Full-factorial Experimental Design for the Investigation of Tribological Properties of PP / TiO₂-Clay Composites

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Abstract

In the current study, titanium dioxide (TiO₂) and clay reinforced polypropylene (PP) composites were produced by using melt compounding method in a twin-screw extrusion machine. Composite pellets were then subjected to injection moulding by using a microinjection moulder. Tribological properties of the composites were investigated using a ball-on-disc tester. Tribo-tests were performed at ambient temperature and dry sliding conditions. Full-factorial experimental design was used to find the main factors on the tribological characteristics of the samples. Filler type, filler content, applied load and turning speed were studied as main factors. It was seen that the wear rate increases, and the coefficient of friction (CoF) decreases by increasing the load and turning speed. The wear rate and the CoF decreases by using clay instead of TiO₂. 5% (wt) was found to be optimum clay concentration for minimizing the wear rate and CoF.

Keywords: Polypropylene, Titanium dioxide, Clay, Wear, Experimental design.

PP / TiO₂-Kil Kompozitlerin Tribolojik Özelliklerinin Tam-Eşlendirmeli Deney Tasarımı ile Araştırılması

Öz

Bu çalışmada, titanyum dioksit (TiO₂) ve kil katkılı polipropilen (PP) kompozitler çift vidalı bir ekstrüzyon makinesi kullanılarak ergiyik harmanlama yöntemi ile üretilmişlerdir. Kompozit peletler ardından bir mikro-enjeksiyon kalıplama makinesi kullanılarak enjeksiyon kalıplamaya tabi tutulmuşlardır. Kompozitlerin tribolojik özellikleri bir ball-on-disk test cihazı kullanılarak araştırılmıştır. Tribo-testler oda

sıcaklığında ve kuru kayma şartlarında gerçekleştirilmiştir. Numunelerin tribolojik özellikleri üzerindeki ana faktörleri tespit etmek için tam-eşlendirmeli deneysel tasarım kullanılmıştır. Takviye tipi, takviye içeriği, uygulanan yük ve dönme hızı ana faktörler olarak seçilmiştir. Yük ve dönme hızının artmasıyla aşınma oranının arttığı, sürtünme katsayısının (CoF) ise azaldığı görülmüştür. TiO₂ yerine kil kullanarak, aşınma oranı ve CoF'nin azaldığı tespit edilmiştir. %5 kil miktarının aşınma oranı ve CoF'yi azaltmak için optimum konsantrasyon olduğu tespit edilmiştir.

Anahtar Kelimeler: Polipropilen, Titanyum dioksit, Kil, Aşınma, Deneysel tasarım.

1. Introduction

Polymers are being used as one of the alternatives to metallic materials owing to their high strength/weight ratio and adequate mechanical and wear resistance properties. Nevertheless, polymers have lower strength, wear and creep resistance than metals. The wear resistance properties of polymers can be improved with the incorporation of reinforcing and/or lubricating organic/inorganic fillers. Polypropylene (PP) is vinyl polymer with methyl group $(-H_2C-CRH-)_n$ and characterized by good mechanical and chemical features. PP was selected because of its advantageous properties like good mechanical resistance and being suitable for melt based production methods [1-3]. The montmorillonite clay and titanium dioxide (TiO₂) are the most demanding reinforcements for polymer matrix composites and numerous studies can be reached in the literature [4-6]. They found that the reinforcement phase was a beneficial effect on the mechanical properties of the composite materials in the studies carried out. Aydemir et al. [7] was found that the mechanical characteristics of PP were also improved with the addition of nano-TiO₂ into the PP matrix. In current study, a systematic approach (2⁴ full-factorial design) was conducted for critical parameter determination for wear testing of PP / TiO₂-clay based composites. After that, the wear mechanisms of the test samples were discussed. Following the study, it is hoped that this will pave the way for further works on different properties of PP / TiO₂-clay based composites.

2. Experimental Setup

2.1. Synthesis of PP / TiO₂-Clay Composites

Polypropylene (PP) pellets were from Rabigh R&P Co. (Rabigh, Saudi Arabia) with a MFI (melt flow index) of 1.1 g/10 min (ASTM D1238), density of 0.921 g/cm³ (ASTM D792 Method A) and melting point (DSC) of 122 °C. Natural montmorillonite clay (Nanomer I.44P) surface-modified with dimethyl dialkyl ammonium was supplied by Nanocor Inc., and TiO₂ powder was purchased from Sigma-Aldrich Co. Firstly, PP pellets, TiO₂ and montmorillonite were dried in a low temperature oven at 80 °C for 12 h to remove adsorbing water. Composites were produced by melt blending method using a twin screw (co-rotating) extruder machine (Gulnar, Turkey) (screw dia. $D=16$ mm, and length/dia. (L/D) ratio 40). The temperature zones was controlled at 50 °C, 180 °C, 190 °C, 195 °C, 200 °C and 205 °C, respectively, from the feeding to the mold exit. The first step was to feed the PP pellets into the extruder and then feed the reinforcements downstream into the fully melted polymer to prepare composites. The rod-shaped composite was hardened in a water bath and chopped by an in-situ pelletizing unit. Before injection molding, chopped composites were dried again at 60 °C for overnight. The composite pellets were then injection molded into the cylindrical samples (about 8 mm thick and $\phi 29$ mm in diameter), using a micro-injection moulder (12 ml, Xplore IM 12, The Netherlands). The mold and barrel temperatures and injection pressure were 25 °C, 225 °C and 6 bars, respectively. The molded samples were then polished to about 7.5 mm thick with SiC abrasive papers (up to 2000 mesh). The surface of the polished composite was cleaned with distilled water and dried before tribo-tests.

2.2. Friction and Wear Tests

Tribo-properties of the composites were investigated using a ball-on-disc tester. The composite discs were fitted on a horizontal turntable driven by a motor of variable speed. The counterpart ball was loaded horizontally by adding weights to a cantilever as shown in Fig. 1. This is different from classic pin-on-disc testers where disc was located in a horizontal position. Similar test configurations like used in this study can be found in the literature [8-10]. The foremost advantage of the test rig is reducing the wear debris in

the track that can change the wear mode and resistance [11]. The counterpart used in the wear tests was $\phi 5$ mm Al_2O_3 ball (hardness of 17.2 GPa). The Al_2O_3 ball was chosen because of its high hardness and non-reactive characteristic to the samples used in this work.

Figure 1. Schematic of tribo-tester

The coefficient of friction (CoF) values of the composites were obtained from a load-cell sensor. A digitiser module (Mantracourt DSC-SUEASC) and a software (Instrument Explorer 1-6.8) were used for getting data. The steady-state coefficient of friction (μ_s) was calculated as the average of all CoF values from steady-state point until the end of wear test. Applied normal loads ranged from 2.5 to 5 N, the sliding speeds in the center of the wear track ranged from 0.275 to 0.550 m/s (disc rotations of 500 and 1000 rpm), and the test duration was kept at 10 min. The wear track diameter was 10.5 mm in the tests. Tests were conducted at room temperature (20 ± 2 °C, humidity $28 \pm 2\%$). Firstly, the counterpart balls were cleaned using alcohol and dried carefully. F_f frictional force was recorded during the wear tests. The wear track widths obtained from optical images of the samples were considered as the wear rate values of the composites.

2.3. The Design of Experiment Method

This method is a good way to optimize a complex study, which helps to get data about the optimum levels of different variables. Factorial design is used to find the main effects and interactions of different test parameters. In a full-factorial experimental design analysis each setting of each parameter appears with each setting of each other parameter. Most common test design is one with all input factors set at two levels each. The levels are called +1 and -1 or high and low, respectively. If there are z factors each at two levels, the design has 2^z runs. In this study, two-levels and four-factors, ($2^4 = 16$ tests) full-factorial experimental design was used to determine the tribological properties of the composites. MINITAB software was chosen to analyze the results on the effect of different factors and their interactions on wear track width and CoF using “smaller-the-better” method below. Where, y is the respective characteristic, n is the observation number and S/N is the signal-to-noise ratio.

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

3. Experimental Results

The factors and the levels used in this work are listed in Table 1. All the tests were done in a random order for eliminating the effect of experimental errors on the test results. The results of 2^4 experimental design are listed in Table 2, and coefficient of friction values of selected samples against sliding distance is given in Fig. 2.

Table 1. Levels of the parameters for L16 full-factorial design

Symbol	Factor / Parameter	Level 1	Level 2
A	Normal Load (N)	2.5	5
B	Sliding Speed (rpm)	^a 500	^b 1000
C	Reinforcement	TiO ₂	Montmorillonite
D	Content (wt.%)	1	5

^a corresponding to 0.275 m/s.

^b corresponding to 0.550 m/s.

Figure 2. CoF values of the samples against sliding distance

Optical microscope images of the wear track on different samples are shown in Fig. 3, 4 and 5. Abrasion, adhesion and fatigue (wave formation) are the main wear types seen in polymeric materials [12,13]. Because of the abrasion, all sample worn surfaces showed macroscopic scratches. The stuck wear debris particles were found in the wear tracks despite the vertical style of the tribo-tester. In the case of clay (%5) reinforced PP composite (at 2.5 N and 1000 rpm) fatigue wear mode was also found. Such findings are often seen due to fatigue wear caused by repeated cycles of an asperity of the counter material on the worn surface. Fatigue wear also increases with increasing stress or defect concentration on the sample [12].

Figure 3. Optical microscope image of the wear track on clay (1%) - PP composite, 2.5 N, 1000 rpm

Figure 4. Optical microscope image of the wear track on clay (5%) - PP composite, 2.5 N, 1000 rpm

Table 2. Experimental results

Experiment No	Normal Load (N)	Sliding Speed (rpm)	Reinforcement Type	Content (wt.%)	Wear Track Width (μm)	Coefficient of Friction
1	2,5	1000	Clay	5	897,1	0,202
2	5,0	500	TiO ₂	1	972,7	0,318
3	5,0	1000	Clay	1	1057,7	0,308
4	2,5	1000	Clay	1	894,9	0,328
5	2,5	500	TiO ₂	1	829,0	0,398
6	5,0	1000	Clay	5	967,3	0,245
7	2,5	500	Clay	1	780,5	0,415
8	5,0	1000	TiO ₂	1	1091,5	0,267
9	2,5	1000	TiO ₂	5	867,2	0,307
10	2,5	500	TiO ₂	5	736,7	0,441
11	2,5	1000	TiO ₂	1	934,9	0,295
12	5,0	500	Clay	5	829,5	0,247
13	5,0	500	TiO ₂	5	885,7	0,365
14	2,5	500	Clay	5	737,4	0,336
15	5,0	500	Clay	1	853,2	0,393
16	5,0	1000	TiO ₂	5	1199,6	0,262

Figure 5. Optical microscope image of the wear track on TiO₂ (1%) - PP composite, 2.5 N, 500 rpm

The pareto charts of the effects for wear track width and CoF are given in the Fig. 6. The factors and the interactions that extend past a specific line are accepted as important. The basis of the graphs is outside the goal of current work, but can find more in the reference [14]. From the results, the most important factors on the wear rate are as follows: Sliding speed, normal load, reinforcement and content, respectively.

The factors that have the important effect on the CoF are as follows: Sliding speed, normal load, content, and reinforcement, respectively, as seen in the Fig. 6. Furthermore, interaction of reinforcement and content (CD) was found potentially important for CoF. But sliding speed seems to be most effective factor among them.

The main effects plots for wear track width and CoF are given in the Fig. 7. The main effects plot shows the relative influence of the effects of the factors. The effect's sign shows the directions of the effect. The wear rate increases and the CoF decreases by increasing the sliding speed and the normal load. The reinforcement type and the reinforcement content had the same effect on both the wear track width and the CoF. The wear rate and the CoF decreases by using clay instead of TiO₂. 5% (wt) was found to be optimum clay concentration for minimizing the wear rate and CoF.

Figure 6. Pareto chart of the effects for (a) wear track width and (b) CoF

The interaction effects plots for wear track width and CoF are given in the Fig. 8. This plot gives the mean response of the factors at all combination of the settings. When the lines are not parallel, it shows an interaction between the two factors. From the graphs, as seen in the Fig. 8, no interaction was found between the factors. From the interaction effects plots for CoF, as seen in the Fig. 8, one interaction comes to the front: The reinforcement type and the reinforcement content. In the case of the reinforcement type and the reinforcement content interaction, the concentrations of 1% for TiO_2 and 5% for clay were found to be the optimum selection to minimize the CoF.

This situation is seen as a requirement to obtain the lowest CoF value. When other parameters are examined, it will not be possible to talk about any interaction since there are no intersecting lines. Clay also has a similar structure like graphene nanoplatelet [15] and also boron nitride nanoplatelet [16] and can offer improvements in composite properties.

Figure 7. Main effects plots for (a) wear track width and (b) CoF

Figure 8. Interaction effects plots for (a) wear track width and (b) CoF

4. Conclusions

PP / TiO₂-clay based composites were prepared by melt extrusion method and the effect of different factors on the wear properties of the composites was characterized. The following results were obtained from the study.

1. The important factors on the wear rate are as follows: Sliding speed, normal load, reinforcement, and content, respectively.
2. The factors that have the important effect on the CoF are as follows: Sliding speed, normal load, content, and reinforcement, respectively.
3. The wear rate increases and the CoF decreases by increasing the sliding speed and the normal load.
4. The wear rate and the CoF decreases by using clay instead of TiO₂. 5% (wt) was found to be optimum clay concentration for minimizing the wear rate and CoF.
5. In the case of the reinforcement type and the reinforcement content interaction, the concentrations of 1% for TiO₂ and 5% for clay were found to be the optimum selection to minimize the CoF.

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