



# Hydroxyapatite nanorods/poly(vinyl pyrrolidone) composite nanofibers, arrays and three-dimensional fabrics: Electrospun preparation and transformation to hydroxyapatite nanostructures

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## ABSTRACT

Electrospinning has been recognized as an efficient technique for fabricating polymer nanofibrous biomaterials. However, the study of electrospun inorganic biomaterials with well-designed three-dimensional (3-D) structures is still limited and little reported. In this study hydroxyapatite (HAp) nanorods with an average diameter of  $\sim 7$  nm and length of  $\sim 27$  nm were synthesized through a simple precipitation method and used for the fabrication of inorganic/organic [poly(vinyl pyrrolidone) (PVP)] composite nanofibers by electrospinning in ethanol solution. 3-D fabrics and aligned nanofiber arrays of the HAp nanorods/PVP composite were obtained as precursors. Thereafter, 3-D single phase HAp fabrics, tubular structures and aligned nanofiber arrays were obtained after thermal treatment of the corresponding composite precursors. Cytotoxicity experiments indicated that the HAp fabric scaffold had good biocompatibility. In vitro experiments showed that mesenchymal stem cells could attach to the HAp fabric scaffold after culture for 24 h.

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## 1. Introduction

Bone is a multiphase composite, with the main constituents of bone being collagen matrix and assembled hydroxyapatite (HAp) crystals  $[\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2]$ . HAp is the major inorganic constituent in human bone [1]. Synthetic HAp particles, films, coatings and porous skeletons with high biocompatibility are widely used in various biomedical applications [2]. Numbers of methods have been used for the synthesis of HAp, such as the wet chemical, mechano-chemical [3,4] and sol-gel methods [5,6]. HAp materials with different morphologies, including nanorods [7], plate-like nanocrystals [8], nanoparticles [9] and three-dimensional (3-D) structures [10–13] have been synthesized. They can be used as a kind of ideal biomaterial for application in tissue engineering, drug/gene delivery systems and other fields [14–16].

Electrospinning has been recognized as an efficient technique for fabricating polymer nanofibers which can be widely used in biomedical areas [17,18]. A great number of polymer and composite nanofibers have been prepared by electrospinning [19]. Recent developments in fiber electrospinning have shown that it is a promising way to produce future advanced composite systems. For instance, nanofibers are ideally suited to form a scaffold on

which multi-functional components can be hierarchically organized [20]. Electrospun fibers can also form well-designed patterns with complex ordered architectures using patterned conductive collectors [21,22]. Cell adhesion, growth and biomineralization on hybrid membranes are usually better than on pure polymer membranes [23–25]. However, investigations on electrospun inorganic biomaterials with well-designed 3-D structures are still limited and little reported. However, the production of non-woven HAp fibers by electrospinning using a solution containing polyvinyl alcohol has been reported [26,27].

In this study HAp nanorods were synthesized through a simple precipitation method and then incorporated into PVP nanofibers to form HAp nanorods/PVP composite nanofibers through electrospinning. 3-D fabrics with different shapes and aligned nanofiber arrays of the HAp nanorods/PVP composite were obtained by changing the collectors used during electrospinning. 3-D fabrics, tubular structures and aligned nanofiber arrays of single phase HAp were obtained by thermal treatment of corresponding composite precursors. Cytotoxicity experiments indicated that a HAp fabric scaffold had good biocompatibility.

## 2. Experimental section

All chemicals were analytical grade reagents and were used as received, without further purification. For the preparation of HAp

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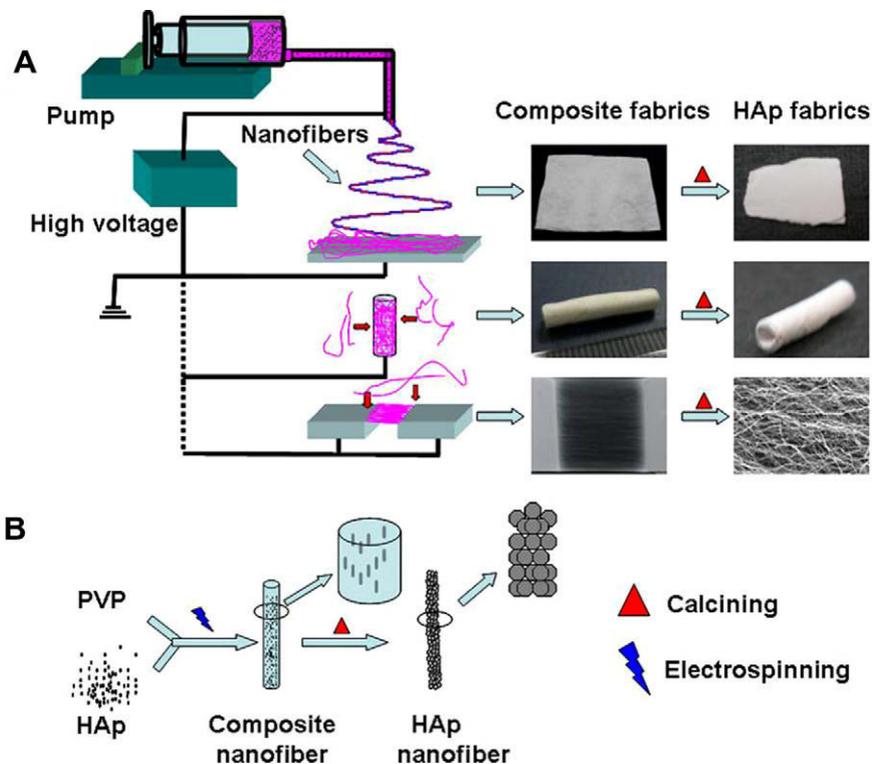


Fig. 1. Scheme of the strategy for fabrication of HAp/PVP composite nanofibers and fabrics (A) and the process of formation of HAp nanofibers (B).

nanorods 0.916 g of  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$  and 5.334 g of acrylamide were dissolved in 30 ml of deionized water under continuous stirring. An aliquot of 0.508 g of  $(\text{NH}_4)_2\text{HPO}_4$  was dissolved in 15 ml of deionized water and was then slowly added to the above solution. The pH value of the solution was kept at 10 using  $\text{NH}_3 \cdot \text{H}_2\text{O}$ . The reaction system was aged for about 1 h. The product was separated by centrifugation and washed with deionized water and absolute ethanol several times.

Thereafter, as prepared HAp nanorods and 0.8 g of PVP (K-90) were dispersed in 10 ml of absolute ethanol and used for electrospinning. As a control experiment a solution of PVP in ethanol at a concentration of 8 wt.% was used for electrospinning under the same conditions. The solution for electrospinning was fed into a plastic syringe with a needle (inner diameter  $\sim 210 \mu\text{m}$ ). A syringe pump (WZS-50F6, Smiths Medical Instruments, China) was used to

feed the solution to the needle at a rate of  $1.0 \text{ ml h}^{-1}$ . The voltage applied was 12 kV using a high voltage power supply (BGG6-358, BMEI, China). A grounded foil and stainless steel tube were used to collect nanofibers at a fixed distance (15 mm from the needle tip). Aligned nanofibers were obtained with two parallel grounded aluminum slats. To prepare HAp nanofibers and fabrics the HAp/PVP composites were used as precursors. They were heated to  $600^\circ\text{C}$  and held at this temperature for 6 h in air in a muffle furnace (Shanghai Yi-Feng Electrical Furnace Co.). The strategies for fabrication of HAp/PVP composite nanofibers and fabrics and single phase HAp nanofibers and fabrics are presented in Fig. 1.

Transmission electron microscopy (TEM) was performed with a JEOL JEM-2100F field-emission transmission electron microscope with an accelerating voltage of 200 keV. Scanning electron microscopy (SEM) was performed with a JEOL JSM-6700 field-emission

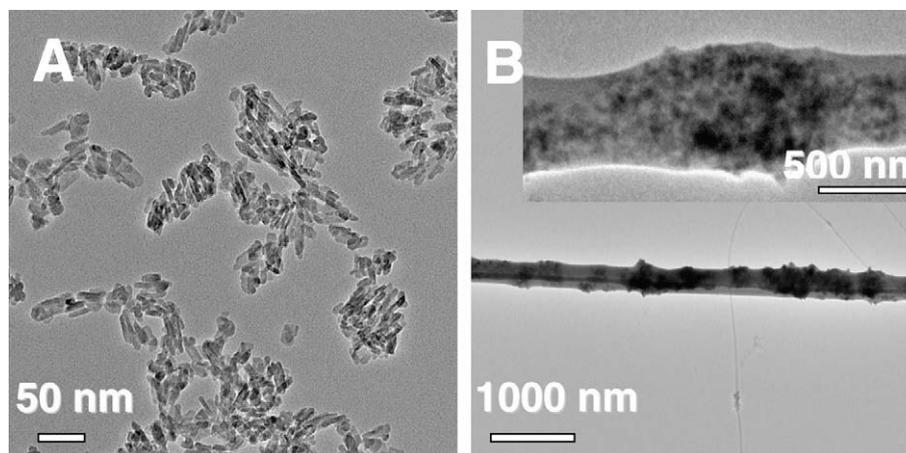


Fig. 2. Transmission electron micrographs of HAp nanorods (A) and HAp nanorods/PVP composite fibers (B).

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