



# PHYSICAL CHEMISTRY 2004

## *Proceedings*

*of the 7<sup>th</sup> International Conference  
on Fundamental and Applied Aspects of  
Physical Chemistry*

*Volume I and II*

September 21-23, 2004  
Belgrade, Serbia and Montenegro



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ISBN 86-82457-12-x  
Title: Physical Chemistry 2004. (Proceedings)  
Editors A. Antić-Jovanović and S. Anić  
Published by: The Society of Physical Chemists of Serbia, Student-  
ski trg 12-16, P.O.Box 137, 11001 Belgrade, Serbia  
and Montenegro  
Publisher: Society of Physical Chemists of Serbia  
Printed by: "Jovan" Printing and Published Comp;  
300 Copies; Number of Pages: x + 906; Format B5;  
Printing finished in September 2004.  
Text and Layout: Aleksandar Nikolić

*300 – copy printing*

## SYNTHESIS OF BCP AND BCP/PLGA BIOMATERIALS BY ULTRASONIC SPRAY PYROLYSIS

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### Abstract

The most significant mass part of the bony tissue consists of calcium phosphate ceramics. This paper describes method of obtaining fine particles of biphasic calcium phosphate (BCP) as well as fine particles of biphasic calcium phosphate covered by polymer, poly-lactide-co-glycolide (BCP/PLGA), by ultrasonic spray pyrolysis. Methods used for characterization of obtained particles of BCP and BCP/PLGA are Infrared Spectroscopy (IR) and Scanning Electron Microscopy (SEM) techniques.

### Introduction

Materials which have structure and characteristics similar to the natural bone are called bioceramic materials [1,2]. For the reparation and replacement of the bony tissue we use calcium phosphate whose characteristics are being improved with polymer. We used spray pyrolysis [3,4,5] for obtaining fine particle of BCP which are at the later stage covered by polymer, poly-lactide-co-glycolide. All samples of obtained particles of BCP and BCP/PLGA are characterized by Infrared Spectroscopy (IR) and Scanning Electron Microscopy (SEM) techniques.

### Experimental Procedure

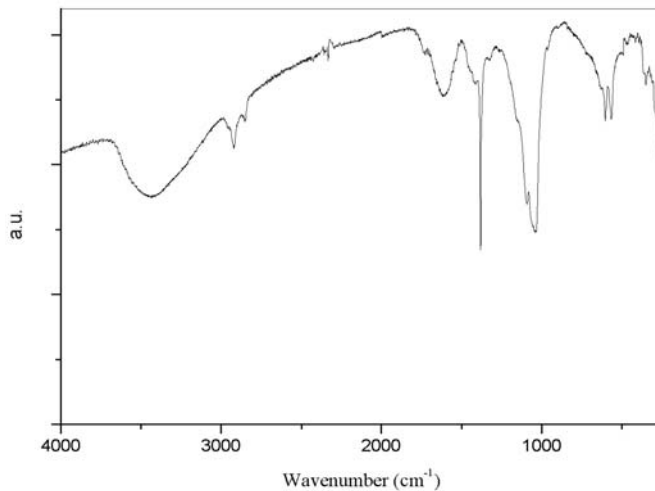
Biphasic calcium phosphate was made by precipitation from  $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$  and  $(\text{NH}_4)_3\text{PO}_4$  in the alkali environment. After 24h the suspension is filtered through Bihner's hopper with vacuum. The obtained gel ( $\approx 40$  g) is dissolved in 500 ml of water and that makes precursor solution for the spray pyrolysis. The concentration of precursor solution is 0,002 M. Spray pyrolysis is performed at  $T=400^\circ\text{C}$  with air as carrier gas. The flow of the gas is 60 l/h and the frequency of the ultrasonic atomizer is  $\nu=1,7$  MHz. Fine biphasic calcium phosphate particles obtained by spray pyrolysis are covered with the polymer by being added in to the solution of poly-lactide-co-glycolide (PLGA) in chloroform ( $\approx 0,3\%$ ) and then at the later stage methanol is added into the obtained polymer solution at the appropriate ratio. The solution after that is precipitated.

All samples are characterized by IR and SEM techniques. The IR measurements were performed on Perkin-Elmer 983G Infrared Spectrophotometer, using the KBr pellet technique, in the frequency interval of  $250\text{-}4000\text{ cm}^{-1}$ .

The morphology of obtained particles BCP and BCP/PLGA were examined by scanning electron microscope (SEM) JEOL JSM-5300. The powder samples for SEM analysis were coated with gold using the PVD process.

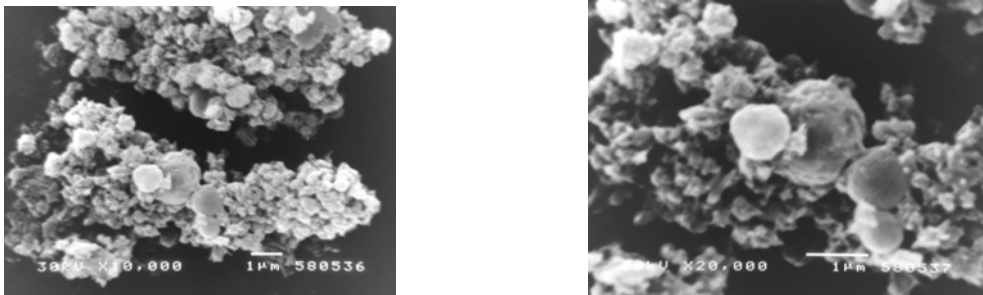
## Results and Discussion

All typical bands for BCP are shown in the IR spectrogram in Fig 1. The bands that correspond to the wave numbers 1093 and 1037, and 606 and 568  $\text{cm}^{-1}$ , belong to the  $\nu_4$  and  $\nu_3$  asymmetrically stretching vibrations for the phosphate group. The symmetrical stretching modes ( $\nu_1$  and  $\nu_2$ ) of  $\text{PO}_4^{3-}$  ion were also found at around 957 and 468  $\text{cm}^{-1}$ . The liberation and stretching modes of the  $\text{OH}^-$  were detected at around 630  $\text{cm}^{-1}$  and 1612  $\text{cm}^{-1}$ , respectively, while the band on 3400-3700  $\text{cm}^{-1}$  belongs to the  $\text{OH}^-$  group of the water molecule.



**Figure 1.** The IR spectra of the obtained biphasic calcium phosphate particles by the spray pyrolysis method

Figure 2 shows the morphology of the BCP particles obtained by spray pyrolysis.



**Figure 2.** Solid particles of biphasic calcium phosphate produced at 400 °C by spray pyrolysis

Scanning Electron Microscopy (SEM) of the obtained powder shows that powder particles, based on biphasic calcium phosphate, obtained with spray pyrolysis, have spherical shape. However, it also shows that powder contains the particles of the irregular shape. The surfaces of the particles are not smooth. They represent aggregation of the particles, which are in submicron range.



Figure 3. SEM micrographs of particles of biphasic calcium phosphate covered by polymer poly-lactide-co-glycolide

SEM micrographs of the BCP/PLGA show characteristic details of the agglomerates and particle gathering.

### Conclusion

The spray pyrolysis method is used to synthesize the particles of BCP, which are afterward covered with polymer PLGA thus obtaining the composite material BCP/PLGA. The particles have specific morphology characteristics which are determined with the parameters of the spray pyrolysis process. The particles have spherical and irregular shape with rougher surface.

### Acknowledgements

The Ministry of Science, Technologies and Development of Republic of Serbia provided financial support under grant no. 1243.

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