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Thermal Characterization of Calcium Phosphates for Biomedical Use

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Abstract Photoacoustic spectroscopy (PAS) has been used for thermal characterization of diverse materials. The use of PAS has become an important tool since it is a nondestructive technique. Furthermore, its use to measure also optical absorption spectra has advantages over the usual transmission measurements due to important features such as the fact that scattered light does not disturb the measurements significantly and also the sample does not need to be prepared to have good quality surfaces. By using a heat transmission configuration of the photoacoustic technique, the thermal properties of biological samples have been investigated. In the present study, the photoacoustic technique is applied to obtain the thermal diffusivity of hydroxyapatite (HA) [$Ca_{10}(PO_4)_6(OH)_2$]. Complementary studies of X-ray diffraction and energy dispersion spectroscopy (EDS) were performed.

Keywords Calcium phosphate · Hydroxyapatite · Photoacoustic spectroscopy

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1 Introduction

Photoacoustic spectroscopy (PAS) is ideally suited for measuring the absorption spectrum of opaque materials, as it depends on thermal as well as optical properties of the sample, and provides different information from reflectance measurements. PAS involves measurement of heat produced as an excited species relaxes by a non-radiative path. The exciting light is chopped at a suitable frequency, and the resulting modulated heat flow is detected as pressure fluctuations by using a microphone and a lock-in amplifier. As the exciting light is scanned in wavelength, a spectrum similar to the optical absorption spectrum is obtained in which the response is proportional to both the absorption cross section and the thermal diffusivity of the sample [1]. In the present study, PAS was applied in order to obtain optical absorption spectra and the thermal diffusivity of synthetic calcium phosphates. HA80 is the sample without thermal treatment, and HA800 is the sample with thermal treatment.

2 Materials and Methods

Stoichiometric hydroxyapatite (HA) was prepared by the chemical reaction of $10Ca(OH)_2 + 6H_3PO_4 \rightarrow Ca_{10}(PO_4)_6(OH)_2 + 18H_2O$. It was dried at 80 °C to obtain the (HA80) sample and furthermore it was thermally treated to 800 °C to obtain the (HA800) sample. For all the samples, the X- ray diffraction patterns have been obtained by using a X-ray diffraction meter Kristalloflex 805, with 40 kV and 20 mA. The optical absorption spectra of these samples ranged from 300 nm to 800 nm, and energy dispersive spectroscopy (EDS) was performed.

3 Optical Absorption Spectra

Optical absorption spectra were obtained in the range of 300 nm to 800 nm by using a homemade photoacoustic (PA) spectrometer. The PA experimental set-up is showed in Fig. 1. This setup consisted of a 1000 W xenon lamp (Oriel), a variable frequency mechanical chopper, set at 17 Hz, a monochromator, and an air-filled brass cell with a condenser microphone. The PA signal from the microphone provided the input to the signal channel of the lock-in amplifier (SR-850) that is interfaced to a personal computer, displaying the wavelength-dependent signal amplitude and phase simultaneously. In order to take into account the Xe lamp emission, the spectrum signal was normalized to the signal obtained from charcoal powder.

4 Results and Discussion

Figure 2 shows the optical absorption spectra obtained by PAS of the HA80 and HA800 samples in the range from 300 nm to 800 nm. In the case of the thermally treated sample (HA800), this showed an absorption peak located around 430 nm. This is a well known size effect, called "blue shift" produced by the very fine powder of several materials [2].

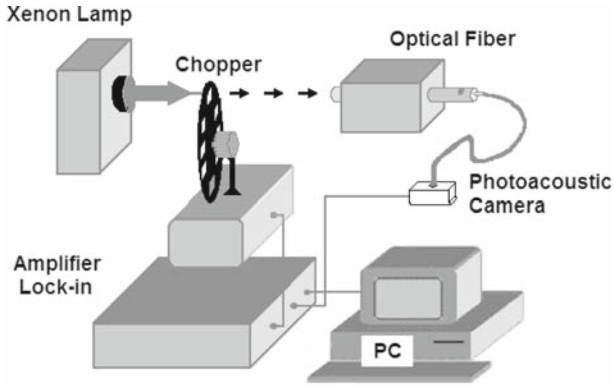

Fig. 1 Experimental setup

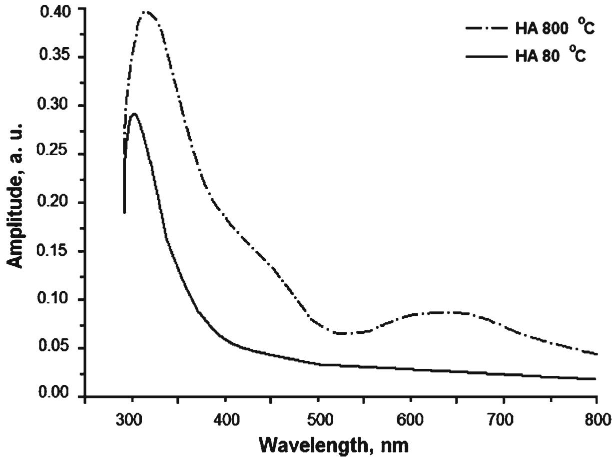

Fig. 2 Optical absorption spectra obtained by PAS

Table 1 Chemical composition by EDS

Sample	Ca		P	
	(at%)	Element (mass%)	(at%)	Element (mass%)
HA 80	71.67	76.60	28.33	23.40
HA 800	75.74	80.16	24.26	19.84

Figure 3 represents the X-ray diffraction patterns of the samples. These patterns show that the crystallinity of HA80 is increased after thermal treatment (sample HA800). The thermal treatment was applied for 2 h.

On the other hand, the EDS results, reported in Table 1, indicate that the chemical composition does not change after thermal treatment.

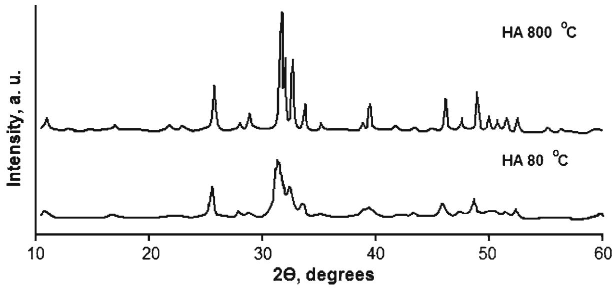

Fig. 3 X-Ray diffraction patterns

5 Conclusions

In the present study, PAS was applied to obtain optical absorption spectra of HA80 and the thermally treated (HA800) samples. The optical absorption spectra of these samples showed differences between them. In the case of the HA800 sample, a peak is located around 430 nm which corresponds to changes in the HA structure. Complementary X-ray diffraction studies confirmed that the crystallinity of the thermally treated sample was increased. On the other hand, EDS indicates that the composition was not changed with thermal treatment.

References

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