일반강연 I -13

Synthesis and characterization of silicone-containing polyamideimide and its gas separation

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1. INTRODUCTION

Polyimides containing siloxane moiety(poly(imide siloxane), or polysiloxaneimide) have been synthesized because of their some merits over polyimide itself.

Polyimides have excellent thermal and mechanical properties but their poor solubility and processibility in their fullly imidized form give disadvantages in applications. Incorporation of siloxane units make it possible to increase solubility and processibility, and also impart impact resistance, low moisture uptake, low dielectric constant, thermo-oxidative resistance, good adhesion properties to substrate and etc..

Incorporation methods of siloxane groups into the polyimide was mainly copolymerization or terpolymerization between oligomeric dimethylsiloxane and aromatic dianhydride. A few methods of introducing siloxane units in functional groups of polyimide was reported. In our laboratory poly(amideimide siloxane) and poly(imide siloxane) were prepared and the study about their thermal kinetics was performed.

In separation membrane area, polysiloxaneimides was utilized in pervaporation and gas separation. Polyimides in gas separation show high selectivity and very low permeability, and introduction of siloxane segments increase permeability with low decrease in selectivity.

We aimed to introduce silicone segments into poly(amic acid) state and synthesize polymer partially imidized, and also show the gas separation characteristics of the synthesized polymer

2. EXPERIMENTAL

2.1. Synthesis of poly(amic acid)(PAA)

6-FDA was dissolved predetermined amounts of NMP with magnetic stirring in the three-necked round bottomed flask equipped with nitrogen inlet and outlet. After 6-FDA was dissolved completely, ODA was added, and the

reaction was performed for 10 hours, at room temperature, under nitrogen atmosphere with stirring. Poly(amicacids)(PAA) was obtained in clean solution.

2.2. Synthesis of silicone containing polyamicacid(SPAA)

SPAA was synthesized *in situ* in the PAA solution state. TEA and $SOCl_2$/THF solution was dropped into the PAA solution. Amine-terminated oligomeric dimethylsiloxane(ODMS)/THF solution was dropped into PAA solution and the reaction was performed for 10 hours. After the reaction was completed, reacted solution(SPI) was precipitated into methanol/n-hexane mixture. The precipitates were washed with distilled water several time, and then dried in vacuum oven for 24 hours at 45°C. Solid content and the ratio of THF/NMP were varied with ODMS contents.

3. RESULTS AND DISCUSSION

Reaction of ODMS was confirmed by FT-IR spectra of the SPAA which is 2967($-CH_3$), 1078 and 1015(Si-O-Si), 836(Si-CH_3)cm^{-1}. Their intensity increased with the amount of silicone reacted with PAA. From TGA with SPAA, we confirmed the degradation temperature of siloxane moiety is about 220C. NMR showed the reacted and unreacted parts of the polymer. Gas separation showed that the permeability of oxygen increased and the selectivity decreased with the increasing siloxane contents

4. REFERENCE

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