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Wear characterizations of POM reinforced with carbon nanotubes (POM/CNT) using paraffin oil dispersion technique

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Abstract

Wear of polyoxymethylene (POM) is considered as its key design parameter of polymer gears and some mechanical applications and also controlled on service time span. This work investigates the influence of carbon nanotubes (CNT) on the specific wear rate of POM/CNT nanocomposites by using pin on disk test rig (sliding only). The CNT were synthesized using a fully automatic machine via the arc-discharge multi-electrode technique and then dispersed in POM matrix to manufacture test specimens. The CNT weight percentages were varied within the range 0 - 0.03 wt.% in three different operating medium (air, distilled water and mineral oil). The wear mechanism was examined by microscopy. The mechanical and thermal properties of POM/CNT were studied using calorimetric analysis and by mechanical tensile testing. Thermal and mechanical properties were improved to an optimum CNT ratio of 0.02 wt.% due to the improvement in crystallinity of POM and decrease the fusion defects. The crystallinity degree increased by 7% and melting temperature also increased. The results further indicate that the specific wear rate (Ws) for CNT/POM containing 0.03 wt. % CNT in air and water medium was improved by adding 73% and 66% respectively compared to virgin POM. On the other hand, the mechanical properties tensile strength and Young's modulus increased by 31% and 29% respectively.

Key words; POM, Carbon nanotubes, Nanocomposite polymer, Paraffin oil Dispersion, Wear resistance, Mechanical tensile properties, Thermal properties.

1. Introduction

Polyacetals or polyoxymethylene (POM) are thermoplastic polymers widely used in mechanical engineering applications prone to high friction such like, polymer gears, seals, gear rotor pump, aerospace, biomedical and automobile elements. This is because POM has high mechanical strength and unique properties such as excellent abrasion resistance, low friction coefficient, fatigue resistance, noiseless performance, lightweight, oil-less conditions, mouldability, higher melting point and low cost [1-10]. As such POM is considered as a major class of tribopolymers widely used in wear applications.

Recent trends in tribopolymers research aims to improve to further improve their properties using nanoreinforcement. In the literature, several methods have been used for nanofiller dispersion to improve these properties and especially on uniform nanofiller dispersion during synthesis POM nanocomposites. For instance, Sun et al. (2008) used twin-screw extruder to

prepare POM/Al₂O₃ nanocomposites in order to study the effect of inorganic nanoparticles on tribological properties of POM nanocomposites [11]. Also, Leszczyńska and Pielichowski (2012) used twin-screw extruder to blend the ternary composition of POM/ thermoplastic polyurethane (TPU)/montmorillonite (OMMT) in two stages, and then studied the inference of nanofiller on the thermal stability and structure [12]. Das et al. (2014) used Brabender to mixed POM/clay nanocomposites at 215°C, screw speed of 50 rpm for 10 min, then the surrency was compression moulded in a hot press at 215°C under constant pressure (20 MPa) to produce POM/clay nanocomposites film [13]. These studies reported that the methods of dispersion employed gave a homogeneous dispersion and also improved the POM nanocomposites properties in general.

Carbon nanotubes (CNT) have been successfully utilized to increase the wear resistance of the polymeric material [14-16]. However, works on CNT/POM composites are scarce in the literature due to poor compatibility of POM with other materials [17]. Zhao et al. (2010 and 2011) used ultrasonic irradiation and HAAKE MiniLab twin-screw extruders to blend CNT by POM and produce a nanopolymer have homogenous dispersion. Additionally, they studied the structure, mechanical and thermal conductive properties. The result shows that, CNT significantly increased the mechanical properties and while excessive addition of CNT led to the poor dispersion and agglomeration of CNT in POM matrix, and decline of mechanical properties [17-19]. Jiang et al. (2014) dispersed CNT after modification in POM using twin-screw extruder to investigate the structural properties of a microinjection molded part and a conventional injection molded part for CNT/POM nanocomposite and its conductive properties were studied [20].

Our previous research studies focused on improving the dispersion of CNT/POM nanocomposite and we have consequently made some significant steps forward. In particular, Yousef et al. (2013) [**] manufactured CNT/POM spur gears produce by machined polymer nanocomposites. It was noted that the traditional methods for dispersion are not useful in this case, because these methods are expensive and need a long time to prepare the same, in addition decrease in the crystallinity of polymer nanocomposites after every process was noted. Furthermore, the study also developed a new method for CNT dispersion that used paraffin oil as assistance material during the injection process. The results showed that the new dispersion method was an effective way of improving the wear resistance especially for CNT/POM spur gears studied [21]. In a follow-up work, Yousef et al. (2014) refined the CNT dispersion using paraffin oil technique to overcome bubble formation and unmelted pellets problems. The study went on to synthesis a series of CNT/POM that included spur, bevel, helical and worm gears [22]. In the previous studies, the wear resistance of POM gears (rolling and sliding) was improved by adding 0.02 wt. % CNT. This work aims to continue the previous research studying the influence of added CNT on the specific wear rate of POM/CNT nanocomposites by using pin on disk test rig (sliding only).

2. Experimental

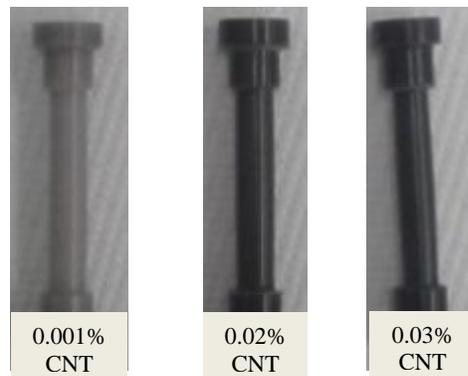
2.1 Materials and samples preparation

The CNT were synthesized using a fully automatic machine via the arc-discharge multi-electrode technique. The synthesized CNT had an average diameter of 10 nm and an average length of 2.5 μm [23]. CNT/POM composites containing 0 (POM), 0.001 (CNT/POM0.001),

0.01 (CNT/POM0.01), 0.2 (CNT/POM0.02) and 0.3 (CNT/POM0.03) wt% of CNT were prepared using an injection moulding die that was designed by the Yousef et al. (2014) to produce a short bars with a diameter of 40 mm and length of 78 mm. The POM powder (KOCETAL® K700) was supplied by El-Slam Company, Cairo, Egypt. Paraffin oil was used to adhere the CNT with the acetal polymer pellets after stirring for approximately 5 min [22]. To produce the study specimens (flanges), the CNT/acetal pellets were firstly are fed into a 400-g capacity hopper. An electric heater was then used to increase the mixing chamber temperature to 175 °C followed by screw threading to push the melt granules along the heater. The liquid was finally injected into the molded die to form the short bar with a diameter of 45 mm and a length of 87 mm then cooling.

2.2 Mechanical testing of CNF/UHMWPE nanocomposites

Mechanical tensile properties of POM and the nanocomposites specimens were measured by a LLOYD LR 10K universal testing machine with a load cell 500 N and cross head speed of 10 mm/min. As shown in Fig. (1a), the standard specimens were prepared according to ASTM E8 standard also used in the previous studies [21]. All tests were conducted at the ambient temperature.



(a) Images of typical tensile specimens



(b) Images of typical CNT/POM wear specimens

Figure (1): Tensile and wears specimens

The POM/CNT nanocomposites test specimens were prepared as a square shaped columns (20 mm x 20 mm x 2 mm) using turning machine for the facing and milling machine for setting the four sides. The tested samples had 0, 0.001, 0.01, 0.02 and 0.03 wt.% CNT content as shown in Fig. (1b). The wear tests were conducted by using a pin-on-disc wear tester (schematized in

Fig. (2a)) at room temperature under dry sliding and (water and oil) lubricated conditions. The flat samples were tested using a steel pin at a linear velocity of 1 m/s, normal load of 12 N and duration of 120 minutes. POM/CNT nanocomposites samples were placed on the rotating disc with a stationary stainless steel pin that has end probe point diameter about 200 μm [24]. Before conducting the test, the samples were first cleaned using ethanol and then with distilled water at test completion. Each sample set was tested three times to measure the wear loss (by weight) using a high sensitivity electronic weighing balance with accuracy (10^{-4} gram) at small, medium and large wear track as shown in Fig. (2c).

For water and oil mediums, lubricant housing part was produced to keep the CNT/POM samples in the wet medium as shown in Fig. (2b). The specific wear rate for CNT/POM was calculated by the equation (I & II) [16]:

$$W_s = \frac{\Delta m}{\rho \times F_n \times L} \quad \text{Equation (I)}$$

$$L = \pi d \quad \text{Equation (II)}$$

where W_s ($10^{-6} \text{mm}^3/\text{Nm}$) is the specific wear rate, Δm (mg) is the mass loss of the specimen, ρ (g/ml) is the density of the specimen, F_n (N) is the normal load and L (m) is the total sliding distance and can be calculated by equation (III), where d (mm) is the wear track. Each sample was tested three times with different wear track (small, medium and large). The specific wear rates were calculated at each wear track and the average value recorded.

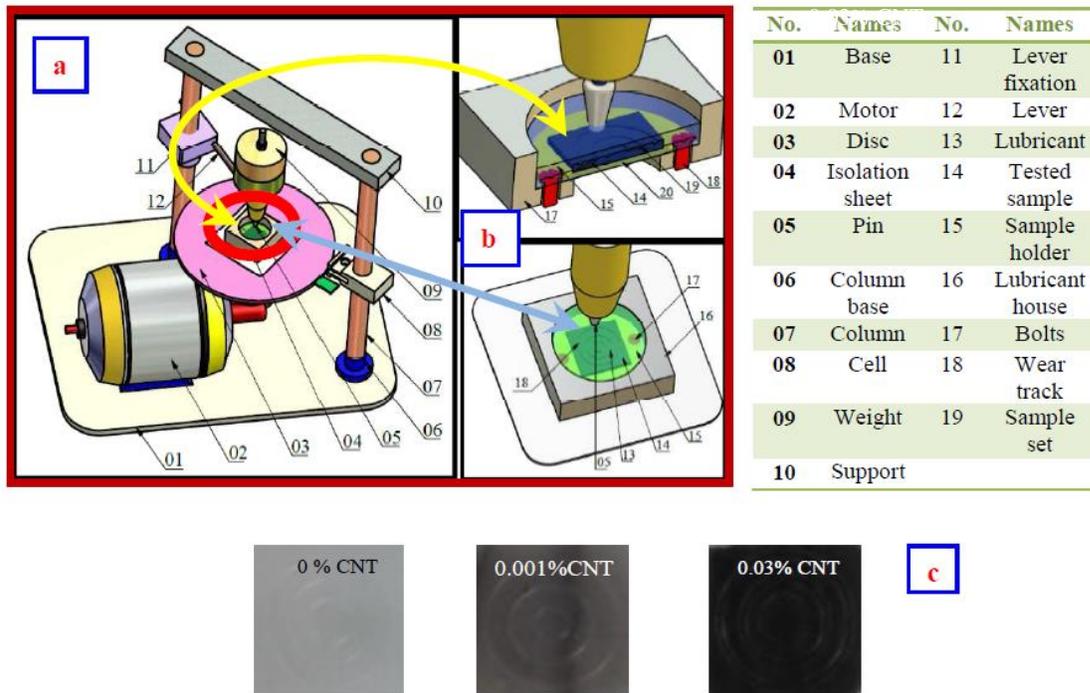


Figure (2): Pin on disc test rig and a resulting wears tracks a) Scheme drawing of pin on disc test rig after development, b) Lubricant house and c) CNT/POM wears tracks

2.3 Characterizations

The melting temperatures, onset temperature and apparent enthalpy of POM and CNT/POM were measured by Differential Scanning Calorimeter (TA INSTRUMENTS Thermo DSC Q 100 Thermo Model 970701.901 DSC). Firstly, the sample was heated until 250°C with rate of 10 °C per minute in nitrogen ambient, and then cooled by using a cooling water. The crystallinity can be calculated with the following equation (III);

$$X_c (\%) = \frac{\Delta H_c}{(1 - \emptyset) \Delta H_m^o} \times 100 \quad \text{Equation (III)}$$

Where ΔH_c is the apparent enthalpy of crystallization of sample, ΔH_m^o is the melting enthalpy of 100% crystalline POM and equal 326 J/g [25], and \emptyset is the weight fraction of CNT and paraffin oil in the POM composites at the ideal system, but in this case the amount of paraffin oil was very small compared to CNT, therefore \emptyset is the weight fraction of CNT only.

The density of CNT/POM composites was calculated using Archimedes principle. The samples were weighted in air and ethanol as an immersion medium using a high sensitivity electronic weighing balance with accuracy (10^{-4} gram). The following formula (V) has been used to calculate the density of CNT/POM [26];

$$\mathcal{P} = \frac{m(dry)}{m(dry) - m(wet)} \times \mathcal{P}(Ethanol) \quad \text{Equation (V)}$$

Where the (\mathcal{P}) the density of ethanol = 0.73 g/ml, m (dry) weight the sample in air and m (wet) weight the sample.

Scanning Electron Microscope SEM BPI-T was used to carry out SEM morphological investigations of the internal surface of the CNT/POM nanocomposites with 0.02 wt. % For the SEM investigations the samples were coated in vacuum with a very thin gold film to make them electrically conductive. The samples were then mounted on an aluminum stab with a conductive adhesive film. The electron acceleration voltage was of 10 kV and photos were recorded at different magnifications in order to have the scale of 5 μ m. Also, Electron Microscopy (EM-Hirox digital microscope KH 8700) was used to observe the wear mechanism of POM and it's composite.

3. Results and discussion

3.1 Physical and mechanical properties

The dispersion of CNT in POM has been examined using scanning electron microscopy (SEM) as shown in Fig. (3) and it is clear there is an excellent dispersion of CNT in POM (homogeneous dispersion) and strong cross linking.

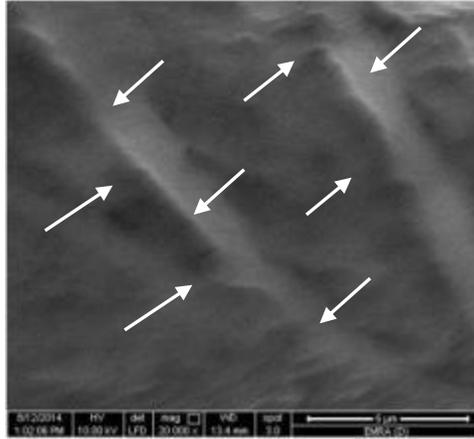


Figure (3): SEM image CNT/POM short bar

From **Table I**, it was evident that the density of POM and CNT/POM composites was nearly the same. It was noted that because the ratio of nanofiller material to the volume of nanocomposite was very small, there were no change in density. Also the addition of CNT did increase the melting temperature and crystallinity of CNT/POM as shown in **Fig. (4)** and **Table I** which indicated that CNT acted as effective heterogeneous nucleating agents to facilitate the crystallization of POM [16].

Table I. Density values and DSC parameters of POM and its nanocomposites

Sample code	Density (g/mL)	T_{Onset} (°C)	T_m (°C)	ΔH_c (J/g)	X_c (%)
POM	1.42 ± 0.0009	159.7	165.5	148	45.4
CNT/POM0.001	1.41 ± 0.0010	159.6	167.7	155	47.6
CNT/POM0.01	1.40 ± 0.0008	160.1	170.6	157	48.2
CNT/POM0.02	1.40 ± 0.0036	160.7	170.9	158	48.5
CNT/POM0.03	1.41 ± 0.0009	160.3	171.5	156	47.9

The mechanical property measurements of POM and CNT/POM composites (**Table II**) showed improvement of the mechanical properties of 0 and 1 wt. % CNT compared to that recorded in previous work [21]. The results indicated an increase in CNT/POM crystallinity (from 45.4% to 48%, a 7% improvement) led to increase in the tensile strength (from 45 MPa up to 58MPa, an improvement of 31%) and Young's modulus (from 1700 MPa up to 2200MPa, an improvement of 29%) hence enhanced materials performance. Moreover, the CNT nanofiller showed a higher ratio of the surface area to volume and lead to rapid interaction and more mixing between CNT and POM thus improved the mechanical properties until 0.02 wt.% CNT [27]. It should be noted that the CNT studied had a higher surface area to volume ratio. It is suspected that this lead to rapid interaction and more mixing between CNT and POM, thus improved the mechanical properties until 0.02 wt.% CNT mark [27]. The addition of paraffin oil to the nanocomposite during injection process led to decrease in the fusion defect during injection process which is advantageous [28]. As noted earlier, poor dispersion and agglomeration of CNT occur in POM and decline of mechanical properties after 0.02 wt.% CNT in the POM/CNT matrix.

Table II. Mechanical tensile properties of POM and its nanocomposites

Parameter	CNT amount [%]					
	0.0	0.001	0.01	0.02	0.03	1.0
Tensile strength (MPa)	45	52.3 ± 1.7	55.6 ± 2.1	58.2 ± 1.3	57.4 ± 0.9	57
Max. Strain (%)	7.2	7.2 ± 0.38	7.0 ± 0.37	6.8 ± 0.38	6.9 ± 0.46	6.7
Young's Modulus (MPa)	1718	1821 ± 36	1834 ± 25	2218 ± 58	1923 ± 33	1931

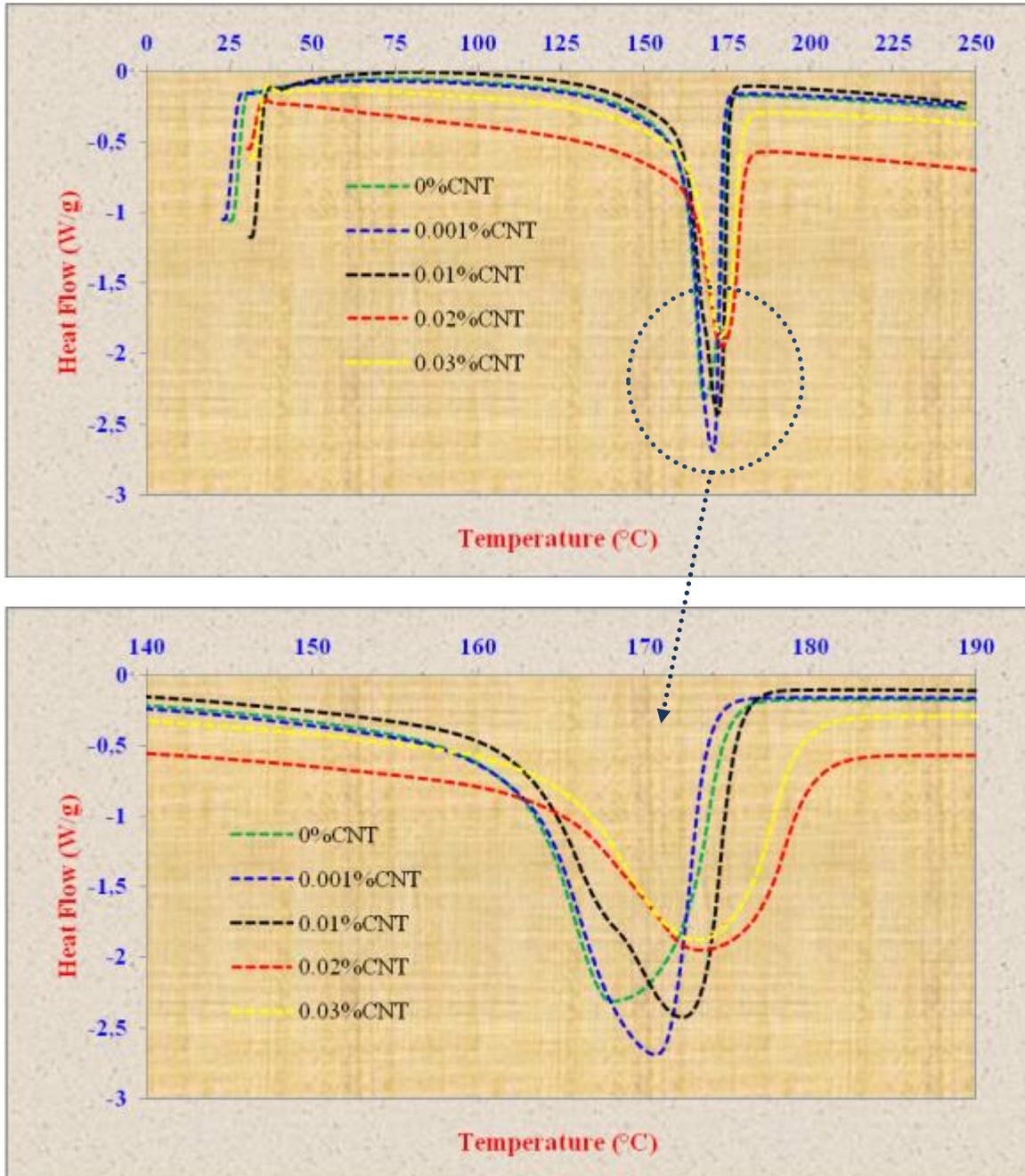


Figure (4) Typical DSC curves of POM and CNT/POM nanocomposites

3.2 Wear tracks and mechanism

The material removal rate (materials removed in the form of particles) due to the sliding between POM nanocomposites samples and metallic pin depend on the wear track size. This means the mass or weight loss increased when the wear track increased since the total sliding distances increased. Every sample was tested three times with different wear track (small, medium and large) and **Table III** illustrates the values of wear tracks.

Table III. Diameter values of wear tracks in POM and its nanocomposites

Wear track diameter [mm]	CNT amount [%]				
	0.0	0.001	0.01	0.02	0.03
Dry operating medium					
small	5	5.5	5.8	6	5
medium	9.3	10	10.3	11.5	10.4
large	13.6	14	15.16	15.4	16
Water operating medium					
small	6.5	5.1	5.29	6.46	5.6
medium	9.6	11.6	11.44	12.35	10
large	13.7	13.8	14.31	16.62	14
Oil operating medium					
small	4.8	6	4.9	4.6	5.1
medium	8.4	11.2	10.7	11.8	9.8
large	13.4	17	16	17.5	14.8

The wear loss (in form mass loss) was weighted by a high sensitivity balance with accuracy (10^{-4} gram). Figure 5 shows the relationship between weight loss (mg) and CNT content at each track, and also illustrate the variation of the weight loss of tested polymers nanocomposite at different medium. It is clear from Fig. (5A-C) that apart from operating condition (air, water and oil), the weight loss for pure POM polymer decrease with the increase in the percentage of CNT at small wear track, whereas with the increase in size of wear track the weight loss also increased. Furthermore, POM have the largest losses in the weight in air, oil and water respectively while POM/CNT0.03 showed lower level in water, air and oil respectively. Next, the specific wears rate were calculated for all samples at each percentage as shown in Fig. (6), to study the effect of wear track size on the final wear value. To check the accuracy of the wear which were measured by weight and calculated by Ws; the specific wear rate of POM for example in water improved by around 12% more than measured by weight loss, so this study focused on the Ws. The result shows that Ws of POM/CNT0.03 in air showed the better polymer nanocomposite composition followed by water and then oil respectively.

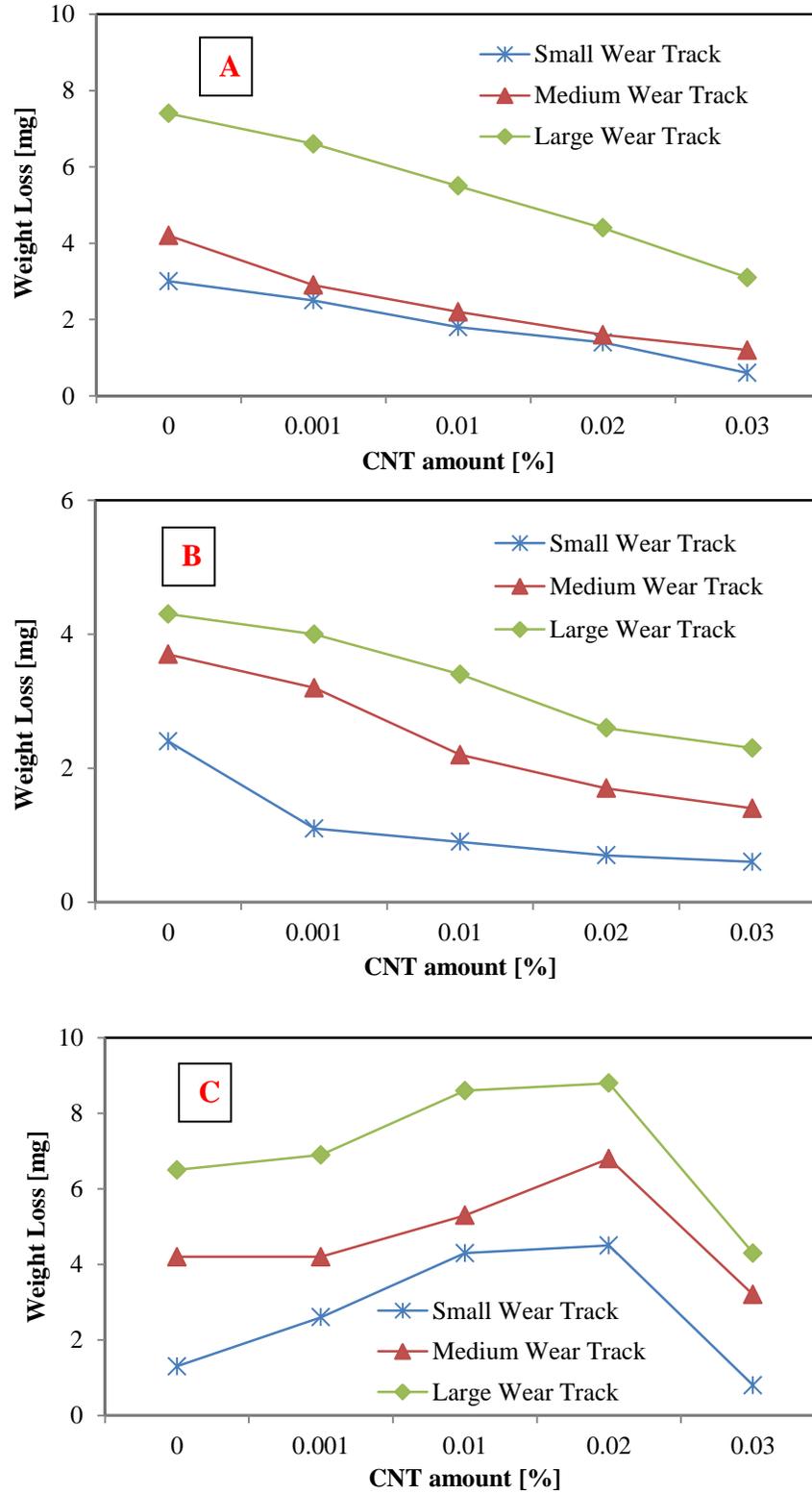


Figure (5) Weight loss of small, medium and large wear tracks of POM and its nanocomposites in (a) air medium (b) distilled water and (c) mineral oil

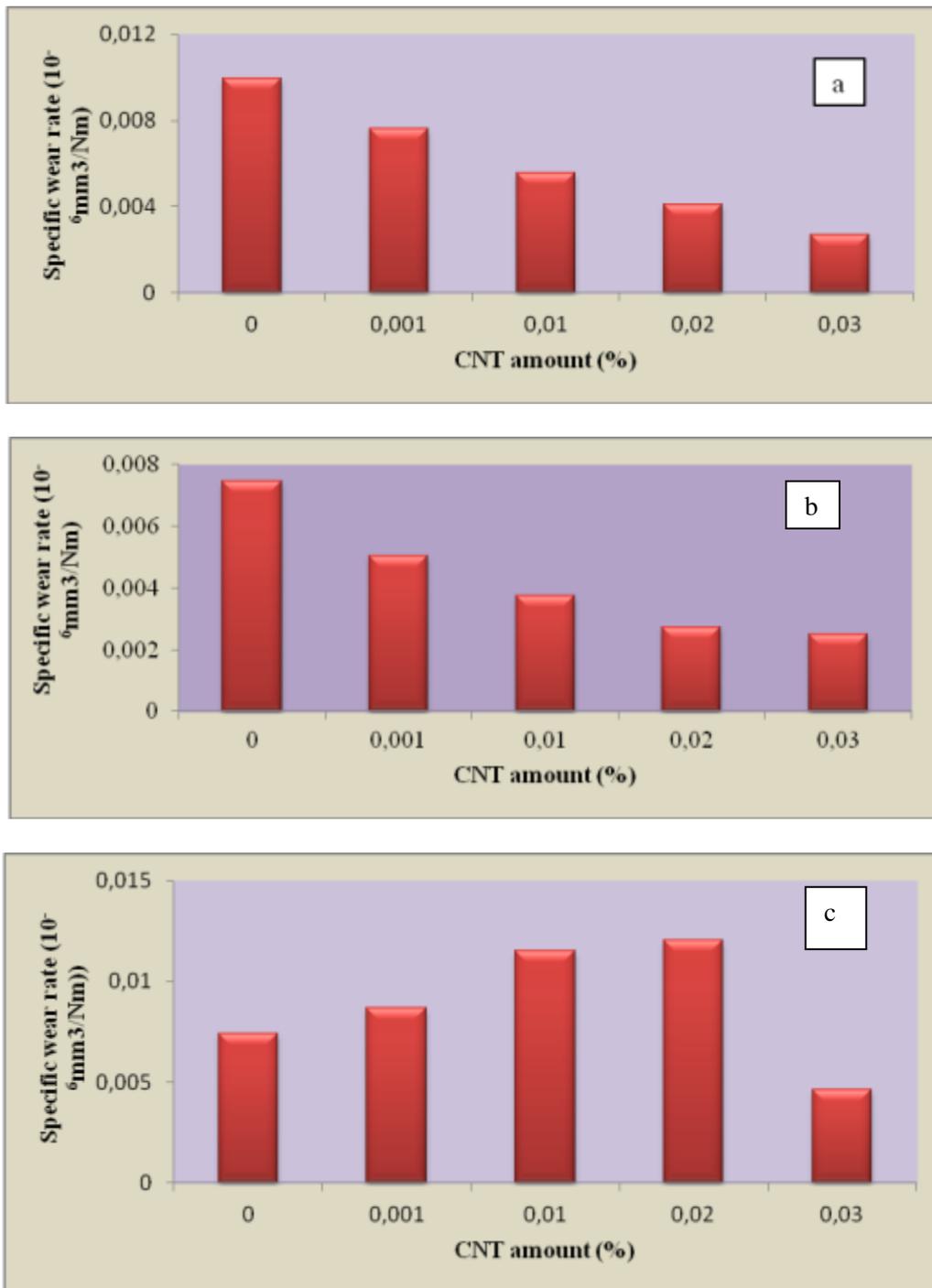


Figure (6) Specific wear rate for POM and its nanocomposites in (a) air medium (b) distilled water (c) mineral oil.

In order to investigate the inference of the wear mechanism of POM and its composite, the optical microscopy was used to examine the worn surfaces. Each sample had three wear tracks as

shown in Fig. (7a,b). The cross section of each track can be represented by trapezoid shape and have two inclined surfaces and curved base. Additionally two tip points resulted from the thickness of established track as shown in the inset of Fig. (7c). For the two tip points and curved base of wear track, failure damages is visible under naked eye at the contact points. On the other hand, it was difficult to observe the two true shape inclined surfaces due to the lens of microscopy that are not perpendicular to the sample surface. So the examination and analysis were focused on the medium wear tracks of POM sample in air ambient because the white color of pure POM during the scanning process reflects intense light declining the quality of the image and the details view.

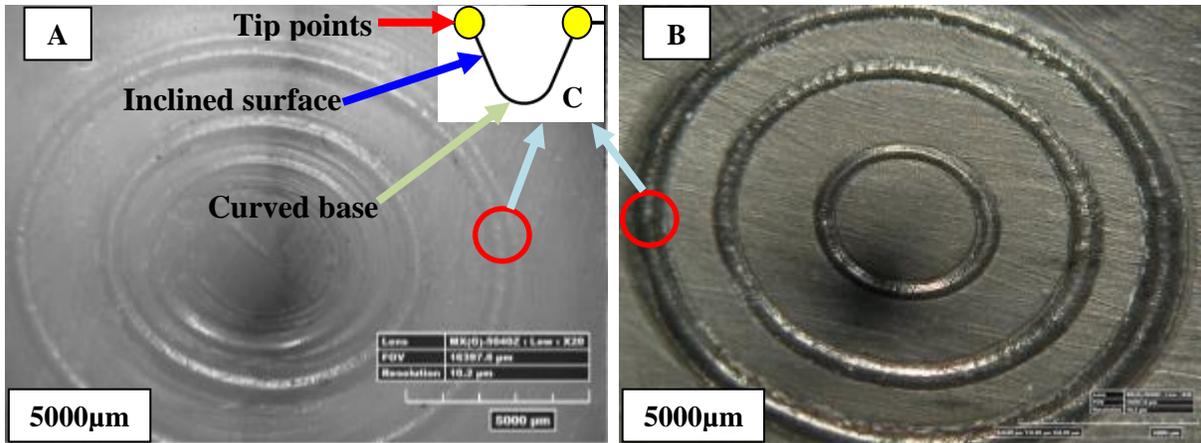


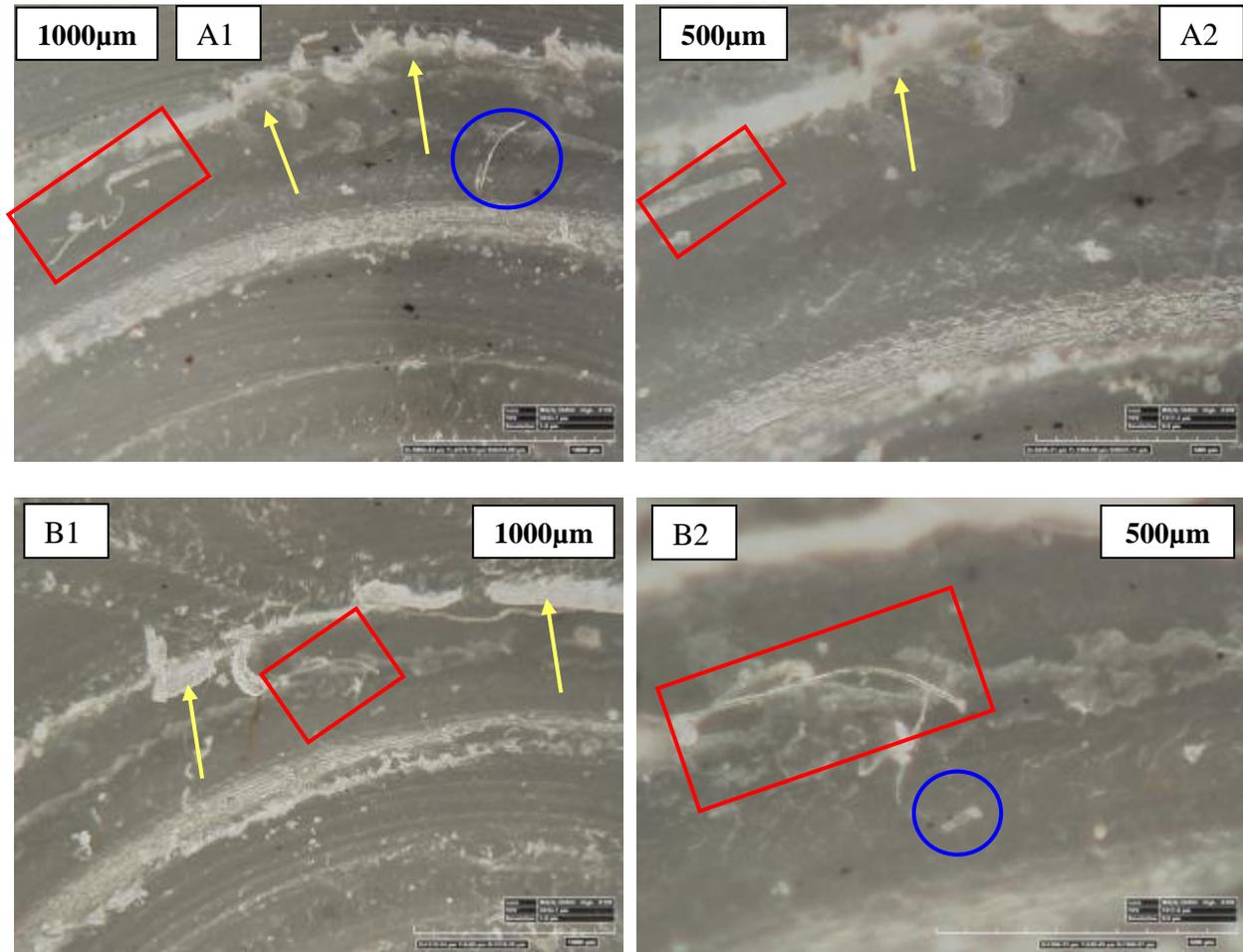
Figure (7) EM images of wear tracks for (A) POM, (B) CNT/POM and (C) Wear tract cross section

In Figs. (8-10) the electron microscopy images of POM and its nanocomposites have been inspected. All samples were scanned with the same scale are 500 and 1000 μm , except for the image that had all wear tracks scale is 5000 μm . In air medium we have compared the images of pure POM and of POM/CNT0.001, POM/CNT 0.01 and POM/CNT 0.03% samples (Fig.8). In water medium comparison was done on the images of POM/CNT0.001, POM/CNT 0.01 and POM/CNT 0.03% samples (Fig.9) while in oil medium the image of the POM/CNT 0.03% sample only (Fig. 10). The wear mechanisms in air, water and in oil are discussed in the follow up sections.

3.2.1 Wear mechanism in air

As a result of continues sliding between POM and its composite and the stain steel pin for 120 min, this lead to thermal softening and melting of the surface layer materials [29], this layer increase by the time and causes a several scratches in inclined surfaces and curved base. The damage particles materials welded to gather under the influence of the heat generated inside the wear tract to produce chip from like producing machining process, then this chip was exited through two inclined surfaces and the sliding effect. Finally, the chip is stuck on the tip points, where the end of sliding effect. Figure (8A1-D2) shows that, the amount and length of these chips decrease with increase of the CNT percentage (as indicated in the images by circles)

particularly at 0.03 wt.%, due to the following causes; a) the addition of paraffin oil during injection process to POM led to decrease the viscosity of the thermoplastic polymer and then obtained a well mixing (dispersion) with nanofiller [30], b) paraffin oil was used in this case as a lubricant oil and similarly CNT but as a solid lubricant even in the event of agglomeration, because CNT remains stuck with the tested surface, and c) the melting temperature of POM grows with increasing the CNT amount in the POM/CNT nanocomposites, as shown by calorimetric data. This lead to disabled the surface layer thermal softening occur early, thus decreasing the wear rate as clearly indicated in this case, where the maximum at 0.03 wt.% CNT. In fact, the melting temperature was measured inside the wear track also both in the virgin POM and in the POM/CNT0.03 nanocomposite. The result showed that the temperature increased by 5% inside the wear tracks in both the materials.



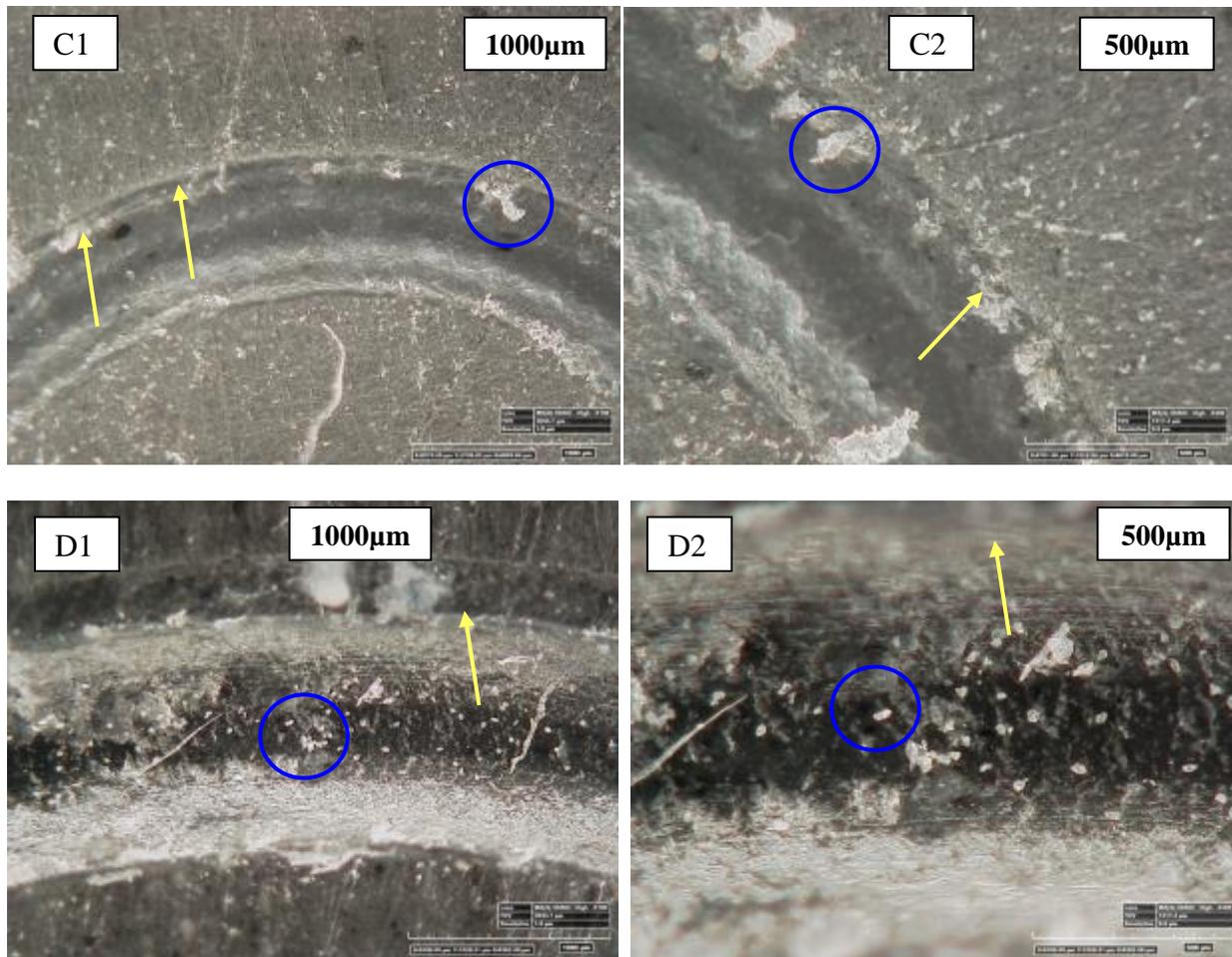


Figure (8) EM of the worn surface in air of (A) POM, (B) POM/CNT0.001 (C) POM/CNT0.01 and (D) POM/CNT0.03

3.2.2 Wear mechanism in distilled water

Distilled water is much cheaper compared to mineral oils and is also considered as an environmentally friendly lubricant if they leak into several polymeric components. Additionally, distilled water is often used as cooling source for polymeric parts which are sliding together [31]. Wear generally in polymeric materials depend on the heat generated during sliding process, therefore distilled water used to disable temperature rise (cooling), thus decrease the rate of materials have been removed. The shape of failure damages in this case are small particles not chips such as wear mechanism in air ambient, due to the same reasons in the previous case additionally used distilled water as a lubricant and cooling. It is worth mentioning the rate of material removed in this case at 0.02 and 0.03 wt. % CNT the same nearly, because the CNT agglomeration did not remains stuck with the tested surface but dissolved in the distilled water and became the CNT concentration in the two samples. Finally, the shape at the tip points

which are represented the limitations of the wear track are smooth in particular with increase of the of CNT concentration as shown in Fig. (9).

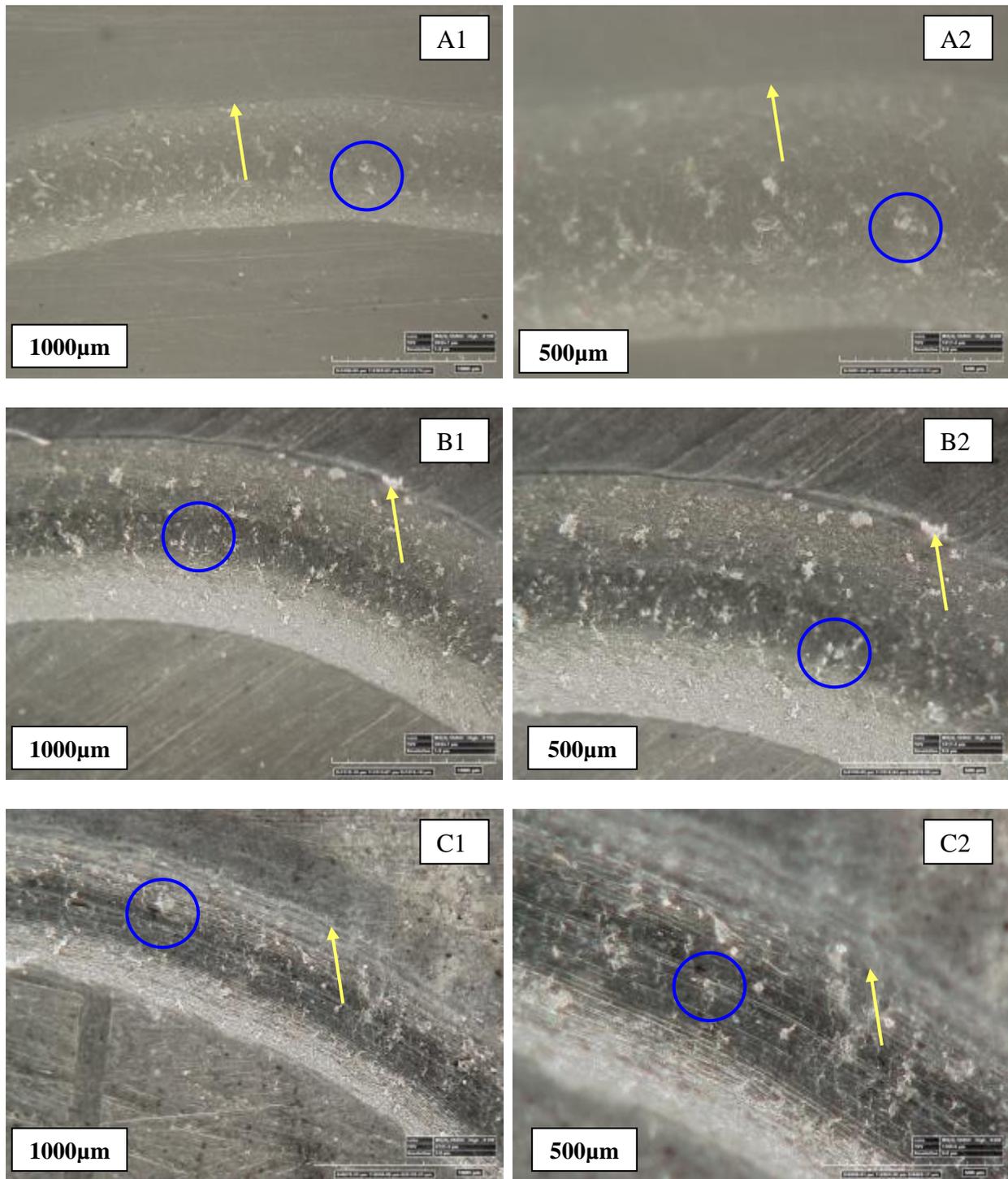


Figure (9) SEM of the worn surface in water (A) POM/CNT0.001, (B) POM/CNT0.01 and (C) POM/CNT0.03

3.2.3 Wear mechanism in oil ambient

Mineral lubricant such as oils and greases are considering the organic materials and if employed with polymeric materials can react, thus reduce the performance of these materials particular tribological behaviours, but the wear rate still increase until 0.02 wt.%. then significant decrease occurred at 0.03 wt.% perhaps due to increase in the ambient temperature. Consequently, this led to dispersed CNT which was removed from the tested sample in the oil under the rotational speed effect. Therefore, the oil characterizations such as viscosity, flash point, fire point and other properties had improved [32]. This is leading to decrease the friction between pin and the tested sample thus wear rate was decreased too, and resulting to a very smooth wear track limitation as shown in Fig. (10).

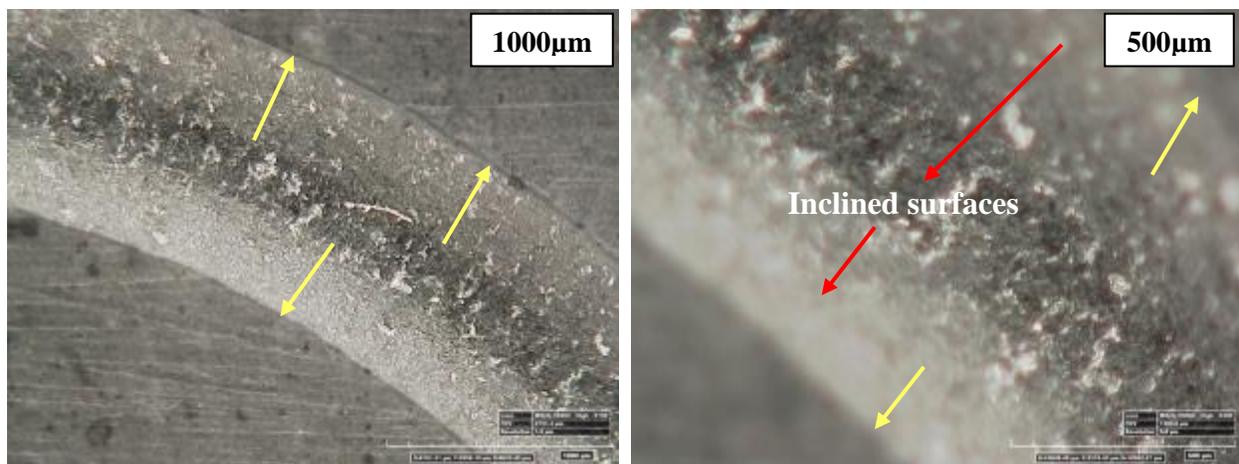


Figure (10) Wear track for POM/CNT 0.03% in oil lubricant

In summary, the average specific wear rate in air medium for the polymer consider the best condition and this result similar to Meng et al. (2009) [16].

Additionally, POM having 0.03 wt. % of CNT produced the best specific wear rate sample especially in ambient air and having average significant improvement of about 73% for sliding wear resistance. It should be noted that earlier study [21,22] reported the wear improvement about 15%, however the measured the gear wear reported was coupled i.e. sliding and rolling wear resistances. In our case we measured only one type of wear (sliding wear), therefore the final wear resistances having a significant increased. So, Fig. 11 shows the topography of the worn surface for POM/CNT0.03 at small, medium and large diameters wear tracks in air, water and mineral oil ambient which consider the better sample. Finally, the most wear mechanism failure is abrasion wear, for comparison, Figure 12 shows the wear in from (weight loss and specific wear rate).

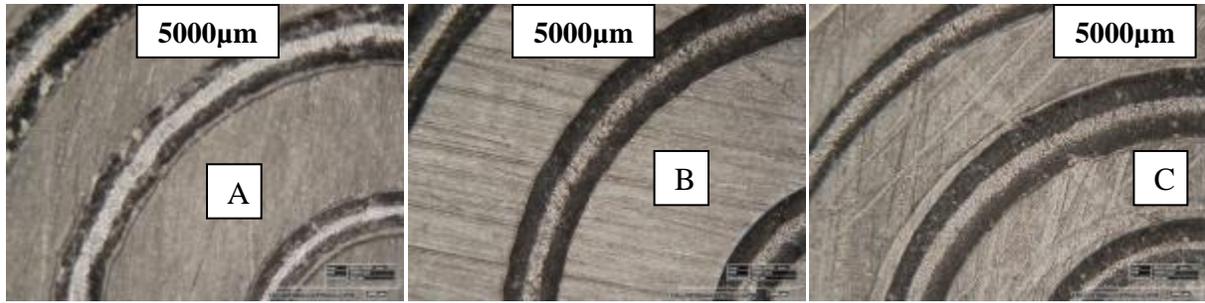


Figure (11) Wear tracks for CNT/POM 0.03% in a) Air, b) Water and c) oil; magnification of 5000 μ m.

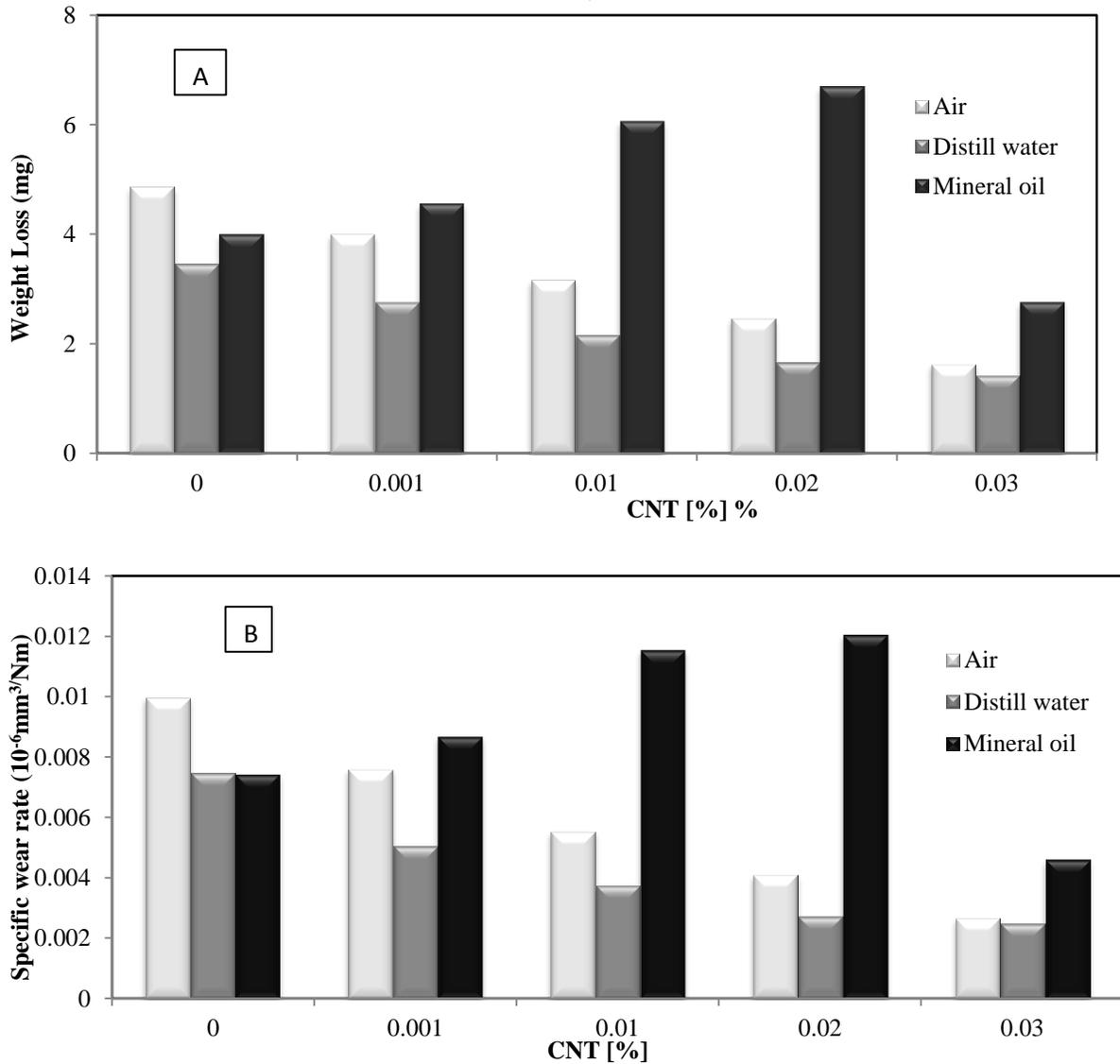


Figure (12) Effect of CNT contents on a) Average weight loss and b) Average specific wear rate of POM and its nanocomposites under dry sliding and lubricating conditions

Conclusion

In this paper, the authors studied the wear, mechanical and thermal characterizations of CNT/POM using pin-on-disc test rig, mechanical and thermal properties. The CNT/POM nanocomposites were synthesized by adding the paraffin oil as an assistant material during injection process: with POM as a base material and CNT as nanofiller material to obtain the desired dispersion. POM nanocomposite synthesized contained 0.001, 0.01, 0.02 and 0.03 wt. % CNT. For the wear, the tests were carried out under dry and wet (distill water and mineral oil) medium at normal load 12 N and test duration 120 minute. The results have indicated that Ws of POM having 0.03 wt. % CNT in air and water medium was improved by adding 73% and 66% respectively comparing with virgin POM. On the other hand, the mechanical properties precisely strength and modulus increased by about 31% and 29% respectively. Also crystallinity degree increased by 7% and melting temperature also had increased. The results showed that the higher percentage of CNT provided a better reduction in specific wear rate, while the air medium provided the greatest reduction in wear rate while the oil medium had the least influence in wear rate as expected.

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