ROUGHNESS, WEAR AND THERMAL ANALYSIS OF UHMWPE NANOCOMPOSITES AS ACETABULAR CUP IN HIP JOINT REPLACEMENT

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ABSTRACT

Reinforcement UHMWPE by nano additives represented by carbon nanotubes (CNTs) and nanohydroxyapatite (nHA) with four weight fractions (1, 2, 3 and 5%) has a vital role to the improvement of surface roughness and wear resistance of produced nanocomposites. A surface roughness of new nanocomposites was decreased with an increasing weight fraction of each additive especially with CNTs due to the larger surface area of this nanofiller compared with nHA particles that achieve lower area.

The results of wear rate indicated the reduction in this rate with reinforcing and this reduction increases with an increasing weight fraction of each additive especially with nHA due to the better filling of the voids by HA particles compared with CNTs that have a hollow structure and may be left some holes in the bulk composite.

The calculated volumetric rate also gave the same results as in weight loss as a function to the weight fraction of additives. The density was increased for prepared nanocomposites due to filling the pores compared with pure UHMWPE which has many holes filled by air. The increasing in density for UHMWPE/nHA composites was higher than UHMWPE/CNTs composites due to the good filling of the pores.

Thermal analysis was done for prepared nanocomposites and the results gave rising in melting and crystallinity temperature for composites compared with pure UHMWPE.

KEYWORDS: Hip joint Replacement, Acetabular Cup, Nanocomposite, UHMWPE, Nano Additives, Wear Rate & Roughness

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1. INTRODUCTION

Nowadays, the nanofillers are used to produce nanocomposites with good improvements in most properties. Better dispersion of nanofillers within the matrix achieves high performance and enhancing the interfacial properties of these nanocomposites for biomedical applications and structural applications [1-5]. In general, the biopolymers like UHMWPE and PMMA has unique physical, chemical and mechanical properties as a matrix due to superior wear resistance along with high fracture toughness and biocompatibility compared to other polymer that used as implants for tissue applications after reinforced by nanofillers[6-9].

UHMWPE used in the manufacturing of acetabular cup in hip joint replacement, but there is a problem due to wear phenomena formed particles leading to aseptic loosening and then the failure of this joint can occurs [10]. Many types of filler can be used to improve the wear performance of UHMWPE through production of
composites like fibers and nanopowder with polymers [11]. There was a work focused on adding MWCNT up to 1 wt% to improve the nano composites with limiting the embrittlement of these composite [12].

The improving of wear resistance is related to cross-linking and reinforced materials that affecting the atomic or molecular arrangement, confirming a specific crystal structure [13]. Karuppiah et al. discovered that the crystallinity effects on the wear resistance through increasing the crystallinity that decreases the friction force and the scratch depth [14]. Similar results were suggested by Wang and Ge [15].

Jawad [16] (2010) studied the wear rate behavior of polyester reinforced by microsize particles of SiO2. Pin on disc machine with variable speed has been used to conduct the experimental work. The results show that the wear rate of such material decreases when using higher weight fraction and smaller particle size.

In the current research, there is an attempt to improve the roughness and wear resistance through the reinforcing UHMWPE by adding four weight fractions of CNTs and nHA to produce nanocomposite as acetabular cup for hip joint supported by DSC analysis to confirm the increasing in crystallinity temperature of UHMWPE nanocomposites.

2. EXPERIMENTAL PROCEDURE
2.1 Materials and Methods

Ultrahigh molecular weight polyethylene Purchased from (MAX PIPE INDUSTRY Co. Ltd) with an average molecular weight (5.5×10^6 g/mol), density (0.935 g/cm^3) and average particle size of (20-50 µm) was used as the matrix to prepared eight nanocomposites. These nanocomposites were reinforced with four weight fraction (%) of each carbon nanotubes (CNTs) and nanohydroxyapatites (nHA).

Multi-walled carbon nanotubes (MWCNTs) (>95%, with an average diameter(30-80 nm) and lengths of 10 - 30µm) were syntheses via AAO templates as describes in our previous paper [17], while nano hydroxyapatite (nHA) was supplied as a nano-particles from (Merck, Darmstad, Germany Company) with average particle size (80 nm).

Four weight fractions include 1, 2, 3 and 5% of each nanoadditives to produce nanocomposites that prepared by weighing of chosen nanofillers (CNTs and nHA) to reinforce polymer matrix UHMWPE and mixed with 30 mL of ethanol and then stirred the mixture by hot magnetic stirrer for 45 min and 60 ºC to disperse the additive in solution. The final mixture (Ethanol + Additive + UHMWPE) was put in siliphon paper and input inside the dry oven for 20 min at 60 ºC, after draying it left in atmospheric for 72 hrs. to evaporate the residual ethanol.

The hydraulic hot press was used to fabricate UHMWPE nanocomposites. After the previous steps, the final produced mixture was put in a hot plate of the hydraulic press with a temperature range of (195-200 ºC) and then pressed under 12 MPa for 1.5 hrs at polymer Department in materials engineering college, Babylon university. Cooling the molds were done in the air to room temperature to get specimens and then they cut by CNC laser machine according to international standard specifications for each test in this research which agreement with ASTM standard.

2.2 Properties Measurement
2.2.1 Surface Roughness Test

The surface roughness test is achieved by the profilometer device from (Mahr Company, type Pocket Surf, USA), which supplied with a surface analyzer (sharp diamond stylus) and the maximum distance that can be move is (11mm). The profile of the surface irregularities and recess which characterized the surface by made scale. Each specimen was
tested three times on the different site at the same time and the average value can be estimated. The shape of specimens was disc shape with 4 cm in diameter and 0.4 cm in thickness.

2.2.2 Wet Wear

Wet wear of nanocomposites was calculated by weighing the pins of nanocomposites before and after the test using balance (accuracy: $10^{-4}$ g) in order to estimate the wet wear rate using the following relation [18]:

$$ W_v = \frac{\Delta m}{\rho \times t} \left( \frac{\text{mm}^3}{\text{sec}} \right) $$

(1)

where: $W_v$ is the volumetric wear rate, $\rho$ the density, $\Delta m$ the mass loss of the specimen after wear test and $t$ the abrading time.

The solution used in wet wear test was human body fluid when nanocomposites pins are cyclically rotated on SS 316 L disc with 75 kg load. The pins were rod shape with a square cross-section ($4 \times 4 \text{ mm}^2$) and put inside the cavity mold of the stainless steel shaft, the dimensions of the pin are 8 mm length and 4 mm thickness.

2.2.3 Density Test

The prepared nanocomposites were tested according to ASTM standard (D-792)[19] and the specimens weights were measured according to the Archimedes method by accurate balances. In this test, the volume of the sample should not be less than ($1\text{ cm}^3$) and free from oil, grease, and dust. The specimens were tested must be weighed in air and immersed in distilled water, and density can be obtained by applying equation (1) and (2) as follow:

$$ \text{Specific Gravity (S.G)} = \frac{W_D}{W_D - W_i + 0.02} $$

(2)

where: $W_D$: Mass of the sample in a dry state (g), $W_i$: Mass of the sample after immersing and suspended in water (g), and 0.02: Mass of practically immersed wire.

The density ($g/cm^3$) can be obtained by multiple specific gravities by the density of distilled water which is equal to (0.9975) [19, 20]:

$$ \text{Density} = S.G \times 0.9975 $$

(3)

2.2.4 DSC Analysis

The differential scanning calorimetry (DSC) test was performed according to (ASTM D3418) [21] using thermal analysis instrument (differential scanning calorimeter DSC) made by (Perkin-Elmer Corporation), type (Shimadzu-DSC-60), to measure the characteristic of thermal stability represented by melting and crystallinity temperature.

The measurements were carried out on specimens have mass (10 mg) at DSC programmed between (25 to 300 $^\circ C$) with heating rate of specimen (10 $^\circ C$/min) by increasing of temperature with scanning up to (300 $^\circ C$) and hold at this temperature between (5-10) min followed by cooling to room temperature. The specimen was placed in an aluminum crucible (cell) with a lid, but the reference crucible (cell) was blank.
3. RESULTS AND DISCUSSIONS

3.1 Surface Roughness of UHMWPE Nanocomposites

Figure (1) shows the relationship between the weight fraction of nano additive (CNTs and nHA) and surface roughness of nanocomposites. This figure indicates that the reinforcing with nanoparticles leads to the clear reduction in surface roughness due to fill the voids and get smoother surface with less defects. This reduction increases with increasing the weight fraction of additives and the addition of nHA gives lower roughness than addition of CNTs in nanocomposites which attributed to the nature and structure of additive type and the ceramic particles (nHA) that are very fine compared with organic molecules (CNTs) to decrease the surface roughness for the prepared nanocomposite [22].

![Figure 1: Surface Roughness for UHMWPE Nanocomposites with Different Additives](image)

The reduction in surface roughness was reached to (0.384 μm) for (UHMWPE/5%CNTs) and to (0.671 μm) for (UHMWPE/5%nHA) compared with the value of (0.883 μm) for pure UHMWPE.

3.2 Wet Wear of Polymeric Pin

The large problem happens in hip joint replacement (HJR) when be implanted in the human body is the wear debris that can be seen in polymer part for an acetabular cup that in publicity made from UHMWPE, where the debris consists of elements of implant failure to cause mechanical failure or inflammability.

In a current study, we used UHMWPE as matrix material for an acetabular cup and it was reinforced with nanomaterials include (CNT and nHA) to improve the mechanical properties for this part of the hip joint replacement. The effect of nanofillers in polymers for this application leads to decrease the wear rate of the contacting element.

Figure (2) shows the relationship between weight loss and weight fraction of additives in nanocomposites. This figure indicates the reduction in wear rate for nanocomposites compared with pure UHMWPE and the nHA gave more reduction in rate than CNTs due to the fine particles of hydroxyapatite that fill the voids to get less wear rate compared with nanotubes.
Ultra-high molecular weight polyethylene is a polymeric material that may be used in tribotechnical applications such as bearings, gears, and may be used in orthopedic applications for joints replacement due to its good properties like friction and wear resistance. Addition of carbon nanofibers into Ultra-high molecular weight polyethylene allows for improvement of tribological properties of the composite samples. It is clear that the addition of CNF with (0.1-0.5 wt%) give lowest wear rate during test.

The calculation of volumetric rate in mm$^3$/sec was done and from Figure (3), it can be noted that the volumetric rate decreasing when the weight fraction increasing for two nano additives to produce (UHMWPE/CNTs) and (UHMWPE/nHA) nanocomposites. This means that the nanofiller can be improved the wear resistance of nanocomposites. And that due to the filling the holes and voids in the polymeric matrix and enhance the cohesive among chains in product [23].

Also from Figure (3), can be seen that the volumetric wear rate of (UHMWPE/nHA) nanocomposite is lower than wear rate of (UHMWPE/CNTs) nanocomposite due to nature and structure of nano additive, where the particles have more ability to fill the voids compared with nanotubes and then give more cohesive matrix to withstand the contacting with other surfaces.

3.3 Density of UHMWPE Nanocomposites

The density of UHMWPE nanocomposite can be calculated according to Archimedes principle through weighing the sample in air and gas-free distilled water (0.998) at (27 °C) in an immersion medium using an electronic weighing balance with accuracy (10$^{-4}$ g). The measurement of density for composite is an essential indicator to know the lighter
composites, typically the composites have lightweight than metals and ceramics. Also, it is one reason behind the use of composites in engineering and biomedical applications.

Figure (4) shows the relationship between the weight fraction of (CNTs and nHA) in polymer matrix UHMWPE and the density of prepared nanocomposites. This figure indicates the increasing of density after reinforcing by nano additives, where the true density of CNT and nHA is (2.1) and (3.05) g/cm$^3$ respectively. In addition, these particles are made to diminish or fill the voids and spaces within the UHMWPE polymer matrix, i.e., the increasing in density in the presence of HA are due to fill the spaces between particles [24]. As a result, the nanoadditives gave denser composites with the same volume through filling the pores and voids instead of air. The ability of nHA particles to fill the voids is higher than nanotube (with special length) to filling, and then the density of nanocomposites reinforced with nHA was higher than that nanocomposites reinforced by CNTs.

![Graph showing density of UHMWPE nanocomposites with different additives](image)

**Figure 4: Density of UHMWPE Nanocomposites with Different Additives**

The highest density is (0.9533) g/cm$^3$ for (UHMWPE/5%nHA) followed by (0.9475) g/cm$^3$ for (UHMWPE/5%CNTs) nanocomposites compared with the density of polymer without reinforcing (0.931 g/cm$^3$). It can also be noticed that the density of UHMWPE slightly increased because the ratio of nanofiller to the volume of the nanocomposite is very small and the densities of the nanofillers are very low.

### 3.4 DSC Analysis

The thermal characteristics of UHMWPE nanocomposites of the first heating run are shown in Figures. (5) and (6). All nanocomposites show a characteristic endothermic melting peak, the melting temperature for pure UHMWPE is 138.87 °C. Reinforcing with CNTs led to little increasing in melting temperature ($T_m$) to reach 139.13, 139.05, 141.26 and 139.98 °C for weight fraction of 1, 2, 3 and 5% respectively. The highest $T_m$ was for UHMWPE/3%CNTs nanocomposite due to the better cross-linking between 3% CNTs and polymeric matrix as showed an improvement of mechanical properties. Generally, the reinforcing with nHA particles also gave higher melting temperature to record the values 139.33, 138.36, 140.08 and 140.55 °C at 1, 2, 3 and 5 % respectively.
After melting, we can see the peaks of crystallization and the crystallinity temperatures ($T_c$) was increased for nanocomposites compared with pure UHMWPE matrix due to the presence of CNTs and nHA crystals within polymer chains. The higher thermal stability of the nanocomposites as compared with the pure UHMWPE is attributed to the formation of a cross-linked network upon chain scission and improved compactness of the polyethylene. The melting temperatures ($T_m$) and crystallinity temperatures ($T_c$) are listed in Table (1).

| Sample                  | $T_m$ ($^\circ$C) | $T_c$ ($^\circ$C) |
|-------------------------|------------------|------------------|
| Pure UHMWPE             | 138.87           | 229.13           |
| UHMWPE/1%CNTs           | 139.13           | 232.43           |
| UHMWPE/2%CNTs           | 139.05           | 235.33           |
| UHMWPE/3%CNTs           | 141.26           | 232.35           |
| UHMWPE/5%CNTs           | 139.98           | 231.53           |
| UHMWPE/1%nHA            | 139.33           | 233.66           |
| UHMWPE/2%nHA            | 138.36           | 229.85           |
| UHMWPE/3%nHA            | 140.08           | 229.44           |
| UHMWPE/5%nHA            | 140.55           | 233.35           |

4. CONCLUSIONS

Adding CNTs and nHA to UHMWPE led to improving surface roughness, volumetric wear rate, density and thermal stability of produced nanocomposites due to the filling the voids in composite body and good cross-linking that occurs between nanofillers and matrix to get new composites with better properties for an Acetabular cup of hip joint replacement.

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