

CHAPTER V

CONCLUSION AND SUGGESTION

5.1 Conclusion

The syntheses of solvent absorption-desorption core/shell beads of acrylates were carried out by seeded and one-stage suspension polymerizations. In this research, several reaction parameters affecting the structure and properties of the core/shell beads were investigated. The results can be summarized as the following.

5.1.1 One-Stage Suspension Polymerization

Appropriate condition for the preparation of poly(methyl methacrylate)/polystyrene bead could be concluded as follows:

- Amount of styrene:methyl methacrylate:ethyleneglycol dimethacrylate monomer (46.5:46.5:7.0) : 100 %wt
- Suspending agent concentration (based on the aqueous phase) : 0.7 %wt
- Initiator concentration (based on the oil phase) : 0.4 %wt
- Toluene:isooctane : 70:30
- Inhibitor concentration (based on the aqueous phase) : 0.11 %wt
- Polymerization time : 10 hours
- Polymerization temperature : 75°C

- Agitation rate : 160 rpm
- Homogenizing time : 10 minutes
- Homogenizing speed : 2000 rpm

- The suspending agent (PVP K-90) is the important parameter in the suspension polymerization, which affected the particle size. The average particle size decreased with increasing the suspending agent concentration and the polymer beads are coalesced when suspending agent concentration was less than 0.5 %wt. The appropriate of concentration was 0.7 %wt.

- Ethyleneglycol dimethacrylate was used as the crosslinking agent in this work. The crosslinking agent helps stabilize and maintains the morphology of the bead during solvent absorption and desorption because of its crosslinking network. However, increasing the crosslinking agent decreased the absorption property. Therefore, the appropriate concentration of ethyleneglycol dimethacrylate was 7 %wt.

- The concentration of benzoyl peroxide of 0.4 %wt is the suitable condition for the appropriate swelling property. A suitable concentration of the initiator should be neither too low nor too high to limit the crosslinking sites, which produced the polymer network with a maximum swelling property.

- The mixed diluent between a good solvent (toluene) and poor solvent (isooctane) is one of the important parameter to control the porous structure of the bead. The appropriate diluent composition (toluene:isooctane) was 70:30.

- The inhibitor was added to the reaction mixture to prevent the secondary nucleation of polymer particles in the aqueous phase. The appropriate inhibitor concentration was 0.11 %wt.

- The polymerization time influenced the % monomer conversion and the absorption property of the bead polymer, the longer the polymerization time, the higher the % monomer conversion. The decrease of the swelling ratio was caused by the increase of the higher crosslinking density. Therefore, the appropriate polymerization time was 10 hours.

- The polymerization temperature affected the swelling property of the bead polymer. When the polymerization temperature increased, the swelling ratio also decreased. The appropriate polymerization temperature was 70°C.

- The agitation rate affected the average particle size. The higher agitation rate, the smaller particle was obtained. When the rate of agitation was decreased to 100 rpm, the coalesced bead formed. Therefore, the appropriate agitation rate was 160 rpm.

- The average particle size of poly(methyl methacrylate)/polystyrene beads were in the range of 0.42-0.59 mm.

5.1.2 Seeded Suspension Polymerization

5.1.2.1 Seeded Suspension Polymerization of Methyl Acrylate

Appropriate condition for the preparation of poly(methyl acrylate)/polystyrene bead could be concluded as follows:

Composition	PMA seed	PMA/PS
- Amount of methyl acrylate:ethyleneglycol dimethacrylate monomer (85.0:15.0)	: 100 %wt	-
- PMA seed particle	-	: 3 g.
- Amount of styrene:ethyleneglycol dimethacrylate monomer (85.0:15.0)	-	: 100 %wt (10 g)
- Suspending agent concentration (based on the aqueous phase)	: 0.2 %wt	: 0.2 %wt
- Initiator concentration (based on the oil phase)	: 0.4 %wt	: 0.4 %wt
- Toluene:isooctane	: 80:20	: -
- Inhibitor concentration (based on the aqueous phase)	: 0.16 %wt.	: 0.16 %wt.
- Polymerization time	: 10 hours	: 10 hours

- Polymerization temperature	: 75°C	: 75°C
- Agitation rate	: 250 rpm	: 250 rpm

The cross-section of poly(methyl acrylate)/polystyrene bead has two parts. The inner bead is poly(methyl acrylate) and the outer bead is polystyrene. This result indicates the core-shell morphology.

5.1.2.2 Seeded Suspension Polymerization of 2-Ethylhexyl Acrylate

Appropriate conditions for the preparation of poly(2-ethylhexyl acrylate)/polystyrene bead could be concluded as follows:

Composition	P(2-EHA) seed	P(2-EHA)/PS
- Amount of 2-ethylhexyl acrylate:ethyleneglycol dimethacrylate monomer (93.0:7.0)	: 100 %wt	: -
- P(2-EHA) seed particle	-	: 3 g.
- Amount of styrene:ethyleneglycol dimethacrylate monomer (93.0:7.0)	: -	: 100 %wt (10 g)
- Suspending agent concentration (based on the aqueous phase)	: 0.2 %wt	: 0.2 %wt
- Initiator concentration (based on the oil phase)	: 0.4 %wt	: 0.4 %wt
- Toluene:isooctane	: 60:40	: -
- Inhibitor concentration (based on the aqueous phase)	: 0.16 %wt.	: 0.16 %wt.
- Polymerization time	: 10 hours	: 10 hours
- Polymerization temperature	: 75°C	: 75°C
- Agitation rate	: 250 rpm	: 250 rpm

The cross-section of poly(2-ethylhexyl acrylate)/polystyrene bead has two parts. The inner beads is poly(2-ethylhexyl acrylate) and the outer bead is polystyrene. The core-shell morphology is thus confirmed.

- Poly(methyl methacrylate)/polystyrene, poly(methyl acrylate)/polystyrene and poly(2-ethylhexyl acrylate)/polystyrene were able to absorb the aromatic hydrocarbon solvent more than the aliphatic hydrocarbon solvent, because the solubility parameters of aromatic solvent and the polymer beads are very close.

- The estimated solubility parameters of poly(methyl methacrylate)/polystyrene, poly(methyl acrylate)/polystyrene, and poly(2-ethylhexyl acrylate)/polystyrene are 18.8, 19.0 and 19.0 ($\text{MPa}^{1/2}$), respectively.

- As for the absorption-desorption kinetics and diffusion coefficient of toluene:isooctane in the polymer beads, the initial stage of absorption with a high slope was about 20 minutes and the swollen beads reached a stationary state in equilibrium with the surrounding solvent within 60 minutes. When swollen bead was placed on the filter paper, the solvent diffused into the filter paper immediately. The initial stage of desorption was about 15 minutes, and the swollen bead lost all retained solvent within 60 minutes after the start of the desorption process. The poly(methyl methacrylate)/polystyrene and poly(methyl acrylate)/polystyrene beads could be used to absorb and desorb toluene:isooctane solvents for many cycles (up to at least 3 cycles). But poly(2-ethylhexyl acrylate)/polystyrene could be used to absorb and desorb toluene:isooctane solvents for only one time. The extent of the diffusion coefficient was changed after many cycles of absorption-desorption because the network of bead was relatively changed. The diffusion coefficients of toluene:isooctane solvents in poly(methyl methacrylate)/polystyrene, poly(methyl acrylate)/polystyrene, and poly(2-ethylhexyl acrylate)/polystyrene were in the range of $0.1242\text{-}0.1743 \text{ mm}^2\text{min}^{-1}$, $0.0711\text{-}0.0777 \text{ mm}^2\text{min}^{-1}$ and $0.7614 \text{ mm}^2\text{min}^{-1}$, respectively.

5.2 Suggestion for Future Work

Synthesis of the core/shell morphology of acrylated beads by seeded suspension polymerization and one-stage suspension polymerization for absorption-desorption organic solvent should be further studied as follows:

a). Using SPG (Shirasu Porous Glass) to prepare the core/shell beads in order to obtain the polymer beads with a uniform composite.

b). Changing the other type of mixed diluent in the monomer phase during the polymerization.

c). Changing the addition method of styrene monomer in seeded suspension polymerization from the styrene monomer being swollen in the seed particles to the addition of the styrene monomer continuously to the reaction mixture from a microfeeder.