CHAPTER 3

RESULTS AND DISCUSSION

3.1 Particle properties

In this research, Flame spray pyrolysis was employed for the synthesis of pure ZnO and ZnO/MgO nanocomposites. Zinc naphthenate and magnesium acetate tetrahydrate dissolved in ethanol were used as Zn and Mg precursors, respectively. Pure ZnO and ZnO/MgO nanocomposites were characterized by X-ray diffraction (XRD), Scanning Electron Microscopy (SEM) and energy-dispersive X-ray spectrometry (EDS), Transmission Electron Microscopy (TEM) and Brunauer-Emmett-Teller (BET) analysis.

3.1.1 X-ray diffraction analysis (XRD)

The XRD patterns of flame-made ZnO and ZnO/MgO nanocomposites were shown in Figure 3.1. The samples were highly crystalline, and all peaks were confirmed to be the hexagonal structure of ZnO by comparison with standard file of ZnO [JCPDS file No. 79-205] and the cubic structure of MgO [JCPDS file No. 74-1225]. The diffraction peak for 20 wt% ZnO/MgO nanocomposites was the broadest as compared to other patterns, suggesting relatively small particles. XRD analysis revealed not only that the MgO was successfully deposited on the ZnO, but also that the deposited MgO comprised a crystalline phase.

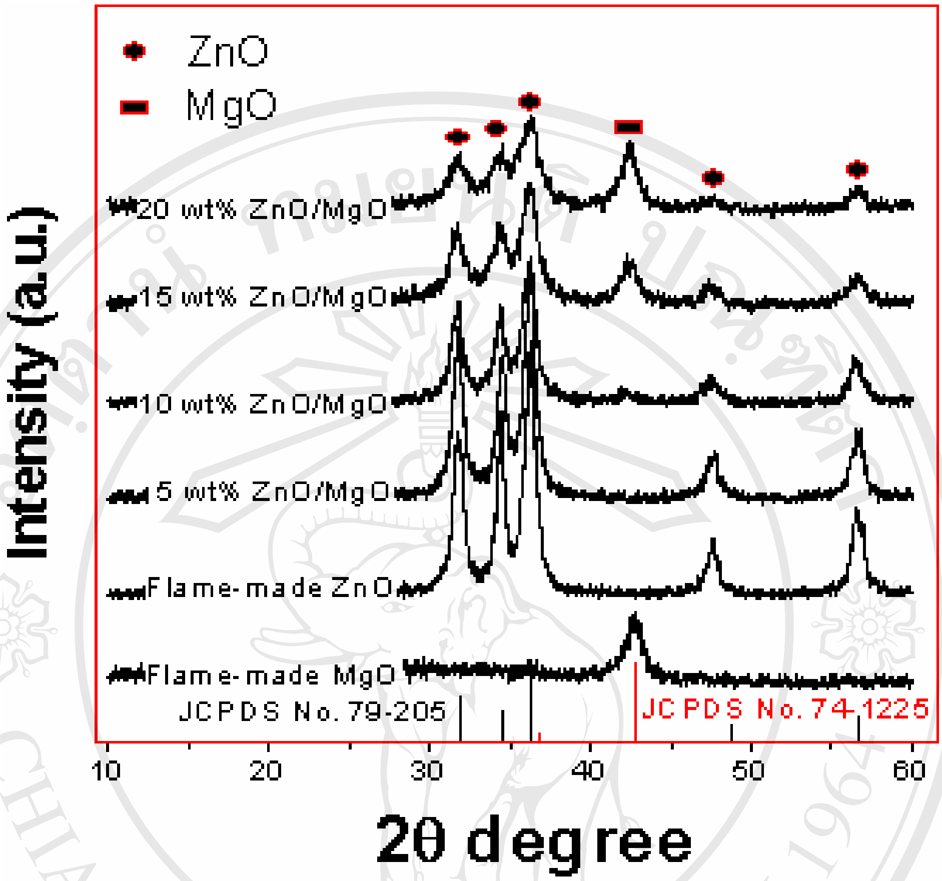


Figure 3.1 XRD patterns of flame-made ZnO and ZnO/MgO nanocomposites

3.1.2 Brunauer-Emmett-Teller (BET) analyzer

The average diameters of BET-equivalent particle (d_{BET}) were calculated using SSA_{BET} equation and the density of ZnO and MgO by considering their weight content for different doping as shown in Table 3.1. It can be assumed that the nanoparticles were rather agglomerated and were degassed at 150°C prior to the experiment, which can be affected a larger particles with decreasing SSA. The specific surface area of pure ZnO, pure MgO and 5-20 wt% ZnO/MgO nanocomposites were measured by nitrogen adsorption after degassing the sample at 150ºC were 88.85, 198.38, 162.78, 177.99, 181.73 and 146.84 m^2/g, corresponded to BET equivalent diameter of 12.04, 8.45, 6.70, 6.24, 6.23 and 7.86 nm, respectively. From these data, it can be deduced that as the amount of MgO loading have effect to the particles size of ZnO, suggesting relatively with TEM measurement.

Table 3.1 SSA_{BET} and BET equivalent diameter of pure ZnO, pure MgO and 5-20 wt% ZnO/MgO nanocomposites

Samples	**Specific surface area SSA_{BET} (m^2/g)**	**Diameter d_{BET} (nm)**
Pure ZnO	88.85	12.04
Pure MgO	198.38	8.45
5 wt% ZnO/MgO nanocomposites	162.78	6.70
10 wt% ZnO/MgO nanocomposites	177.99	6.24
15 wt% ZnO/MgO nanocomposites	181.73	6.23
20 wt% ZnO/MgO nanocomposites	146.84	7.86

3.1.3 Scanning Electron Microscopy (SEM)

The morphology of highly crystalline flame-made (a) pure ZnO (b) pure MgO and (b-f) 5-20 wt% ZnO/MgO nanocomposites at high magnification (200K) for SEM analysis were shown in Figure 3.2. The SEM micrographs clearly showed nano-structural morphologies of ZnO nanoparticles prepared by FSP. A rather segregate of nanoparticles was observed in most cases. A typical SEM micrograph in Figure 3.2 (a-f) showed the rather agglomerated nanospheroidal particles with an average diameter of 10-20 nm. From these data, it was found that the rough morphology and the rough particles size were not changed with the increasing MgO doping levels. Nevertheless, the accurate sizes and morphology of the nanoparticles can be estimated from the TEM analysis. While the SEM images provide 3-D morphology to estimate particle sizes, TEM images can reveal internal structure and a more accurate measurement of particle size and morphology.

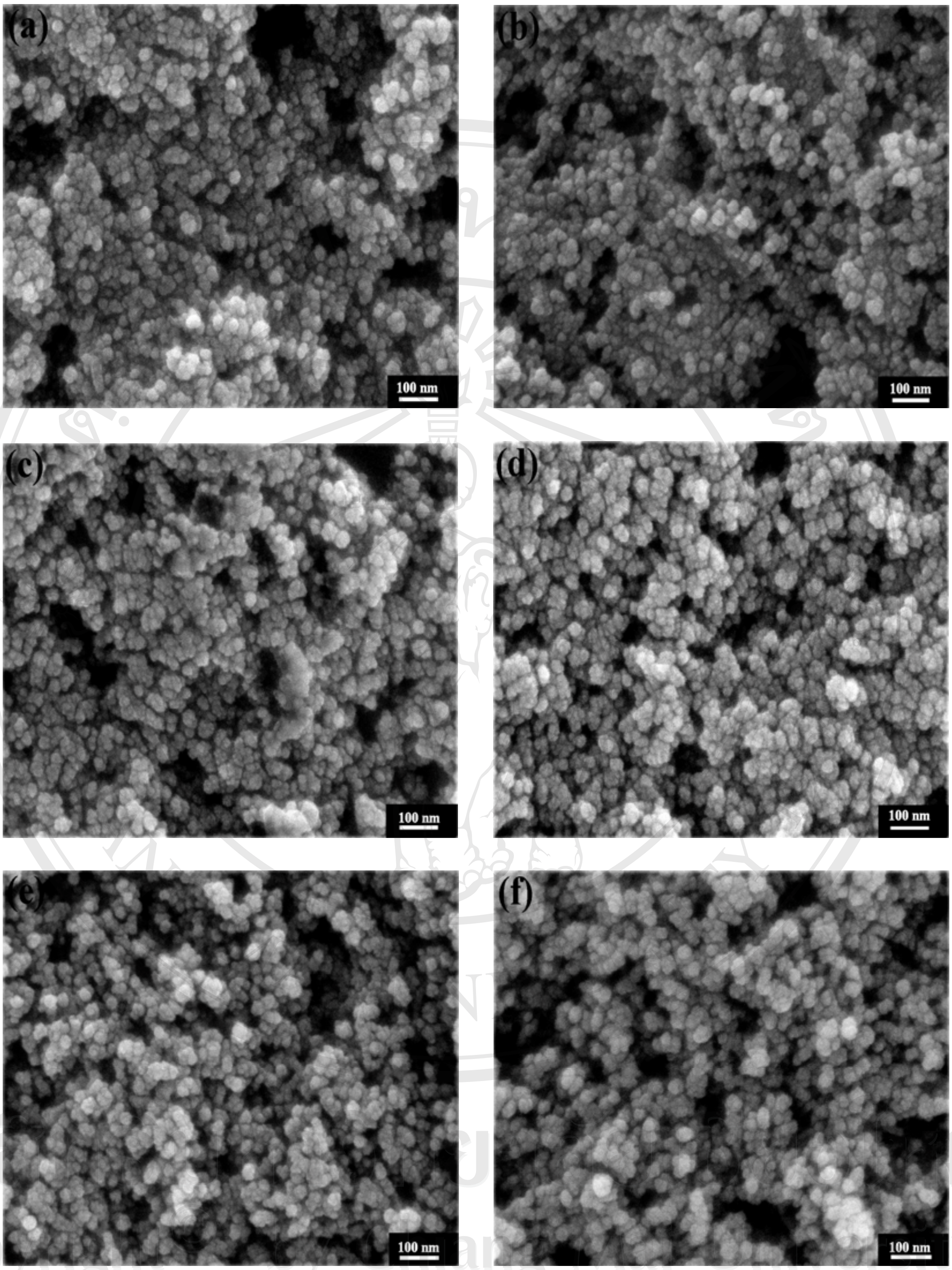
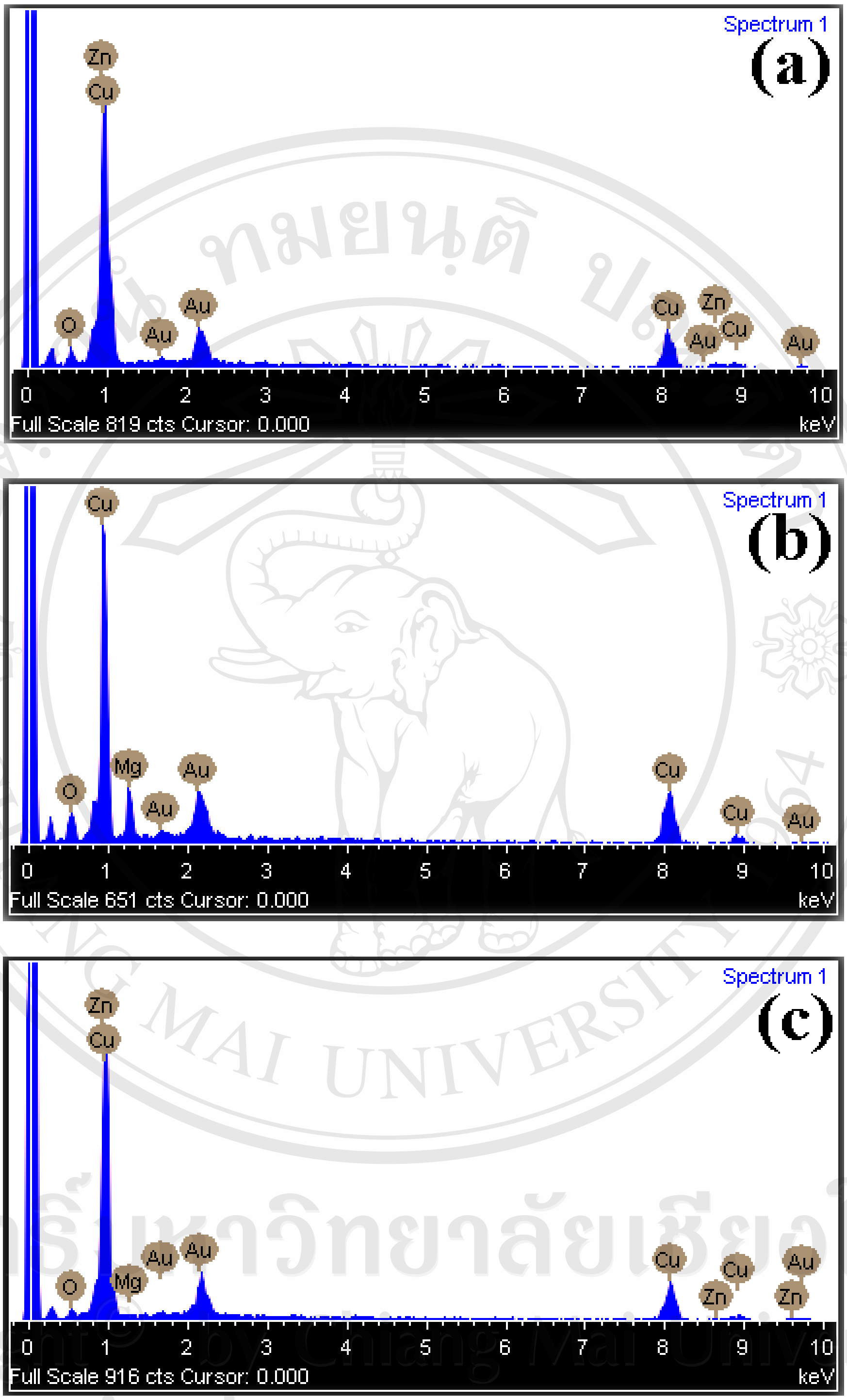


Figure 3.2 SEM micrographs of highly crystalline flame-made (a) pure ZnO, (b) pure MgO (c) 5 wt%, (d) 10 wt%, (e) 15 wt% and (f) 20 wt% of ZnO/MgO nanocomposites

3.1.4 Energy Dispersive X-Ray Spectrometry (EDS)

Figure 3.3 showed the EDS spectrum of chemical elements of pure ZnO, pure MgO, 5 wt%, 10 wt%, 15 wt% and 20 wt% of ZnO/MgO nanocomposites. The signals of EDS spectrum corresponded to Zn, Mg and O elements. From these results, the Cu and Au spectrum were also evidently showed in the pattern, which caused by the contaminations from copper trap and Au sputtering. The Zn, Mg and O signals arose from the particles of ZnO/MgO nanocomposites. The element composition data from EDS confirmed MgO was actually in ZnO/MgO nanocomposites. However, the concentrated amount of element compositions was slightly changed depending on the different selected area in EDS analysis.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(a)
Zn
Cu
O
Au
Au
Cu
Zn
Au
Cu
Au
0 1 2 3 4 5 6 7 8 9 10
Full Scale 819 cts Cursor: 0.000
keV
Spectrum 1
(b)
Cu
Mg
O
Au
Au
Cu
Cu
Au
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Full Scale 651 cts Cursor: 0.000
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(c)
Zn
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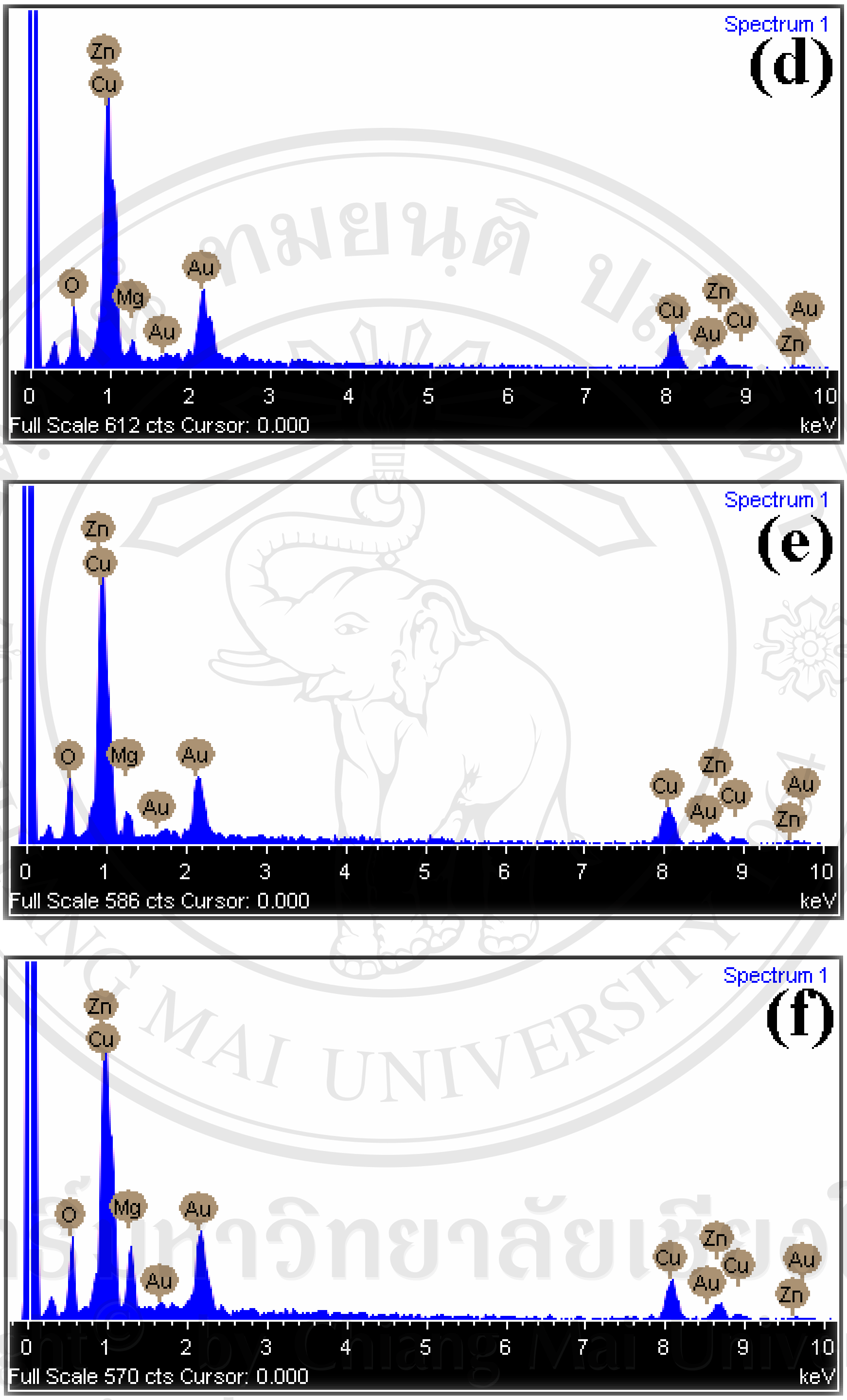

Figure 3.3 EDS analysis of (a) pure ZnO, (b) pure MgO, (c) 5 wt%, (d) 10 wt%, (e) 15 wt% and (f) 20 wt% of ZnO/MgO nanocomposites

3.1.5 Transmission Electron Microscopy (TEM)

The accurate sizes of nanoparticles were confirmed by TEM bright-fields images. Figure 3.4 (a), (b), (c), (d), (e) and (f) showed the TEM bright-field images of pure ZnO, pure MgO, 5 wt%, 10 wt%, 15 wt% and 20 wt% of ZnO/MgO nanocomposites, respectively. The corresponding diffraction patterns were shown in the insets. The diffraction patterns illustrating the spot patterns of the hexagonal structure of ZnO and the cubic structure of MgO, indicating the ZnO and MgO nanoparticles were highly crystalline, which was in good agreement with the XRD patterns. The crystallite sizes of ZnO spheroidal and hexagonal particles were found to be in the range of 5-20 nm. ZnO nanorods were found to be approximately 5-15 nm in width and 15-30 nm in length. Also, the crystallite sizes of MgO spheroidal particles were found to be in the range of 5-10 nm. 5-20 wt% ZnO/MgO nanocomposites spheroidal particles were found to be in the range of 5-15 nm. 5 wt% and 10 wt% of ZnO/MgO nanocomposites were found to be approximately 5-10 nm in width and 10-20 nm in length. Figure 3.4 (b-f) show the sizes of pure MgO and 5-20 wt% ZnO/MgO nanocompoistes were very small, when compared with the size of ZnO nanoparticles.

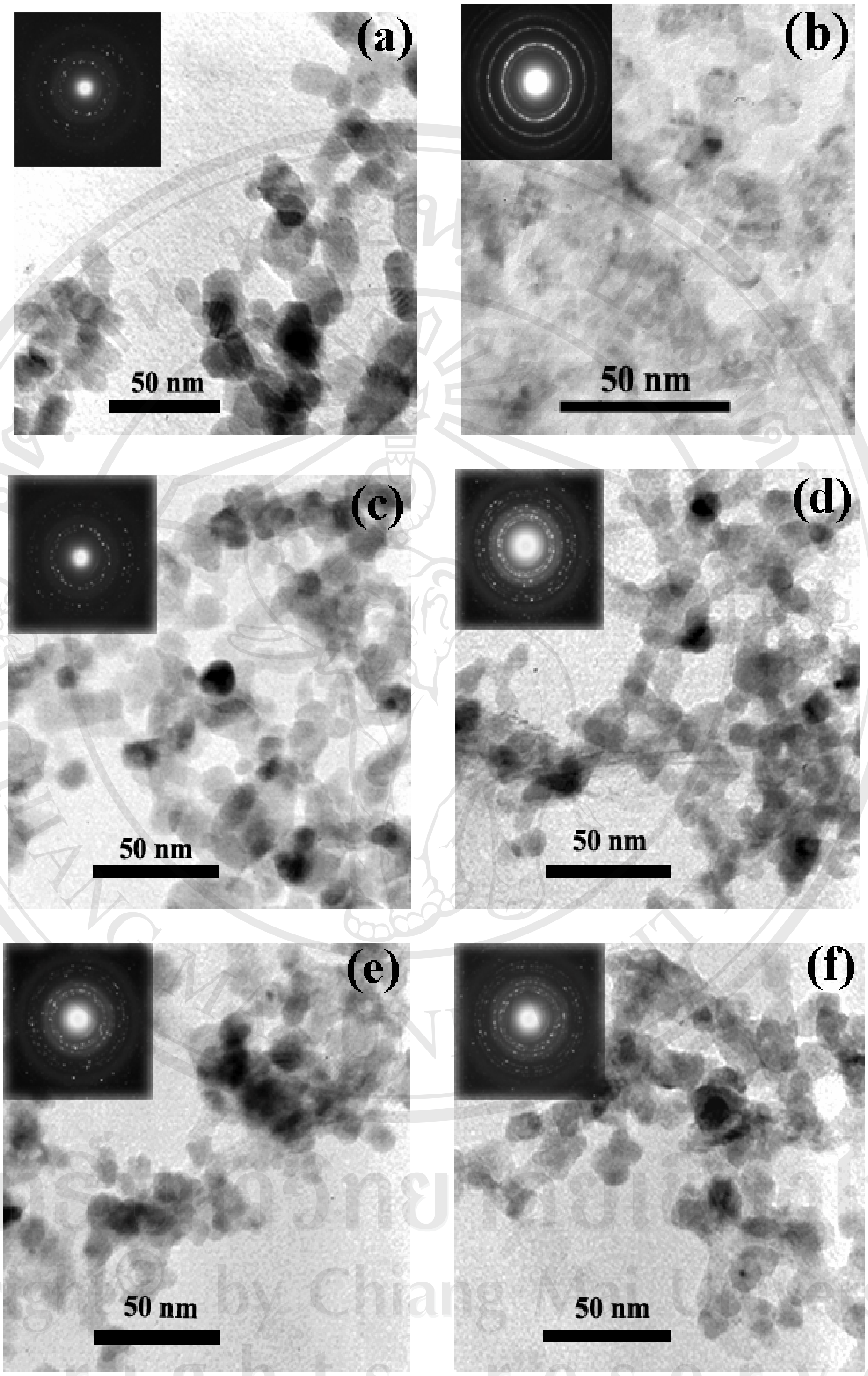

Figure 3.4 TEM bright-field images of (a) pure ZnO, (b) pure MgO, (c) 5 wt%, (d) 10 wt%, (e) 15 wt% and (f) 20 wt% of ZnO/MgO nanocomposites