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RESEARCH ARTICLE

Study of α-Fe_2O_3@ZnO nanoleaves: Morphological and optical study

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Abstract: In this paper, α-Fe_2O_4@ZnO nanoparticles (NPs) were synthesized by coprecipitation method in the presence of PVP and EG surfactants. The samples were charactrized by x-ray fluorescence (XRF), x-ray diffraction (XRD), scanning electron microscopy (SEM), high resolution transmission electron microscopy (HRTEM), and fourier transform infrared spectroscopy (FTIR). The XRD results exhibited rhombohedral α-Fe_2O_3 and wurtzite structure of ZnO. The SEM images showed that the NPs changed from rod-shape to nanoleaves particles after heat treatment. The TEM studies displayed the formation of Fe_2O_3@ZnO core-shell of as-synthesized NPs. The stretching vibrations peaks in FTIR in the wavenumber of 532 cm^{-1} and 473 cm^{-1} ascribed to the Fe and Zn groups. The XRF data indicated decreasing of the Fe weight percent from 22% Wt. to 25% Wt., after heat treatment.

Keywords: α-Fe_2O_4@ZnO, Nanoleaves, XRF, Crystal structure, FTIR

1 Introduction

Metal oxide semiconductors (MOS) are recognized as the best option for electronic and industry applications[1–5]. The MOS examples such as TiO_2[6–10], CeO_2[11–14], Fe_2O_3[15–18], ZnO[19–21], Al_2O_3[22–25], V_2O_5[26–30], Co_2O_3[31–33], and composites such as FeCo[34–36] and FePt[37–39], and coupled MOS such as Fe_2O_3-CeO_2[40–43], SiO_2-TiO_2[44], Ce-doped Al_2O_3[45,46], Fe-doped Al_2O_3[47–50], Fe-doped TiO_2[51–54], Ce-doped TiO_2[55–57], Co-doped ZnO[58,59] are greatly used in optoelectronic applications and photocatalytists. However, study shows that the optical performance of MOS is limited due to rapid electron-hole recombination, light corrosion, and poor activity in sunlight. This factor has led scientists to do more research to improve the optical properties in visible light by reducing electron-hole recombination[60]. Among the various semiconductors application, zinc oxide (ZnO) has received much attention due to its unique structural, optical and surface properties. ZnO has a suitable bandgap about 3.73 eV and on the other hand its electron mobility is more than TiO_2 with the same bandgap. The optical properties activity of ZnO is limited to the light radiation in the ultraviolet region due to its wide bandgap, and electron-hole recombination[61]. Fe_2O_3 is also MOS that has been recognized as an important semiconductor which is active in visible light with a short band gap length of about 2.4 eV. In addition, iron oxide with magnetic properties has been proposed as an attractive idea for the separation of nanocatalysts from the liquid phase and has been studied in various photocatalytic processes of the liquid phase[62–64]. On the other hand, Fe_2O_3 is able to send electrons into the wide band gap of MOS with a wide band gap like ZnO, which helps to reduce electron-hole recombination. Therefore, it can be expected that by activating the two ZnO and Fe_2O_3 semiconductors, an active MOS in visible light will be achieved[65]. Fe_2O_3@ZnO coupled semiconductor increases the optical activity because of efficient electronhole separation at the interface. According to several investigations, Fe_2O_3@ZnO nanocomposites have been fabricated using various synthesis such as hydrothermal[66], sol-gel[67], solution combustion[68], and emulsion[69] method. However, many of these methods suffer from several drawbacks such as long reaction time, requirement of high reaction temperature. Coprecipitation synthesis is regarded as a versatile tool for the synthesis of MOS NPs and is a simple and economic route to synthesize the Fe_2O_3@ZnO NPs[70]. Present study refers to the synthesis of Fe_2O_3@ZnO via a direct coprecipitation method and study the morphological and optical properties. The novelty of this work is the synthesis of Fe_2O_3@ZnO NPs with new precursors and solvent and study the new morphology of the fern-like Fe_2O_3@ZnO

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nanoleaves by changing the temperature.

2 Experimental details

α-Fe_2O_4@ZnO NPs were synthesized by coprecipitation method by iron nitrate ($Fe(NO_3)_3.9H_2O$) and zinc nitrate ($Zn(NO_3)_2.9H_2O$) precursors. At the beginning, 2 g iron nitrate was dissolved in 60 mL deionized water with stirring at room temperature. After 20 min, 2 g of zinc nitrate precursor was added to the solution and then 1 g of polyvinylpyrrolidone (PVP) stabilizer was added to the solution. After 5 min 10 mL ethylene glycol (EG) was slowly added to the solution. The temperature was increased to 90 °C and pH value of solution was measured about 4. The product were evaporated for 4 hours, cooled to room temperature and finally annealed at 650 °C for 3 hours. The specification of the size, structure and optical properties of the as-synthesis and annealed samples were carried out. XRD was used to identify the crystalline phase and to estimate the crystalline size. The XRD pattern were recorded with 2θ in the range of 4-85o with type X-Pert Pro MPD, Cu-Kα: λ = 1.54 $\overset{\circ}{A}$. The morphology of the samples was characterized by FESEM with type KYKY-EM3200, 25 kV and exact size of the NPs wa determined by TEM with type Zeiss EM-900, 80 kV. Stretching bound of the samples was characterized by FTIR with WQF 510. The elemental analysis of the NPs was carried out by Spectro Xepos ED-XRF spectroscopy.

3 Results and discussion

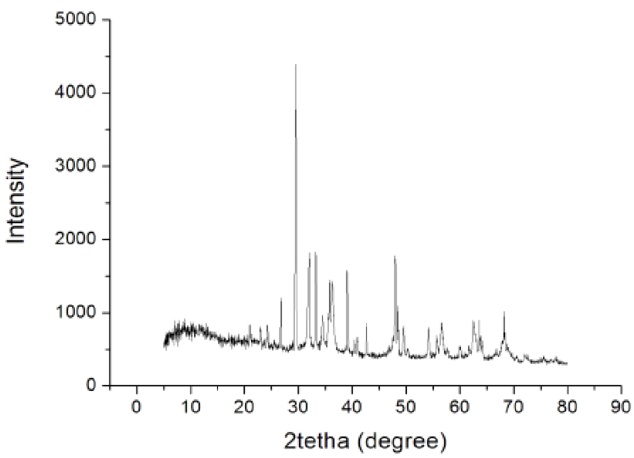

Figure 1. XRD patterns of as-prepared and annealed Zinc ferrite samples

Figure 1 exhibits the XRD spectrum of the annealed α-Fe_2O_4@ZnO NPs. The peaks correspond the α-Fe_2O_3 and ZnO NPs exhibit all the diffraction peaks of the hexagonal wurtzite ZnO and rhombohedral α-Fe_2O_3, respectively. The peaks formed at 2θ angles of 31.98°, 35.73°, 36.28°, 47.98°, 56.55°, 62.49°, 66.33° and 68.17° are indexed the diffraction peaks at (100), (002), (101), (102), (110), (103), (200) and (112) respectively, represent the hexagonal wurtzite of zinc oxide. The peaks formed at 24.15°, 33.24°, 39.05°, 49.53°, 53.96°, 57.45°, and 63.60° which are correspond to the diffraction peaks at (012), (104), (113), (024), (300), (122), and (300) respectively, suggesting the rhombohedral α-Fe_2O_3 structure. Moreover, peaks formed at 26.46°, 29.46°, 42.60° and 54.14o indicating the α-Fe_2O_3@ZnO structure. The crystallite size of the ordered α-Fe_2O_3@ZnO samples have been calculated from full width at half maximum (FWHM) and Debye- Scherrer formula[71]. The crystallite size of the NPs are determined around 42 nm.

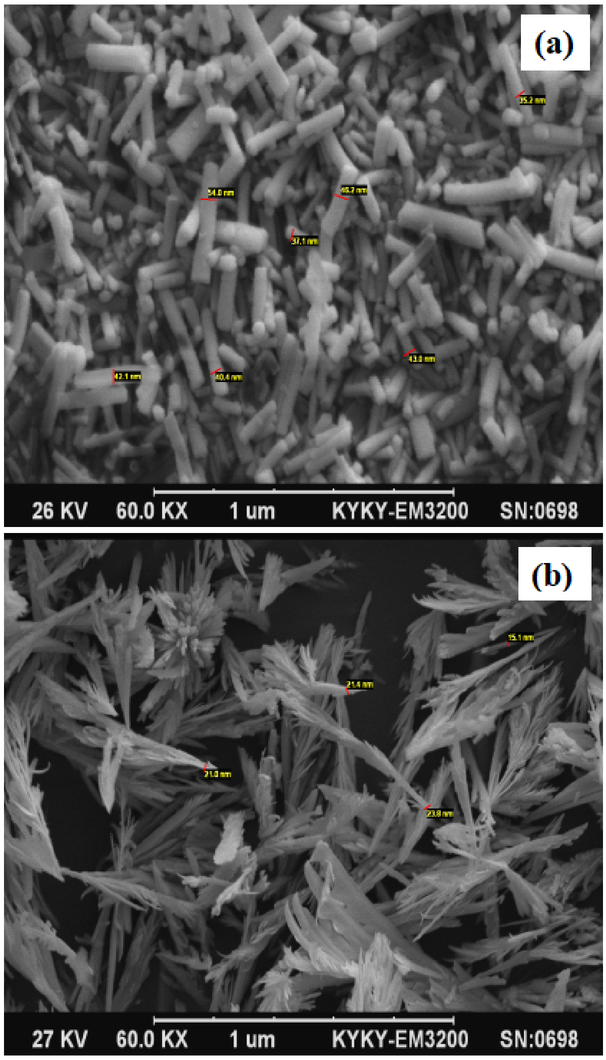

Figure 2. FESEM images of the (a) as-synthesized and (b) annealed samples

SEM analysis of the as-prepared and annealed α-Fe_2O_3@ZnO samples was done to investigate the morphology of the samples. Figure 2(a) indicates the SEM

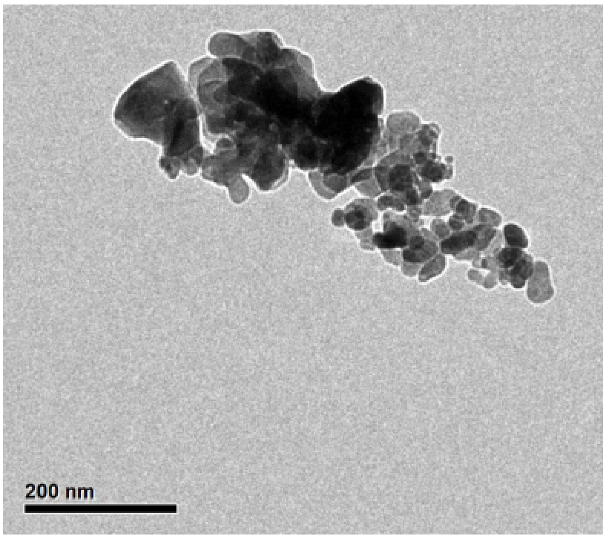

Figure 3. HRTEM image of the as-prepared Zinc ferrite sample

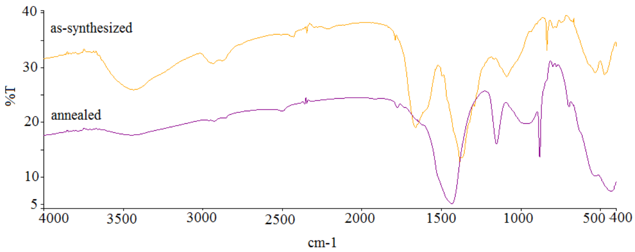

Figure 4. FTIR spectrum of (a) as-synthesized and (b) annealed α-Fe_2O_3@ZnO samples

image of the as-prepared samples and Figure 2(b) shows the SEM image of the annealed NPs at 650 °C for 3 hours. The morphology of the samples show that the samples change from rod-shape to fern-like Fe_2O_3@ZnO nanoleaves after heat treatment[70]. In fact, the PVP and EG stabilizers are removed by annealing process and the atomic and molecular interactions change between particles that lead the fern-like nanoleaves formation[72–77].

In order to measure the actual size of the NPs the TEM analysis was done Figure 3 shows the as-prepared TEM image of α-Fe_2O_3@ZnO core-shell nanopaerticles with average diameter of 48 nm prepared by coprecipitation method which is in accordance with XRD analysis[78–81]. From the TEM, Fe_2O_3 particles are formed as nuclei magnetic and zinc oxide as the shell of these NPs. It can be seen from TEM image, the core-shell formation lead to the aggregation and agglomeration of the NPs.

Figure 4 shows the FTIR spectra of the as-synthesized and annealed α-Fe_2O_3@ZnO NPs in the range of 400-4000 cm^{-1} frequencies to identify the chemical bonds as well as functional groups in the compound. The large broad band at 3436 cm^{-1} is attribiuted to the O-H stretching groups. The spectra of the annealed sample demonstrates that the O-H band reduces and it shifts to higher energy values at 3459 cm^{-1} which shows that O-H groups are chemically bonded to the oxide structure[82]. The sharp peaks at 1662 cm^{-1} and 1384 cm^{-1} are due to bending vibration of C = O respectively. The peaks in the frequencies of 532 cm^{-1} and 473 cm^{-1} are assigned to the stretching vibrations of Zn-O and Fe-O bonds[83, 84].

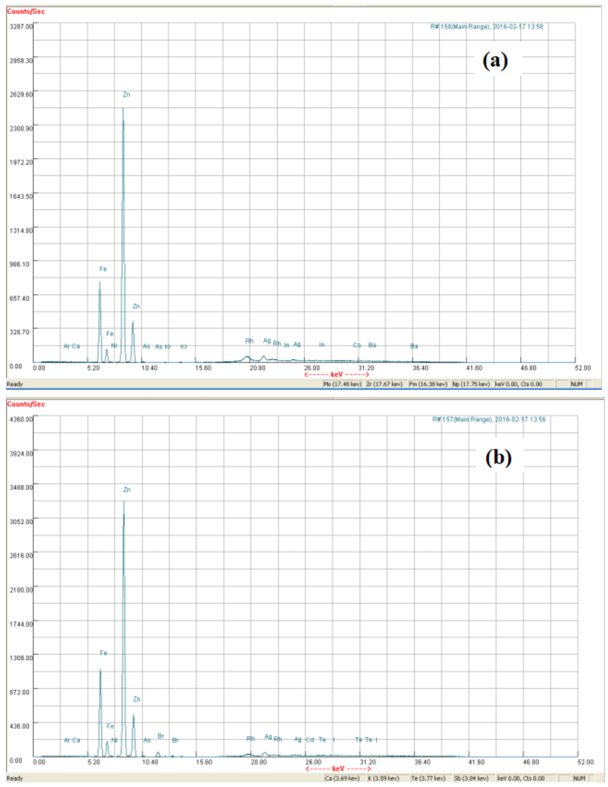

Figure 5. XRF analysis of (a) as-prepared and (b) annealed Zinc ferrite NPs

XRF analysis of α-Fe_2O_3@ZnO samples prepared by coprecipitation method is presented in Figure 5. The as-synthesized and annealed samples confirms the existence of Zn and Fe elements. XRF exhibits the peaks of Fe and Zn elements with less Pb, Sb, Bi and Sn contaminations. The XRF data revealed increasing of the Fe weight percent from 22% Wt. to 25% Wt., by annealing. It may be because of increasing of the Fe^{3+} into the crystal lattice. The value of Fe^{3+}/ ZnO is obtained 0.28 for the as-prepared NPs, while it is achieved 0.32 for the annealed samples suggesting an increase in the Fe content.

4 Conclusion

α-Fe_2O_3@ZnO NPs were successfully fabricated by simple coprecipitation method. The XRD spectra exhibited the rhombohedral α-Fe_2O_3 structure and wurtzite ZnO structure. The SEM images indicated that the NPs changed from rod-shape to fern-like Fe_2O_3@ZnO nanoleaves after heat treatment. TEM image showed the formation of core-shell α-Fe_2O_3@ZnO NPs. FTIR analysis ascribed the presence of Fe and Zn stretching bound and it also confirmed the formation of α-Fe_2O_3@ZnO bound. Finally, the XRF analysis demonstrated an increase in Fe content into the crystal lattice.

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