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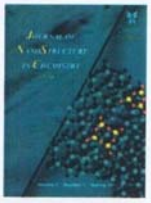

Thermal Degradation of Nano Hydroxyapatite/PMMA Composite

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ABSTRACT

This study is focused on the role of nano hydroxyapatite particles on the degradation behavior of nano HA/PMMA composites. In order to achieve a proper and homogeneous distribution of HA particles in the polymer matrix, mixer milling process was applied. Thermo gravimetric analysis was conducted using TGA analyzer. It is observed that the degradation of samples takes place in a single stage and the addition of nano HA to PMMA promotes degradation behavior of samples.

1. Introduction

Acrylic-based bone cements are widely used in dentistry and orthopaedic surgery [1]. Intense research is being carried out to improve the thermal, mechanical and biological properties of bone cements. Some studies include addition of small quantities of ingredients such as carbon [2], graphite [3], aramid [4], bone particles [5], polyethylene [6], titanium [7], ultra high molecular weight polyethylene [8], PMMA fibers [9], tricalciumphosphate (TCP) [10] or hydroxyapatite (HA) [11] in the cement matrix.

HA is a biocompatible material and since it is osteoconductive it strongly integrates with bone [12]. Therefore, addition of nano HA into bone cement formulations can enhance biocompatibility and improves the thermal degradation.

In the present study we report the degradation behavior of nano HA/PMMA composites, using thermogravimetric analysis and make an attempt to study the role of HA powder to degradation of polymer matrix.

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2. Experimental

2.1. Materials

Cold-cure Acrylic powder (Acropars, Marlic) and Methyl Methacrylate (Acropars, Marlic) were used to produce Polymethylmethacrylate (PMMA) as the polymeric base of composite. Hydroxyapatite nano powders (catalogue No. 2196, Merck) were used as reinforcement phase. Figure 1 shows transmission electron microscope micrograph from nano size hydroxyapatite. The average length of nano powders is 50 nm. The purity of hydroxyapatite nano powders that are used in this research is 98%.

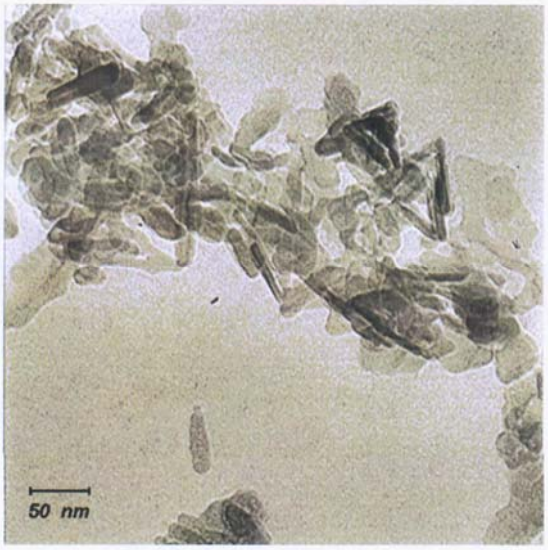

Figure 1: TEM micrograph of nano sized hydroxyapatite

2.2. Sample Preparation

For the preparation of samples, weighed amounts of PMMA and HA powders were mixed by a mixer mill (Retsich MM400, Germany) for 10 min with a vibration frequency of 20 s^{-1}. The weight ratio of balls to powders was kept constant 8 (60 gram balls and 7.5 gram powders). After mixer milling, the same ratio of the solid/liquid components were used in the preparation of compositions. 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 room temperature of 25 ± 1 °C and the homogeneous dough obtained was kept for 2-4 min (depended on the sample) to reached the sticky state. In this step the dough was molded in compression molding method. After passing about 15 min the dough was hardened and the sample was brought out the mold. The compositions of the samples are shown in Table 1.

Table 1 The details of the composition of each composite sample

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

To observe both size and shape of nano size hydroxyapatite powders, a transmission electron microscope (TEM), LEO 919 AB, was used. For sample preparation, powders were first mixer milled for 10 minutes and then dispersed in ethanol and sonicated for 1 hour. Afterward, dispersed powders were placed on grid for taking photo.

2.3. Thermogravimetric Analysis (TGA)

The thermal degradation behavior of nano HA/PMMA composites was investigated using a Shimadzu TGA-50. Samples of about 3 mg were heated from 25 to 520 °C at 10 °C min^{-1} under air atmosphere.

3. Results and Discussions

3.1. Thermal Degradation Behavior

Figure 2 shows the thermal gravimetric curves of various samples. The thermograms show that the degradation of these samples takes place in a single stage. Also, it can be seen that the thermal degradation of neat PMMA occurred earlier than that of the nano HA/PMMA composites. With the addition of HA increasing in amount, the blend started to degrade later and later.

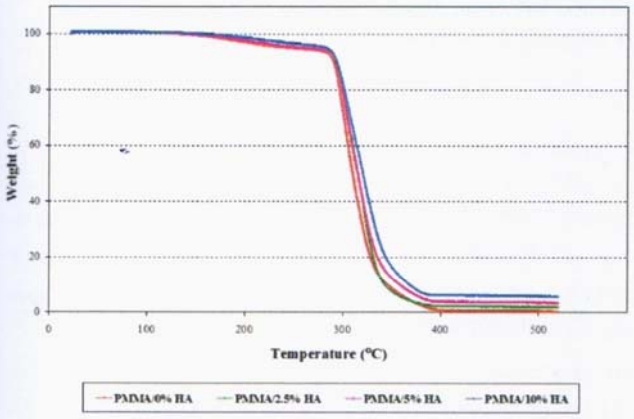

Figure 2: The TGA curves of various nano HA/PMMA samples

Table 2 lists all the calculated values from the curves including the degradation temperature in different weight percents (95, 90, 70, 50, 30 and 10) and the weight residue at 500°C.

Sample	PMMA/0% HA	PMMA/2.5%HA	PMMA/5% HA	PMMA/10% HA
$T_{-5\% wt}$ (°C)	248	282	257	283
$T_{-10\% wt}$ (°C)	290	293	292	294
$T_{-30\% wt}$ (°C)	301	305	303	308
$T_{-50\% wt}$ (°C)	310	316	314	321
$T_{-70\% wt}$ (°C)	321	325	326	334
$T_{-90\% wt}$ (°C)	346	343	357	368
Residue at 500°C (%wt)	0.5	2.1	3.7	5.9

Table 2 The calculated results of TGA curves in various samples

The results of TGA tests (in table 2) show that the addition of HA to PMMA promotes degradation behavior of samples in different temperatures. Furthermore, we can see that a low content of nano-HA particles can significantly increase residue weight of acrylic resin at high temperatures. Totally, it can be inferred that the higher thermal stability of HA causes to promote the thermal stability of PMMA at high temperatures significantly.

4. Conclusions

The results of the study of the role of nano hydroxyapatite particles on the degradation behavior of nano HA/PMMA composites are remarked as belows:

1. Mixer milling process can be a good method for mixing the Acrylic powder and nano HA to fabricate the final composite.
2. Degradation of all samples (pure PMMA and nano HA/PMMA composites) takes place in a single stage.
3. The addition of nano HA to PMMA promotes the degradation behavior of pure PMMA.
4. By increasing the amount of nano HA the degradation of samples takes place at higher temperatures.

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