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Investigation of Compression and Hardness for UHMWPE Bio-composites as Internal Bone Plate Fixation

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HIGHLIGHTS

- Hybrid composite specimens of the fracture fixation device were made by the hot pressing technique.
- The weight fraction of nanoparticles and Types are most significant on the properties.
- The compression strength, hardness, and density increased with increasing nanoparticle weight fraction.

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ABSTRACT

Bone plates are essential for bone fracture healing because they modify the biomechanical microenvironment at the fracture site to provide the necessary mechanical fixation for fracture fragments. This paper addresses the use of composite bone plates in healing long-bone fractures such as transverse fractures of the femur. However, stress shielding in the bone due to metal plates can be reduced by designing implants with Bio-composites that involve Ultra high molecular polyethylene reinforced (UHMWPE) with Nano hydroxyapatite (n-HA) and Nano titanium dioxide (n-TiO_2) particles at different weight fraction (0,1.5,2.5,3.5and 4. 5%) and 5% of carbon and Kevlar fibers. FRIT spectrum was used to identify the incorporation between the matrix and Nano particles, and the shifting in main peaks confirmed the good cross-linking within the composite structure. The specimens thus prepared were subjected to a compression test, hardness test, and density. The results indicated that UHMWPE+4.5%n-HA+CF hybrid biocomposite has the highest compressive strength and hardness properties. In contrast, UHMWPE+4.5%TiO_2+CF has the highest density, which increased with increasing percentages of weight fraction of Nano-particles, where the compression strength 53 MPa, hardness property ranges 65.6 shore D, and density 1.09 (g/cm^3). According to the current study's findings, it is possible to create bio-composites as internal fixation device with improved performance by placing different fiber reinforcements.

1. Introduction

When a human bone fracture occurs, various internal fixation devices, such as bone plates, are placed at the fracture site to help stabilize the bone structure. Typically, internal fixation is carried out via open surgery using plates, screws, and wires [1]. Metal materials such as stainless steel and titanium and their alloys are not the ideal bone plate considering the adverse effects on callus formation and fracture healing caused by the high modulus of elasticity and biomechanical mismatch to the bone. Therefore, metal plates can cause regional osteoporosis and stress fractures with long-term implantation in the body. Therefore, a second operation may sometimes be required to remove the metal plates. [2]**.** To resolve these problems, polymer-based composite materials, which have less stiffness, high fatigue strength, and good radiolucency, have been proposed for bone plate fixations as an alternative to metal materials. Ultra-high molecular weight polyethylene (UHMWPE) is a widely used polymer in medical applications because of its high chemical resistance, biocompatibility, and mechanical and tribological properties. To further improve its mechanical properties and tribological response, fillers/reinforcements are incorporated into the polymer. Studies have been done to develop polymer-based composite materials as bone implants using natural fiber [3]. Synthetic fibers with unidirectional lamina [4], discontinuous short fiber [5], and braided fiber as reinforcement [6]. Balakrishnan Studied the mechanical, morphological, biocompatibility, and crystallization properties of HDPE/HA composites for bone replacement applications. [7]. Hashim investigated mechanical properties like (tensile, compression, and compact tension) under a flow of phosphate-buffered saline PBS at 37 ◦C of bone plate made from Epoxy and PMMA reinforced with natural fiber [3]. Alsoufi synthesized bio-ceramic components, including alumina (Al_2O_3) and titanium oxide (TiO2) to high-density polyethylene matrix composites for orthopedic applications (bone fracture plate, bone cement, bone graft, and hip replacement) [8]. Olewi found that

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the density of manufactured nanocomposites is due to the filling of the pores was increased in comparison to pure UHMWPE, which has many voids filled with air. These Nano additives are represented by carbon nanotubes (CNTs) and Nano-hydroxyapatite (n-HA) with four weight fractions (1, 2, 3, and 5 percent). Due to the effective filling of the pores, UHMWPE/n-HA composites increased in density more than UHMWPE/CNTs composites. [9]. Also, they studied Shore D hardness. It was found that the hardness is higher in the presence of CNTs than in the presence of n-HA, which is attributed to the purely mechanical properties of each additive compared to pure UHMWPE.[10]. The nanoparticle size directly affects the mechanical and physical properties and produces a biocomposite with suitable properties not for bone repair but for bone substitution [11]. Rija investigated the effect of adding Nano Al_2O_3 particles on the mechanical properties and inflammation behavior of Nano TiO_2/PEEK bio composite using an animal model [12]. Soundhar and Jayakrishna fabricated epoxy polymer composites reinforced with CTS nanoparticles for bone plate applications. They found that adding CTS improved strengths making it suitable for orthopedic applications[13]. Kureemun presented the application of carbon fiber/epoxy layers to cover the flax fiber/epoxy inner core, notably increased compressive properties from only flax fiber/epoxy[14].Qiao used n-HA/PA 66/GF plate for canine femur shaft fracture, they found n-HA/PA 66/GF plate have good strength in vivo which was the first important factor for an internal fixing plate[2].The aim of this research was introduce a new bone plate fixation materials for broken bones by manufacturing hybrid bio composites and study the effect of two types of particles (n-HA , n-TiO2) and two types of fibers (Kevlar and carbon fiber) in UHMWPE matrix. More changes in the qualities necessary for bone plate applications can be made by looking at the compressive strength, hardness, and density of various types of fillers and fibers in the polymer matrix as well as choosing the best component and Nano filler ratios.

2. Materials and Preparation of Bio-Composite

Materials used in femoral bone plate fixation for this research are ultra-high molecular weight thermoplastic polyethylene-UHMW-PE polymer powder with molecular weight 600-700 (104 g/mol.), density (0.93-0.94) (g/cm^3), and purity (≥99%) from LUOYANG MAX PIPE INDUSTRY as matrix materials, hydroxyapatite Nanopowder (with an average particle size of 50.28 nm) and titanium(IV) oxide Nanopowder (anatase phase with an average particle size of 20 nm), both from (Xian Real and Hangzhou Union in Biotechnology Company/China), as reinforcement material. The reinforcement materials were weighed by weight fraction (0,1.5, 2.5,3.5, and 4.5%). First, the powder particles are dispersed in ethanol with an ultrasonic device for 45 min for n-HA and 30 min for n-TiO_2. Then the UHMWPE is added to the nanoparticles simultaneously with mixing by mechanical mixing for 30 min to n-HA and 15 min to n-TiO_2 at 1500 rpm. To violate the ethanol, the mixture was placed in an oven at 60 C° for 2 hours and allowed to stand for 48 hours, tightly dry. After that, the mixture was placed in a mold and pressed in a hydraulic press at a temperature of 180 C° and a pressure of 12 MPa for one hour. Then the mold was allowed to air cool to room temperature to obtain the composite sheet, and then select the best composite properties were reinforced with two types of fibers (Kevlar and carbon) as one layer that led to obtaining hybrid nanocomposites. Table 1 shows the prepared samples. The samples were cut with a CNC laser machine according to the present study's ASTM standard for each test.

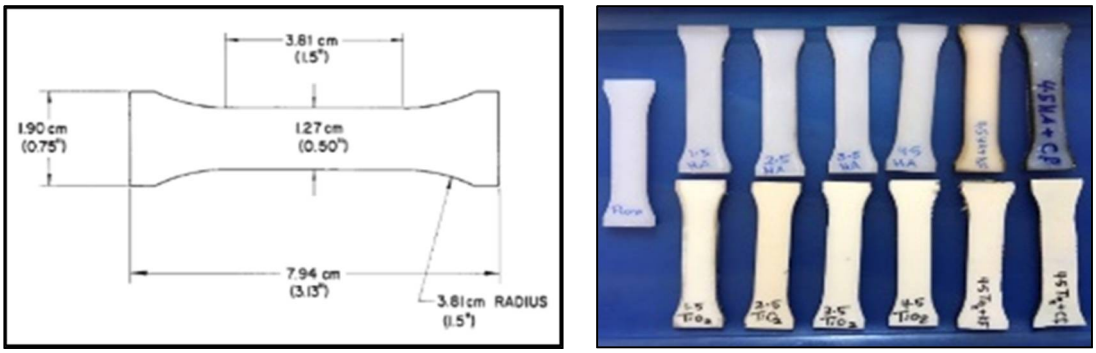
Table 1: Samples of Bio-Composite Materials

Groups	Wt.%
A	Neat UHMWPE
	UHMWPE - x HA Where x= (1.5, 2.5, 3.5, 4.5) wt.%
B	UHMWPE - x TiO_2 Where x= (1.5 , 2.5, 3.5, 4.5) wt. %
C	UHMWPE- 4.5 HA
	UHMWPE- 4.5 HA – 5% KF
	UHMWPE – 4.5 HA – 5% CF
E	UHMWPE- 4.5 TiO_2
	UHMWPE- 4.5 TiO_2– 5% KF
	UHMWPE – 4.5 TiO_2– 5% CF

3. Experimental Setup

3.1 Compressive Strength Test

As illustrated in Figure 1 a and b the test samples are cut from sheets following ASTM D695- 02a [15] The test is carried out until the sample fails at a speed of 1.3 mm per minute. Then, the stress-strain measurements are collected. The mechanical properties are calculated every time using the average data from the five tested samples [16].

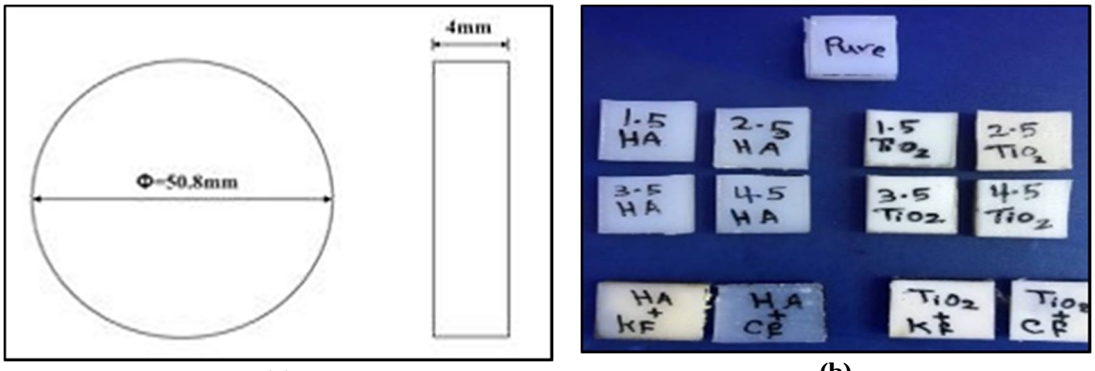


(a) **(b)**

Figure 1: (a) Standard Specimen of compression test, (b) Sample of the specimens for compression test

3.2 Hardness Test

To get the average value of these readings, a hardness test is necessary to measure the material's resistance to indentation, shore D, with a load equivalent to 50 N for a measurement duration equivalent to (15 sec) in seven different sites from the surface of the composite samples[16] . According to ASTM (D2240) [17]. Figure 2 a and b shows the prepared samples.



(a) **(b)**

Figure 2: (a) Standard Specimen of Hardness test, (b) Sample of the specimens for hardness test

4. Density Test

The test can be done according to the ASTM D-792[18, 19] by measuring the weight of the samples according to the Archimedes method by accurate balance using the displacement method. To determine the density, use the following formula:

$$\text{Specific gravity (S.G)} = W_D / (W_D - W_1 + 0.02) \quad (1)$$

Where:W_D: Mass of dry sample (g) W_I: Mass of the sample after submersing and suspended in water (g) and 0.02 mass of engaging wire

5. FTIR Spectroscopy

The (FTIR) test was achieved according to (ASTM E1252). After placing the specimen inside the device, the FTIR test was carried out in the air. Fourier transform analyses were performed for pure UHMWPE, n-HA, and n-TiO2 and UHMWPE nanocomposites reinforced by Kevlar and carbon fibers. Infrared spectrums were obtained in absorption and were set to operate in the range of (400 – 4000 cm^{-1}) at the thickness of specimens between the (4 mm) as rectangular rod form with cross-sectional area equal to (0.16 mm^2)[20].

6. Results and Discussion

Figure 3 shows the compression strength for the first and second groups (n-HA / UHMWPE) and (n-TiO_2/UHMWPE) biocomposites. Compression strength values for both groups enhanced as the weight fraction of both types of particles increased, and the maximum compressive strength was obtained at 4.5% for the two types of particles. This is due to the nature of bonding and the strengthening mechanism. It can also be affected by the compressive strength of n-HA and n-TiO_2 particles, which are significantly higher than UHMWPE[21]. From Figure 4 It can also be noticed that the addition of n-HA particles has a noticeable effect on the compression strength of composite specimen more than the n-TiO_2 particle. This is due to the improvement of the mechanical properties that are associated with the addition of HA particles, which have high compression strength compared with TiO_2 particles. Hence, the values of the compression strength increased from (21 MPa) for UHMWPE (as referenced) to (41.5 MPa) for (UHMWPE-4.5% n-HA) composite[19].

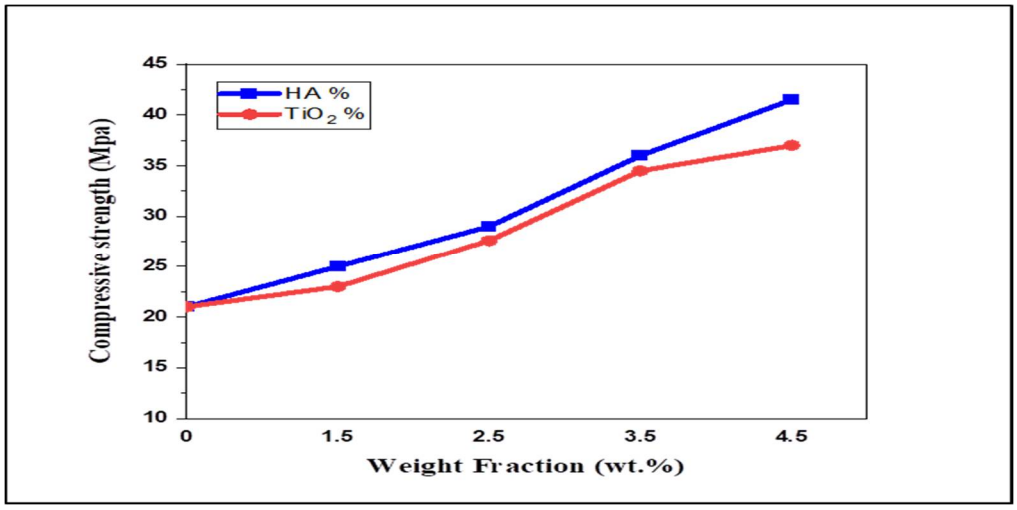


Figure 3: Compression Strength of Bio Composite materials as a function of nanoparticles (Wt.%)

Figures 4 shows the compressive strength of (n-HA and n-TiO_2) nanocomposites and the compression strength of the hybrid nanocomposite for the C and D groups, respectively. It can be observed that when adding carbon fiber or Kevlar fiber to the UHMWPE nanocomposite, the compression strength increases. This may result from the fact that carbon and Kevlar fibers are recognizable by their higher compressive strength than the UHMWPE matrix. As a result, the compressive strength of the hybrid composite was enhanced. Also, Figure 4 shows that the compression strength values for the hybrid nanocomposite specimens reinforced by carbon fiber are greater than those of Kevlar fiber. This is due to the characteristic that distinguishes carbon fiber from Kevlar fiber. The former has higher compression strength than the latter, besides the weakness of the Kevlar fibers toward the axial compression load, as it has anisotropic properties[18]. Figures 4 also demonstrate that the hybrid nanocomposite specimens' compression strength values raised when adding particles n-HA are greater than those obtained when adding n-TiO_2 particles. This is a result of the enhancement in mechanical characteristics produced by the incorporation of n-HA composites as compared to n-TiO_2 composites. Hence, the higher values of compression strength approach (53 MPa) for hybrid composite. (UHMWPE-4.5 n-HA% -carbon fiber) [22,23], which are below the extent of human bone. However compressive strength of human bone is about 100 MPa,[24,25].

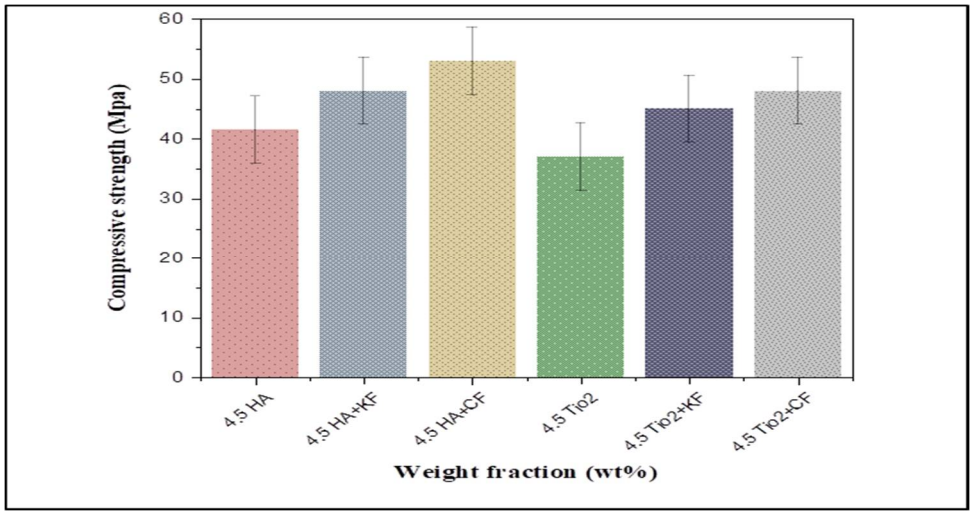


Figure 4: Compression Strength of Hybrid Bio Composite materials as a function of nanoparticles (Wt.%) and Type of fibers

Figure 5 shows the hardness for the first and second groups: UHMWPE /(n-HA) and (UHMWPE /n-TiO_2) biocomposites. It is clear that the hardness values for both groups improved as the weight fraction of both types of particles increased, and the maximum hardness was obtained at 4.5% for the two types of particles. This is because these particles have higher hardness and brittleness than the UHMWPE matrix [9]. Further, the wettability and bonding strength at the interface (between the matrix and these particles) contribute to forming a stiffer surface by inhibiting matrix mobility along the stress direction [26]. Also, Figure 5 shows that the addition of n-HA particles has a more significant effect on the hardness of composite specimens than the addition of n-TiO_2 particles. This is due to the different chemistry, surface roughness, and geometry of HA versus TiO_2 particles. Thus, the hardness values increased from (59.2) for UHMWPE (as referenced) to (64.2) for (UHMWPE-4.5%n-HA) composite[27 ,28 ,29].

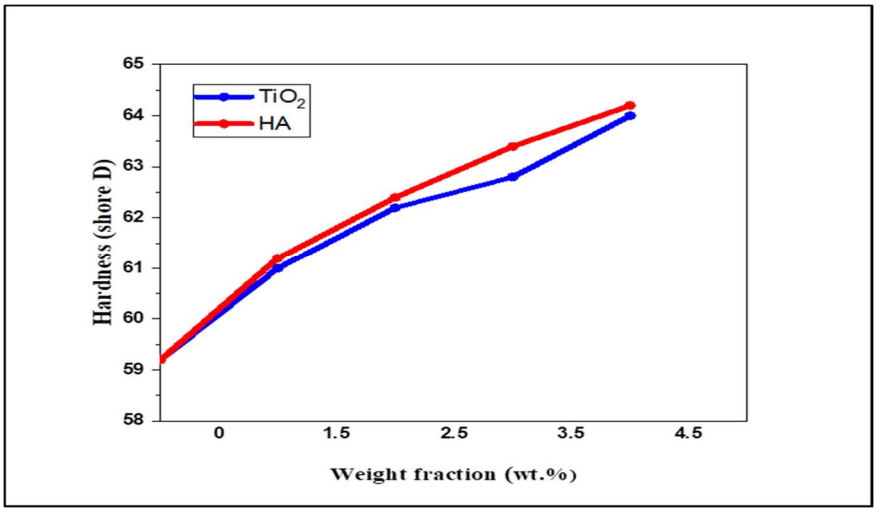


Figure 5: The hardness of Bio composite materials as a function of nanoparticles (wt.%)

Figure 6 shows the relationships between the hardness of (n-HA and n-TiO_2) nanocomposites and hybrid nanocomposites. It is observed that when adding carbon fiber or Kevlar fiber to the UHMWPE nanocomposite, the hardness increases. This may be explained by the reality that carbon and Kevlar fibers are recognized by their higher hardness than the UHMWPE matrix. As a result, the hybrid composite specimens' hardness was increased. This is because these particles have higher hardness and brittleness than other reinforced materials, such as (carbon fiber and Kevlar fiber) and UHMWPE matrix materials with low hardness values. Furthermore, high wettability between the matrix and this article makes the hybrid composite surface harder, which leads to restricting the movement of the matrix and low resistance to the load applied to it. Moreover, this figure shows the hardness values for the specimens of hybrid composite reinforced by carbon fiber are higher than the hardness values for specimens of hybrid composite that reinforced by Kevlar fiber. That is because carbon fibers have higher mechanical properties, are stiffer, and are harder than matrix polymer and Kevlar fiber. Therefore, the greater hardness values rise to (65) for hybrid composite (UHMWPE-4.5% n-HA-carbon fiber).

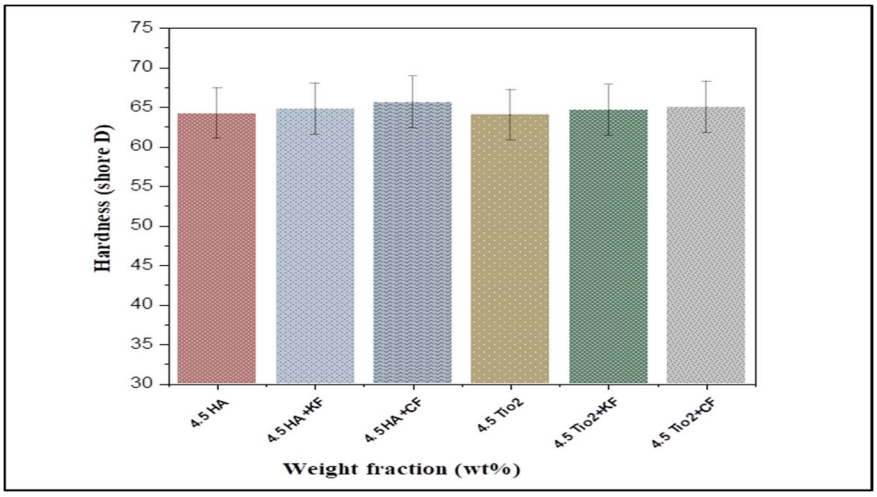


Figure 6: Hardness (Shore-D) of Hybrid Bio Composite materials as a function of nanoparticles (wt.%) and Type of Fiber

Figure 7 shows the density for the first and second groups: (UHMWPE/n-HA) and (UHMWPE / n-TiO_2) biocomposites. It is observed that the density values increased with increasing the weight fraction of both types of particles for both groups, and the maximum density was obtained at 4.5% for the two types of particles. These particles have a higher density value than the UHMWPE matrix. Additionally, these particles are designed to reduce or fill in the voids and spaces within. The UHMWPE matrix. Also, this Figure shows that the additions of TiO_2 particles have a noticeable effect on the density of composite specimens more than the HA particles. Therefore, the observed density values for the second group (TiO2-UHMWPE) Composite specimens have higher densities than those of the first group of composite specimens (HA-UHMWPE). As a result, adding nanoparticles made denser composites with the same volume by filling the pores and voids rather than air [30].

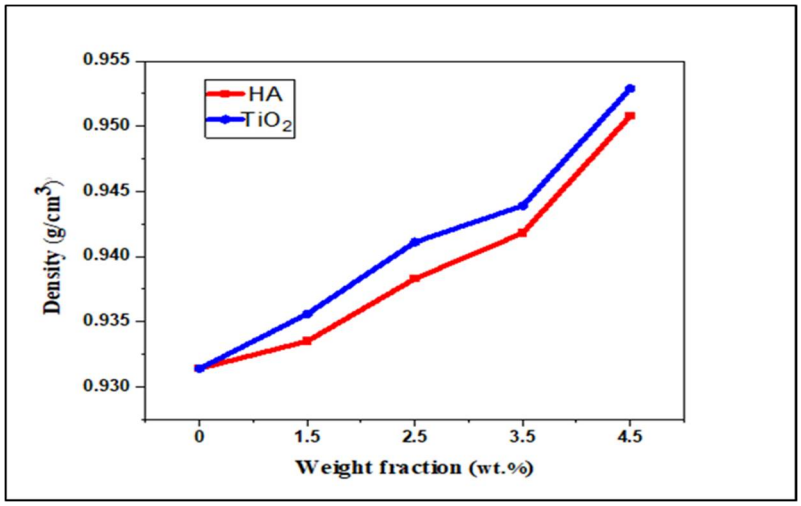


Figure 7: The density of Bio-composite materials as a function of nanoparticles wt.%

Figure 8 show the relationships between the density of (n-HA and n-TiO_2) nanocomposites and the density of the hybrid nanocomposite. The density of UHMWPE nanocomposite increases with woven carbon fiber or Kevlar fiber. This might be explained by the fact that the UHMWPE matrix is less dense than carbon fiber and Kevlar fiber, which led to the hybrid composite specimens having more density. Also, it can be observed from these figures that the density values for the nanocomposite reinforced by carbon fiber are higher than the values for the specimens of nanocomposite reinforced by Kevlar fiber. This is because carbon fiber has a larger density than Kevlar fiber, which are the features that set the former apart from the latter. Moreover, the maximum density values increased when adding carbon fiber to TiO2 nanocomposite specimens are more than the density values when adding Kevlar fiber to HA nanocomposite specimens. This is due to the density of TiO_2 particles being greater than the density of HA particles. Thus, the higher values of density reach (1.09gm/cm^3) for hybrid composite (UHMWPE-4.5% TiO_2-carbon fiber)[31]. Typically hybrid composite is lighter than metal plates like 316 L stainless steel alloy. This is another reason behind the use of hybrid composite in orthopedic fixation [32].

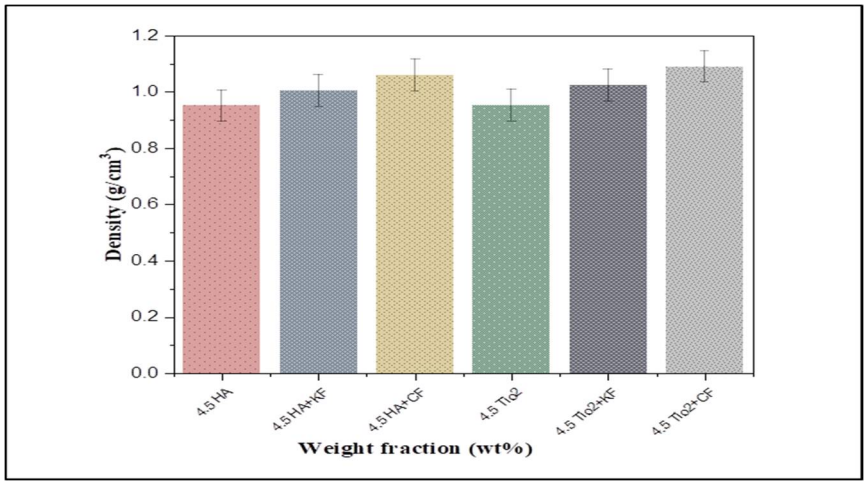


Figure 8: The density of Hybrid Bio Composite materials as a function of nanoparticles (wt.%) and Type of Fiber

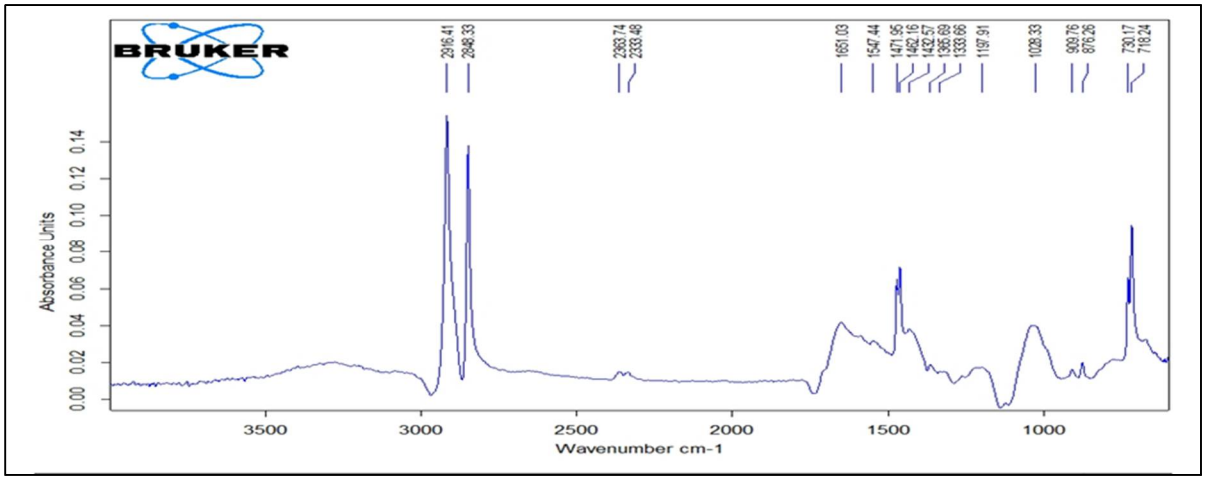
Figure (9-a) is the IR spectrum of UHMWPE. In this figure, many bands were represented, such as the bands at 2916.41 cm^{-1} and 2848.33 cm^{-1} for –CH_2– stretching, bands at 1462.16 cm^{-1} for CH_2 bending and the band at 718.24 cm^{-1} for CH_2 rocking, as shown in Table 2.

Table 2: The absorption bands of the IR spectrum characteristic of UHMWPE

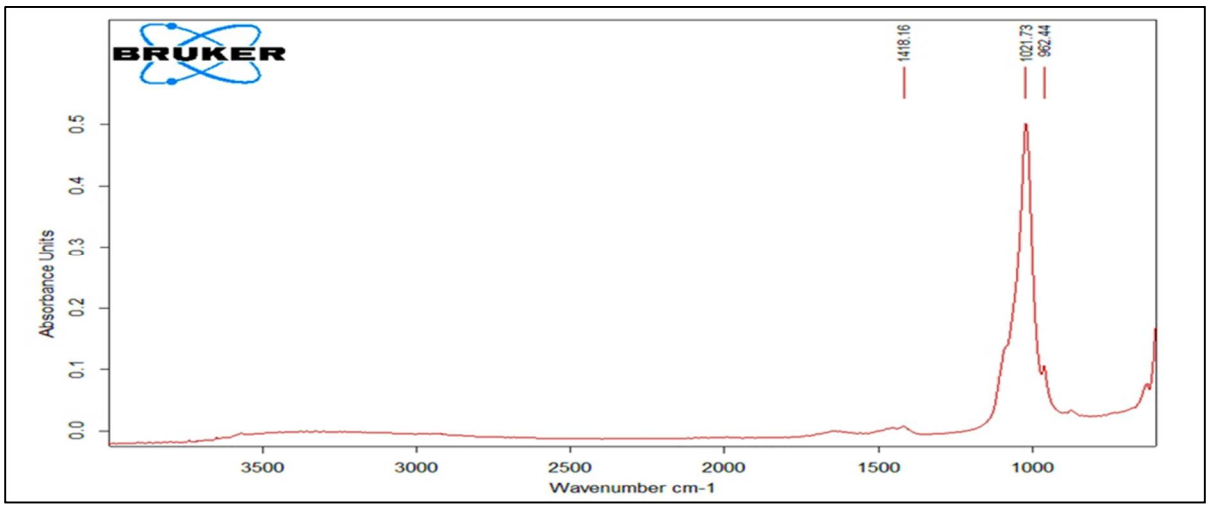
Type of bond	**UHMWPE(cm^{-1})**	**Reference**[33]
CH- Stretching	**2848.33**	**2851**
CH- Stretching	**2916.41**	**2919**
CH2- Bending	**1462.16**	**1462**
CH2- Rocking	**718.24**	**719**

Figure (9-b) is the IR spectrum of HA. In this figure, the absorption peak at 1418.16 cm^{-1} corresponds to the contracting vibration peak of O-H in H_2O; the absorption peak at 1021.73 cm^{-1} corresponds to the key band of PO^{-3}_4. The absorption peak at 962.44 cm^{-1} corresponds to the key band of PO_4^{-3}[34].

Figure (9-c) is the IR spectrum of TiO_2 representing an intense and wide band centered at ~3301.31 cm^{-1} attributed to the O−H stretching, as well as peaks at 1652.70 and 1033.34 cm^{-1} arising from the bending vibration of coordinated H_2O and Ti−OH [35] The peak at ~667.77 cm−1 is related to the Ti−O−Ti stretching, and that at 2361.12 cm^{-1} it has been assigned to TiO_2 lattice vibrations [36].



(a)



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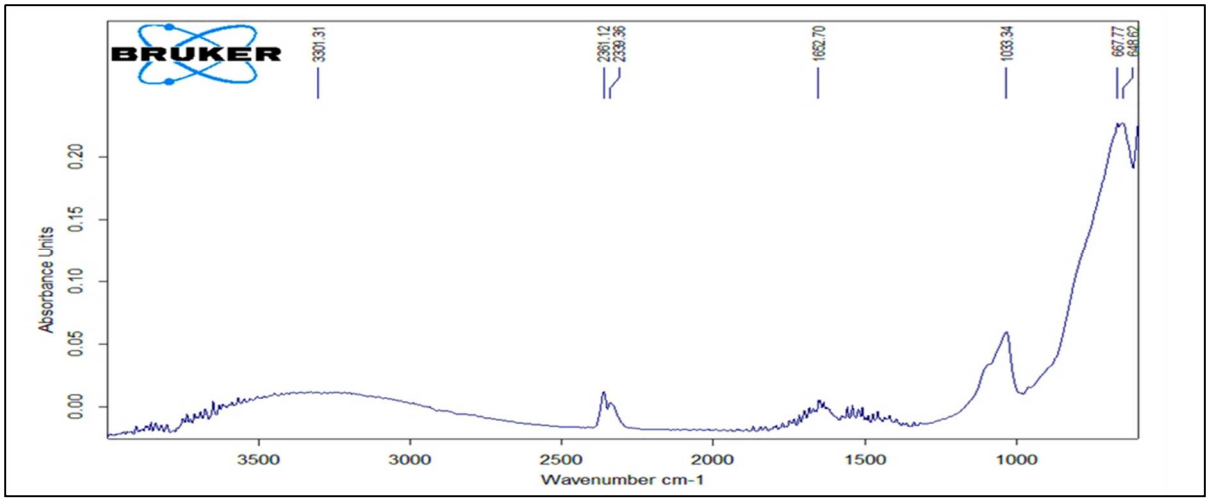

(c)

Figure 9: (a)FTIR spectrum of UHMWPE, (b)FRIT spectrum of n-HA, (c) FTIR spectrum of n-TiO_2

Figure 10 represents the IR spectrum of UHMWPE/HA Nanocomposites with 1.5 %, 2.5%, 3.5%and 4.5% of n-HA by weight fraction. At the wave number of 718.74 cm^{-1}, it is the rocking deformation of polyethylene[37]. At the wave number of 1462.44 cm^{-1}, it is the bending deformation of polyethylene. Similar behavior could also be observed at the wave numbers 2848.57 cm^{-1} and 2916.62 cm^{-1}, which indicate asymmetric stretching of CH_3-CH_2- and symmetrical stretching of -CH_2-CH_2- groups[38]. At the wave number of 1028.85 cm^{-1}, the orthophosphate group gives a broader band for Nano-HA/UHMWPE sample. Furthermore, there are some absorption peaks at the wave number of 1650.50 cm^{-1} for both samples, representing the wave number of carbonyl absorption peaks. Thus, it indicates oxidation in UHMWPE and Nano-HA/UHMWPE samples preparation from 3000-3500 cm^{-1}[39]. Oxidation can weaken the mechanical performance of UHMWPE [40]. So other sample preparation methods can be taken to eliminate oxidation.

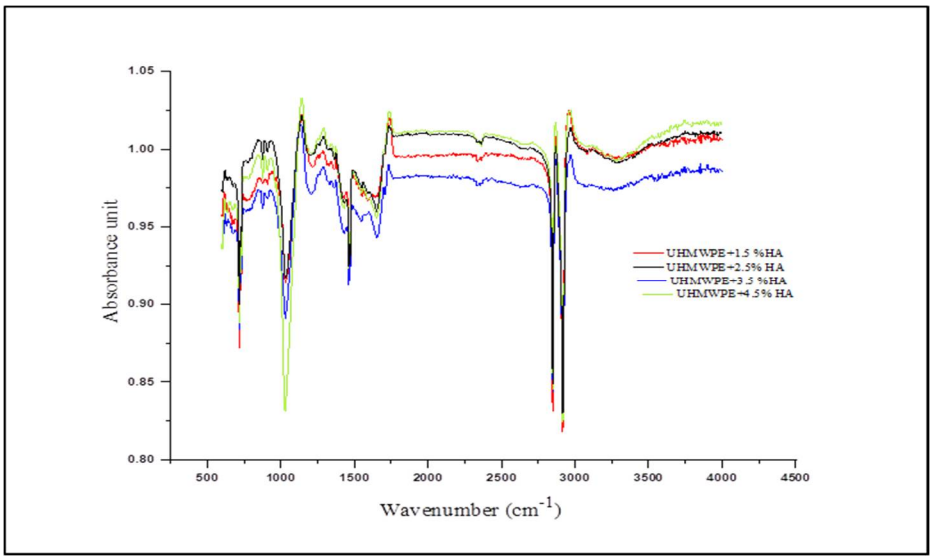

Figure 10: FTIR spectrum of (UHMWPE + x% n-HA) Bio-composites

Figure 11 represents the IR spectrum of UHMWPE/TiO_2 Nanocomposites with 1.5 %, 2.5%, 3.5%and 4.5% of n-TiO_2 by weight fraction. The modes detected for the UHMWPE Reference sample show peaks at 2916.23 cm^{-1} and 2848.28 cm^{-1}, which correspond to C-H's asymmetric and symmetric stretching modes. The mode at 1462.17 cm^{-1} represents the in-plane bending vibration of C-H, and the modes at 717.66 cm^{-1} are related to CH_2 rocking vibrations, which are attributed to the high degree of polymerization and long molecular chains of UHMWPE[41]. Incorporating the n-TiO_2 particles in the UHMWPE polymeric matrix promotes the C-O interaction, as it was detected in the modes at 1651 cm^{-1} (C=O) and 1262.11 cm^{-1} (C-O). The detection of such modes indicates that the n-TiO_2 particles are interacting with the polymeric chains; thus, the degradation of UHMWPE is caused by their photocatalytic activity [42].

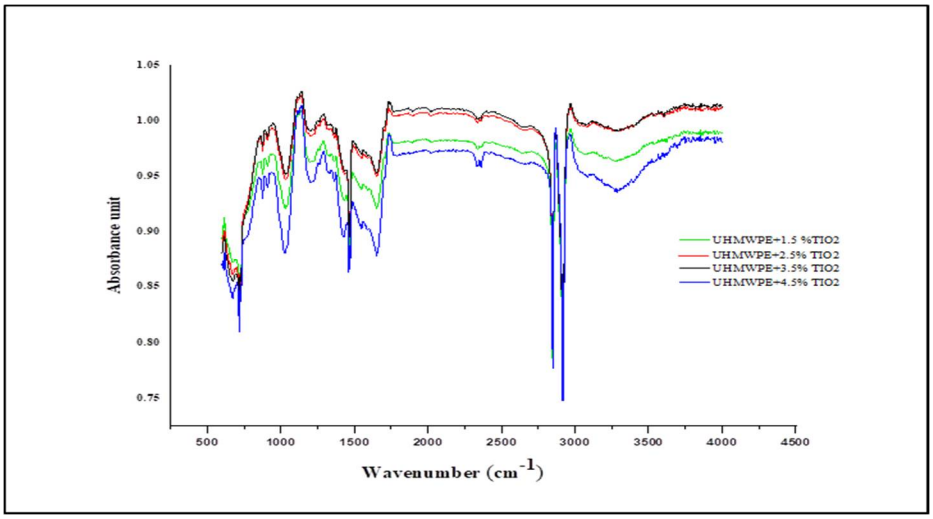

Figure 11: FTIR spectrum of (UHMWPE + x% n-TiO_2) Bio-composites

Figure 12 and 13 represent the IR spectrum of UHMWPE-4.5%n-HA with Kevlar fiber or Carbon fiber, UHMWPE- 4.5%n-TiO_2 Kevlar fiber or Carbon fiber hybrid composites, respectively. Again, it can be deduced that the chemical structure of the UHMWPE matrix was not observably altered by the addition of nanoparticles and KF or CF to the biocomposite specimens. Also, no new and manifest peaks shift are observed, which can prove the absence of cross-linking in these specimens.

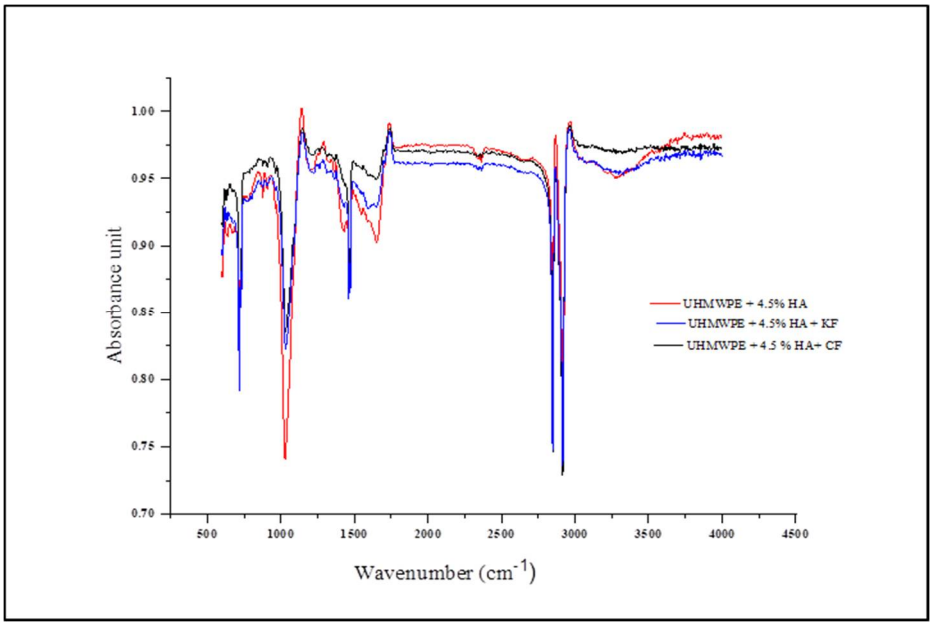

Figure 12: FTIR spectrum of (UHMWPE + 4.5% n-HA), (UHMWPE + 4.5% n-HA + KF) and (UHMWPE + 4.5% n-HA + CF) Bio-composites

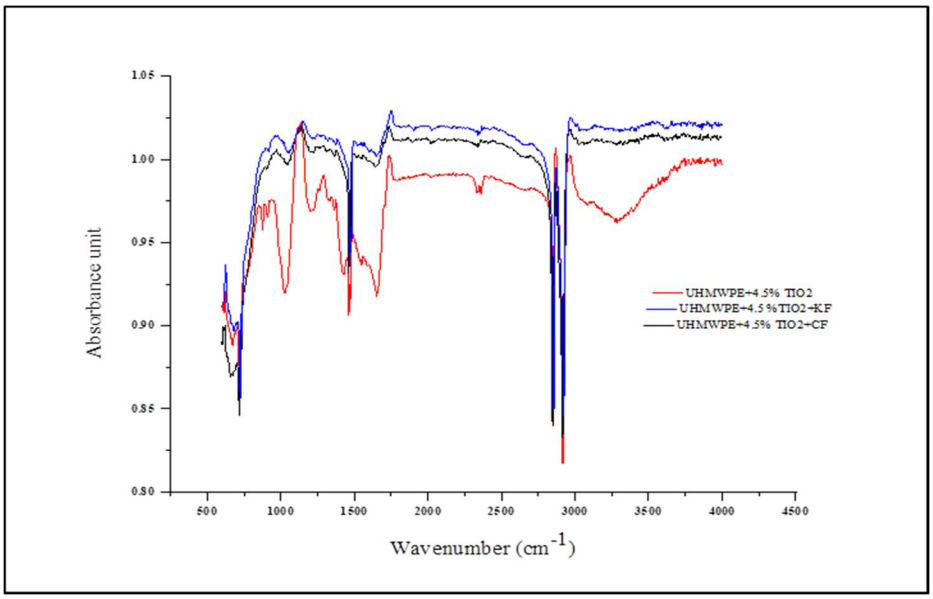

Figure 13: FTIR spectrum of (UHMWPE + 4.5% n-TiO_2), (UHMWPE + 4.5% n- TiO_2 +KF) and (UHMWPE + 4.5% n-TiO_2 + CF) Bio-composites

7. Conclusion

According to the experimental results, the properties of UHMWPE biocomposites increased with increased weight fraction of (n-HA and n-TiO_2) particles, and hybrid composites specimens increased with the addition of Carbon and Kevlar to both types of the nanocomposite for bone plate fixation. Moreover, hybrid composites specimens with Carbon fiber are higher than hybrid composites specimens with Kevlar fiber:

1) Compression strength increases with each increase in weight fraction of both types for nanoparticles, and the largest value is (53 MPa.) for UHMWPE+4.5 n-HA +CF.
2) Hardness increases with each increase in the weight fraction of both types for nanoparticles, and the highest hardness value was (65 Shore-D) for UHMWPE+4.5 n-HA +CF.
3) Density increases with each increase in weight fraction of both types for nanoparticles, and the highest density value was (1.09 Shore-D) for the specimen's UHMWPE+ 4.5% TiO_2 +CF.
4) The FRIT bands of Bio-composite materials are shifted by the addition of nanoparticles, and the main bands of the polymer were shifted by the addition of fibers without creating a chemical bond which indicated good incorporation between reinforcements (nanoparticles, fibers) and matrix, which referred to a better combination among them.

Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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