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Experimental design in the study of wear properties of polymer composites

Nurlan Gurbanov[1], Kamala Ismayilova[1], Mustafa Yunus Askin[2]

[1] Department of Mechanical and Materials Science Engineering, Azerbaijan State Oil and Industry University, Azerbaijan
[2] Materials Research and Development Center, Iron & Steel Institute, Karabuk University, Turkey

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This study consists of two stages. In the first stage, the wear properties of HDPE (high density polyethylene) matrix composites reinforced with carbon fiber (CF) and basalt fiber (BF) at different ratios were investigated in artificial body fluid in accordance with ASTM G33 standard; in the second stage, the Taguchi method was used for the experimental design of wear tests of the polymer composites. It was established that the composite samples reinforced with 10% CF and 10% BF had the best wear performance especially under low loads (10N and 30N). Although the combination of CF and BF with PE-g-MA (maleic anhydride) (10CF/3MA and 20BF/6MA) provided certain improvements in wear performance, it was observed that wear rate increased as the load increased. It was noted that the wear rates for polymer composite samples increased depending on the concentration of fibers and the increase in normal load.
Keywords: HDPE, carbon fiber, basalt fiber, adhesive wear, experimental design

Експериментальне проектування з вивчення зносостійкості полімерних композиційних матеріалів. *Н. Гурбанов, К. Ісмаілова, М. Ю. Ашкін*

Це дослідження складається з двох етапів. На першому етапі властивості зносу композитів на основі матриці ПВП (поліетилен високої щільності), армованих вуглецевим волокном (УВ) і базальтовим волокном (БВ) у різних співвідношеннях, досліджувалися в штучній рідині організму у відповідності зі стандартом ASTM G33, а на другому етапі метод Тагучі використовувався для експериментів. Під час випробувань на зношування було зазначено, що зразки композитів, армовані 10% УВ і 10% БВ, мали найкращі характеристики зношування, особливо при низьких навантаженнях (10 Н і 30 Н). Хоча поєднання УВ і БВ з PE-g-MA (малеїновим ангідридом) (10 УВ/3МА і 20 БВ/6МА) забезпечувало певні поліпшення характеристик зносу, було зазначено, що швидкість зносу збільшувалася зі збільшенням навантаження. Під час експериментального проектування було зазначено, що швидкість зношування зразків полімерних композитів збільшувалася залежно від концентрації волокон та збільшення нормального навантаження.

1. Introduction

The wide range of applications of polymer composites is due to the fact that these materials allow the creation of durable and high-performance products that can be adapted to various industrial needs. Therefore, polymer composites have become an indispensable material in modern engineering and design applications. Recent scientific studies on the mechanical and tribological properties of these materials and their potential for industrial scale applications have yielded important results [1-3].

Zum et al. stated that the friction and wear properties of fiber-reinforced polymer matrix composites largely depend on the properties of the components (matrix and fiber) that make up the polymer composites and the properties

of the interface formed between them. These properties are related to the type of polymer matrix and reinforcement material, the distribution of the reinforcement element in the structure, the shape of the fiber, the volume fraction of the fiber, the thickness of the fiber, the orientation of the fiber in the matrix, the properties of the counter surface material and the hardness of the reinforcement material relative to the matrix [4] .

Suresha et al. investigated the properties of various end grain fibre reinforced polymer matrix composites (FRPMC) under high load dry wear, adhesion wear, material transfer and fatigue conditions. They observed that the wear of FRPMC depends on the sliding speed, normal loads and sliding path. At the same time, it is claimed that wear is reduced due to surface softening in composite samples [5].

Zhang et al. studied wear properties of various fillers such as short carbon fiber, graphite, poly-tetrafluoroethylene and nano-TiO_2 in polyethylene matrix on a block ring device. It was noted that that short carbon fibers used as reinforcement material had a positive effect on the wear resistance of composite materials [6].

Dhand et al., studied basalt fiber reinforced polymer composites. They argued that the ratio of fiber in the composites does not improve the impact resistance of the composites, but their wear resistance increased [7].

Akinci et al. investigated the abrasion wear of LDPE matrix composite samples reinforced with different proportions of basalt fiber under three different loads. It was noted that the samples of the composite reinforced with 30% basalt fiber had a 90% lower wear rate under a load of 20 N compared to pure LDPE [8].

Gogoleva et al. investigated the existing tribological properties of ultra-high molecular weight polyethylene (UHMWPE) composites of basalt fiber reinforcement material. It was established that the wear resistance of composites reinforced with 5% basalt was approximately 7.7 times higher than pure UHMWPE [9].

Hanchi and Eiss reported that the samples of polyetheretherketone (PEEK) matrix composite reinforced with 30% carbon fiber had better wear performance compared to pure PEEK matrix composites [10].

In the study reported by Sclippa and Piekarski, it was observed that composite samples containing 20% carbon fiber by weight had higher wear resistance than pure high density polyethylene (HDPE) matrix composites. However, the test was carried out under only one load and the carbon ratio was kept constant [11].

Kishore et al. presented SEM images of wear surfaces of glass fiber reinforced polymer composites under dry sliding wear. They found that in polymer composites, delamination occurs at the matrix-fiber interfaces with increasing sliding distance [12].

Rao et al. investigated the wear behavior of natural fiber reinforced polymer composites in a pin-on-disc device using the Taguchi method according to the L9 orthogonal array [13].

Petrokovets et al. found that the friction coefficient of polymer composites depends on wear conditions [14].

There are a number of works devoted to increasing the wear resistance of polymer and polymer matrix composite materials and to reduce the friction coefficient values [7,9,15,16]. Although polymer matrix composite materials provide lower strength compared to metal matrix composites, they are widely used today. The main reason for this is that polymer composites are lighter than metal composites [2, 17-19].

For the last thirty years, the tribological behaviors of polymers and polymer composites have been intensively investigated experimentally and theoretically. Depending on the experimental conditions and target material properties, different trends in wear are observed. During experimental studies it was established that the wear resistance of polymer matrix composite materials is 2-3 times lower than that of metals [13, 18-21].

Considering the importance of composite materials in industrial applications, determining their wear resistance is of great importance. The main purpose of this study is to determine the wear rates of polymer composites in different concentrations in artificial body fluids using experimental and factorial methods and to create a scientific database on this topic.

2. Wear mechanisms of polymer composites and experimental design

Wear is defined as damage that occurs as a result of mechanical interaction between two surfaces that move relative to each other. Wear is the loss of material as a result of physical and chemical changes occurring under the influence of external factors on contact surfaces that cannot be restored [22]. The literature describes various wear mechanisms such as ad-

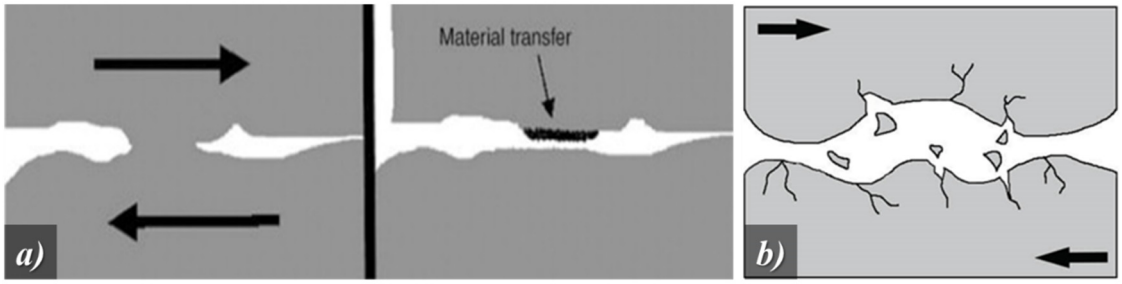

Fig. 1. Wear mechanisms occurring in polymer composites; a) adhesive wear, b) abrasive wear [24].

hesion, abrasion, corrosion and surface fatigue occurring in different materials [5,16,22].

The wear mechanisms commonly observed in polymer and polymer matrix composites are adhesive and abrasive wear [23]. Adhesive and abrasive wear modes occur during plastic contact. In cases where there is plastic contact between similar materials, wear of adhesive bonds occurs at the contact boundary. In cases where fracture is expected to occur as a result of strong adhesion at the contact interface, the resulting wear is defined as "adhesive wear" [22-24]. In abrasive wear, material is removed or displaced from a surface by hard particles (Figure 1) [23,24].

The tribological properties of polymer composites seem to be quite complex, since they depend on the properties of polymer material and the counter surface, the type and ratio of the reinforcement element added to the matrix material, as well as the process conditions such as the applied load, sliding speed, sliding distance and environmental conditions [4,25]. To increase the service life of polymeric materials and polymer composites, it is very important to correctly select a pair of materials that correspond to the operating conditions [26]. In tribology studies, the most suitable polymer-polymer or polymer-metal material combination should be selected in order to reduce the friction coefficient and increase the wear resistance [24,26].

The fact that two basic mechanisms of friction (adhesion/shearing and deformation) can act simultaneously on the surface of polymer composite materials is visually shown in Figure 2. As can be seen from the figure, when the "hard protrusion" is pressed onto the polymer surface, both a "Shearing zone" (a thin layer in which the adhesion and shear effects at the interface are concentrated) and a "Deformation zone" (an area in which the material is volumetrically crumpled or deformed) are simultaneously formed on the surface (Figure 2) [22,23,27].

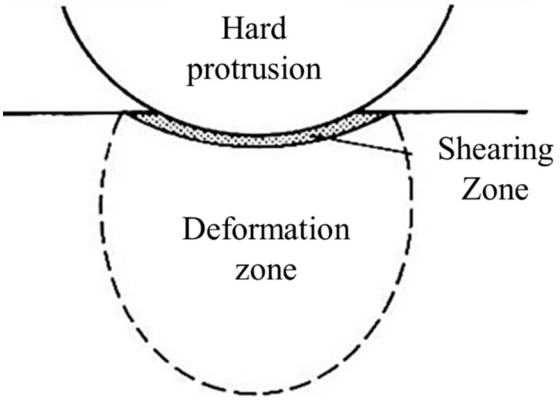

Fig. 2. Schematic representation of friction distribution regions in polymer composites.

Detection of complex tribological changes in polymer composites is a critical issue for maintaining the material structure and selecting suitable materials under operating conditions. This formal modeling provides an efficient and systematic approach. Experimental design methods provide important information for optimizing costs, collecting systematic data, and obtaining reliable results [28]. Factorial maintenance, the Taguchi method and Response Surface Methodology (RSM) solutions are frequently used in wear performance analysis. The method to be selected depends on the complexity of the system and the details of the parts examined. In the literature, the Taguchi method is available as an effective, systematic and economical method for determining wear rates of polymer composites [29,30]. This method is a critical tool in understanding the tribological assembly of materials and making the most appropriate material selection.

3. Materials and methods

3.1. Materials

In the composites, HDPE with a density of 0.956 g/cm^3 was used as a matrix material, and carbon coated with 1.5-3% polyurethane-based resin and chopped basalt fibers with a diameter

Table 1. Compositions of samples used in the study.

Coding of composite samples	Compositions of samples (wt%)	Compatibilizer Composition (wt%)
1.	100 HDPE	-
2.	10 CF/90 HDPE	-
3.	20 CF/80 HDPE	-
4.	30 CF /70 HDPE	-
5.	10 CF /87 HDPE	3 PE-g-MA
6.	20 CF /74 HDPE	6 PE-g-MA
7.	30 CF /61 HDPE	9 PE-g-MA
8.	10 BF /90 HDPE	-
9.	20 BF /80 HDPE	-
10.	30 BF /70 HDPE	-
11.	10 BF /87 HDPE	3 PE-g-MA
12.	20 BF /74 HDPE	6 PE-g-MA
13.	30 BF /61 HDPE	9 PE-g-MA

of 13-20 μm were used as reinforcing materials. Polyethylene-graft-maleic anhydride (PE-g-MA) supplied by Xcar was used to ensure adhesion between the polymer matrix element and the reinforcement phases.

3.2. Method of Manufacturing Composites

Polymer matrix composite samples were produced by melt mixing technique using a co-rotating twin-screw extruder with a screw diameter of D=16 mm and a length/diameter (L/D) ratio of 40 in accordance with the compositions given in Table 1. Melt mixing method is one of the simple and common methods used in the synthesis of polymer matrix composites [31]. Twin-screw extruder prevents the re-accumulation of reinforcement materials in the polymer matrix and ensures homogeneous distribution of fibers in the matrix. The die pressure of the used machine was set as 6.7 bar and the temperature profile of the extruder lower zone during the production phase was set as 50, 200, 205, 210, 215 and 220 °C from the feeding zone to the heating zones, respectively. The extruded material was removed from the die in the form of rods, passed through a water bath at room temperature and granulated using a granulator. Then, it was dried in an oven at 80 °C for 12 hours. The granules were prepared using a 12 ml capacity micro injection molding device.

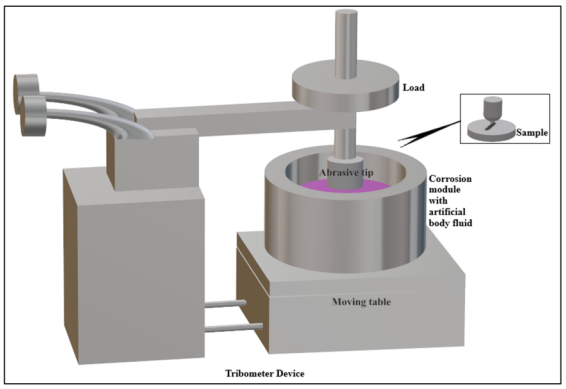

Fig. 3. Schematic view of the wear testing device.

2.3. Wear and Structural characterization

2.3.1. Wear

The wear tests of the composite samples were carried out in a UTS brand wear tester with linear reciprocating motion type in accordance with the ASTM G33 standard in artificial body fluid. The schematic view of the wear test is given in Figure 3. The wear test was carried out using 6 mm diameter AISI52100 steel balls. The samples were subjected to a total sliding distance of 100 m under different loads (10 N, 30 N, 50 N) with a stroke distance of 20 mm in each cycle. Artificial body fluid (Hank's salt solution containing phenol red) was used to carry out the wear tests of the composite samples in accordance with the ASTM G33 standard.

2.3.2. Structural characterization

A ZEISS brand EVO 40 model scanning electron microscope (SEM) was used to examine the distribution of matrix and fiber elements within the structures of composite samples produced for wear tests. A NIKON brand shuttle pix portable digital stereo microscope was used to examine the wear marks on the sample surfaces after the wear tests performed on polymer composites in artificial body fluid.

2.3.3. Theoretical design of wear experiments

In any experiment, standard approaches have been developed regarding the factors affecting the final value and the number of levels of these factors. Experimental design is a useful method that allows finding optimum values even in complex systems with a large number of variables. In a matched design, the effects of the main factors and the interactions between different factors can be estimated with a high level of accuracy. In a perfectly matched design, each level of each factor is matched with

Table 2. Factors and Levels for L16 Fully Matched Experimental Design.

Symbol	Factor/parameter	Level 1	Level 2
A	Fiber concentration (%)	10	30
B	PE-g-MA (% of fiber content)	0	9
C	Normal load (N)	10	50
D	Slining distance (m)	100	100

Table 3. Wear test results of polymer composites.

Coding of composite samples	Normal Loads			Slining Distance (m)
	10N	30N	50N	
1.	0.0167	0.1163	0.3127	100
2.	0.0148	0.0377	0.0632	100
3.	0.0056	0.083	0.24	100
4.	0.0079	0.0572	0.2171	100
5.	0.0091	0.1126	0.2212	100
6.	0.0052	0.0727	13.21	100
7.	0.0025	0.0654	0.1889	100
8.	0.0138	0.1152	0.1969	100
9.	0.01027	0.0741	0.2124	100
10.	0.0077	0.0643	0.1903	100
11.	0.0018	0.0783	0.2907	100
12.	0.0026	0.0673	0.2317	100
13.	0.0016	0.0599	0.3114	100

the levels of the other factors. A commonly used design is the experimental design in which each input factor has two levels. In such a design, the levels are called 'high' and 'low' or '+1' and '-1' (or '2' and '1'). If there are k factors with both levels, the perfectly matched design will have $2k$ cycles. In this study, the 2-level 4-factor (L16) perfectly matched experimental design method was used to determine the effects of the factors affecting the tribological properties and the interactions between the factors. The criterion used in the experimental design is the "lowest best criterion" given in formula (1).

$$\frac{S}{N} = -10 log \frac{1}{n}\left(\sum y^2\right) \quad (1)$$

Here, S/N is the signal-to-noise ratio, n is the total number of observations, and y is the characteristic of interest. The factors for the L16 experimental design and their corresponding levels are given in Table 2.

4. Results and Discussion

The wear tests of HDPE matrix composite samples reinforced with CF and BF at different concentrations in artificial body fluid was carried out under three different loads (10 N, 30 N and 50 N) in accordance with ASTM G33 standard. The wear volume loss (W_r) of polymer composites was calculated by multiplying the wear surface area of the samples by the wear depth. The worn surface area of the composite samples was determined using a profile projector device (Nikon, V-20B). The wear rates (W_V) of the polymer composite samples were calculated according to the formula (2):

$$W_r = \left(\frac{W_V}{dL}\right) = \frac{\left(\frac{2abc}{3}\right)}{d} \quad (2)$$

where W_r is the wear rate of composite samples $\left[mm^3/m\right]$, W_V is the volume loss in composite samples $\left[mm^3\right]$, d is the sliding distance of samples $\left[m\right]$, b is the wear depth of composite samples $\left[mm\right]$, a is the wear width of composite samples $\left[mm\right]$, and c is the stroke $\left[mm\right]$.

The wear test results of HDPE matrix composite samples with different fiber concentrations, calculated according to Formula 2, are given in Table 3, and the graph is given in Figure 4.

From the graph in Figure 4, it is determined that the wear rates of the samples prepared from pure HDPE are 0.0167 mm^3/m, 0.1163 mm^3/m and 0.3127 mm^3/m under the loads of

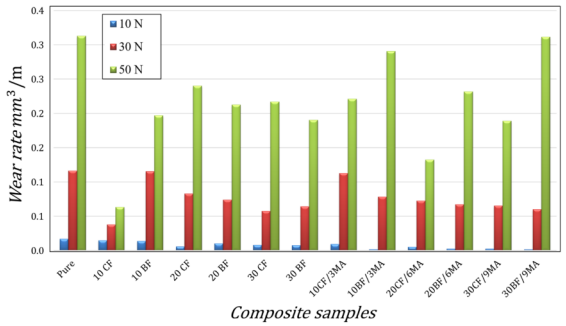

Fig. 4. Wear rates of polymer composite samples with different fiber concentrations.

10N, 30N and 50N, respectively. The lowest wear rate in pure HDPE samples was observed under the load of 10N. However, a significant increase in the wear rate was recorded as the load increased.

It was established that polymer composites reinforced with CF and BF at different concentrations (10%, 20% and 30%) provided positive effects on wear rates when compared to pure HDPE (high density polyethylene) in wear tests performed under different loads (10N, 30N and 50N) in simulated body fluid. Indeed, in wear tests performed on composite samples with different fiber concentrations, it was determined that the best wear performance under loads of 10 N and 30 N was obtained in samples reinforced with 10% CF and 10% BF. However, the best wear resistance at a load of 50 N was determined for composites reinforced with 20% CF.

The interface bond compatibility between the matrix and fiber significantly affects the wear resistance of the composite. In the selection of matrix and fiber, the compatibility between them is an important factor that determines the strength and durability. The bond strength of the compatible interface directly affects the strength of the composite [4,32].

The effect of PE-g-MA compatibilizer at different concentrations (3%, 6% and 9%) on enhancing the adhesion between HDPE and fibers on the wear rate of polymer composite samples was studied; and it was found that the combination of CV and BP with PE-g-MA has a positive effect on wear resistance, especially at low loads (10 N and 30 N). However, when the load increased to 50N, the effect of PE-g-MA compatibilizer on polymer composite samples decreased and the wear rates increased.

In the wear experiments carried out on polymer composite samples, a regular increase was observed in the wear rates of all samples in parallel with increasing load values. In addition, polymer composite samples exhibited lower wear rates compared to pure HDPE, indicating that fiber reinforcement increased the wear resistance. Especially at low loads (10% CF and 10% BF), polymer composite samples exhibited superior wear resistance and it was observed that determining the appropriate fiber concentration was of critical importance.

Table 3. Parameters and levels of the L16 fully matched design for CF and BF.

Exp No.	Parameters and Levels			
	A	B	C	D
1.	1	1	1	1
2.	1	1	1	2
3.	1	1	2	1
4.	1	1	2	2
5.	1	2	1	1
6.	1	2	1	2
7.	1	2	2	1
8.	1	2	2	2
9.	2	1	1	1
10.	2	1	1	2
11.	2	1	2	1
12.	2	1	2	2
13.	2	2	1	1
14.	2	2	1	2
15.	2	2	2	1
16	2	2	2	2

In polymer composites, the type, size and shape of the reinforcing material and the applied chemical treatments play an important role in the mechanical and tribological properties of the composites [32]. For example, short fiber lengths reduce their load-bearing capacity and lead to rapid wear, while fibers that are too long can lead to fiber pullout and cause even more wear [2,4,33]. Therefore, the use of fibers below the critical length can negatively affect the stress transfer efficiency [32-34]. The shearing effect during the melt mixing process resulted in the formation of shorter fibres, and this was confirmed by the SEM images of the cryofractured surfaces (Figure 7).

In order to determine the factors affecting the tribological properties and the interactions between the factors during the experimental studies of HDPE matrix composite samples with different fiber concentrations, the 2-level 4-factor (L16) fully matched experimental design method was used and the results were examined according to formula 1. The parameters and levels of the L16 fully matched design for HDPE matrix composite samples with different

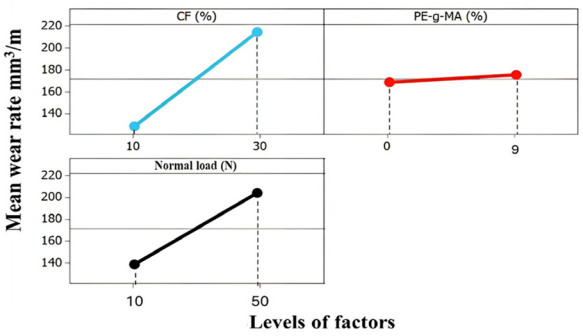

Fig. 5. Main effects for carbon fiber reinforced composites.

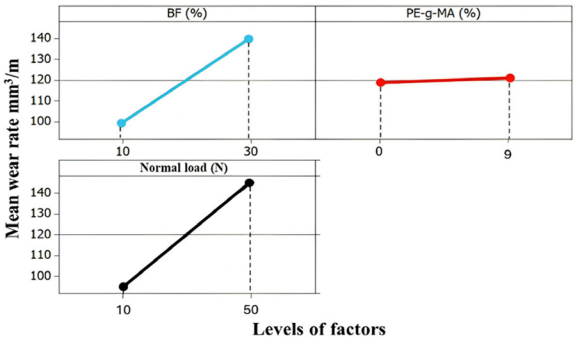

Fig. 6. Main effects for basalt fiber reinforced composites

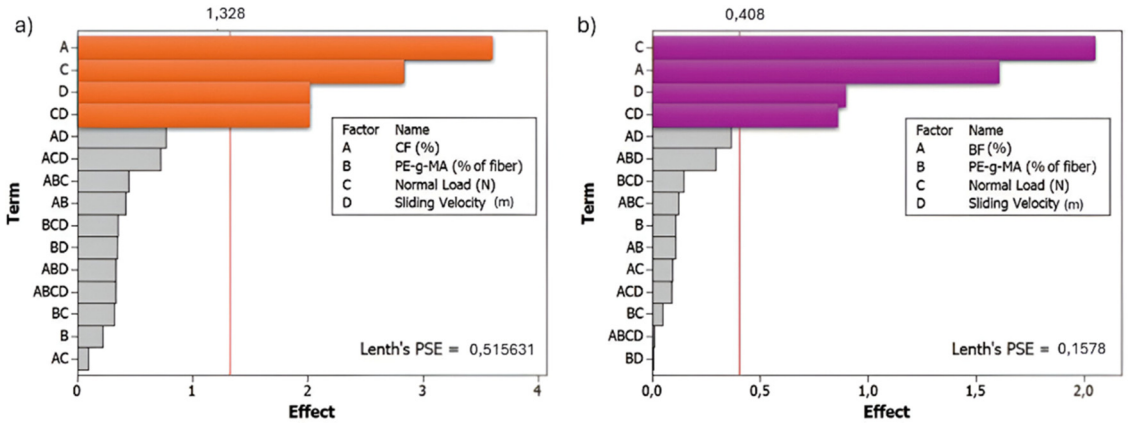

Fig. 7. Optical microscope images of wear surfaces of polymer composite samples reinforced with a) 10 CF, b) 10 CF/3 MA, c) 20 CF, d) 20 CF/6 MA, e) 30 CF, f) 30 CF/9 MA.

KF and BF concentrations are summarized in Table 3.

The main effect graphs for wear rates in Figures 5 and 6 show the relative importance of the effects of various parameters. Figure 5 shows the relationship between the average wear rate of carbon fiber reinforced polymer composites and different factor levels. A significant increase in the average wear rate is observed when the carbon fiber (CF) ratio increases from 10% to 30%. This suggests that the increased carbon fiber content may reduce the wear resistance or cause higher wear under certain conditions. At the same time, a very small increase in the wear rate is observed as the ratio of the compatibilizer (PE-g-MA) increases. This minimal change may indicate that PE-g-MA does not have a direct significant effect on wear. When the normal load is increased from 10 N to 50 N, the average wear rate increases significantly. This is in line with the general wear theory that higher loads cause higher wear.

The relationship between the average wear rate and different factor levels for basalt fiber reinforced composite samples is given in Figure 6. A significant increase in the wear rate is observed with an increase in the BF ratio from 10% to 30%. This shows that in order to improve the wear resistance of the material, it is necessary to carefully select the fiber ratio. It is seen that PE-g-MA used as compatibilizer has a very low effect on the wear rates of the composite samples. This minimal change indicates that the effect of PE-g-MA on the wear behavior may be limited. At the same time, when the load applied to the composite samples is increased from 10 N to 50 N, a significant increase in the average wear rate occurs. This result is consistent with the wear theory; higher loads cause more friction on the material surface and thus more wear. The wear rate increases significantly as the applied load increases.

In interaction graphs, all possible interaction combinations are considered. Parallel lines (not intersecting) lead to the conclusion that there is no interaction.

Optical microscope images of damaged surfaces of polymer matrix composite samples after abrasion tests in body fluids are given in Figures 7 and 8. Wear marks formed after the abrasion tests of HDPE matrix composite materials reinforced with different ratios of CF

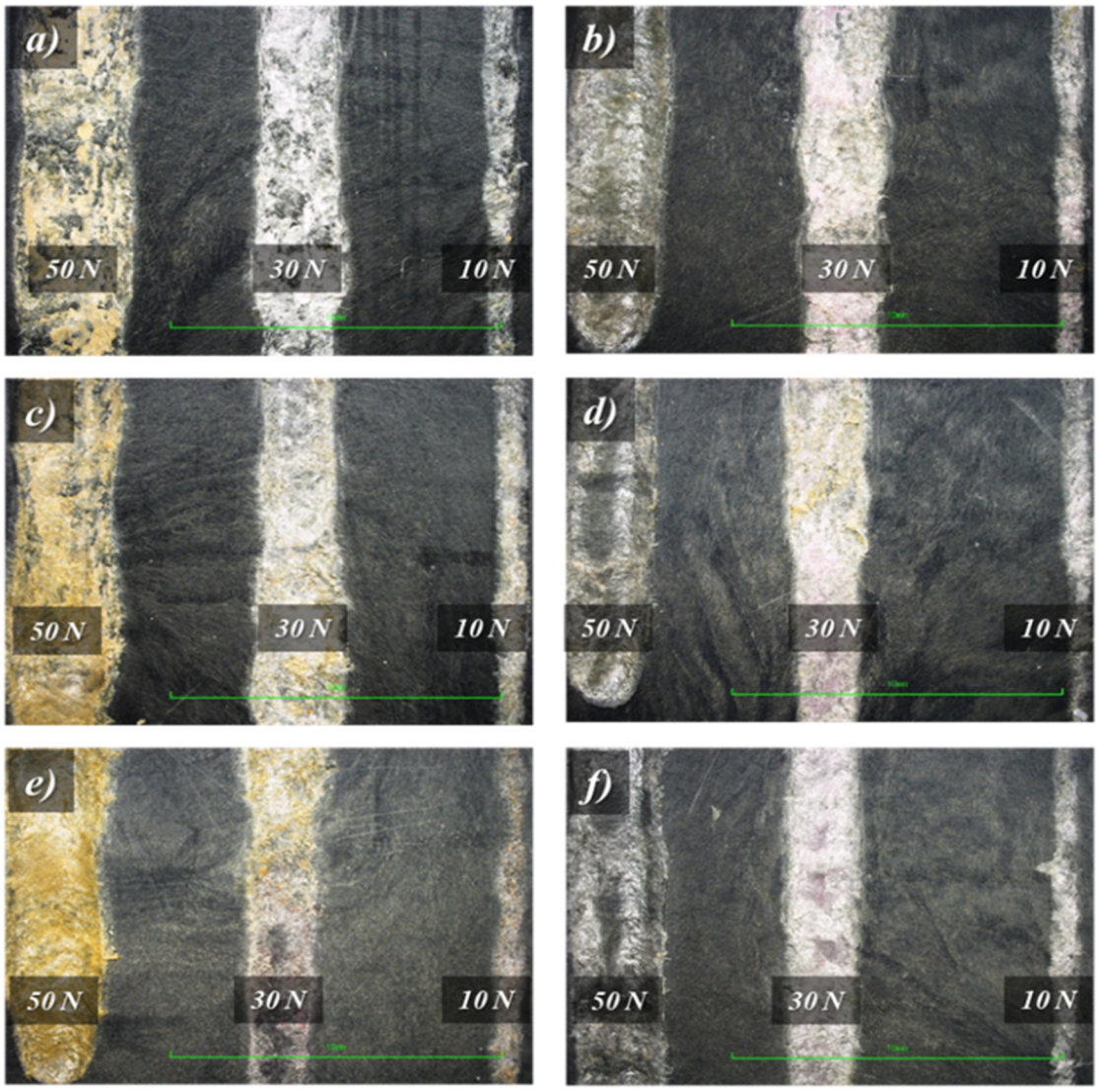

Fig. 8. Optical microscope images of wear surfaces of polymer composite samples reinforced with a) 10 BF, b) 10 BF/3 MA, c) 20 BF, d) 20 BF/6 MA, e) 30 BF, f) 30 BF/9 MA.

and BF under 10N, 30N and 50N loads in accordance with ASTM G33 standard indicate that adhesive and abrasive wear mechanisms are generally dominant in composite samples (Figures 7-8).

Some sources show that the wear phenomenon can be investigated by the appearance of the trace formed by wear. Images of these marks may show indentations, peeling, scratches, polishing, gouges, nicks and scratches. [35,36]. The wear phenomenon can also be examined from the point of view of the physical mechanisms by which damage occurs [35,37,38].

The wear marks of polymer composite materials shown in Figures 7 and 8 demonstrate differences in the sizes of the wear marks formed in the samples depending on the amounts of different applied loads and the fixed sliding distance. When comparing, it is evident that that the wear marks formed in the samples without compatibilizer (Figure 7 a,c,e and Figure 8 a,c,e) are more pronounced depending on the applied loads.

The mechanical and tribological properties of polymer composite materials depend on the interfacial interaction between the matrix and fiber (reinforcement) [4,39]. This interfacial interaction can be improved by surface treatment of the fibers [40] or by adding compatibilizer to the matrix [41]. Based on the positive reports of previous studies on polymer composites in the literature [39-43], in this study, PE-g-MA compatibilizer was used at different ratios to improve the adhesion between the HDPE matrix material and carbon and basalt fiber reinforcing elements in composite structures. It was also

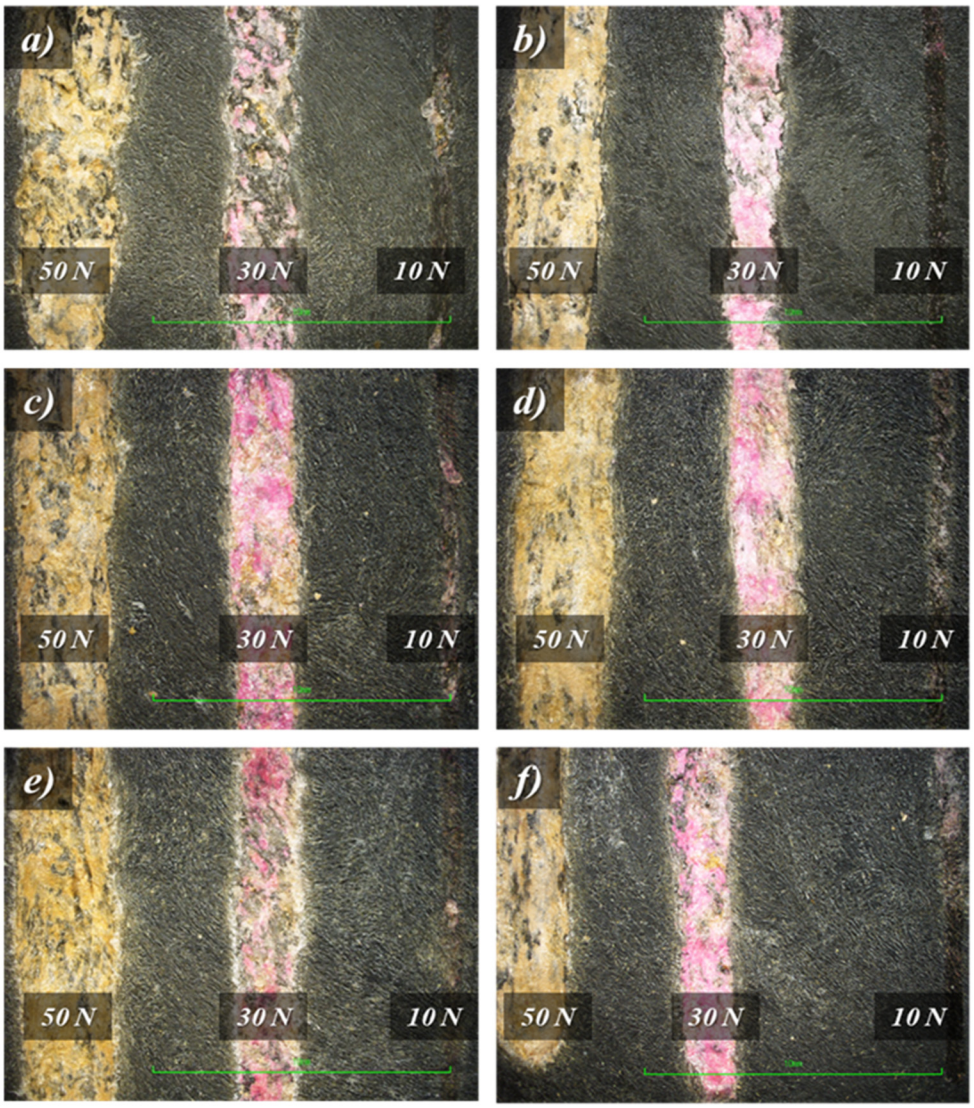

Fig. 9. SEM images of polymer composite samples reinforced with a) 20 CF, b) 20 CF/6 MA, c) 20 BF, d) 20 BF/6 MA.

observed after wear tests that the compatibilizer material significantly affected the adhesion between the fiber-matrix interfaces in the composite structures (Figure 7-8). In their study, Lu et al. modified wood fiber/HDPE composites using PE-g-MA compatibilizer. SEM analysis of unmodified composite samples showed that the wood fiber was mainly physically attached to the thermoplastic matrix, whereas for MAPE-reinforced composites, chemical bonds were observed at the interface [44]. The response of polymer matrix composite materials to external loading is extremely complex [39]. Wear damage in composite specimens caused by static loads consists of matrix deformation, matrix cracking and fiber damage (Figures 7-8).

For a better interpretation of the tribological properties of polymer composites, Figure 9 shows SEM images of composite samples of medium concentration. The SEM images showed that the carbon and basalt fiber reinforcements were unevenly distributed throughout the HDPE matrix and agglomerated. The fluctuations in the wear rates of the polymer composites may be due to this situation.

It was noted that the selection of the correct component ratios in polymer composites provides the most suitable image separation characteristics for determining the wear resistance of samples. In general, the load applied during mechanical and wear testing of fiber-reinforced polymer composites is transferred to the fibers through the fiber-matrix assembly. Therefore, their structure and properties have an important influence on the composite material mechanics, tribological and physical properties. In

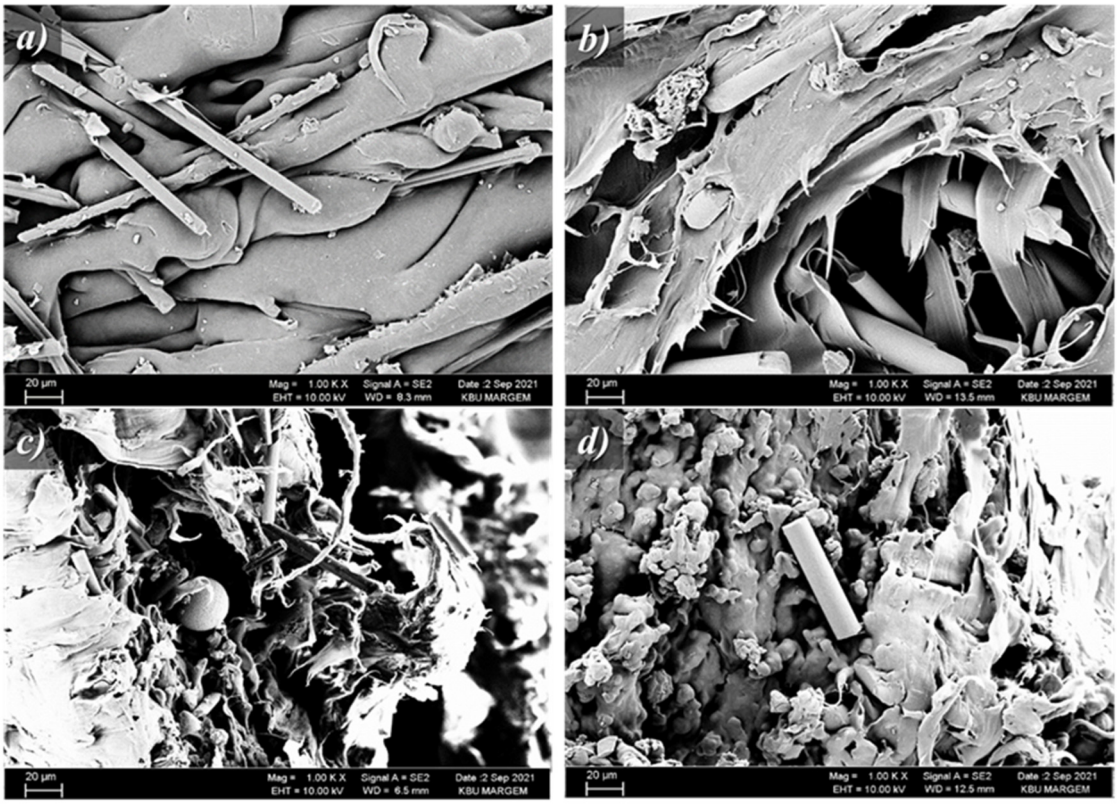

Fig. 10. SEM images of a) 20 CF, b) 20 CF/6 MA, c) 20 BF, d) 20 BF/6 MA reinforced polymer composite samples.

particular, the breaking strength is an important parameter that controls the strength and hardness of the composite material. To obtain a composite with good mechanical and tribological strength and to make the most efficient use of the fibre, an optimum standard must be achieved throughout the material.

Conclusions

HDPE matrix composite samples with different fiber concentrations were subjected to abrasion tests in accordance with ASTM G33 standard in artificial body fluid and the following results were obtained.

During the abrasion tests, it was found that composite samples reinforced with 10% CF and 10% BF exhibited the best abrasion resistance especially at low loads (10N and 30N).

A significant effect of PE-g-MA additive used as compatibilizer in polymer composites on the abrasion resistance of the samples was observed.

As a result, the fiber ratio and loading in polymer composites act as factors that significantly influence the abrasive behavior of the samples, and these parameters should be carefully controlled to achieve optimal abrasion resistance.

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