SONOCHEMICAL PREPARATION OF HYDROXYAPATITE / POLY (D,L LACTIDE CO GLYCOLIDE) COMPOSITE

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INTRODUCTION

In recent years there have been many attempts in the direction of developing materials and techniques to impart suitable biological and mechanical properties to synthetic composites to be used in the replacement and reconstruction of the human hard tissue. Composite materials made from hydroxyapatite and a polymer – natural or synthetic – are highy applicable for bone tissue recovery, i.e. as implants where they work by accelerating bone reconstitution induced by various injuries [1-3]. Since now, this material had been synthesised moustly by emulsion or emulsion modified methods [4-7].

In our previous work, we have reported the porcedure for synthesis of HAp the field of ultrasound and investigated the influence of the presence of various urea concentration in the reaction medium on the morphological properties of Hap synthesized [8]. In the present work, we report the application of ultrasound for the preparation of nano-sized PLGA/HAp composite particles of spheric morphology. Microscopy analysis results revealed that using the ratio 90 wt% of PLGA in relation to 10% Hap in the steps of synthesis in the field ultrasound highly uniform and spheric particles with diameter of 250-300 nm were obtained. The presence of both PLGA and HAp in these particles was confirmed by IR spectroscopy.

RESULTS and DISCUSSION

In order to confirm the presence of both organic and inorganic component, i.e. DLPLG and HAp, respectively in the sample, IR spectroscopy was undertaken. The corresponding IR spectrum of sample with 90:10 %wt DLPLG:HAp ratio is given in Fig. 1. These spectra clearly show characteristic bands of DLPLG at 2998, 2950, 2853, 1762, 1455, 1423, 1397, 1273, 1174, 1130, 1094, 751 and 710 cm^{-1}, from one, and HAp characteristic bands at 1036, 961, 632, 601, 563 and 472 cm^{-1}, from another side.

Figure 2 represents XRD pattern of DLPLG/HAp composite. Wide preak with maximum at $2\theta = 15^0$ corresponds to amorphous phase of composite, i. e. to DLPLG as polymeric part. All the other peaks corresponds to crystal part of composite and they were assigned as hydroxyapatite.

SEM micrographs, represented in Figs. 3a, 3b and 3c, revealed morphology of synthesized particles. Sample with DLPLG content (90wt%) consists of almost perfect spheres and uniform in size in the range of under 150 up to 320 nm. Figure 3b showes randomly marked sizes of some these spheres. These spheres have self assembled, regular space organisation, as can be seen in Figures 3a and 3c.

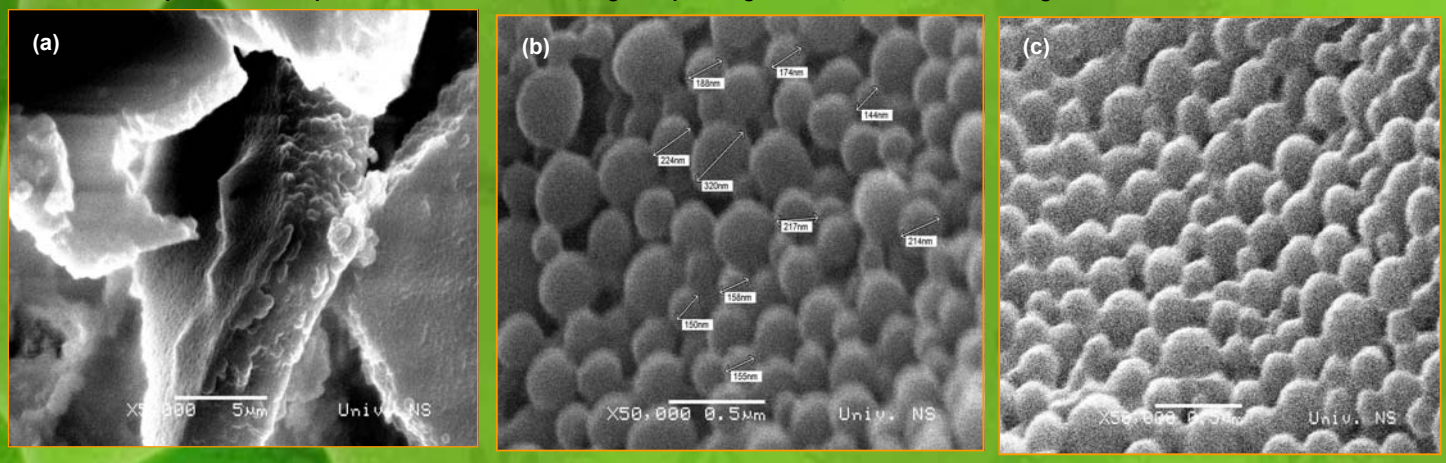

Fig 3: SEM micrographs of sample with DLPLG : HAp = 90 : 10 : (a) plate-like macrostructure built upon spherical particles, (b) sizes of some randomly chosen particles, (c) self assembled organization of spherical particles.

METHODS

In the first step, HAp was synthesized by homogeneous precipitation method in the field of ultrasound (as described in [4]). After preparation, HAp was mixed with ethanol and additionally treated in the field of ultrasound with parameters amp. = 80 %, T_{max} = 80 ^{0}C and t = 10 min. So treated apatite was than mixed and dispersed in polymer solution, DLPLG (2% DLPLG in acetone) in ultrasonic field with following parameters: T = 25 °C, t = 2 min., amplitude = 20 %. Precipitation started by adding ethanol drop wise to the reaction vessel with this dispersion cooled using ice at T = 8 °C in the field of ultrasound. Ratio DLPLG: HAp was 90:10 wt%. Sonochemical treatment was continued until all ethanol, as insolvent, was added. When precipitation was finished, obtained colloid was mixed with PVP (100 mL 2mmol/L) as surfactant solution. Reaction mixture was shortly centrifuged to spin down the pellet that was air dried afterwards.

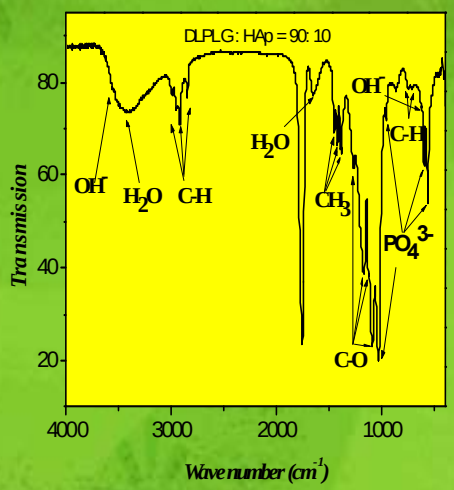

Fig 1: IR spectrum of DLPLG / HAp composite

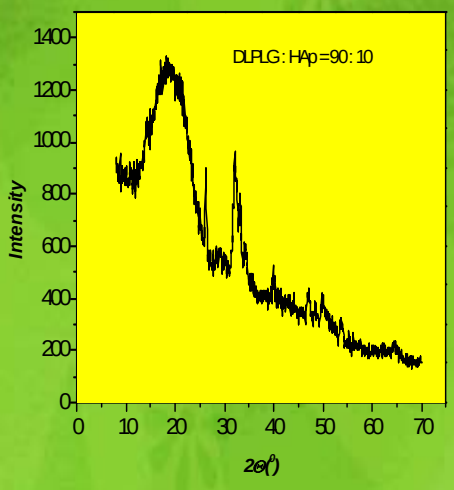

Fig 2: XRD pattern of DLPLG/HAp composite

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