

Research Article

Morphological of Zinc Ferrite Nanostructure Using the Hydrothermal Synthesis

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Abstract. Over the last two decades, many methods for synthesizing various ZnFe_2O_4 nanostructures of uniform size and shape, such as nanospheres, nanoflowers, nanorods, and nanotubes, have been developed. It seems challenging to investigate a practical and cost-effective method to synthesize cubic ZnFe_2O_4 magnetite with cubic morphological properties in an aqueous solution. The morphology of zinc ferrite nanostructures is mostly random and non-uniform. The objective of this study is to create reasonably uniform single-crystal zinc ferrite nanocubes. The hydrothermal method was used to successfully sample zinc ferrite by varying the amount of (En) ethylenediamine (0 - 2.4 mL) in the solution. The outcomes demonstrated that the nanocubes, with a particle size of nearly 48 nm, had a more uniform morphology than other samples. The morphology and dimensions of the samples were examined using scanning electron microscopy in conjunction with energy dispersion X-ray spectroscopy.

Keywords: *Zinc Ferrite, ZnFe_2O_4 , Nanocubes Morphology, Hydrothermal.*

A. INTRODUCTION

There are numerous ways to obtain ferrite, which can be found in three different crystal systems. The ability to prepare virtually unlimited quantities of solid solutions opens up the possibility of modifying ferrite's properties for a wide range of uses (Sugimoto et al., 1999). New and exciting research areas have been created as a result of the potential to create ferrite in the form of nanoparticles, with revolutionary implications for biotechnology and electronics. The historical advancement of ferrite science and technology since roughly 1940 has been extensively documented (Valenzuela et al., 2012). After the 1950s, the significance of ferrite came to the attention of the general public (Wijn, 1970).

The shape-controlled synthesis of spinel ferrites has drawn a lot of interest because the morphology of these nanocrystalline spinel ferrites affects their magnetic properties. Numerous techniques for creating distinct ZnFe_2O_4 nanostructures of uniform size and shape have been developed over the past 20 years, including those for creating nanospheres (Zhu et al., 2008), nanoflowers (Deng et al. 2019), nanorods (Ly et al., 2010), and nanotubes (Moriya et al., 2019). Thermal decomposition of suitable organometallic compounds has recently yielded magnetic nanocubes; however, this method is toxic and energy-intensive (Wang et al., 2010). Uniform ZnFe_2O_4 nanocubes in solution, however, have yet to be reported. It appears difficult to investigate a cost-effective strategy for synthesizing ZnFe_2O_4 magnetite with cubic morphology in solution.

Numerous studies have shown that nanotechnology presents a great means of producing high-quality treated nanoparticles without harmful effects on people (Ghasemi et al., 2022; Bora et al., 2014; ghrke et al., 2015). However, nanostructured magnetic materials that are sufficiently large and have a high surface-to-volume ratio are attracting more attention than their rivals and are being taken into consideration for their potential wide range of applications (Taha et al., 2019). Currently, magnetic metal oxide and its compounds as spinel structure

(AB_2O_4) are effective electro-catalysts for wastewater and sewage treatment (Zie et al., 2017; El Nahrawy et al., 2019). These nanomaterials are employed in a number of fields, including gas sensors, catalysis, Opto-magnetic devices, electromagnetics, pigments, electrochemical devices, converters, drug delivery, and photovoltaics (Ikawa et al., 2017). ZFO nanoparticles with various nanostructured morphologies, including rod-like, flower-shaped, and spherical, were synthesized to better understand the critical role of nanostructure morphology in determining performance (Li et al., 2012).

One of the recent research topics that has gained traction is the preparation of nanostructures based on spinel ferrite. This is due to the strong tendency of ferrite nanoparticles to aggregate, making it difficult to exploit their unique physical properties. The material preparation methodology is widely acknowledged to be related to morphology, which further determines the physical properties of the material of interest. The morphology of zinc ferrite nanostructures is generally random and non-uniform. The purpose of this research is to create relatively uniform single-crystal zinc ferrite nanocubes.

B. METHOD

Sinopharm Chemical Reagent Co. provided zinc acetate dihydrate $Zn(CH_3COO)_2 \cdot 2H_2O$, J&K Chemical Reagent Co. supplied iron(II) chloride tetrahydrate ($FeCl_2 \cdot 4H_2O$), Sigma-Aldrich offered ethylenediamine ($C_2H_8N_2$), zinc chloride tetrahydrate $ZnCl_2 \cdot 4H_2O$, zinc nitrate hexahydrate $Zn(NO_3)_2 \cdot 6H_2O$, Sodium Hydroxide (NaOH) and nonahydrate Iron(III) nitrate $Fe(NO_3)_3 \cdot 9H_2O$ from Sinopharm. All chemicals are of analytical grade and are used without further purification.

The hydrothermal method is used to produce zinc ferrite nanoparticles. As precursors, iron (II) chloride tetrahydrate [$FeCl_2 \cdot 4H_2O$] and zinc acetate dihydrate [$Zn(CH_3COO)_2 \cdot 2H_2O$] were used. First, a mixture of 17 mL 0.1 M $Zn(CH_3COO)_2 \cdot 2H_2O$ and 33 mL 0.1 M $FeCl_2 \cdot 4H_2O$ was dissolved in the deionized water one by one for 2 hours at room temperature with constant stirring. The solution was placed in a 100 mL Teflon beaker with constant stirring, and then (En) ethylenediamine (0-2.4 mL) and NaOH were added, followed by stirring for the remainder of the time. thirty minutes the homogeneous solution was sealed in a Teflon-coated stainless-steel autoclave and kept at 180 C for 24 hours. After synthesis, the product was filtered and washed several times with distilled water and ethanol before drying in air at 120°C for 2 hours. Figure 1 displays the flowchart for the fabrication process.

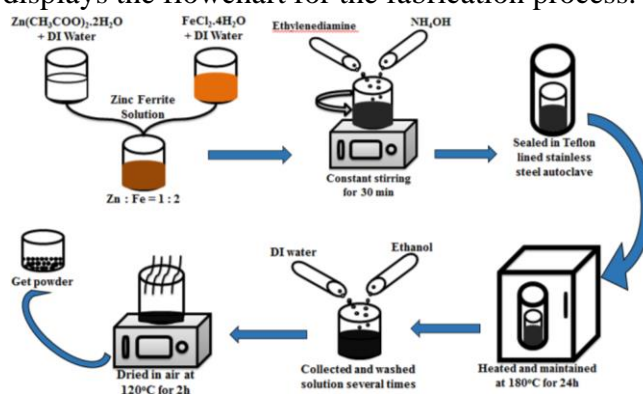


Figure 1. Flowchart of the hydrothermal method for zinc ferrite.

Furthermore, the material characterization utilizes Scanning Electron Microscopy (SEM) to provide a highly magnified image of the surface of a material that strongly resembles what one would expect if one could actually "see" the surface visually. SEM resolutions can be as low as a few nm, and magnifications can be easily adjusted from 10x to 300,000x. Dimensions and morphology were investigated using scanning electron microscopy with energy dispersion X-ray spectroscopy (FESEM, HITACHI S4800). Prior to measurements, our

samples were coated with Pt, as sample coating is required in the field of electron microscopy to enable or improve sample imaging.

C. RESULTS AND DISCUSSION

Figures 2(a)-(f) show SEM images of zinc ferrite samples prepared with En 0 - 2.4 mL. (f). The solution in the Teflon cup prepared with the same total volume was 60%. Figures 2(a)-(c) show the uniformity of shape morphology and the increase in particle size as En concentration increases until it reaches 0.8 mL. The particle size is 30.1; 38.5; and 49.6 nm for solutions prepared with 0; 0.4; and 0.8 mL En, respectively.

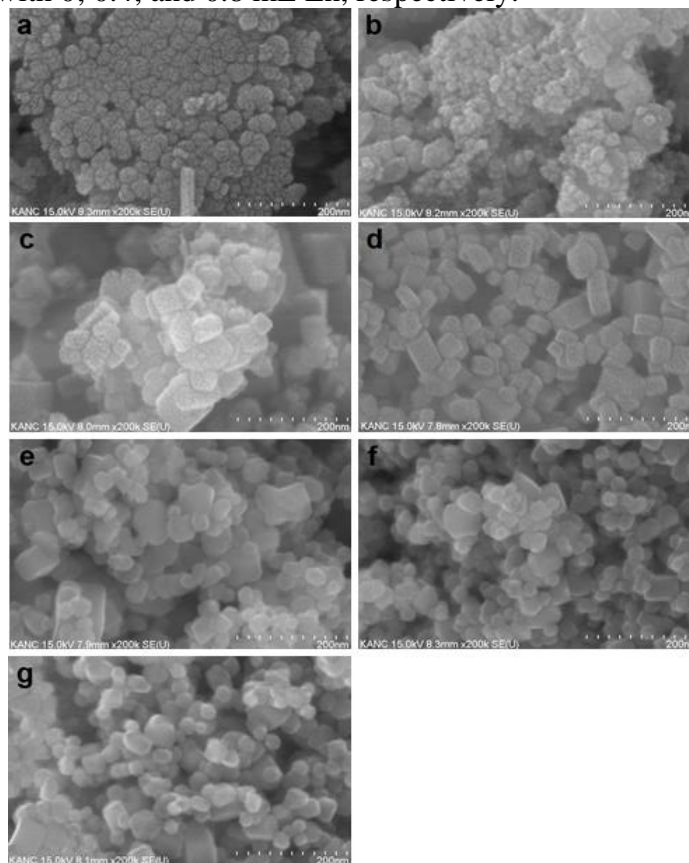


Figure 2. SEM images of zinc ferrite nanostructures prepared with En 0 - 2.4 mL samples

The particle size was then reduced by increasing En, as shown in Figure 2 (d) - (g). The particle size for 1.2 is 47.6; 44.7; 38.2; and 35.4 mL En; 1.6; 2.0, and 2.4 mL En. The samples obtained different morphological shapes based on the SEM examination, so we selected three samples for additional studies that had a more homogeneous morphology compared to the other samples.

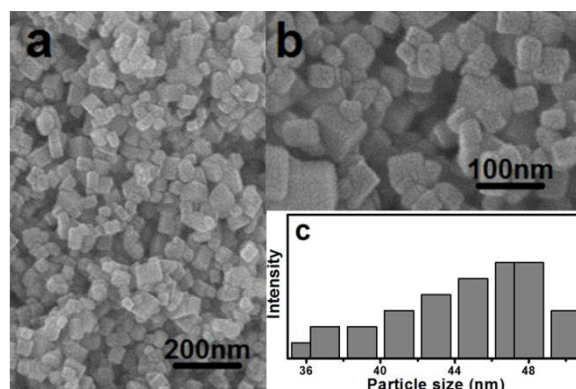


Figure 3. SEM images of zinc ferrite samples prepared with 1.2 mL of En and nanocubes

Once the amount of En is 1.2 mL, the morphology shifts to cubic, as illustrated in figure 3, with various magnifications to demonstrate more clearly the transition in morphology, when the magnification is 100K, as shown in figure 3 (a), the morphology is demonstrated to be uniform cubic, then magnification increases at 200K, almost all cubes are shaped with different sizes, as shown in figure 3. (b).

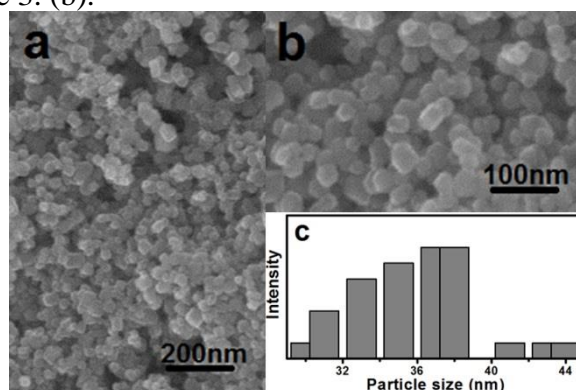


Figure 4. SEM images of zinc ferrite samples prepared in 2.0 mL En by combining nanocubes and nanospheres.

SEM investigations not only demonstrated a cubic form, however, the addition of 2.0 mL of En disclosed a morphological mix, as shown in Figure 4; throughout this case, the particles do not prove a uniform shape; some particles persist cubic in shape, but some of them changed to develop a more spherical, as seen in figure 4(a) with the magnification of 100K.

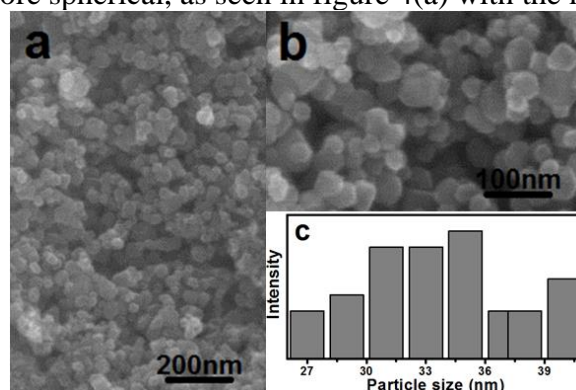


Figure 5. SEM results for zinc ferrite samples prepared by 2.4 mL En with nanospheres.

After adding about 2.4 mL of En, our sample also experienced morphological alterations to become spherical. This phenomenon begins with the form changing from the edge of the cubic surface to become not sharp and shaped like a sphere, so we call these sample

nanospheres. Figure 5(a) does not show a perfectly spherical shape but also is not cubic, so this morphology is similar to the shape of a dodecahedron. The particle size in this sample is the smallest of the other samples.

Table 1. Description of zinc-ferrite samples, and experimental values obtained from analysis of SEM images

| Sample | Particle size (nm) |
|------------------------------------|--------------------|
| Nanocubes | 47.6 |
| Mixing (nanocubes and nanospheres) | 38.2 |
| Nanospheres | 35.4 |

Table 1 displays the change in particle size and morphology in the sample caused by an increase in En during the synthesis process. The particle size in nanocubes was discovered to be the largest compared to the others. If this issue is looked into further, it can also affect the material's magnetic, optical, and electromagnetic properties.

D. CONCLUSION

The hydrothermal process for the synthesis of zinc ferrite nanostructures with various morphological forms was successfully demonstrated in this study. A uniform size distribution of nanocubes obtained from SEM observations revealed morphological differences. Ethylenediamine was increased to 1.2 mL, and as it was increased further, the zinc ferrite's morphology changed to become spherical. The uniformity of the shape of the nano cube causes the particle size to increase. The smallest particle size, on the other hand, is obtained when the morphology is slightly rounded. The magnetic, optical, and electromagnetic properties of the material may change as a result, if we can look into this further.

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