

ϵ -caprolactone-grafted dextran 공중합체의 합성과 전기 방사에 관한 연구

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Synthesis and electrospinning of ϵ -caprolactone-grafted dextran

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1. Introduction

Grafting process consists of the reversible protection of the hydroxyl groups of the polysaccharide backbone by silylation, followed by the ring-opening polymerization (ROP) of ϵ -caprolactone (CL) initiated by the free remaining hydroxyl groups of the partially silylated dextran in the presence of tin-based catalysts. The last step relies upon the removal of silylating groups under mild acidic conditions yielding the desired amphiphilic graft copolymers.

The purpose of this research is to synthesis of ϵ -caprolactone-grafted dextran copolymer (CGDC) and to study the electrospun structures of the copolymers by means of scanning electron microscope (SEM)

2. Experimental

2.1 Materials

Dextran (average mol wt% 10,400), 1,1,1,3,3,3-hexamethyldisilazene (HMDS), and CL were purchased from Sigma Co. and Aldrich Co. respectively. Toluene and dimethylsulfoxide (DMSO) were obtained from Showa Chemical Co. Dextran and CL were dried under CaH_2 for 48 h, distilled under reduced pressure and stored under inert atmosphere.

2.2 Grafted copolymer synthesis

6g of previously dried dextran was dissolved in 110 ml of dried DMSO in three-necked flask and nitrogen was passed. After the complete dissolution of dextran, 61.2 ml of HMDS was added and heated at 60°C for 20 h. In the course of the reaction, 36 ml of dried toluene were added. Silylated dextran was recovered by precipitation into heptane. In the second step, 2.5 g of partially silylated dextran containing 3.15 mmol of remaining hydroxyl groups was dried by three azeotropic distillations of toluene, then dissolved in 54.5 ml of dried toluene 1.85 ml of a stannous octoate ($\text{Sn}(\text{Oct})_2$) solution in toluene was added into the flask containing silylated dextran solution. 11.6 ml of dried CL was added and the reaction was carried out at 100°C for 68 h. The silylated graft copolymers recovered by precipitation in heptane, filtration and drying. Deprotection of silylated dextran hydroxyl groups were carried out by dissolution in THF and addition of 1 ml of an aqueous HCl solution (1M).

2.3 Electrospinning

CGDC was electrospun with using a 10 wt% in the THF solution.

2.4 Characterization

The chemical structure of the samples was identified by $^1\text{H-NMR}$ (FT-NMR spectrometer, Model JNM-EX 400(Japan)). The solvent for NMR experiment was deuterated chloroform(CDCl_3 , Aldrich). The glass transition temperature (T_g) was measured by DSC(TA Intrument DSC 2010). All samples were scanned in a dried nitrogen atmosphere at a heating rate of $10\text{ }^\circ\text{C}/\text{min}$. FT-IR spectra were recorded using a BIO-RAD Excalibur spectrometer. Electron micrographs of the electrospun nonwoven was observed by SEM (scanning electron microscope, GSM-5900, Jeol, Co. Japan).

3. Results and Discussion

Figure 1 shows $^1\text{H-NMR}$ spectrum of silylated dextran. The protection yield has been determined by $^1\text{H-NMR}$ from the relative intensities of the trimethylsilyl groups at 0.1 ppm and the glucosidic protons multiplet centered at 4.9 ppm using CDCl_3 as solvent.

CGDC was prepared by the ROP of CL with remaining hydroxyl groups of silylated dextran. Thus the resulted copolymers were applied for electrospinning after deprotection of silylated CGDC copolymer.

4. Conclusion

In this study, we synthesized the amphiphilic CGDC. The structure of the product synthesised was confirmed by $^1\text{H-NMR}$, FT-IR, and DSC. This polymer is further studied for electrospinning process.

Acknowledgements

This work was supported by Ministry of Health and Welfare, Republic of Korea (02-PJ10-PG8-EC01-0032) and MTT Co. Ltd.

5. References

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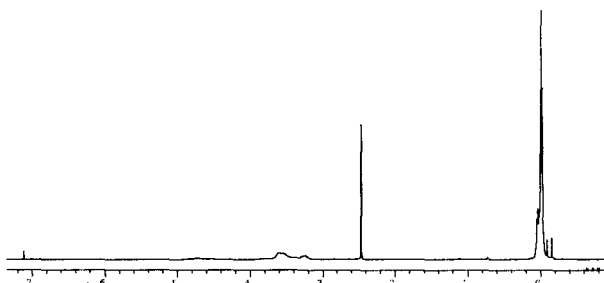


Figure 1. $^1\text{H-NMR}$ spectrum of silylated dextran