한국섬유공학회 춘계학술발표회 논문집, 제 34 권 제 1 호 **06A03**

단당류로부터의 새로운 생분해성 폴리에스터 합성

박필규, 박종래

서울대학교 재료공학부, 고차구조형유기산업재료연구센터, 환경고분자설계연구실

Novel biodegradable polyester from monosaccharides

Pil Kyu Park, Chong Rae Park

Hyperstructured Organic Materials Research Center and
Enviro-Polymers Design Laboratory,
Department of School of Material Science & Engineering,
Seoul National University

1. Introduction

Recently, polymers from sugar have attracted much attention due to their biodegradability and biocompatibility [1, 2]. Bioabsorbable polymers that have side functional attributes benefit a number of applications in biomedical materials. The pendant functional groups provide active sites for crosslinking and grafting as well as the opportunity to attach bioactive substance to modulate cellular response for crosslinking for tissue engineering application, for example [3, 4, 5, 6].

In this study, we tried to prepare a novel polyester based on aldaric acids and ethylene glycol as monomers for the synthesis of hydroxylated polyester.

2. Experimentals

2.1 Materials

Galactaric acid(mucic acid) was obtained Aldrich Co. and used without further purification. Ethylene glycol(EG) and dimethyl sulfoxide(DMSO) were first purified with molecular sieves for several days and then distilled under reduced pressure.

Antimony oxide, tin oxide, sulfonic acid, and zinc chloride used as catalyst, were analytically pure and used without further purification.

2.2 Synthesis of Bis-hydroxyl ethylene galactarate(BHEG)

Galactaric acid, excess EG and catalyst were added into a nitrogen-purged reaction vessel equipped with Dean Stock trap, and the reactant mixtrue was

heated to 140~150℃ to proceed condensation for 12 hours.

The reactant was cooled to room temperature, precipitated using non-solvent in order to remove excess EG, and dried under vacuum at 50℃.

mp: 135℃(decomp.). IR (KBr): 3500 (sharp, free O-H, stretch), 3300 (broad, O-H stretch), 2942 (C-H, stretch), 2885(-CH_2-, stretch), 1730 (C=O, stretch), 1453 (-CH_2-, bend). ^{1}H NMR (DMSO, δ): 4.32 (s, -C$\underline{H_2}$-), 4.13 (s, 2H, H-2 and H-5), 4.01 (s, 2H, H-3 and H-4), 3.82 (s, terminal -C$\underline{H_2}$OH). ELAM. ANAL. Calcd. for $C_{10}H_{14}O_{10}$ (294): C, 40.81%; H, 4.80%; O, 54.39%. Founded: C, 39.76%; H, 4.83%; O, 55.41%.

2.3 Synthesis of Poly(ethylene galactarate)

Galactaric acid, BHEG, catalyst, and DMSO as a solvent were added into a nitrogen-purged reaction vessel equipped with distilled condenser, and the reactant mixtue was heated to 140~150℃ to proceed condensation polymerization for 12~24 hours.

The reactant mixture was cooled to room temperature, precipitated using non-solvent in order to remove solvent, and dried under vacuum at 50℃.

2.4 Measurement

IR spectra were recorded with Midac Prospect FT-IR spectrometer as KBr pellets. ^{1}H NMR spectra were obtained on Joel Lambda-300 NMR spectrometer with DMSO as a solvent and tetramethylsilane(TMS) as a internal standard. Differential scanning calorimetry(DSC) analyses were performed with a Perkin-Elmer DSC7 microcalorimeter. The inherent viscosity was measured in DMSO with an Ubbelohde viscometer at 25±0.1℃.

3. Result and Discussion

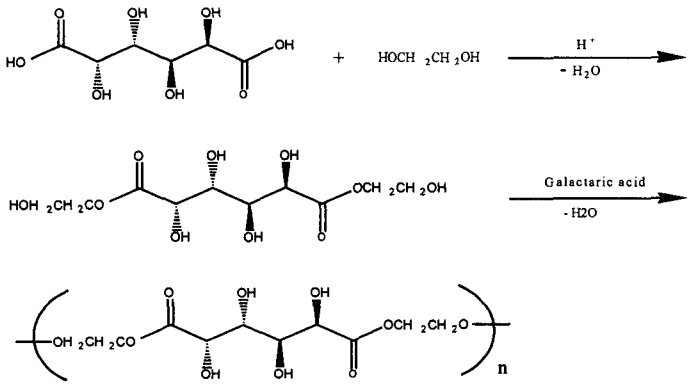
Poly(ethylene galactarate) were prepared via the BHEG synthesis as reported in scheme 1. This process involves two steps. In the first one, BHEG was prepared by thermal condensation of excess EG and galactaric acid.

BHEG was charactrized by ^{1}H NMR, FT-IR, DSC, Elemental Analysis, ect. In the second step, because BHEG has no melting temperature, polyester was prepared by thermal condensation of BHEG and galactaric acid. Molecular weight of polyester was about 10,000

Lewis acid catalysts, were better than proton acid catalyst because proton acid catalysts may cause galactric aicd to hydrolyze.

4. Reference

1) Kiely. D. E., J. Polym. Sci A: Polym Chem., 38, 594(2000)
2) Kiely, D. E., J. Am. Chem. Soc., 116, 571(1994)
3) E. Ranucci, Macromol. Rapid Commun., 21, 680(2000)
4) Z. Zhang, J. Polym. Sci A: Polym Chem., 39, 232(2001)
5) Scott. G., Polymer Degradation and Stability, 68, 1(2000)
6) M. Ajioka, Bull. Chem. Soc, Jpn., 68, 2125(1995)



Scheme 1. Synthetic routes of Poly(ethylene galactarate) via BHEG