RESEARCH PAPER

Microwave Synthesis of Different Morphologies of Lead Ferrite Nanostructures and Investigation of Magnetic Properties

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ARTICLE INFO

Article History:
Received 14 October 2016
Accepted 29 November 2016
Published 01 January 2017

Keywords:
Lead Ferrite
Microwave
Nanocomposites

ABSTRACT

The lead ferrite ($PbFe_{12}O_{19}$) nanoparticles were prepared by a simple and short time microwave method. Lead nitrate, iron nitrate nine hydrate, surfactants, and ethylene glycol were used as precursor materials. The effect of surfactants on the morphology and particle size of the magnetic products was investigated. The prepared magnetic products were studied by X-ray diffraction, scanning electron microscopy, and Fourier transform infrared spectroscopy. Single phase hexagonal ferrite nanoparticles with average particle size of 50 nm were obtained in synthesize temperature of 850°C. Alternating gradient force magnetometer approves magnetic property of the hexaferrite nanostructures. The values of both saturation magnetization and coercivity strongly depend on the particle sizes. The obtained hexagonal ferrite nanoparticles exhibit a hard magnetic feature with a suitable saturation magnetization.

How to cite this article
Nabiyouni G, Halakouie H, Ghanbari D. Microwave synthesis of different morphologies of lead ferrite nanostructures and investigation of magnetic properties. J Nanostruct, 2017; 7(1):77-81. DOI: 10.22052/jns.2017.01.009

INTRODUCTION

Hexagonal ferrites $MFe_{12}O_{19}$ (M = Ba, Sr, and Pb) are well known as permanent magnetic materials. They have magnetoplumbite structure and exhibit large spontaneous magnetization with strong anisotropy along the c-axis [1]. Moreover, hexagonal ferrites are technologically very useful materials due to their application in permanent magnets, high-density magnetic recording media, and microwave devices [2–4]. The conventional solid mixture for the preparation of hexagonal ferrite powders involves a high temperature, resulting in the loss of the fine particle nature. Several solution techniques including co-precipitation [5], sol–gel [6], hydrothermal [7], and ultrasonic spray pyrolysis [8] are used to prepare magnetic nanoparticles.

The research in magnetic oxides driven by the prospective applications of these materials and on the novel properties that they possess as nanocrystalline particles, powders and thin films has received a lot of interests [9–11]. In particular, Pb–M hexaferrite ($PbFe_{12}O_{19}$) crystallizes at temperatures lower than which happen for Ba and Sr–M hexaferrites. Thus makes it interesting for magnetic recording media [12–15]. The preparation of lead hexaferrite, as bulk material, presents some problems due to lead evaporation during thermal treatment and sintering process [15,16].

In this work we synthesis nanoparticles by microwave method. Microwave systems provide the opportunity to complete reactions in minutes, and have manifold applications in academic and industrial environments alike. There are some important parameters in microwave synthesis such as choosing solvent, kind of reactions, of heating time and microwave power which can

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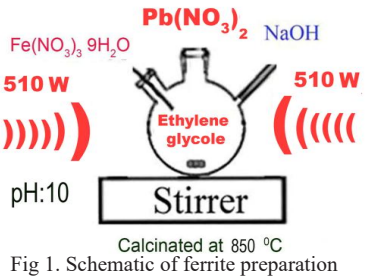

Fig 1. Schematic of ferrite preparation

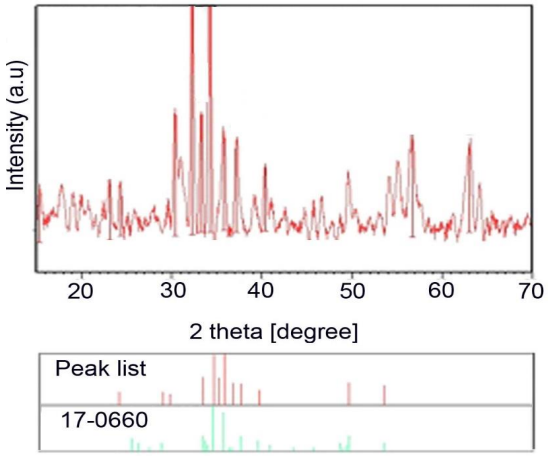

Fig. 2. XRD pattern of the $PbFe_{12}O_{19}$ nanoparticles

affect on synthesized products.

Because of uniform heat distribution, ultra heating, choice of heating or selectivity, the reaction rate increases and subsequently reduce the time and energy required for synthesis of different nanomaterials. Thus many products with various structures have been synthesized by this method [17-20].

MATERIALS AND METHODS

Materials and Instruments

$Pb(NO_3)_2$, $Fe(NO_3)_3$ $9H_2O$, NaOH, NH_3 32%, ethylene glycol and acetone were purchased from Merck and all the chemicals were used as received without further purifications. Room temperature magnetic properties were investigated using a vibrating sample magnetometer (VSM) device, made by Meghnatis Daghigh Kavir Company (Iran) in an applied magnetic field sweeping between ±10000 Oe. XRD patterns were recorded by a Philips, X-ray diffractometer using Ni-filtered CuK_α radiation. SEM images were obtained using a LEO instrument model 1455VP. Prior to taking images, the samples were coated by a very thin layer of Pt

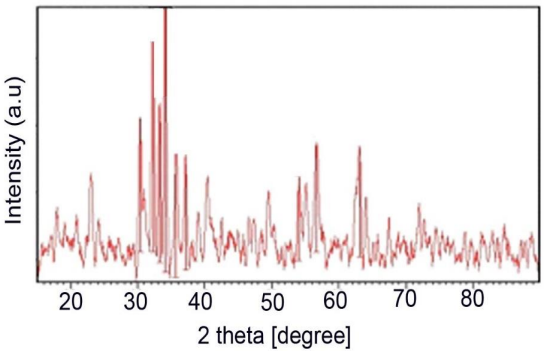

Fig 3. XRD pattern of the $PbFe_{12}O_{19}$ by SDS

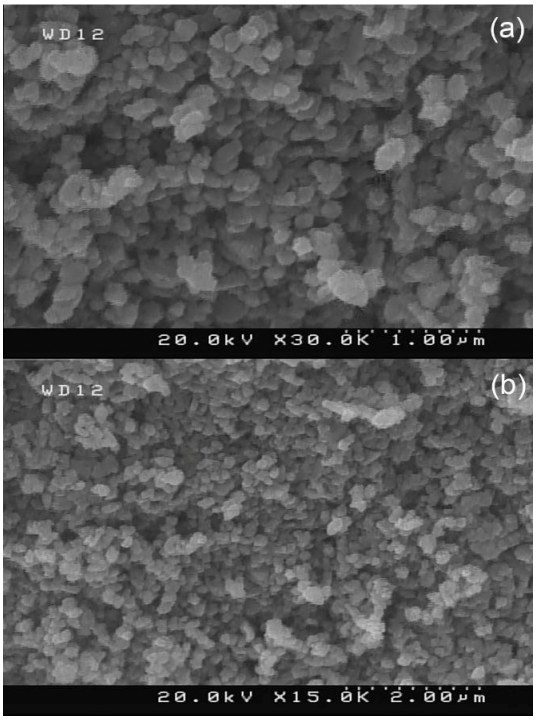

Fig. 4. SEM images the lead ferrite nanoparticles synthesized by SDS.

(using a BAL-TEC SCD 005 sputter coater) to make the sample surface conductor and prevent charge accumulation, and obtaining a better contrast.

Synthesis of $PbFe_{12}O_{19}$ nanoparticles

0.012 mole of $Fe(NO_3)_3$ $9H_2O$ and 0.001 mole of $Pb(NO_3)_2$ and surfactant (SDS,C-TAB and citric acid) were dissolved in 100 ml of ethylene glycol and put under microwave with 510 W. 16 ml of NaOH solution (1M) was then slowly added to the solution until reaching pH to around 10. A brown precipitate was then centrifuged and rinsed with distilled water. Finally the obtained precipitate was calcinated at 850 °C and its colour goes

from brown to black. Fig. 1 shows the schematic diagram for experimental setup for nanoparticles preparation used in the microwave procedure.

RESULTS AND DISCUSSION

The XRD pattern of $PbFe_{12}O_{19}$ nanoparticles is shown in Fig. 2. The pattern consists with the typical diffraction pattern of pure hexagonal phase (JCPDS No.: 17-0660) with P63-mmc space group.

The crystallite size measurements were also carried out using the XRD data and using Scherrer equation:

$$D_c = K\lambda/\beta Cos\theta$$

where β is the width of the observed diffraction peak at its half maximum intensity (FWHM), K is the shape factor, which takes a value of about 0.9, and λ is the X-ray wavelength (CuK_α radiation, equals to 0.154 nm). The average crystallite size was found to be about 18 nm.

The XRD pattern of $PbFe_{12}O_{19}$ nanoparticles synthesized by SDS surfactant is illustrated in Fig. 3. The pattern reveals the typical diffraction patterns of pure hexagonal phase (JCPDS No.: 17-0660) with P63-mmc space group and consists with pure lead hexa ferrite.

SEM images of $PbFe_{12}O_{19}$ nanoparticles prepared using SDS surfactant are illustrated in Fig. 4. In this condition, mono-disperse product with mediocre size of around 80 nm was synthesized.

Fig. 5 shows TEM image of lead ferrite nanoparticles synthesized by SDS.

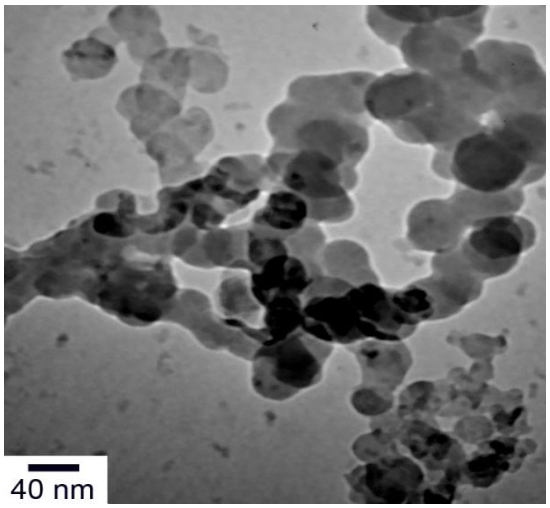

Fig. 5. TEM image the lead ferrite nanoparticles synthesized by SDS

Fig. 6 illustrates SEM images of $PbFe_{12}O_{19}$ nanoparticles synthesized by CTAB surfactant. It is observed that nanoparticles are homogenous and rod-like nanostructures were formed.

Fig. 7 shows SEM images of $PbFe_{12}O_{19}$ nanoparticles synthesized using citric acid. It seems homogenous hexagonal plate-like nanoparticles were synthesized. The average particle size is found to be around 60 nm, showing that, particle size, morphology and magnetic property of samples can easily be controlled by using different surfactants.

Hysteresis loop for PbFe magnetite nanoparticles prepared with microwave method is shown in Fig.8. Interestingly from Fig. 9, it was observed that surfactant free sample has coercivity of 800 Oe, which is much less than which for product prepared by SDS (about 3000 Oe). The outcomes indicate the direct effect of morphology and particle size on the magnetic property of the prepared ferrite [21-24]. Saturation magnetization of this sample is also lower than ferrite which prepared with SDS and it is around 26.7 emu/g. Fig.9 shows that Saturation magnetization for lead ferrite nanoparticles synthesised by SDS is around 43 emu/g.

Fourier transform infrared (FT-IR) spectrum of synthesized nanoparticles was recorded in the

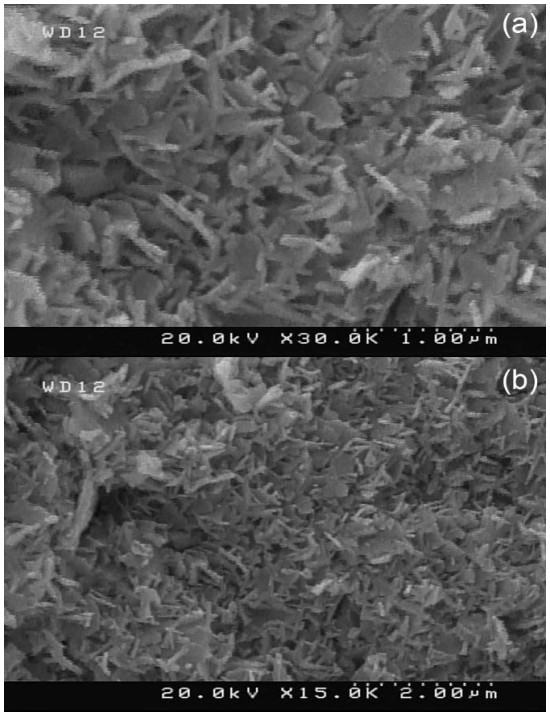

Fig 6. SEM images of $PbFe_{12}O_{19}$ nanoparticles obtained by CTAB

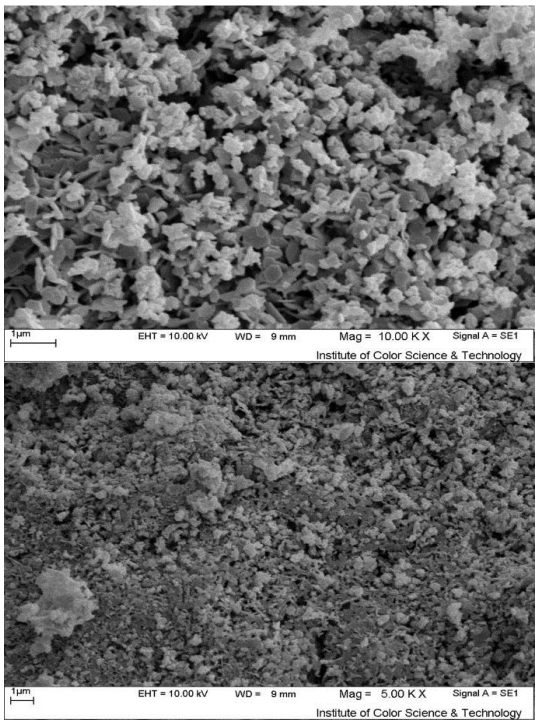

Fig. 7. SEM images of $PbFe_{12}O_{19}$ nanoparticles with citric acid

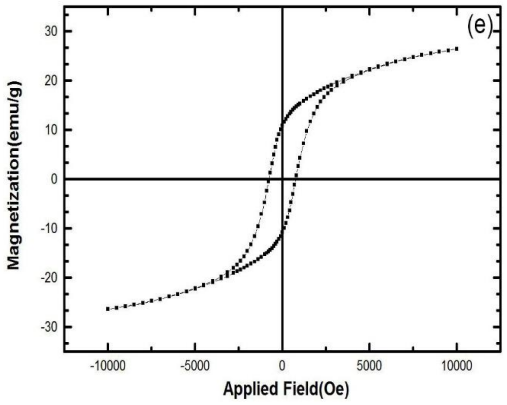

Fig. 8. AGFM loop of $PbFe_{12}O_{19}$ calcinated at 850 °C

range of 400–4000 cm^{-1} and result is shown in Fig. 10. Absorption peaks around 400 to 600 cm^{-1} are related to metal-oxygen Fe-O and Pb-O bonds. The spectrum exhibits broad absorption peaks between 3500-3600 cm^{-1}, corresponding to the stretching mode of O-H group of hydroxyl group that are adsorbed on the surface of nanoparticles.

CONCLUSION

Synthesis and characterization of $PbFe_{12}O_{19}$ nanoparticles were reported. Effect of precursor

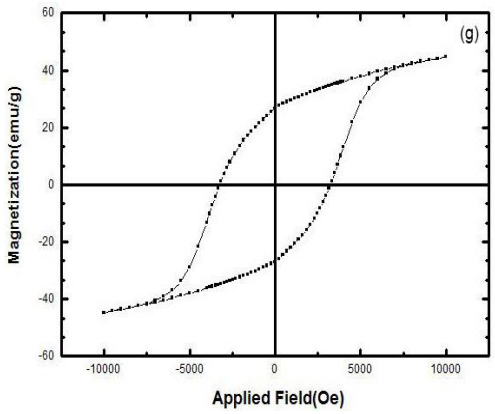

Fig. 9. AGFM loop of $PbFe_{12}O_{19}$ by addition of SDS

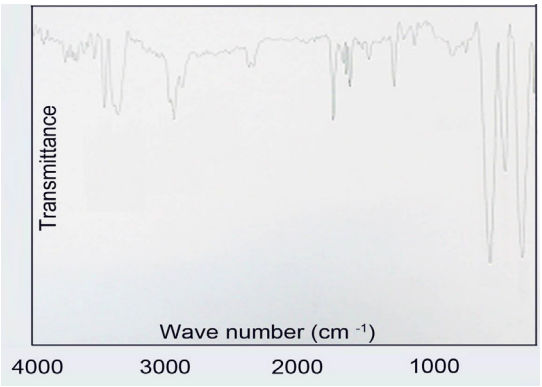

Fig. 10. FT-IR spectrum of $PbFe_{12}O_{19}$ nanoparticle

and surfactant on the morphology and particle size of the products was investigated. SEM images show that it is possible to change formation and nanoparticle size by adding surfactants. AGFM confirmed that nanoparticles exhibit ferromagnetic behaviour. However, by adding of surfactant, coercivity of magnetic nanoparticles was increased. The results also show that microwave method is a suitable approach for preparation of lead ferrite as promising candidate for industrial applications.

ACKNOWLEDGEMENT

This work has been supported financially by Arak University Research Council (AURC) under the Grant Number of 94-2113.

CONFLICT OF INTERESTS

The authors declare that there is no conflict of interests regarding the publication of this paper.

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