CHAPTER V
CONCLUSIONS

5.1 Conclusions

The formation of nanometer size titanium dioxide particles in the microemulsion system of n-heptane/$TiCl_4$/NaCl/AOT anionic surfactant reacted with NH_3 was studied. NaCl concentration was varied to obtain o/w, bicontinuous, and w/o microemulsions. Increasing the NaCl concentration increases the micellar size of o/w and bicontinuous microemulsions, which decreases the micellar size and amount of water in w/o microemulsion as shown in the results studied by dynamic light scattering and coulometer. Adding $TiCl_4$ decreased the micellar size. Higher $TiCl_4$ concentration causes smaller micellar sizes in all types of microemulsion, but did not affect the amount of water. The weight ratio of oil to brine phases showed an insignificant effect on both micellar size and amount of water in the w/o microemulsion phase, but lowering weight ratio causes larger micellar size of o/w microemulsion and smaller micellar size of bicontinuous microemulsion.

XRD, SEM, TEM and BET surface area analyzer characterized precipitates of TiO2 obtained in different microemulsions and the results were compared with commercial TiO_2 (P25). The conclusions can be drawn as follows:

1. pH condition of precipitation step did not affect the phase of titanium dioxide in the calcination step. Titanium dioxide was formed at the optimum pH of 5.0-6.0 and titanium tetrahydroxide at pH 8.0 and higher.
2. The phase of titanium dioxide was changed from brookite to anatase and to rutile with increasing the calcination temperature. The optimum temperature for the anatase was 400-500°C.
3. Titanium dioxide synthesized in different types of microemulsion has different characteristics and particle sizes. The precipitation of titanium dioxide in o/w microemulsion was similar to the precipitation

in bulk aqueous solution in which the solution also contained components of oil, surfactant, and salt. The precipitated particle had a surface area and particle size similar to the commercial titanium dioxide P25. For bicontinuous microemulsion, titanium dioxide had a polydispersion in the particle size. Titanium dioxide synthesized in w/o microemulsion was smallest in size and highest specific surface area with high porosity and crystallinity of the particle and only anatase phase was formed. Salt concentration is a factor controlling the size of particle in w/o microemulsion. Increasing NaCl concentration decreases micellar size and amount of water and thus decreases the particle size of precipitated titanium dioxide. In addition, increasing NaCl concentration also increases the amount of anatase. The results from SEM showed no particle agglomeration and TEM results showed the observed particle sizes were in the range of 40.0-13.0 nm.

5.2 Future Directions

The current study has succeeded in the formation of titanium dioxide particles in microemulsion of AOT anionic surfactant. Even though, these experiments elucidated the idea as described above, however, it is just a primary investigation for the nanomaterial development. In order to understand more fundamental phenomena of nanoparticle formation, further studies are recommended for future works as follows:

5.2.1 Controlling the Titanium Dioxide Formation

Scanning salt concentration to control the micellar size was studied in this work. Other parameters, including types of surfactant and oil or composition of the microemulsion solution also can affect the micellar size. Therefore, the influences of these variables on the particle formation in microemulsion are needed for further investigation.

5.2.2 Other Particles Synthesizes Based on Microemulsion Technique

The microemulsion technique can produce the narrow size distribution and smaller in size of titanium dioxide. The other types of particle such as metals (Pt, Ag, Au), metal oxides (SiO_2, Fe_2O_3), and metal halides including mixed oxide particles should be studied with microemulsion technique to improve the quality of the production particle.

5.2.3 Applications of Titanium Dioxide Nanoparticle Obtained from Microemulsion Technique

Titanium dioxide synthesized in this study is applicable for chemical sensor and photocatalytic study.