PREPARATION AND CHARACTERISATION OF HYROPHILIC/HYDROPHOBIC COMPOSITE SCAFFOLDS

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Introduction

Composite of hydrophilic and hydrophobic polymers are capable of exhibiting mutually complementary properties for biomedical applications that cannot be achieved using individual compound [1].

Chitosan is biocompatible; however it has low mechanical strength. Poly (ε-caprolactone) (PCL) possesses superior mechanical strength; however, the absence of cell recognition sites on the surface leads to poor cell adhesion and the hydrophobic surface may cause undesirable protein adsorption from blood, especially in *in vivo* environment [2]. It is therefore, expected that composite mixtures of these two polymers would have an enhanced mechanical properties compared with neat chitosan and improved biocompatibility over pure PCL.

The primary objective of this study was to explore the composite of hydrophobic (PCL) and hydrophilic polymers (chitosan). Emulsion lyophilisation was used to produce three-dimensional (3D) porous composite scaffold. The effect of weight ratio of two components on mechanical behaviour was investigated

Experimental

Materials

Chitosan (medium molecular weight) and PCL (M_w 65,000) were purchased from Sigma; Dichloroform (DCM) and glacial acetic acid were obtained from Ajax Chem; Sodium hydroxide (Merck) was used to prepare 0.2 M NaOH aqueous solution. Phosphate buffer saline (PBS) was prepared by dissolving one PBS tablet (Sigma) in 200 ml MilliQ water. MilliQ water was used for the preparation of solutions.

Composite scaffolds preparation

Stable emulsion mixtures of 1.5% (w/v) chitosan solution in 0.2 acetic acid and 5% (w/v) PCL solution in DCM with different weight ratios (100/0, 75/25 and 50/50) were prepared by using ultrasonic sonication (Hielschar UP400S) under 20mV for two minutes to form homogenous mixtures of two phases. The emulsion mixtures were frozen at -20 ºC overnight, followed by lyophilisation. The samples were immersed in 0.2 M NaOH to neutralise the acid residue and then stored in PBS for characterisations.

The microstructure of the samples was examined by scanning electron microscopy (SEM, Philips XL30). Prior to SEM analysis, the dry samples were gold coated.

The swelling ratio was evaluated in PBS at 37 °C by gravimetrical method.

Fourier transform infrared (FTIR) spectroscopy (Varian 660-IR) was used to determine molecular interaction between these two polymers using 4 cm^{-1} resolution, averaging for 32 scans.

The compressive properties of the samples were tested in the hydrated state at room temperature. Compression (mm) and load (N) were recorded using Wintest software at a cross head speed of 30 μm/s. The compressive modulus was obtained as the tangent slope of the stress-strain curve.

Results and Discussion

Emulsion lyophilisation was efficient to produce 3D chitosan/PCL composite scaffolds (Fig. 1a). Macro-pores and micro-pores were observed in chitosan/PCL scaffold (Fig. 1c, d). No micro-scale pore can be found in pure chitosan scaffold (Fig. 1b). The porous structure plays an important role in tissue engineering application. The macro-pores allow for homogeneous cell distribution and mass transfer properties. The micro-pores facilitate interconnection throughout scaffolds and further enhance the nutrients and oxygen diffusion.

FTIR spectra indicated the presence of both chitosan and PCL in the scaffold (Fig. 2). Pure chitosan scaffold showed a peak at 1560 cm^{-1}, which corresponds to either the

N-H band for primary amines or amide II , and another small peak was detected at 1659 cm^{-1}, corresponding to the C=O stretch for amide I.

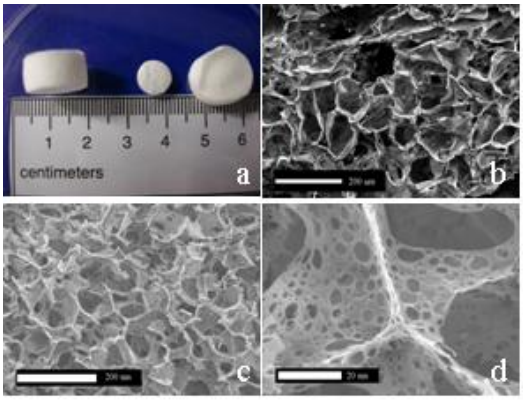

Fig. 1 a: image of chitosan/PCL composite scaffold; SEM images of b: pure chitosan (scale bar 200 μm); c and d: chitosan/PCL composites (scale bar 200 μm and 20 μm, respectively)

The characteristic peak of PCL located at 1724 cm^{-1}, corresponding to carbonyl group. These three peaks were observed in FTIR spectrum of chitosan/PCL composite scaffold. A peak shift (1724 to 1720 cm^{-1}) occurred, which corroborates the molecular interactions between two components.

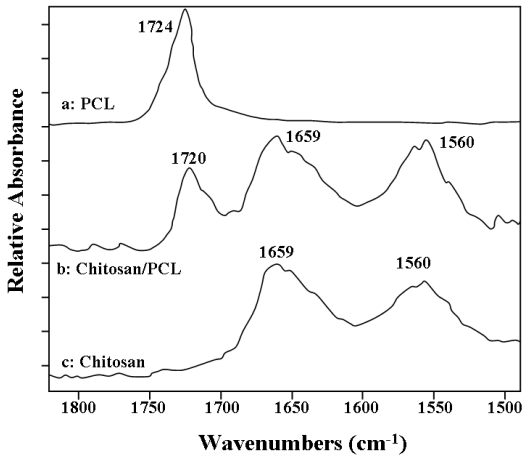

Fig. 2 FTIR spectra of a: pure PCL; b: chitosan/PCL scaffold and c: pure chitosan

The swelling property of chitosan/PCL composites was decreased due to its hydrophobicity. The swelling ratio was declined from 17.2 to 11.6 with the increasing PCL weight ratio (0 to 50 wt %) (Table 1).

Table 1. Swelling ratio and compressive modulus of chitosan/PCL composite scaffolds with different weight ratios

Chitosan/PCL weight ratio	Swelling ratio	Compressive modulus (kPa)
100/0	17.2 ± 1.4	8.2 ± 0.6
75/25	14.5 ± 0.9	19.5 ± 1.7
50/50	11.6 ± 1.2	29.6 ± 1.2

Chitosan/PCL weight ratio had an impact on mechanical behaviour of composite scaffold. The compressive modulus in was increased from 8.2 to 29.6 kPa when the weight ratio of PCL in the sample increased from 0 to 50 wt% (Table 1). However, Increasing PCL weight ratio had an adverse effect on linear stress-strain range (~50% and 30% strain rate for 75/25 and 50/50 composites, respectively), compared with over 75% for pure chitosan scaffold (Fig. 3).

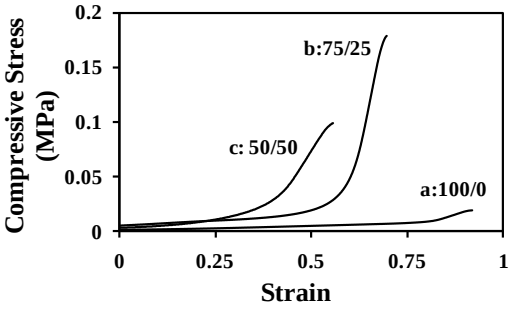

Fig. 3 Stress-strain curves of chitosan/PCL composite scaffolds with different weight ratios a: 100/0, b: 75/25 and c: 50/50

Conclusion

Emulsion lyophilisation was efficient to produce 3D porous chitosan/PCL composite scaffolds. The swelling ratio and mechanical behaviours could be controlled by altering the weight of ratio two components. The composite scaffold has a potential application for regeneration of soft tissues.

Reference

1. Sarasam A and Madihally SV. Characterization of chitosan-polycaprolactone blends for tissue engineering applications. *Biomaterials*, **26** (2005) 5500-5508.
2. Chen C, Chueh J, Tseng H, Huang H, Lee S. Preparation and characterization of biodegradable PLA polymeric blends. *Biomaterials*, **24** (2003)1163-1173.