CHAPTER IV
RESULTS AND DISCUSSION

4.1 Microemulsion

4.1.1 Effect of Surfactant Structure on the Size of Reverse Micelle

With different surfactant structures of TX-100 and AP-135, the microemulsions were formed at different concentrations of surfactants. The microemulsion of AP-135 was formed at low concentration when compared with the TX-100 microemulsion. At the same microemulsion condition and at constant temperature, the micellar size was 9 and 30 nm for the system of TX-100 and AP-135 respectively because of the small size of polar head group of TX-100 which is almost three times smaller than the AP-135. It means that the CMC of AP-135 is lower than that of TX-100 because the AP-135 has two hydrophobic tails to ease the formation of reverse micelle more than the TX-100 that has only one hydrophobic tail. The micellar sizes of these two systems were different almost three times too. Therefore, it also can be explained that the surfactant carrying a small polar head group can have the small size of reverse micelle.

4.1.2 Effect of Water Content and Temperatures on the Size of Reverse Micelle

It was found that at a given temperature, 30 °C the micellar size was reduced with a decrease of water content in both TX-100 and AP-135. For example, in the system of AP-135, the size of reverse micelle was reduced from 40 nm to 30 nm when Wo was decreased from 6 to 2 and for the TX-100 system, the size of reverse micelle was reduced from 22 nm to 9 nm when Wo was changed from 3 to 2 corresponding to an increasing of the molar ratio of water to surfactant.
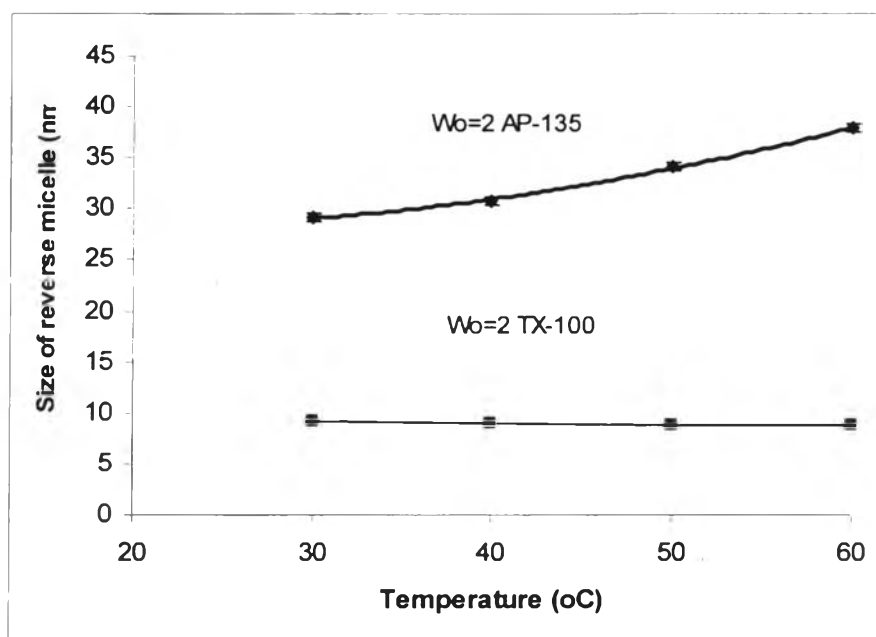
Figure 4.1 Effect of temperature on micellar size of AP-135 and TX-100.

The effect of temperature on the micellar size is shown in Figure 4.1. The temperature was varied from 30 to 60 °C at Wo equal 2. The result shows that an increasing temperature decreased the micellar size of TX-100, but increased the micellar size of AP-135. It might be corresponded to the dehydrate water of polyoxyethylene at the polar head group of TX-100 was removed at high temperature and thus decrease the CMC of surfactant and the size of reverse micelle. While, for the microemulsion of AP-135, an increasing the temperature increases the micellar size due to expansion volume of water in the micellar core.

4.1.3 Effect of Co-surfactant on the Size of Reverse Micelle

The concentration of co-surfactant was varied from 10 to 50 weight percent. It was found that the addition of co-surfactant at low concentration reduced the repulsive force of the surfactant head groups and hence reduces the micellar size (Figure 4.2). However, the addition of co-surfactant more than 20 percent caused the size of reverse micelle to increase because the co-surfactants solubilize in the deeper palisade region of the reverse micelle, and hence the micellar size is expanded.
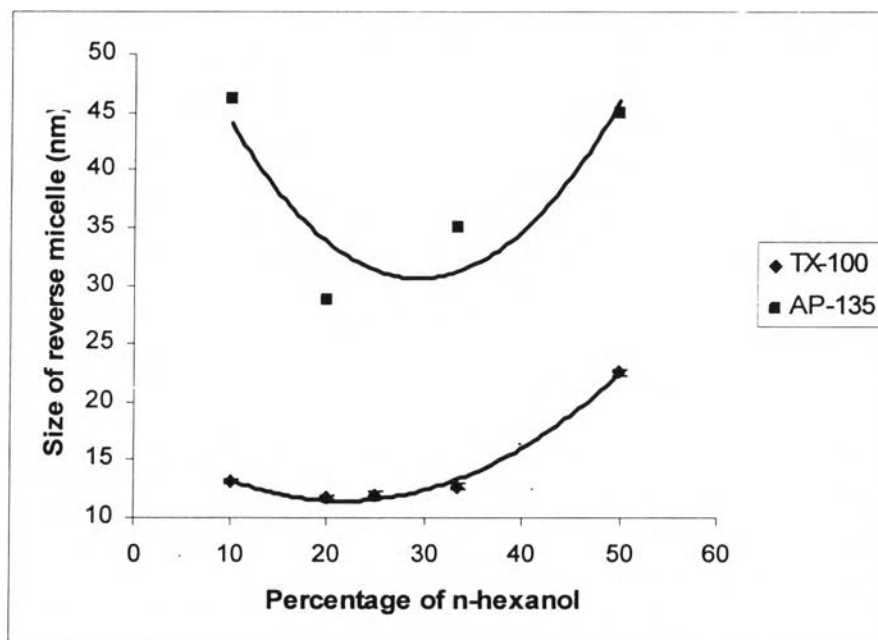
Figure 4.2 Effect of co-surfactant on the micellar size of TX-100 and AP-135.

4.1.4 Effect of Concentration of Metal Salt Precursor on the Micellar Size

The concentration of metal salt precursor was varied from 0.1 M to 0.5 M. The results (Figure 4.3) show that the metal salt concentration has slightly effect on the micellar size. This trend is similar in both system of AP-135 and TX-100. Although the concentration of metal salt precursor should have had no effect on the size of reverse micelles, a slightly increase in size may be corresponding to an increase of repulsive force between the polar groups of surfactant when the tin ion (Sn^{+4}) was bonded to the polar group of surfactant. From the molecular structure of these two surfactants, the direction of negative polar moment is towarding the oxygen atom (δ^{-}) of polyoxyethylene and polyethylene glycol groups while the other hydrocarbon at the polar head group is slightly positive. Thus Sn^{+4} can interact with the O atom resulting in an increasing of the repulsive force between the polar head group of surfactant and Sn^{+4}. Therefore, the micellar size is larger.
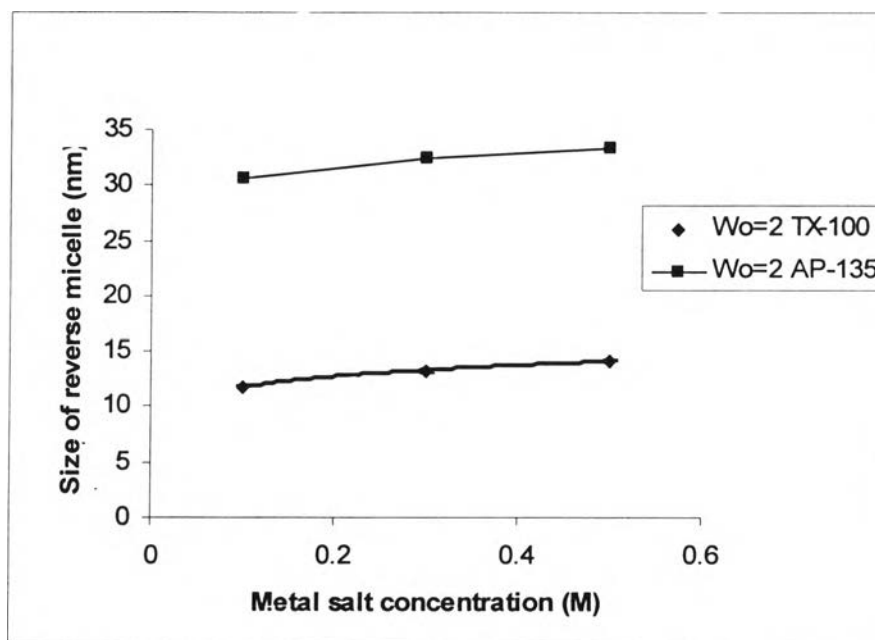
Figure 4.3 Effect of metal salt concentration on the size of reverse micelle.

4.2 Precipitation and Calcination

4.2.1 Effect of Calcination Temperatures on the Morphology of Particles

Tin chloride prepared in the microemulsion of TX-100 at the condition of Wo equal 2, 20 % (wt) co-surfactant, 0.1 M of tin chloride was precipitated at 60 °C and calcined for 2 hour at different temperatures. The calcination temperatures were varied between 550 °C and 900 °C. From the XRD patterns in Figure 4.4, the powders obtained from the microemulsion and the commercial were cassiterize phase or rutile structure and the crystallinity increased as increase the calcination temperature. The surface areas of particles were characterized by Autosorb I. The results show that the surface area of tin oxide powder was 45 m^2/g at low calcination temperature (550 °C), while at 900 °C was 38 m^2/g and the surface area of the commercial tin oxide was 8.61 m^2/g.
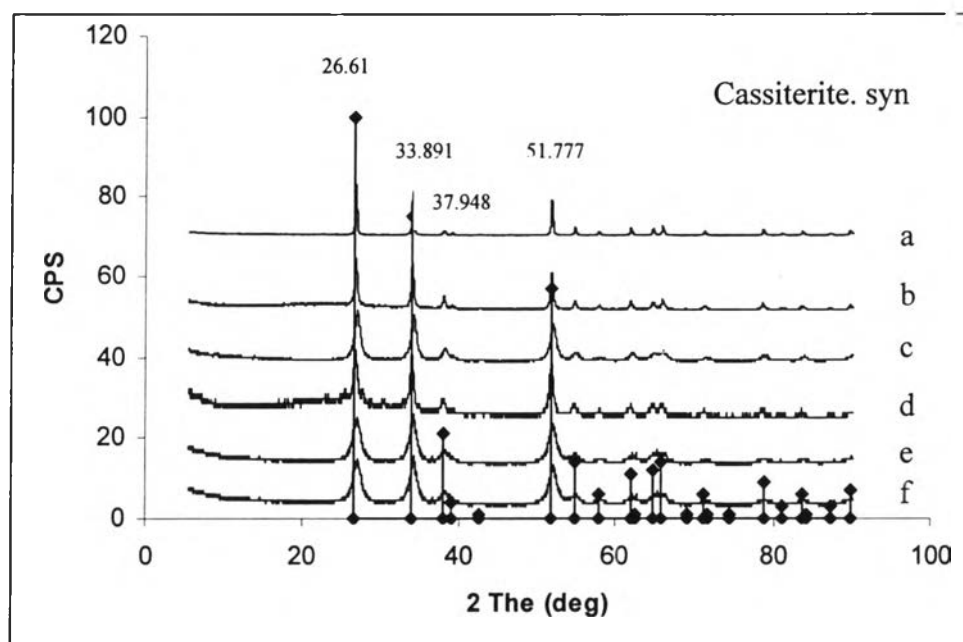
Figure 4.4 XRD patterns of commercial tin oxide (a), the synthesized tin oxide prepared by 0.1 M. of $SnCl_4$ at 900°C (b), 750°C (c), 650°C (d), and 550°C (e), tin oxide prepared by 0.7 M. of $SnCl_4$ at 550°C (f).

In order to estimate the morphology and particle size, SEM and TEM were used to characterize the powder. The micrographs of tin oxide prepared by 0.1 M of tin chloride were calcined at 550 and 900°C are shown the Figures 4.5-4.6 respectively. At low calcination temperature (550°C), the size of the synthesized tin oxide was 8 nm and at high temperature (900°C), the particle size was 15 nm. However, the morphology of synthesized tin oxide still shows homogeneity in size. It can be explained that agglomeration of grain can occur at high temperature.
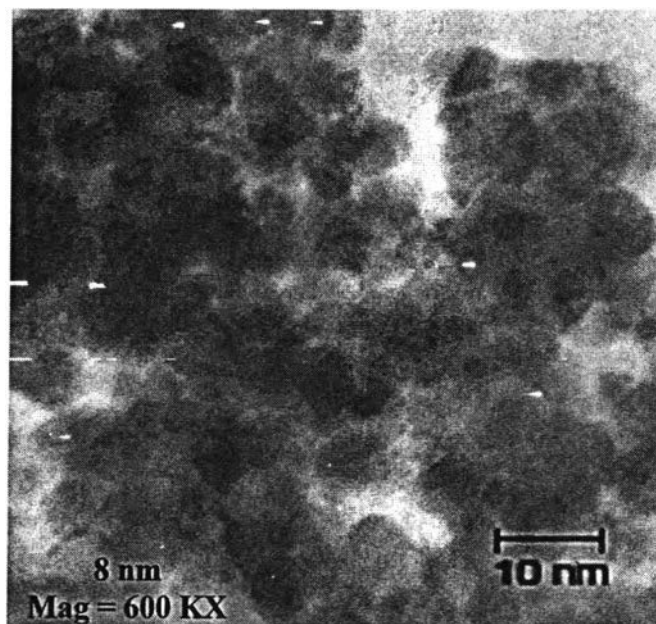
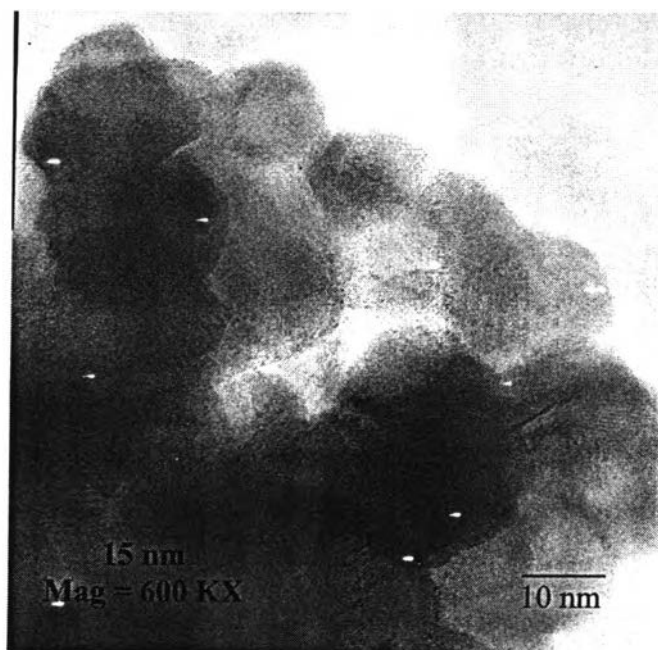**Figure 4.5** TEM of the synthesized tin oxide calcined at 550 °C.

Figure 4.6 TEM of the synthesized tin oxide calcined at 900 °C.

The comparison of the morphology of the synthesized tin oxide and the commercial one are shown in the Figures 4.7-4.12.
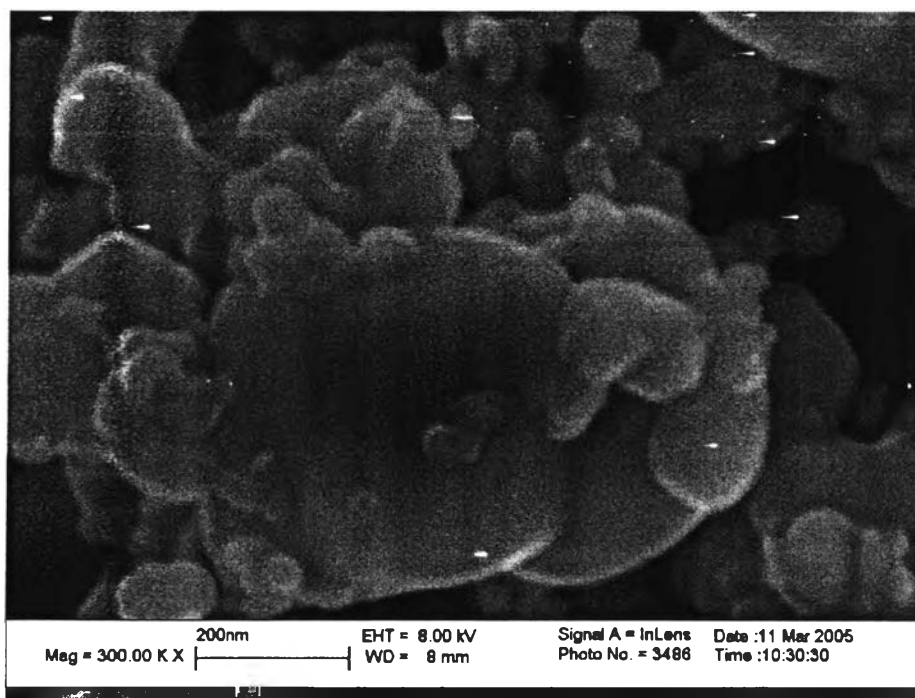

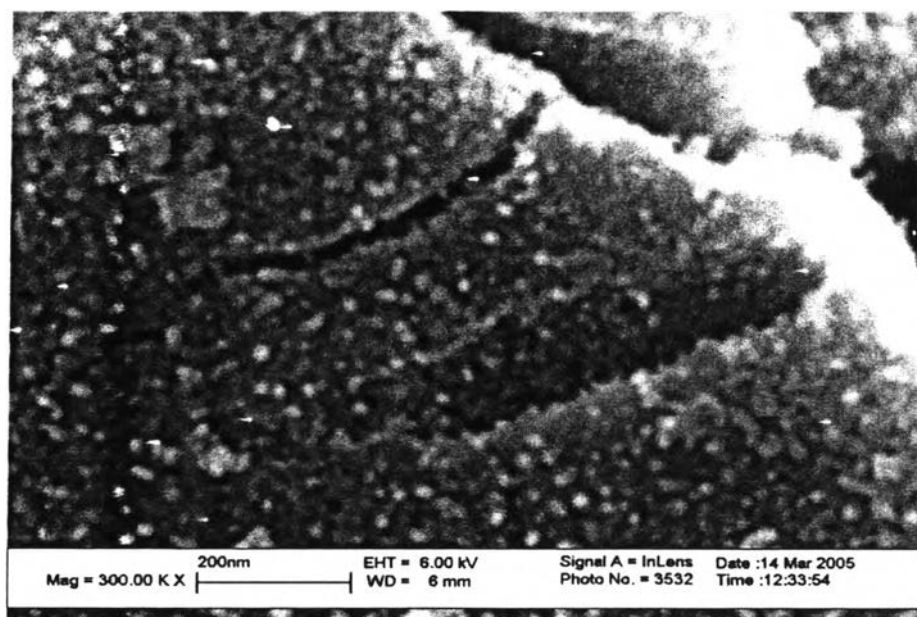**Figure 4.7** SEM of the commercial tin oxide.

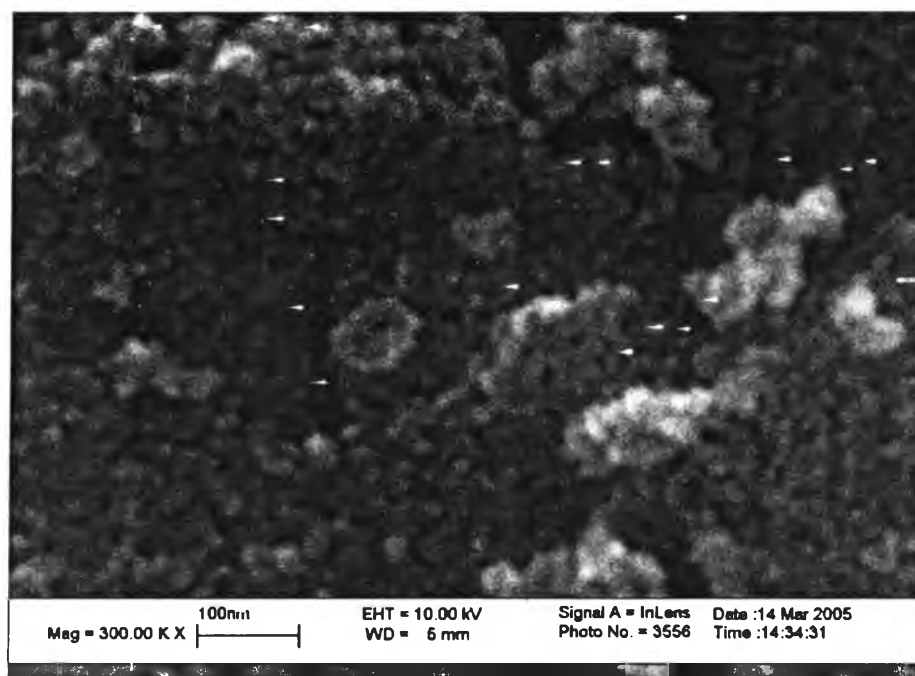**Figure 4.8** SEM of the synthesized tin oxide prepared by 0.1 M. of $SnCl_4$.

| 100nm | EHT = 10.00 kV | Signal A = InLens | Date :14 Mar 2005 |
| Mag = 300.00 K X | WD = 6 mm | Photo No. = 3556 | Time :14:34:31 |

Figure 4.9 SEM of the synthesized tin oxide prepared by 0.7 M. of SnCl$_4$.
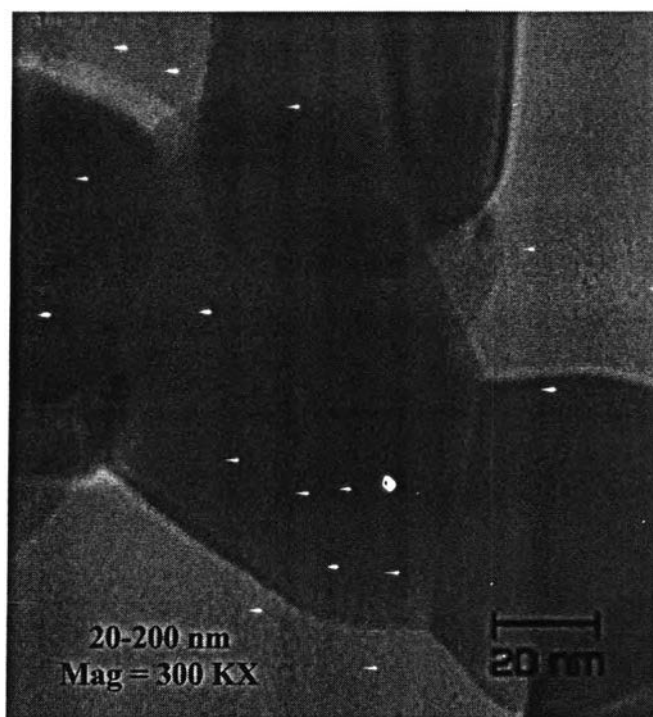

20-200 nm
Mag = 300 KX
20 nm
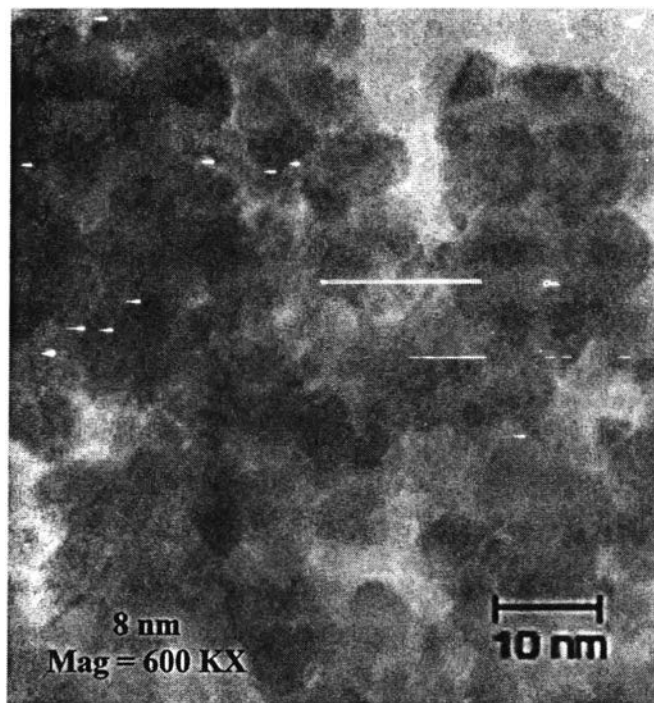
Figure 4.10 TEM of the commercial tin oxide.

Figure 4.11 TEM of the synthesized tin oxide prepared by 0.1 M. of SnCl$_4$.
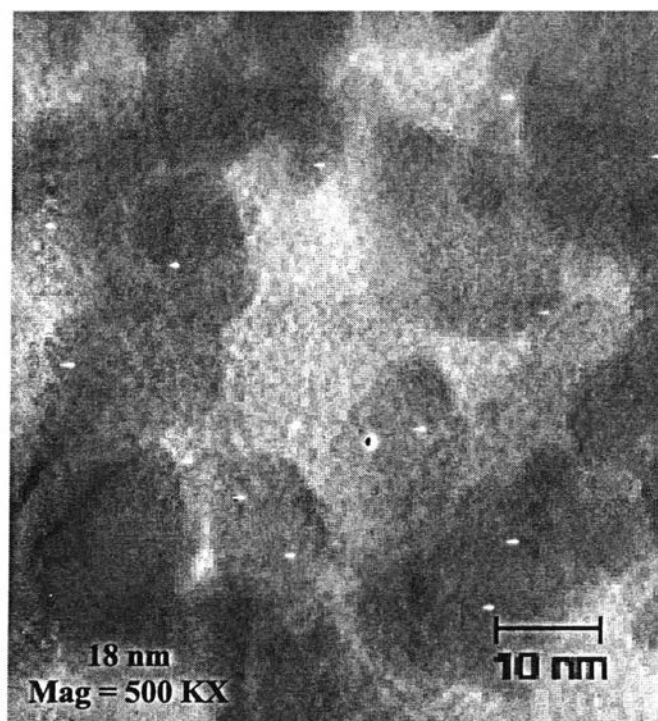

Figure 4.12 TEM of the synthesized tin oxide prepared by 0.7 M of SnCl4 calcined at 550 ^{o}C.

The results show that the size of the synthesized tin oxide calcined at 550 °C that was prepared by 0.1 M. of SnCl$_4$ was 8 nm, while the size of tin oxide prepared by 0.7 M. of SnCl$_4$ was 18 nm, on the other hand, the size of commercial tin oxide was in the range of 20-200 nm. However, the size of synthesized tin oxide increases to 15 nm by increasing of calcinations temperature and still shows homogeneity in size.

4.3 Sensing Application

4.3.1 The Effect of Morphology

SnO$_2$ powders obtained from the microemulsion of non-ionic surfactant (TX-100) with two different concentrations of SnCl$_4$ (0.1 and 0.7 M.), which its surface areas were 45 m^2/g for 0.1 M. SnCl$_4$ and 22 m^2/g for 0.7 M SnCl$_4$. The sensing responses are shown in the Figures 4.13-4.14.
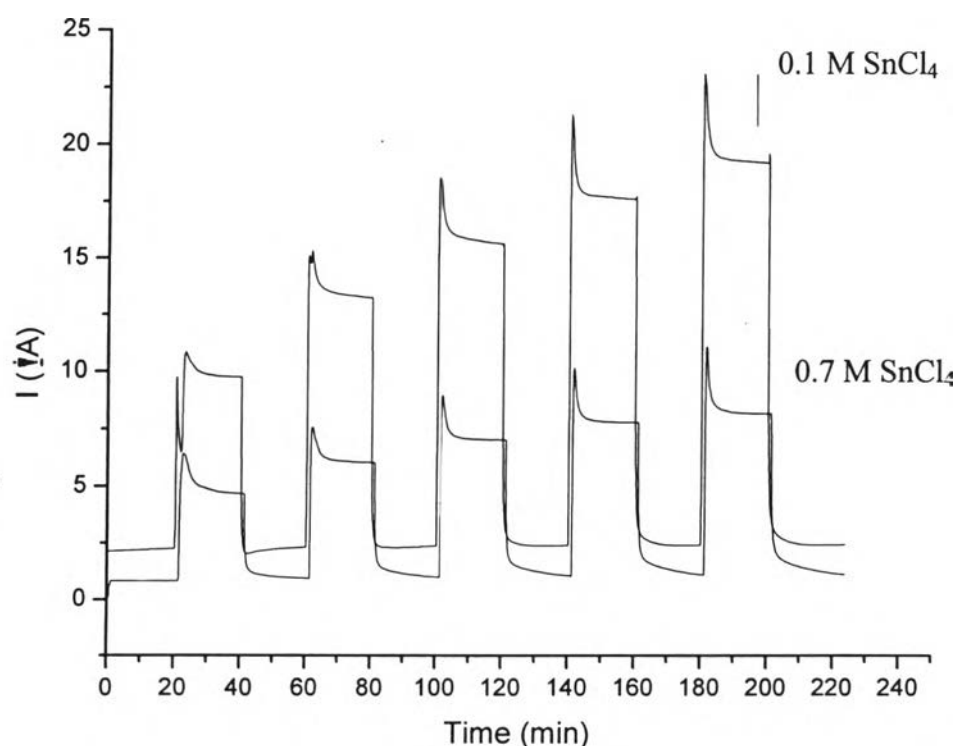

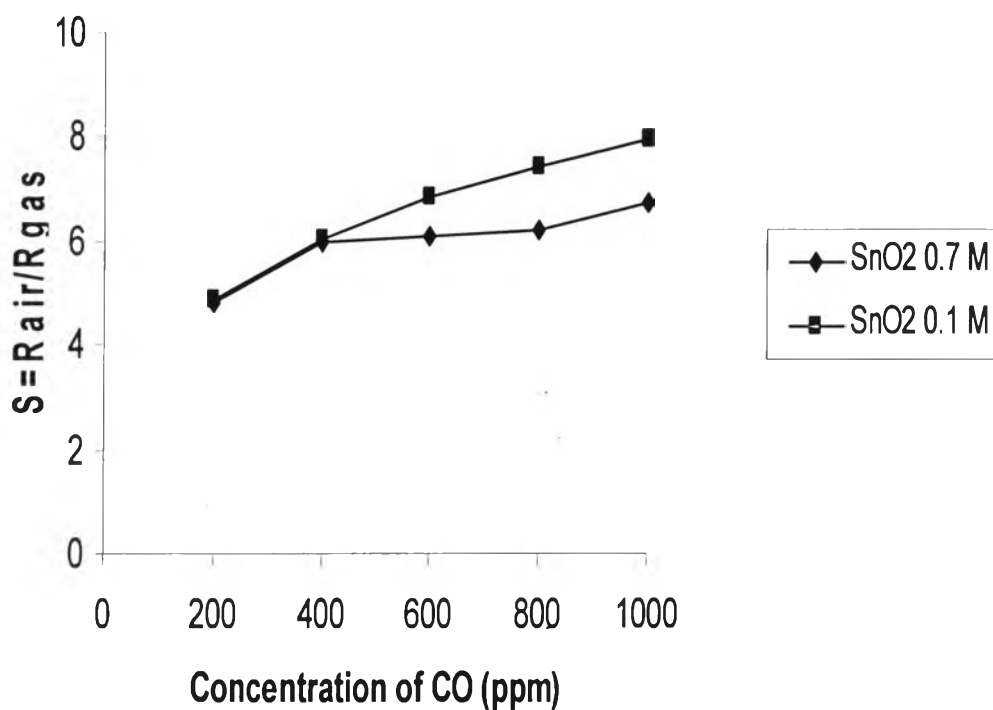**Figure 4.13** Sensing signals of the synthesized SnO$_2$ prepared by microemulsion.

Figure 4.14 The sensitivity of the synthesized SnO_2 at 400 °C.

It was found that the sensing results from SnO_2 powder prepared from 0.1 M $SnCl_4$ was higher than those prepared from 0.7 M $SnCl_4$ due to the smaller in crystal size and higher surface area of tin oxide. The CO sensitivity was also increased with an increase in concentration of the CO gas.

4.3.2 The Effect of Temperature

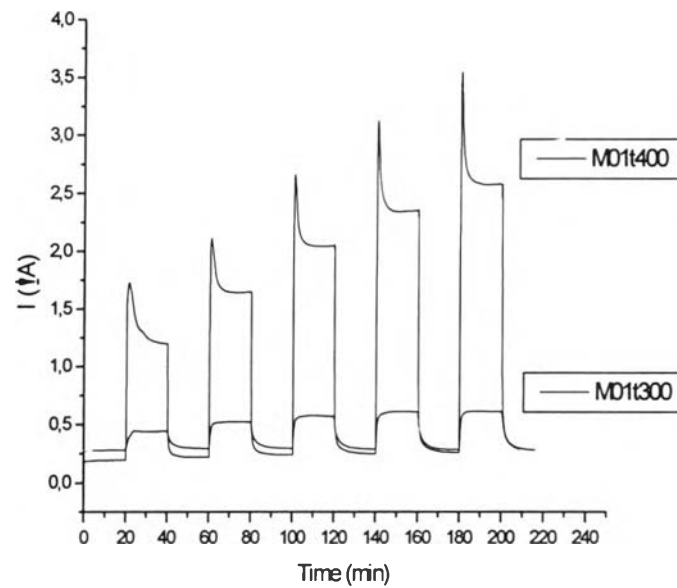The synthesized SnO_2 obtained from 0.1 M. of $SnCl_4$ was tested.

Figure 4.15 The sensing signal of the synthesized SnO_2 at different sensing temperature.
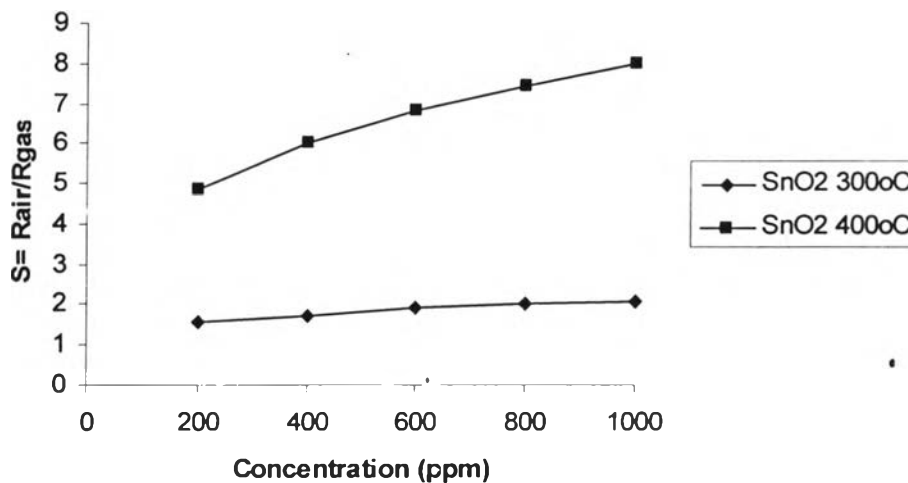

Figure 4.14 The sensitivity of the synthesized SnO_2 at different temperature.

The results show that the sensing signal and the sensitivity increase with an increase in temperature. It might be due to the natural of semiconductor that increases the conductivity at high temperature.