SUMMARY

Synthesis of Poly(di-allylcalix[4]arene) resin as Cationic Heavy Metal

Phenol is one of petrochemical products which is cheap and easy to obtain. This research was done with the intention of convert phenols to valuable products which have higher economic value for example phenol transformation to calix[4]arene. Active site of calixarenes which have important role to the adsorption, extraction, and complexation are the presence of hydroxyl groups located in a line to form cyclic structure and the existence of molecule cavity forming like vase or cup shapes. With regard to calixarenes, it have proved that this compound can be used for heavy metal cationic adsorption. In the form of polymerization, the cyclic hydroxyl groups and calix[4]arenes cavity are arranged in a series forming a tunnel like structure. Based on this phenomenon, this research was carried out to synthesize calix[4]arene polymers having two allyl groups. Due to the existence of the two allyl groups, cross link reaction between calix[4]arene was taken place. This makes the molecules immobile and it can be predicted that the adsorption process for heavy metal cationic will be higher.

In the first year, the synthesis of poly(di-allylcalix[4]arene) from phenol were performed in experimental series as follows: (1) Alkylation of phenol using *p*-t-butylchloride and $AlCl_3$ to p-t-butylphenol, (2) Cyclotetramerization of p-t-butylphenol with NaOH and paraformaldehyde to p-butylcalix[4]arene, (3) Debutylation of p-butylcalix[4]arene using $AlCl_3$ and phenol to tetrahydroxycalix[4]arene, (4) Allylation reaction using allylbromide to allyloxy-dihydroxycalix[4]arene, (5) Claisen rearrangement reaction, (6) Cationic polymerization to diallylcalix[4]arenes.

The second year, the poly-diallylcalix[4]arenes were applicated as an adsorbent of heavy metal cations (Pb(II) and Cr(III)). Adsorbent used in adsorption process were three adsorbent, there are 25,26,27,26-tetrahydroxycalix[4]arene (M), poly-25-27-diallyloxy-26,28- dihidroxy[4]arene (PD) and poly-5,7-diallyl-25,26,27,28-tetrahidoxycalix[4]arene (PCD).

Adsorption was carried out towards Pb(II) and Cr(III) ions in batch system. Several variables including pH, contact time and initial concentration of metal ions were determined. The optimum pH of adsorption of metal ions Pb (II) was pH 5.0 for the three adsorbents, while the adsorption of metal ions Cr (III) at pH 5.5 for adsorbent M and PCD, and those for adsorbent PD was pH 5.0. In addition, the optimum contact time of Pb (II) ion adsorption was 180 minutes for the adsorbent M, while those for the adsorbent PD and PCD were 135 minutes. The optimum contact time of Cr (III) ion adsorption were 180 minutes for the adsorbent M and PD, while those the adsorbent PCD was 135 minutes. The adsorption kinetics of Pb(II) and Cr(III) ions using three adsorbent (M, PD and PCD) followed a pseudo 2^{nd} order kinetics model. Furthermore, The adsorption isotherm of Pb(II) ions using three adsorbent tends to follow the Langmuir isotherm, whereas the adsorption of Cr (III) using three adsorbent tends to follow the Freundlich isotherm. The adsorption capacity of Pb(II) metal ions using M,PD and PCD were 115.03, 102.19 and 125.82 µmole/g, with adsorption energy of 27.69, 28.74 and 28.12 KJ / mole, respectively. Whlie The adsorption capacity of Cr(III) metal ions using M,PD and PCD were 163.98, 178.57 and 238.59 µmole/g, with adsorption energy of 25.63, 27.65 and 25.53 KJ / mole, respectively.