

# Preparation of polycaprolactone film reinforced with CaP whisker

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## Introduction

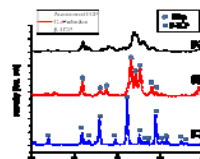
The use of hybrid organic/inorganic composites has attracted increasing interest in biomedical field especially for bone tissue engineering, on account of their improved mechanical and biological properties compared with their individual components [1]. Incorporating reinforcement into the organic polymer is one way to complement the novel properties of biopolymers as mentioned above [2]. Whiskers are generally considered as toughening additives for composite based on biopolymers [3]. Also CaP incorporated composites are expected as a strong candidate for applications such as hard tissue regenerative, in which the bioactive CaP component provides an environment favorable for cells to attach and proliferate, while the biopolymer matrix provides the required flexibility and moldability [4,5]. Therefore in this study, we fabricated CaP whisker-reinforced PCL film for potential applications in biomedical fields.

## Materials and Methods

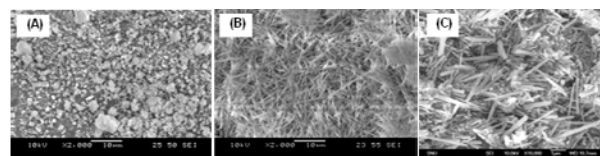
First, tricalcium phosphate was calcined at 900°C for 1 hour. Calcined powders were poured into hydrogen peroxide, and then placed in 90°C oven for 48 hours, tightly sealed, to form CaP whiskers by H<sub>2</sub>O<sub>2</sub> decomposition reaction. The products were collected by filtration. Synthesized whiskers were mixed with PCL-dichloroethane solution with various whisker contents.(5, 10, and 15 wt%) After stirring, the solutions were poured into petri dishes and dried to manufacture composite films. The crystalline phase of the samples was characterized using X-ray diffraction, the microstructures observed using scanning electron microscopy.

## Results and Discussion

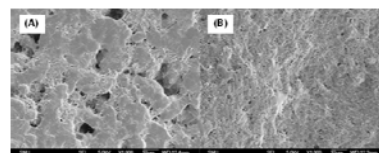
Fig 1. shows the XRD patterns of powders. The diffraction patterns of as-received TCP powder(A) and  $\beta$ -TCP(C) is shown in contrast with the synthesized whisker. That of synthesized CaP whisker(B) shows a typical peak for hydroxyapatite, thus whisker formed in hydrogen peroxide is found to be HA. No other specific peaks were observed. The SEM morphologies of the powders before and after H<sub>2</sub>O<sub>2</sub> decomposition reaction are shown in Fig 2., which show (B) whisker bundles of ~10  $\mu$ m wide and (C) individual whiskers having an average length of 5  $\mu$ m and mean width of 0.3  $\mu$ m. As shown in Fig 3., CaP whiskers are well-spread in PCL films without any agglomeration or phase separation regardless of whisker contents. In addition, mechanical behavior and *in vitro* tests are being performed.



**Fig. 1.** XRD patterns of (A) the as-received TCP powder, (B) the synthesized CaP(HA) whisker, and (C)  $\beta$ -TCP powder



**Fig. 2.** SEM micrographs of (A) the as-received TCP powder, (B) the synthesized CaP(HA) whisker at low magnification, and (C) the synthesized HA whisker at high magnification.



**Fig. 3.** SEM micrographs of (A) PCL/CaP whisker 10 wt%, and (B) 20 wt%

**Conclusions:** Polycaprolactone films reinforced with micron sized CaP whisker were fabricated with various compositions. CaP whiskers were found to be hydroxyapatite without any other phases. Individual whisker had an average length of 5  $\mu$ m and mean width of 0.3  $\mu$ m. CaP whiskers were well-dispersed in PCL matrix. So far, these results suggest that the CaP whisker-reinforced PCL film have potential for biomedical applications. More evaluations are essential to ascertain the effect of CaP whiskers, thus evaluation of mechanical properties and *in vitro* tests are in progress.

## References:

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