Indian Journal of Engineering

ANALYSIS

ISSN 2319 – 7757 EISSN 2319 –7765

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Publication History
Received: 29 April 2016
Accepted: 26 May 2016
Published: 1 July 2016

Citation
Mehar AK, Mahapatra SS, Patel SK. Fabrication, Processing and Assessment of Mechanical behaviour of Hydroxyapatite (HAp) reinforced Polycarbonate (PC) Composite for Bio-medical Applications. *Indian Journal of Engineering*, 2016, 13(33), 386-393

Fabrication, Processing and Assessment of Mechanical behaviour of Hydroxyapatite (HAp) reinforced Polycarbonate (PC) Composite for Bio-medical Applications

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Abstract

A composite can be defined as a material having two or more chemically distinct phases, which at the microscopic scale are separated by a distinct interface. A composite have light weight, high strength to weight ratio and stiffness properties and replaces the conventional materials. Composites are finding applications in many fields ranging from construction to automotive industry and today widely in biomedical field as well. Hydroxyapatite (HAp) is a suitable ceramic material for tissue repair and replacement. In this study, ceramic composites comprising of synthetic HAp reinforced with Polycarbonate (PC) thermoplastic polymer are fabricated and tested to assess mechanical behaviour of ceramic composite. The materials may serve the purpose in biomedical engineering in respect to repair and replacement of fractured bone with artificial bone materials. Many implant materials has been made in the last three decades of metals, alloys, ceramics and polymers etc. Most metals and ceramics are much stiffer than bone tissue, which can result in mechanical mismatch (i.e. "stress shielding") between the implant and the adjacent bone tissue. Because metals are too stiff in addition to their other biocompatibility problems, ceramics are too brittle and polymers are too flexible and weak to meet the mechanical strength while polymers are popular due to their low density, good mechanical strength, and easy formability however, low stiffness, high wear rate and low hardness limit their use in various demanding applications. To overcome these difficulties polymeric bone implants may be used. In this present work, HAp has been synthesized by wet chemical precipitation route. HAp particulates incorporated into polymer matrix through a series of processing stages involving melt compounding, granulating and micro-injection molding. The samples were characterized by X-ray diffraction (XRD), Fourier transform infrared (FT-IR) and Scanning electron microscopy (SEM). The mechanical properties such as tensile, compression, flexural, impact and hardness have been assessed for the composite varying HAp volume percentage in PC polymer. The aim is to produce a material that has similar mechanical properties to that of bone in order to achieve mechanical compatibility in the human body.

Keywords: Artificial bone; Biocompatibility; Hydroxyapatite (HAp); Micro-injection molding; Polycarbonate (PC); Stress shielding.

1. INTRODUCTION

HAp reinforced polymer composites have been developed in recent years as analogue materials for bone replacement, adhesive bone cements and degradable internal fixation devices. The purpose of making such composites is to reinforce the polymer and improve the bone bonding properties of the material. The interfacial bonding between inorganic and organic phase plays an important role in determining the ultimate mechanical properties of the composites [1]. To carry out the experimental work HAp is mixed with PC polymer with different volume percentage. The resulting composite material is injection molded to prepare standard tensile, compression, impact and flexural test specimens. Thereafter, mechanical properties of the prepared specimens are examined. Traditionally metallic materials such as stainless steel, titanium alloys and cobalt-chromium alloys have been widely used as bone implants in orthopedic applications. HAp reinforced polymer composites for different load-bearing orthopedic applications have been developed recently [2,11].

2. METHODOLOGY USED FOR HYDROXYAPATITE PREPARATION

Analytical grade Calcium Hydroxide ($Ca(OH)_2$) powder (Merck, 96%) and Orthophosphoric (H_3PO_4) acid (Merck, 85%) were weighted at molar ratio of Ca/P=1.67 to prepare HAp powder [3,7]. The chemical reaction for wet chemical process may be expressed as follows:

$$Ca(OH)_2 + H_3PO_4 \rightarrow \mathbf{Ca_{10}(PO_4)_6(OH)_2} + H_2O \quad (1)$$

Calcium Hydroxide   Ortho phosphoric acid   ***Hydroxyapatite (HAp)***

Initially, ten grams of calcium hydroxide is weighted in a weighing machine (Mettler Toledo, 0.01 g accuracy). It is mixed with water about 40 times. The solution is stirred by a magnetic stirrer (RemiEquipmentsPvt. Ltd.) at 50^0C to 60^0C for 3 to 4 hours. Then, ortho phosphoric acid is mixed with the solution at the rate of 30 drops/minute through burette and continuously stirred but heat input to the solution is stopped. Care is taken to reach at pH value of the solution at 8 to 10. If pH value of the solution drops below the threshold values, ammonia solution (Merck, 25%) may be added to increase the pH value. After 5 to 6 hours, magnetic stirrer is stopped and the mixer is kept for 12 hours at room temperature. Then, precipitations are collected using a filter paper. The HAp precipitations were dried at 80ºC in an oven and after granulating it manually, it was sintered at 850ºC for 2 to 3 hours in muffle furnace to form non-crystalline HApto crystalline HAp [8,9,10].

3. FABRICATION OF HYDROXYAPATITE/POLYCARBONATE COMPOSITE

Composites with various amounts of HAp (0, 10, 20, 30, and 40 vol. %) were fabricated by mixing through Batch Mixer (Rheomix 600, Germany), granulating, melting and micro-injection molding. Initially, the raw materials (i.e. HAp powder and Polymers) were pre-dried in an oven at 120°C for 4 hours. Dried HAp powder is mixed with PC polymer with a density of 3.15 g/cm^3 and mean particle size of 2.248 µm through Batch Mixer. The mixtures were then granulated into small granules through cuttermanually. The granules were then again pre-dried in an oven at 120°C for 4 hours and melted through Micro-Compounder (XPLORE, 15 ml, DSM, Netherlands) and molded through Micro-injection molding machine to produce standard tensile specimen (ASTM D638), compressive specimen (ASTM D695), flexural specimen (ASTM D790) and impact specimen (ASTM D256). The micro-injection molding machine parameters were injection pressure of 6 to 7 bar, mould temperature of 85°C, melt temperature of 230°C to 240°C, heating in three front zones as well as in three rear zones were maintained at a temperature of 210°C, 220°C and 230°C, rotor speed of 100 rpm, time required for loading, holding, and cooling is of 5 seconds, 30 seconds and 30 seconds respectively, mixing time is of 1 to 2 minutes and nozzle temperature is of 240°C [5,6,12].

4. RESULTS AND DISCUSSION

4.1 ASSESSMENT OF MECHANICAL PROPERTIES

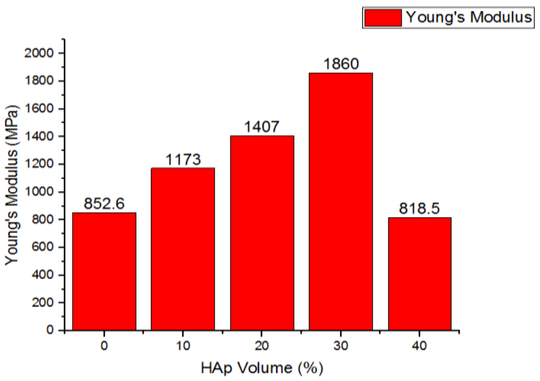


Fig. 1 Graph between Young's modulus and HAp volume of HAp/PC composite

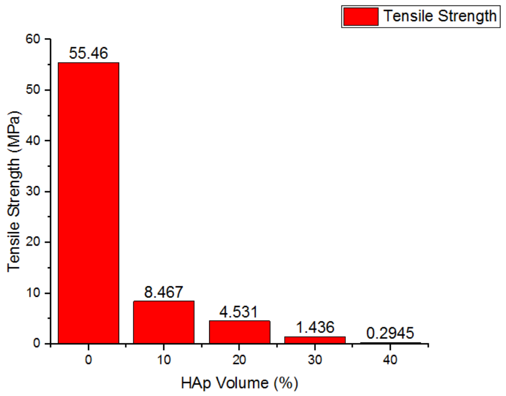


Fig. 2 Graph between tensile strength and HAp volume of HAp/PC composite

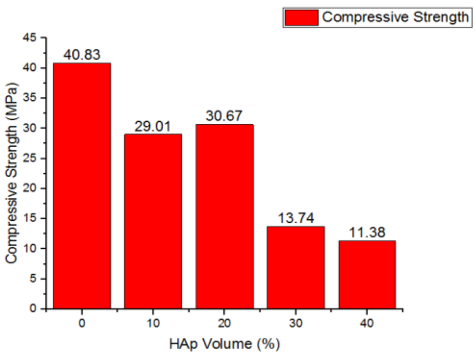


Fig. 3 Graph between compressive strength and HAp volume of HAp/PC composite

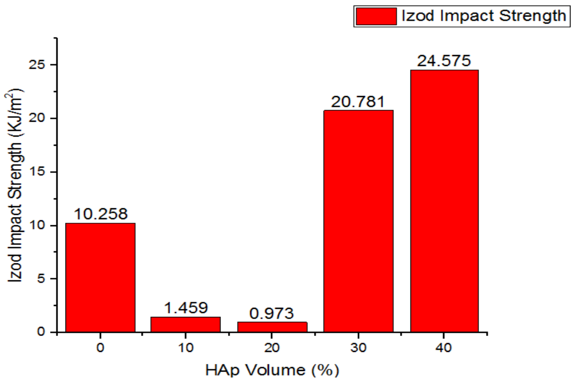


Fig. 4 Graph between izod impact strength and HAp volume of HAp/PC composite

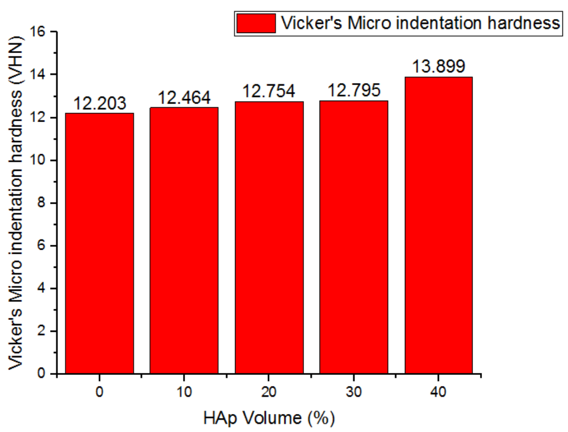


Fig. 5 Graph between vicker's hardness and

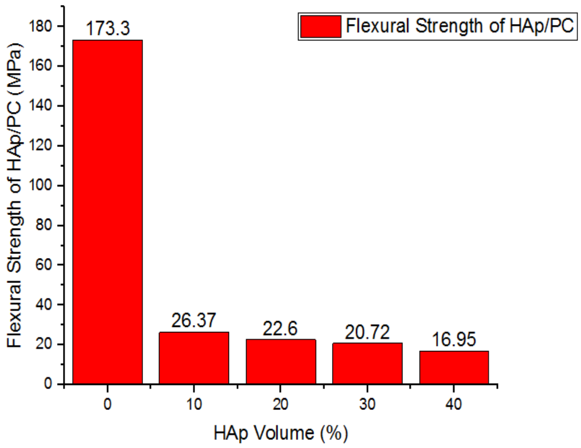


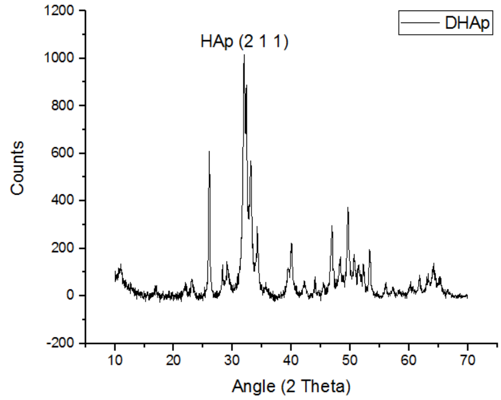
Fig. 6 Graph between flexural strength and

HAp volume of HAp/PC composite HAp volume of HAp/PC composite

It can be seen from Fig. 1 that 30 vol. %HAp/PC having maximum young's modulus. Fig. 2 shows decreasing in tensile strength of HAp/PC as increasing in volume percentage of HAp. It can be seen from Fig. 3 that initially compressive strength of HAp/PC is increasing and then it is gradually decreasing as HAp vol. % increasing. Fig. 4 shows 40 vol. % HAp/PC having maximum impact strength. From Fig. 5 it can be seen that vicker's micro-hardness of HAp/PC increasing asHAp vol. % increasing while Fig. 6shows flexural strength of HAp/PC decreasing as HAp vol. % increasing [4].

4.2. X-RAY DIFFRACTION (XRD) ANALYSIS

XRD analysis was done through X-ray diffractometer (Ultima IV, Japan).



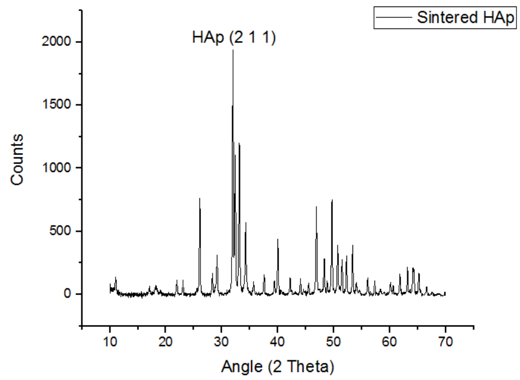
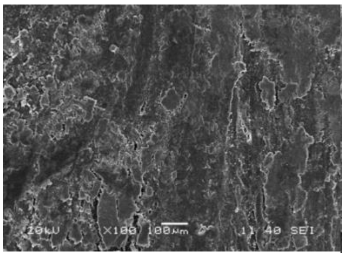


Fig. 7 XRD analysis of Dry HApFig. 8 XRD analysis of sintered HAp

XRD analysis of dry HAp is shown in Fig. 7reveals that there is a very broad peak which shows non-crystalline HAp while XRD analysis of sintered HApshown in Fig. 8 results that there is a very narrow peak as compared to dry HAp means it shows crystalline in nature of HAp.

4.3. SCANNING ELECTRON MICROSCOPE (SEM) ANALYSIS

SEM analysis was done through scanning electron microscope (JEOL JSM-6480 LV). For this initially the samples were fine through auto fine coater (JEOL JFC-1600).



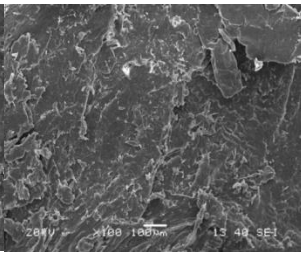
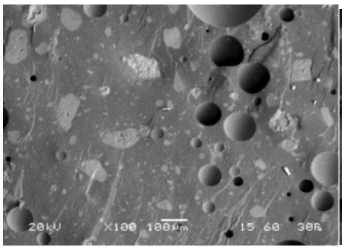


Fig. 9 SEM micrograph of 10 vol.%HAp/PC at the magnification of 100X

Fig. 10 SEM micrograph of 20 vol. % HAp/PC at the magnification of 100X



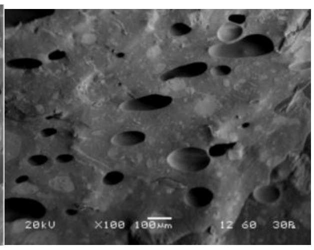


Fig. 11 SEM micrograph of 30 vol. % HAp/PC at the magnification of 100X

Fig. 12 SEM micrograph of 40 vol. % HAp/PC at the magnification of 100X

SEM micrograph of HAp/PC from Fig. 9 to Fig. 12 shows that as HAp content increases from 10 vol. % to 40 vol. %, voids also increases however there is a very good distribution of HAp particles into PC polymer matrix which increases bonding characteristics of HAp/PC composite.

4.4. FOURIER TRANSFORM INFRARED (FTIR) ANALYSIS

FTIR analysis was done through FTIR spectrometer (Perkin Elmer, Model no. Spectrum Two, Serial no. 99302, USA) in sintered HAp with KBr powder in pellet form. For this very small amount of HAp powder is first mixed with KBr powder and then fills it in pellet maker machine with pressure maintained at 10 tons through hydraulic press to make required pellets for FTIR test. An FTIR spectrum of sintered HAp is shown in Fig. 13. FTIR spectra were recorded in the transmission mode in the range 4000 to 400 cm^{-1}. The lower wavelength limit was chosen to encompass the highest known vibration frequency due to a fundamental molecular vibration. Broad peak between 3500 and 3300 cm^{-1} were noticed on IR spectra indicated that the vibration of absorbed water (OH^-) in the apatite lattice in accords with the

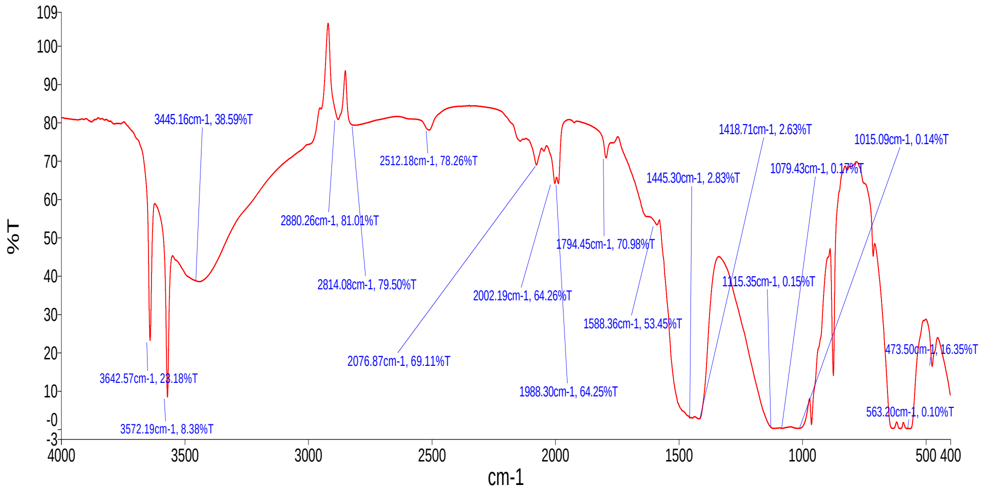


Fig. 13 FTIR of sintered HAp at 850°C

HAp chemical formula ($Ca_{10}(PO_4)_6(OH)_2$). Also there are various vibration modes representing the phosphate functional group. Those vibration modes were found on the FTIR spectra, thus confirming that the calcinedHAp powders were composed of phosphate group. The peaks of phosphate group were represented between 500 and 600 cm^{-1} and 1000 and 1100 cm^{-1}.

5. CONCLUSIONS

1. Wet chemical precipitation technique is chosen widely to synthesize Hydroxyapatite (HAp) in comparison to other techniques as relatively large amount of HAp can be produced in absence of any organic solvents at a reasonable cost.
2. XRD analysis shows that the developed ceramic powder (HAp) is completely pure in phase and having hexagonal structure.
3. SEM micrograph of HAp/PCshows that as HAp content increases from 10 vol. % to 40 vol. %, voids also increases however there is a very good distribution of HAp particles into PC polymer matrix.
4. Broad peak between 3500 and 3300 cm^{-1} were noticed on IR spectra indicated that the vibration of absorbed water (OH^-) in the apatite lattice in accords with the HAp chemical formula ($Ca_{10}(PO_4)_6(OH)_2$). Also the peaks of phosphate group were represented between 500 and 600 cm^{-1} and 1000 and 1100 cm^{-1}.

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