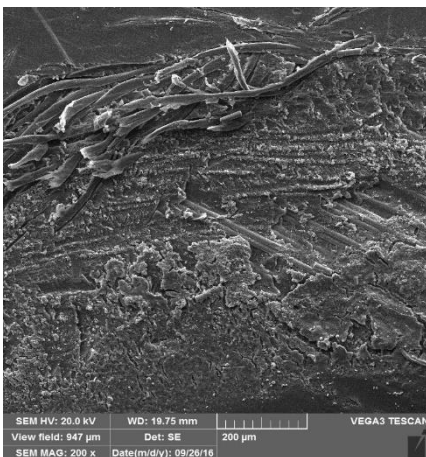
Fig. 15 SEM micrographs taken from worn surface



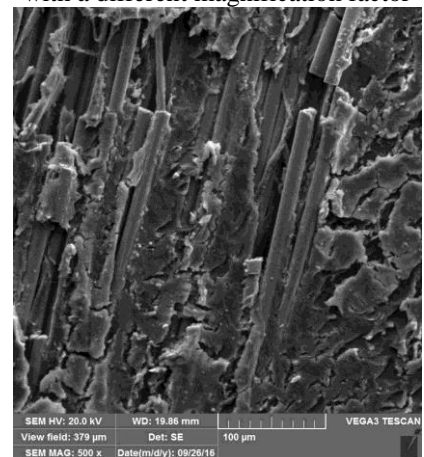
(a) Worn surface of vinyl ester/glass fiber composite



(b) Worn surface of vinyl ester/glass fiber composite with a different magnification factor



(c) Worn surface of vinyl ester/glass fiber/nano-silica composite containing one wt% nano-silica

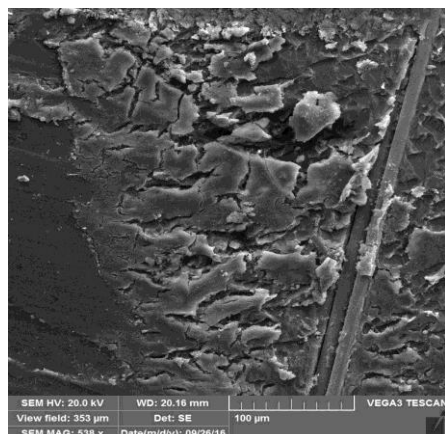


(d) Worn surface of vinyl ester/glass fiber/nano-silica composite containing one wt% nano-silica with a different magnification

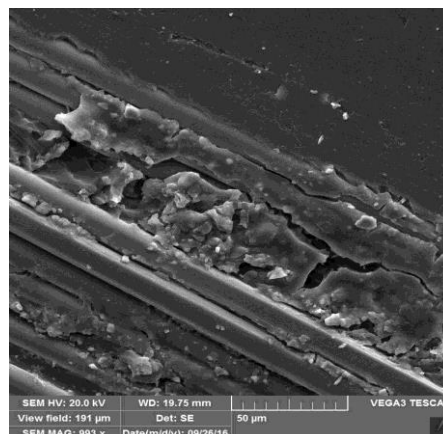
Fig. 16 SEM micrographs taken from wear zone

nanoparticles, the stable value of the COF was decreased. By adding one wt% nano-silica to the glass fiber composite, a 35% reduction was observed in the stable value of the COF.

As shown in Fig. 14, by adding nano-silica to the two-component composite, the percentage of weight loss due to specimen wear by the pin decreases. Therefore, adding nano-silica not only decreases the friction but also reduces



(a) Worn surface of vinyl ester/glass fiber composite



(b) Worn surface of vinyl ester/glass fiber/nano-silica composite containing one wt% nano-silica

Fig. 17 SEM micrographs taken from wear zone boundary

the weight loss due to the wear. Also, the average value of COF for 200 m wear testing of nano-silica/glass fiber composite is presented in this figure. According to this diagram, adding one wt% nano-silica decreases the average value of COF in a 200 m distance by 52.2%. The reason for this decrease in the COF is due to the role of the silica nanoparticles in tolerating a significant portion of the load applied by the pin to the composite material.

According to the SEM images of Fig. 15 captured from the worn surface of the two and three-component composites, it was revealed that the penetration depth of the pin in the two-component composite was much higher than the three-component composite. The width of the circle created due to wear indicates this issue. Also, these images prove the reason for the COF reduction and the weight loss due to the wearing of the three-component composite.

As shown in Fig. 16, the vinyl ester is fully degraded in the two-component composite and the glass fibers are severely failed and degraded. However, by increasing one wt% silica nanoparticles to the vinyl ester, the resulted composite well resisted wear, and the vinyl ester containing nano-silica kept its integrity after traversing 200 m. Also, it is worth noting that the glass fibers are well protected. The critical point is that the fracture and degradation of the glass fibers in the three-component composite is very low and insignificant. Fig. 17 clarifies that the vinyl ester is cracked at the worn boundary due to the 10 N force exerted by the pin. The crack growth in the vinyl ester resin of the two-component composite was much more significant than the crack growth in the vinyl ester resin of the three-component composite containing one wt% nano-silica. It means that the presence of silica nanoparticles improves the mechanical properties of the vinyl ester polymer by preventing the crack growth in the boundary of the wear test. Consequently, this boosts the wear resistance of the material.

#### 4. Conclusions

In the present study, the vinyl ester/glass fiber/nano-silica composites were fabricated with four different weight

percentages of nano-silica. To examine the influence of nano-silica on the wear and mechanical performance of the fabricated nanocomposite, the tensile, three-point bending, Charpy impact, and pin-on-disk wear tests were performed. In the following, the main results of these tests are briefly presented.

- It was found that the three-component nanocomposite containing 0.5 wt% of nano-silica has the maximum failure strength.
- The maximum normalized energy was absorbed by the three-component nanocomposite containing 0.5 wt% of SNPs.
- The SEM images of the binary composite in comparison to the ternary one after performing tensile test demonstrated that the nanoparticles act like a barrier to change the interlaminar and intralaminar crack propagation paths, wherein this barrier leads to an increase in the amount of toughness.
- The SEM images exhibited that the SNPs were properly distributed in the polymer matrix and confirmed the primary role of the nanoparticles in improving the mechanical and wear properties of the nanocomposite.
- By adding one wt% of SNPs to the two-component composite, the average value of COF decreased by an amount of 52.2%. Also, as another significant result for this composite, about 70% decrease in weight loss was achieved after conducting a tribometer test.

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