

CHAPTER V

CONCLUSION

The new chelating polymers for metal ion adsorption were prepared from the reaction of aldehyde functional group in resin with chelating ligands. In the preparation, chloromethylated polystyrene-divinylbenzene (Cl-PS-DVB) was firstly oxidized to aldehydic polystyrene-divinylbenzene (CHO-PS-DVB). An estimated yield of the reaction was 57 %. These resins were characterized by Fourier transform infrared spectroscopy (FT-IR), Fourier transform raman spectroscopy (FT-Raman), thermogravimetric analysis (TGA) and elemental analysis (EA). The FT-IR and FT-Raman spectra showed an appearance of C=O peak but some C-Cl moiety remained. From the TGA result, CHO-PS-DVB was lower thermal stable than the starting matrix. However, the elemental analysis results of Cl-PS-DVB and CHO-PS-DVB were not significantly different. The characterization results revealed that most of chloromethyl units were transformed to aldehyde group.

Then, the two chelating ligand, 2-[3-(2-amino-ethylsulfanyl)-propylsulfanyl]-ethylamine (AEPE) and 2-[2-(2-amino-ethylsulfanyl)-ethylsulfanyl]-ethylamine (AEEE) were synthesized through nucleophilic substitution reaction. The yields of the synthesis of AEPE and AEEE were 79 and 60 %, respectively. The ¹H-NMR, ¹³C-NMR and FT-IR results agreed with the structure of both synthetic ligands. Subsequently, AEPE, AEEE and one commercial available ligand (triethylenetetramine (TETA)), were coupled with CHO-PS-DVB to prepare three chelating resins: AEPE-PS-DVB, AEEE-PS-DVB and TETA-PS-DVB, consequently. The appropriate conditions in preparation of each chelating polymer were shown in Table 5.1. The reaction progress could be observed by the disappearance of the C=O peak and the appearance of a new C=N absorption band in FT-IR and FT-Raman spectra.

All chelating resins were characterized by FT-IR, FT-Raman, EA and TGA. From the FT-IR and FT-Raman spectra that showed a C=N peak and the appearance of C=O peak and the percent of nitrogen content in elemental analysis results, it proofed the coupling of the chelating ligands into the polymeric support. In addition, order of thermal stability was observed from the TGA results as TETA-PS-DVB >

AEEE-PS-DVB > AEPE-PS-DVB > CHO-PS-DVB. By the way, the ninhydrin test indicated some uncoupled amine groups of the chelating ligands.

Table 5.1 Appropriate conditions in preparation of chelating polymers

Chelating polymers	Appropriate conditions		
	Amount of ligand (mmol/g)	Reaction times (hrs)	Temperature of reaction (°C)
AEPE-PS-DVB	2.65	6	50
AEEE-PS-DVB	2.65	6	50
TETA-PS-DVB	2.65	6	Room temperature

Finally, adsorption properties towards heavy metal ions such as Pb(II), Cu(II), Cd(II), Zn(II), Ni(II), Co(II) and Cr(III) were carried out in batch method. The parameter affecting the adsorption efficiency was investigated only pH of solution ranging from 1-7. The optimum pH and maximum metal sorption on the chelating resins were shown in Table 5.2. The selectivity of each chelating resin can be estimated by the observed maximum metal sorption (in $\mu\text{mol/g}$ of resin) at optimum pH. In case of AEPE-PS-DVB and AEEE-PS-DVB, the sorption behavior of both resins was similar. The pH of the solution did not affect the sorption of Co(II), Ni(II) and Cr(II) onto both resins. The order of maximum sorption capacity is Pb(II)>Zn(II)>Cu(II)>Cd(II), Co(II), Ni(II), Cr(II).

Table 5.2 Optimum pH of solution and maximum metal sorption

Chelating polymers	Optimum pH						
	Pb(II)	Cu(II)	Cd(II)	Zn(II)	Ni(II)	Co(II)	Cr(III)
AEPE-PS-DVB	5-7 (1.182)	5-7 (0.456)	6-7 (0.160)	5-6 (0.841)	- (0.136)	- (0.136)	- (0.135)
AEEE-PS-DVB	5-7 (1.091)	5-7 (0.441)	6-7 (0.169)	5-6 (0.841)	- (0.136)	- (0.136)	- (0.096)
TETA-PS-DVB	4-6 (1.062)	7 (0.157)	- (0.053)	- (0.061)	- (0.102)	- (0.068)	- (0.135)

Value in parenthesis is maximum metal sorption ($\mu\text{mol/g}$)

Suggestions for future work

Other parameters in sorption study such as contact time, adsorption isotherm, and interfering ions should be further investigated as well as the potential application for real samples.