



## Influence of Nanoceramic on Some Properties of Polyetheretherketone Based Biocomposites

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### ABSTRACT

*Polyetheretherketone (PEEK) materials belong to a group of high-performance thermoplastic polymers thermoplastic that has been proposed as a substitute for metals in biomaterials. In this research, in order to improve the performances of PEEK, nano titanium dioxide (n-TiO<sub>2</sub>) and nano-hydroxyapatite (n-HAp) were incorporated into PEEK loading up to (1.5 wt%) to fabricate PEEK composites by using a method of melt-blending and hot compressing. Properties, such as compression, density, the morphology of fracture, and element analysis were examined for preparing samples. The results showed that the compression and density properties improved with increased weight fraction for two types of reinforcement, but the higher values obtained at (1.5 wt%) for two types of powders. It was found the higher compression strength and compression modulus obtained when reinforced with (1.5% n-HAp) which equal to (107.632 MPa and 3.991 GPa) respectively, than for samples reinforced with (1.5% n-TiO<sub>2</sub>) which equal to (91.579 MPa and 3.123GPa) respectively, while the density results have opposite behavior, it was found the higher values obtained when reinforced with (n-TiO<sub>2</sub>) than for samples reinforced with (n-HAp) and at (1.5% n-TiO<sub>2</sub>) the higher density, which equal to (1.3656) while at (1.5% n-HAp) which equal to (1.3425). Field emission scanning electron microscope (FESEM) manifested, that the fracture morphology transferred from brittle to ductile when reinforced with nano particles. Also, EDS analysis elucidated an identically uniform distribution of n-TiO<sub>2</sub> and n-HAp.*

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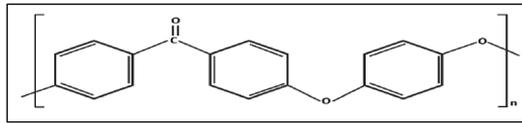
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## 1. Introduction

The dental implants raise the life quality for numerous patients with loss of a tooth. The selection of material for the dental implants is the titanium (Ti) that was first presented via Brånemark since titanium osseointegrates into the bone [1]. Nevertheless, titanium implants possess some difficulties. One of them is a potential allergy to titanium [2]. The other one is the greater stiffness of titanium, which possesses a greater modulus of elasticity (110 GPa) than that for the bone (10 GPa) [3]. This greater stiffness creates high peaks of stress during transfer the load at the interface between bone and implant, which may result in bone damage [4, 5]. Additionally, titanium can result in aesthetic problems owing to its color, which can exist as a greyish color in the peri-implanted soft tissue in a state of mucosal recession around the titanium implant. Presently, an attracted interest has been given to the ceramic dental implants produced from Zirconia due to their biocompatibility, high fracture toughness and, tooth-like color [6]. Nevertheless, Zirconia implants may possess a difficulty that concerns with the distribution of stress around the implant since it owns a high modulus of elasticity (210 GPa) in comparison with to that titanium (110 GPa) [7]. Also, Zirconia implants can result a high peak of stress at the interface between bone and implant like the state with the titanium implants [8-10]. New biocompatible materials and modern technologies have made it feasible to substitute many components of the human body. Purely polymeric materials, which are biocompatible, show better characteristics than the metal and to employ them for prosthetics that are in straight skeletal touch. The reason for that is they need a lower modulus of elasticity in order to become compatible in structure [11]. Polyetheretherketone ((-C<sub>6</sub>H<sub>4</sub>-O-C<sub>6</sub>H<sub>4</sub>-O-C<sub>6</sub>H<sub>4</sub>-CO-)n; PEEK) is the most high-performance thermoplastic material, which is a semi-crystalline thermoplastic polymer with almost (30%–35%) crystallinity [12]. Particularly, (PEEK) and its composites employed as implant materials to replace and repair the hard tissues of the human body have given a rising interest due to their excellent biocompatibility and mechanical properties [13, 14]. Bakar et al. fabricated hydroxyapatite and PEEK ranging from 5 to 40 vol%. It was obtained that the HAp amount in composite affected the tensile characteristics. The dynamic behavior due to tension–tension fatigue depicted that the (PEEK–HA) composites fatigue-life depended upon the content of (HA) in addition to the exerted load. The composite biological behavior was via in vivo assays, and the outcomes revealed high composite bioactivity [15]. Tang et al. investigated the cyclic loading on the (PEEK/HAp) composite with various (HAp) contents and illustrated that the (HAp/PEEK) composite is a promising composite to resist the fatigue for biomedical uses [16]. Wang et al. fabricated HAp/PEEK nanocomposite up to 15% of HAp in the PEEK matrix to improve the bonding between HAp and PEEK matrix. The mechanical, thermal behavior, microstructure, crystallization, and phase structure of the composites were examined. It was found that there is no debonding occurs between the well-dispersed HAp and PEEK matrix [17]. Wu et al. prepared n-TiO<sub>2</sub>/PEEK nanocomposites and assessed the in vitro and in vivo bioactivity of such materials, and the results showed that the nanocomposites bioactivity assessment manifested, that the osteoblasts pseudopods prefer to anchor at the areas where the (n-TiO<sub>2</sub>) exist on the surface [18]. Kumar et al. compared and quantified the osteogenic potential of untreated PEEK, PEEK coated with TiO<sub>2</sub>, and PEEK blended with TiO<sub>2</sub>. These materials were examined for cytotoxicity using human osteosarcoma cells, and alkaline phosphatase (ALP) activity was performed. The results made it evident that the PEEK coated by n- TiO<sub>2</sub> was more versatile biomaterial of selection in the dentistry implant proceeded by PEEK blended with n- TiO<sub>2</sub> and untreated PEEK. The SEM analysis result clearly showed the difference in matrix prior and beyond the adhesion of cells [19].

### 1. Characteristic of PEEK

Polyetheretherketone (PEEK) is a prevailing of PAEK (poly-aryl-ether-ketone) family of polymer that possesses high stability of temperature (more than 300 °C) and a high chemical and mechanical resistance. PEEK will be the first alternative for the metallic constituents in the trauma and orthopedics field [20-23]. It owns an aromatic molecular backbone with the combinations of ether (–O–) and ketone (–CO–) functional sets among the aryl rings, see Figure 1. Also, it possesses a low density (1.32 g/cm<sup>3</sup>), low insolubility, high stability, and permits elastic (3–4 GPa). The melting point (T<sub>m</sub>) temperature and glass transition temperature (T<sub>g</sub>) of PEEK are 334°C and 143°C respectively [24-26].



**Figure 1: Chemical Structure of PEEK**

### I. Inorganic Fillers Modification

Polyetheretherketone (PEEK) was reinforced via nanoparticle filler possesses excellent properties. The nanoparticles are the major modified material for (PEEK) due to the excellent mechanical characteristics, the influence of dimension, and the activity of surface [27].

Hydroxyapatite (HAp),  $[\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2]$ , is a calcium phosphate chiefly aware of its uses in the replacement of bone [28]. In the recent years, HAp reinforced polymer composites have taken like cobalt-chromium alloys and stainless-steel alloys, the biomaterials next generation, for example, (HAp) reinforced polymer composites, with the bioactive (HA) inclusion not only coincides the mechanical characteristics (i.e. strength and stiffness) of the bone, but also imitates the bony tissue via creating a biological response that encourages the growth of bone on implant [29]. Titanium dioxide ( $\text{TiO}_2$ ) is well recognized as mechanically robust, biologically, and chemically inert, super-hydrophobic, antifogging, biocompatible, nontoxic, and cheap [30]. It's broadly utilized in the environmental uses, coatings, toothpaste, foods, cosmetics, paper, and paint due to its green color, low cost, clean, and sustainable innovation that frequently utilized as the polymer matrix reinforcement phase to enhance its mechanical characteristics [31].

The main objective of the present study was to obtain some properties of novel composite materials used as dental implants made by PEEK reinforced with n-HAp and n- $\text{TiO}_2$  separately at different weight fractions (0, 0.5, 1, 1.5) % for two types of reinforcement. A compression test was conducted in order to evaluate the compression modulus and compression strength. Also, a density test was performed to compare the density value for the resultant composites. Moreover, Scanning Electron Microscope (SEM) with energy X-ray dispersive was done to illustrate the morphology of fracture surface with element analysis of polymer composites.

## 2. Materials and Experimental Methods

### I. Sample Preparation

PEEK (551G) granule, Nano hydroxyapatite, and Nano titanium dioxide were purchased from (Jilin Jointure Polymer Co., Ltd, China), (N & R Industries, Inc, China) and (Hangzhou Union Biotechnology Co., Ltd, China) respectively. The size of the HAp and  $\text{TiO}_2$  nanoparticles was 20nm. The biopolymer composites containing (0, 0.5, 1, 1.5) wt% for two types of nano powder were fabricated via a series of processes compounding and hot press process. PEEK granule was dried in an oven 80 °C for 1 hr prior to compounding and hot press. In brief, nanopowder was dispersed in ethanol alcohol using an ultrasonic mixer with variables (90 W power, 0.5 pulses, and (5-10) min) to obtain a homogeneous mixture and then mixing with PEEK granule. After the proper dispersion, the blend was dried in an oven at temperature (90 °C) for a period of 24 h for removing the excess ethanol alcohol.

In the compounding process, compounding was conducted in an internal mixer (Haake) type (HBISYSTEM 90, AHAKE BUCHLER PRODUCT, USA) at (360°C) and (90 rpm) blending speed. The required time for the compounding was modified correspondingly (20 min). Then, the hot press by using a mold with dimension (17\*17\*0.4)  $\text{cm}^3$  was preheated at temperature 200 °C and covered from the top and bottom by a cover withstand a high temperature in order to prevent adhesion of polymeric composite to mold and put in hot press type (TOYOSEIKI, Japan). The temperature in the hot press was adjusted at 360°C when reaching this temperature, the pressure was applied for (15) min, and its value equal (15MPa). The mold was then removed from the hydraulic press and put in the cooling system to be cooled using (5 L/min) jet of water at room temperature. It was then opened, and after that, the composite sheet was taken to cut the specimens by CNC machine for each test.

### II. Characterization

Samples for compression test were prepared according to standard ASTM D695-2002a at across head speed (strain rate) of (5mm/min) this test was done by using a universal testing machine type

(LARYEE, China). The following formula was used for calculation of compression strength and compression modulus [32]:

$$\text{Compressive Strength} = \frac{F}{b h} \quad (1)$$

$$\text{Compression Modulus} = \frac{\Delta\sigma}{\Delta\varepsilon} \quad (2)$$

Where  $F$  = force (N),  $b$  = width of specimen (mm), and  $h$  = thickness of specimen (mm),  $\Delta\sigma$  = changes measured in stress (MPa),  $\Delta\varepsilon$  = change measured in strain.

The density test is performed for specimens via employing the displacement approach depending upon the theory of Archimedes. A simple way to determine the density of a specimen is to weigh it in the air and then weigh it again when it is immersed in a liquid. Water is the most convenient liquid to use. The specific gravity is a measurement of the ratio of material density to the density of gas-free distilled water at (25 °C) without units, as represented in the following equation:

$$\text{Specific Gravity (S.G)} = \frac{W_D}{W_D - W_i + 0.02} \quad (3)$$

Where:

$W_D$ : is of the sample mass in dry state, gm.

$W_i$ : is the sample mass after the immersing and suspending in water, gm.

0.02: is the mass of practically immersed wire.

The specific gravity can be changed to density ( $\text{gm.cm}^{-3}$ ) via multiplying it by  $D$ , which is the distilled water density ( $\text{gm.cm}^{-3}$ ), which equals to 0.9975 [33]. That means:

$$\text{Density} = (\text{Specific gravity}) \times (0.9975) \quad (4)$$

This test was performed on (RADWAG, Poland (EU)) instrument.

The morphology of the sample was obtained by field emission scanning electron microscopy FESEM (TESCAN-type MIRA3, Czech) under a low-vacuum mode with (15 kV) primary electron energy prior to the observation; the specimens were coated with a gold layer. The sample element composition was analyzed via the energy dispersive X-ray (EDS) by employing the same SEM device.

### 3. Results and Discussion

#### I. Compression Test

The relationship between the compression strength of the composite materials and the weight percentage of nanoparticles showed in Figure 2. It was observed the addition of n-HAp and n-TiO<sub>2</sub> as reinforcement, increased in compression strength of composite material with increasing percentage of reinforcement content namely (0, 0.5, 1, and 1.5) % for two types of reinforcement separately. The enhancement in compressive strength of the nanocomposites was contributed to better interactivity of nanoparticles with a polymer chain. That can explain nanoparticles on impeding the cracks movements were led to increment in compression strength. Also, the properties of polymer composites depend on the natural bond and chemical composition of the reinforcement and the degree bound between reinforcement and resin matrix.

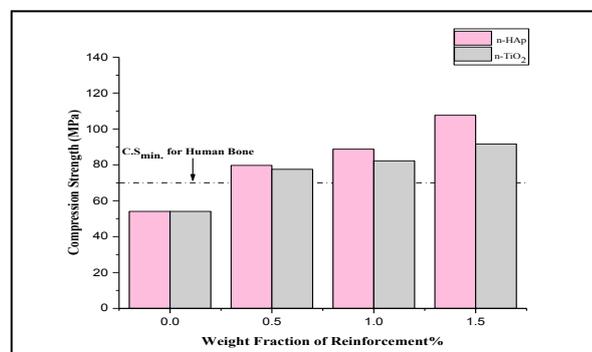
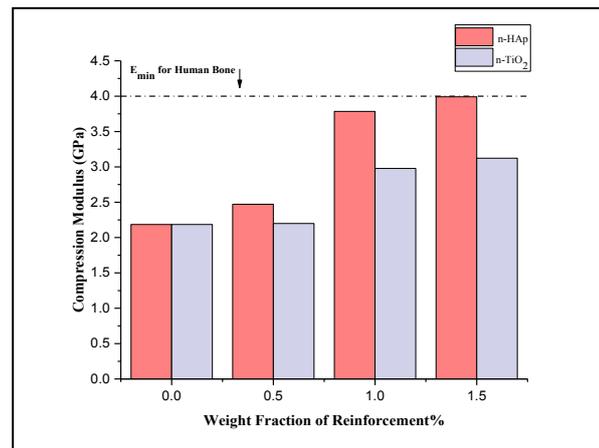


Figure 2: Compression Strength Results for Composite Reinforced with Nano Particles

The maximum compression strength happened at 1.5% for two types of reinforcement. Similar to compression strength, Figure 3 shows the compression modulus also increases with an increasing percentage of n-HAp and n-TiO<sub>2</sub> in the PEEK matrix. Also, it was found that the addition of n-HAp improves the compression properties more than the n-TiO<sub>2</sub>. Furthermore, the compression strength

and compression modulus were improved by (99%) & (83%) respectively, which obtained at (1.5% n-HAp) while at (1.5% n-TiO<sub>2</sub>) which equal to (69%) & (43%) respectively.

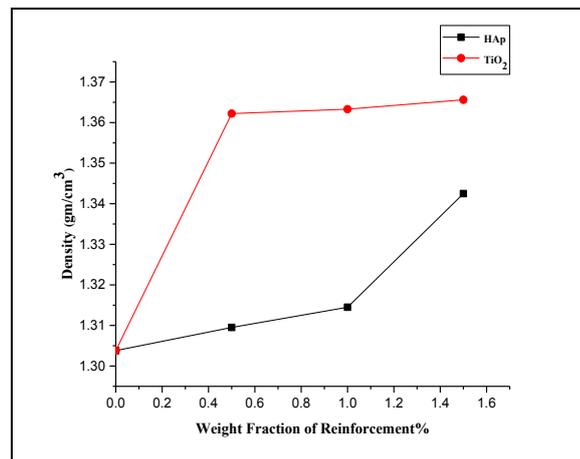


**Figure 3: Compression Modulus Results for Composite Reinforced with Nano Particles**

It is very important for the implant to match the properties of implant materials with human bone. As shown in these figures, when compare the compression strength obtained from compression tests the compression strength value of the pure PEEK was lower than that of human cortical bone (70-280) MPa, while the compression strength for the composites was within range of human bone. Moreover, the compression modulus obtained from experimental tests it was found that the modulus of elasticity of 1.5 % n-HAp nanoparticles reinforced PEEK was within the range of 4-22GPa (the modulus value of human cortical bone (4-22GPa) [34].

## II. Density Test

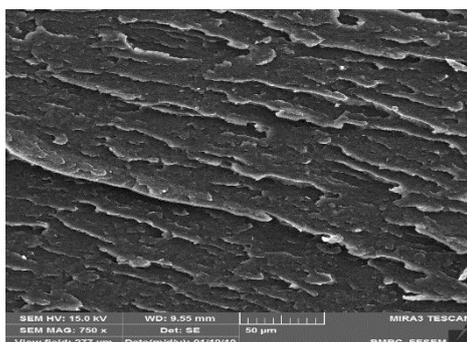
Figure 4 demonstrated the density values for composite samples with different weight fractions of (hydroxyapatite and titanium oxide). The results indicated that the density of biocomposite was closely correlated with the content of nanoparticles, which indicated the increase of density after reinforcing by nanoparticles. This increment contributed due to good bonding between nano particles and PEEK matrix without changing the cross-linked or branched structure of the composite material. Other factors such as Interparticle distance, distribution of reinforcement particles, the weight density difference of reinforcements and matrix polymer, and so on decide the final density [35]. Additionally, such particles are produced for diminishing or filling the spaces and voids within the matrix of PEEK. As a result, the nanoparticles gave denser composites with the same volume through filling the pores and voids instead of air [36]. Also noted from this figure the ability of n-TiO<sub>2</sub> to fill the voids is higher than n-HAp for filling, so the density nanocomposites obtained when reinforced with n-TiO<sub>2</sub> was higher than nanocomposites reinforced by n-HAp. It was worth noting that the composites contain 1.5% n-TiO<sub>2</sub> has the maximum value of density was (1.3656) g/cm<sup>3</sup>, while the density of composites reinforced with 1.5% n-HAp was (1.3425) g/cm<sup>3</sup>.



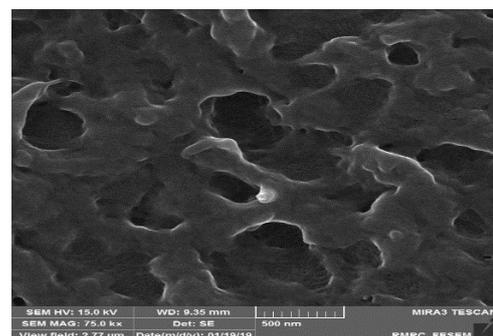
**Figure 4: Density Results for Composite Reinforced with Nano Particles**

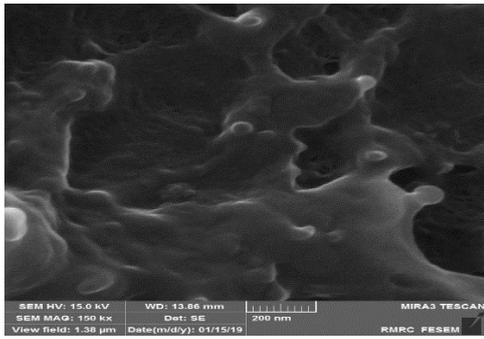
### III. Scanning Electron Microscopy (SEM) with Energy Dispersive X-Ray Spectroscopy (EDS)

The examination of fracture zone results from a tensile test for polymer samples which illustrated in previous work [37] was done by (SEM) technique at both low and high magnification with EDS spectra. It can be observed in Figure 5 for neat PEEK brittle fracture that shows the materials in the glassy state and the brittle fracture of PEEK matrix is observed with the broken shape like a wave at the ambient temperature. The samples containing n-HAp and n-TiO<sub>2</sub> showed that the fracture was ductile and the dark regions are the PEEK matrix while the nanoparticles are visible throughout the image as white spots appeared to become well-embedded into the matrix of PEEK. Nano-particles were measured to become almost (20nm) in diameter that matches the specifications of the manufacturer. Despite the nanoparticles are prevailing across the surface of fracture [38]. On the other hand, it's noticeable nanopores, which indicate the location of particles formed as a result of the fracture samples. The EDS analysis provides an effective means of identification of the elemental composition of specimens as shown in Figure 6. The results showed the similarly uniform homogeneous spread of the (n-HAp) and (n-TiO<sub>2</sub>), and from the EDS spectrums, it was found that the intensity of C peak decreased, while the intensity of n-HAp and n-TiO<sub>2</sub> increased when added to the matrix. Also, the mapping images show that Calcium (Ca), Phosphate (P), and Titanium (Ti) elements are homogeneously distributed throughout the material. Also, this study confirmed the existence of the matrix of the (PEEK) polymer via the observations of Oxygen and Carbon. From the analysis of EDS, it was found that the ratio of Ca/P is 1.67 in the n-HAp which is similar to the human body (1.67).

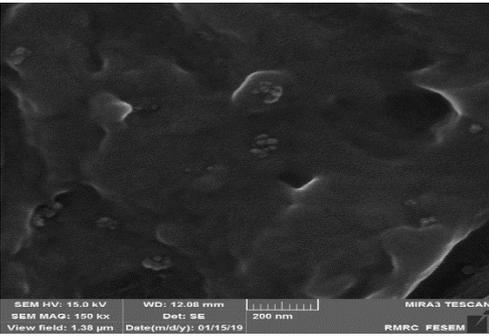
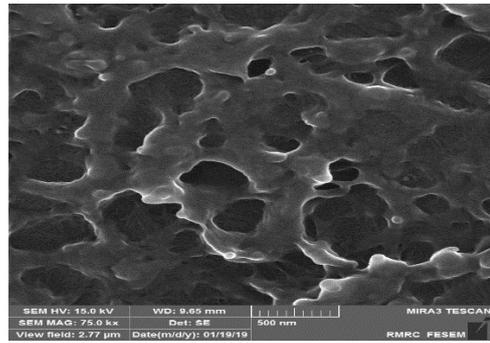


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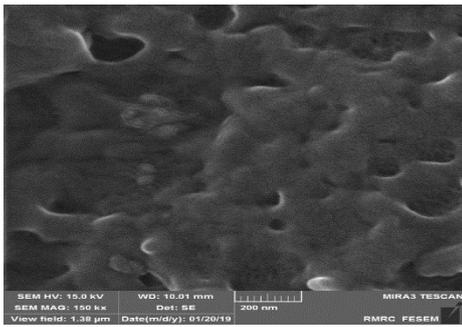
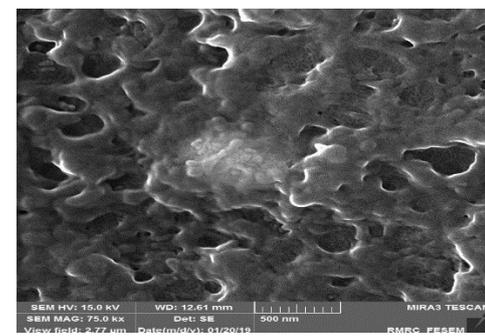




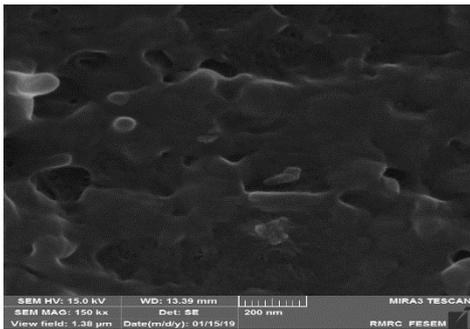
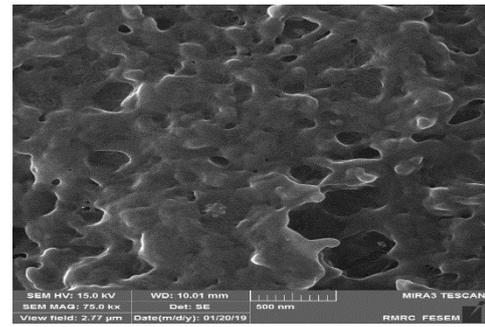
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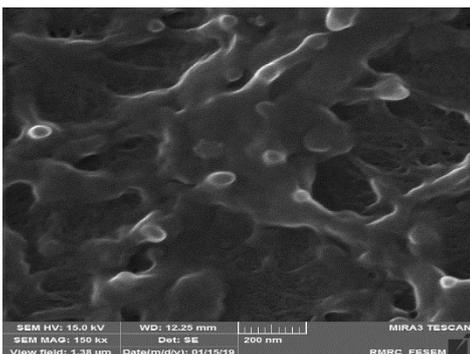
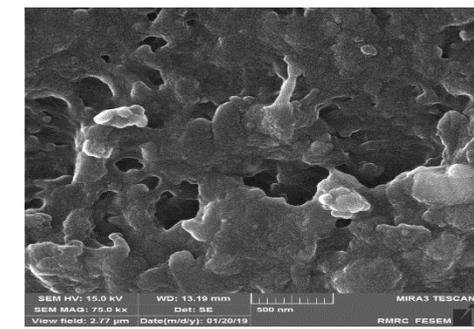
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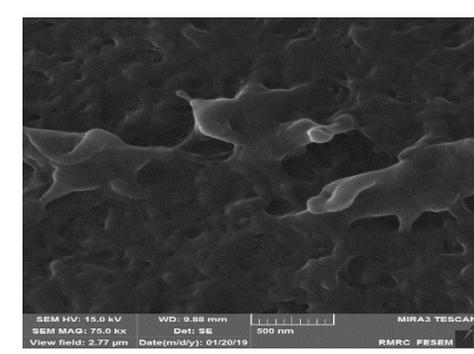
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e)



f)



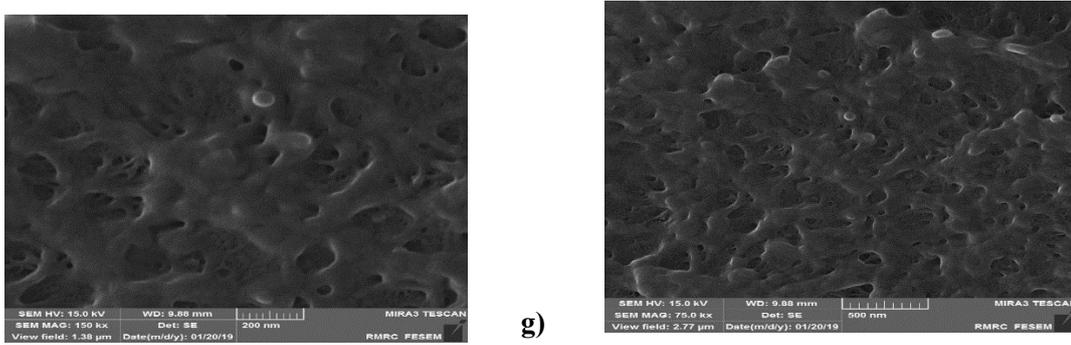


Figure 5: SEM Results for a) Neat PEEK b) 0.5%HAp/PEEK c) 1%HAp/PEEK d) 1.5%HAp/PEEK e) 0.5%TiO<sub>2</sub>/PEEK f) 1% TiO<sub>2</sub>/PEEK g) 1.5% TiO<sub>2</sub>/PEEK

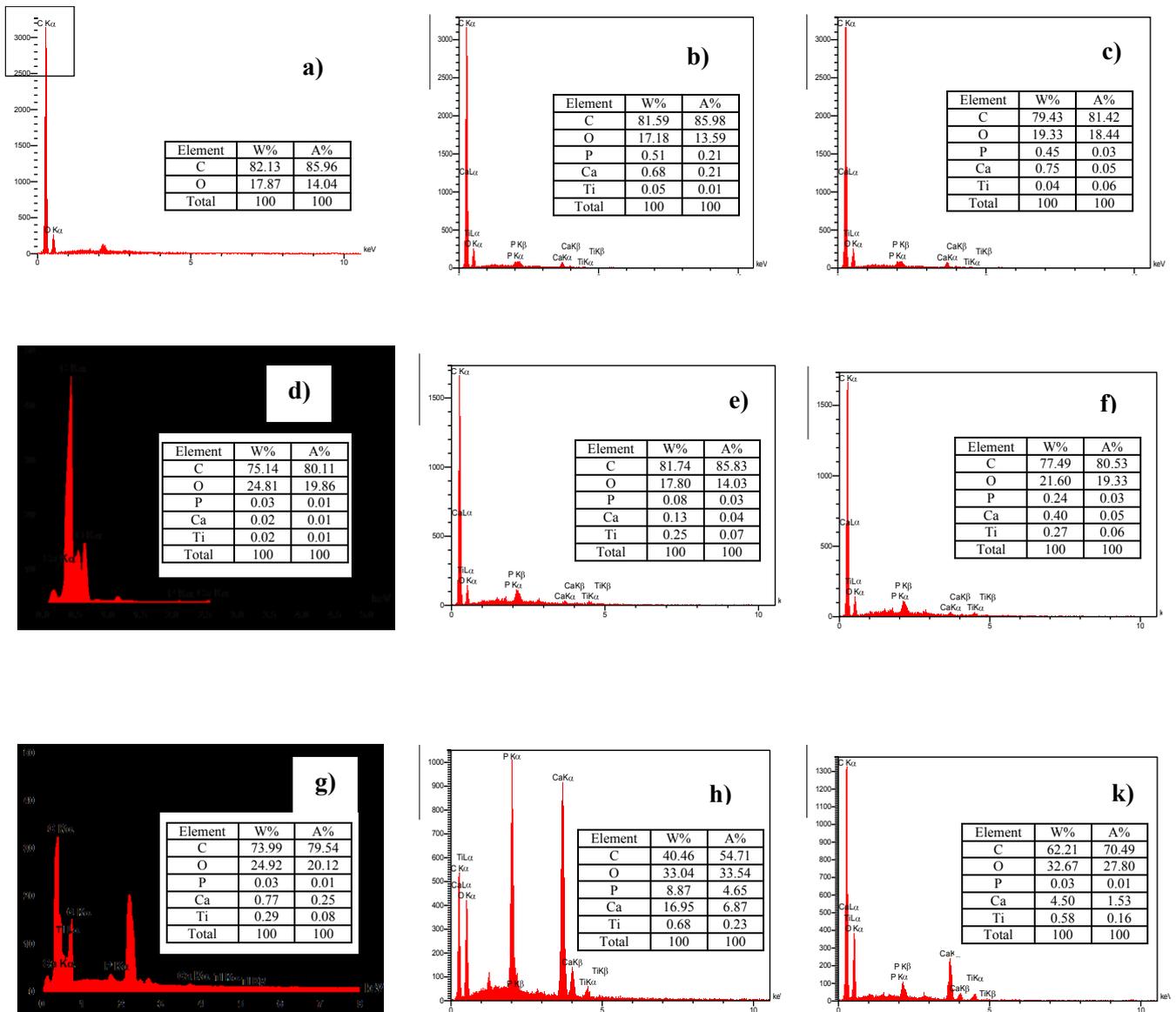


Figure 6: EDS Analysis Results for a) Neat PEEK b) 0.5%HAp/PEEK c) 1%HAp/PEEK d) 1.5%HAp/PEEK e) 0.5%TiO<sub>2</sub>/PEEK f) 1% TiO<sub>2</sub>/PEEK g) 1.5% TiO<sub>2</sub>/PEEK h) n-HAp powder k) n-TiO<sub>2</sub> powder

#### 4. Conclusions

In this study, it was found that:

1. The compression behavior of polymer composite improved by the increased weight fraction of powder, but the higher values obtained at (1.5wt%) for two types of powders. compression strength and compression modulus was improved by (99%) & (83%) respectively, which obtained at (1.5%n-HAp) while at (1.5%n-TiO<sub>2</sub>) which equal to (69%) & (43%) respectively.
2. The density of polymer samples increased with an increasing weight fraction of powder, but the higher values obtained at (1.5wt%) for two types of powders.
3. FESEM shows that the fracture morphology was the transfer from brittle to ductile when reinforced with Nano-particles.

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