# COMPORTAREA LA UZURĂ A COMPOZITELOR POLIAMIDA 6/ POLIETILENĂ DE ÎNALTĂ DENSITATE / NANOARGILĂ

# WEAR BEHAVIOR OF POLYAMİDE 6/ HIGH DENSITY POLYETHYLENE/NANOCLAY COMPOSITES

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***This study investigates the wear behavior of the composites such as wear rate-load, coefficient of friction-load and coefficient of friction-direction relationships. For this purpose, polyamide 6 (PA 6), high density polyethylene (HDPE), montmorillonite as nanoclay (NC) and the compatibilizer maleic anhydride-grafted-polyethylene (PE-g-MA) are used in this study. The HDPE, nanoclay (montmorillonite) and the compatibilizer are added to a PA 6 matrix material and their effect on the wear property of the composite is investigated. The wear tests are performed in a pin-on disk machine to determine these properties. Optical microscope examination of the wear surfaces is conducted by a transmission electron microscope (TEM) in order to find out more about the material characterization. As a result of the experiments, it is observed that 1% nanoclay (montmorillonite) added to the material has a positive effect on the wear. However, the wear is observed to increase with an increase in the rate of the nanoclay.***

***Keywords:*** *Polymer, Nanocomposites, Polyamide 6, High density polyethylene, Wear*

## 1. Introduction

Polymer composites are lightweight and chemically resistant materials. These properties have expanded their usage area in recent years and they have increasingly started replacing metallic materials in such sectors as automotive, construction and chemistry [1 - 3]. Compared with other polymers, polyamides exhibit excellent wear behavior, high chemical resistance and superior mechanical properties, therefore, they are widely used in many areas [4]. The most important polyamides in terms of their mechanical and physical properties are polyamide 6 and PA 66, as their hardness, durability and heat resistance are better than those of all other polyamide types [5]. Nanocomposites have emerged as a new class of composite materials obtained by the addition of reinforcements in the range of 1–100 nm into matrix [6]. Recent studies on nano-structured materials indicate that the addition of nano additives increases hardness and has a positive effect on the combustion, chemical and thermal properties of materials. This positive effect is due to the large surface area of the nanostructures which increases the interaction between the matrix and the nano reinforcement. Another positive feature of nanoscale particles is that they can be easily dispersed in the polymer-matrix. Especially, low rates of nano additions into PA 6 were found to improve the wear behavior under dry sliding conditions. In addition, nanosize reinforcements form a solid film layer on wear surfaces and improve the friction and wear properties of materials [7]. Montmorillonite (MMT) consisting of silicate layers is widely used in polymer nanocomposites [8]. Silicate layers of montmorillonite are stacked together (interstratification) and each silicate layer is 1 nm in thickness. Natural montmorillonite is hydrophilic. Its organophilic property is enhanced by an ion exchange with quaternary ammonium. Modified in this way, it is referred to as organoclay. The compatibility of the organoclay in the polymer is better [9,10]. Since a couple of the polymers have superior tribological properties, most studies have focused on these polymers [11]. The size of the nanoparticles is very small, therefore, when added to the material, they are wrapped by polymer chains and this process increases the bonding strength [12,13]. Srinath and Gnanamoorthy [14,15] and Dasari et al. [16] studied the wear and friction behavior of the material by adding organoclay into PA 6. The results of their study indicate that the wear resistance of the material increases while the coefficient of friction decreases. Dayma et al. [17] found that PA-6/PP-g-MA with differentnanoclay proportions increased the wear resistance of the composite. As a result of the study in which polypropylene and nanoclay were added topolyamide 66 matrix, Suresha et al. [18] determined that the nanoclay decreased the

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coefficient of friction of the material and increased the wear resistance.

This study investigates the effect of montmorillonite as nanoclay addition on the wear properties, such as wear rate-load, coefficient of friction-load and coefficient of friction-direction relationships, of PA 6/HDPE composite with different nanoclay proportions and the compatibilizer. Pin-on-disk tests and Transmission Electron Microscope were employed to characterize the wear traces.

## 2. Materials and Methods

The materials used in the current study were as follows; PA 6 (DSM Akulon F223-D) as the matrix material; 1%, 3% and 5% Montmorillonite (Nanocor I30) as the organoclay; 20% High Density Polyethylene (Petkim); 5% and 10% Maleic anhydride grafted polyethylene (Polybond HKMA 04) as the compatibilizer. Granules were dried at 80 °C for 12 h. The materials were mixed and the mixture was extruded using a Werner & Pfleiderer GmbH. ZSK 25 intermeshing co-rotating twin screw extruder. The length/diameter (L/D) ratio was 48, D = 25 mm. With the twin screw extruder, a compound was obtained by mixing polymer, nanoclay and compatibilizer. The nanocomposites were prepared by mixing the compounds at a cylinder barrel temperature at 230, 240 and 250 °C. The extruder screw speed was set at 500 rpm. The samples were injection molded using a Yonca Injection Molding Machine. The temperatures maintained in the three zones of the molding machine barrel were between 220 and 240 °C (L/D: 24, D: 65 mm) and the injection pressure was set at 100 bar. Table 1 shows the details of the composites.

TEM samples were cut into pieces of 80 nm thickness by a CryoLeica EM UC7 Ultramicrotome at room temperature. TEM investigations were carried out by a ZEISS LEO 906 device in order to determine how the nanoclay was dispersed in the material. The operating voltage was set at 80 kV.

The wear test samples were 6 mm diameter and 50 mm long. Before testing, the surface of a steel disk (Ra= 0.35 μm), was polished by AISI D2 sandpaper to provide a good contact during the sliding experiment. Before each wear test, the disk surfaces were thoroughly cleaned with acetone. Wear tests were performed using a pin-on-disk device with loads of 80 N, 120 N and 160 N, at a sliding speed of 1 m/s for a sliding distance of 10000m. The loss in pin volume was calculated and recorded using the density of the corresponding polymer. The specific wear rate (Ks) [$mm^3$/Nm)] was calculated by:

$$Ks = \frac{\Delta V}{LP}$$

where $\Delta V$ is wear volume, $P$ is the applied load and $L$ is the travelling distance. The coefficient of friction values (μ) were directly obtained from the equipment that recorded the μ value using the following relation:

$$\mu = \frac{Fs}{Fn},$$

where Fs is the frictional force and Fn is the applied load on the sample.

Generally, the specific wear rate is defined as the wear volume normalised by the normal load and the sliding distance. The mass loss (m) was measured after each set of run and volume loss (V); V= *m/p* was found by using the density (p) of the sample. Wear surfaces were examined using a Nikon SMZ 800 optical microscope.

## 3. Results and Discussions

Mechanical properties in composite materials depend not only on the ratio and the shape of reinforcements, but also on the distribution of reinforcements added into composite. The shape of the particles in the matrix in nano–structured materials is especially important. A TEM examination was conducted to determine this distribution. Figure 1 shows a homogeneous distribution of the nanoclay in the matrix and in the

**Table 1**

Composites

| Samples | weight% | | | |
|---|---|---|---|---|
| | PA 6 | HDPE | PE-g-MA | Nanoclay |
| PA 6 | 100 | - | - | - |
| HDPE | 100 | - | - | - |
| PA 6/HDPE | 80 | 20 | - | - |
| PA 6/HDPE/PE-g-MA | 80 | 20 | 5 | |
| PA 6/HDPE/PE-g-MA/Nanoclay | 80 | 20 | 5 | 1 |
| PA 6/HDPE/PE-g-MA/Nanoclay | 80 | 20 | 5 | 3 |
| PA 6/HDPE/PE-g-MA/Nanoclay | 80 | 20 | 5 | 5 |
| PA 6/HDPE/PE-g-MA/Nanoclay | 80 | 20 | 10 | 1 |
| PA 6/HDPE/PE-g-MA/Nanoclay | 80 | 20 | 10 | 3 |
| PA 6/HDPE/PE-g-MA/Nanoclay | 80 | 20 | 10 | 5 |

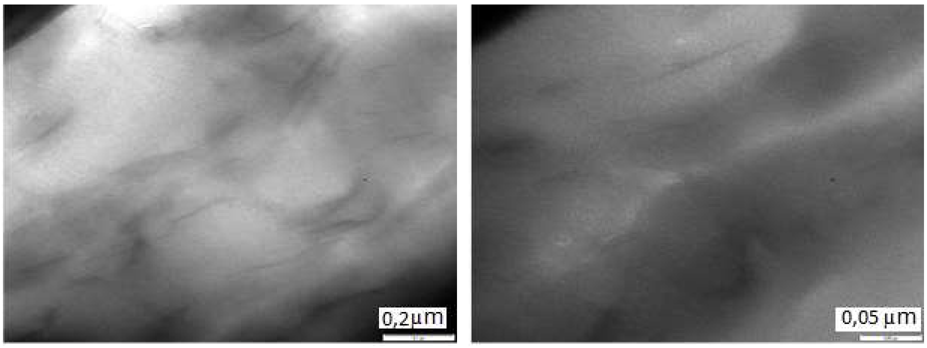



Fig. 1 - TEM images of PA 6/ HDPE/PE-g-MA / Nanoclay(80/20/10/5).

areas of intercalated structures with those containing partial exfoliation structures. In addition, it is observed that nano particles adjust themselves in a way during injection. Hence, the nanoclay can be said to have achieved a uniform dispersion in the PA 6 matrix.

Figure 2 shows that the pure PA 6 had the highest coefficient of friction (0.592) at 1 m/s and at 80 N load. The pure HDPE has the lowest coefficient of friction. The coefficient of friction of the material produced in the ratio of PA 6/HDPE (80/20) was observed to be lower than that of the pure PA 6 while higher than that of the pure HDPE. The coefficient of friction of the PA 6/HDPE (80/20) was reduced 74% by the addition of 5% PE-g-MA and dropped from 0.328 to 0.244 especially at 80 N load. Therefore, it can be claimed that the compatibilizer reduces the coefficient of friction. This reduction is thought to result from the fact that the compatibilizer reduces the surface tension between PA 6 and HDPE, forming new bonds and creates a uniform microstructure. However, when the rate of the compatibilizer content is raised to 10%, this positive effect is eliminated. The highest reduction in the coefficient of friction (44.5%) was observed to occur in the pure PA 6 with an increase in the load.

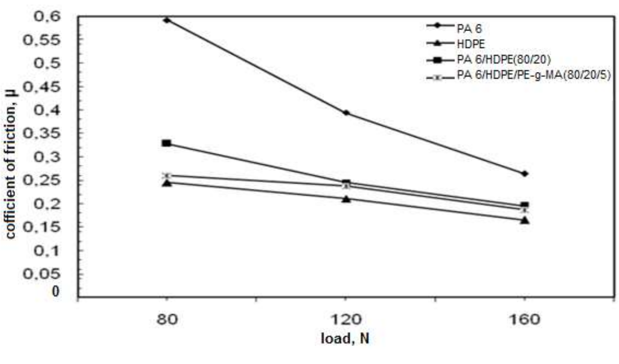



Fig. 2 - Variation of coefficient of friction with applied load for pure PA 6, pure HDPE, PA 6/HDPE (80/20), PA 6/HDPE/C (80/20/5) composites tested against stainless steel (velocity:1 m/s).

The coefficient of friction-load relationship diagrams are shown in Figure 3 with regard to the nanoclay-structured composites. It can be seen that the coefficient of friction was reduced by the addition of 1% nanoclay addition into the material. Srinath and Gnanamoorthy [15] attributed this reduction to a more effective film layer formed between the metal disk and specimens due the presence of nanoclay addition. The lowest coefficient of friction, especially at 80 N and 120 N loads, was observed in the PA 6/HDPE/PE-g-MA/Nanoclay (80/20/5/1) composition. The coefficient of friction increased with an increase in the rate of the nanoclay in the material. The coefficient of friction decreased with an increase in the load. However, the difference between the coefficients of friction decreased with an increase in the load. The coefficient of friction-load relationship at 160 N load reveals that the values are very similar to each other.

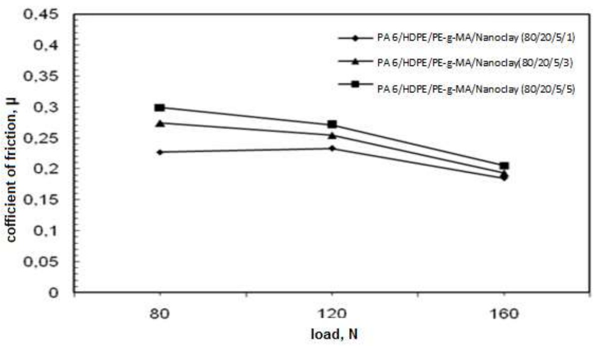



Fig. 3 - Variation of the coefficient of friction at applied load for PA 6/HDPE/C/NC (80/20/5/1), PA 6/HDPE/C/NC (80/20/5/3), PA 6/HDPE/C/NC(80/20/5/5) composites tested against stainless steel (velocity: 1 m/s).

The coefficient of friction-load relationship (at 1 m/s) of the composites produced with the mixing ratios of PA 6/HDPE/PE-g-MA/Nanoclay (80/20/10/1), PA 6/HDPE/PE-g-MA/Nanoclay (80/20/10/3) and PA 6/HDPE/PE-g-MA/Nanoclay (80/20/10/5) are shown in Figure 4. The variation in the coefficient of friction -load relationship with the increase in the compatibilizer content in the material to 10% was examined. As can be seen in the diagram that, the increase in the amount of compatibilizer content from 5% to 10% resulted in an increase in the coefficient of friction. The coefficient of friction increased with an increase in the nanoclay additions in the composite. In general, the coefficient of friction values of the material decreased with an increase in the load. Unal et al. [19] obtained the similar results using different

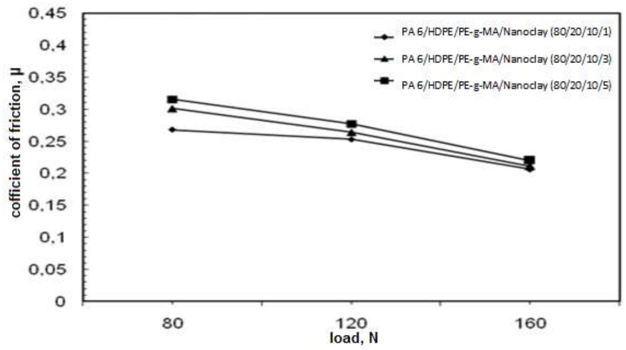



Fig. 4 - Variation of coefficient of friction at applied load for PA 6/HDPE/C/NC (80/20/10/1), PA 6/HDPE/C/NC (80/20/10/3), PA6/HDPE/C/NC(80/20/10/5) composites tested against stainless steel (velocity: 1 m/s).

polymers and polymer reinforced composites in their studies.

Figure 5 shows the wear rate-load relationship of the pure PA 6, pure HDPE, PA 6/HDPE (80/20) and PA 6/HDPE/ PE-g-MA (80/20/5) composites at 1m/s speed. The wear rate values of these materials are quite different from each other. The pure HDPE exhibits the highest wear rate at the applied load. The reason behind this is thought to be the fact that HDPE is softer and has a lower melting temperature than the other ingredients therefore it is affected by the heat generated during the wear process. It is also believed that the applied load has an effect on the wear. The pure PA 6 was worn less than HDPE. The wear rates of both materials decreased with an increase in the applied load. The reason of this reduction is thought to result from the film layer formed on the disk surface during friction. The wear behavior of the material produced in PA 6/HDPE (80/20) composition varied from that of the pure PA 6 and HDPE. Its wear rate is lower than those of the pure materials. Although its wear rate showed a slight downward trend with an increase in the load, this reduction was less than those of the pure materials. From this point of view, it can be claimed that the wear rate of specimens showed a significant reduction. The wear rate of PA 6/HDPE/ PE-g-MA (80/20/5) composite tends to decline a little with an increase in the load. With 5% compatibilizer content, the wear rate decreased at 80 N and 120 N loads. Therefore, PA 6/HDPE/ PE-g-MA (80/20/5) composite can be said to have the lowest wear.

Figure 6 shows the effect of the nanoclay addition on the wear rate. As the figure illustrates, the wear rate trends of all the materials are similar which means that the wear rates of all the materials decreased with an increase in the load. 1% montmorillonite nanoclay added to PA 6/HDPE/ PE-g-MA (80/20/5) composite dropped the wear rate from 10.89 to 10.71 at 80 N load, from 10.49 to 7.75 at 120 N load, and from 80.1 to 7.71 at160 N load. Approximately the same values were obtained when the rate of the nanoclay was increased to 3% while

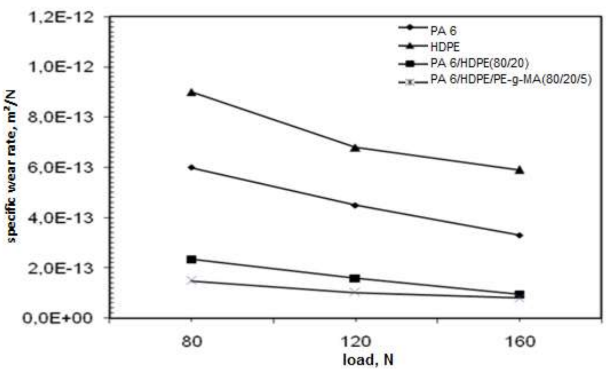



Fig. 5 - Variation of the wear rate atapplied load forpure PA 6, pure HDPE, PA 6/HDPE(80/20), PA 6/HDPE/ PE-g-MA(80/20/5) nanocomposites tested against stainless steel (velocity: 1 m/s).

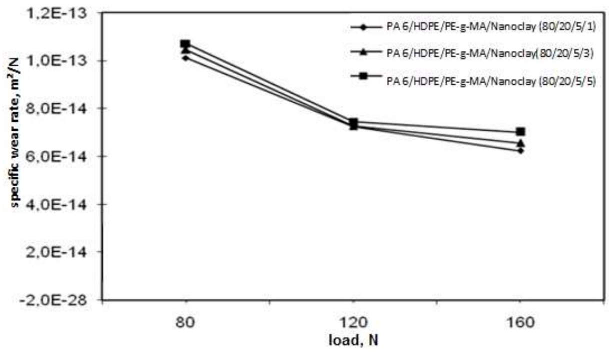



Fig. 6 - Variation of wear rate atapplied load PA 6/HDPE/ PE-g-MA/Nanoclay (80/20/5/1),PA 6/HDPE/ PE-g-MA/Nanoclay (80/20/5/3), PA 6/HDPE/ PE-g-MA/Nanoclay (80/20/5/5) nano composites tested against stainless steel (velocity: 1 m/s)

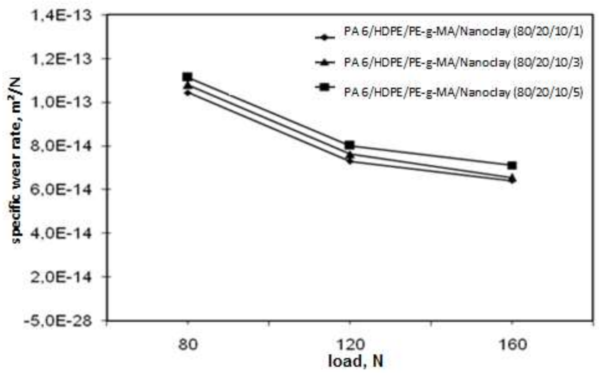



Fig. 7 - Variation of wear rate atapplied load PA 6/HDPE/ PE-g-MA/Nanoclay (80/20/10/1), PA 6/HDPE/ PE-g-MA/Nanoclay (80/20/10/3), PA 6/HDPE/ PE-g-MA/Nanoclay (80/20/10/5) nano composites tested against stainless steel (velocity: 1 m/s)

the wear rate increase when 5% nanoclay was added to the material

The effect of compatibilizer on the wear rate is shown in Figure 7. The variation of the wear rate was compared with the increase in the compatibilizer content up to 10% in the composites. The increase in the compatibilizer content from 5% to 10% resulted in an increase in the wear rate. The possible reason is that the structure of the polymer chains of the matrix material had deteriorated and the plastic zones in the interface increased with an increase in the amount of the compatibilizer. Bhimaraj et al. [20] determined that when the amount of nanoparticle added to the material exceeds 2%, it results in an increase in the wear rate.

The coefficient of friction-sliding distance diagrams are shown in Figures 8, 9 and 10 for the materials at 80 N load and at 1 m/s. The pin-on disk test resulted in an oscillating coefficient of friction which was characterized by an increase especially for the pure PA 6 up to the first 2000 m sliding distance, followed by a decrease. The coefficient of friction decreased with the addition of the compatibilizer to the specimens and it became more stable along the sliding distance. After the first 1000 m sliding distance, the coefficient of friction decreased with the nanoclay additions to the structure and the coefficient of friction became virtually stable. The increase in the amount of nanoclay additions in the structure resulted in an increase in the coefficient of friction. The compatibilizer and the nanoclay added to the material made the coefficient of friction more stable along the sliding distance. Bhimaraj et al. [20] determined that the wear area of the wear surface film layer for the pure PET was 51% while it rose up to 76% when 2% nanoparticle was added to alumina. They state that nanoparticles play an important role in the formation of a film layer on the counter surface and they decrease the coefficient of friction and make it more stable. The coefficient of friction increased with an increase in the amount of nanoclay additions in the composite, which is believed to result from the polymer chains becoming weaker due to increasing nanoclay additions which reduces the total bond surface within the polymer. The compatibilizer and the nanoclay added to the specimens made the coefficient of friction more stable along the sliding distance.

Figure 11 shows the wear traces formed on the disk surface of the materials at 100 N load, at a sliding speed of 1 m/s for a sliding distance of 10000 m. The disk surfaces indicate that abrasive, adhesive or both wear types occurred in the specimens, simultaneously. This could be explained by three mechanisms ploughing, wedging and cutting, and heating effect resulting from the friction, which promoted the softening the polymer surface. PA 6/HDPE/PE-g-MA (80/20/5) shows that the materials adhering to the disk surface increased as a result of the compatibilizer. This situation in the form of adhesive wear decreased the wear by reducing the interaction between the pin and the disk. The studies also show that the compatibilizer positively affects the wear behavior. 1% nanoclay added to the material is observed to reduce the wear. This is due to the fact that the addition of the nanoclay results in a positive effect on the formation of the film layer on the disk surface. It is also observed that the increase in the amount of

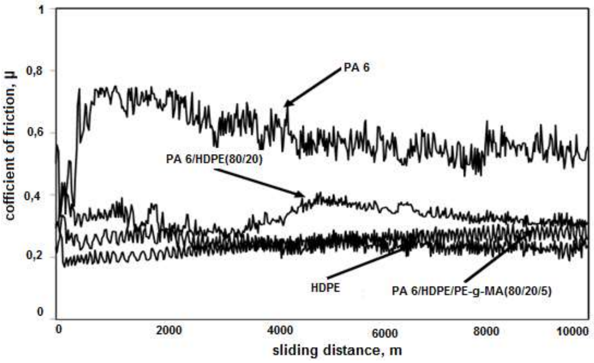



Fig. 8 - Coefficient of friction-sliding distance graph of PA 6, HDPE, PA 6/HDPE (80/20), PA 6/HDPE/ PE-g-MA (80/20/5) materials (load: 80 N, sliding speed: 1 m/s)

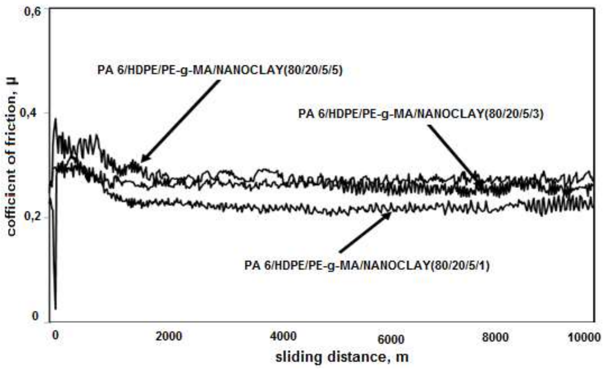



Fig. 9 - Coefficient of friction-sliding distance graph of PA 6/HDPE/PE-g-MA/Nanoclay (80/20/5/1), PA 6/HDPE/PE-g-MA/Nanoclay (80/20/5/3), PA 6/HDPE/PE-g-MA/Nanoclay (80/20/5/5) materials (load: 80 N, sliding speed: 1 m/s).

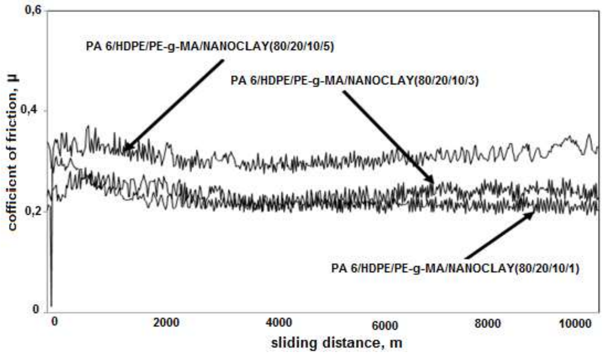
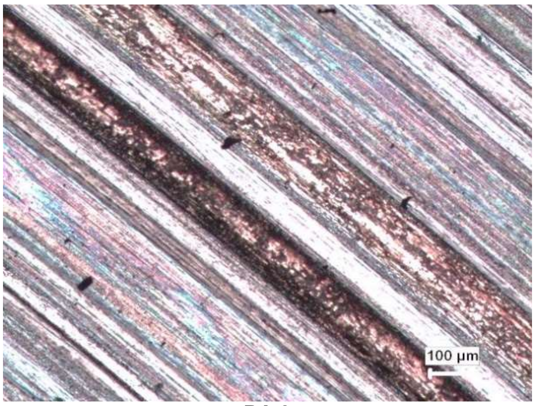
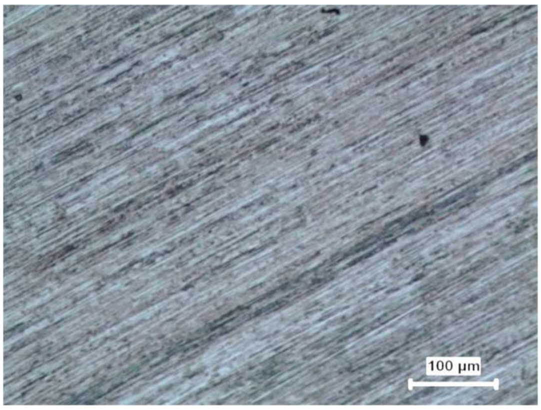



Fig. 10 - Coefficient of friction-sliding distance graph of PA 6/HDPE/PE-g-MA/Nanoclay (80/20/10/1), PA 6/HDPE/PE-g-MA/Nanoclay (80/20/10/3), PA 6/HDPE/PE-g-MA/Nanoclay (80/20/10/5) materials (load: 80 N, sliding speed: 1 m/s).

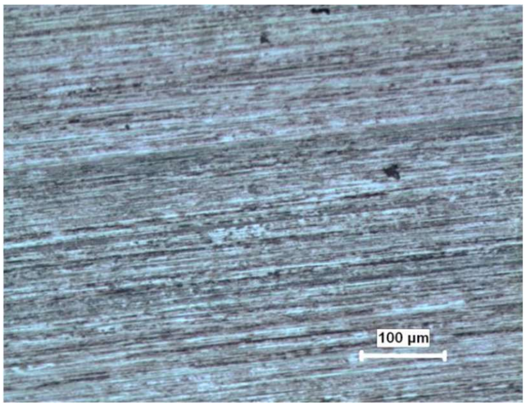
nanoclay addition in the specimens, results in an increase in the wear, and that mainly abrasive wear is operative. This is due to the fact that the nanoclay addition hardened the material and increased the wear by reducing the adhesion. Bhimaraj et al. [20] conducted a study on poly(ethylene) terephthalate (PET) alumina nanoparticles and determined that 1% nanoparticle reduced the wear rate and that the wear rate increased with an increase in the amount of the nanoparticle.



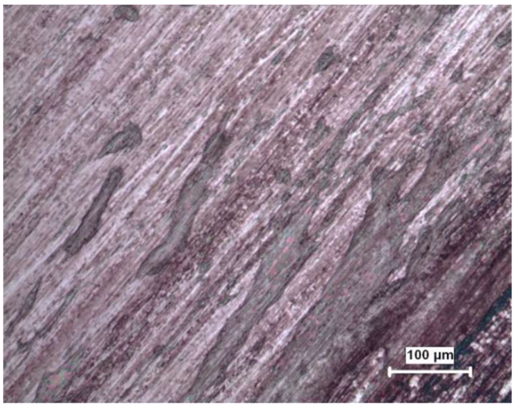

**PA 6**



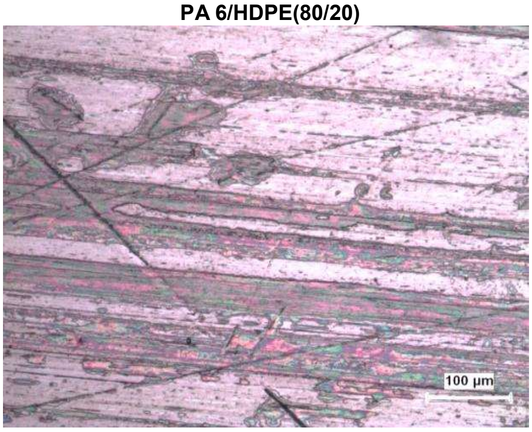

**HDPE**



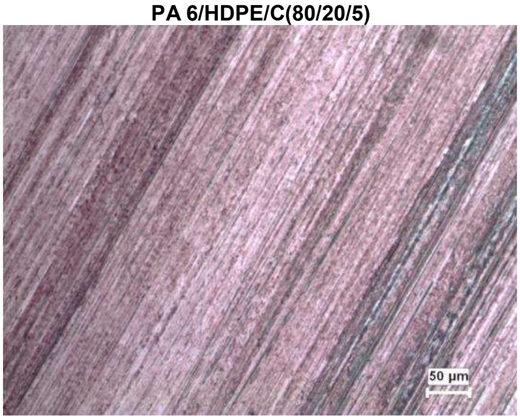

**PA 6/HDPE(80/20)**



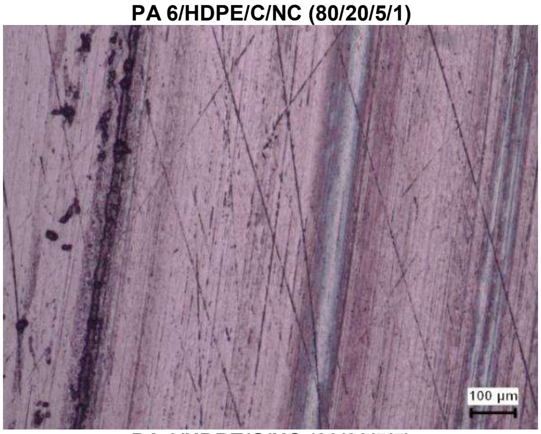

**PA 6/HDPE/C(80/20/5)**



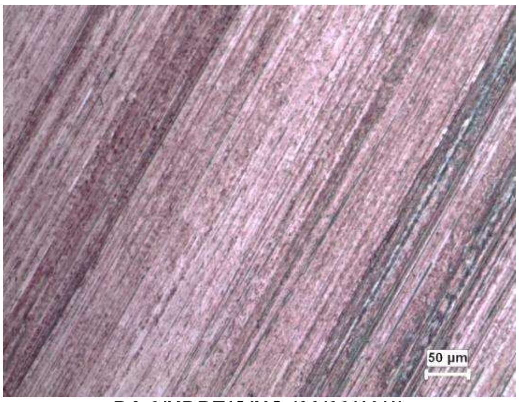

**PA 6/HDPE/C/NC (80/20/5/1)**




**PA 6/HDPE/C/NC (80/20/5/3)**

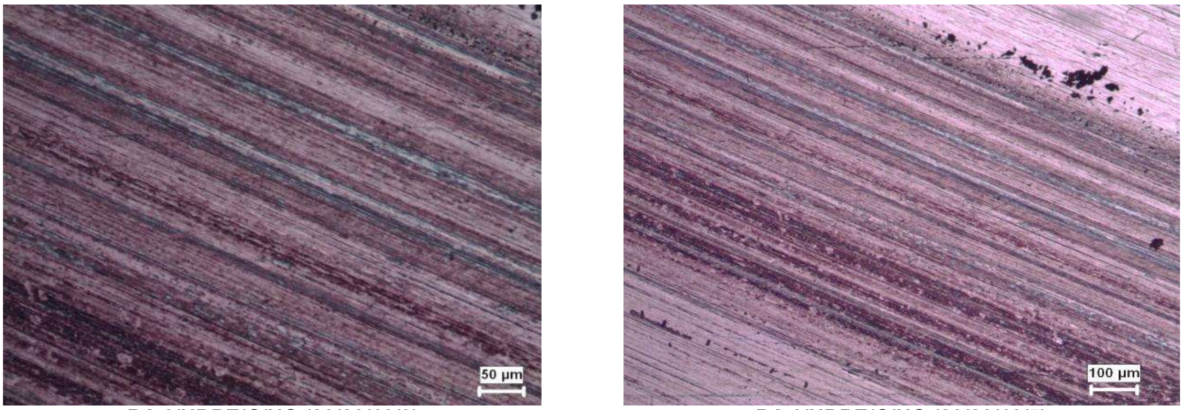



**PA 6/HDPE/C/NC (80/20/5/5)**




**PA 6/HDPE/C/NC (80/20/10/1)**

**Fig. 11 continues on next page**




**PA 6/HDPE/C/NC (80/20/10/3)** **PA 6/HDPE/C/NC (80/20/10/5)**

Fig. 11 - Disk worn surface optic microscopy at 100 N load and 1 m/s sliding speed.

## 4. Conclusions

An experimental study of wear behaviour of PA 6/HDPE composites containing different montmorillonite nanoclay proportions and the compatibilizer at different load values reveals the following conclusions:

- Results of the TEM examination indicates that the nanoclay additions in the matrix material is finely dispersed in the composite. The formation of intercalated and exfoliated dispersions is observed in the composite.
- The coefficients of friction of the materials decrease with an increase in the load. Addition of 1% nanoclay into the composite decreased the coefficient of friction values. However, the increase in the amount of nanoclay in the structure increases the coefficient of friction. 5% compatibilizer added to the structure results in a decrease in the coefficient of friction compared to those of the pure materials. However, increasing the amount of the compatibilizer content to 10% results in an increase in the coefficient of friction.
- The wear rates of all the materials decrease with an increase in the applied load. The wear rate-load relationship with the addition of the compatibilizer indicates that 5% compatibilizer content decreases the wear rate. Increasing the amount of the compatibilizer content to 10% results in an increase in the wear rate. The composites formed by the addition of 1% nanoclay reveal the best results in terms of the wear rate-load relationship. However, the increase in the amount of nanoclay in the composite results in an increase in the wear rate.
- The compatibilizer and the nanoclay added to the material make the coefficient of friction more stable at a shorter distance along the sliding distance.
- The disk surfaces on which wear occurs indicate that the adhesive wear increases with the addition of the compatibilizer into the structure. It is determined that the increase in the amount of the nanoclay leads to a reduction in the attractive force between the polymer chains, and the wear increases and occurs in an abrasive form.

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