Journal of Saudi Chemical Society (2016) 20, S517–S522

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Journal of Saudi Chemical Society

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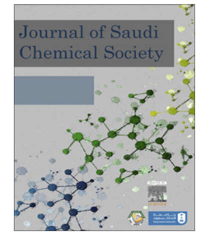

ORIGINAL ARTICLE

Effect of preparation conditions on Nickel Zinc Ferrite nanoparticles: A comparison between sol–gel auto combustion and co-precipitation methods

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Received 18 April 2012; accepted 23 March 2013
Available online 3 April 2013

KEYWORDS

Nano ferrites;
Sol–gel technique;
Co-precipitation;
Grain size;
Single phase

Abstract The experimental conditions used in the preparation of nano crystalline mixed ferrite materials play an important role in the particle size of the product. In the present work a comparison is made on sol–gel auto combustion methods and co-precipitation methods by preparing Nickel Zinc Ferrite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$) nano particles. The prepared ferrite samples were calcined at different temperatures and characterized by using standard methods. X-ray diffraction analysis indicated the formation of single phase ferrite nanoparticles for samples calcined at 500 °C. The lattice parameter range of 8.32–8.49 Å confirmed the cubic spinel structure. Average crystallite size estimated from X-ray diffractogram was found to be between 17 and 40 nm. The IR spectra showed two main absorption bands, the high frequency band v_1 around 600 cm^{-1} and the low frequency band v_2 around 400 cm^{-1} arising from tetrahedral (A) and octahedral (B) interstitial sites in the spinel lattice. TEM pictures showed particles in the nanometric range confirming the XRD data. The studies revealed that the sol–gel auto combustion method was superior to the co-precipitation method for producing single phase nano particles with smaller crystallite size.

1. Introduction

Nanoscale materials promise to be important in the development of various current and future specialized applications. Mixed metal-oxide nanoparticles have been the subject of much interest because of their unusual optical, electric and magnetic properties (Brain et al., 2007; Date et al., 2004; Monica et al., 2007). Ferrites are ceramic ferro magnetic materials with general chemical formula MFe_2O_4, where M represents metallic cations like Fe, Mn, Ni, Co, Zn, Cu, Al or a mixture of these. They crystallize into the spinel structure in which the sites occupied by the cations are of two types, tetrahedral and octahedral sites. MFe_2O_4 type compounds with spinel structure show a variety of novel properties which vary with the nature of the cations, their charge and their site distribution among tetrahedral and octahedral sites (Zang et al.,

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Peer review under responsibility of King Saud University.

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http://dx.doi.org/10.1016/j.jscs.2013.03.003
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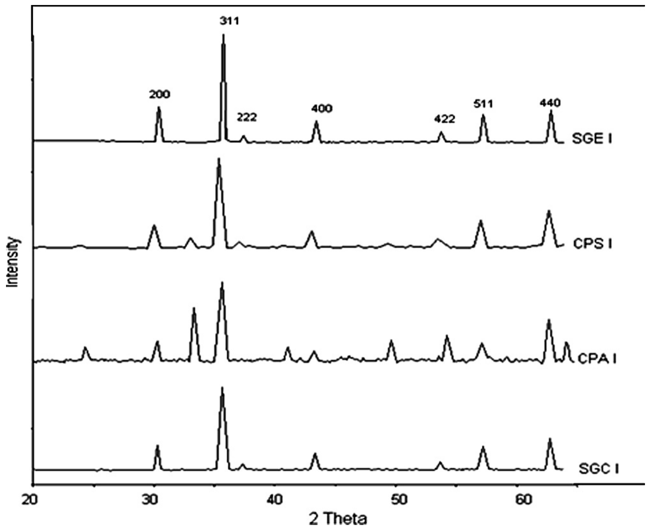

Figure 1 XRD of NZF samples at 550 °C.

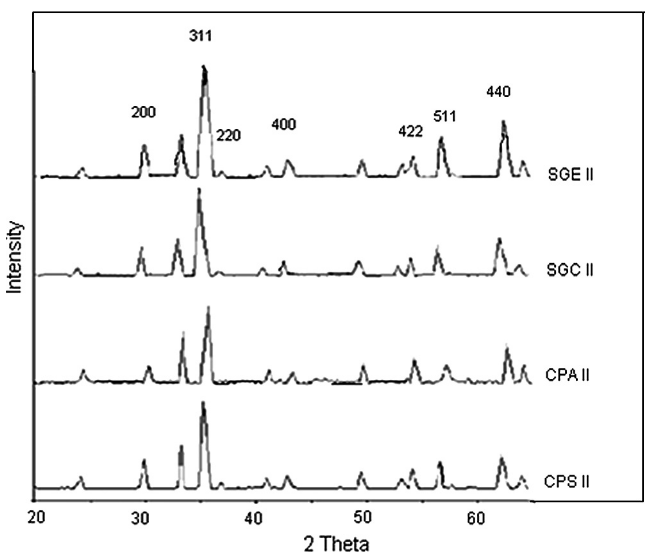

Figure 2 XRD of NZF samples at 800 °C.

2010). The type of cations and their distribution between two interstitial sites in spinel ferrites can be tuned resulting in many interesting properties. It is known that magnetic ferrite nanoparticles are also used extensively for a broad range of applications, such as catalytic applications, drug delivery, cell labeling and sorting, magnetic resonance imaging and sensing as well as therapeutic applications (Wang etal., 2009; Kunduand Mishra, 2008; Costa et al., 2008; Ruiting et al., 2008). The small dimensions of materials show novel chemical, physical and biological properties together with unique physical phenomena and processes. The size reduction of a magnetic material leads to novel properties like superparamagnetism as compared to bulk material due to small volume (Alexandre et al., 2007). So it can find many applications in bio medical fields. Zinc substitution has a remarkable influence in the magnetic properties of a nanoscale system (Ghane et al., 2010; Sathishkumar et al., 2011; Concas et al., 2009). Recently more sophisticated uses of nanoscale materials have been realized.

The conventional methods for the preparation of ferrites have certain limitations such as long heating schedule at high temperatures, higher grain size, higher time consumption etc. The experimental conditions used in the preparation of these materials play an important role in the properties and the particle size of the ferrite nano particles produced. For this reason, a great variety of experimental methods have been used in the production of nano particles, like the sol–gel techniques (Gatelyte et al., 2011; Khorrami et al., 2011; Slama et al., 2009), co-precipitation method (Joy et al., 2004; Arulmurugan et al., 2005; Kim et al., 2001), microwave sintering method (Roy et al., 2004), hydrothermal method (Wang and Kung, 2004), spray-spin-heating-coating method (Liu et al., 2009), refluxing method, pulsed wire discharge method and auto combustion method (Zahi, 2010). Of these, co-precipitation and sol–gel methods are promising techniques for preparing nano ferrites in bulk scale due to the production of homogeneous particles. The Co-precipitation method with constant pH has been widely used because of the high crystallinity, homogeneity and good textural properties of the materials produced. The sol–gel technique has become very popular recently for the preparation of a variety of mixed-metal oxides, nanomaterials and nanoporous oxides due to the high chemical homogeneity, low processing temperatures and the possibility of controlling the size and morphology of particles. It has been demonstrated that the sol–gel process offers considerable advantages such as better mixing of the starting materials and excellent chemical homogeneity in the final product (Veith et al., 2005).

The Ni–Zn ferrite is a spinel with crystalline structure belonging to the cubic system and is generally represented by the formula $Ni_{1-x}Zn_xFe_2O_4$. Spinel ferrites with different Ni–Zn compositions are of great interest due to their potential applications in microelectronics, magneto-optics and as a microwave device component (Bhattacharjee et al., 2011; Thakur et al., 2009; Shobana and Sankar, 2009). It is a known fact that particle size and morphology of the nano particles may be controlled by the reaction conditions. In the present study Nickel Zinc Ferrite (NZF) nano particles are prepared through two co-precipitation methods as well as two sol–gel auto combustion methods and a comparison is made on these methods by studying the structural properties of NZF nanoparticles. A comparison is also made with respect to the annealing temperature of the nanoparticles. The obtained nanoparticles are characterized by different standard techniques such as X-ray diffraction, FTIR spectroscopy and transmission electron microscopy.

2. Experimental

2.1. Synthesis of nanoparticles

The mixed metal oxide system $Ni_{0.5}Zn_{0.5}Fe_2O_4$ was prepared by two co-precipitation methods as well as two sol–gel auto combustion methods. In the first co-precipitation method, calculated amounts of $Fe(NO_3)_3{\cdot}9H_2O$, $Zn(NO_3)_2{\cdot}6H_2O$ and $Ni(NO_3)_2{\cdot}6H_2O$ were weighed accurately and dissolved in doubly distilled water. The precipitation was carried out at a controlled pH of 9.5 using 1:1 NH_3 solution. It was then heated at 95 °C with constant stirring for 4 h followed by oxidation using 30% H_2O_2. The precipitated ferrite was washed well,