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Flexural, Impact, Rheological and physical Characterizations of POM Reinforced by Carbon Nanotubes and Paraffin Oil

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Abstract

The paraffin oil dispersion technique innovated in the recent years to synthesize bulk polymer nanocomposite materials have a uniform dispersion. This research work aims to study the effect of added carbon nanotubes (CNT) on flexural, impact and rheology behaviours of polyoxymethylene (POM) reinforced by 0 - 0.03 wt. % of CNT using paraffin oil dispersion technique. The wettability and lamellar thickness were measured and rheological performance investigated using a parallel plate rheometer. The flexural and impact mechanical properties were also evaluated. The fracture surfaces were then examined by microscopy. The results showed that the energy to break, flexural strength and modulus increased proportionally with the addition of the amount of CNT in the matrix. For the rheology behaviour, the viscosity decreased at the low percentage of CNT, and then increased with increase the percentage weight ratio of CNT in the POM matrix. It was also noted that the water contact angle rose with the increase the CNT percentages.

Key words; Carbon nanotubes, POM, Paraffin oil dispersion technique, Rheological, Fracture properties and Flexural properties

1. Introduction

Currently, polyoxymethylene (POM) is used in many industrial sectors for biomedical applications such as, removable partial dentures to vehicular structures in automotive applications. This owes to the fact that POM is a rigid material with good energy absorption capability compared to other thermoplastic polymers [1-3].

To increase the service lifetime, carbon nanotubes (CNT) have been considered in recent years as nanofiller materials incorporated in POM to improve the desired physiochemical properties [4, 5]. The results showed minor improvements, but dominated by defects such as poor compatibility and dispersion due to POM's high viscosity [6]. Next, CNT/POM with high compatibility and uniform dispersion have been synthesized by application of a small amount of paraffin oil (PO) and appears to be a viable solution to the CNT poor compatibility and dispersion [7]. It is worth mentioning that a new dispersion technique for PO in POM has been investigated in the recent literature and reported to produce a significant improvement in mechanical properties, in particular the strength and modulus by about 22 and 30% respectively.

A 6% increase in the melting temperature and 7% improvement in the degree of crystallinity were also noted in the thermal properties [8].

Regarding the wear behaviours of CNT/POM reinforced by PO, Yousef et al. (2013 and 2014) measured the wear rate (sliding and rolling) of four types of CNT/POM gears (spur, helical, bevel and worm) using TS universal test rig [7, 9]. The results showed that the CNT had a significant positive effect on the wear rate reattached until 32%. Through similar testing method, a pin on disk was employed to measure the sliding wear rate of CNT/POM in three different operating medium (air, distilled water and mineral oil). The results showed that the air medium provides the greatest reduction in wear rate while the oil medium the lowest [8]. On the other hand, POM characterized by low water absorption lead to increase the contact angle, thus improved the wettability [10].

CNT/POM is one of the rapidly evolving areas of polymer nanocomposite research and considering a strong competitor for some soft metals. The available literature, primarily deals with the improved mechanical, wear and thermal behaviours of CNT/POM. So far, only a handful of research works has been published on the effect of rheological parameters variation by using rheometrics system (parallel or cone plates) and capillary viscometer [11-16]. The evaluation of melt rheological properties of POM and its composites in terms of complex viscosity, storage modulus and loss modulus with angular frequency is important. However the correlation of flexural, impact, rheology, thermal and wettability behaviours of CNT/POM is missing. This paper therefore aims to study the effect CNT and PO on the missing behaviours. The wettability and lamellar thickness were measured and rheological performance investigated using a parallel plate rheometer. The flexural and impact mechanical properties were also evaluated and damage investigated using microscopy.

2. Experimental

2.1 Materials and Preparation

The POM used in this study is a commercial grade powder (KOCETAL® K700) supplied by El-Slam Company, Cairo, Egypt. 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 [17, 18]. CNT/POM composites containing 0.001, 0.01, 0.2 and 0.3 wt% of CNTs were prepared using an injection moulding die that was designed in a previous work by the Yousef et al. (2014) to produce a short bar. Paraffin oil was used to attach the CNTs with the POM pellets after stirring for approximately 5 min [7]. Each prepared sample has a code according to percentage of CNT as illustrated in Table (I). All tested samples were cut or machined by turning and milling machine and had surface roughness 0.23 μm.

Table I. Synthesis samples codes

CNT (% wt.)	0	0.001	0.01	0.02	0.03
Sample code	PC_0	PC_{01}	PC_1	PC_2	PC_3

The flexural specimens of CNT/POM were prepared according to (BS EN ISO 178:2010) standard (three point bending test) with the following dimensions 80 x 10 x 4 mm^3. LLOYD LR 10 k universal testing machine with load cell 500 N and crosshead speed of 1.25 mm/min was employed to measure the flexural load and modulus at the ambient temperature, then the flexural stress (σ_f), flexural strain (ε_f) and flexure modulus (E_f) were calculated by the following equations (I-III) [1];

$$\sigma_f = \frac{3FL}{2bt^2} \quad \text{(I)} \qquad \varepsilon_f = \frac{6\delta t}{L^2} \quad \text{(II)} \qquad E_f = \frac{FL^3}{4\delta b t^2} \quad \text{(III)}$$

where F (N) is the flexural load, L (mm), b (mm), t (mm) and δ (mm) is the span length, width, thickness and defection of the specimen respectively.

The impact tester (Model: CEAST Resil Impactor) equipped with a 25 Joule slug (ASTM D256-10 international protocols) and impact speed 3.7 m/s was used to measure the energy to break of POM and CNT/POM. The notch 2.5 mm deepness was performed by a CEAST NOTCHVIS with an angle of cut of 45°. The notched samples were tested at ambient temperature. Five flexural and impact tests were performed to ensure the reliability of the test results. In addition, optical microscopy (Model: Hirox digital microscope KH 8700) was used to examine the fracture surfaces of the flexural and impact specimens.

The wettability or contact angle (θ˚) of POM and its composites in distilled water and mineral oil were evaluated by using a device have a high resolution camera. This device was particularly assembled at Messina University by Torrisi et al. (2002) to measure θ [19], through depositing a drop of liquid at room temperature on the horizontal surface of the sample using a 0.5 microliters calibrated syringe. A 5 µl drop was deposited on the sample surface and θ was measured after 2 second [20]. Ten drops were measured for each sample. Finally, Jimage software was used to capture and analyze the contact angle.

Gibbs-Thomson equation *(*IV*)* provides one of the convenient ways to calculate the lamellar thickness (d_c) of the polymer and its composites, particularly in our case POM and CNT/POM nanocomposites [21].

$$d_c = \frac{2\sigma_e}{\Delta h_f}\left(1 - \frac{T_m}{T_m^o}\right)^{-1} \qquad \text{(IV)}$$

Here, T_m^o = 187+273 °K is the extrapolated equilibrium melting temperature of a POM crystal of infinite thickness, T_m (°K) is the melting peak absolute temperature of pristine POM and CNT/POM (measured by TA INSTRUMENTS Thermo DSC Q 100) [8], σ_e = 122 $J.cm^{-2}$ is the lamellar basal surface free energy, and Δh_f = 3.78 x 10^9 $J.cm^{-3}$ is the heat fusion per unit volume [22].

Regarding the evaluation of melt rheological parameters variations (complex viscosity and storage modulus with angular frequency) of the pristine POM and CNT/POM nanocomposite, was carried out by means of a rotational rheometer (Mod. SR5, Rheometric Scientific) equipped with an environmental controller. The experiments were performed with a parallel plate geometry, diameter 25 mm. The tested round samples shown on Fig. (1) had 25 mm in diameter and thickness of 1mm. The tests were conducted in the linear viscoelastic regime at 1% applied strain controlled, temperature sweeps of 180°C, frequency range from 1 to 100 rad/s, and the gap between tested sample and upper hot metal rod of 0.05 mm [13].

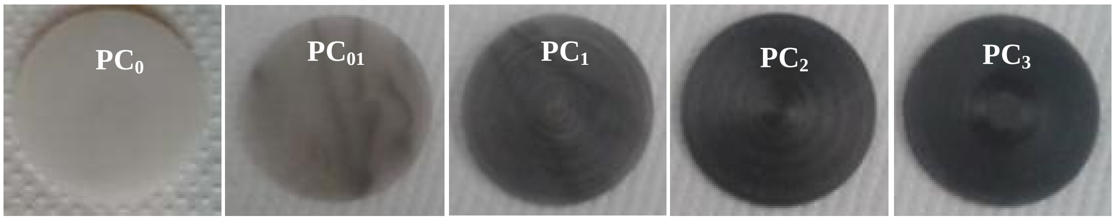


Figure (1) The POM and CNT/POM rheology specimens

3. Results and discussion

3.1 Flexural Properties

The flexural stress–strain curve was determined to investigate the effect of CNT on the flexural properties of POM as shown in Fig. (2A). The trend in variation of σ_f and E_f of CNT/POM nanocomposites at various amounts of CNT is presented in Fig. (2B, C). It can be seen that σ_f increases with increasing ε_f until around 128 % of all samples. This point represent the maximum value of σ_f and after this point σ_f was decreased with increases ε_f. Also, it was clean that σ_f and E_f for PC_0 and PC_{01} similar approximately close to 106 MPa , due to the smaller amount of nanofiller material and this is leading the nanofiller are not enough to cover the all surface POM thus poor dispersion or not uniformly.

PC_1 is considered as the starting point of uniform dispersion σ_f and E_f increases gradually by about 4% and 24% respectively. With the increase of the CNT amount until PC_2, significant increasing was noted for σ_f and E_f i.e. about 20% and 27% respectively. This may be associated to excellent dispersion noted at this percentage of CNT [8]. Additionally, the σ_f and E_f of the CNT was noted to be higher than that of the POM, leading to increasing the flexural modulus of the CNT/POM composite [23-25]. Also, the crystallization behaviour of POM (PC_0) was improved CNT presence [8]. After this amount of CNT for PC_3 sample, σ_f and E_f decreased (13% and 21% respectively) due to the agglomeration of CNT in the POM matrix leading to poor dispersion.

To better understand the failure mechanism, optical microscopy analysis was used to observe the fracture surfaces of two flexure samples (PC_2 and PC_3), which had the higher flexure strength values respectively. During flexure tests, the test sample is exposed to two types of stresses; tension and compression. Usually, compression is located on the inside (contact with force tip) and tension on the outside. In the ideal case; the crack propagation starts at outside surface which experience the maximum bending strength [26]. Figure (3A, B) shows the corrupted surface of PC_2 and PC_3, it is clean that the failure crack in PC_2 relatively similar to ideal case, and the thickness of tension zone was lower than compression zone, due to well dispersion leading to increase σ_f and E_f as shown in the Fig. (3A). Regarding $PC_{3,}$ the thickness of tension and compression zone was relatively equalled as shown on Fig. (3B). Furthermore, the crack failure appeared inside the cross section (in the middle) due to CNT agglomeration as shown in the red circles (Figure 3B), leading to decrease σ_f and E_f again.

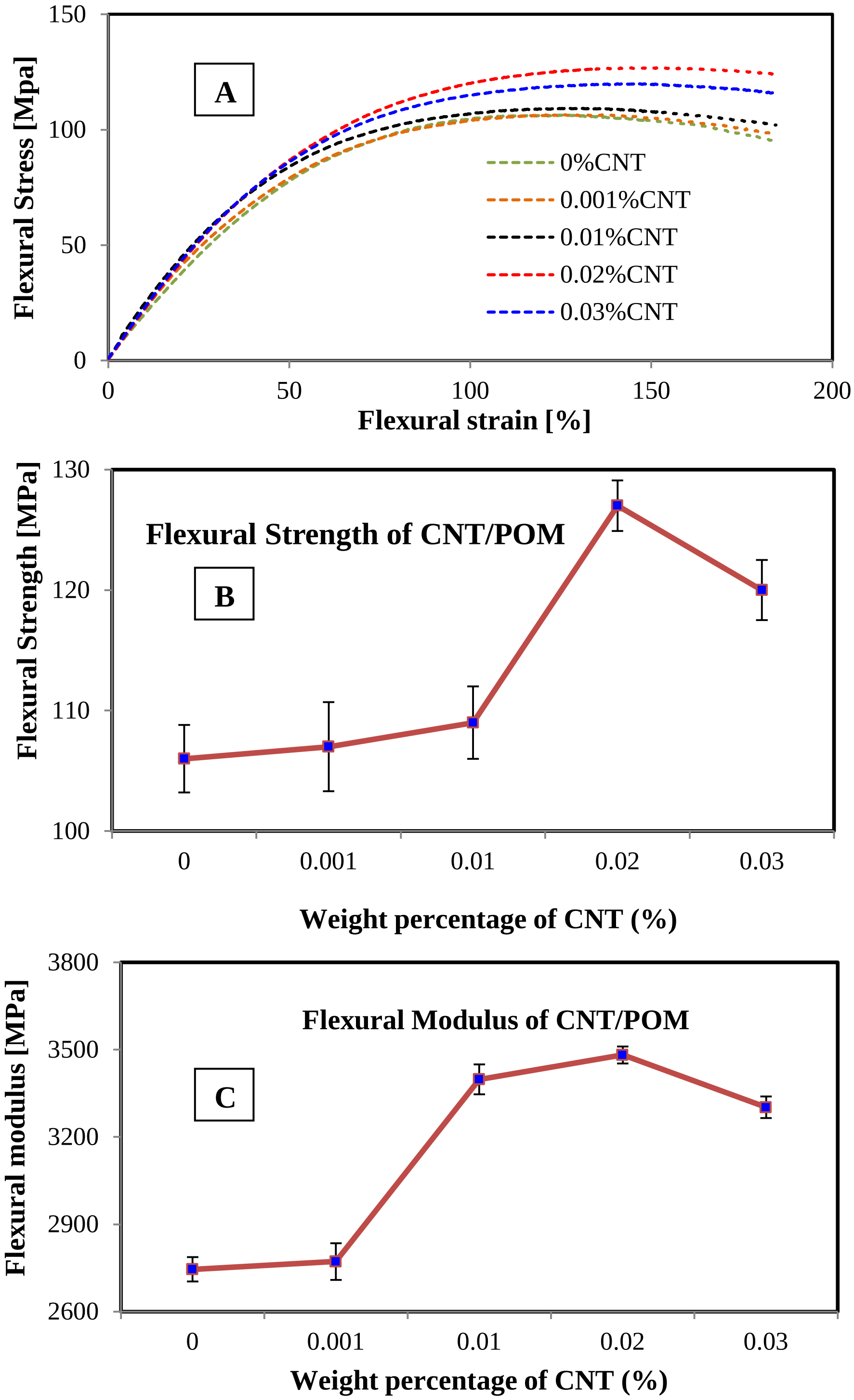


Figure (2) Relationships between (A) flexural stress– flexural strain, (A) flexural strength vs the CNT amount, and (B) flexural modulus vs the CNT amount of POM and CNT/POM composites

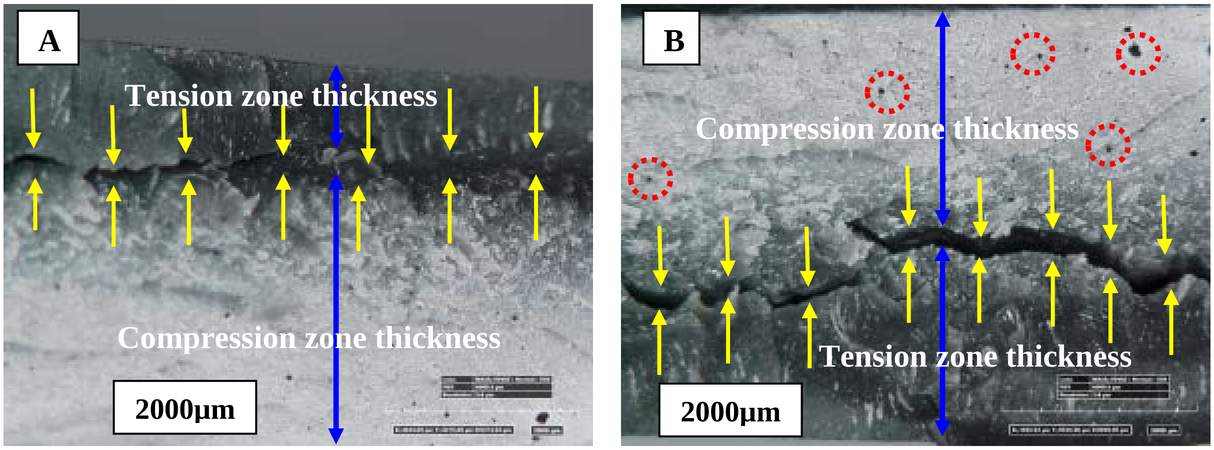


Figure (3) Optical microscope images of flexural fracture surfaces (A) PC_2 and (B) PC_3

3.2 Impact Properties

Figure (4) shows the CNT/POM impact specimen had a very smooth V-notch and also shows the influence of CNT addition on Izod impact strength of POM. As shown in the figure, the impact strength of PC_{01} was slightly improved ~8% compared by PC_0 due to poor dispersion. At PC_1 and PC_2, significant increasing were occurred ~23% and ~29% respectively, due to the increasing of CNT amount leading to more coalescence between CNT nanoparticles and POM thus impact strength increases. In addition, CNT had impact strength more than POM, also, increased energy absorption during the impact process [27]. Finally, impact strength of PC_2 was decreased due CNT agglomeration [28].

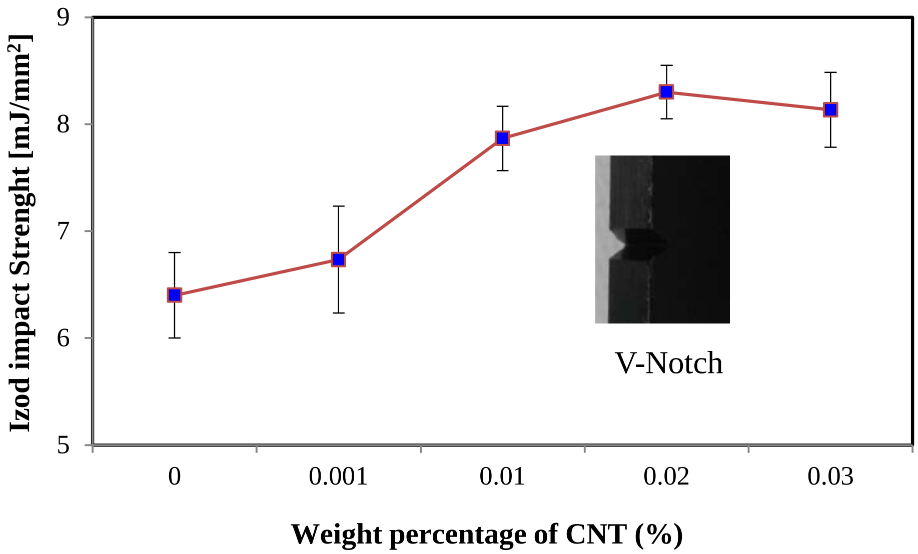
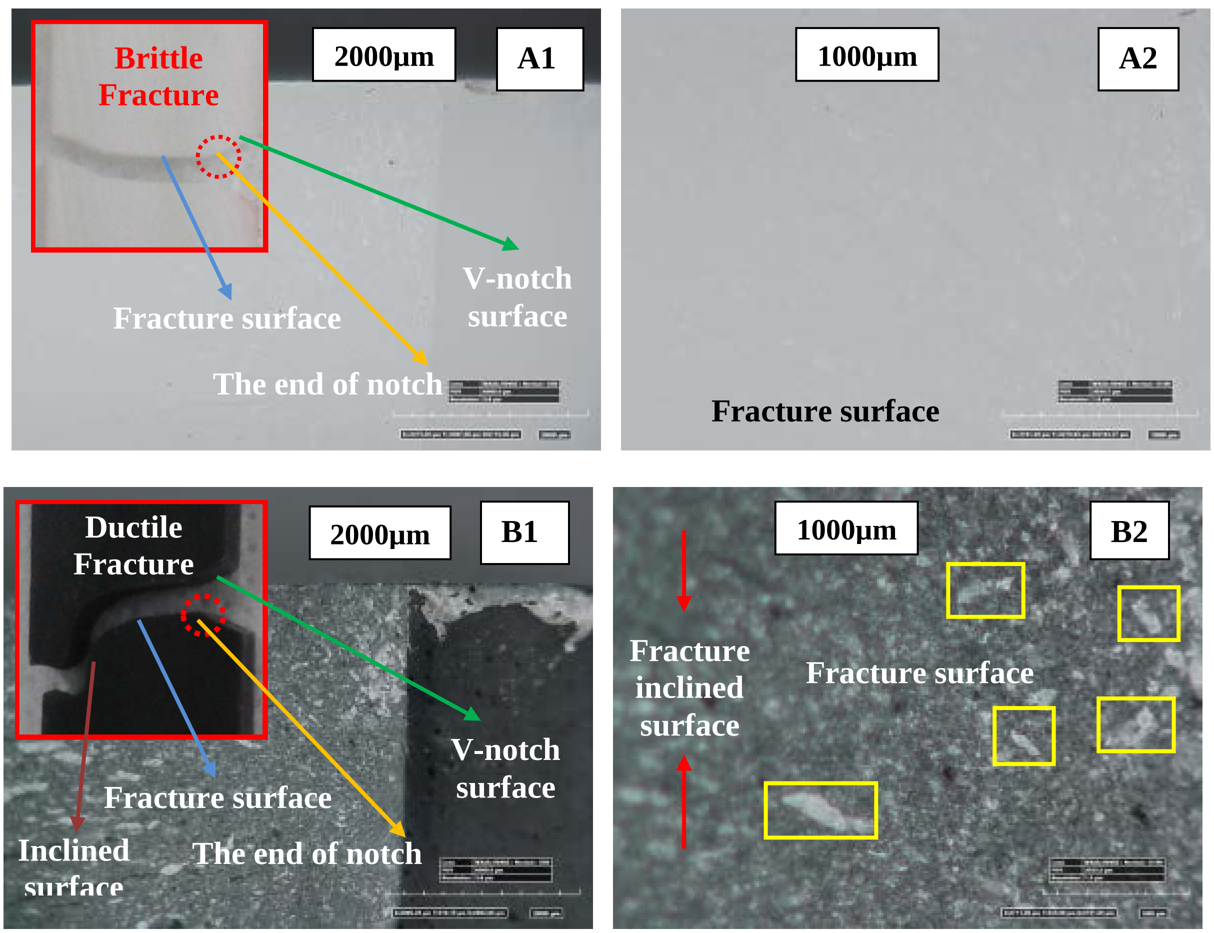


Figure (4): Impact strength of pure POM and CNT/POM nanocomposites

Optical microscopy also was used to inspect the impact damage surfaces for PC_2 and PC_3. Every damage surface had three features, V-notch surface, intersection line (intersection between V-notch and fracture surface), and the fracture surface area. The examination focused on the fracture surface area. Generally, in thermoplastic materials there are two modes of failure, i.e. brittle fracture (weak) and ductile fracture (tough) [29]. Brittle and ductile fracture is characterized by linear relationship and plastic yielding respectively. The brittle fracture is considered more serious because it does happen suddenly. Previous studies on the plastic yielding of CNT/POM showed that the plastic yielding increased by adding CNT until 0.02 wt.% then decrease gradually at 0.03 wt. % and linear relationship was noted [8].

All features of impact fracture (V-notch, intersection line and fracture area) are shown in Fig. (5). The inspection focused on the impact fractured area. As shown in Fig. (5A) the fractured surface of PC_0 was very clean, and this meant a brittle fracture (flat surface). By addition CNT in particularly $PC_{2,}$ some smaller particles were still sticking in the damage surface (inside the yellow squares) and this meaning ductile fracture (winding or spline surface) as shown in Fig. (5B). Through increasing the amount of CNT (PC_3), brittle fracture reappeared with relatively smooth (inside the white circles) as shown in Fig. (5C). Accordingly, the failure modes are compatible with Yousef (2015) [8].



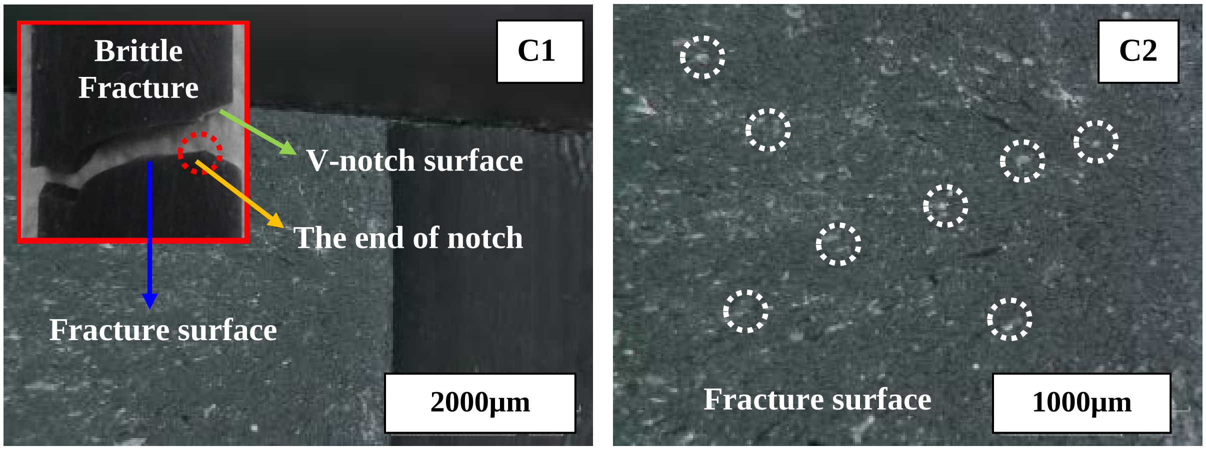


Figure (5) Optical microscope images of impact fracture surfaces (A) PC_0, (B) PC_2 and (C) PC_3

3.3 Lamellar Thickness

Figure (6) shows the effect of CNT amount on lamellar thickness of POM polymer single crystals. It is shown that the lamellar thickness increases with increasing the amount of CNT, due increase the crystallization [8, 30].

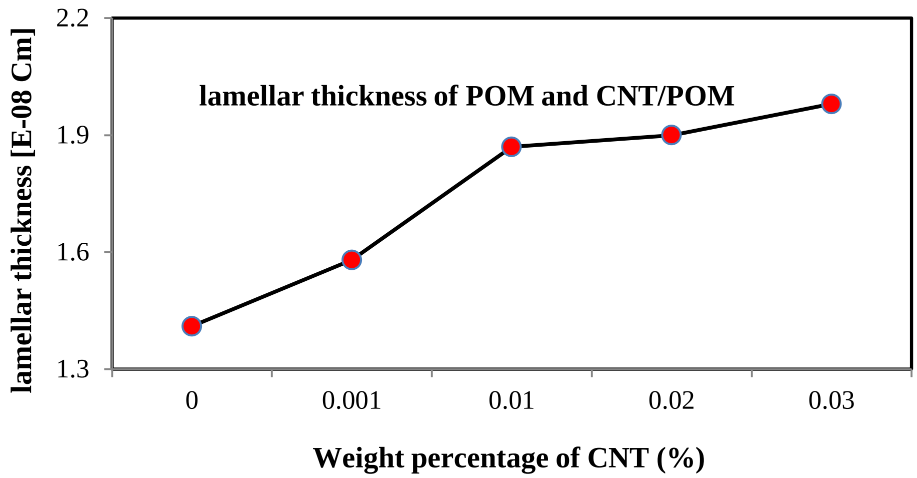


Figure 6: Relation between lamellar thickness and CNT amount

3.4 Wettability

In general, the wear rate of polymeric materials under the wet (distilled water and mineral oil) conditions depends on many factors such as, the friction coefficient and wettability. Borruto et al. (1998) suggested that the reduction in the coefficient of friction leads to increase the contact angle (θ) of the polymers [31]. Golchin et al. (2013) confirmed these results and draw the relationship between θ and the specific wear rate (Ws) for several polymers including POM, the result showed that POM had a best Ws after UHMWPE and PET respectively. In addition, θ had the same trend of Ws and POM had distilled water θ=70˚ [32]. In the last paper, the authors studied the wear behaviour of POM and CNT/POM in distilled water and mineral oil ambient [8]. Table [II] illustrates the distilled water θ measurements and Ws of POM and CNT/POM. It is clear that, a significant rise in θ was noted with increasing the amount of CNT until PC_2, then gradually increasing at PC_3. The increasing of θ led to decreasing the wettability of the POM nanocomposites [33]. This improved as a result particular modification enhanced by the CNT presence [34]. Also, mineral oil θ was measured for comparison. The results showed that θ is equal to 180 º, i.e., a liquid film is formed on a solid surface where a perfect wetting occurs [35]. This means the wettability of distilled water is better than mineral oil. Also, the wear rat in distilled water considers the best and this agree with Yousef et al. (2015). Finally, Fig. (7) shows the relationship between distilled water θ vs. Ws of POM and CNT/POM [8].

Table II. The average value of contact angle and specific wear rate of POM and its composites

Sample code	PC_0	PC_{01}	PC_1	PC_2	PC_3
Average contact angle (˚)	69	71	74	79	80
Specific wear rate in water ($10^2 mm^2/N$) [8]	74.61	50.51	37.55	27.32	24.94

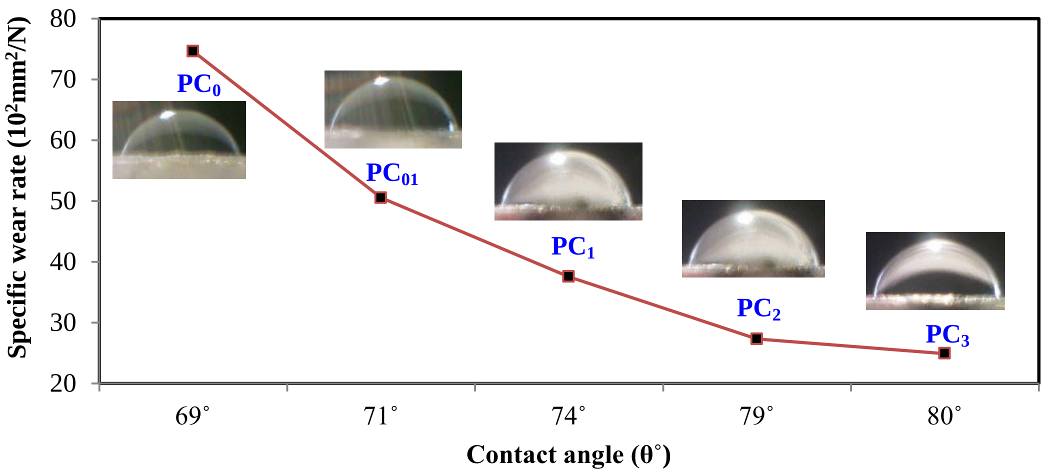


Figure 7: Specific wear rate vs. water contact angle of polymers

3.5 Rheological Analysis

Generally, the high viscosity (η) for thermoplastic materials such as; POM and UHMWPE represent the main obstacle for blends the nanofiller materials with polymers - for more precisely, high η means bad dispersion [36]. So, Galetz et al. (2007), Yuezhen et al. (2007) and Wood et al. (2011) used PO as a solvent and assisted melt material during the mixing process respectively then extracted PO to decrease η of UHMWPE - the result was uniform dispersion [37-39]. By the same token, Yousef et al. (2013, 2014) blended PO with CNT/POM without extraction. Thus, melt rheological analysis for CNT/POM was required in order to find a relationship between viscosity and dispersion. It is worth mentioning that POM and it's composites at low frequencies (below 1 rad/s) has a liquid-like behaviour. So, η was measured at 1 rad/s [13]. Figure (8a) show η against frequency curves for POM and CNT/POM. As observed, PC_{01} has lower η (1200 Pa.s) comparing pristine POM (2800 Pa.s), this is a result of adding PO not CNT because many previous studies showed that the adding of nanofiller materials led to increased η not vice versa [40]. Additionally, the amount of CNT in this sample was too small when compared with other samples, but in our case η decreases as a result of adding PO. In confirmation of this, η increased gradually by surge in the amount CNT until PC_2, which had higher crystallinity [8] but still lower than pristine POM. Meanwhile, the η of PC_3 increased more than pristine POM due to CNT agglomeration. Accordingly, POM and CNT at 0.02 wt.% are completely compatible. Also, storage modulus (G') against frequency was plotted for POM and CNT/POM as shown in Fig. (8b). The result shown that G' have the same trend of η but on the contrary. Finally, the presence of the PO in the CNT/POM blends decreased the diffusion defect, and then increased the crystallinity, thus decreased the viscosity of the blends until 0.02 wt.% and hence a better dispersion.

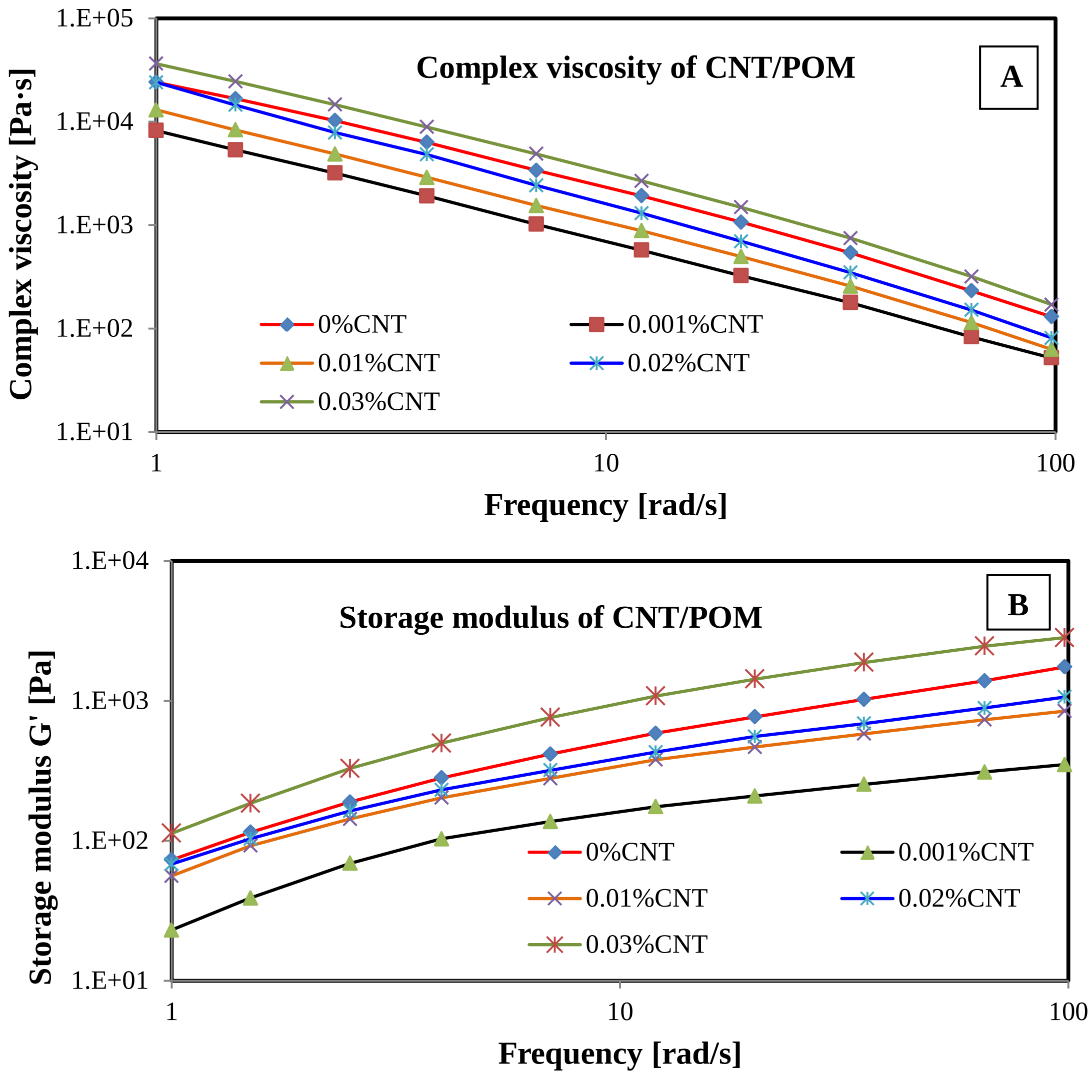


Figure 8: Variation of A) complex viscosity and B) storage modulus with angular frequency for CNT/POM

Finally, despite that the rheological characterization of POM reinforced by CNT was hidden due to its high viscosity, however, CNT is a rigid-rod similarities with macromolecular systems and has a higher ratio of surface area to volume, this leads to rapid interaction and more cross-linking between CNT and POM, hence form a nematic phase which lead to increase the shear force necessary to break these bonds hence increase the viscosity [41-43]. Also, the agglomeration is one of the reasons for increasing the viscosity. Therefore, PO was used as a thin film to cover the pellet surfaces, then abrasive the CNT with a uniform distribution on the pellet surfaces after manual mixing thus decrease the agglomeration during the injection process. In addition, the adding of PO leads to create more mobile POM chains and thus reduce the viscosity and allow penetration of the CNT into the POM grains [44].

In summary, this paper presented an economical POM nanocomposite compared to other thermoplastic polymers has a good energy absorption and very useful in biomedical and automotive applications, the nanocomposite has been prepared using the PO dispersion technique. Also, the paper presented another method to observe the dispersion of CNT inside the polymer matrix beside the other dispersion methods through examining the viscosity, where low viscosity represents the best dispersion and vice versa.

Conclusions

In this study, flexural, impact, rheology, thermal and wettability behaviours of CNT/POM nanocomposites containing 0, 0.001, 0.01, 0.02 and 0.03 wt. % of CNT were investigated. Based on the results the following conclusions were drawn:

1. The flexural strength, modulus strength and impact strength of POM were increased by the addition of CNT and paraffin oil until 0.02 CNT wt.% associated to the increase in lamellar thickness of the polymeric crystals and reduced the fusion defect. Also CNT had a more rigid and energy absorption compared to POM. After 0.02 CNT wt.% decrease in desired properties was noted and related to CNT agglomeration in POM matrix.
2. With the increasing CNT amount, the contact angle of CNT/POM increased under distilled water condition, but then decreased the wettability - thus increase the water absorption resistance and this lead to improvement in the wear rate. A completely comparative absorption was recorded under mental oil condition.
3. The addition of paraffin oil had a major effect on the complex viscosity and storage modulus of POM, where these characterizations were decreased, then increased again by increase the amount of CNTs until 0.02 wt.%, which represented the nearly similar values to virgin POM.

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