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Improvement of mechanical and thermal properties of high energy electron beam irradiated HDPE/hydroxyapatite nano-composite

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HIGHLIGHTS

- HDPE/HAP nano-composite sample were prepared in nano-powder form.
- All the samples were made in thin sheet form using warm press system.
- The samples were subjected to irradiation under 10 MeV EB in different doses.
- Mechanical and thermal properties of the samples were compared.
- Mechanical and thermal parameters are strongly dependent on HAP dispersion.

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ABSTRACT

In this research work, the nano-composites of high density polyethylene/hydroxyapatite samples were manufactured via two methods: In the first method, the granules of high density polyethylene and nano-structure hydroxyapatite were processed in an internal mixer to prepare the nano-composite samples with a different weight percentage of the reinforcement phase. As for the second one, high density polyethylene was prepared in nano-powder form in boiling xylene. During this procedure, the hydroxyapatite nano-powder was added with different weight percentages to the solvent to obtain the nano-composite. In both of the procedures, the used hydroxyapatite nano-powder was synthesized via hydrolysis methods. The samples were irradiated under 10 MeV electron beam in 70–200 kGy of doses. Mechanical, thermal and morphological properties of the samples were investigated and compared. The results demonstrate that the nano-composites which we have prepared using nano-polyethylene, show better mechanical and thermal properties than the composites prepared from normal polyethylene granules, due to the better dispersion of nano-particles in the polymer matrix.

1. Introduction

In recent years, due to the growing number of the traffic accidents, and also increasing human life expectancy, most of people in the world have to use at least one orthopedic or dental implant. Natural bone is a nano-composite consisting of a mineral fraction including small apatite crystals and non-stoichiometric calcium phosphate, a mixture which affers excellent mechanical resistance (Glimcher, 2006). Pure hydroxyapatite (HAP) is $Ca_{10}(PO_4)_6(OH)_2$ which is similar to the chemical composition of HAP found in the hard tissues of the body such as the bone and teeth (Afshar et al., 2003). The high density polyethylene (HDPE)/HAP composites were developed by Bonfield and coworkers (Bonfield et al., 1981) and have been used as clinical application of the bone implant for over 20 years (Tanner et al., 1994). In the wake of this procedure, the scientists focused on developing of the mechanical and morphological properties of this composite. There were many ways to produce the polyethylene (PE)/HAP composites (Kamei et al., 1997). High density polyethylene (Pandey et al., 2006; Wang and Porter, 1994) and ultra-high molecular weight polyethylene reinforced by HAP were produced and studied (Crowley and Chalivendra, 2008; Shokrgozar et al., 2010). There were a variety of sizes, morphologies and shapes of HAP to improve the mechanical properties of the composites such as micro-sized particles and nano-sized particles (Bonfield et al., 1981; Crowley and Chalivendra, 2008; Fekete et al., 1999; Fu et al., 1993; Huang et al., 1997; Roeder et al., 2003; Wang et al., 1997).

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Several methods have been used to combine PE and HAP such as extrusion (Fang et al., 2007; Huang et al., 1997; Wang and Porter, 1994), blending with compounding (Homaeigohar et al., 2006), mixing (Shokrgozar et al., 2010; Soltani et al., 2013), using solvents (Carmen et al., 2006), hot- milling prior to injection molding (Pandey et al., 2006). On the other hand, one of the ways to improve the physical properties of the HAP/HDPE composite is to use radiation. The effects of gamma irradiation on the mechanical and morphological properties of HDPE/HAP composite prepared via the solution method has been studied (Carmen et al., 2006). Another important scientific issue in preparing the composite instance of implants is homogeneity dispersion of the nanoparticles used as the reinforcement phase. In fact, agglomeration is one of the major problems in composites.

In our previous work, the effect of high energy electron beam irradiation on the mechanical, thermal and morphological properties of the PE/HAP nano-composite has been investigated (Soltani et al., 2013).

In this research work, it was tried to prepare the HDPE/HAP nano-composite samples with a perfect homogeneity of reinforcement phase via using HDPE powder, and to compare the obtained results with the sample prepared, using the normal granule of HDPE, under the influence of high energy electron beam radiation in different doses.

2. Experimental procedure

2.1. Materials

The HDPE supplied by Bandar Imam Petrochemical Company HDPE-HI0500 with a melt flow index of 4.0–6.0 g/10 min and a density of 0.96 g/cm^3 was used in this investigation. Synthetic nano-structured HAP was obtained via hydrolysis method (Shih et al., 2004). The procedure of the method is that dicalcium phosphate dehydrate ($CaHPO_4 \cdot 2H_2O$, DCPD) (Merck, Germany) and $CaCO_3$ (Merck, Germany) mixed in 500 ml of 2.5 M NaOH at the Ca/P ratio of 1.67, at 75 °C in a high speed agitator for 1 h. After hydrolyzes, it was cooled in ice water. The precipitates were filtered and rinsed five times in deionized water. Then the powder was dried at 60 °C for 12 h. Finally, the HAP powder was sintered at 400 °C for 4 h.

2.2. Samples preparation

Nano-composites samples were prepared via two methods. In the first procedure, granules of HDPE with the different weight percentages (w%) of 10%, 15% and 25% HAP were directly subjected to melt, by carrying out mixing process in an internal mixer, Brabender 350E, made in Germany, at 180 °C at 60 rpm for 8 min. In the second approach, first the HDPE was dissolved in xylene at 110 °C under continuous stirring. At this stage, the nano-structure HAP in the mentioned different w% were added and mixed under stirring for about 20 min to reach a good dispersion of HAP particles in the solution. Then ethanol was poured into the suspension for precipitating the composite in a nano-powder form. The aggregates were filtered and dried in the vacuum oven, at 40 °C for 24 h, in order to expel the remained xylene. The prepared nano-composite samples, via the both mentioned methods were compressed in a Dr. Collin warm-press instrument, Laboratory Platen Press (Type 200P), made in Germany, at 180 °C under 100 bar pressure for 8 min, for preparing sheets of 1 ± 0.1% mm thickness.

2.3. Sample irradiation

The samples were irradiated under 10 MeV electron beam in a Rhodotron type electron accelerator machine, TT200 model, in different absorbed doses from 70 to 200 kGy at a fixed dose rate.

2.4. XRD analysis

X-ray diffraction (XRD) analysis was performed by a STOE STADI-MP system, Darmstadt, Germany, using CuKα radiation (1.5456 Å wavelength), in the 2θ angle range of 20°–60°. The grain size of the prepared HAP powder was evaluated using the Scherer's equation indicated in Eq. (1).

$$t(nm) = \frac{0.89 \times \lambda(nm)}{B(rad) \times \cos\theta} \quad (1)$$

where t is the grain size, λ is the wavelength of the X-ray, B is the full width at half maximum (FWHM) of the peak in X-ray pattern, and θ is Bragg's angle.

2.5. FTIR spectroscopy

Fourier transform infrared spectroscopy (FTIR) was carried out for the samples in wave number range of 400–4000 cm^{-1} using a Bruker Tensor 27 Spectrophotometer.

2.6. Hot-set testing

A Hot-Set UT 6050HS heating oven was used to determine the thermal elongation of the samples. All measurements were done in accordance with the DIN standard (DIN 57472-602, VDE 0472, 1983). The temperature of the oven was adjusted at 200 °C with a measuring period of 15 min. The thermal elongation (ε) was calculated using formula shown in Eq. (2).

$$\varepsilon(\%) = \frac{L_1 - L_0}{L_0} \quad (2)$$

where L_0 and L_1 were the initial and final length, respectively.

2.7. Microscopic techniques

A transmission electron microscopy system (TEM) EM208S series was used to study and determine the size of the nano-particles and morphology of the composite. The surfaces of the nano-composite samples were examined using a scanning electron microscopic (SEM) system, Philips PSEM 500.

2.8. Swelling test

The swelling ratios of the nano-composite samples were measured to determine their radiation induced crosslinking. The sample were weighed, immersed in xylene at 90 °C for 12 h, removed, dried and weighed again. The swelling ratio (SW) was calculated according to Eq. (3).

$$SW(\%) = \frac{W_t - W_0}{W_0} \times 100 \quad (3)$$

where W_t and W_0 were the sample weights after and before extraction, respectively.

2.9. Gel content

The gel content of the irradiated samples was determined by extracting the sample in boiling xylene for 24 h (Afshar et al., 2003). After that the samples were dried, the remaining insoluble were weighed. The average gel content was calculated according to Eq. (4).