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Mechanical and Tribological Characteristics of PMMA Reinforced by Natural Materials

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Abstract
The current paper focuses on studying the mechanical and tribological properties of Polymethyl methacrylate (PMMA) reinforced by natural materials. Two different natural powders, corn cobs, and miswak particles were used with different loading fractions, 2.0, 4.0, 6.0, 8.0, and 10 wt.%. The results showed a good enhancement in the mechanical properties of PMMA composites comparing with pure PMMA. The improvement in mechanical properties is accompanied by an improvement in tribological properties. Eexperimental results illustrated that PMMA composites with a weight fractions of 8.0 % corn cobs and 6.0 % miswak recorded the optimal tribological and mechanical characteristics among other weight fractions. Furthermore, the microscopic results showed a change in the wear mechanism of the PMMA due to the incorporation of the natural materials.

***Keywords**:* PMMA-Composites; Corn Cob particles; Miswak particles; Friction and Wear rate; Mechanical properties;

1. Introduction

Polymers get a lot of attention in various industrial applications, so making innovation and development in their characteristics is a major focus of many researches [1-4]. Polymethyl methacrylate (PMMA) is one of thermoplastic polymer is distinguished as transparent, tough, and rigid that widely used in various industrial applications [5]. PMMA can be performed well as alternative shatterproof windows, LED lights, car windows, motorcycle windshields, and lenses. Furthermore, PMMA is frequently used as dental cavity fillings, fabricate dentures, and bone cement. This may be due to it is distinct biocompatibility, and ease of forming with high aesthetic degrees [6]. This may lead to PMMA being one of the most common materials in denture fabrication [7]. Dental acrylic materials are made up of two main components, the first is a powder (PMMA modified form), while the second is a liquid component (Methyl methacrylate) called a solvent or a monomer. Many researches focused on dental materials reinforced by micro and nanometer-size materials to create a new composite that has enhanced mechanical and wear resistance characteristics [8]. Metal powders and oxides form are getting some attention, which undoubtedly improves the mechanical strengths and thermal properties. Platinum nanoparticles are used as filler in PMMA denture acrylic to enhance the mechanical characteristics [9]. Human hair fibers and copper fibers, 2mm length and 200 μm diameter, were added to PMMA resin to change the impact strength and color stability properties [10]. Ceramic particles of ZrO_2- Al_2O_3-SiO_2 are used as filler in PMMA composite, which fabricates through the sol-gel technique. Indeed, the fundamental target of the addition is to evaluate the mechanical strengths via SEM, X-ray diffraction (XRD), and Vickers hardness test [11]. Tribological characteristics and hardness, Shore D, of PMMA denture base resin reinforced with nanographene (NG) and hybrid SiO_2 - TiO_2, have been significantly improved. The hardness of PMMA/NG/SiO_2/TiO_2 composite increases by 18% compared with pure PMMA [12]. The heat cure modes and curie period have been significantly affecting the hardness and the friction coefficient of the PMMA matrix reinforced with TiO_2 nanoparticles [13, 14]. The friction coefficient of the PMMA/TiO_2 composite is decreased gradually by increasing of TiO_2 filler amount. However, it has been observed that gradually increasing of loading of TiO_2/ZnO nanoparticles leads to reduce the friction coefficient and wear rate of the PMMA composite [15].

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Moreover, the flexural strength and the thermal characteristics of the PMMA composite are significantly enhanced by ZnO nanotubes [16].

Also, the loading PMMA resin of the synthetic and natural fibers takes into account [8]. Fiberglass is good enough to realize the preferable requirement dental material. Therefore, good bonding between fiberglass and PMMA is used to improve the adhesion and superior biocompatibility of PMMA/Fiberglass composite [17]. The physical, thermal, biocompatibility and chemical properties of PMMA resin reinforced with glass fiber were reviewed, also the factors that may be involved in a denture fracture [18]. Two sets were used as reinforced materials in dental composites. Barium glass particles, 2 μm, and silanated glass fibers,1.4 mm length, 7–13 μm diameter, with filler content of 0%, 2.5%, 5.0% and 7.5% is examined. Based on the experiments, it can be concluded that hybrid fillers significantly increased fracture toughness and reduced shrinkage rates [19]. Study the effect of the reinforcing system consists of nylon-6 was dissolved in hexafluoroisopropanol, with fracture of weight 10%, and multi-walled carbon nanotubes MWCNTs, with fracture of weight 0.5% and 1.5%, on the flexural strength of a PMMA dental resin [20]. Moreover, the wear and friction coefficient go down directly for both the hot- and cold-cured PMMA/MWCNTs composite. While the hardness of the hot-cured resin increased in parallel with the increase in MWCNTs filler amount [21]. Thermogravimetric analysis (TGA) was used to examine the dispersions of MWCNTs in the PMMA matrix. Fatigue resistance, flexural strength, and resilience of PMMA/MWCNTs composite were compared with a commercially pure PMMA [22]. PMMA reinforced by hydroxyapatite (HA) nanoparticles with filler amounts of 0, 0.2, 0.4, 0.6, and 0.8 wt.% was tested to evaluate the tribological and mechanical properties [23, 24]. The results indicate that increasing the concentration of HA nanoparticles leads to enhance the stiffness, compressive yield strength, toughness, ductility, and hardness of the PMMA/HA composite. While the friction coefficient and wear loss are declining.

The incorporation of new materials such as those that can be obtained from plants, crops, animals, or other sources is one of the alternatives to reduce some outstanding problems. These materials are characterized by being environmentally safe, easy to recycle, and available in abundance from renewable natural sources [25]. The mechanical properties of the PMMA composite can be improved by interacting between the oil palm empty fruit bunch fiber and matrix. Utilization of palm fiber in cellulose form, with 0.5 mm thickness, leads to enhanced flexural strength and flexural modulus [26]. It was believed that the incorporation of miswak fiber, with a length of (2, 6, and 12 mm) and filler content (3, 6, and 9 wt. %), into the PMMA matrix, can improve the mechanical and physical properties [27]. On the other hand, PMMA reinforced by fibers, miswak, and bamboo showed an enhancement of compression strength with high loading of fibers content. Therefore, the treatment of the fibers with alkaline solution helps the bonding strength to increase [28]. Another attempt has also been made based on filling the PMMA resin with pomegranate peels, an average diameter of 53.38 nm, and powder of dates powder, an average diameter of 93.78nm, with loading filer of 0.4, 0.8, 1.2, and 1.6%. From the results, it is possible to verify that the PMMA composite confirmed a positive change of mechanical and physical properties [29].

In the current work, two sets of PMMA composites samples were fabricated by adding corn cobs and miswak micro-particles to PMMA denture resin. First, the micro-particle of the selected natural materials was produced in a uniform particle size. Second, the composite samples were produced at different weight fractions of the corn cobs and the miswak micro-particles of 2.0, 4.0, 6.0, 8.0, and 10 wt.%. Third, the chemical composition was investigated utilizing XRD. Finally, then the mechanical and tribological properties of the produced composites were studied to evaluate the effect of adding the selected natural material into the properties of the PMMA composite.

2. Materials and Experimental

2.1. Materials

The resin used is a quick prepared product, which is a self-curing and cold cure poly (methyl methacrylate) acrylic material. PMMA powder (purchased from Cold Cure, Acrostone Dental & Medical Supplies, Cairo, Egypt) was used in this research. A hardener liquid (from Acrostone Dental) was added to resin A hardener liquid was added to the resin to help start and accelerate the mixing process. The main characteristics of the utilized PMMA are illustrated in table 1.

Corn is one of the widely cultivated crops in Egypt. Corn cob is the innermost part of the corn that the kernels grow around. Corn cobs were collected after the kernels were removed and dried in the summer sun for 3 months to remove the moisture. After drying, the corn cobs were crushed manually using a mortar and pestle. The crushed parts were pulverized using the grain miller at 300 rpm, then passed through a microplate sieve to obtain the uniform particle size of 150 μm and finer, as shown in Fig. 1. The typical and chemical properties of corn cob powder were illustrated in Table 2 [30].

Table 1: Typical Properties of acrylic PMMA.

Physical Properties		Mechanical Characteristics	
Color	White	Compressive Strength [MPa]	75-131
Density [gm/cm^3]	1.18	Compressive Modulus [GPa]	2.76-3.3
Particle Size [µm]	50-105	Fracture Strength [MPa]	7.92%
Water Solubility	0.3%	Fracture Modulus [GPa]	2.4-3.45
		Hardness, Rockwell Scale M	80-100

Miswak stick is one of the widely cultivated in the arab world specially in Saudi Arabia. The Miswak sticks were cleaned with water before drying into an oven under a constant temperature of 100°C for 72 hrs to get them completely dried. The drying stage follows the cutting process of the sticks and then was pulverized using the grain miller at 250 rpm. The powder was sieved using microplate sieves with uniform precision pores (1 to 100 µm), as shown in Figure 2. The characteristics of miswak particles were displayed in Table 3 [31].

Fig. 1. Photograph of corn cobs with a size of 150 µm

Table 2: Typical and chemical properties of Corn Cob powder.

Typical Properties		Chemical Characteristics	
Color	Wheat	Ash Content	1.33%
Bulk Density [gm/cm^3]	0.64	Lignin	35.2%
Water Solubility	9.0%	Cellulose	41.5%
PH	5	Hemicellulose	13.0%
Specific Gravity	1.1	Others	8.97%

Fig. 2. Photograph of miswak particles

Table 3: Typical and chemical properties of Miswak powder.

Typical Properties		Chemical Characteristics	
Color	Brown	Ash Content	1.7%
Bulk Density [gm/cm^3]	0.52	Volatile Matter	0.65%
Water Solubility	3.7%	Moisture	7.92%
PH	6.2	Total loss of Ignition	89.8%
Specific Surface Area [m^2/g]	0.5		

2.2. Preparation of Composite Samples

The matrix is a quick prepared material that produced by mixing methyl methacrylate powder and hardener with a ratio of 2:1. Specimens were mixed manually at room temperature in a glass container by dispersing corn cobs and miswak particles into a PMMA matrix using a rotating mixer at about 300 rpm from 10 min. As the stirring continues, the viscosity of the mixture gradually increases due to the partial dissolution of PMMA particles to hardener liquid. The dough was casted into the samples' cylindrical mold in a 25 mm × 8 mm using compression molding under pressure of 14 MPa for 30 minutes, as shown in Fig. 3. Furthermore, in order to evaluate the optimum filler weight fraction, PMMA composites were prepared with five different compositions as shown in Table 4.

Table 4.: Variation of PMMA Corn Cob and Miswak particles (%)

Code	Compositions of PMMA Samples		
	PMMA	Corn Cobs	Miswak
Pure PMMA	100%	-	-
Corn2	98%	2%	-
Corn4	96%	4%	-
Corn6	94%	6%	-
Corn8	92%	8%	-
Corn10	90%	10%	-
Miswak2	98%	-	2%
Miswak4	96%	-	4%
Miswak6	94%	-	6%
Miswak8	92%	-	8%
Miswak10	90%	-	10%

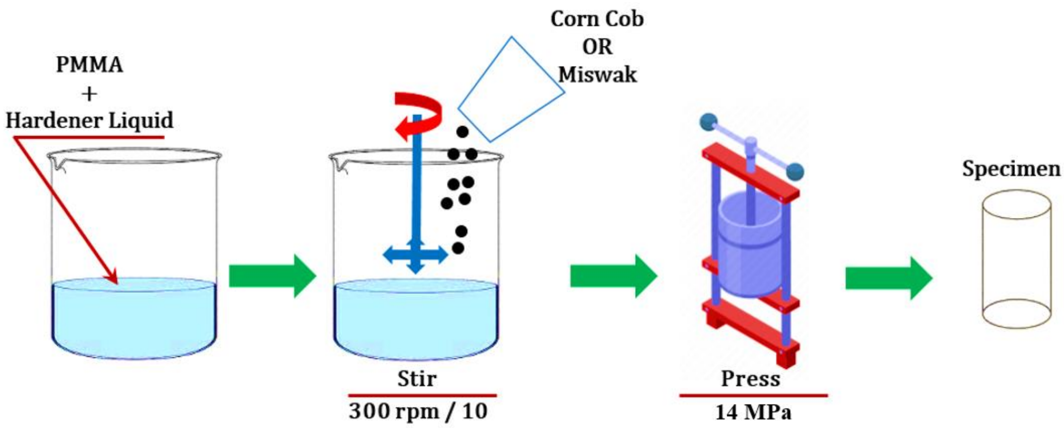

Fig. 3. Schematic diagram of the PMMA/composite samples' preparation

2.3. Testing and Characterization

The mechanical properties of the PMMA/composites were examined via hardness and compression tests. The hardness test was determined via the durometer Shore D device with sharp cone point (SR 0.1mm), 35° included angle, measuring range of 0~100 ± 0.5 HD and dwelled for up to 10s, based on the ASTM standard D2240 [32]. For each sample, the average reading values were measured five times along the specimen surface. United High Capacity Smart Universal Hydraulic (DFM-300KN) was used to perform the compression test in accordance with the ASTM standard D1621. System equipped with a 300 KN capacity load accuracy +/- 0.5%, encoder resolution of 0.0005 mm and conditioned at 25 °C and 50 % RH. The three test specimens are prepared from each composition to evaluating their compressive properties. The stress-strain curves were measured automatically by the machine, and compressive yield strength and modulus of elasticity were estimated.

A reciprocating pin-on-disk tribometer, with 50 mm stroke according to ASTM G99-95 [33], was used to test the tribological behavior of PMMA composites specimens under dry contact conditions at 29°C and relative humidity of 55%, as illustrated in Figure 4. The reciprocating strategy was selected to simulate the actual motion of denture base in the human mouth. The samples were tested as a cylindrical pin and the disk was a rectangular plate made of stainless steel or PMMA. Different two types of plates were used to simulate the natural conditions. PMMA disk was suitable in case of friction can occur between teeth made of PMMA material, [34].

Furthermore, sometimes the dentist uses stainless steel crowns to protect the children's teeth [35]; so that, the sliding PMMA composites against stainless steel disk were also performed. Surface roughness tester utilized to precisely gauge accurate roughness of the stainless steel and PMMA disks (Ra = 0.025 µm, and Rz = 0.177 µm) and (Ra = 0.018 µm, and Rz = 0.159 µm), respectively. Cleaning the samples' surfaces before each experiment was conducted using acetone and then dried with a heat gun to remove any contaminants. The experiments were done under different normal loads of 6, 8, 10, and 12 N at 0.4 m/s of sliding velocity. Wear tests were performed by estimating the weight loss between the initial and final weights of samples. For each specimen individually, the experiment was repeated five times under the same conditions and the average values were recorded and standard errors were calculated. To evaluate the wear mechanism, scanning electron microscope SEM microscope (JCM-6000Plus; JEOL, Tokyo, Japan) was used to inspect the worn surface morphology of PMMA composites. The samples were washed, dried completely via air, and then coated with a thin film of platinum and images were scanned using SEM.

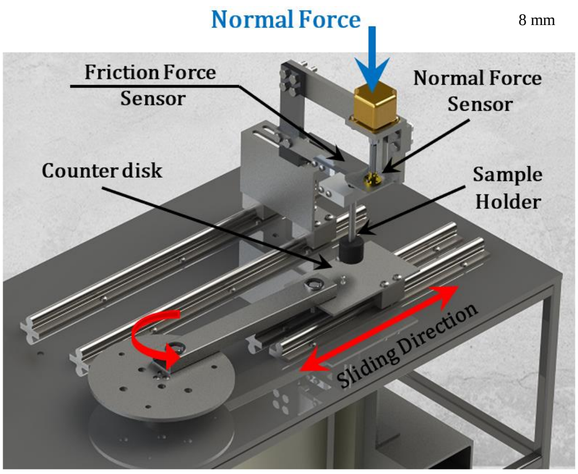

Fig. 4. Sketch of the reciprocating tribometer

3. Results and Discussion

Before the evaluation of the mechanical characteristics of the produced composites, the chemical composition of the pure materials and composites were extracted using XRD, as shown in Fig. 5-7. In Fig. 5, the pure PMMA showed some peaks at 2θ of 13.6°, 30.7°, and 41.8° which is consistent with other studies [34] and the peeks are not sharp which indicate the complete amorphous nature of the poly(methyl methacrylate) [36]. The XRD for the corn cob showed a main peak at 21.8° which is very close to the alpha cellulose XRD pattern [37]. The diffractogram of Miswak raw material exhibited several intense diffractions which cannot indicate the crystallinity of the raw powder. However, the Miswak XRD result may be attributed to the existence of the inorganic superfluity moieties which is doubtful to be calcium-based mineral. Consequently, more studies should be conducted to fulfill the identity of the Miswak compound. Fig. 6 and 7 show a disappearance of the corn cob and Miswak peeks which confirm the well-distribution of corn cob and miswak particles in the PMMA matrix. Furthermore, the XRD patterns of the PMMA/corn cob and PMMA/Miswak composites showed also an amorphous nature. These results indicate that the structural characteristics of PMMA were not influenced by the incorporation of corn cob or the miswak powder and there was no chemical reaction occurred among the matrix and reinforcements materials.

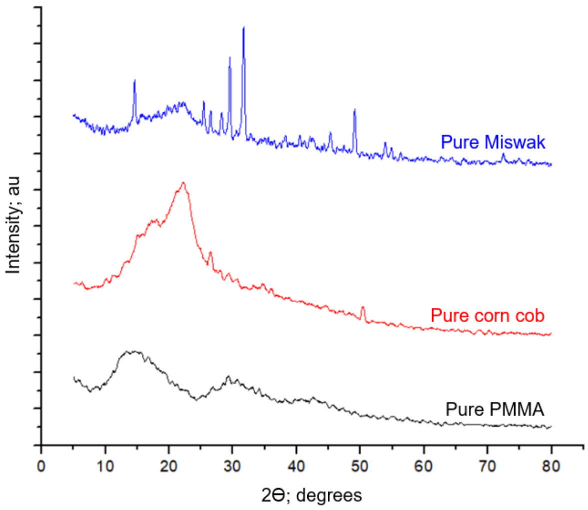

Fig. 5. X-ray diffraction patterns of pure poly(methyl methacrylate) (PMMA), corn cob, and Miswak.

PMMA is commonly used as a denture base material, which is susceptible to compression loads. Compressive yield strength and modulus of elasticity of all PMMA/composites samples were measured based on the aforementioned standards. Figure 8 displays the effect of corn cobs particles filler amount on the mechanical properties of the PMMA. Compared with pure PMMA, the compressive yield strength and modulus of elasticity receded up to 37.8% and 30.5%, respectively, when filler amount reached 6.0 wt.% CCs particles. Nevertheless, the addition of 8.0 wt.% CCs particles to PMMA composites has enhanced remarkably the compressive yield strength as well modulus of elasticity up to 19.4% and 11.61%, respectively. On the other hand, the pure PMMA was examined along with the filler amount of miswak particles in an attempt to improve the PMMA composite's properties. In Figure 9, it can be noted that adding miswak particles to the mixture weakens its properties, but with increasing filler amount the trend starts to get better. Consequently, the synthesis of Miswak6 enhanced the compressive yield strength and modulus of elasticity with an augmentation percent of 32.1% and 21.43%, respectively.

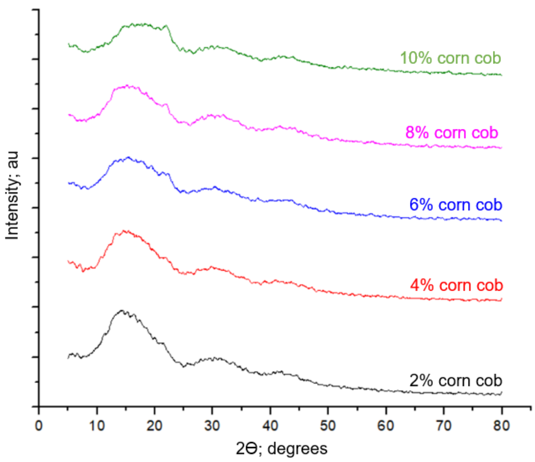

Fig. 6. X-ray diffraction patterns of PMMA/corn cob composites.

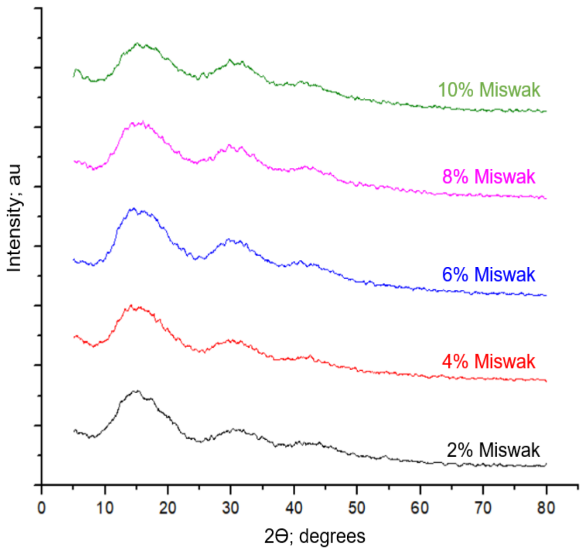

Fig. 7. X-ray diffraction patterns of PMMA/Miswak composites.

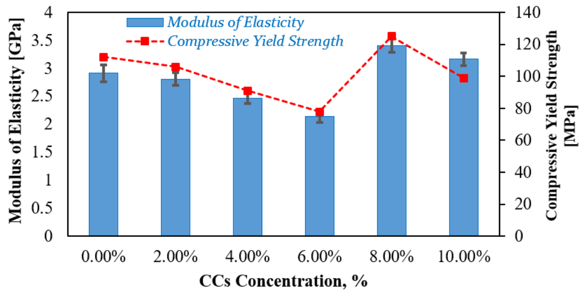

Fig. 8. Compressive yield strength and modulus of elasticity of PMMA/Corn cobs samples.

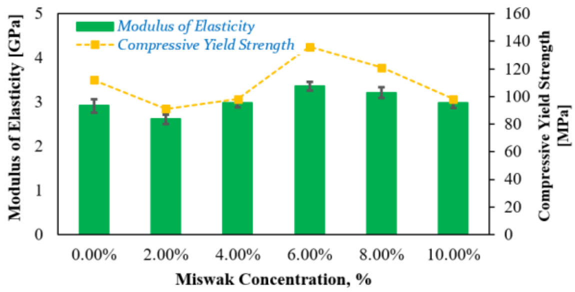

Fig. 9. Compressive yield strength and modulus of elasticity of PMMA/Miswak samples.

Figure 10 illustrates that the pure PMMA has a hardness of 81 ± 3 (D index). The addition either of CCs or miswak particles to PMMA composites has significantly improved the hardness along with the filler amount. PMMA/Miswak composite (6%) exhibited the maximum enhancement of the hardness of 87 (D index), with an improvement of up to 7.4% compared with pure PMMA. Additionally, the hardness increased by 6.17% (86 D index) for PMMA/Corn cob composite (8%). Consequently, it could be concluded that the PMMA/composites reinforced by 8.0 wt.% and 6.0 wt.% of CCs and miswak particles, respectively have a significant effect in improving the mechanical properties of the poly(methyl methacrylate).

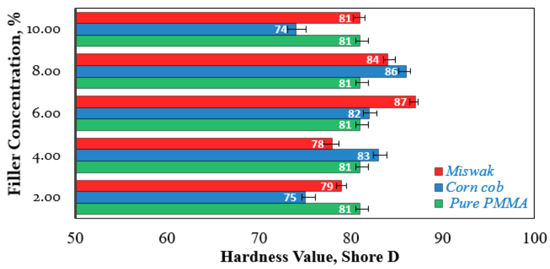

Fig. 10. The hardness of PMMA composites with different filler amount.

To study the tribological characteristics, both PMMA and stainless steel were used as the counter disk to simulate the actual frictional contact surfaces of dentures. Figure 11 illustrates the relation between friction coefficient and the filler amount of the corn cobs particles under different applied loads rubbing against PMMA surface. The results reveal that a low filler amount of CCs particles is counter-effective on the friction behavior as observed for Corn2 and Corn4. Frictional behavior of PMMA/CCs composite begins to improve when filler amount increases up to 6.0 wt.% (Corn6) and achieve the best result for the 8.0 wt.% (Corn8). Nevertheless, it may be noted that the filler amount increases above 8%, leading to a regression of enhancement of the friction behavior of Corn10. The same observations are true when testing that set of samples against stainless-steel surface as displayed in Fig. 12. In both cases, the addition of 8.0 wt.% filler amount of the corn cobs particles reduced the friction coefficient compared with pure PMMA. From this, it can be concluded that the reducing in the friction coefficient for Corn10 with an average of 18% and 14.5% at rubbing against PMMA and stainless-steel surfaces, respectively. This enhancement may be due to the presence of lignin in the composition of CCs powder, which can form a self-lubricating layer on the contact area helps to reduce friction between rubbing surfaces [39-40].

Figures 13 and 14 display the influence of filler amount of miswak particles on the friction coefficient comparing with pure PMMA. All samples were examined under the same operating conditions that were mentioned before. From the results, it can be indicated that adding a low filler amount of miswak particles plays a role in reducing the coefficient of friction. It can be verified that the general trend of friction behavior of the samples begins to improve as the PMMA is filled with 2.0 wt.% of miswak particles, and the improvement continues with increasing the filler amount up to 6.0 wt.%. In contrast, the friction coefficient significantly increases gradually for samples with a filling amount of 8.0 wt.% or higher. The same observations appear in both cases, PMMA and stainless-steel disks, and exhibit the same behavior. Compared with pure PMMA, Miswak6 gave the most appropriate friction behavior with both contact surfaces achieving reduction ratios of up to 14% and 15.7% at rubbing against PMMA and stainless-steel surfaces, respectively. Finally, it can be realized that a low filler amount of miswak particles act as modifiers, in which it exhibited an enhancement on the friction performance.

As well the samples slid against PMMA and stainless-steel surfaces, the weight loss due to the normal load was measure and the specific wear rate was calculated. The wear rate curves of PMMA/CCs samples are shown in Fig. 15 and 16. It is clear that the addition of 2.0 wt.% of corn cobs particles led to a reduction in the specific wear rate by approximately 8.27% compared with pure PMMA.

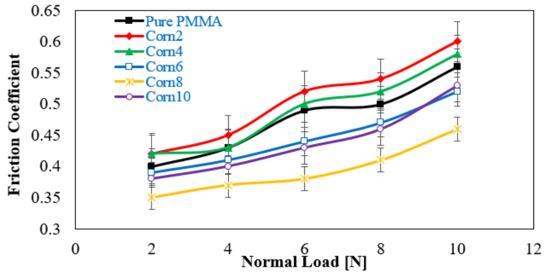

Fig. 11. Friction coefficient of PMMA/Corn cobs samples against PMMA surface.

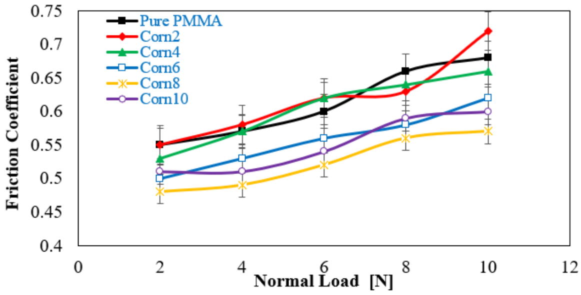

Fig. 12. Friction coefficient of PMMA/Corn cobs samples against a stainless-steel surface.

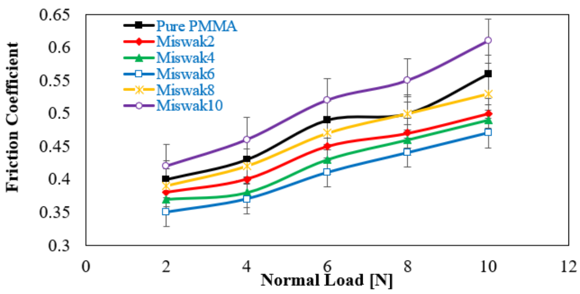

Fig. 13. Friction coefficient of PMMA/Miswak samples against PMMA surface.

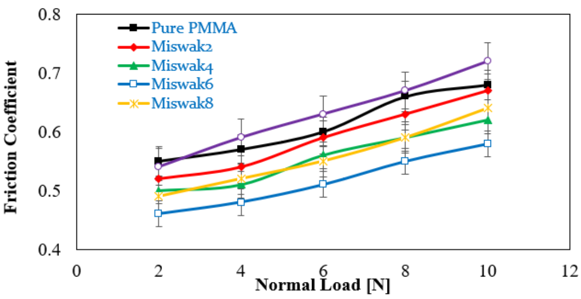

Fig. 14. The friction coefficient of PMMA/Miswak samples against a stainless-steel surface.

While the maximum reduction of 14.5% in the wear rate occurs for the 8.0 wt.% of CCs (Corn8). By testing samples against a stainless-steel disk, the wear rate reduces and it can notice that Corn8 exhibited an improvement in wear rate up to 21.1%.

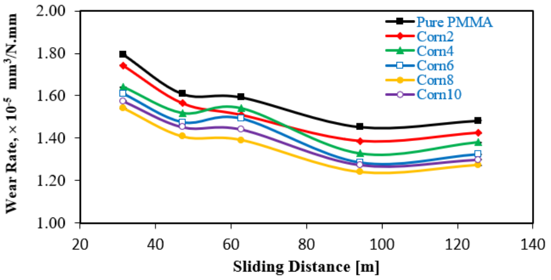

Fig. 15. Wear rate of PMMA/Corn cobs samples against PMMA surface.

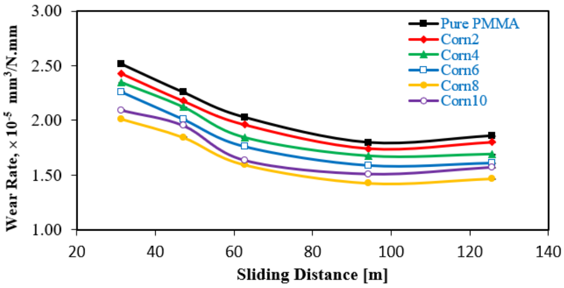

Fig. 16. Wear rate of PMMA/Corn cobs samples against a stainless-steel surface.

In case of utilizing miswak particles as a reinforcement, an enhancement of the wear resistance of the PMMA composite, as illustrated in Fig. 17 and 18. By observing the occurrence of weight loss during the tests, the results indicate that increasing the miswak particles' filler content reduced the wear rate of the PMMA composites. It means that the wear resistance increased when increasing the filler content of the miswak particles. Miswak6 achieves the best improvement results as the wear rate is reduced by about 20% and 22.7% at sliding against PMMA and stainless-steel surfaces, respectively. It can be concluded that the addition of CCs or miswak particles to the pure PMMA improve the polymer mechanical properties, in which; the bonding strength between CCs or miswak particles and the PMMA resin was enhanced and led to an improvement in the load-carrying capacity [41-42]. Consequently, reinforcing PMMA composite by 8.0 wt.% of CCs or 6.0 wt.% miswak would enhance the mechanical and tribological properties.

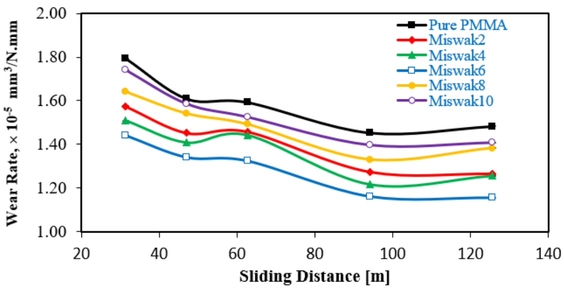

Fig. 17. Wear rate of PMMA/Miswak samples against PMMA surface.

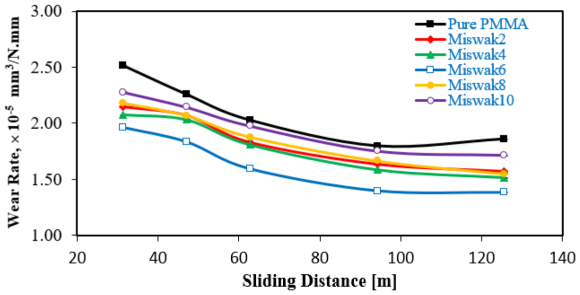

Fig. 18. Wear rate of PMMA/Miswak samples against a stainless-steel surface.

As shown in the results of the experiments, the incorporation of 8.0 wt.% of CCs and 6.0 wt.% miswak into PMMA led to an enhancement in the tribological properties compared with pure PMMA. Consequently, the worn surfaces of pure PMMA and the two composite samples were evaluated utilizing SEM to identify the wear mechanism, as shown in Fig. 19. It is clear that in the case of pure PMMA, the surface has some deteriorated layers resulted from ploughing and peeling during the friction test against PMMA and stainless-steel counter faces. The eliminated layers may lead to a shear resistance yielded to an increase in the friction coefficient during the sliding process [43]. Consequently, the wear mechanism for pure PMMA could be a delamination wear mechanism which usually increases the friction coefficient and decrease the wear resistance [44]. On the other hand, an enhancement in the rubbed surfaces of 8.0 wt.% of PMMA/CCs and 6.0 wt.% PMMA/Miswak composites could be observed. This enhancement in the surface profile could be attributed to the enhancement in the mechanical properties which were recorded for those composites. Consequently, smooth surfaces with some cracks and debris with unnoticeable deteriorated layers, a fatigue wear mechanism, which enhances the tribological results.

4. Conclusions

In the current study, two types of natural materials, Corn cobs (CCs) and Miswak were used with different loading fractions as a reinforcement for poly(methyl methacrylate), PMMA. The natural materials were processed to be used as a micro-scale reinforcement material, up to 150 µm. The mechanical and tribological properties of the PMMA/CCs and PMMA/Miswak composites with different loading fractions were evaluated. Results showed that the optimal loading fraction to be used from CCs and Miswak were 8 wt.% and 6 wt.%, respectively. Utilizing these loading fractions showed an enhancement in the mechanical properties which led to an enhancement in the tribological characteristics.

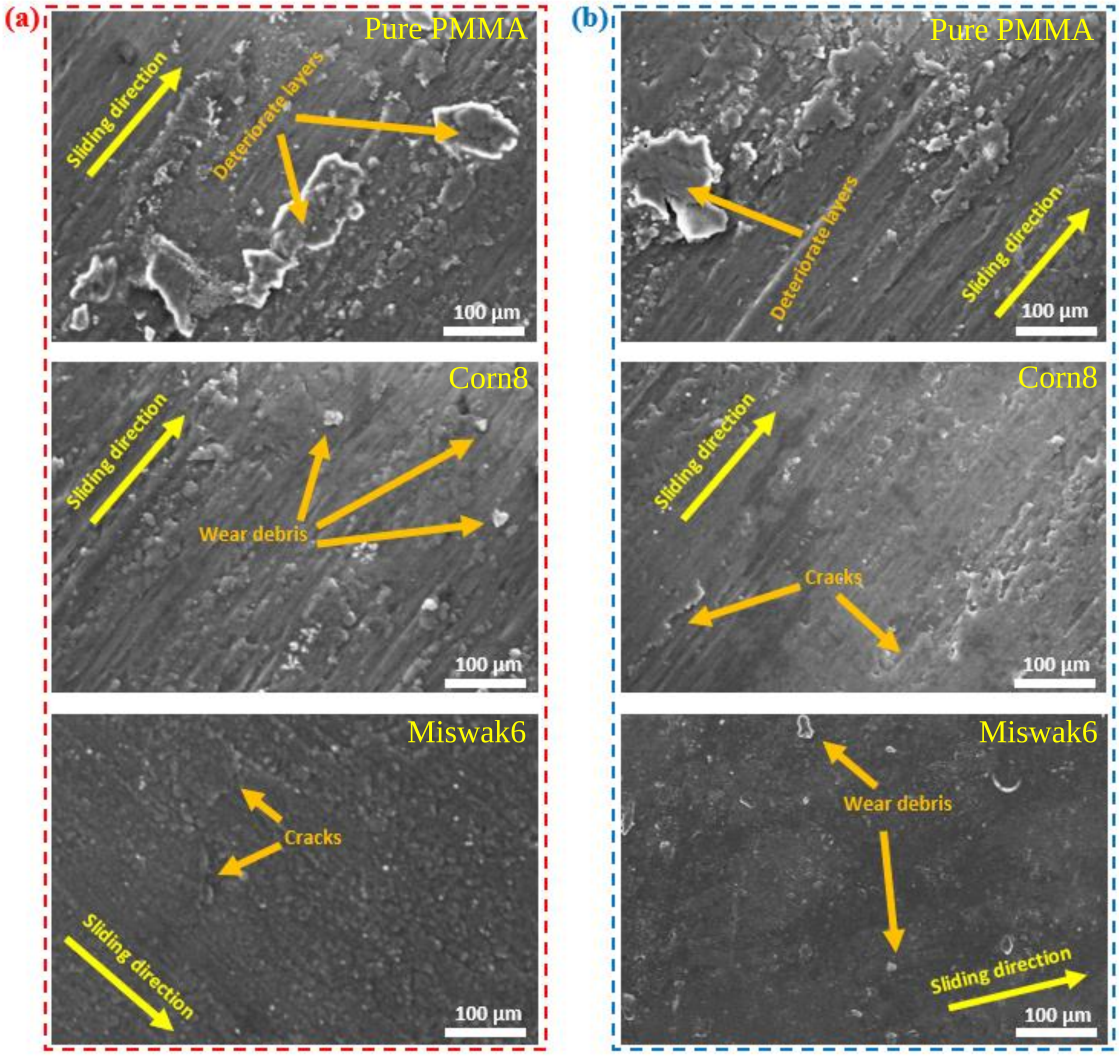

Fig. 19. Scanning electron micrographs of the surface of the pure PMMA, PMMA/corn cobs (8 wt.%), and PMMA/Miswak (6 wt.%) composites, after being rubbed against (a) stainless steel and (b) a PMMA counterpart.

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