Effect of Hydroxyapatite Filler on Mechanical Properties of PE/HAp Composite as a Candidate for Bone Repair

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Abstract. Polymer is one material that can be used as a fixation to repair fractured or broken bones. However, polymers are soft and ductile, so modifying them by adding hydroxyapatite as a filler is necessary. Polyethylene is a high-density polymer with more potent material properties to be utilized as a matrix. The PE-HAp composites were synthesized by compacting and heating the composition percentage of Hap 25%, 35% and 45%. Based on the characterization results using XRD, FTIR, and hardness test instruments, it is concluded that the addition of HAp composition results in better composite mechanical properties. The material properties are improving, increasing the hardness value (shore A) by 63 shore A. The hardness value increases because the composite properties are more compact, and the PE matrix physically binds the HAp filler. This is reinforced by XRD and FTIR characterization results, with no new compounds formed and no new molecular vibrational patterns in the FTIR spectrum.

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INTRODUCTION

Research on developing polymer-based composites as biomaterials for prosthetics is growing along with the advancement of biomaterial technology. One of the applications of biomaterials in the medical field is bone substitution, which supports the body and protects the fracture area by maintaining its shape during healing.

Commercially, one of the materials that can be used as bone substitution is hydroxyapatite (HAp). Hydroxyapatite with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$ is an example of powdered apatite. It is the main inorganic component in bones and teeth [1], but HAp has the limitation of being brittle so that it is easily broken. HAp must be modified by adding a polymer as a matrix to qualify as a bone substitute material. The material used as a matrix must have non-toxic, osteoconductive, biocompatible, biodegradable, and non-carcinogenic properties [2]. Polyethylene (PE) is a high-density polyethylene with more potent material properties that can be utilized as a matrix to manufacture bone-replacement biomaterial composites. In addition, PE is highly resistant to chemicals and is economically priced.

The synthesis of PE-HAp composites was carried out by compacting and heating methods, hoping to improve the hardness properties of the composites. In this study, the synthesis of modified PE-HAp composites with variations in HAp composition was carried out. The results of the synthesis of PE-HAp composites were characterized using XRD instruments for phase analysis, FTIR for functional groups in composites, and analysis of mechanical properties carried out by hardness tests.

This research aims to synthesize PE-HAp composites in the form of pellets and determine the mechanical properties and physical properties of PE-HAp composites.

METHODOLOGY

Tools And Materials

The equipment used included an analytical reader, compacting machine, furnace, Zwick shore A, X-ray diffraction (XRD) (Philips type Shimadzu 610), and FT-IR (Tensor 27). The materials used were hydroxyapatite and polyethylene granules (Mr PE = 15,000 g/mol).

Synthesis of PE-HAp composites

Synthesis of PE-HAp composite by compacting and heating method. PE and HAp with a total weight of 6 grams at a percentage of HAp of 25%, 35%, and 45% (w/w) and put into a square mould with a size of 15 cm × 15 cm and a thickness of about 0.5 mm. The mould was then inserted into a hot felting device at 180°C and pressed with an initial pressure of 50 kg cm-2 for 1 minute. The sample was then subjected to additional pressures of 100 kg cm^{-2} for 1 min and 120 kg cm^{-2} for 5 min, for a total time of 7 min. Afterwards, the samples were removed from the hot felts and transferred into the cold felts.

Characterization of PE-HAp composites

The synthesized PE-HAp composites were characterized using Zwick shore A for hardness test, XRD for phase analysis, and FTIR to analyze functional groups in PE-HAp composites.

RESULTS AND DISCUSSION

Synthesis of PE-HAp Composites

Synthesis of PE-HAp Composites by Compaction and Heating Method. The synthesis of PE-HAp composites was carried out by cold compaction method, with the percentage of Hap at 25%, 35%, and 45% (w/w). The compressed composite (felts) is brittle, and of low strength, so heating is carried out at 180oC to homogenize the PE-HAp composite and increase its mechanical strength. Composites are alloys of two or more substances that combine

physically and do not undergo chemical reactions between the substances. The synthesis of PE-HAp composites was carried out by hot felting and cold felting methods.

When the composite melts, the crystallinity of PE decreases [3] and has an irregular particle arrangement, resulting in a physical reaction with HAp. HAp can fill the amorphous particles of PE and physically bond to improve its mechanical properties.

Characterization of PE-HAp Composites

Shore A Hardness Test

The mechanical properties of PE-HAp composites determine the quality of the composite application. In this study, the effect of Hydroxyapatite composition on the mechanical properties of composites was studied. The hardness test using the Zwick Shore A tool can determine mechanical properties, so the hardness value is obtained, as shown in Figure 1.

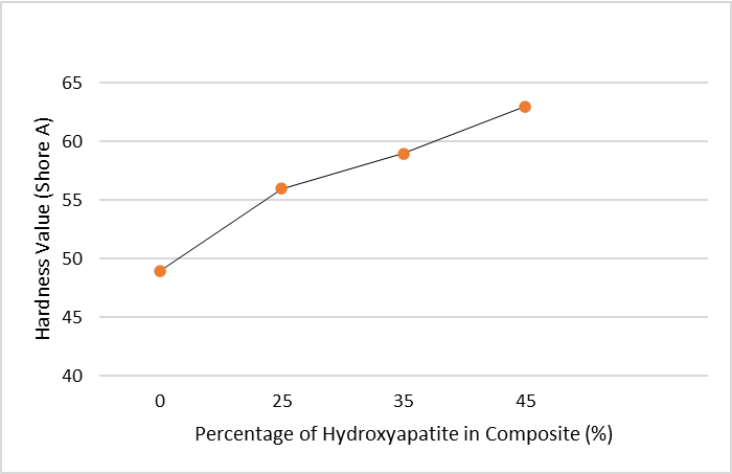


FIGURE 1. Hardness values of composites against HAp composition

The composition ratio of hydroxyapatite addition also influences the hardness value of the PE-HAp composite. The most excellent hardness value is at 45% HAp percentage of 63 shore A. In the 25% HAp percentage composition, there is a strong interaction between PE as a matrix and HAp as a filler, forming a more compact matrix

Phase Characterization with XRD

XRD characterization was used to analyze the phase and degree of crystallinity of PE-HAp composites before and after gamma radiation. Based on the diffractogram pattern in Figure 5 shows a specific phase for PE polymer at angles $2\theta = 19.4°$, 21.5°, and 23.95°. This is to the research of Kim et.al (2010) that the specific PE phase at $2\theta = 21.44°$, 23.83° and Jaggi et al. (2012) at $2\theta = 26$, 44° and 21.39°, 23.56° and 36.13° [4,5].

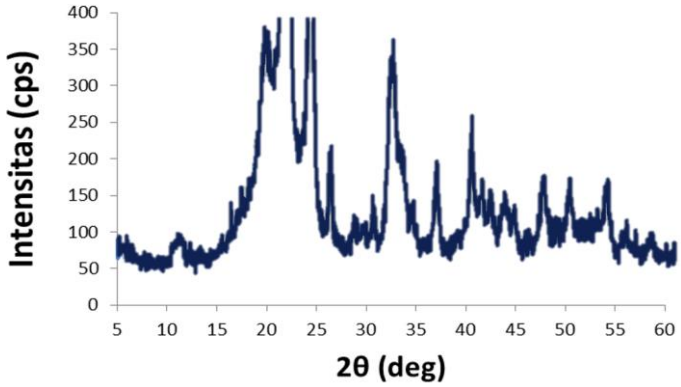


FIGURE 2. XRD spectra of PE-HAp

The specific diffractogram patterns of HAp (Figure 5) are at angles 2θ = 25.85º, 26.22º, 32.1º, 36.4º and 39.85º. This is by the Joint Committee Powder Diffraction Standards (JCPDS) database number 50-0584 for pure HAp at angles 2θ = 18.6º, 21.18º, 24.56º, 25.42º, 26.12º, 31.03º, 32.29º, and 36.44º.

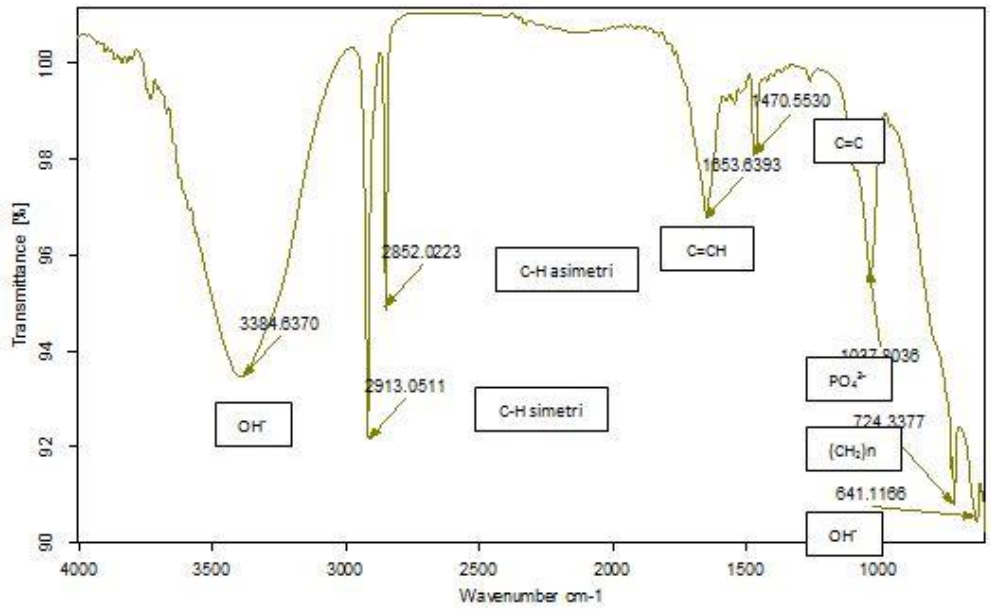
Characterization of Function Groups with FT-IR

FTIR characterization aims to determine the functional groups in PE-HAp composites formed by the felt method. PE molecules have specific vibrations at wave numbers 2915, 2850, 1467 and 720 cm^{-1} [6], while the functional groups identified in HAP include phosphate groups (PO_4^{3-}) at wave numbers 1024-1092 cm^{-1}, carbonate groups (CO_3^{2-}) at wave numbers 1420 and 1456 cm^{-1}, and hydroxyl groups (OH^-) at wave numbers around 3576 and 632 cm^{-1} [7]. The vibrational bands of the synthesized PE-HAp composite are presented in Table 3.

TABLE 3. Vibrational bands synthesized on PE-HAp composites

Wavenumber (cm^{-1})	Vibration	Molecular
2913,05	Symmetry bendC-H	PE
2852,02	Asymmetry bendC-H	PE
1653,64	Asymmetry bend CO_3^{2-}	HAp
1470,55	Groove C=C	PE
1027,80	Asymmetry bend PO_4^{3-}	HAp
724,34	Bend $(CH_2)_n$	PE
641,12	Bend OH	HAp

FTIR spectra provide information on the nature of the reaction between PE-HAp composites. The FTIR spectra suggest that the HAp filler is physically bound to the PE matrix. This can be seen in the typical peaks of each HAp and PE spectra, and no new absorption bands were found as new molecules formed [8,9].



CONCLUSION

The synthesis of PE-HAp composites was carried out by hot compressing and heating methods. The percentage ratio of HAp is 25%, 35%, and 45% (w/w) to PE/HAp composite. The percentage of 45% has more complex material properties with a hardness value of 63 shore A. XRD diffractogram patterns of HAp 25.85º, 26.22º, 32.1º, 36.4º and 39.85º. The presence of HAp filler does not significantly affect the mechanical properties because the interaction of HAp filler with the PE matrix is a physical reaction.

REFERENCES

1. Kehoe S, 2008. Optimisation of Hydroxyapatite (HAp) for Orthopaedic Application via the Chemical Precipitation Technique [Thesis] School of Mechanical and Manufacturing Engineering. Dublin: Dublin City University.
2. Wang M. 2004. Bioactive Ceramic-Polymer Composites for Bone Replacement. Singapore: Nanyang Technological University. *Journal.* School of Mechanical and Production Engineering.
3. Kusumawati. 2012. Pengaruh Radiasi Gamma Terhadap Sifat HDPE untuk *Tibial Tray*. [*Skripsi*]. Yogyakarta: Sekolah Tinggi Teknologi Nuklir-BATAN.
4. Jaggi, H.S; Kumar, Y; Satapathy, B.K; Ray,A.R; dan Patnaik,A. 2012. Analytical interpretations of the structural and mechanical response of high-density polyethylene/hydroxyapatite biocomposites. Journal Materials and Design (36) 757–766
5. Kim, B.J; Byun, J.H dan Park, S.J. 2010. Effects of Graphenes/CNTs Co-reinforcement on Electrical and Mechanical Properties of HDPE Matrix Nanocomposites. Korea: Univ.Incheon 402-751. Dept. *Journal* of Chemistry, Inha. Vol.31, No.8.
6. Ilie & Setnescu. 2009. Radiation-Induced Aging Effects in Polymeric Cable Insulators at CERN. Compilation of the DSC and ATR-FTIR data on irradiated Cern Cables. European Organization For Nuclear Research Organisation Européenne Pour La Recherche Nucléaire. CERN TE –VSC.
7. Muntamah. 2011. Synthesis and characterization of hydroxyapatite from blood clam shells (Anadara granosa, sp) [Thesis]. Bogor: Graduate School, Bogor Agricultural University.