CHAPTER VI
CONCLUSIONS

7.1 Conclusions

In this work, the single (TiO_2, SnO_2) and/or mixed metal oxides (Nb-TiO_2, TiO_2-SnO_2) were synthesized by two microemulsion systems. The first one was the system of anionic surfactant (*n*-heptane/water/NaCl/ sodium bis (2-ethylhexyl) sulfosuccinate (AOT)). The second was nonionic surfactant system of cyclohexane/water/TX-100. The applications were divided into two sections; the liquid and gases study.

For the liquid phase study, the microemulsion system of *n*-heptane/$TiCl_4$/NaCl/AOT anionic surfactant, the size of micellar reactors and the amount of NaCl in the microemulsion control the growth of the TiO_2 by thermodynamic stabilization, where the reverse micelle acts as a constraint. This also controls the growth and structure of the titanium dioxide. Titanium dioxide synthesized in microemulsion, P25, and bulk precipitate were different in their characteristics and particle size. The preparation technique affected the TiO_2 microstructure. Titanium dioxide obtained from bulk precipitation had a broad distribution of particle size, with a larger average particle size. P25 had a smaller size (30 nm) but contains also TiO_2 in rutile form and not only anatase. Titanium dioxide synthesized in microemulsion was pure, crystalline anatase phase and possessed the smallest particle size (7-20 nm) and the highest specific area, showing therefore the best photocatalytic activity in phenol degradation. In fact, TiO_2 synthesized from microemulsion showed the fastest rate of phenol decomposition related to its smaller crystal size and higher surface area to unit volume. The change in the mean crystal size clearly determined the fraction of the exposed titanium active sites to reactant molecules and increased the chance for electrons and holes to reach the surface and react with phenol.

For gas phase study, a nanostructure with the high thermal stability of niobium doped TiO_2 (Nb-TiO_2) was synthesized using the water-in-oil (w/o) microemulsion system of n-heptane/water/sodium bis (2-ethylhexyl) sulfosuccinate

(AOT) surfactant. It was compared with undoped TiO_2. It was found that the Nb-doped TiO_2 at 3-5 mole% clearly hinders the anatase-to-rutile phase transition and inhibits the grain growth in comparison with pure TiO_2. The nanostructure of anatase could be maintained even after the powder was fired at 850°C. In a CO sensing study, it was found that the sensitivity of CO is significantly increased with an increase in the thermal stability of Nb-doped TiO_2. This shows that nanostructured Nb-doped TiO_2 is promising for environmental monitoring.

Nanostructured mixed metal oxides (Nb-doped TiO_2 (TN) and SnO_2 mixed with Nb-doped TiO_2 with 2 different ratios; 10 to 1 (TSN 101), and 1 to 1 ratio (TSN 11)) were synthesized using reverse micelles microemulsion of a nonionic surfactant (brine solution/1-hexanol/Triton X-100/cyclohexane). TN was found to have the highest surface area with only anatase phase with a mean crystal size of 14 nm. TSN 101 and TSN 11 had less surface area with two phases (solid oxide and anatase). TSN 101 showed various crystal sizes of 10 and 50 nm while TSN 11 had a very fine and uniform mean crystal size of 10 nm. The effect of film morphology and firing temperature on CO sensitivity was studied. It was found that the sensitivity of TSN 11 was clearly high (about 11) when compared to those films obtained from TN and TSN 11. The firing temperature had little effect on film crystallinity. All types of films presented good thermal stability. No change in film morphology was observed before or after CO sensing. The electrochemical impedance spectroscopy (EIS) of TSN 11 was also studied. The changes in the Nyquist plot were related to the difference in mean crystal sizes of sample.

Both nanostructured single and mixed metal oxides showed to be promising new semiconductor oxides for either liquid and gas phase applications. The right combination of the mixed metal oxides can also enhance the powder properties and can be applied for the crucial conditions in real applications.

7.2 Future Work

Since the microemulsion was proved to be a promising technique for synthesis nanostructured metal and mixed metals oxides in laboratory scale with an excellent both in chemical and electronical properties. Therefore, the up-scaled production should be studied for synthesize a large volume of nanostructured powder for industrial applications. On the other hand, production cost need to be optimized. The study on the reused organics might be considered as an important factor for cost reduction.