CHAPTER V
CONCLUSIONS

5.1 Conclusions

The formation of nanometer-scale silica particles in DP6 nonionic W/O microemulsion from the hydrolysis of tetrabuthyl orthosilicate (TBOS) catalyzed by aqueous ammonium was carried out. The chemical system was based upon the complex colloidal solution that was stabilized with surfactant (DP6), and other polar molecules (i.e., water, ammonia, butanol). This colloidal system was characterized by the interplay among molecular FTIR spectroscopy, TEM micrograph, and light scattering (DLS). The conclusions from this study can be drawn as follows:

1) The results show that the rate of TBOS hydrolysis in microemulsion solution was a pseudo-first order reaction. The silica particle growth was limited by the TBOS hydrolysis reaction. The value of particle size distribution decreased during the growing period.
2) The rate of TBOS hydrolysis was very slow, while the inter-micellar mass exchange had intermediate rate. The diffusion of TBOS molecules to the water-oil surface of microemulsion was the fastest rate.
3) Since the size of synthesized silica particles was larger than the size of initial micelle droplets, the silica particle growth strongly depends on the inter-micellar mass exchange.
4) The alkyl chain length (OR group) of alkoxide precursor influences the hydrolysis reaction. The hydrolysis rate will increase when the alkyl

chains of silica alkoxide reduce, due to the depletion of partial charge distribution in the alkoxide molecule.

5) The TBOS hydrolysis in W/O microemulsion system produced the silica particles that were ranged from 25 to 50 nm The size distribution was virtually less than 10 nm.
6) Size distribution of silica particles can be controlled by using butanol. Since butanol acts as co-surfactant, it can increase the monodispersity by with only small amount.
7) Increasing of butanol concentration leads to the increasing polydispersity of formed silica particles, due to the equilibrium of hydrolysis reaction to be disturbed.

5.2 Future Directions

The current study has succeeded in the formation of silica particles in W/O microemulsions from the hydrolysis of tetrabuthyl orthosilicate (TBOS). Even through, this study elucidates new ideas as described above, however, it is just a primary investigation for the colloidal application development. In order to understand more the fundamental phenomenon of colloidal particle formation, further studies are recommended for future works as follows:

5.2.1 Population Model of Silica Particle Formation in Microemulion System

The size distribution of silica particle is preserved constantly for the entire growing period. This size distribution represents a result of the nucleation of silica particles through coagulation of silica species among microemulsion droplets. To simulate this nucleation process, a population balance model (i.e., Smoluchowski equation) with an adjustable collision kernel, k_{ij}, for each pair

of colliding species with the sizes i and j, can be used. If k_{ij} is a function of i and j, that reflects the shape of the actual rate of coagulating silica specie pairs between microemulsion droplets. This population balance model can reproduce the profile of observed size distribution of silica particles. Therefore, this population balance simulation can be used to study the relationship between the size distribution of silica particles and the stability of polymeric silica species in microemulsion droplets.

5.2.2 Controlling the Silica Particle Formation

This study was carried out by adding butanol to control the uniformity of silica particles. The results show that size distribution was ranged in width less than 10 nm. Other parameters, including the types of surfactant and oil as well as the composition of the solution can also affect the uniformity of silica particles formed. Therefore, the influences of these variables on the formation of silica particles in W/O microemulsions are needed for further investigation. To determine the effects of alkyl group of silica alkoxide precursor, types of surfactant and oil, and special additives on the silica particle formation.

5.2.3 Other Inorganic Particles Synthesis Utilizing W/O Microemulsion System

Since thermodynamically stable and monodispersed-compartmentalized of aqueous portion of W/O microemulsions, causes to produce efficiently the narrow size distribution inorganic particles. The effective hydrocarbon tail group can protect the occurrence of particle flocculation. The high potential of alkoxide hydrolysis reaction can produce the stable particle from polymerizing the hydrolyzed species. For example, TiO_2 particles can be produced by using the hydrolysis of tetrabuthyl orthotitanate

(TBOT). Due to this precursor consisted of titanium atom which is a transition metal, it can be hydrolyzed with aqueous component. Therefore, the particular controlled microemulsion reactor is needed for the particle formation.