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Research Paper

# Comparison of Tribological Behaviours of Nano $SiO_2$ and $ZrO_2$ Reinforced Polyester Matrix Composite Materials

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**Abstract:** This paper aimed to examine and develop the tribological behaviour by adding nano reinforcements into the polyester matrix. Nanocomposite materials were obtained by adding nano-sized, 0.5%, 1%, 2% $SiO_2$ and $ZrO_2$ reinforcements to isophthalic polyester. These materials were mixed with mechanical and ultrasonic methods and poured into open molds. The coefficient of friction and wear rates were obtained using the pin-on-disc test device. Optical and SEM surface images of the composites were examined. The experiments were conducted at room temperature and an average of 70% humidity. Tribological studies were made under a load of 2, 5, 10N and at a sliding speed of 0.5 and 0.1 m/s. It was seen that the nano reinforcements to the polyester matrix improved the tribological properties of the material.

**Keywords**: Wear, coefficient of friction, nanocomposite material, $SiO_2$, $ZrO_2$

## 1. Introduction

With the development of technology, the need for superior materials is increasing. Therefore, with the studies in materials science, traditional materials are replaced by more qualified and more performance materials. Composite materials are the source of inspiration for these new high-quality and performance materials [1].

Today, polymers are used in almost every sector and field, as they are low density, easily mouldable, suitable for different purposes, chemically inert and corrosion-resistant materials. Due to these superior properties, studies are carried out to expand polymer materials' usage area and develop their mechanical, tribological, and surface properties. It is possible to improve various properties of these materials with reinforcements to the polymer matrix [2].

He et al. investigated the wear properties of the composite obtained by adding nano $SiO_2$ into the polymer matrix, and they got decreases of approximately 27% and 47.4% in the average friction coefficient and wear rate of nano-$SiO_2$ reinforced composites [3]. Zhaohong et al. investigated the tribological properties of the composite material obtained with nano $SiO_2$ reinforced into the polymer material. It has been determined that the friction properties have improved. In addition, the best friction properties between 1-5% of the reinforcements were obtained in 3% nano $SiO_2$ reinforced composites [4]. Li et al., thanks to the addition of nanographene as a reinforcement, achieved reductions of approximately 35% and 48% in the determined average friction coefficient and wear rate of polymer composites [5]. Beckford et al. aimed to increase the abrasion resistance of the composite by adding nano $SiO_2$ particles into the PTFE polymer matrix. They found that the wear

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resistance increased with 1.7% and 3.3% $SiO_2$ reinforcement. It has been determined that the 3.3% $SiO_2$ reinforced composite had lower peaks and grooves than 1.7%; therefore, it had higher abrasion resistance [6]. In another study, the abrasion resistance of the composite was obtained by reinforcing 0.5-10% nano-sized $ZrO_2$ into the epoxy matrix. With 0.5% $ZrO_2$ reinforcement, they have improved to 95% in wear rate. No reduction in wear rate was observed at higher reinforcement ratios [7].

Tribological properties of $ZrO_2$/$SiO_2$ nanoparticle-reinforced composites were investigated by Li et al. The studies determined that the friction coefficient and the wear rate decreased with nano $ZrO_2$/$SiO_2$ reinforcement. They also proved that surface modification to the surface nanoparticles improves the tribological properties of the composite [8].

Zhang et al. investigated the effect of nano $TiO_2$ reinforcement in PTFE, graphite, and short carbon fibre reinforced hybrid epoxy composite on wear performance. They show that wear properties improved with 5% nano $TiO_2$ reinforcement [9]. Nadia et al. investigated the wear behaviour of the nanocomposite obtained by adding nano $Al_2O_3$ and nano $SiO_2$ reinforcements into the epoxy matrix. The addition of 2% nano $SiO_2$ significantly improved the nanocomposite's wear performance. It is observed that there had been an increase in wear rates after the 2% reinforcement ratio [10].

As a result of the literature research on polymer matrix composite materials, it is seen that studies on the development of the tribological and surface properties of these materials are given importance. It has been determined that variables such as the type of reinforcement used, the amount of reinforcement, matrix material, applied load, and speed affect the material properties. In this study, the effect of nanoparticles added to the thermoset matrix to the tribology and surface performance of the material was investigated. In this context, polyester matrix, one of the most frequently used materials as a thermoset polymer, was chosen. Composite materials were obtained by adding nano-sized $SiO_2$ and $ZrO_2$ as reinforcement materials.

## 2. Materials and Method

Isophthalic polyester (1/100 hardener) was used as a matrix material. It has been determined that the composite material obtained by adding nano-sized silicon dioxide ($SiO_2$) and zirconium dioxide ($ZrO_2$) powders into the polyester matrix has better properties. The properties of the materials used in the studies are shown in Table 1. Silicon dioxide ($SiO_2$) and zirconium dioxide ($ZrO_2$) powders are used because they have a special place due to their excellent stability, high strength, high fracture toughness, superior wear resistance, high hardness, and excellent chemical resistance [11].

**Table 1.** The properties of the materials

| Materials | Properties | Brand |
|---|---|---|
| Polyester resin (POLYLITE 721-800) | Isophthalic | Reichhold™ |
| Silicon dioxide ($SiO_2$) | 10-20 nm | Alfa-Aesar™ |
| Zirconium dioxide ($ZrO_2$) | 12-25 nm | Alfa-Aesar™ |

Powders were dried in an oven at 80 °C to remove any moisture in the nanoparticles. Then, nanoparticles were reinforced to the polyester resin in determining amounts (%0,5-%1-%2) and mixed with a mechanical mixer at 250 rpm for 1 hour. At the end of this process, vacuuming was done to remove the bubbles formed in the resin. The premixed resin was transferred to an ultrasonic mixer and mixed for 15 minutes at 30% amplitude. Mixing was done in ice water so that the heat generated during ultrasonic mixing did not degrade the structure of the resin.

After adding the hardener to the prepared composite and mixing, vacuuming was performed again. The resulting mixture was poured into silicone and Teflon-based open moulds and cured in the mould at room conditions for 24 hours. Finally, the materials were kept in a drying oven at 80 °C for six hours post-curing.

## 3. Experimental Work

Wear tests were performed on pin-on-disc wear device according to ASTM G99 [12] standard. The diameter of the test specimens is 6 mm, and the length is 36 mm. 100Cr6 cold work tool steel was used as the disc material. Before the experiments, the samples were dried at 80°C for 4 hours to remove the moisture. The surfaces of the samples were polished with 2400 sandpaper. Then, the sample and disc surfaces were cleaned with alcohol and dried. Each experiment was repeated at least three times. The data was automatically saved to the computer during the experiments, and the average values were taken. Experiments were carried out under room conditions and average humidity of ~70%. The sliding speeds were 0.5 m/s and 1.0 m/s. The applied loads were 2N, 5N and 10N. The sliding distance was 250 m.

Before and after the test, the pin samples were weighed on a balance with a sensitivity of 0.0001 g. Specific wear rate was calculated according to Equation 1. k is the specific wear rate, Δm is the difference between the final and initial weight, ρ is the density, and d is the sliding distance [13].

$$k = \frac{\Delta m}{\rho . d} \ (mm^2 N^{-1} m^{-1}) \quad (1)$$

## 4. Result and Discussion

Figure 1 shows the contribution ratio of nano-reinforced composites and the changes in friction coefficients at 0.5 m/s sliding speed depending on the load. Reinforcements to the polyester resin changed the friction behaviour of the composites.

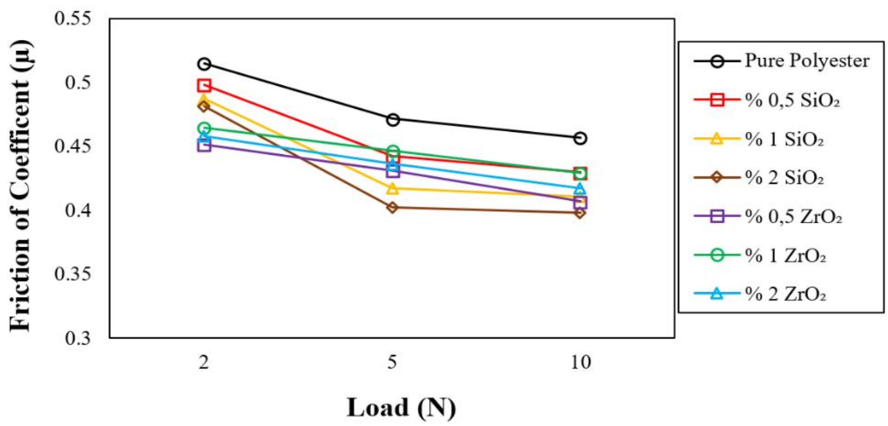


**Figure 1.** Variations of the friction coefficients of nanoparticle reinforced polyester composites at 0.5 m/s depending on the reinforcement ratio and the load

The lowest friction coefficient at 0.5 m/s occurs in the 2% nano $SiO_2$ reinforced polyester composite under 10N load in nanocomposites. It is seen that the coefficient of friction decreases with the increase of the reinforcement ratio. With the addition of 2% nano $SiO_2$, the friction coefficient decreased by 13%. This can be explained as the reinforced nanoparticles separating under the increasing load, making a rolling motion at the interface, and facilitating sliding, thus minimising the contact of the polymer with the disc surface, and reducing the friction coefficient.

He et al. found a decrease in friction coefficients by adding $SiO_2$ into the polymer matrix. They concluded that with the inclusion of nano $SiO_2$ reinforcements, more polymer material in polymer/$SiO_2$ composites is prevented from interacting with the friction surface, thus leading to lower friction coefficient values and specific wear rate [3]. Friedrich et al. examined the effect of various reinforcements on the wear and friction performance of the material; it can be concluded that the friction coefficient decreased from ~0.55µ to ~0.3µ with nano $TiO_2$, which they included at a rate of 5% in the PA-6,6 polymer matrix, thus nano reinforcements could reduce the friction coefficient of polymers. [14].

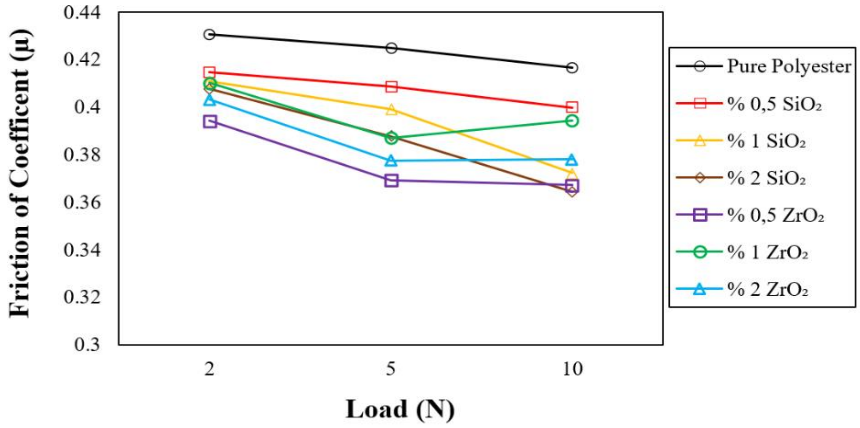


**Figure 2**. Variations of the friction coefficients of nanoparticle reinforced polyester composites at 1 m/s depending on the reinforcement ratio and the load

Figure 2 shows the contribution rate of nano-reinforced polyester composites and the changes in the friction coefficients at 1 m/s sliding speed depending on the load.

In nanocomposites, the lowest friction coefficient at 1 m/s occurs in the 2% nano $SiO_2$ reinforced polyester composite under 10N load. It is seen that the coefficient of friction decreases with the increase of the reinforcement ratio. The friction coefficient decreased by 12.5% with 2% nano $SiO_2$. Garcia et al. They investigated the tribological properties of the composite material obtained by adding nano-sized $SiO_2$ particles to the PA-6 polymer matrix. A decrease in the friction coefficient of the 2% nano $SiO_2$ reinforced composite was observed compared to the pure PA-6 matrix at a sliding speed of 0.1 m/s and under 1N load. It was determined that the friction coefficient of the pure PA-6 matrix decreased from 0.45 to 0.20 with a 2% nano $SiO_2$ addition. [15]. In the friction coefficient of nano $ZrO_2$ reinforced composites, under 10N load, an increase of 1% and 2% is observed compared to the 0.5% reinforced composite. With increasing load and sliding speed, the temperature increase on the friction surface and the non-homogeneous distribution of nanoparticles can cause this. At the same time, nanoparticles breaking off from the surface in mixtures above the optimum reinforcement ratio can increase the friction coefficient by causing abrasive wear. The lack of a similar mechanism in $SiO_2$ reinforced composites is explained by the fact that nano $SiO_2$ powders have higher interfacial bond strength and dispersion ratio with the matrix than $ZrO_2$ powders [16]. Friedrich et al. showed that the dispersion rate of nano $SiO_2$ particles in the matrix is better than nano $ZrO_2$ particles. While the optimum reinforcement ratio is 1.5% by volume in nano $ZrO_2$ particles, it can increase to 3.4% in $SiO_2$ reinforcements [14].

Figure 3 shows the changes in the wear rate of nano-reinforced polyester composites at 0.5 m/s depending on the reinforcement ratio and the load. Wear rates increase with increasing load. The nano reinforcements to the polyester matrix reduced the specific wear rate. While the lowest specific wear rates were under 2N load, the best wear performance was in 2% $SiO_2$ reinforced composite with a 78% reduction at 10N load compared to pure polyester.

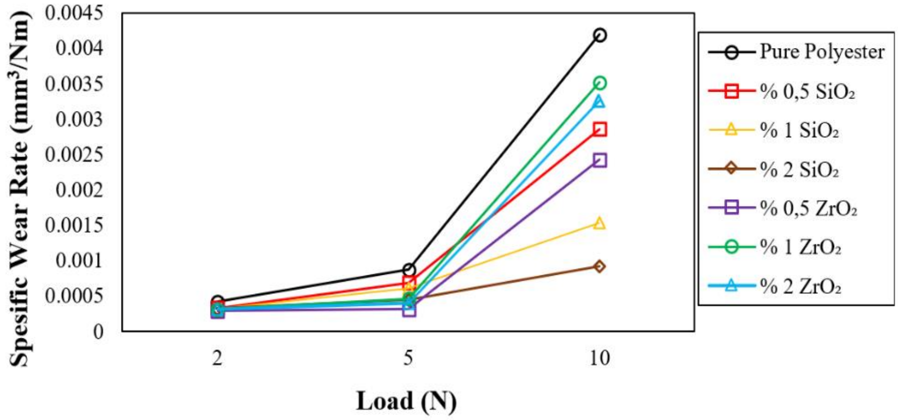


**Figure 3**. Variations of the wear rate of nanoparticle reinforced polyester composites at 0.5 m/s depending on the reinforcement ratio and the load

Garcia et al. examined the tribological properties of the composite obtained by adding 2% nano $SiO_2$ into the PA-6 polymer matrix. They found that the wear rate of the 2% nano $SiO_2$ reinforced composite decreased from 5.29x10-5 to 2x10-7 [15]. In addition, the wear rates of 0.5% $ZrO_2$ reinforced composites at 10N load decreased by 42%, 63% with 1% $SiO_2$ reinforcement, 32% with 0.5% $SiO_2$ reinforcement, 22% with 2% $ZrO_2$ reinforcement and 16% with 1% $ZrO_2$ reinforcement compared to pure polyester. When we look at the literature, Wang et al. proved that nano $SiO_2$ reinforcements show better abrasion performance than nano $ZrO_2$ reinforcements and that dispersion in $ZrO_2$ particles is difficult. Therefore, the agglomeration problem is more.

The wear performance of nano-reinforced composites depends on the homogeneous distribution of nano-reinforcements in the composite. The type, size, surface area, ratio of nanoparticles and mixing mechanisms affect their homogeneous distribution [17].

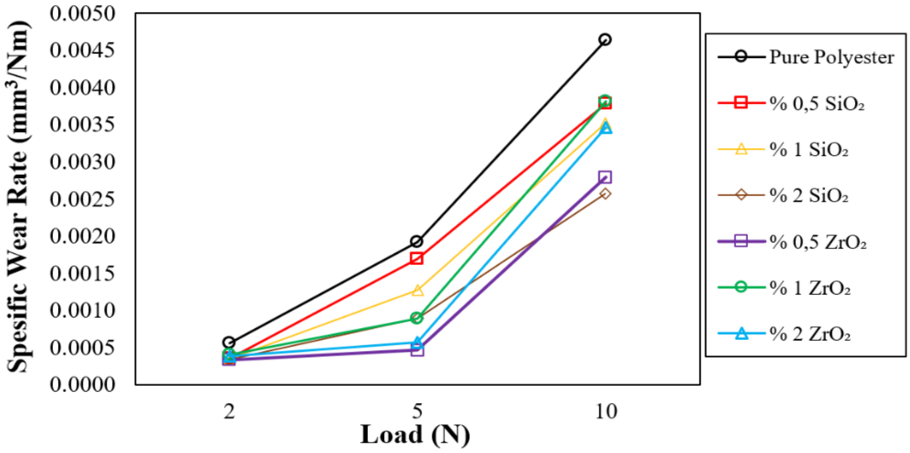


**Figure 4**. Variations of the wear rate of nanoparticle reinforced polyester composites at 1 m/s depending on the reinforcement ratio and the load

Figure 4 shows the changes in wear rates of nano-reinforced polyester composites at 1 m/s, depending on the reinforcement ratio and the load. Wear rates increase with increasing load. The nano reinforcements to the polyester matrix reduced the specific wear rate. The specific wear rate increases with the increase in sliding speed.

While the lowest specific wear rates were under 2N load, the best performance in composites was in 2% $SiO_2$ reinforced composite with a 44% reduction at 10N load compared to pure polyester. The wear rates of 0.5% $ZrO_2$ reinforced composites at 10N load decreased 39% compared to pure polyester. In other results, decreased of wear rates 24% with 1% $SiO_2$ reinforcement, 18% with 0.5% $SiO_2$ supplementation, 25% with 2% $ZrO_2$ reinforcement and 17% with 1% $ZrO_2$ reinforcement. It is thought that the interfacial bond strength between the matrix and the reinforcement decreases since the $ZrO_2$ reinforcement content causes agglomeration at a certain rate. For this reason, with the increase in the sliding speed, it is easier for the particles to break off from the sample surface and for the hard particles to have an abrasive effect on the surface. At the same time, with the increasing nano reinforcement, the homogeneity of the nanopowders in the resin decreases, forming agglomeration areas in the composite by showing agglomeration behaviour. Thus, it becomes easier for the hard nanoparticles to break off the matrix during sliding contact [18].

Friedrich et al. examined the tribological properties of the nanocomposite obtained by adding nano $TiO_2$ into the epoxy matrix; the wear rate decreased to 5x10-6 mm3/N with 5% nano $TiO_2$ to the pure epoxy with 47x10-6 mm3/Nm wear rate. However, wear performance decreases at higher reinforcement ratios than 5% reinforcement, if not lower than pure epoxy [19]. Oleiwi at al. determined that the wear resistance of the composite material obtained by adding nano $SiO_2$ to the polyester matrix is to two-fold increase increased compared to pure polyester [20]. Garcia et al. investigated the tribological properties of the composite by adding 2% nano $SiO_2$ to the PA-6 polymer matrix. While the wear rate of pure PA-6 polymer was 5.29x10-5, it was determined that the wear rate decreased to 2x10-7 with 2% nano $SiO_2$ reinforcement, but the wear rate increased to 2.81x10-5 with 14% $SiO_2$ reinforcement [15]. Considering these studies, it is determined that the wear performance may decrease if the optimum reinforcement ratio is exceeded.

### 4.1. Optical Microscope and SEM Images

Generally, materials must have stable friction performance, low friction coefficient and high wear resistance with a low wear rate [21]. This section gives optical and SEM images of the samples that meet these conditions. Optical microscope images of pin and disc wear surfaces of $SiO_2$ $ZrO_2$ reinforced polyester composites under 10N, and 0.5 m/s velocity conditions are given in Figure 5.
The wear marks of pure polyester are in the sliding direction, and there are surface damages. With the $SiO_2$ reinforcement, the particles that break off during wear fill the rough spots between the surface and the sample and reduce the wear marks. At the same time, it is seen that a thin film layer is formed on the disc surfaces. With the increasing additive ratio, the transfer film layer decreases with the effect of hard particles. The amount of material separated on the disc decreased with the nano additive ratio. With the $SiO_2$ and $ZrO_2$ reinforcement, the particles that break off during wear fill the rough spots between the surface and the sample and reduce the wear marks. At the same time, it is seen that a thin transfer film layer is formed on the disc surfaces. The amount of material separated on the transfer film layer and disc has decreased with the effect of hard particles and the nano additive.

It is observed that the friction coefficient decreases with the increase of $SiO_2$ additive. When we look at the disc's surface, it can be said that the traces are reduced, so the nanoparticles facilitate the sliding.

Optical microscope images of pin and disc wear surfaces of $SiO_2$ and $ZrO_2$ reinforced polyester composites under 10N load, and 1 m/s velocity conditions are given in Figure 6. The wear marks of pure polyester are wavy in the direction of sliding. With the $ZrO_2$ reinforcement, the particles that break off during wear fill the rough spots between the surface and the sample and reduce the wear marks. As the sliding speed increases, the scratches deepen. It is thought that hard nanoparticles that break off due to particle aggregation enter the interface and increase the damage. A transfer film layer is seen on the disc surface.

The lowest surface damage occurs in the 0.5% $ZrO_2$ added polyester composite. It is thought that hard nanoparticles that break off due to particle aggregation at other additive rates increase the damage by entering the interface. There is a transfer film layer on the disc surface. The least and thinnest transfer film layer occurs in 0.5% $ZrO_2$ added composite.

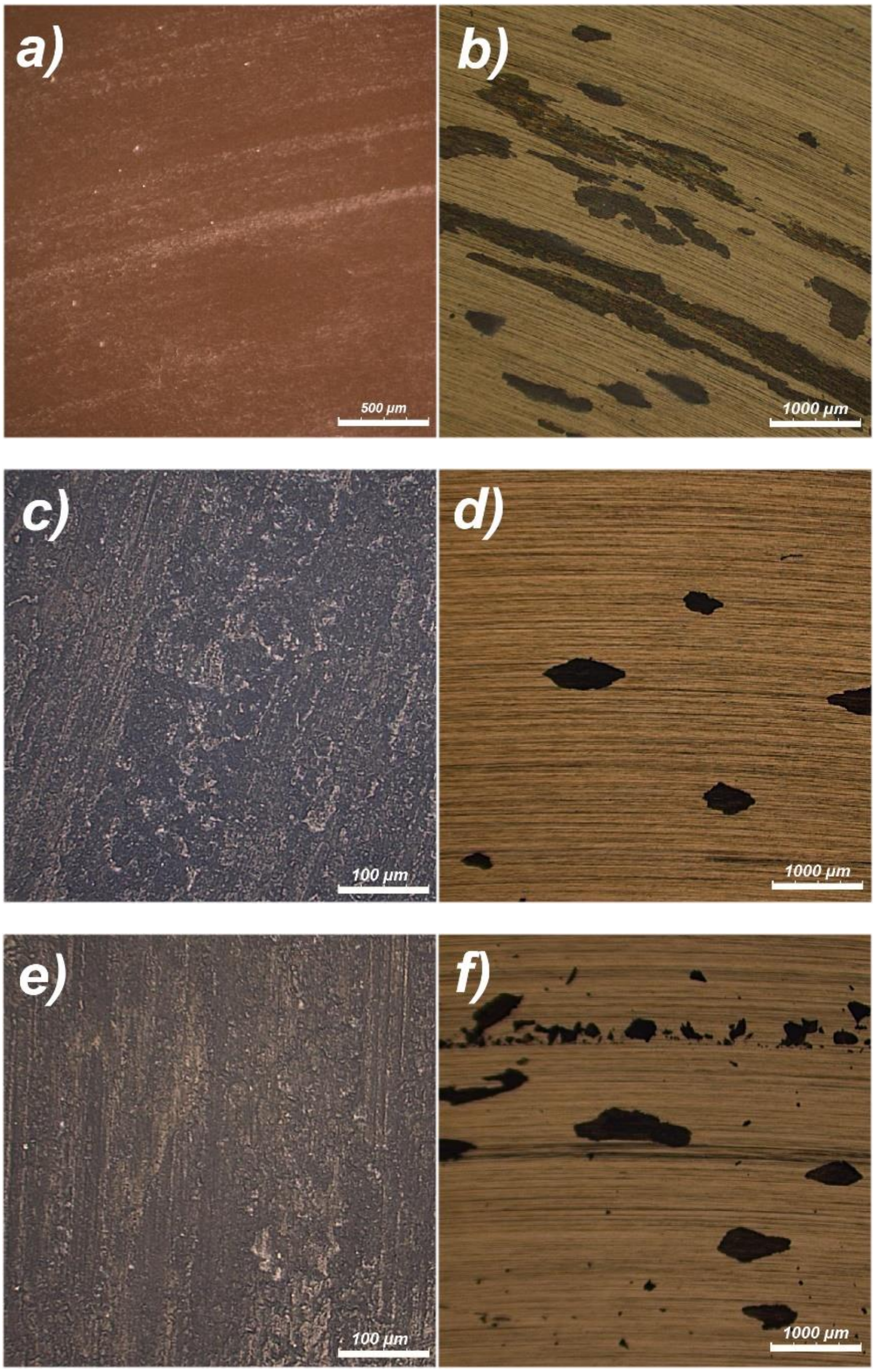


**Figure 5.** Optical microscope images of pin and disc wear surfaces under 10 N and 0.5 m/s a) Pure polyester pin, b) Pure polyester disc, c) 2% nano $SiO_2$ reinforced pin, d) 2% nano $SiO_2$ reinforced disc, e) 0.5 % nano $ZrO_2$ reinforced pin, f) 0.5 % nano $ZrO_2$ reinforced disc

SEM images of pure polyester and reinforced polyester composites were made in the figures below. Figure 7 shows the surface image results of the isophthalic polyester used as a matrix under 1000x magnification.

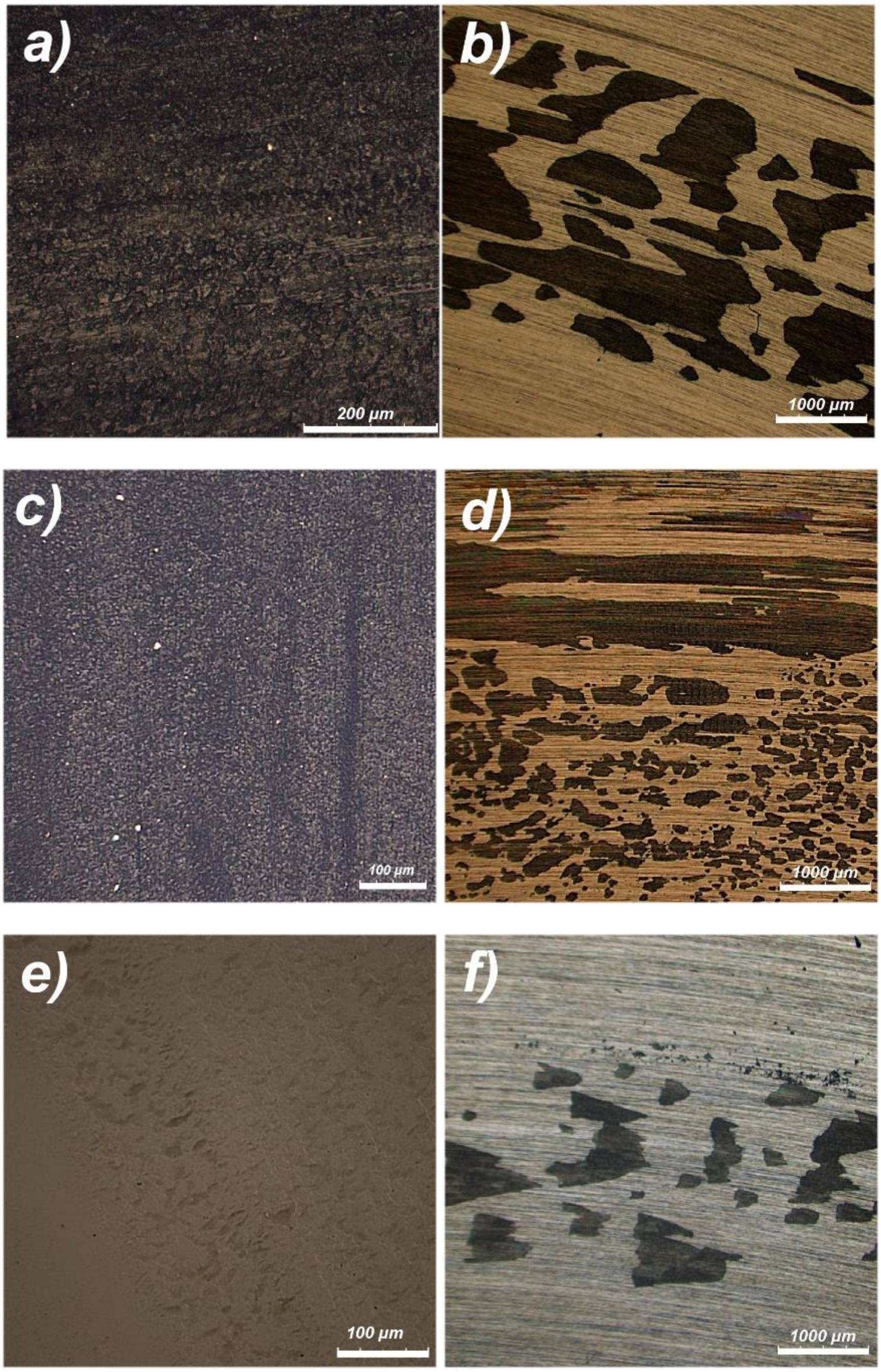


**Figure 6.** Optical microscope images of pin and disc wear surfaces under 10 N and 1 m/s a) Pure polyester pin, b) Pure polyester disc, c) 2% nano $SiO_2$ reinforced pin, d) 2% nano $SiO_2$ reinforced disc, e) 0.5 % nano $ZrO_2$ reinforced pin, f) 0.5 % nano $ZrO_2$ reinforced disc

When we look at the broken surface image of 0.5% nano $SiO_2$ reinforced polyester composite in Figure 8, it is understood that the agglomerated particles show a homogeneous distribution even if they are partial. In the surface image of 2% nano $SiO_2$ additive polyester composites, the density of nanoparticles has increased even more. With the increasing contribution rate, an increase occurs in the atomic percentage. As a result of the analysis, the distribution of the nano $SiO_2$ reinforcement in the matrix and the changes in the composite internal structure caused by the differences in the additive ratio and agglomeration are seen. The distribution of the increased additive ratio in the matrix is consistent with the wear and friction results.

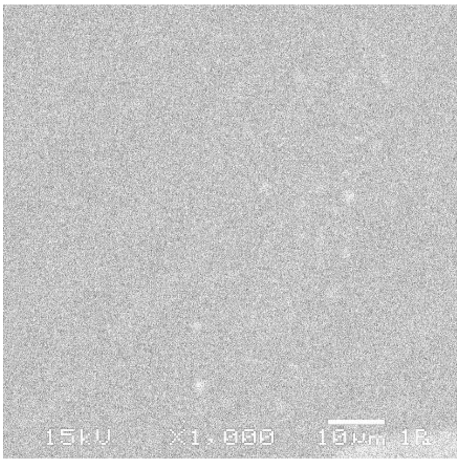


**Figure 7.** SEM images of pure polyester

SEM images of nano $SiO_2$ reinforced polyester composites were performed. Figure 8 shows the surface images of the $SiO_2$ reinforced polyester composite under 1000x magnification.

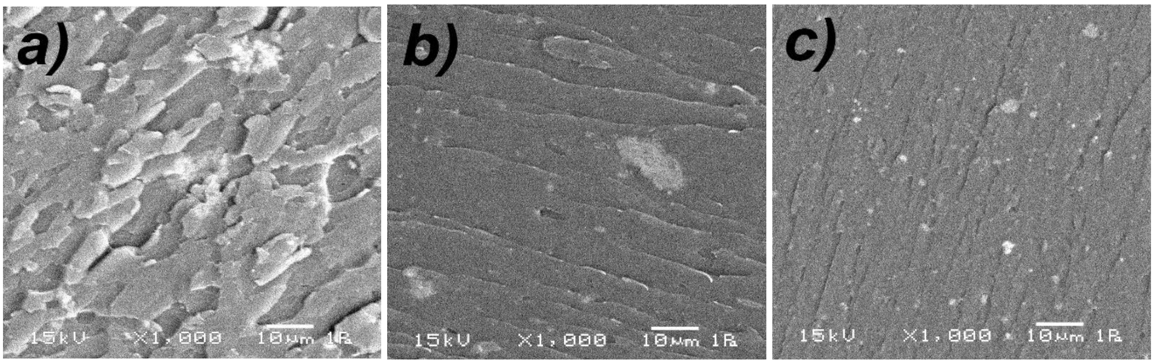


**Figure 8.** SEM images of nano $SiO_2$ reinforced polyester composites a) 0.5% $SiO_2$ b) 1% $SiO_2$ c) 2% $SiO_2$

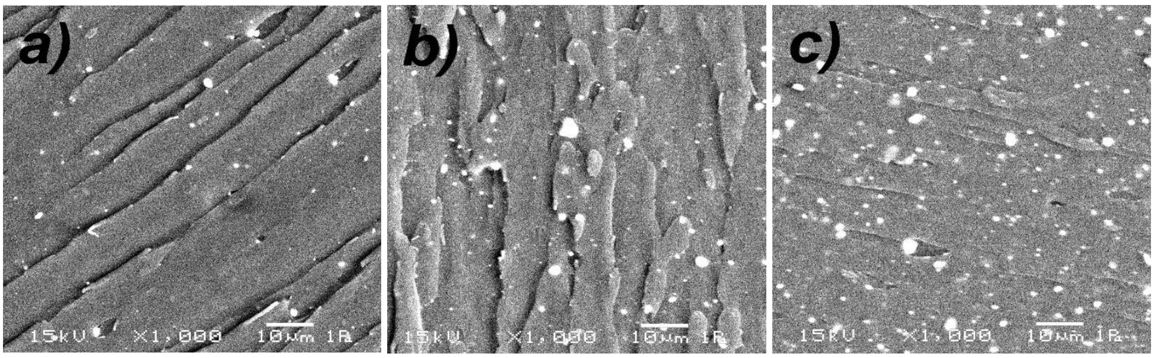


**Figure 9.** SEM images of nano $ZrO_2$ added polyester composites a) 0.5 % $ZrO_2$ b) 1 %$ZrO_2$ c) 2 % $ZrO_2$

SEM images of nano $ZrO_2$ reinforced polyester composites were performed. Figure 9 shows the surface images of the $ZrO_2$ reinforced polyester composite under 1000x magnification.

When we look at the broken surface image of 0.5% nano $ZrO_2$ added polyester composite in Figure 9, it is understood that the particles show a homogeneous distribution. It is seen that the most homogeneous mixture occurs in 0.5% nano $ZrO_2$ added composites, and agglomeration is minimal. Zhang et al. explained that nano $SiO_2$ additives show better distribution than nano $ZrO_2$ additive, and the optimum additive ratio percentage is higher in nano $SiO_2$ additives [22].

## 5. Conclusions

In this study, composite materials were prepared by adding nano-sized $ZrO_2$ and $SiO_2$ reinforcements into the polyester matrix. The tribological properties of composite materials were investigated. It was seen that the nano reinforcements to the polyester matrix improved the tribological properties of the material.

- With nano reinforcements to isophthalic polyester resin, wear, and friction properties are improved.
- The best wear and friction performance were obtained in 2% nano $SiO_2$ reinforced and 0.5% $ZrO_2$ reinforced composites.
- A general decrease in friction coefficients of pure polyester and nano-reinforced composites were observed with increasing load in experiments. However, there is an increase in wear rates.
- The friction coefficients decrease as the sliding speed increases, while the wear rates increase.
- Adhesive and abrasive mechanisms were effective in wear mechanisms.

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## Authors' Contributions

All tests, optical microscopy and SEM examinations were performed by MA and SCA. MA conducted the literature review. The design of the wear tester was made by NO and MO. Test samples were prepared by MA and SCA. AA is the project manager. MA and NO wrote the article. NO and AA made final checks of the article. All authors have read and approved the final version of the article.

## Competing Interests

The authors declare that there is no conflict of interest.

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