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Synthesis and characterization of $BaFe_{12}O_{19}/MgFe_2O_4$ nanocomposite powders

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ABSTRACT

The composite powders of barium-magnesium ferrite were prepared using sol-gel auto-combustion and microwave methods. The crystal structure and the shape of nanocomposites have been compared. X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Vibrating Sample Magnetometer (VSM) were used to investigate the structural, morphological and magnetic properties of nanopowder. The formation of pure crystallized nanocomposites occurred when the precursor was heat-treated at 800°C for 2 hours. As a result, the composite powders consist of $BaFe_{12}O_{19}$ and $MgFe_2O_4$ phases. The average crystallite size of the synthesized nanoparticles which were obtained by sol-gel auto-combustion and microwave methods were 32 and 65 nm, respectively.

Keywords: *$BaFe_{12}O_{19}$; $MgFe_2O_4$; Ferrites; Sol-Gel auto-combustion method; Microwave method.*

INTRODUCTION

Ferrites are materials that have significant importance because of the large number of technological applications they find as well as because they can be used as model materials and thus provide an opportunity for better understanding of the magnetic interactions in nanoscale [1]. Ferrites are superior magnetic materials which widely used in microwave and electrical industries. They exhibit high electrical resistivity combined with useful ferromagnetic behavior. Their applications range from simple function device, such as small permanent magnets to sophisticated devices for the electronic industry. Some interesting applications of these materials are magnetic media used in computers, recording devices, and magnetic cards, among others [2-5]. One of the attractive properties of ferrites is the possibility to prepare different compositions and thereby modify the magnetic properties. Recently, one of the challenges is to improve the magnetic properties of soft ferrites such as saturation magnetization, magnetic hysteresis, demagnetizing force and anisotropic energy.

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Researchers are trying to produce hard and soft ferrites by using simple methods. In view this, many studies have focused on new systems, such as $CoFe_2O_4/ZnFe_2O_4$ [6], earth-iron-boron [7] and Fe/Z-type ferrite [8].The results suggest that coupling exchange exists between the nanoparticles and the interaction significantly influences magnetization and coercivity of the composite powders. Masala et al. [9] reported that exchange interaction between hard and soft magnetic phases improve the microwave absorption and magnetic properties of nanocomposites.

Among these materials, combining between hexagonal ferrite and spinel ferrite are intensively investigated by several scientists. Chen et al. [10] concluded that the absorption performance of $SrFe_{12}O_{19}/ZnFe_2O_4$ composite powders which were fabricated by sol-gel technique may be attributed to exchange coupling interaction between hard and soft phases. Shen et al. [11] investigated the magnetic properties of $SrFe_{12}O_{19}/Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite in their research. They have pointed to this thread that the nanocomposite magnets combining a high saturation magnetization of the soft phase and high coercivity of the hard phase will be recognized as the next generation of permanents.According to studies, we went onto investi gate the structural, morphological and magnetic properties of $BaFe_{12}O_{19}/MgFe_2O_4$ as hard/soft composite powders. $BaFe_{12}O_{19}$, is well-known for its high performance permanent magnetic and good mechanical hardness which has attracted considerable attention in recent years owing to its high coercivity, high curie temperature, relatively large magnetization, and the superior chemical stability and corrosion resistivity. $BaFe_{12}O_{19}$ has been used as microwave absorbing agent in 2–18 GHz to eliminate unwanted electromagnetic signals, which may interfere with electronically controlled systems and be harmful to health [12-15].

$MgFe_2O_4$ particles of micrometer-range size are reported to exhibit greater magnetic heating than others. Recently, magnetic hyperthermia treatment has been investigated for use in conjunction with a drug delivery system (DDS) so as to enable heating of only cancer tissue. A requirement for use with a DDS is that particle size must be in the range 10–100 nm for accumulation in cancer tissue [16-18]. $MgFe_2O_4$ has many applications in high-density recording media, heterogeneous catalysis, adsorption, sensors and magnetic technologies. $MgFe_2O_4$, is a kind of soft magnetic material with diamagnetic substitution for Mg^{2+} and Fe^{3+} ions, that has attracted the attention of a number of research workers. Since Mg^{2+} is diamagnetic, a weaker coupling between Fe^{3+} cations reduces the magnetic properties in $MgFe_2O_4$ which also yield super-paramagnetism at room temperatures, so $MgFe_2O_4$ is placed in soft magnetic materials category [19].

Herein, the composite powders were synthesized by using sol–gel auto-combustion and microwave methods. The crystal structure and phase constituents of the composite powders were also studied. Finally, the magnetic properties of product which was prepared with sol-gel auto-combustion was compared with product obtained from microwave method.

EXPERIMENTAL

The starting materials were barium nitrate, $Ba(NO_3)_2$, magnesium nitrate hexa hydrate, $Mg(NO_3)_2.6H_2O$, iron (III) nitrate nona hydrate, $Fe(NO_3)_3.9H_2O$,citric acid, $C_6H_8O_7$,and ammonia, all of analytic purity. Reactants were supplied from Merck Company and used without purification. Deionized water was used for the preparation of all the samples.

Synthesis of $BaFe_{12}O_{19}/MgFe_2O_4$ Nanocomposite by Sol-Gel Auto-Combustion Method

Appropriate amount of $Ba(NO_3)_2$, $Mg(NO_3)_2.6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ were dissolved in deionized water. Citric acid was then added into the prepared aqueous solution to chelate Ba^{2+}, $Mg^{2+}$and Fe^{3+}. The mixed solution was neutralized to pH 7 by adding ammonia and then stirred continuously at 70˚C until a viscose gel was obtained. The obtained gel was ignited to form a loose powder. Finally, powder was calcined at 800˚C for 2 hours with a heating rate of 10˚Cmin^{-1} to obtain barium hexaferrite and magnesium ferrite phases.

Synthesis of $BaFe_{12}O_{19}/MgFe_2O_4$ Nanocomposite by Microwave Method

Metal nitrates were dissolved in deionized water followed by the addition of citric acid. An ammonia solution was added to adjust the pH value to 7. The solution was slowly evaporated at 70˚C until a viscose gel was formed. The obtained gel was used as a precursor of microwave technique. In the final stage, the precursor was placed in the microwave and heated at 120˚C for 10 minutes to form a loose powder and then the product was calcined at 800˚C for 2 hours with a heating rate of 10˚C min^{-1} to obtain nanocomposite.

Characterization

The resulting powders were further characterized by the powder X-ray diffraction (XRD) technique (CuKα radiation, model STOE).The average particles size of the different phases, were determined from the line widths of the diffraction peaks using Scherrer equation:

$$d=(0.9)\lambda/\beta \cos\theta$$

Where d is the grain diameter, β is half-intensity width of the relevant diffraction, λ is X-ray wavelength and θ the diffraction angle.

A Philips XL-30 scanning electron microscope was used to characterization the morphologies and microstructure of the samples.

RESULTS AND DISCUSSION

XRD Patterns

The XRD patterns in Figure 1.a and 1.b indicates that $BaFe_{12}O_{19}/MgFe_2O_4$ nanocomposites have formed by using two methods. The diffraction peaks appeared in the XRD patterns can be indexed with the standard patterns for $BaFe_{12}O_{19}$ (JCPD 00-027-1029) and $MgFe_2O_4$ (JCPD 00-036-0398).

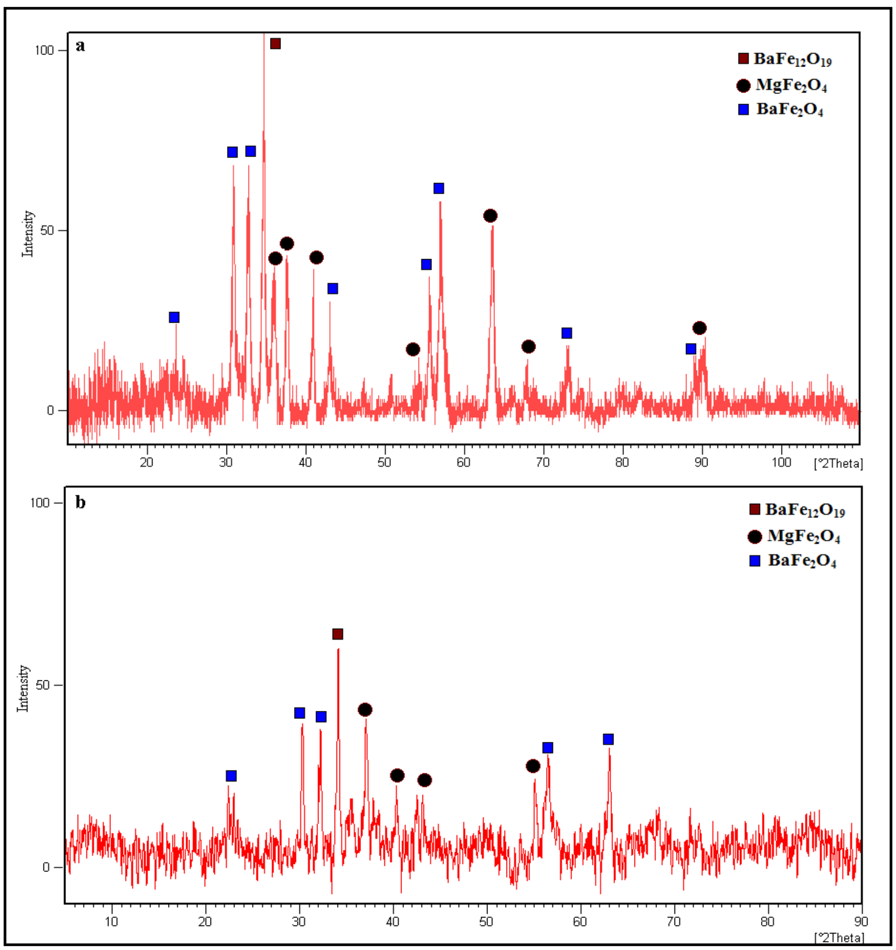

Fig. 1. X-ray diffraction patterns $BaFe_{12}O_{19}/MgFe_2O_4$ nanocomposites prepared by a) sol-gel auto-combustion method and b) microwave method.

Well-defined sharp peaks in the XRD pattern of sample which was treated by sol-gel auto-combustion method indicates the good crystalline quality and confirm the formation of nanocomposite. However Intensity of these peaks in the XRD pattern of sample which was treated by microwave route demonstrated that this approach was not appropriate as well as first technique. In both cases the X-ray patterns confirm the formation of hard-soft ferrite, but results showed that the samples included $BaFe_2O_4$ as a sub-phase. The average crystallite size for the resulting nanopowders were 32 and 65 nm for samples which were produced by sol-gel auto-combustion and microwave methods, respectively.

SEM Images

According to the electron microscopy data in Figure 2a and 2b, particles are agglomerated and exhibit non-uniform size distribution in the sample which was obtained by microwave method (Figure 2b) that was adapted with XRD calculations for this sample. While the particles synthesized by sol-gel auto-combustion method (Figure 2a) were shaped in spherical form and some extent agglomerated particles were observed. So, it can be clearly concluded from XRD patterns and SEM images that the structural and morphological properties of sample which was synthesized by sol-gel auto-combustion method was better than the sample which was treated by microwave route.

Magnetic Properties

Magnetization versus applied field for the sample which was treated by sol-gel auto-combustion route was shown in Figure 3. In this case, the magnetic properties of this sample compared with barium hexaferrite nanoparticles, $BaFe_{12}O_{19}$, which were prepared by the same method.

It is observed that the saturation magnetization and the coercivity of $BaFe_{12}O_{19}$ nanoparticles decrease from 55.98 emu/g and 5100 Oe to 34.84 emu/g and 4500 Oe for $BaFe_{12}O_{19}/MgFe_2O_4$ nanocomposites. The variations may be attributed to the interphase interactions at the surface of two phase ferrites. On the other hand, comparing the results of this study with similar studies conducted on the synthesis of $MgFe_2O_4$ nanoparticles [18, 19] shows that the magnetic properties of the pure magnesium spinel ferrite nanoparticles were improved in hard-soft nanocomposite.

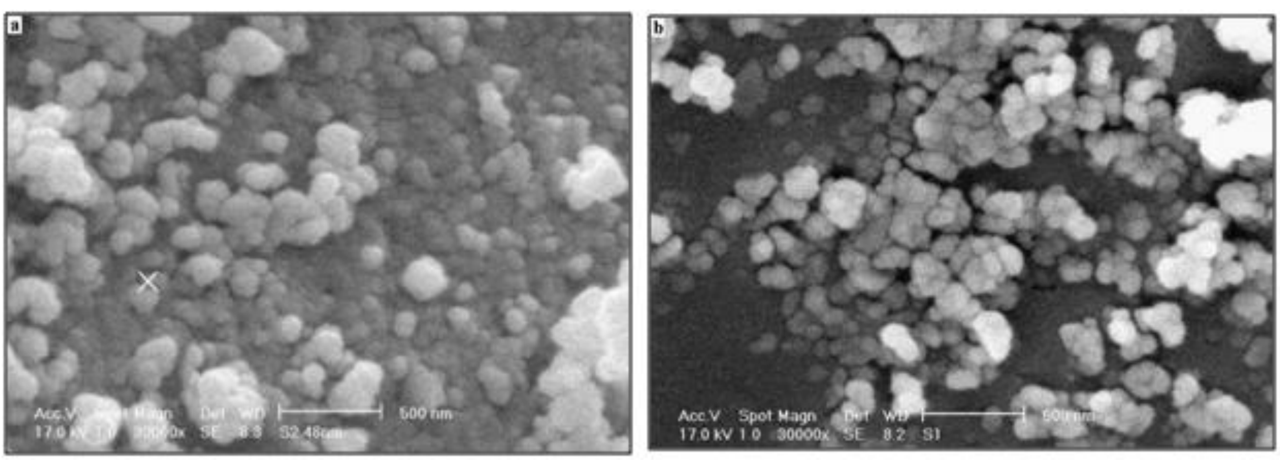

Fig. 2. SEM images of $BaFe_{12}O_{19}/MgFe_2O_4$ nanocomposites prepared by a) sol-gel auto-combustion method and b) microwave method.

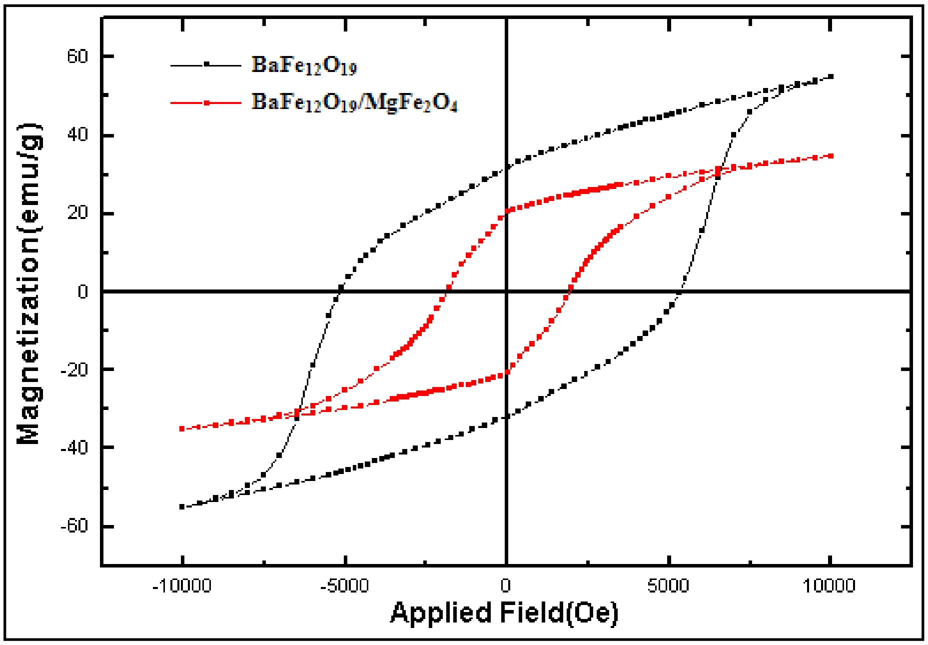

Fig. 3. Magnetization versus applied field for $BaFe_{12}O_{19}/MgFe_2O_4$ nanocomposites and $BaFe_{12}O_{19}$ nanoparticles which treated by sol-gel auto-combustion route.

CONCLUSIONS

We succeeded in synthesizing $BaFe_{12}O_{19}/MgFe_2O_4$ferrite nanocomposites by using sol-gel auto-combustion and microwave methods. XRD patterns reveal that the hexaferrite $BaFe_{12}O_{19}$ and spinel $MgFe_2O_4$ were formed. The XRD pattern of microwave treated sample has not shown sharp peaks but using both methods lead to formation of hard/soft nanocomposite. Investigation of microstructure with SEM revealed that nanoparticles were shaped in spherical form in both cases but the sample which was treated by sol-gel auto-combustion method had uniform structure. As a result, combustion considerably accelerates the formation of the nanocomposite. Results showed that the magnetic properties of $MgFe_2O_4$ nanoparticles increased. It is demonstrated that the interaction between the hexagonal and spinel ferrite could be form the nanocomposite with good magnetic saturation and coercive force. Of course, the magnetic properties of barium hexaferrite were expected to decline as hystersises loops showed.

Obviously, the change of the reaction conditions to achieve optimal values of the parameters affecting on the method of preparation such as, PH, temperature, mole ratio and solvent can be effective on the structural and morphological properties of samples. In the future we will try to synthesis $BaFe_{12}O_{19}/MgFe_2O_4$ferrite nanocomposite by using sol-gel auto-combustion and microwave methods with different weight ratios of barium ferrite to magnesium ferrite and focus on optimized conditions for preparation of nanocomposite by these techniques.

REFERENCES

[1] Silvaa, J., Britoa, W., Mohallem, N., (2004), Influence of heat treatment on cobalt ferrite ceramic powders. *Mater. Sci. Eng. B*, 112: 182–187.

[2] L. Nalbandian, A. Delimitis, V.T. Zaspalis, E.A. Deliyanni, D.N. Bakoyannakis, E.N. Peleka, (2008), Hydrothermally prepared nanocrystalline Mn–Zn ferrites: Synthesis and characterization. *Microp. Mesopor. Mat*., 114: 465–473.

[3] Venkataraju, C., Sathishkumar, G., Sivakumar,K., (2010), Effect of cation distribution on the structural and magnetic properties of nickel substituted nanosized Mn–

Zn ferrites prepared by co-precipitation method. *J. Magn. Magn. Mater.*, 322: 230–233.

[4] Heer, W.A., (2000), Nanomagnetism characterization of nanophase materials. *Wiley-VCH*.

[5] Nalwa, H.S., (2002), Magnetic nanostructure. *American Cientific Publishers*, USA, (chapter 1–4).

[6] Masala, O., Hoffman, D., Sundaram, N., Page, K., Proffen, T., Lawes, G., Seshadri, R., (2006), Preparation of magnetic spinel ferrite core/shell nanoparticles: Soft ferrites on hard ferrites and vice versa. *Solid State Sci*., 8:1015.

[7] Maeda, T., Sugimoto1, S., Kagotani, T., Tezuka, N., Inomata, K., (2004), Effect of the soft/hard exchange interaction on natural resonance frequency and electromagnetic wave absorption of the rare earth–iron–boron compounds. *J. Magn. Magn. Mater*., 281: 195–205.

[8] Liu, J.R., Itoh, M., Machida, K.I., (2006), Magnetic and electromagnetic wave absorption properties of *α*-Fe/Z-type Ba-ferrite nanocomposites. *Appl. Phys. Lett*., 88: 062503–1.

[9] Masala, O., Hoffman, D., Sundaram, N., Page, K., Proffen, T., Lawes, G., Seshadri, R., (2006), Preparation of magnetic spinel ferrite core/shell nanoparticles: Soft ferrites on hard ferrites and vice versa. *Solid State Sci.*, 8: 1015–1022.

[10] Chen, N., Mu, G., Pan, X., Gan, K., Gu, M., (2007), Microwave absorption properties of $SrFe_{12}O_{19}/ZnFe_2O_4$ composite powders. *Mate. Sci. Eng. B*., 139: 256–260.

[11] Song, F., Shen, X., Liu, M., Xiang, J., Microstructure, magnetic properties and exchange–coupling interactions for one-dimensional hard/soft ferrite nanofibers. *J. Solid State Chem.*, 185: 31-36.

[12] Wang, L., Zhang, Q., (2008), The effect of pH values on the phase formation and properties of $BaFe_{12}O_{19}$ prepared by citrate–EDTA complexing method. *J. Alloys Compd*., 454: 410-414.

[13] Ghasemi, A., Hossienpour, A., Morisako, A., Saatchi, A., Salehi, M., (2006), Electromagnetic properties and microwave absorbing characteristics of doped barium hexaferrite. *J. Magn. Magn. Mater*., 302: 429-435.

[14] Zhang, X., Duan, Y., Guan, H., Liu, S., Wen, B., (2007), Effect of doping MnO_2 on magnetic properties for M-type barium ferrite, *J. Magn. Magn. Mater*., 311: 507-511.

[15] Gómez, P.H., Torres, C., Francisco, C., Muñoz, J.M., Alejos, O., Iñiguez, J.I., Raposo, V.O., (2006), Effect of sintering conditions on the magnetic disaccommodation in barium M-type hexaferrites. *J. Magn. Magn. Mater*., 304: e766-e768.

[16] Watanabe, T., Ichikawa, H., Fukumori, Y., (2002), Tumor accumulation of gadolinium in lipid-nanoparticles intravenously injected for neutron-capture therapy of cancer. *Eur. J. Pharm. Biopharm.*, 54: 119–124.

[17] Pradeep, A., Priyadharsini, P., Chandrasekaran, G., (2008), Sol– gel route of synthesis of nanoparticles of $MgFe_2O_4$ and XRD, FTIR and VSM study. *J. Magn. Magn. Mater*., 320 (2008) 2774– 2779.

[18] Sasakia, T., Oharaa, S., Nakaa, T., Vejpravova, J., (2010), Continuous synthesis of fine $MgFe_2O_4$ nanoparticles by supercritical hydrothermal reaction, *J. Supercrit. Fluid*., 53: 92–94.

[19] Modi, K.B., Joshi, H.H., Kulkarni, R.G., (1996), Magnetic and electrical properties of Al^{3+}-substituted $MgFe_2O_4$, *J. Mater. Sci*., 31: 1311-1317.

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