Tribological and Rheological Investigations of MWCNT/PMMA Polymeric Composites and Hybrid Compounds

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Abstract

The Nano scale self-lubricating compounds were prepared by in-situ free radical polymerization with the dispersion of MWCNT ultrasonic molecules embedded in the polymer matrix to enhance the tribological mechanical properties as well as the production of hybrid compounds to detect the tibial changes on the polymer surface, dry friction under the conditions of slip was studied, the friction coefficient was found For hybrid compounds slightly lower than the coefficient of friction of Nano composites under multiple loads and constant velocities, enhancements of Nano composites in rheological behavior were investigated and the influencing factors were determined: shear stress, shear viscosity, as well as shear rates. By using visual inspection, tracking of the behavior of compounds during extrusion was carried out and obtaining the shapes of the threads resulting from extrusion, which are usually important matters whose results depend on many industries.

Key Words: In-situ Free Radical Polymerization, Friction, Melt Fracture, Surface Quality.

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Introduction

Tribology contains special criteria for friction, wear resistance and lubrication of slippery surfaces. Here, the role of nanotechnology and its great impact on design, reducing the rate of friction, and increasing the wear resistance are highlighted (Reddy, 2011) (Rogers et al., 2014) (Choudhury et al., 2013). The use of nanomaterial's promotes extending the life of systems, prevents chemical and / or mechanical damage and controls temperature and moisture transfer (Delogu et al., 2017) (Davim, 2013). Nanotechnology has been used in many fields such as materials and manufacturing, Nano electronics, medicine and engineering, energy, and biotechnology, as well as food (Menezes et al., 2013) (Gnerre et al., 2010) (Gorrasi & Sorrentino, 2015). Lubrication is commonly used to reduce the friction and wear between two hard surfaces moving with a layer of lubricant, whether solid, liquid, or gas, while preserving the surfaces. Both friction and wear are very complex mechanisms. In older systems, many dynamic process machines are lubricated with fluids of various types. These fluids impede the optimum lubrication of the dynamic surfaces and leave traces, pollution and cost of the system, and it suffers from corrosion in high temperature conditions. Recently, there has been a trend for environmental systems that use self-lubricating oils to eliminate the problems associated with lubrication in the aforementioned systems represented by corrosive particles and dropping points from motor machinery as well as frequent and complex cleaning in some cases (Briscoe & Sinha, 2008).

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As for self-lubrication in terms of rheology: rheology is a branch of physics that deals with the formation and flow of matter under pressure and temperature. Rheology tracks the properties of a substance (viscosity, molecular weight, particle size, etc.) that prevent it from acting under the influence of mechanical force or high temperature. Rheology differs from fluid dynamics by its interest in the three conventional states of matter rather than just liquid and gases. Important issues related to thixotropic properties appear in many different applications, for example as an additive that enhances the required flow behavior. Among these products, the organoclay, from the reaction of organic cations with smectite clays, is one of the most widely used solid lubricants for solvent-based coatings (Sadiku-Agboola et al., 2011). Among the rheological measurements (shear, extended viscosity, pressure, storage modulus, and loss modulus), which serve to understand the identity of materials and their ability to work (Nesaei, 2019). High molecular weight rheology has a critical position in characterizing materials, as well as molecular weight distribution, and subgroups. Flow measurements can be used to determine the boundary conditions by which problems are prevented before the material is introduced into the extruder (Subcommittee, 2006). Multi-walled carbon nanotubes are characterized by being concentric layers stacked of several layers (cylinders) of graphene coiled with equal spacing (0.34 nm) (Al-amshany et al., 2019). The characteristics of carbon nanotubes were reported and largely dependent on size, morphology, and diameter. Carbon nanotubes are produced using various methods: arc vaporization, laser ablation, chemical vapor deposition, electrolysis, etc., carbon nanotubes are very promising as fillers and stiffeners for polymer-based compounds due to their generally better and more important structural and functional properties. It is the high aspect ratio, high mechanical strength, and high quality electrical properties (Al-amshany et al., 2019) (Mittal et al., 2015). Polymer / CNT compounds are characterized by their dependence on the dispersion of the carbon nanotubes in the matrix, the interactions between the carbon nanotubes and the polymer, and affect the performance of the carbon atoms on the walls of the chemically stable carbon nanotubes due to the aromatic nature of the van der Waals bond and because of this, the CNT reinforcements are considered inert so the transport is the effective load across the CNT / Matrix interface is very weak. And to enhance the bonding between CNT / polymer (P. Ma et al., 2010), the orientation towards modifying the surface properties of the carbon nanotubes by activation includes chemical activation, which enhances the covalent bonding of functional groups on the surface of the carbon nanotubes, or it is a physical activation, where the non-covalent interactions are formed between the surface of the carbon nanotubes and the molecules used to modify the properties. Interlayer (Jatti & Singh, 2015). Researchers' interest has focused on developing organic nanomaterials for many applications due to their unique properties such as semiconductors, catalysts, optical properties, friction reduction, reduced magnetism (Sukkar et al., 2019), lubrication of moving parts, system cleaning, anti-wear and improved performance and engine cooling (Shen et al., 2001). Lubricant oil is the basis in the final product specifications of the materials as in adding nanoparticles of copper oxide (CuO) and titanium oxides (TiO_2) in SAE 15W40 motor oil to produce high-quality properties, coupled with revealing the thermal conductivity, stability, and viscosity properties of the nano-lubricants., Pour point and flash point as quality criteria, so the CuO particles showed better function and effect on the motor oil (Sukkar et al., 2019). Nanofluidics are one of the most important applications of nanotechnology in many scientific and engineering techniques when adding nanoparticles to any liquid that produces a nanofluid. When added to a lubricant, the addition of nanoparticles to the base oil (nanofluids) is formed (Shen et al., 2001) (S. Ma et al., 2010).

Material and Method

The methyl methacrylate (MMA) (≥99.5%) monomer as liquid, the benzoyl peroxide as a white granules, MWCNT (Mw=12.01, appearance: black powder, Tm= 3550 °C, Bp= 4027 °C, Average Particle Size= <50 nm, Specific Surface Area= >100 m2/g (BET), and Bees wax ($C_{15}H_{31}COOC_{30}H_{61}$, Tm = 75°C were all supplied from the local Iraqi market.

1. Spicemen Preparation

The PMMA matrix was fabricated by an in situ polymerization system, consisting of BPO powder and liquid MMA. In the following experiments, MWCNTs were selected at ratios (0.05, 0.1, 0.2, 0.5 wt%) to study the effect of nanofillings on the tribological performance of PMMA compounds. The PMMA and BPO powder were kept in a desiccator for 24 hours for dehumidification before use. The liquid mixture was MMA with 100 g and BPO 0.9% by weight as a basis, thus starting the polymerization reaction of PMMA with the action of PBO and after mixing evenly and before treatment, the pure PMMA obtained from the polymerization molds was formed using glass sheets (20 cm x 20 cm) with a length (15 cm A rubber tube, four paper holders, the glass sheets are met and sealed by the paper holders, the material is injected into the mold with a syringe, the mold was inserted into the oven for 8 hours at a temperature of 85 ° C. In view of the difficulties facing the scientific researcher in the practical aspect when dealing with (MWCNT) in obtaining the optimum diffusion and the formation of polymers / MWNCT, therefore, in this research, the surface of MWCNT was treated in two ways: **First:** Sulfuric acid (H_2So_4) and Nitric acid (HNo_3) were mixed in a ratio of [1:3] respectively, adding MWCNT to it and placing the component in Ultrasonic Bath for 30 minutes and neutralizing the acidity of the solution to reach [PH=7]. An oven was brought to a temperature of 100 and the component was heated for 3 hours and the component milled manually، MWCNT Was used with proportions (0.05, 0.1, 0.2, 0.5 wt%) using in-situ free radical polymerization and Ultra-sonication technique for best dispersion and finally production of a PMMA/MWCNT. The hybrid compound consists of more than two materials bearing the characteristics of each substance included in its composition and in this paper a self-lubricating hybrid compound was produced by mixing (50 wt% wax + 50 wt% PMMA / MWCNT) and mixing the mixture with acetone as a buffer, Ultra-sonication technique for best dispersion, then it is brought into Oven at 100°C then the component is milled by hand and used in the same proportions as PMMA / MWCNT.

Table 1. Nano-Polymeric composites and Hybrid compounds

N	Sample Code	Sample Composition
1	Pure PMMA	100 g wt. % MMA + 0.9 wt. % PBO
2	PMMA/MWCNT1	PMMA + 0.05 % wt. PMMA/MWCNT
3	PMMA/MWCNT2	PMMA+ 0.1% wt. PMMA/MWCNT
4	PMMA/MWCNT3	PMMA+ 0.2 % wt. PMMA/MWCNT
5	PMMA/MWCNT4	PMMA+ 0.5 % wt. PMMA/MWCNT
6	Hybrid1	PMMA+ 0.05 % wt. Hybrid
7	Hybrid2	PMMA+ 0.1 % wt. Hybrid
8	Hybrid3	PMMA+ 0.2 % wt. Hybrid
9	Hybrid4	PMMA+ 0.5 % wt. Hybrid

Hybrid	PMMA/ MWCNT	Pure PMMA

Figure 1. Samples produced by in-situ polymerization

Reinforcement Filler (PMMA/MWCNT) Behavior & their Effect on Tribology

A Pin-on-disk Instrument evaluated dry friction and wear properties of unfilled PMMA, PMMA/ MWCNT and Hybrid. A Aluminum ball which fixed in a pin-shaped fixture was used to slide against each sample. The diameter of the ball (10mm), temperature during tests: RT, applied force range: 1

to 12 N, distance: 500 m, and speed: 110 rpm. The dimension of the samples for wear test was (1 mm width, 2mm long, 1.26 thickness). All samples were prepared to get smooth surface. Each experiment was repeated, At the end of each test, the evolution of the friction coefficient with the sliding distance was measured directly by the CSM tribometer. Wear coefficient of friction illustrates as shown in figure(2). This study investigated the effects of carbon nanotubes on tribological performance under dry and slippery conditions and under dry friction. It was observed that carbon nanotubes act as a solid lubricant, and under lubrication conditions, it could be classified as a type of lubricant additives. Under dry slip conditions, the addition of carbon nanotubes can lead to a significant reduction in friction and corrosion, carbon nanotubes formed a carbon layer covering the contact surfaces as well as due to the low shear property of carbon nanotubes, the friction coefficient of (PMMA/MWCNT 4) was decreased (at 6 N from 0.6 to 0.34 and at 12 N from 0.7 to 0.31). While the friction coefficient of (Hybrid 2) was decreased (at 5 N from 0.56 to 0.1 and at 12 N from 0.7 to 0.37) Under dry friction as shown in figure (2,a,b), the effective time of MWCNTs is limited, and the coefficient of friction decreases as the MWCNTs content increases. The wear mechanism under dry friction is mainly adhesive wear.

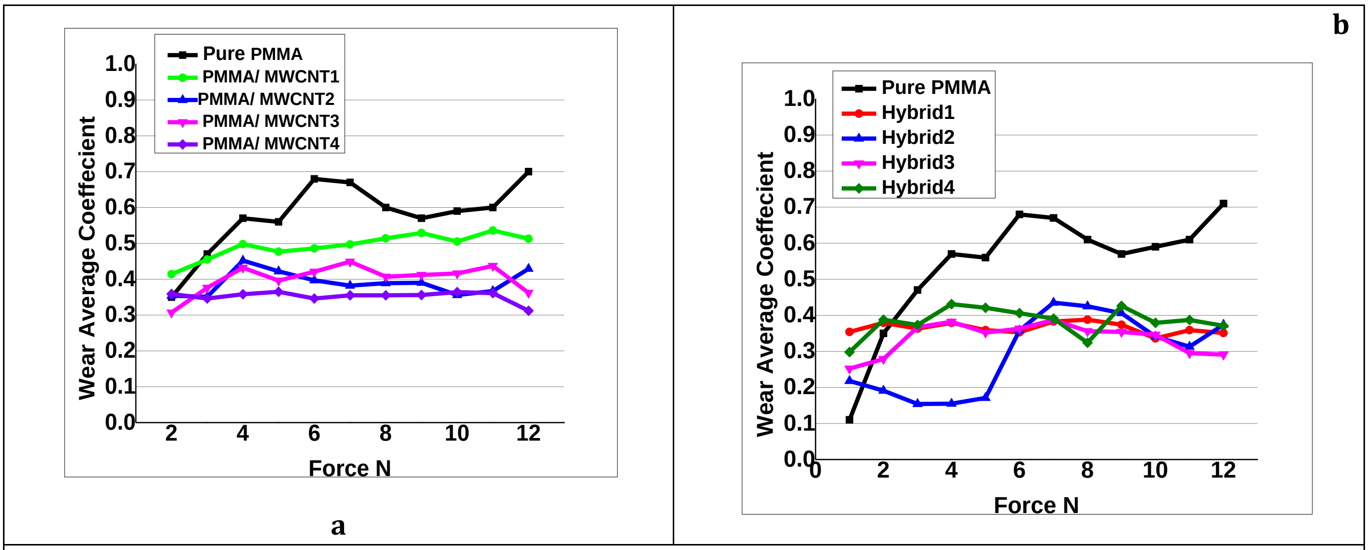


Figure 2. Effects of Wear Coefficient friction with Different PMMA/MWCNT wt.% And Loads

1. The Effect of In-Situ Free Radical Polymerization

In situ polymerization consists of chemical reactions that generate phases of stiffening at the molecular level and the compound is dynamically stable within the matrix structure, wherein the nanoparticles are dispersed using ultrasound in a liquid monomer and an initiator is added using a suitable heat source that produces a thermoplastic nanocomposite, and the surfaces are The nanomaterial-reinforced polymers are free from contamination, by adding CNT to the pre-polymer with relatively low viscosity, achieves successful dispersion, and the polymerization is described by its progression. The polymer grows around it, wraps the carbon nanotubes, forms covalent or non-covalent bonds that are stronger than nanocarbon and produces compounds enhanced with mechanical, electrical and tribological properties (Zhang et al., 2012). A polymer was taken as a host or a polymer mixture that contains a host polymer and combine additives chemically identical or compatible with the host polymer and the polymer or other polymers compatible with the host polymer to impart the changing surface properties due to spontaneous surface separation or due to flow-induced migration. The additives must be in the form of a branched molecule of low molecular weight, and an optional nucleus or final groups must be present. Using a termination tool we add end groups of functional branches to the arms or branches attributed to the additive. And it is chosen based on compatibility with the job group added to the end of the branch. For example, a silane alkyl separator containing one or more fluorine, bromine, or amine hydroxyl groups will be selected, respectively, to impart the functions of fluorine, bromine, amine or hydroxyl at the end of the branch (Archer & Qian, 2011).

2. Hybrid Compounds Characteristics

Hybrid compounds are materials that are manufactured by combining two or more different types of materials within one common matrix. There are several definitions of hybrid compounds for describing them as a strengthening material incorporated in a mixture of different matrices or as a material that is strengthened by incorporating it into two or more additives and packaging materials present in one matrix. Hybrid compounds occupy a more advanced position than fiber-reinforced compounds and have a wide range of desirable applications. An accurate description of the performance of the hybrid compounds is the sum of the individual components with a more consistent balance between the inherent advantages and disadvantages. Hybrid compounds can supplement the benefits of one type of properties lacking in the other type (Jamir et al., 2018). Polymer nanomaterials can be arranged over a wide range into the following classes: fibrous, laminar, and particle. Nanomaterials either with amorphous or crystalline of 1D, 2D and 3D structures are made by encapsulating fine or nanoparticles in an organic polymer matrix, or inorganic metal / metallic oxides or ceramic material, accordingly the physical and chemical properties of the materials are subject to change. It has been reported that organic-inorganic and inorganic-based hybrid polymer nanomaterials possess thermal, mechanical, electrical and electrochemical properties that outweigh separate properties (Pielichowski & Majka, 2018).

The Rheological of PMMA/ MWCNT

Capillary rheometer is used in industry and in academia to evaluate the rheological performance of polymer molten at high shear rates before treatment in industry, as shown in Fig. (3). Among the causes of polymer flow in capillary flowmeter: First, "represents Capillary flow in any mold forming flow: inlet, fully developed sites, and exit site points. Second, the study of capillary flow to aid in solving problems of inlet pressure drop, swelling, and molten fracture. "Should be taken into account (Tadmor & Gogos, 2013). Apparent shear viscosity from capillary flow from The derivation of the Hagen- Poiseuille law for capillary flow (Thomas, 1962):

$$\tau_{app.} = \frac{R}{2}\frac{P}{L} \qquad (1)$$

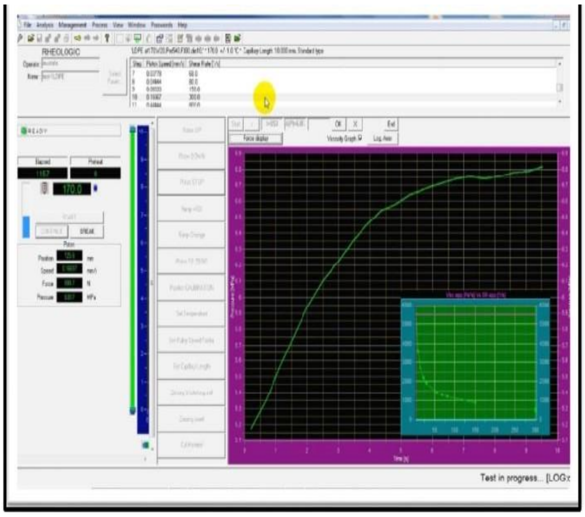
$$\gamma^{\cdot}_{app.} = \frac{4\,Q}{\pi R^3} \qquad (2)$$

$$\eta_{app.} = \frac{\tau_{app.}}{\gamma^{\cdot}_{app.}} \qquad (3)$$

Where: P= the pressure drop (N/m2);
Q = the volume flow rate (m3 /s);
R = the capillary radius (m); and
L = the capillary length (m).

Specifications	***Units***	***Value***
Maximum Force Range	KN	20
Barrel Type	——	Single Bore
Barrel Working Length	mm	290
Barrel Working diameter	mm	15
Standard capillary diameters range	mm	0.25 up to 2
Temperature Range	°C	50 - 500
Temperature Accuracy	°C	± 0.2
Force Transducers	KN	1 - 20
Pressure Transducers	MPa	3.5 -200
Weight (without options)	Kg	130
Shear rate range	s^{-1}	0.1 – 36000
Preheating range	s	1 - 3600

A



B

Figure 3. A) Specifications of the capillary rheometer SR20, and, B) Software Program to Run the Capillary Rheometer

Capillary rheometer device (INSTRON CEAST, Country of Origin Italy), which is available in laboratory of college of materials engineering/University of Babylon, as shown in Fig.(4). Capillary rheometer systems are built in accordance with ISO 11443, ASTM D3835; international standards for rheology testing. This capillary rheometer simulates the process conditions, measuring the plastic materials flow behavior that characterizes the rheology of materials through several capillary die Control the device by a software program enables users to run the capillary rheometer. It provides the complete flow curve of tested materials and comparison charts for material curves reference. Customized data export allows easy pst-processing analysis and feeding for software simulation programs. The 'flow curve', which is a graph showing the apparent shear stress and apparent shear rate, is widely used for the rheological composition of the polymer in the molten state. Here, discussions focus on the phenomenon of cracking and sliding, which may include friction and fracture. The use of apparent shear stress and apparent shear rate will be low in characterizing friction and fracture. Therefore, the Tarst curves for quantification are as follows: pressure (P) at the inlet of the mold versus the external velocity of the mean (V), the relationships of average extrusion velocity (V) from the volumetric flow rate Q are found to determine the flow in the circular die (Using & Rheometers., 2014):

$$V = \frac{4\,Q}{\pi\,d^2} \qquad (4)$$

Where d is the diameter of the die.

The apparent shear stress (τ_{app}) can be calculated as:

$$\tau_{app} = \frac{4L}{d}P \qquad (5)$$

Where (L=Length of the die), And the apparent shear rate is given:

$$\dot{\gamma}_{app} = \frac{32\,Q}{\pi d^3} = \frac{8V}{d} \qquad (6)$$

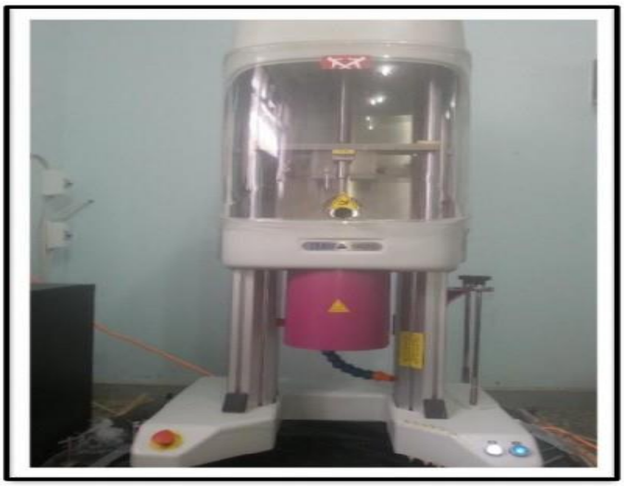


Figure 4. Capillary rheometer SR20 device

Fig. (5) shows the flow curve of pure PMMA,PMMA with MWCNT and Hybrid it seems that the use of these MWCNT, Hybrid cause a decrease in the extrusion press. The trend of the shear viscosity of PMMA with MWCNT initially followed the typical pseudo-plastic shear-thinning behavior and the shear viscosity decreased significantly, when the shear rate increased from approximately 50 to 400 s^{-1}, then change in the shear viscosity tended to be flatter, in contrast Hybrid[1], Hybrid[2] showed gross melt fracture from 50 to 400 s^{-1}, Hybrid3, Hybrid4 showed flatter shear viscosity from 50 to 900 s^{-1}. During the sonication process, there is a break and fluctuation in the polymeric chain, so the molecular weight increases, the viscosity and the shear stress increase accordingly. As the chains are broken up, the molecular weight distribution tends to be the broad molecular weight distribution.

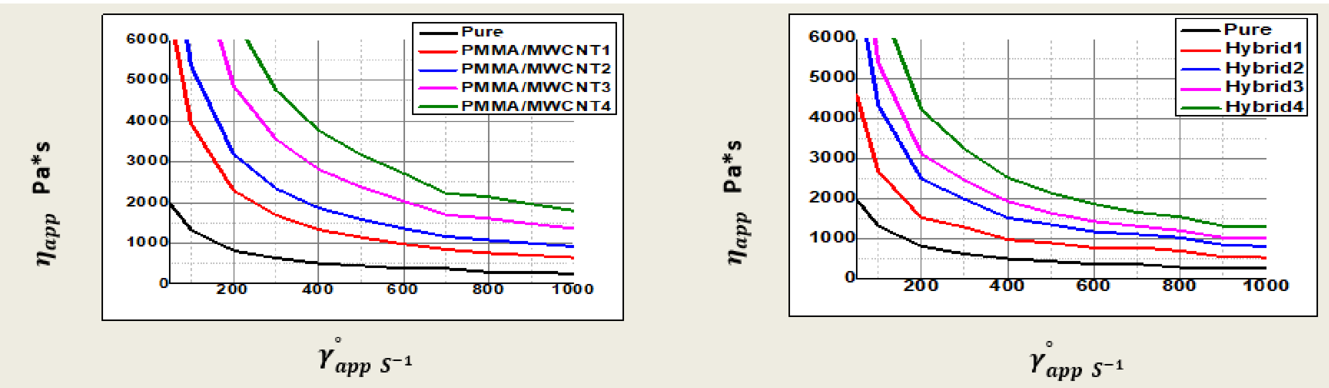


Figure 5. Shear viscosity behavior of Pure PMMA melt, PMMA/MWCNT melt with the shear rate increasing in a capillary at die 1 mm 240°C (a) PMMA/MWCNT (0.05,0.1,0.2, and 0.5 wt.%), (b) Hybrid (0.05,0.1,0.2, and 0.5 wt.%)

The shear stress behavior of PMMA / MWCNT is obvious from the figure (6,(a)) from 100 to 800 s^{-1}, Shear stress increased with shear rate increased, shear stress tend to be flatter. While shear stress behavior of hybrid causes turbulent extrusion lead to a variety of the elastomeric effects. Hybrid shear stress curves are perfectly continuous with change in the slope around hybrid 3, hybrid4 from 50 to 800 s^{-1} as shown in fig. (6,(b)). The additives cause a decrease in the viscosity and thus a decrease in the processing temperature. In the case of primary stable extrusion, wall stress is evaluated based on adhesion between capillaries and fluid, which is not dependent on the emission pressure. The first critical stress (w1) is formed by the sudden increase of slope. When the shear stress increases, shark skin is observed on the extruded material, which is a periodic phenomenon of High frequency that expands with increasing wall shear stress and flow rate, and can be observed to arise from the slip and uniaxial expansion occurring at the exit of the die (Mackley et al., 1998). The third region after the second critical pressure (W2) is the sliding stick, where two distinct topologies invert the shape of the extrusion that is smooth lustrous (slip), and it can be defined as the single instability between a polymer extrusion adopted by pressure oscillation at a constant transfer rate (Taliadorou et al., 2007), Surface instabilities in extrusion, significance and impacts in industry have been reported. In fact, the emergence of shark skin or the cyclical 'sticky slip' effects of extrusion mixtures arises with each increase in production rates and results in a maximum rate of processing in most processing stages based mainly on the extrusion of polymeric materials and suspensions (Birinci & Kalyon, 2006), The wavy fracture zone we observe the effects of the total extrusion deformation which is the last zone, whose effect is more severe than that of a shark skin with a fracture depth equal to the diameter of the extrusion. The initiation mechanisms are related to the convergent flow dynamics at the inlet of the template(Kim & Dealy, 2002).

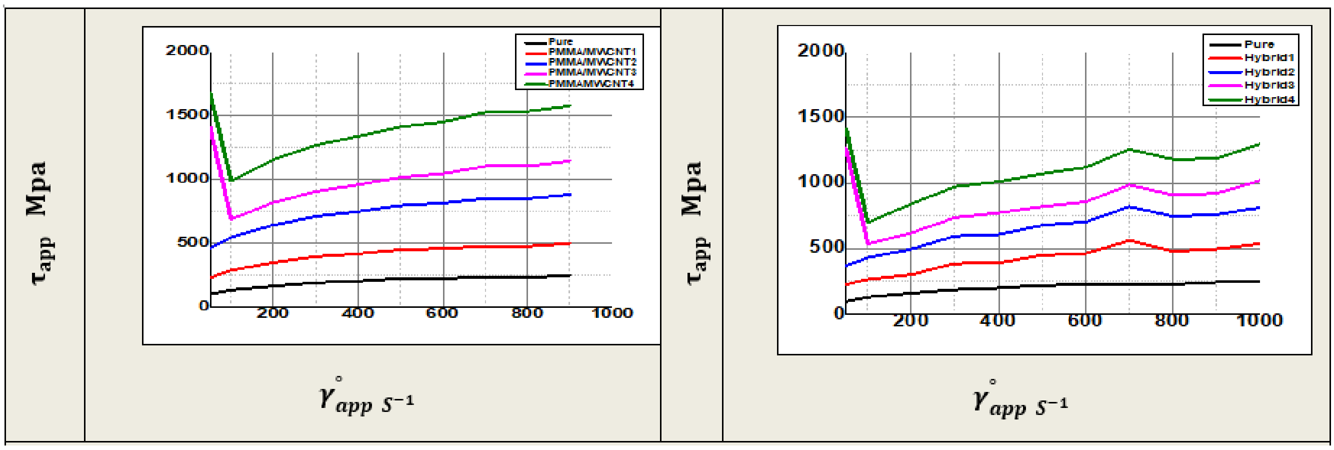


Figure 6. Shear stress behavior of Pure PMMA melt, PMMA/MWCNT melt with the shear rate increasing in a capillary at die 1 mm 240°C (a) PMMA/MWCNT (0.05,0.1,0.2, and 0.5 wt.%), (b) Hybrid (0.05,0.1,0.2, and 0.5 wt.%)

Polymers are subjected to shear stress in curing processes, and the polymer long chain suffers from deformations. Particles tend to turn due to shear stress and to turn again at the end of the shear process. The rewinding process (sometimes known as relaxation) may not be completed in the event of rapid post-shear cooling. These unwinding / rewinding operations lead to a variety of phenomena commonly known as the elastomeric effects: swelling and melt fracture. Extrusion deformation arises when the extrusion process occurs at high rates. Then the shear rate exceeds the critical shear rate to break a particular polymer melt that is initially in the mold entry region when the material is directed into the capillaries from the die reservoir. Where the melt movement is in the part parallel to the mold. Other effects occur on the die wall, as the melt fracture phenomenon occurs when extruded through the small diameter die with high shear rate, Process occurs at high rates. Then the shear rate exceeds the critical shear rate to break a particular polymer melt that is initially in the mold entry region when the material is directed into the capillaries from the die reservoir. Where the melt movement is in the part parallel to the mold. Other effects occur on the die wall, as the melt fracture phenomenon occurs when extruded through the small diameter die with high shear rate (Kay et al., 2003). In general, there is an instability that may occur in the capillaries Pure PMMA, PMMA / MWCNT and Hybrid extrusion. First, there is a critical shear stress in which low-amplitude periodic deformations exist on the extrusion surface causing surface melting fracture. When high shear stress is present, the flow creates massive deformations that appear on the material surface upon extrusion known as the total melt fracture. The polymer shows diffuse defects on the surface, the real problem being the inability to have simple quantification of the turbulent nature of extrusion. These observations can only be recorded on the basis of the visual aspect and the accurate description indicating the increasing degradation with an increase in the flow rate (shear rate). In all cases, irregular pressure at an extremely high flow rate (shear rate) causes turbulent extrusion. It can be seen whether the flow is stable or unstable through the extrusion profile of the molten polymer through a die: at a low flow rate the flow is smooth, the extrusion is smooth and its cross section is constant, in this case the flow is quite stable. However, after the critical flow rate, the flow becomes unstable and extrusion defects abound, affecting its surface or volume(Kay et al., 2003). These instabilities are generally defined under the term (dissolving fracture) (Schreiber et al., 1960). There is more than one way to verify the instability of the polymer melt flow, namely, firstly, the experimental method, and secondly, the theoretical method. The experimental method includes an indirect approach that represents the classical rheology and the resulting experimental data are recorded as: shear stress, pressure, shear drag and flow rate during the individual instability test, and based on these data results for the treatment condition, the materials are studied. However, this approach includes inadequate basics for investigating entry flows due to their persistent disappearance within the extrusion matrix. The second method - includes the direct direction of optical measurements, which gives the perception of kinetic motion (velocity, deposited pressure) by the interaction of light with the flowing liquid, but the integration of the appropriate optical visualization system with the flow field without affecting it. (PTV), laser Doppler velocity (LDV) and flow refraction (Manual, n.d.).

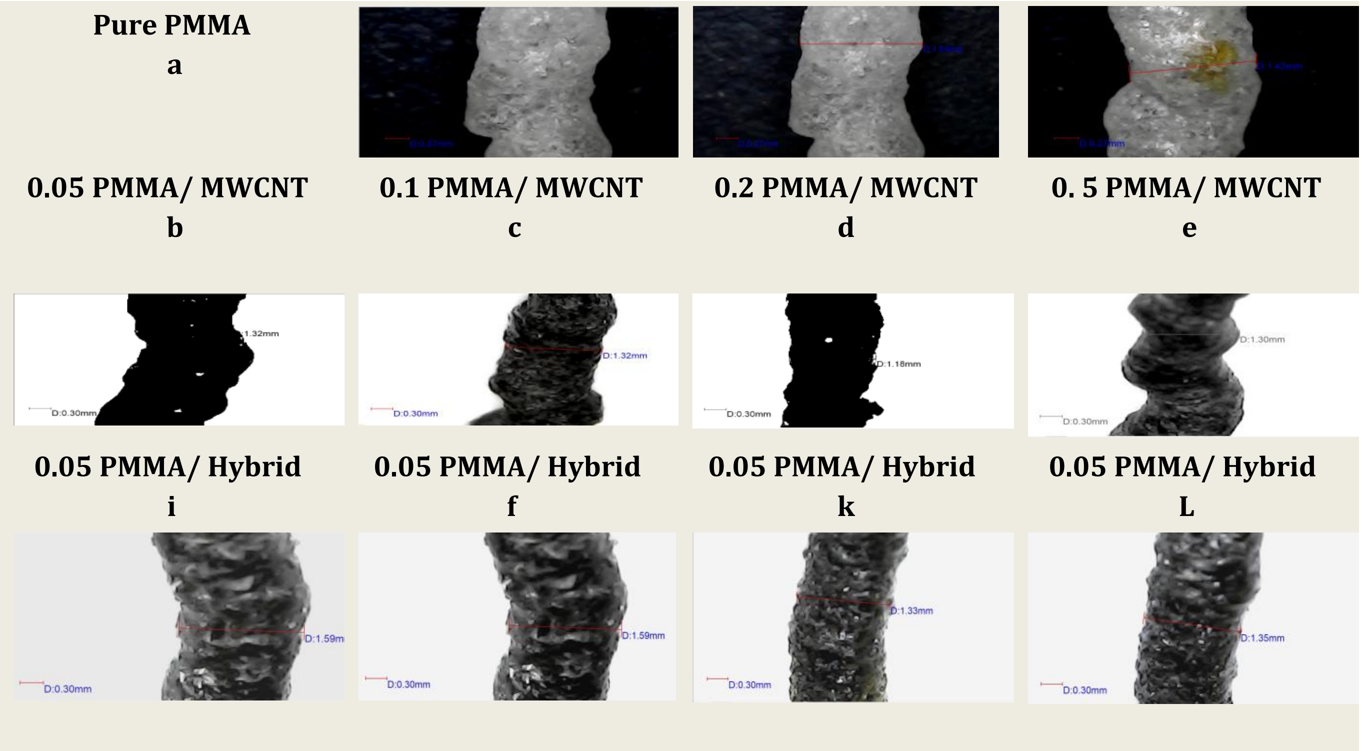


Figure 7. Example of Extrusion Defects:(a,b,d,e) Volume Defects,(c,k,L), and (i,f) Sharkskin

Conclusion

The major conclusions on the basis of experimental results are as follows:

1. In-situ free radical polymerization is quite acceptable technique for producing pure PMMA, PMMA loaded with solid lubricant and nanoparticles.
2. The Wear coefficient test results for PMMA/MWCNT & PMMA/Hybrid showed an improvement in the corrosion rates which decreased to (0.7% and 0.8%) respectively.

3. The flow of PMMA/MWCNT and Hybrid compound results in capillary rheometer showed that the shear rate increases with the shear viscosity decreasing and increases with shear stress increasing.
4. Determine the critical area to be able to change the surface quality and relate its results to pressure, shear stress and shear rate. This provides a determination of the appropriate boundary conditions for a capillary thermometer or polymer processing machine to produce the ideal surface quality, and the characteristics of the final product to save time, effort and energy.

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