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Synthesis and characterization of nanocrystalline spinel Zinc ferrite prepared by sol-gel auto-combustion technique

S. Khorrami[1], F. Gharib[2], G. Mahmoudzadeh[1,*], S. Sadat Sepehr[1], S. Sadat Madani[1], N. Naderfar[1], S. Manie[1]

[1]Department of chemistry, North tehran branch, Islamic azad university, Tehran, Iran
[2]Department of chemistry,Shahid beheshti university,Tehran, Iran

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Abstract

Sol–gel auto-combustion is a unique combination of the combustion and the chemical gelation processes. In this work, nanosize (d) powders with compositions of $ZnFe_2O_4$ were synthesized by sol–gel combustion method. The puffy, porous brown powders as-combusted was calcined at the temperature of 750–1000°C for 4 h. These powders are characterized by X-ray diffraction and scanning electron microscopy. Particle size and morphology of nanocrystalline were determined by XRD and SEM, respectively. The average particle size was estimated about 17 nm for calcined powders. Surface area for zinc ferrite synthesized by this method is 36 m^2/g.

Keywords: *Sol–gel auto-combustion, Nanocrystalline, $ZnFe_2O_4$, X-ray diffraction, Scanning electron microscopy*

1. Introduction

Nanosize particles exhibit unique chemical and physical properties [1,2]. In particular nanocomposite materials composed of nanometric metal and metal oxide particles embedded in vitreous matrices reveal a variety of interesting magnetic, electric and catalytic properties [3–5]. Different techniques have been suggested for the preparation of nanophase materials namely vapour deposition [6], ball milling technique [7], reversed micelles [8], Langmuir–Blodgett film [9] and self-assembled monolayers [10]. In addition, the sol–gel process has been used for the preparation of nanocomposite materials [11,12]. Zinc ferrite is not only interesting in basic researches in magnetism, but also has great potential in technological application.

* Corresponding author: Golrokh mahmoudzadeh
Chemistry Department, North Tehran Branch,
Islamic Azad University, Tehran, Iran
Tel +98 914 3434088, Fax +98 21 22949657
Email Gmahmoudzadeh@yahoo.com

Spinel ferrites possessing the cubic structure are described by the formula (A) [B] $_2O_4$, where (A) and [B] indicate tetrahedral and octahedral cation sites, respectively, in a FCC anion (oxygen) sublattice. Bulk $ZnFe_2O_4$ has a normal spinel structure with Zn^{2+} ions in the A-site and Fe^{3+} ions in the B-sites [13]. The sol–gel auto-combustion method is used here has many simple processing steps. It has the advantages such as using inexpensive precursors and reducing in consumption of external energy by usage of auto-combustion process. The synthesis also results in the formation of nano-sized, homogeneous and highly reactive powder [14]. Sol–gel combustion process offers a molecular-level mixing of the precursors, which is capable of improving chemical homogeneity of the resulting powders to a significant extent. However, since this method makes use of the exothermicity of redox reaction, agglomeration commonly exists in the ceramic powders synthesized by sol–gel combustion method. Agglomeration is commonly divided into soft agglomeration and hard agglomeration according to the strength of attractive force between particles [15]. Several preparation conditions such as dilution, fuel/oxidant ratio, pH and temperature can have an impact on the formation of the ferrites and their properties [16].

2. Materials and methods

2.1. Sample preparation

Zinc ferrite powders with compositions of $ZnFe_2O_4$ were synthesized by sol–gel combustion method. The detailed process can be described as follows .The analytical grade $Fe(NO_3)_3{\cdot}9H_2O$, $Zn(NO_3)_2{\cdot}4H_2O$ and Thiourea (CS $(NH_2)_2$ were used as raw materials. The appropriate amount of nitrates and thiourea as fuel was first dissolved into deionized water to form a mixed solution. The molar ratio of nitrates to thiourea is 1:2. The pH value of solution was adjusted to about 9.5 using ammonia. Then, the mixed solution was poured into a dish and heated at 70-100 $^{\circ}$C under constant stirring to transform into a dried gel. The removal of water from the dried gel is a major problem in the sol–gel synthesis. Being ignited, the dried gel burnt in a self-propagating combustion way to form loose powder. This process was continued for 3 to 4 hours, after which the transparent solution turned to viscous brown gel followed by foaming of the gel. The foamy gel was kept on a pre-heated oven at 350°C which caused its spontaneous ignition. The combustion reaction was completed within a few seconds and loose powder was formed. This was crushed and ground thoroughly. The puffy, porous brown powders as-combusted was calcined at the temperature of 750–1000°C for 4 h with a heating rate of 10 $^{\circ}$C/min.

2.2. Characterization

The phase identification of the burnt powder of $ZnFe_2O_4$ is done using XRD Philips Model: XPERT- MPD). The structural morphology is investigated using SEM (Philips Model: XL30).

3. Results and Discussion

3.1. Structural and morphological analysis

Figure 1 shows the XRD pattern of calcined powder, which was synthesized by sol-gel auto-combustion technique. Structural characterization was performed by X-ray diffractometer (XRD) having Cu K*a* radiation. All the as-burnt powders are single-phase ferrites with spinel

structure. To get more information, the nanocrystallite sizes of calcined powders were calculated from X-ray peak broadening of the diffraction peak (227) using the Scherrer formula:

$$D = k\lambda / \beta \cos\theta$$

Where D is the crystallite size, λ is the wavelength of X-ray radiation (0.15405 nm for Cu Kα), θ is the Bragg angle and β is the full width at half maximum (FWHM) of the most intense diffraction peak (227) and k is an instrumental constant. The average particle size is from 17 nm for powders calcined at the temperature of 750–1000°C for 4 h.

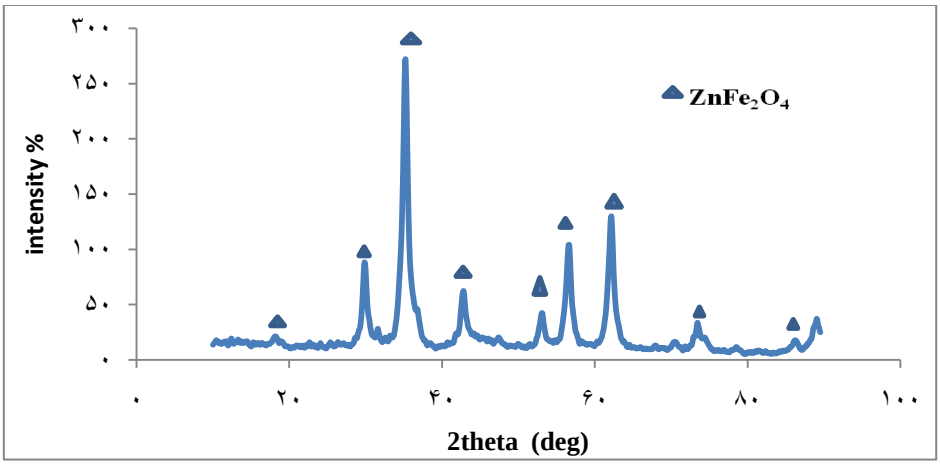

Fig.1. XRD pattern for zinc ferrite synthesized by sol–gel auto-combustion.

The SEM photograph for ferrites prepared with thiourea fuel is shown in Figure 2. The morphology of the oxides is mostly spherical with some agglomeration. Surface area for zinc ferrite synthesized with this method is 36 m^2/g.

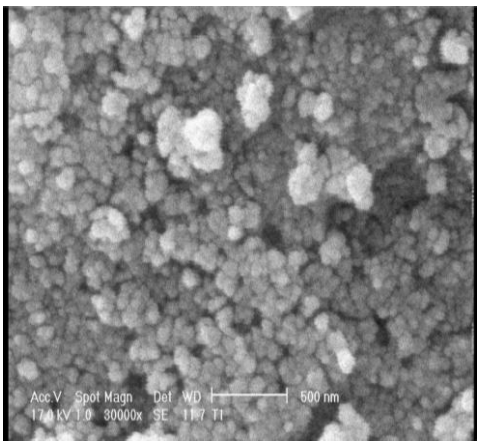

Fig.2. SEM photograph of the as-burnt $ZnFe_2O_4$ powder.

4. Conclusion

Nanosized $ZnFe_2O_4$ powders with compositions of $ZnFe_2O_4$ were synthesized by sol–gel combustion method. Sol–gel auto-combustion is a unique combination of the combustion and the

chemical gelation processes. The average particle size is from 17 nm for powders calcined at 750–1000°C. The observed results revealed that single-phase $ZnFe_2O_4$ nanoparticles with surface area 36m^2/g could be obtained when the dried gel is calcinated.

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