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This is to certify that Prof./Dr./Ms./Mrs/Mr. SEYED MOJTABA ZEBARJA

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INSTITUTE OF MACROMOLECULAR SCIENCE AND ENGINEERING
Perumbaikadu P.O.
Kottayam
Kerala-686 028
India

Convener
Prof. (Dr.) Sabu Thomas

Organized By :-

Institute of Macromolecular Science and Engineering (IMSE)
Parumbaikadu P.O, Kottayam, Kerala, India

Ayurveda-und Venen-Klinik
St. Primus Weg 68, A-9020 Klagenfurt,
Austria

Beijing University of Chemical Technology
Bei San Huan East Road 15#,
Chaoyang District, Beijing, China

Centre for Nanoscience & Nanotechnology
Mahatma Gandhi University
Priyadarshini Hills P. O., Kottayam, Kerala, India

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A Study on Mechanical Properties of PMMA/ Hydroxyapatite nanocomposite

S. M. Zebarjad, S. A. Sajjadi, T. Ebrahimi Sdrabadi, A. Yaghmaei
Department of Materials Science and Metallurgical Engineering,
Faculty of Engineering,
Ferdowsi University of Mashhad, Mashhad, Iran
E-mail: Zebarjad@um.ac.ir

This study is focused on the role of nano hydroxyapatite particles on the mechanical properties of PMMA/HA nanocomposites. In order to achieve a proper and homogeneous distribution of HA particles in the polymer matrix, mixer milling process was applied. Wear, compression and three-point bending tests were conducted. It was observed that wear rate decreased by increasing in HA content in both atmosphere and artificial saliva. The results of compression tests showed that the addition of 2.5 percent HA to PMMA promoted ultimate compressive strength, yield strength and modulus while caused to decrease elongation at break. Also it was elucidated that addition of HA more than 2.5 wt.% caused a decrease in both ultimate compressive strength and compression yield strength and an increase in elongation at break. The results of three-point bending tests on the PMMA cements containing 2.5 percent HA demonstrated the maximum bending strength value and modulus among all the HA containing formulations. However there was no direct proportionality between the results of bending tests and the HA content and the addition of HA to PMMA (up to 10 wt.%) did not change the bending properties significantly.

Keywords: PMMA, Nano hydroxyapatite, Mechanical properties.

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Department of Materials Science and Metallurgical Engineering, Faculty of Engineering, Ferdowsi University of Mashhad, Mashhad, Iran

Abstract: This study is focused on the role of nano hydroxyapatite particles on the mechanical properties of PMMA/HA nanocomposites. Compression and three-point bending tests were conducted. The results of compression tests showed that the addition of 2.5 percent HA to PMMA promoted ultimate compressive strength, yield strength and modulus while caused to decrease elongation at break. Also it was elucidated that addition of HA more than 2.5 wt.% caused a decrease in both ultimate compressive strength and compression yield strength and an increase in elongation at break. The results of three-point bending tests on the PMMA cements containing 2.5 percent HA demonstrated the maximum bending strength value and modulus among all the HA containing formulations. However there was no direct proportionality between the results of bending tests and the HA content and the addition of HA to PMMA (up to 10 wt.%) did not change the bending properties significantly.
Keywords: PMMA, Nano hydroxyapatite, Mechanical properties.

Introduction

Polymethylmethacrylate (PMMA) is widely used as bone cement to secure orthopedic implants to the skeleton [1]. Because of its limited mechanical properties and poor compatibility with bone, the clinical use of this cement is accompanied by complications. Some examples of these limitations are brittleness and shrinkage of PMMA, void production in processing [2-4], lack of adherence to the bone [5,6] and exothermic reaction of polymerization which can damage bone tissue [7]. To improve the noted deficiencies several investigators have studied bioceramic-reinforced polymer composite materials. Hydroxyapatite (HA)-reinforced polymer composites are instances of these materials that can be used as bone cement, filling bone defects, coating of joint replacement prosthesis and dental implants. Several investigators have studied HA-reinforced PMMA (PMMA/HA) as a potential bone cement. In fact addition of HA to the bone cement can improve biocompatibility and also enhance the

mechanical properties of the cement because of its both biocompatibility and osteoconductivity. Depending on HA amount added, mechanical and thermal properties of the cement can differ. Because there are few papers that study about addition of nano hydroxyapatite in PMMA, this study tries to fill this gap. In this research, mechanical properties of nano HA-reinforced PMMA cement were investigated by using three point bending, compressive and wear test.

Experimental Procedure

Cold-cure Acrylic powder (Acropars, Marlic) and Methyl Methacrylate (Acropars, Marlic) were used to produce Polymethylmethacrylate (PMMA) as the polymeric base of composite. HA powders were used as reinforcement phase. For the preparation of samples, weighed amounts of PMMA and HA powders were mixed by a mixer mill. The solid part was consisted of the mixed PMMA and HA powders and the liquid part was consisted of the PMMA monomer, the inhibitor and the catalyst. The weight ratio of the solid/liquid components was kept 5/3.5 in all samples. For the cement dough preparation, the powder and the liquid parts were manually mixed together for 30 sec at temperature of 25 ± 1 °C and the homogeneous dough obtained was kept for 2-4 min (depended on the sample) to reach the sticky state. The compositions of the samples are shown in Table 1. To observe both size and shape of nano size HA powders, a transmission electron microscope, LEO 919 AB, was used. Figure 1 shows transmission electron microscope micrograph from nano size hydroxyapatite. The powders are flake in shape and their average size is about 50 nm.

Table 1. The details of the composition of each composite sample

Notation of samples	**HA (wt.%)**	**milling time (min)**
PMMA neat	0	0
PMMA	0	10
PMMA/2.5HA	2.5	10
PMMA/5HA	5	10
PMMA/10HA	10	10

Two kind of mechanical tests have been utilized to evaluate the properties of PMMA/nano-hydroxyapatite composites. Three-point bending and compression tests were used. In three-point bending test, for each formulation 5 samples were tested with the strain rate of 5 mm/min. The dimensions of the samples were 3.3*10*64 mm and the 5 samples were also used for each formulation in compressive strength test.

The load rate was 2 mm/min and the dimensions of the samples were 4*4*8 mm. In both tests universal tensile machine Zwick model was utilized. For each run, 3 samples were tested and the average values were tabulated.

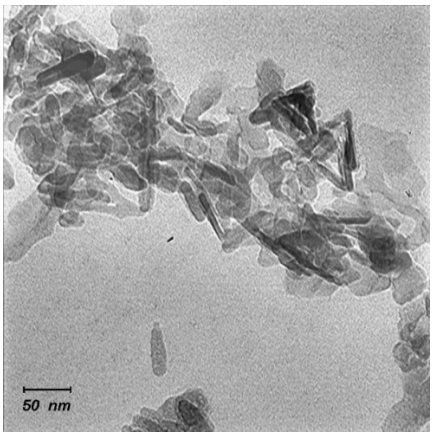

Figure 1- Transmission electron microscope micrograph of nano sized hydroxyapatite.

Results and Discussions

The average values of ultimate compressive strength (σ_C), elongation at break (ε_{fC}), compressive elastic modulus (E_C) and compression yield strength (σ_y) of the cement samples are given in Table 2. According to the results, PMMA cements containing 2.5 percent HA had the maximum value of ultimate compressive strength, elastic modulus of compression and compression yield strength among all other formulations. Also the minimum value of elongation at break was also in this sample. It was observed that HA addition more than 2.5 wt.% caused a decrease of ultimate compressive strength and compression yield strength. Although the results of elongation at break were very close to each other, and there was no direct proportionality between the results of elastic modulus of compression and the HA content. In most, but not all, composites, ultimate compressive strength decreases with increasing filler content and average particle size. This is because during the setting and cooling stage of PMMA, since the shrinkage of PMMA matrix is more than the shrinkage of HA particles, HA particles would be firmly entrapped and squeezed in the PMMA matrix. As a result of this, a circumferential tensile stress forms in the bone cement matrix that is adjacent to HA particles. This stress is a hoop stress and it simulates the case of weak bonding between HA particles and PMMA matrix. The average values of ultimate bending strength (σ_b), elongation at break (ε_{fb}) and elastic modulus of bending (E_b) are given in Table 3. PMMA cements containing 2.5 and 10 percents HA demonstrated the maximum bending strength value (67 MPa)

among all the HA containing formulations. Furthermore increasing the HA content caused the slight increased of elongation at break. Also the result showed that elastic modulus of bending in PMMA cements containing 2.5 percent HA had the maximum value between the samples. It can be inferred that the addition of HA to PMMA (up to 10 wt. %) did not change the bending properties significantly. But, it can be said that the mixer milling process had the significant effect on increasing of bending strength and elongation at break in PMMA samples (PMMA-neat and PMMA samples).

Table 2. The results of the compression test of each composite sample

Sample	σ_C (MPa)	ε_{fC} (%)	E_C (GPa)	σ_y (MPa)
PMMA neat	222	4.0	29	131
PMMA	188	4.1	26	98
PMMA/2.5HA	254	3.7	36	124
PMMA/5HA	203	4.1	24	83
PMMA/10HA	186	4.2	26	83

Table 3. The results of the three-point bending test of each composite sample

Sample	σ_b (MPa)	ε_{fb} (%)	E_b (GPa)
PMMA neat	59	3.0	1.9
PMMA	65	3.5	1.8
PMMA/2.5HA	67	3.5	2.0
PMMA/5HA	64	3.6	1.8
PMMA/10HA	67	3.7	1.9

Conclusions

There was no direct proportionality between the results of compression tests and the HA content.

The addition of HA to PMMA (up to 10 wt. %) did not change the bending properties significantly and the mixer milling process caused the significant changes of mechanical properties of PMMA.

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