CHAPTER V

Conclusion

The new route of synthesis of poly(p-epoxystyrene) and poly(p-epoxystyrene-co-styrene) with different ratio of p-epoxystyrene and styrene has been achieved.

It started from styrene and p-chloromethylstyrene monomer which was prepared by using the method of Kondo et al. The chloromethylation of 2-phenylethyl bromide, and then dehydrobromination of p-(2-bromoethyl)benzyl chloride gave p-chloromethylstyrene with the overall yield of 31.5%. Poly (p-chloromethylstyrene) and poly(p-chloromethylstyrene-co-styrene) were synthesized using solution polymerization. Five different composition ratios of p-chloromethylstyrene (F_1) and styrene (F_2) in copolymers were prepared, i.e, 0.1:0.9 , 0.25:0.75 , 0.5:0.5 , 0.75:0.25 and 0.9:0.1. By this way the certain numbers of the chloromethyl groups in the polymer could be managed, whereas it could not be controlled by the direct chloromethylation of polystyrene. Poly(p-chloromethylstyrene) had the peak average molecular weight ($\bar{M}p$) of 180,050 and the glass transition temperature (T_g) of 116.44°C. The copolymers had the peak average molecular weights of 50,120, 99,650, 110,200, 119,855, and 177,675 and the glass transition temperature (T_g) of 110.50, 110.64, 112.24, 112.93 and 113.37, respectively. The elemental analysis indicated that the more p-chloromethylstyrene in the

feed was used, the more chloromethyl groups in the polymer were present which was correspond to the monomer reactivity ratio of styrene and p-chloromethylstyrene.

Oxidation of the chloromethyl group in the polymer led to the formation of carboxaldehyde group. The complete conversion required excess reagent and the reaction time of 6 hours . Then the epoxidation was performed to convert the carboxaldehyde group to the epoxide group using a phase transfer catalyst. This afforded the better yield on the epoxidation than the other epoxidation methods because of the less ring opening during the reaction. It was found that the epoxidation content increased as the numbers of the chloromethyl groups increased. However, it was upto around 2.6 mmole/1g.polymer. The glass transition temperature of the copolymers were 108.5, 125.5, 138.8, 142.5, and 142.6, respectively while the glass transition temperature of poly(p-epoxystyrene) was 142.2. The copolymer with the monomer composition ratio of 1:1 was the most appropriate one which required 15 hours of reaction at 15°C and 68.20% conversion was obtained.

Being sensitive to light, the photoresist should be kept in the dark with antioxidant. Actually, it should be characterized once the synthesis was completed. Due to the lack of the characterization equipment, the resists synthesized in this research had to be sent to Macromolecule Laboratory in France. It turned out that the resists were crosslinked which

might occur during the delivery or probably during the epoxidation. Therefore, the characterization of the resists could not be obtained.