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HARDNESS AND TRIBOLOGICAL PROPERTIES OF PMMA COMPOSITE REINFORCED BY HYBRID GRAPHENE AND TiO_2 NANOPARTICLES USED IN DENTAL APPLICATIONS

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ABSTRACT

Due to the PMMA resin has a widespread in dental restorations, the current study focused on assessing the tribological properties of the composite filled with nano-filling. The hybrid nano-filler used in this research is graphene and TiO_2 nanoparticles. Loading filler amount of TiO_2 nanoparticles 0.25, 0.5, and 0.75 wt. %, while graphene adds with amount of 0.5 and 1.0wt. %. The reciprocating tribometer was used to perform the tribological properties of the composites. Moreover, the hardness was evaluated via durometer Shore D. It was found that a good enhancement in the mechanical and tribological properties of PMMA composites. According to experimental results, it can conclude that, PMMA nanocomposites with filler content of 1.0 wt. % of graphene and 0.25 wt. % TiO_2 nanoparticles recorded the best wear resistance performance comparing with pure specimen.

KEYWORDS

PMMA-Composites, Friction, Wear rate, TiO_2 nanoparticles, Graphene.

INTRODUCTION

Recently, interest of the utilization of polymers has increased in several fields, which has made the pursuit of polymers development unstopped. In many fields, reliance on polymers has become a master plan due to its distinct properties. Nanomaterials have always had a prominent role in reducing friction significantly, which reduces fuel consumption and reduces energy losses when used as an additive to engine oils, [1 – 3]. Nanomaterials have always been of outstanding importance in the plastics industry. While fillers lead to targeted development in some mechanical or physical properties of polymers, [4 – 8].

Restorative dental materials can be divided into four directions: metals, polymers, ceramics, and composites. Polymers play a key role in denture base resins. Light curing acrylic resin are one of the common methods of fillings used in dental surgeries, [9 - 11]. The distinct characteristics of Polymethyl Methacrylate (PMMA) make it a suitable alternative material for many applications of biomaterials such as bone cement and dental fillings, [12 - 14]. Polymerization of MMA molecule is used as a hardener in the manufacture of dental resin,

[15]. PMMA is an acrylic resin with good surface and mechanical properties, and it is easy to form but is not sufficient to match the target wear resistance, hardness, and mechanical strength. Based on the foregoing, pure PMMA is not a preferred material for dental surgeries. Therefore, much research focused on studying the effect of dispersion of different nanomaterials in acrylic resin as an attempt to improve the various properties of the resin.

The mechanical and tribological attributes of PMMA reinforced by low content of Al_2O_3 nanoparticles was examined. PMMA filled with 0.2 %, 0.4 %, 0.6 %, 0.8 %, and 1wt. %. of Al_2O_3 nanoparticles were performed. It was found that, PMMA integrated with 0.6wt. %. of Al_2O_3 nanoparticles, had the best compressive yield strength, and higher hardness. Moreover, results indicated that PMMA specimen filled by 0.8 wt.% of Al_2O_3 nanoparticles had higher fracture toughness, [16]. In general, Al_2O_3 nanoparticles is an element with a prominent role in enhancing the compression strength, fracture, and hardness of improving the properties of the PMMA resin, [17 - 18]. PMMA, incorporated with hybrid Al_2O_3 nanowires and ZrO_2 nanoparticles at various filler amount (wt. %) was investigated. Thus, it can be concluded that PMMA resin content up 0.5 wt. % of Al_2O_3 nanowires and 0.7 wt. % of ZrO_2 nanoparticles achieve better mechanical and tribological performance, [19]. Furthermore, set of specimens were evaluated to investigate the effect of adding ZrO_2 with various filler amount to dental base PMMA. It was found that, the surface and mechanical performance for the composite clearly improved, [20 - 23]. The wear resistance of PMMA resin filled by SiO_2 or Al_2O_3 nanoparticles was evaluated with different filler content of 0.1%, 0.3 %, and 0.5 wt. %. The results showed that, specimens filled with Al_2O_3 nanoparticles achieved better results than their counterparts filled with SiO_2 nanoparticles, [24]. Hybrid nano-filler of ZrO_2-Al_2O_3-SiO_2 was dispersed in PMMA resin using sol-gel technique were paper. The specimens were examined their properties via SEM, XRD and Vickers hardness, [25].

Furthermore, both the hot and cold cured methods were evaluated to investigate the effect of adding multi-walled carbon nanotubes MWCNTs with various filler amount to dental base PMMA. It was found that, the wear and friction coefficient for the composite clearly decreased. On the other hand, specimen prepared via hot-cured showed higher hardness than others prepared by cold-cured method, [26]. Thermogravimetric analysis was used to assess the mixing and dispersion quality of MWCNTs. Results of flexural strength and fatigue of PMMA/MWCNTs specimens proved a significant improvement compared to the pure PMMA specimen, and the expected results were achieved, [27]. Also, the hydroxyapatite nanoparticles dispersed through the resin to increase the efficiency of the composite properties, [28]. PMMA filled by hydroxyapatite nanoparticles was performed to evaluate the tribological and mechanical performance of the resin. It can be concluded that, the increase of filler amount of hydroxyapatite nanoparticles helps to improve the tribological and mechanical properties.

To further develop and enhance the bonding between the reinforced nanofiller and polymeric resin, more than one factor has been taken into consideration to reduce the bonding problems between the filler and the resin. Nano-graphene is added to the hybrid SiO_2-TiO_2 nanoparticles to enhance the tribological properties of PMMA dental base material, [29]. It can be noticed that, adding 0.4 wt. % of nano graphene to PMMA nano

composite, dispersed with 0.4 wt. %. of SiO_2 - TiO_2 nanoparticles, reduces friction coefficient and wear rate while the hardness is significantly up to 18 %. By three different mediums, nano graphene oxide was dispersed through a resin to performed PMMA composite. The three methods go through mixing nanoparticles and PMMA powder directly. In the second case, the nanoparticles incorporated into the hardener MMA, but in the third case, the nanoparticles are dispersed through water and added to the resin. It can observed that, the third method displays the best homogeneous composite, [30]. Different filler content of graphene silver nanoparticles, 1.0 and 2.0 wt. %, were evaluated to examine the antibacterial effects. Dental PMMA nanocomposite is significantly improve the antibacterial effects, [31].

The aim of this current work is to perform PMMA incorporated with nano-graphene and SiO_2 nanoparticles to evaluate the mechanical and surface properties of the PMMA base dental material.

EXPERIMENTAL

Specimens Preparation and Experiments

Materials

For various dental restoration purposes, acrylic resins based on poly (methyl methacrylate) were used as the base material. PMMA is a resin depending on two components, one is a powder called modified PMMA and the other is a liquid called MMA that is based on it as a hardener. PMMA resin is purchased from Acrostone Co., Egypt. The hybrid of graphene and TiO_2 nanoparticles is adopted as the filling material. The properties of the different materials are presented as follows, as listed in Tables (1 - 3).

Table 1. PMMA resin mechanical properties

Tensile Strength (MPa)	70
Density (gm/cm^3)	1.18
Water Absorption (%)	0.3
Hardness, Shore D	75 - 85

Table 2. Nano-graphene

Purity %	99.5
Thickness (nm)	6
Diameter (µm)	24
Specific Surface Area (m^2/g)	150
Color	Grey

Table 3. TiO_2 nanoparticles

Purity %	95.9
Average Particle Size (nm)	18 - 35
Specific Surface Area (m^2/g)	150 - 550
Density (gm/cm^3)	2.2
Color	white

Specimens Preparation

The powder to liquid ratio is one of basic lineaments that control and affect the durability of PMMA composite. This work targets specimen mixing ratio of 2:1. The composition is prepared via adding the hardener MMA to the PMMA powder in the same proportion as mentioned above. Once mixed, the stirring process begins at a speed of up to 200 rpm for 10 minutes at room conditions. This is followed by the process of pouring the mixture into a cylindrical mold with dimensions of 10 mm in diameter and 20 mm of height. Finally, the mixture is pressed into the mold for 2 hours under a pressure of 10 bar. The same procedure is followed to produce nanocomposites. Hybrid nano-fillers Graphene/TiO_2 were performed with different filler content as displayed in Table 4.

Table 4. Specimen mixing ratios

Specimen	PMMA	Gr	TiO_2
S00	100	0	0
S01	99.25	0.5	0.25
S02	99	0.5	0.5
S03	98.75	0.5	0.75
S11	98.75	1.0	0.25
S12	98.5	1.0	0.5
S13	98.25	1.0	0.75

A reciprocating pin-on-disk tribometer based on ASTM standard G99-95 was used to perform the friction and wear resistance of the specimens, as illustrated in Fig 1. The set-up drive unit is an electric motor (220V AC 50HZ/0.5HP) with rotational variable speed, controlled with voltage control unit. Nano-composite and pure PMMA specimens were installed into holder and stainless-steel alloy plate were used as counter-face surfaces. Surface roughness tester utilized to precisely gauge accurate roughness of the stainless-steel plate, R_a = 0.023 µm, R_q = 0.029 µm, and R_z = 0.179 µm. The friction force can be estimated via a load cell, as strain gauges are found. The software is provided to take the friction coefficient data and plot the charts of the tested specimens. Experiments were conducted under three different amounts of normal load of 2, 4, 6, 8, and 10 N at 0.1 m/s of sliding velocity. The sandpaper with grit size of 1200µm were used as counter-face to examine the wear behavior of nano-composite specimens. Wear tests were done each exactly 120 sec., and the weight loss was considered by weighing the specimens before and after the test. The wear rate was determined as follows:

$$W_r = \Delta m / L \rho F_n$$

Where the sliding distance L, material density ρ and applied load F_n, and proportional to the weight loss Δm.

The worn surfaces were performed using an electronic microscope (OLYMPUS BX53M, USA). The 3D and 2D images were performed to assess the wear tracks and pits happened

on the sliding surfaces. Furthermore, ASTM standard D2240 procedure is applicable to perform and evaluate of hardness properties using the durometer Shore D device. The hardness value was examined three times along the specimen surface and then hardness average value was calculated. The average values were calculated with standard errors.

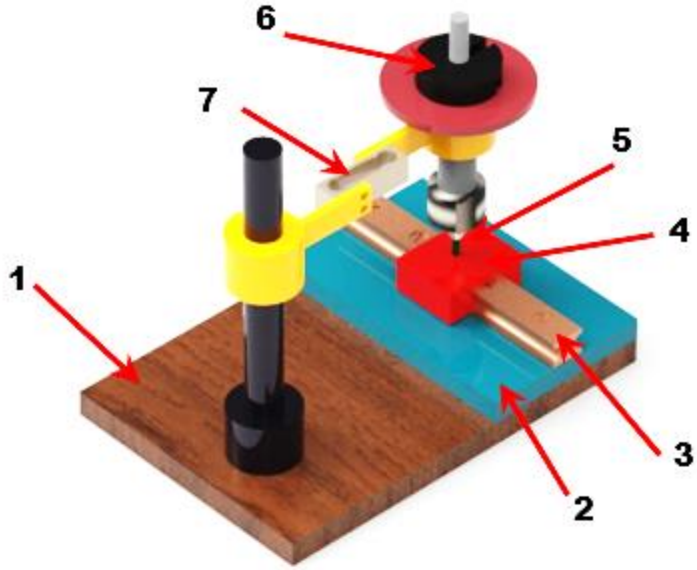

Fig. 1 Sketch of the reciprocating tribometer.
1. Base, 2. Disk, 3. Linear Bearing, 4. Sandpaper,
5. Specimen, 6. Normal Load, 7. Load Cell.

RESULTS AND DISCUSSION

The current study is focused on studying the mechanical and tribological properties to perform the behavior of PMMA/Gr/TiO_2 nanocomposites and determine the preferable adding amount. The tribological characterizations of PMMA specimens were performed by examining the friction coefficient and wear rate of the two specimen sets. The friction coefficient of the first specimen set, was illustrated in Fig. 2. It was declared that the pure PMMA specimen (S00) has the high friction coefficient values at various normal loads. It is evident from the results that, nano-fillers help to significantly reduce the coefficient of friction. As the resin begins to be filled with the hybrid nano-fillers, the friction values begin to decline. The specimen (S02) with composition of 0.5 wt. % of both of graphene and TiO_2 nanoparticles, showed outstanding results. From this it can be concluded that, specimen (S02) gives a reduction of the friction coefficient average of 15 % comparing with the pure specimen (S00). It has also been noted that the increase in TiO_2 nanoparticles above the limit of 0.5 wt. % leads to the coefficient of friction rises again, specimen (S03). This may be due to the fact that, the increase of TiO_2 nanoparticles leads to its spread across the surface of the specimen, which increases the chances of its presence in the areas of friction and plays the role of an abrasive material. The same trends of graphs were achieved when examining the other set of specimens as displayed in Fig. 3. Nevertheless, it may be observed that the specimen (S11) with composition of 1.0 wt. % of graphene and 0.25wt. % of TiO_2 nanoparticles, causes a reduction of the friction coefficient average of 17 % less. In this set

of specimens, the increase in TiO_2 nanoparticles amount caused a high rate of friction. The comparison between the best specimen of the two sets was displayed in Fig. 4. Based on that, the specimen (S11) achieved the desired results and improved better than those achieved with the other specimen (S02). It is possible to rely on the fact that graphene is a self-lubricating material that helps significantly reduce friction in the contact areas. Finally, it can be recognized that low filler amount of TiO_2 nanoparticles with 1.0 wt. % of graphene act as modifiers, in which it exhibited a reduction on the friction coefficient.

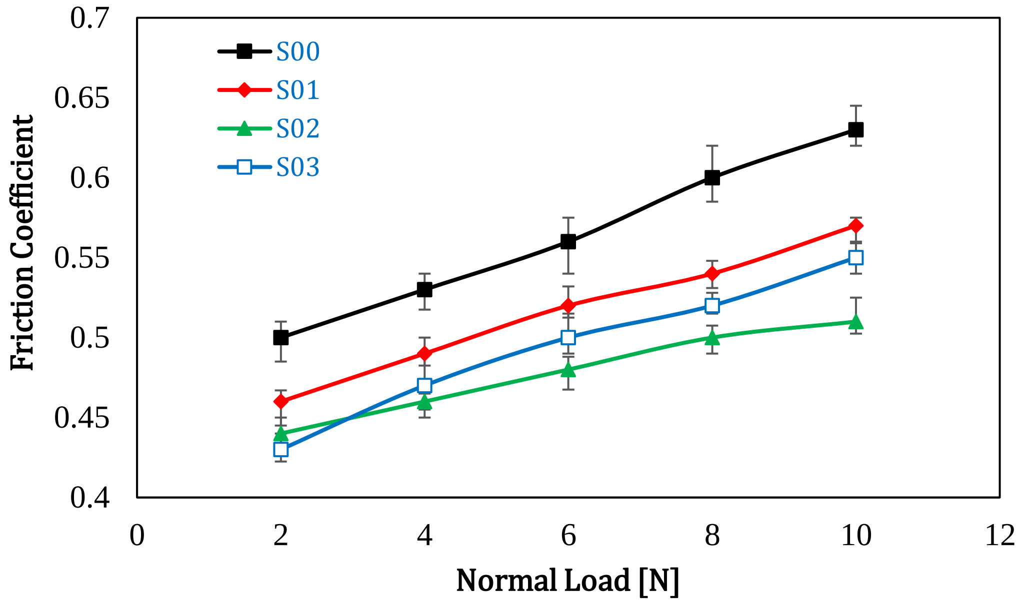

Fig. 2 Friction coefficient of PMMA nanocomposite specimens with 0.5wt. % graphene.

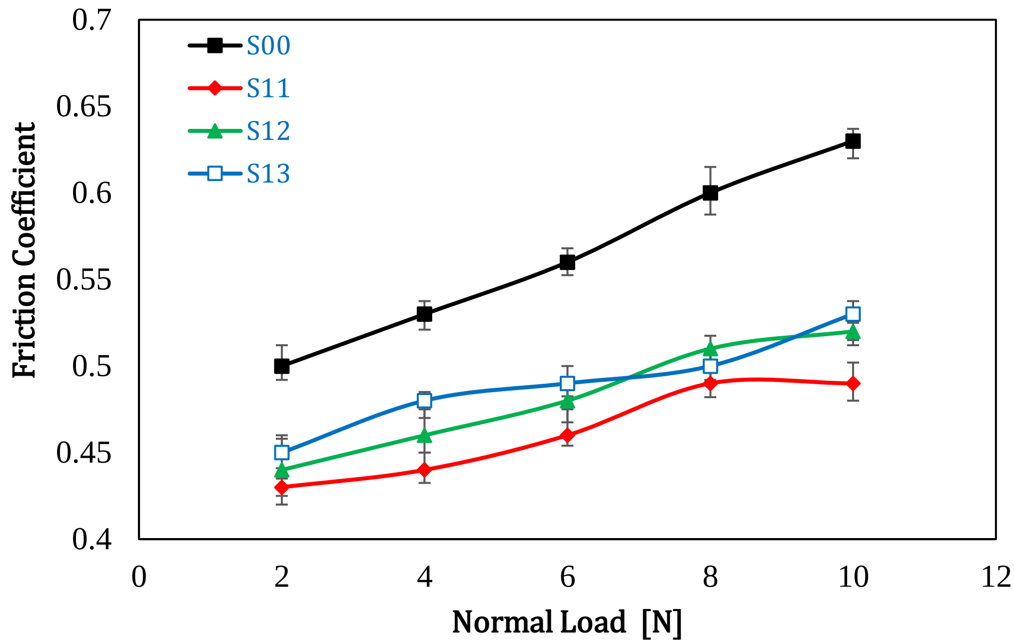

Fig. 3 Friction coefficient of PMMA nanocomposite specimens with 1.0wt. % graphene.

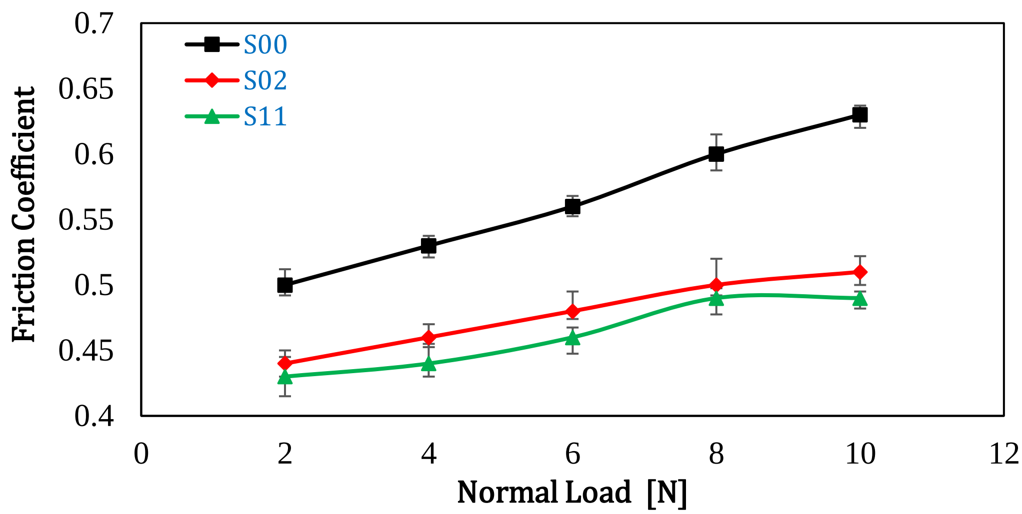

Fig. 4 Comparison of friction coefficient of PMMA nanocomposite specimens.

To give a clearer view of the tribological properties, the wear rate of the specimens was studied. The weight loss due to the normal load and the frictional load were used to measure the wear rate. Regarding the first group, it is possible to note that loading a filler amount of TiO_2 nanoparticles may obviously improve the wear rate compared to pure PMMA. While the maximum improvement reaches to average 18.5 % in the wear rate occurs for specimen (S02), as illustrated in Fig. 5. It can be evaluated that the spherical shape of TiO_2 nanoparticles has a rolling effect that helps to reduce the wear and improves the strength of the composite. Also, it cannot overlook the role of graphene as a self-lubricating material. On the other side, for second set, it is easy to verify that the increase in the filler amount of

graphene leads to reduce the weight loss of the PMMA composite. It can be noticed from Fig. 6 that reinforcing 0.25 wt. % of filler amount of TiO_2 nanoparticles (S11) achieves improvement of the wear resistance nearby 19% compared to pure PMMA, as displayed in Fig. 6. Figure 7 illustrates that specimen (S11) offers better tribological performance compared to the specimen (S02). Accordingly, the good dispersion and improve of the bonding strength between nanoparticles and the PMMA resin, helps the load-carrying capacity to improve.

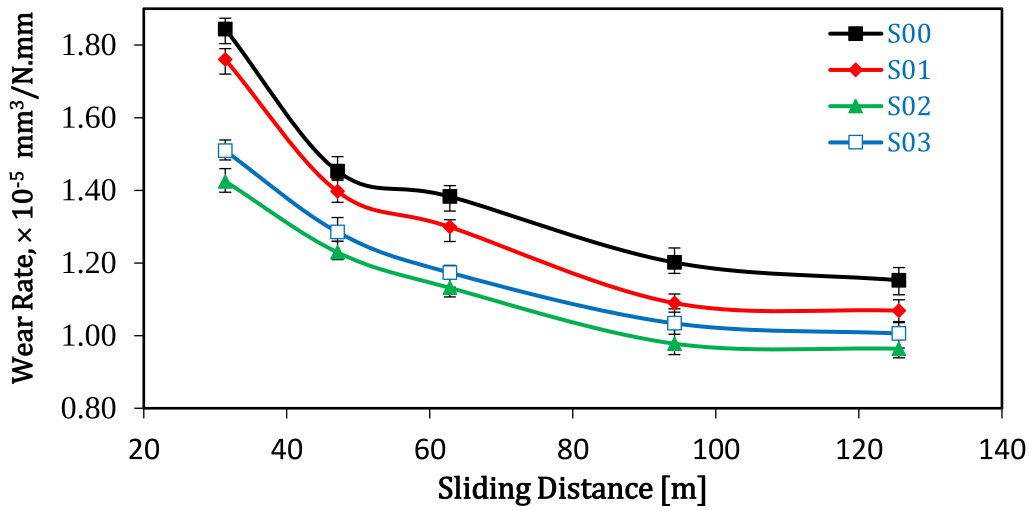

Fig. 5. Wear rate of PMMA nanocomposite specimens with 0.5wt. % graphene.

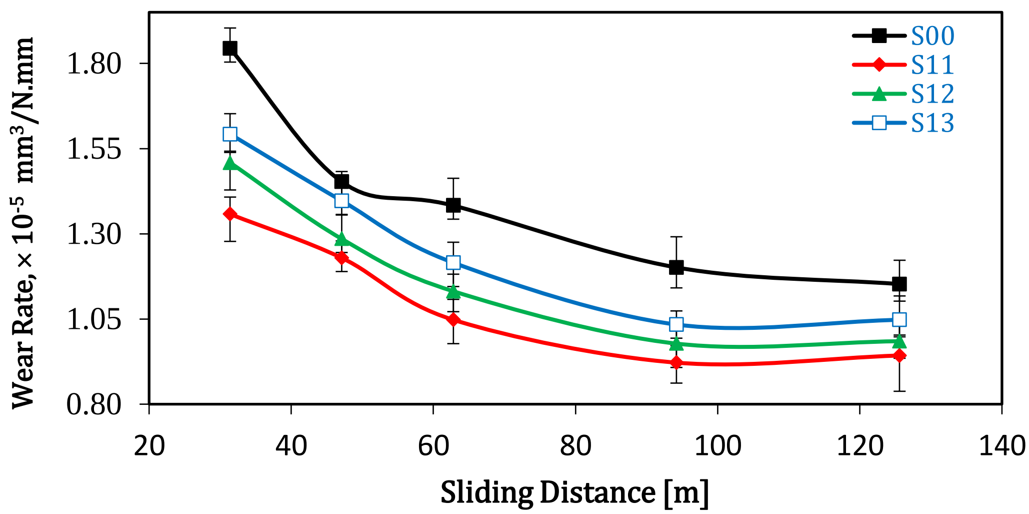

Fig. 6. Wear rate of PMMA nanocomposite specimens with 1.0wt. % graphene.

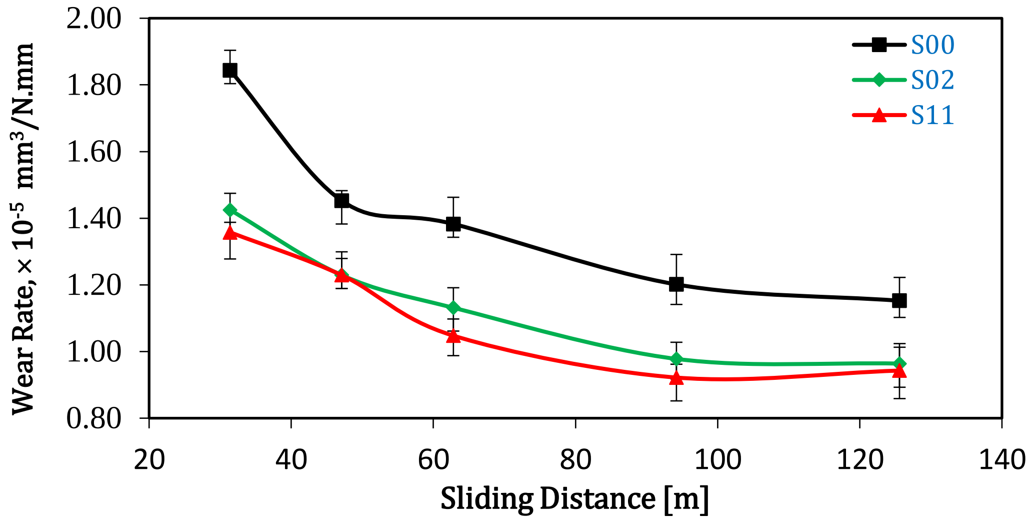

Fig. 7. Comparison of wear rate of PMMA nanocomposite specimens.

3D images of worn surfaces of all specimens were scanned after rubbing against stainless-steel disc to evaluate the wear mechanisms. Using electron microscope with magnifications of up to 200x, worn surfaces topography and 3D profile of the surface roughness were analyzed. Figure 8 displays detailed image of the wear marks on PMMA loading with hybrid graphene/TiO_2 nanoparticles, which is evident that the wear tracks formed through surfaces of specimens. It can be clearly noticed in that the sliding surface of PMMA specimen (S00) exhibits rougher surface, which leads spread pits and grooves. Moreover, there were less surface roughness and reduce the pits and grooves on worn surface of PMMA specimens filled by hybrid filler. It can be noticed from Figure that PMMA specimens (S02) and (S11) exhibit the smoothest and less damaged surfaces. Consequently, the dispersion of TiO_2 nanoparticles through the PMMA resin is a main reason leads to the surface roughness is smoother and in the enhancement of the wear resistance ability. While the graphene content of specimens helps the resin to form a lubricated layer leads to reduce the frictional forces.

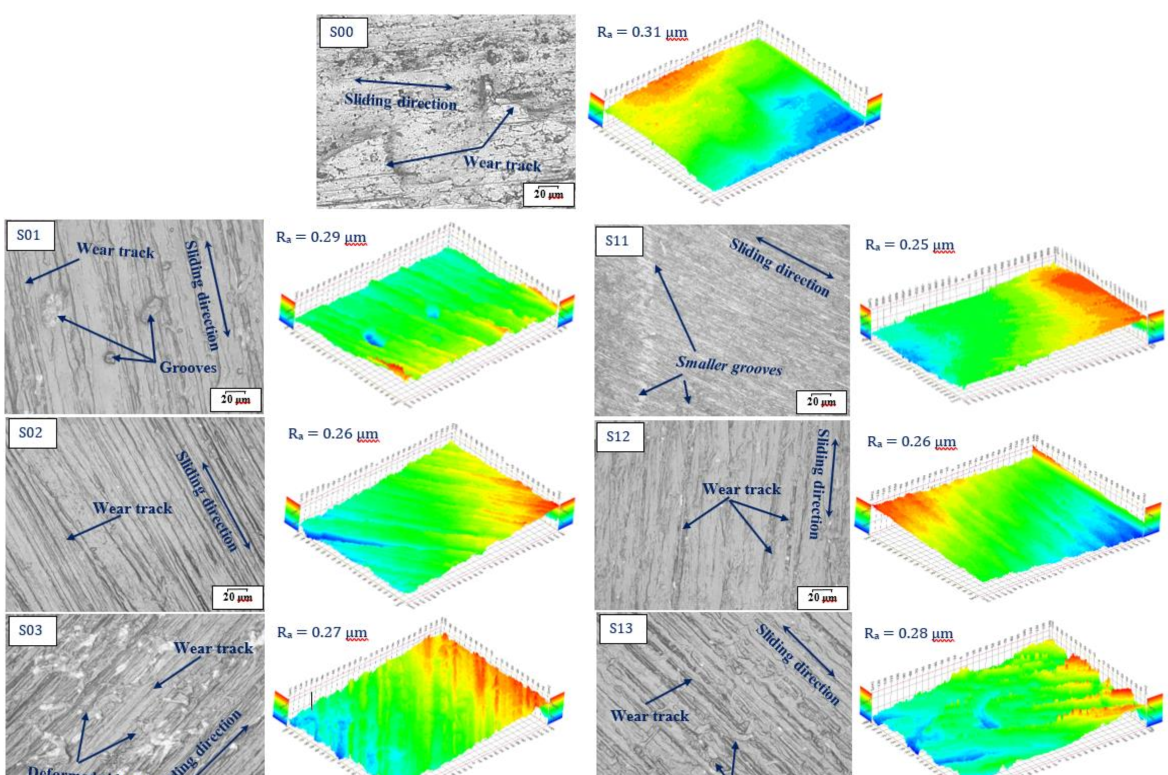

Fig. 8. 3D topography images of worn surfaces of PMMA/Graphene/TiO_2 composites.

The surface hardness of the specimens, the focus of the study, was measured in order to ensure the good bonding between the resin and filler particles. From Figure 1 it can be noticed that the pure specimen (S00) have a hardness of 81.2 (D index). While the specimens with nano-filler have significantly improved the hardness with all filler content. It can be observed that, the first set, loading of 0.5 wt. % of graphene, achieved slightly lower results than its counterparts in the other set, 1.0 wt. % of graphene. Based on the results presented previously, it becomes clear that, TiO_2 nanoparticles have the main role to enhance the strength of the composite. Specimen (S13) exhibited the best improvement of the Shor D hardness of 87.1 (D index), with an improvement up to 7.2% compared with pure PMMA.

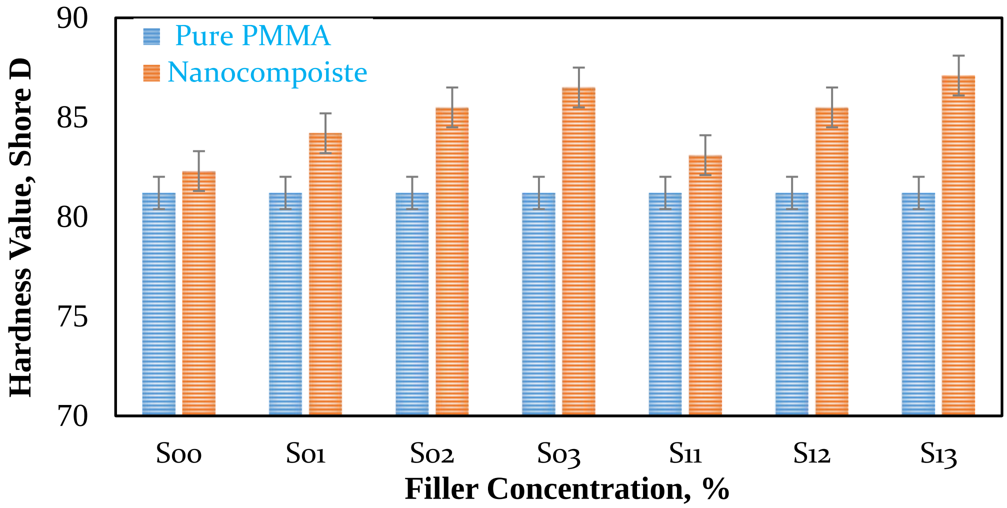

Fig. 9 Hardness values of PMMA nanocomposite specimens.

CONCLUSIONS

Through the current study, it can conclude the following:

1. **PMMA nanocomposite could be successfully filled by hybrid nanoparticles with better properties comparing with pure PMMA.**
2. **PMMA specimens with loading amount of 0.5 wt. % of graphene and 0.5wt. % TiO_2 nanoparticles and 1.0wt. % of graphene and 0.25 wt. % TiO_2 nanoparticles recorded a good enhancement comparing with pure PMMA**
3. **The friction coefficient reduces with average of 15 % and 17 % for the specimens (S02) and (S11), respectively.**

4. **The wear rate reduces with average of 18.5% and 19% for the specimens (S02) and (S11), respectively.**
5. **Specimen with filler content of 1.0wt. % of graphene and 0.75 wt. % TiO_2 nanoparticles, exhibits the maximum improvement of the hardness up to 7.2 % compared with pure PMMA.**

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