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# Microstructure, Friction and Wear Analysis of PMMA – based Composites with Solid Lubricant

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*Abstract* - (Methyl methacrylate) was prepared by in situ free radical polymerization by mixing BPO granules and liquid methyl methacrylate (MMA) mixture. The percentage of solid lubricant was studied on the friction and wear properties of the PMMA polymer using Pin-On - Disk technology. Fourier transform infrared (FTIR), and electron microscopy (SEM), were used to characterize the PMMA synthesis of a solid lubricant. The results showed that the coefficient of friction and the wear rate of the PMMA-based compound, compared to that of unfilled PMMA, can be significantly reduced by filling with a solid lubricant. As the percentage of solid lubricant mass filling increases, the wear rate of the PMMA compound of the solid lubricant increases under dry slip conditions. The main corrosion mechanism for unfilled PMMA is adhesive corrosion. Whereas, the main wear mechanisms of PMMA solid lubricants are abrasive wear and corrosion.

Keywords - Material property, PMMA, Polymeric materials, Solid lubricant, Stress analysis, Wear damage.

## INTRODUCTION

Polymers are still limited in their distinctive shape due to their thermal stability and properties of durability, stiffness and strength. The degradation of most thermoplastics occurs at relatively low temperatures. This behavior limits the use of most thermoplastic non-reinforced polymers in tribological applications at elevated temperatures. Given the weak corrosion rate of most basic polymers, various methods are presented to improve the tribological behavior of the polymers. One method is hardening with suitable fillers, blending with other polymers and adding lubricants [1]. In flexible plastics such as composites, erosion particles are created by the following mechanisms: [2][3] (a) adhesion and deformation during sliding, (b) plowing by superimposed solid protrusions or solid particles on the sliding surface, and (c) discharge resulting from indentation cracks below the surface. Corrosion debris resulting from these mechanisms is dispersed particles, or these particles are transported to the surface by mechanical entanglement or by the adhesion property of the opposite surface. As for dynamic and stable wear, the removal of wounds is constantly progressed by deformation and fracture resulting from the effects of wear. Plowing also plays a distinct role in the process of surface wear and the formation of abrasive particles. The wear of a sample depends on a certain roughness, the extent of wear depends on the particle size [4]. Polymer and its compounds are facing an increasing demand in many industrial applications such as loading materials, pulleys, seals, gears, cameras, wheels, piston rings and clutches where the self-lubricating properties are utilized and operated to move away from the old methods and methods of liquid lubrication or grease and by using it, pollution and its associated problems are produced [5]. However, when contact is present between the sliding facade, friction and wear are prominent [6]. A study has been conducted on the mechanism of PTFE transmission and wear. Movable PTFE films can reduce or increase the friction force depending on the differences in adhesion mechanisms in the contact area during layer formation. Low values of activation energy indicate that the corrosion of PTFE is mainly related to the breakage of weak molecular bonds and shear between surfaces and the slip of the accumulated crystal structure formed in the band structure of the materials [7]. While it was proven in similar studies conducted on PEEK compounds filled with CuS and PTFE powders during friction with a steel tool under ambient sliding conditions and adding PTFE resulted in reducing both the rate of wear and friction while the friction coefficient increased with the addition of CuS. Which indicates that the rate of corrosion of the composite depends on the ability to create transfer films on steel surfaces, which is a mechanical process that the particles of the removed material are confined to the cracks of the opposite surface [7][8][9]. The roughness of the corresponding surface is sometimes required to achieve an efficient transfer appropriate to a specific medium. Friction processes are often accompanied by residues as a result of erosion and rubbing of the two surfaces, which leads to the formation of a friction layer through which a qualitative determination can be given and a description of the slip performance of that surface. These tribological processes can

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be understood separately for both polymers and compounds. It is possible to define the coefficient of friction (COF) as the ratio between the frictional force ( $F_f$ ) and the normal load ( $F_N$ ), as shown in the equation below.

$$\mu = \frac{F_{\rm f}}{F_{\rm N}} \dots \dots \dots \dots \dots \dots (1)$$

The friction between two surfaces resulting in plowing with debris and crusts of the solid isotope and adhesion can be expressed as mechanical interactions at an atomic level as in the deformation of fillings. The shear method and the rupture of the material around the contact area between the two surfaces and around it during sliding has a real effect on friction [10]. Bowden and Tabor demonstrated in 2005 that the true contact zone has a significant effect and is indirectly proportional to friction [11]. There are two phenomena involving under friction and causing energy loss: adhesion  $F_a$ , and  $F_d$  deformation [12].

$$F_f = F_a + F_d \dots \dots (2)$$

Often the high pressure and the shear of the bonds between the surfaces that are in a relative movement is friction called adhesion friction and heat is generated as a result of the shear to the site melting of some types of materials and junctions arise at the local contact points [13]. The self-lubricating compound should be providing effective support for the dynamic stresses caused by the applied load as well as the occasional friction stresses. If the polymer / polymer compound cannot support these stresses, it leads to conditions: rapid wear (plastic deformation, brittle fracture, etc.). Catastrophic failure, increased wear down to the point where the polymer will support the contact area at low dynamic pressures, producing less deformation than wear. Stress is a factor that must be taken care of with self-lubricating compounds. A part shall be designed so that the self-lubricating compound is able to support the dynamic stresses of slip and roll. In the event that the self-lubricating polymeric compound possesses sufficient capacity to carry the impacted load, then there are different types of wear depending on the polymer, the additives used and the sliding conditions. In order to provide optimum lubrication, a kind of "shear layer" must be activated between the sliding surfaces. The shear layer has the ability to inhibit the adhesive and "plowing" reactions that occur in the contact area of moving surfaces and reduce friction, as well as reduce stresses in a larger area of the polymer compound. In most results, and in general, the thinner the cutting layer, the greater the effectiveness [14]. Wear is a process that takes place at the interfaces between the interacting bodies under dry slip conditions. The technological revolution has generated many advanced engineering products, most of which include high-speed sliding or rolling or small-dimensional geometric shapes for hostile environments, which can only be developed and included in the use fields by successfully overcoming the challenges of corrosion processes through an understanding and awareness of previous experiences and past and contemporary studies. Mechanical examples include: gas turbine engines, car engines, transmissions, tires and brakes, in the field of medicine: artificial human

joints, artificial teeth, artificial Knee, and in the technological field: hard drives for storing data and an increasing number of electromechanical devices for home and industrial use [15]. The simplest way to reveal how much material has been lost due to corrosion. It gives the total amount of mass removed, but the distribution of wear depth in the contact surface remains unknown. Moreover, worn surfaces can be examined with the help of optical microscopes and scanning and transmission electron microscopes [16]. wear rate was obtained by using the equation below [17]:-

Wear rate (W.R) =  $\Delta W/\pi D.N.t$  ...... in gm/cm (3) where:-D is the sliding circle diameter (cm), t=sliding time (min), N = steel disc speed (rpm)

## **EXPERIMENTAL DETAILS**

## 1. Material

The methyl methacrylate (MMA) ( $\geq$ 99.5%) monomer as liquid, the benzoyl peroxide (BPO), Calcium Streat, Paraffin wax, and Bees wax were all supplied from the local Iraqi market. Calcium Streat (Ca(C<sub>18</sub>H<sub>35</sub>O<sub>2</sub>,Tm=170 °C)<sub>2</sub> is a compound made of calcium with a group of solid organic acids extracted from edible materials. [18]. Bees wax (C<sub>15</sub>H<sub>31</sub>COOC<sub>30</sub>H<sub>61</sub>, Tm= 75 °C), it is consists of carbohydrates as raw materials mainly [19]. Praffien wax (C<sub>20-40</sub>H<sub>42-82</sub>, Tm= 145 °C) its main advantages are high melting point and very small particle size, with the particle size at 1-2µm [20].

## 2. Specimen Preparation

The PMMA matrix was synthesized by a in-situ free radical polymerization, consisting of a BPO powder and an MMA liquid. In the following experiments, three kinds of solid lubricant with ratios (0.05,0.1,0.2,0.5 wt.%) were selected to study the influence of the solid lubricant percentages on the tribological performance of PMMA composites. PMMA and BPO granules were kept in a desiccator for 24 h to remove moisture before used. The liquid mixture was mainly MMA with 100g g and BPO 0.9 wt %. The filler and the MMA/BPO mixture with the corresponding mass were mixed and stirred in a mixer for 30 min. After, the solution mixture (MMA, BPO) was gradually mixed and put into the oven vacuum to get rid of air that causes bubbles. Thus the polymerization reaction of PMMA will be initiated by the action of PBO and After mixing evenly and before curing, the generated PMMA produced by The polymerization molds were formed using glass sheets, a (15 cm) long rubber tube, and four paper holders, Against the glass sheets and sealed by paper holders, the materials are injected into the mold using a syringe, The mold was brought into the oven for 8 hours at a temperature of 85 °C.

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TABLE 1 Polymeric Composites				
Ν	Sample Code	Sample Composition		
1	Pure PMMA	PMMA without additives		
2	PMMA/Pwax1	PMMA+0. 05 %Paraffin wax		
3	PMMA/Pwax2	PMMA+0. 1 %Paraffin wax		
4	PMMA/Pwax3	PMMA+0. 2 %Paraffin wax		
5	PMMA/Pwax4	PMMA+0. 5 %Paraffin wax		
6	PMMA/Bwax1	PMMA+0.05 %Beeswax		
7	PMMA/Bwax2	PMMA+0.1 %Beeswax		
8	PMMA/Bwax3	PMMA+0.2 %Beeswax		
9	PMMA/Bwax4	PMMA+0. 5 %Beeswax		
10	PMMA/CS1	PMMA+0.05 %Calcium stearate		
11	PMMA/CS2	PMMA+0.1%Calcium stearate		
12	PMMA/CS3	PMMA+0.2 %Calcium stearate		
13	PMMA/CS4	PMMA+0.5 %Calcium stearate		

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TADLE 1

## 3. Material Characterization

# • FTIR

Fourier-transform infrared spectrometer is a technique employed to distinguish organic, non-organic and polymeric groups sited in substances, it was utilized to characterize molecular structure of (Pure PMMA, Pwax/ PMMA, Bwax/ PMMA and CS/PMMA) and interpret bonds between Pure and sulf lubricant materials. The instrument employed for this work is (IR Affinity-1) type made in (Kyoto, Japan) and situated in polymer and petrochemicals industries department/ material engineering collage/Babylon University. The procedure is executed under ASTM E1252.

• Wear Test

The Pin-on-disk tool evaluated the dry friction and wear properties of unfilled PMMA and PMMA lubricant. An aluminum ball mounted in pin-shaped fixtures was used to slide against pure PMMA and sulfur-lubricated PMMA. Ball diameter (10 mm), temperature during tests: RT, range of applied force: 1 to 12 N, distance: 500 m, speed: 110 rpm. The dimensions of the wear test samples were (1 mm wide, 2 mm thick, 1.26 mm thick). All samples were processed to obtain a smooth surface. Each experiment was repeated, at the end of each test the evolution of the friction coefficient along with the slip distance was measured directly by the CSM frictionometer. Unlike the ring on the disc, during the test of the pin on the disc, no reverse movement occurs and no breaks occur in the lubricating film between the wear components. So it can be assumed that there is pure sliding friction between the components. The loading condition in the pin-to-disk test is determined by the normal load, slip speed, initial temperature of test medium, and slip distance between damaged couplings.

#### Micro-Hardness Test

It is a method of determining a material's hardness and resistance to penetration in case of if test samples are very small or thin, or when small regions in a composite sample or plating are to be measured detailed information can be provided precisely and about surface features of materials that have a fine microstructure, are multi-phase, non-homogeneous or prone to cracking. ASTM E384 is a method of measuring micro-hardness by pushing a pyramid shaped diamond indenter into a sample and then measuring the size of the indent using a microscope. This method covers Knoop micro-hardness using an elongated pyramid indenter and Vickers micro-hardness using a square (equilateral) pyramid indenter. The indenter is pushed into the sample using a fixed load Hardness profiles from the surface to the core of a sample allow measurement of effective case depth or depth of decarburization. High surface hardness will give good wear resistance and the results are reported in a table per sample. The hardness is determined by measuring the depth of indenter penetration or by measuring the size of the impression left by an indenter [21].

$$HN = \frac{2Fsin\left(\frac{136}{2}\right)}{d^2} = 1.854 \ \frac{F}{d^2}.....(4)$$

Contact Angle Test

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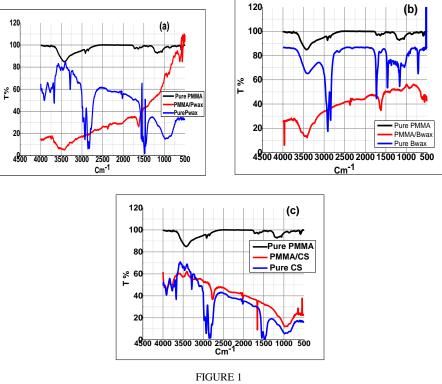
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The contact angle is the angle between the circumference of the water droplet and the surface, and through it is possible to know the amount of wetness of the surface [22]. The used instrument is (SL 200C - Optical Dynamic I Static Interfacial Tensiometer & Contact Angle Meter which manufactured in KINO Industry Co., Ltd., USA with contact angle range (0° to 180°). Wettability can be defined as the ability of the liquid to spread over the surface of the substrate. Hydrophilic surface can be determined when the contact angle is low while, for hydrophobic surfaces the contact angle is large. The contact angle is that angle formed by the intersection of the liquid-solid interface and the confrontation of the liquid vapor and is geometrically calculated by applying the tangent from the point of contact along the three-phase contact line. (When the contact angle is less than 90 degrees, it leads to suitable surface wetting, and the liquid is spread over large of areas on the surface; While contact angles greater than 90 degrees lead to unwanted surface wetting, so the liquid contracts to form a combined liquid droplet [23].

#### **RESULTS & DISCUSSION**

## 1. FTIR Results

The Figure (1,(a)) the intensity of the bands observed in the spectrum (O-H Stretch) starts increasing with the emergence of a doublet at (3757 - 3456) cm<sup>-1</sup>, indicating the structural transformation of the molecule, also, at 3903-3672 cm<sup>-1</sup> for Pure Pwax and 2846 cm<sup>-1</sup> for PMMA/Pwax, respectively, but show took a wrinkled shape for Pure PMMA . The spectrum (CH2; CH3 Stretch) shows this doublet (2947 - 2846 cm<sup>-1</sup>) for Pure Pwax and 2924 cm<sup>-1</sup> for Pure PMMA, but less shifting for PMMA/Pwax at 2846 cm<sup>-1</sup>, the C-O stretching bands observed at 1550 cm-1 in Pure PMMA is less shifting in PMMA/Pwax. The Figure (1,(b)), (C – O Stretch) at 1550-1600 cm<sup>-1</sup> respectively for PMMA is higher shifting in PMMA/Bwax K peak appear at 1643 cm<sup>-1</sup>. The (O-H Stretch) here less shifted with the emergence of a doublet at 3572 - 3433 cm<sup>-1</sup> for PMMA/Bwax. The (CH<sub>2</sub>; CH<sub>3</sub> Stretch) here less shifted at 2384 cm<sup>-1</sup> for PMMA/Bwax, (C=C Stretch) less shifted at 1643cm-1. Figure (1,(c)),( O-H Stretch) for PMMA/CS show decrease of this peak in the same position for Pure PMMA [24]. While (R-CO<sub>2</sub> band ) present in PMMA/CS spectrum only [25], (CH<sub>2</sub>; CH<sub>3</sub> Stretch) is less shifting for PMMA/CS at 1635 cm<sup>-1</sup>.



FTIR SPECTRUM FOR (A) PURE PMMA, PMMA/PWAX, AND PURE PWAX, (B) PURE PMMA, PMMA/BWAX, AND PURE BWAX, AND, (C) PURE PMMA, PMMA/CS, AND PURE CS

TABLE 2							
ABSORPTION AREAS FIG.(1,(A))							
Pure PMMA	Pure Pwax	PMMA/Pwax					
3448 cm <sup>-1</sup>	3502 cm <sup>-1</sup>	$3757 - 3456 \text{ cm}^{-1}$					
	3903-3672 cm <sup>-1</sup>	3565 cm <sup>-1</sup>					
2924 cm <sup>-1</sup>	2947 – 2846 cm <sup>-1</sup>	2846 cm <sup>-1</sup>					
1550-1600 cm <sup>-1</sup>	1519 cm <sup>-1</sup>	1635 cm <sup>-1</sup>					
	ABSORPTION A   Pure PMMA   3448 cm <sup>-1</sup> 2924 cm <sup>-1</sup>	ABSORPTION AREAS FIG.(1,(A))   Pure PMMA Pure Pwax   3448 cm <sup>-1</sup> 3502 cm <sup>-1</sup> 3903-3672 cm <sup>-1</sup> 3903-3672 cm <sup>-1</sup> 2924 cm <sup>-1</sup> 2947 - 2846 cm <sup>-1</sup>					

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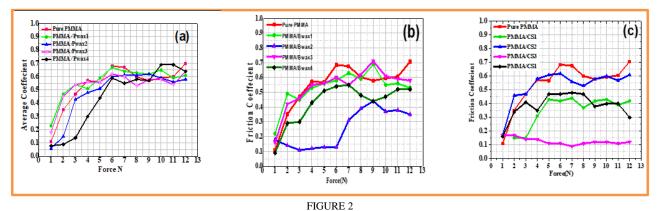
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ABSORPTION AREAS FIG.(1,(B))							
Type of bond	Pure PMMA	Pure Bwax	PMMA/Bwax				
O-H Stretch	3448 cm <sup>-1</sup>	3417 cm <sup>-1</sup>	3572 - 3433 cm <sup>-1</sup>				
C - O Stretch	1550-1600 cm <sup>-1</sup>	1774 cm <sup>-1</sup>	1643 cm <sup>-1</sup>				
CH <sub>2</sub> ; CH <sub>3</sub> Stretch	2924 cm <sup>-1</sup>	2916 cm <sup>-1</sup>	2384 cm <sup>-1</sup>				
C=C Stretch	1743 cm <sup>-1</sup>	1759 cm <sup>-1</sup>	1643 cm <sup>-1</sup>				

TABLE 3

ABSORPTION AREAS FIG.(1,(C))						
Type of bond	Pure PMMA	Pure CS	PMMA/CS			
O-H Stretch	3448 cm <sup>-1</sup>	3919 - 3672 cm <sup>-1</sup>	3919 - 3672 cm <sup>-1</sup>			
		3479 cm <sup>-1</sup>	3479 cm <sup>-1</sup>			
CH <sub>2</sub> ; CH <sub>3</sub> Stretch	2924 cm <sup>-1</sup>	2962 - 2769 cm <sup>-1</sup>	2769 cm <sup>-1</sup>			
C - O Stretch	1550-1600 cm <sup>-1</sup>	1550 cm <sup>-1</sup>	1635 cm <sup>-1</sup>			
R-CO <sub>2</sub> band			1658 cm <sup>-1</sup>			

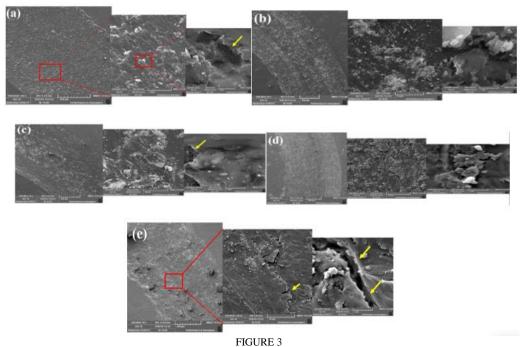
TABLE 4



AVERAGE COEFFICIENT UNDER APPLIED LOADS (A) PMMA/ PWAX, (B) PMMA/ BWAX, AND (C), PMMA/CS

## 2. Friction Under Self-lubricating Environmental Conditions

Figure (2) shows the friction coefficient curves changing with the applied force of the Pure PMMA and PMMA based a variety solid lubricant materials. In general, the presence of the solid lubricants greatly reduces the coefficient of friction, as shown in Figure (2,a) a decrease in the friction coefficient of PMMA/ Pwax (0.5 wt. %) at (5 N) from 0.55 to 0.45 and this agreed with [26] [27]. While Figure (2,b) shows the friction coefficient change with the normal forces for PMMA and PMMA based Beeswax lubricant and the results show that the average coefficient was decreased from 0.55 for pure PMMA to 0.1 for PMMA/0.1Beeswax, and this may be due to increasing the ability of the migration of the beeswax additive, which causing a significant decrease in the coefficient of friction. Figure (2, c) shows the effect of the presence of Calcium Streat with different ratios on reducing the coefficient of friction of PMMA. It can be seen that the friction coefficient was decreased significantly from 0.55 for pure PMMA to 0.1 for PMMA/CS (0.2 wt.%) at normal load 5N which agreed with [28]. In terms of increased corrosion resistance and associated mechanical properties, calcium stearate was found to be the most effective micro-filler for high molecular weight matrix, which reduces the surface temperature of the sample (the final product). Observations of worn surfaces by SEM provide knowledge about both, the role that lubricant solids play in the reduction of wear rate, and the acting wear mechanisms. Fig.(3) shows the worn surfaces of the PMMA and PMMA/Pwax composite filled with additional solid lubricant tested under normal load 12N, the worn surfaces of the 0.1 and 0.2 composite were much smoother compare with the worn surface of pure PMMA composite. As shown in Fig. 3(b and c) where the worn surface of pure PMMA showed a clear plastic deformation due to adhesive nature, breakage of the Pure PMMA matrix occurs at the interface Fig. (3,a). With increasing the weight percentage of Pwax to 0.5% caused increase in wear rate due to a loss of mechanical properties because of solid lubricant materials assisted softening polymeric.



DETAILED MORPHOLOGY OF WEAR SURFACE OF PMMA FOLLOWING MICRO ABRASION UNDER (12N) LOAD AS A FUNCTION OF SLIDING DISTANCE: (A) PURE PMMA, (B) PMMA/PWAX1, (C) PMMA/PWAX2, (D) PMMA/PWAX3, (E) PMMA/PWAX4

As shown in Fig. (4) where the worn surface of PMMA/Bwax showed a clear enhancement in wear resistance. The resulting surface is smoother and this indicates that the lubricating materials reduce the intermittent shear stress and thus reduce the adhesion friction compound and this confirms the ability of beeswax to migrate through PMMA structure to the interface and decreasing the coefficient of friction and therefore improve wear resistance.

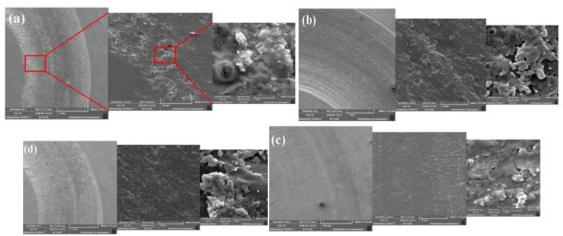


FIGURE 4

DETAILED MORPHOLOGY OF WEAR SURFACE OF PMMA FOLLOWING MICROABRASION UNDER 12 N LOAD AS A FUNCTION OF SLIDING DISTANCE: (A) PMMA/BWAX1 (B) PMMA/BWAX2 (C) PMMA/BWAX3 (D) PMMA/BWAX4.

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On the other hand, the surface of the worn composites containing CS (Fig5) a clear enhancement in wear resistance. The resulting surface is smoother and this indicates that the lubricating materials reduces friction but does not protect against abrasive particles Since calcium stearate is relatively soft in corrosion cannot save the matrix from the influence of fixed abrasive particles, as in UHMWPE-CS compounds which are not suitable for use in corrosive conditions [29].

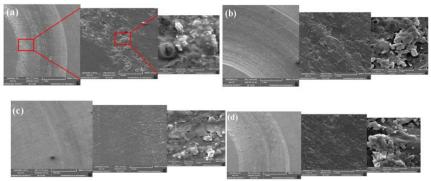
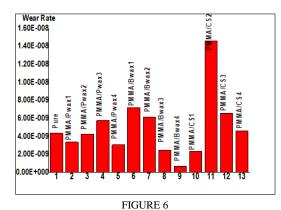


FIGURE 5

DETAILED MORPHOLOGY OF WEAR SURFACE OF PMMA FOLLOWING MICROABRASION UNDER 12 N LOAD AS A FUNCTION OF SLIDING DISTANCE: (A) PMMA/CS1 (B) PMMA/CS2 (C) PMMA/CS3 (D PMMA/CS4

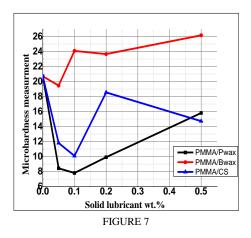
## 3. Wear Rate

After calculating the wear rate from the equation (3), from figure (6), it was found that the Bwax(0.5 wt.%) had a higher resistance compared to (Pwax,CS). The reason for this may be attributed to the distinct migration activity of the Beeswax towards the surface and at the same time it left the bulk without affecting it.



EFFECT OF APPLIED LOADS ON WEAR RATE FOR PURE PMMA,

SULF-LUBRICATED PMMA AT CONSTANT SLIDING TIME OF 180 MIN



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#### MICRO HARDNESS MEASUREMENT SCHEME

## 4. Hardness Test Results

Vickers hardness test method is an accurate micro hardness test to measure the resistance offered by the polymer composite material when the load applied over the surface area of indentation. Vickers Pyramid number (HV) or Diamond Pyramid hardness (DPH) is the unit for measuring the Vickers hardness number. Readings were taken on well-polished samples immediately after the indentation made as it highly depends on the elastic recovery of the material and surface homogeneity. The results showed that the presence of solid lubricants Paraffin wax and calcium streat has a slight effect on decreasing the hardness of PMMA, while in the case of beeswax there is no real effect on the PMMA hardness, Fig. (7).

## 5. Hydrodynamics of Hydrophobic Surfaces

Through the Fig. (8), an increase in the contact angle value was observed for all samples that used a solid lubricant with it, but with varying proportions, as we notice the samples filled with beeswax that had a high contact angle with water compared to the rest of the types, and the reason for this may be due to the difference in the ability and rate of migration of particles Solid lubrication to the surface, where the results proved that the Beeswax had a high percentage on the surface. This behavior was reflected in most of the surface properties of the composite material that were previously discussed.

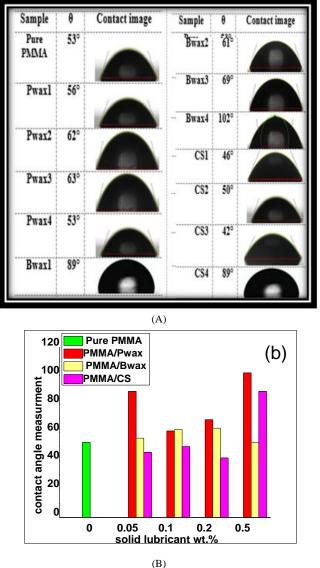


FIGURE 8 (A) CONTACT ANGLE OF EXPERIMENTAL RESULT (B) CONTACT ANGLE SCHEME

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## CONCLUSION

In the current research, three different types of solids lubricating with PMMA were used during the in situ polymerization process, respectively (Pwax, Bwax, CS) to improve the tribological treatment performance of PMMA, and the results were:

- 1) The results of the wear coefficient for the resulting PMMA/Pwax composites showed an improvement in the rates of friction coefficient which decreased to (0.8%), PMMA / Bwax decreased to (0.7%), and PMMA / CS decreased to (0.8%).
- The Wear rate test results for PMMA/Bwax4 composites showed an improvement in the corrosion rates which decreased to (0.6%).
- PMMA / Bwax showed improvement in contact angle tests for resulting composites and achieved higher contact angle values for hydrophobic surfaces.

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