Effect of Porosity and Pore Size on Microstructures and Mechanical Properties of Poly-ε-Caprolactone (PCL)-Hydroxyapatite (HA) Composites

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Statement of purpose: Addition of hydroxyapatite (HA) to polycaprolactone (PCL) has been reported to improve the biological and mechanical properties of the biodegradable scaffold. However, little knowledge is known about changes in microstructural and mechanical properties that accompany variations in the porosity and pore size in PCL+HA scaffolds for bone tissue engineering applications in regard to structural compatibility. In this study, porous scaffolds composed of PCL and HA were developed and evaluated for microstructural and mechanical properties, with the aim to develop an optimized PCL+HA scaffold for bone tissue engineering.

Methods: The polycaprolactone (Mn 80000) was dissolved in tetrahydrofuran (Sigma, USA) at 40˚C. HA particles (≤40µm particle size) were suspended in the solution at a weight ratio of 1:3 to PCL. Sieved NaCl (212-355µm or 355-600µm particle size) was mixed into PCL+HA slurry at various ratio to the PCL+HA (Table 1). The viscous mixture was dried on a glass mold with thickness 5mm. After leaching out NaCl in excessive distilled water, scaffolds were dried and weighed. The porosities of the scaffolds were determined, and the morphology and component analyzed by SEM and EDX. The mechanical properties of scaffolds, both tensile strength and Young's modulus, were determined by loading tensile force on scaffolds with the dimension of 20x10x5mm using an Instron (Universal Materials Testing Machine, USA) at a crosshead speed of 2mm/min. Statistical comparisons were made by ANOVA, and $p<0.05$ was considered as statistically significant.

Table 1: Group Designation

Group	A1	A2	A3	B1	B2	B3	C
NaCl:(PCL+HA) (*w/w*)	4	1	0.25	4	1	0.25	0
NaCl particles (µm)	212-355	212-355	212-355	355-600	355-600	355-600	

Results/Discussion: The porosities of scaffolds were in direct proportion to the porogen ratio ($p<0.001$), in which the porosities of A1 and B1 were up to 74.69±2.9% and 77.48 ±2.7% respectively. Moreover the large porogen size led to a little higher porosity. The SEM images revealed that PCL+HA networks with obvious interconnections only developed in the scaffolds with highest porosity (groups A1 and B1) (Figure 1 A), where the larger porogen particles (355-600µm) produced more frequent and wider interconnection opening than did the smaller group (212-355µm). The lower porosities resulted in dispersed pores and sporadic interconnections. At the high magnification the HA particles or clusters were observed on the pore wall, which contributed to rough surfaces associated with the higher porosity scaffolds (Figure 1 B). Otherwise smoother surfaces appeared gradually with the reduction of the porogen ratio. The SEM pictures revealed that PCL tightly bound the HA particles, in the form of either HA impregnating into PCL or the exposure of HA particles on PCL surface. From the EDX spectrum, the peaks of calcium and phosphorus were prominent, confirming that HA was integrated into the composites. These data suggest that for the purpose of promoting cellular ingrowth and angiogenesis, scaffolds with sufficient porosities, interconnections and microtopography with protruded HA particles can be achieved by increasing the porogen ratio and size.

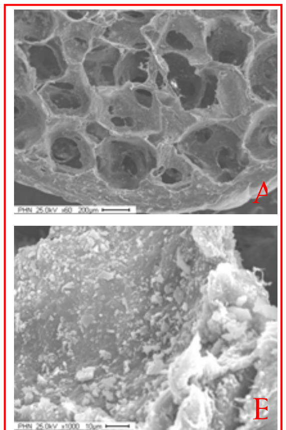
Fig.1: scaffold morphology (group B1): A, ×60; B, ×1000

The stress-strain curves generated by mechanical testing showed that the tensile strength and Young's modulus of scaffolds were influenced not only by the porogen ratio but also by the porogen sizes. Elevating the ratio of porogen weakened the mechanical properties ($p<0.001$). Given the same porogen ratio, the larger porogen size led to increased tensile strength but reduced Young's modulus, with the exception of Groups A1 and B1 ($p<0.05$). These differences were influenced by the following factors. (1) Various porogen size and ratio contributed to the distinct morphology of PCL+HA scaffolds, especially the thickness of pillars between pores that determine the pattern of HA incorporation into PCL. (2) The combined effects of mechanical properties of PCL and HA might result in that either stiffness and brittleness of single pillar increased locally where the amount and effects of HA were dominant, or higher tensile strength were achieved by the sufficient deformation locally where the continuity and plasticity of PCL were not interrupted by the HA particles. The tensile strengths of most scaffolds (group A2, B2, A3, B3 and C) were comparable to that of human cancellous bone (1.2-7.4MPa) [1, 2], while the Young's modulus of human cancellous bone (50-400MPa) were much larger than that of most scaffolds, except for Groups A3 and C. This suggests that sufficient mechanical support, as well as avoidance of stress-shielding during the healing of bone defects can be achieved with these scaffolds.

Conclusions: This system allowed us to modify the properties of PCL+HA scaffolds, including porosity, microtopography and mechanical properties, by adjusting porogen ratio and size; although combinations of properties do result in trade offs. By exploring the mechanism of PCL and HA incorporation, and evaluating the global influence of the composites, further studies should allow us to equilibrate these characteristics by modulating the volume fractions and arrangements of components in PCL+HA composites.

References:

1. Marra KG. J Biomed Mater Res. 1999; 47: 324-335.
2. Black J. 1998; Handbook of biomaterials Properties.