

Poly(lactic acid)와 dialkyl phthalate 혼합물에서의 열유도 상분리

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Thermally induced phase separation in poly(lactic acid)/dialkyl phthalate system

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1. Introduction

Poly(lactic acid) is known as a biodegradable, biocompatible and bioresorbable material. It is widely used for the tissue engineering and drug delivery system as porous scaffolds. The scaffold should be fulfilled some requirements : (i) biocompatibility, (ii) biodegradability, (iii) high porosity, (iv) interconnected pore network and (v) good mechanical strength and flexibility[1]. Thermally induced phase separation (TIPS) in a polymer solution is a simple method for fabrication of scaffolds[2, 3]. One of the advantages of this technique is that various porous structures can be easily obtained by adjusting various thermodynamic and kinetic parameters[4, 5]. Nevertheless the difficulty in the morphology control limited its application. The TIPS technique has been used commercially to produce microporous membranes for filtration, but the pore size of the resultant membrane is too small to be applied for cell seeding.

In this study we are concerned with TIPS in poly(lactic acid) solutions. Both of poly(l-lactic acid) (PLLA) and poly(dl-lactic acid) (PDLLA) with different molecular weight were used to investigate the

effects of the crystallization of polymer in the same solvent system. A series of dialkyl phthalates was adopted as a solvent. The interaction between polymer and solvent was systematically controlled by changing the number of carbon atoms in the alkyl chain of the phthalates. The experimental phase diagrams were constructed by optical microscopy with a hotstage and by differential scanning calorimetry. And the theta temperatures estimated from the extrapolation of the critical temperatures with different cooling rate. The morphology formed by TIPS process was observed by scanning electron microscopy.

2. Experimental

Semi-crystalline PLLA and amorphous PDLLA were purchased from Polysciences Inc. (Warrington, USA). In this study we used three kinds of PLLA and four kinds of PDLLA with different molecular weight. The solvents used were 1,2-dialkyl phthalates with different number of carbon atoms in the alkyl chain. The solvent power could be controlled by changing the number of carbon atoms in the alkyl chain.

The liquid-liquid phase separation temperatures were observed by an optical microscope (Leica DMLB). Thermal treatments were done in a hotstage (Mettler FP 82) and controller (Mettler FP 80 HT). In the range of the experimental temperature, the evaporation of the solvent was negligible.

The films were prepared via temperature quenching experiment and the samples were fractured in liquid nitrogen to observe the morphology of the cross section. Cross sections of the samples were examined using a Philips 535M scanning electron microscopy (SEM).

3. Results and Discussion

The experimental phase diagrams for poly(lactic acid)/dialkyl phthalate systems were constructed. Liquid-liquid phase separation temperature of PDLLA solutions and PLLA solutions was determined by optical microscopy and crystallization temperature of PLLA solutions was measured by DSC. When the molecular weight of PLLA is increased, the liquid-liquid phase separation occurs at a higher temperature and the crystallization temperature remains almost constant.

As the number of carbon atoms of alkyl chain in the phthalate was decreased, the interaction between polymer and solvent increased and the liquid-liquid phase separation temperature decreased considerably while the crystallization temperature was not significantly influenced.

The final morphology of PLLA solutions obtained by the competitive phase separations was investigated. The pore size increased when a poor solvent was used and when quench temperature was decreased, which gave a higher extent of liquid-liquid phase separation to progress further before the crystallization occurred. When the polymer concentration was low and the PLLA molecular weight was decreased, the rate of the liquid-liquid phase separation is increased because of low viscosity and easiness of diffusion, so large pores were obtained.

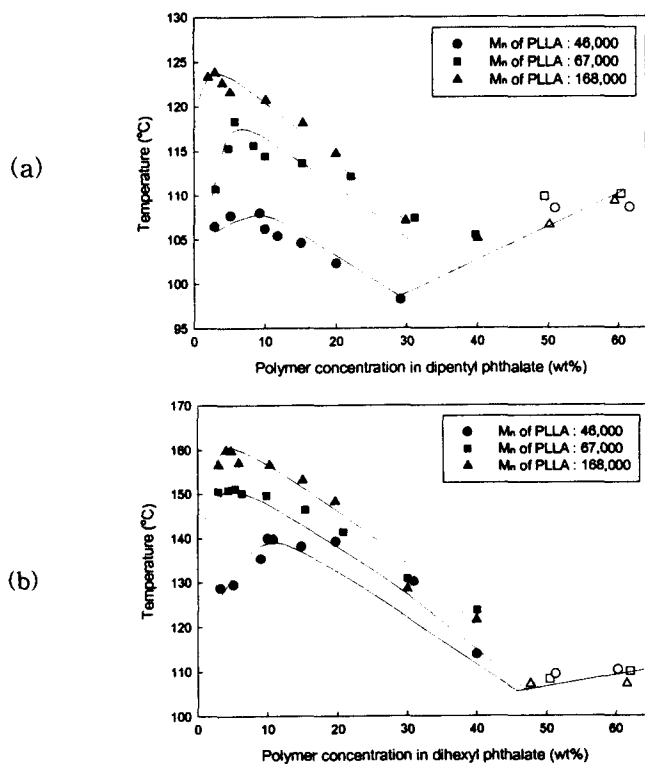


Figure 1. Liquid-liquid phase separation temperature determined by optical microscopy (filled symbols) and crystallization temperature by DSC (open symbols) for PLLA solutions in dipentyl phthalate (a) and in dihexyl phthalate (b)

4. References

- [1] A. G. Mikos, Y. Bao, L. G. Cima, D. E. Ingber, J. P. Vacanti, and R. Langer, *J Biomed Mater Res*, **27**, 183 (1993)
- [2] C. Schugens, V. Maquet, C. Grandfils, R. Jerome, and P. J. Teyssie, *J Biomed Mater Res*, **30**, 449 (1996)
- [3] Y. S. Nam, and T. G. Park, *Biomaterials*, **20**, 1783 (1999)
- [4] D. R. Lloyd, S. S. Kim, and K. E. Kinzer, *J Membrane Sci*, **64**, 1 (1991)
- [5] S. W. Song, and J. M. Torkelson, *Macromolecules*, **27**, 6389 (1994)