ECMS 2018

Preparation and Identification of $BaFe_2O_4$ Nanoparticles by the Sol-Gel Route and Investigation of Its Microwave Absorption Characteristics at Ku-Band Frequency using Silicone Rubber Medium

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Received: 22 April 2018; Accepted: 14 May 2018; Published: 18 May 2018

Abstract: In the last decade, spinel structures have been widely explored due to widespread applications in the antibacterial nanocomposites, memory devices, catalysts, photocatalysts, high frequency devices, and electromagnetic absorbing materials. In this study, $BaFe_2O_4$ spinel structure were synthesized through the sol-gel method using low sintering temperature and identified by vibrating sample magnetometer (VSM), X-ray powder diffraction (XRD), Fourier transform infrared (FT-IR), field emission scanning electron microscopy (FE-SEM), and vector network analyzer (VNA) analysis. Results showed that uniform and pure crystal structure of $BaFe_2O_4$ nanoparticles have been prepared based on the sol-gel method. Finally, $BaFe_2O_4$ nanoparticles were blended by silicone rubber to characterize microwave absorption properties of the nanocomposite at ku-band frequency. According to the VNA results, $BaFe_2O_4$/silicone rubber nanocomposite with 1.75 mm thickness absorbed more than 94.38% of microwave irradiation along the ku-band frequency and the maximum reflection loss of the $BaFe_2O_4$/silicone rubber nanocomposite was 51.67 dB at 16.1 GHz.

1. Introduction

The magnetic materials of normal spinel ferrites with general chemical formula MFe_2O_4 have various application owing to type of M cation that M is the divalent metal cation (M^{2+}= Ba^{2+}, Sr^{2+}, Co^{2+}, Mg^{2+}, Zn^{2+}, Cu^{2+}, Mn^{2+}, and etc.). The intrinsic properties of the $BaFe_2O_4$ nanoparticles such as: high magnetic saturation and coercivity, high chemical and mechanical resistance, and high curie temperature have indicated it as a good candidate for microwave devices, radar absorbent materials, permanent magnets, drug deliveries, photocatalytic catalysts, credit cards, and etc. The methods of synthesizing spinel ferrites have large effect on its properties and applications. In the recent decade, extensive researches have been done to improve synthesis methods, which increase crystal purity, decrease size, and control morphology of nanostructures**.** Diverse methods have been used to prepare of $BaFe_2O_4$ nanoparticles such as: spray pyrolysis, co-precipitation, microemulsion, ball milling, and hydrothermal [1–3]. The crystallinity, size, and shape of nanostructures are the most influential factors on the properties of nanomaterials [4]. Most of methods require a high calcination temperature about 800-1000 °C [2,5,6]. In this research, single-phase of ferrite nanoparticles was prepared by the sol-gel method with a low sintering temperature. Moreover, microwave absorption of the $BaFe_2O_4$ nanoparticles were investigated using silicone rubber polymeric matrix.

2. Experimental

2.1. Materials and Instruments

Barium nitrate from Sigma-Aldrich and citric acid, iron (III) nitrate nonahydrate, and ammonia solution were purchased from Merck. Silicone rubber was obtained from ELASTOSIL® M4503. Wacker RTV-2.

Tescan Mira2 presented SEM micrograph of the nanoparticles. The crystal structure of nanostructures was investigated using Philips X'Pert MPD instrument, operated on 40 mA and 40 kV current with Co tube and the wave length of =1.78897 Å. Shimadzu 8400 S FT-IR revealed chemical structure of the sample. The magnetic hysteresis loop was obtained using IRI Kashan VSM. Microwave absorption properties were investigated by Agilent technologies, E8364A.

2.3. Synthesis of $BaFe_2O_4$ Nanoparticles

Barium ferrite nanoparticles were prepared by the conventional sol-gel method. Firstly, metal salts and citric acid with stoichiometric ratios were dissolved in distilled water and then, pH of the solution was rised until alkaline medium by the ammonia solution. Finally, the solution was dried and calcined at 450 or 650°C for 4h to compare the results.

2.4. Preparation of $BaFe_2O_4$/Silicone Rubber Nanocomposite

The $BaFe_2O_4$ nanoparticles were blended with silicone resin and then hardener was added with 20 Wt. % to mold $BaFe_2O_4$/silicone rubber nanocomposite and study microwave absorption of the nanocomposite.

3 Results and Discussion

3.1. Phase Identification Analysis

Figure 1 depicts the XRD patterns of the samples synthesized by the sol-gel method and calcined at 450 or 650 °C for 4 h. The pattern of $BaFe_2O_4$ calcined at 650 °C exhibits that all the obtained peaks correspond with JCPDS number of [00-046-0113]. The XRD patterns indicate that by enhancing the calcination temperature from 450 to 650 °C, the BaCO3 (JCPDS: [00-005-0378]) crystalline phases were disappeared and pure phase of BaFe2O4 nanoparticles has been synthesized. The size of the BaFe2O4 nanoparticles was 10.2 nm based on the Scherrer equation.

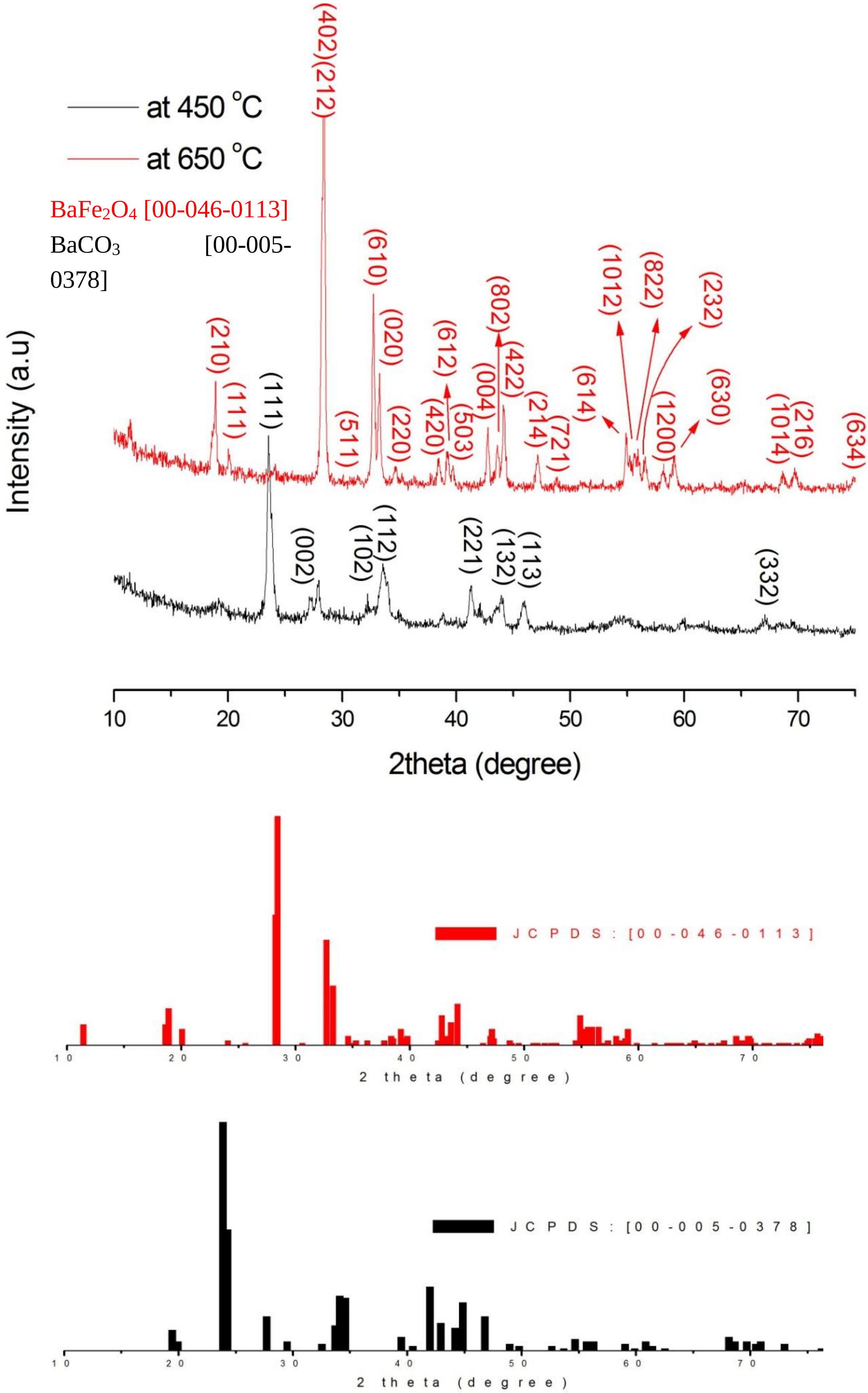

Figure 1. XRD patterns of $BaFe_2O_4$nanoparticles calcined at 450 or 650 °C.

3.2. FE-SEM Morphology

The morphology of $BaFe_2O_4$ nanostructures at 650 °C was investigated using FE-SEM micrograph as shown in the Figure 2. $BaFe_2O_4$ nanoparticles have a poly crystalline structure with average size about 70 nm.

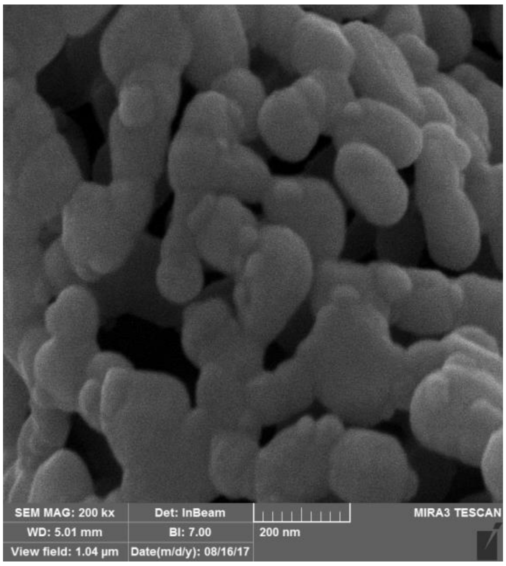

Figure 2. FE-SEM micrograph of $BaFe_2O_4$ nanoparticles.

3.3. FT-IR Spectroscopy

The FT-IR analysis is used to determine the structure and measurement of chemical species. According to the result showed in Figure 3, the peaks at 497.12, 618.30, and 764.16 cm^{-1} are related to stretching vibrations of Ba^{2+}-O^{2-} and Fe^{3+}-O^{2-} in the octahedral and tetrahedral sites as well as the peaks at 1053.63 and 1111.98 cm^{-1} are associated to vibrations of M-O-M (M= Ba^{2+} or Fe^{3+}) in the finger print region corresponded to orthorhombic crystalline structure of prepared BaFe2O4 nanoparticles[2,7,8]. The peak at 1630.34 cm^{-1} and broadband absorption at 3434.51 cm^{-1} are assigned to the bending and stretching vibration of O-H bond associated to adsorbed water as well as remained hydroxyl functional groups on the surface of the nanoparticles[5,6].

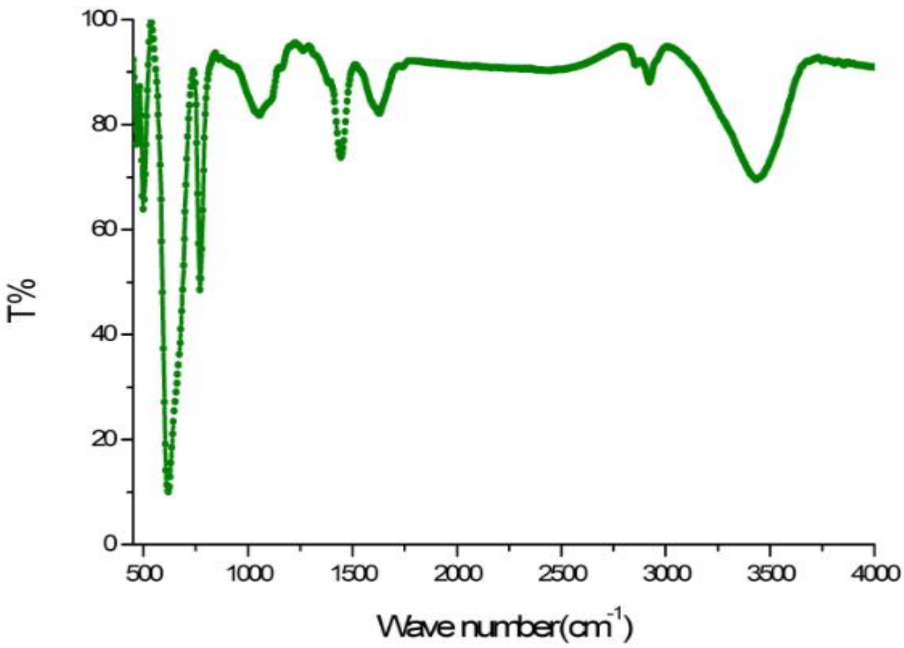

Figure 3. FT-IR spectrum of $BaFe_2O_4$ calcined at 650 °C.

3.4. Magnetic Properties

Magnetic properties of the $BaFe_2O_4$ nanoparticles were explored using VSM, operated at 25 Hz frequency, -15 < kO_e < 15 field, and room temperature. The result demonstrated that saturation magnetization (M_s), remanent magnetization (Mr), and coercivity (Hc) were 0.5 emu/g, 0.2 emu/g, and 4471.0 Oe, respectively (Figure 4.). Numerous researchers have investigated magnetic parameters of M-type BaFe12O19 nanoparticles, exhibiting Ms = 41, 54.97, and 75.54 emu/g as well as Hc= 5450, 4964.5 and 2800 Oe [9-12], showing more paramagnetic properties in comparison to synthesized BaFe2O4 nanoparticles.

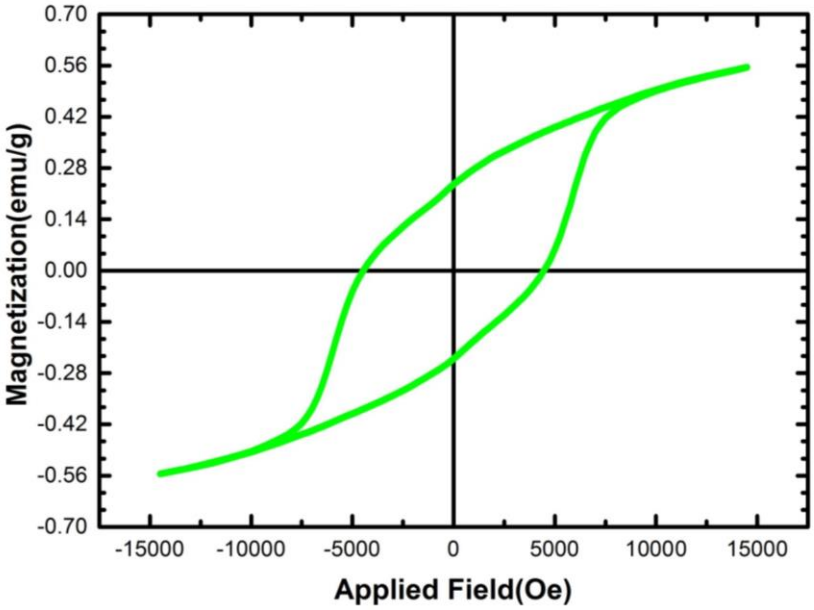

Figure 4. The hysterisis loop of $BaFe_2O_4$ calcined at 650 °C.

3.5. Microwave Absorption Properties

The transmission line theory equation indicates that microwave absorption properties of the materials are generally related to permittivity and permeability of the absorbers [13-16]. According to results, BaFe2O4/silicone rubber nanocomposite with 1.75 mm thickness absorbed more than 94.38% of microwave irradiation at ku-band frequency and the maximum reflection loss of the BaFe2O4/silicone rubber nanocomposite was 51.67 dB at 16.1 GHz (Fig. 5.). Table 1 exposes comparison of presented study with some previously published researches. Broadband and intense microwave absorption of the BaFe2O4/silicone rubber nanocomposite are originated form proper impedance matching, multiple scattering, and interfacial polarization led to more microwave attenuation[17-20].

Table 1. Comparison of presented study with some previously published researches.

Particles	Max RL (dB)	Diameter (mm)	Absorption bandwidth (GHz) < -10 dB	Ref.
$BaFe_{12}O_{19}/Fe_3O_4$	33.6	2.5	1.3	[21]
$CoFe_2O_4$	14	3	2	[22]
$BaFe_{12}O_{19}/CoFe_2O_4$	10	5	-	[23]
$BaFe_{12}O_{19}$	16.1	3	3.8	[24]
$BaCu_{0.5}Mg_{0.5}ZrFe_{10}O_{19}$	9	2.1	-	[25]
$BaFe_{12}O_{19}$	7	2.5	-	[11]
$Ba_{0.25}Sr_{0.75}$ $Fe_{11}(Ni_{0.5}Mn_{0.5})O_{19}$	3.6	4	-	[26]
$BaFe_{12}O_{19}$	10.7	3	-	[12]
$Ba_{0.2}Sr_{0.2}La_{0.6}MnO_3$	22.36	2	2.67	[4]
$BaFe_2O_4$	51.67	1.75	<5.6	Presented study

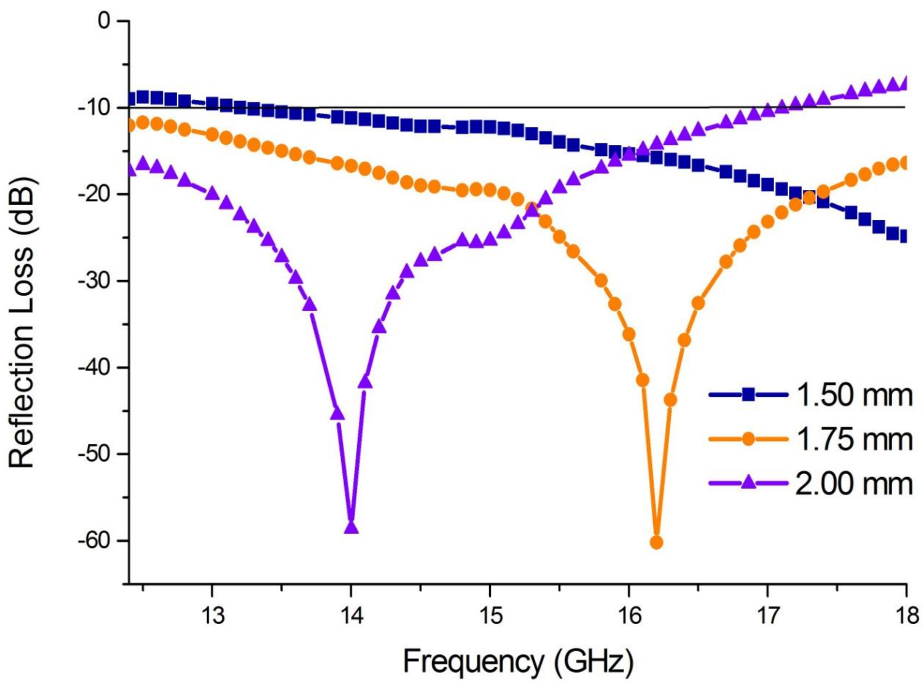

Figure 5. The Reflection losses of $BaFe_2O_4$/silicone rubber nanocomposite at various thicknesses.

4. Conclusions

The obtained results demonstrated that $BaFe_2O_4$ nanoparticles were prepared through the sol-gel method using low sintering temperature as well as confirmed that the heat treatment has a significant effect on the crystal purity of the nanostructures. According to the XRD patterns, phase impurities of nanoparticles disappeared when temperature was enhanced. The FE-SEM micrograph exhibited uniform morphology for $BaFe_2O_4$ nanostructures. The FT-IR curve approved metal-oxide bonds of $BaFe_2O_4$ nanoparticles have been synthesized at the low temperature. Finally, VNA results illustrated that maximum reflection loss of $BaFe_2O_4$/silicone rubber nanocomposite was 51.67 dB at 16.1 GHz as well as the nanocomposite absorbed more than 94.38% of microwave irradiation along the ku-band frequency with a thickness of 1.75 mm. The results introduce $BaFe_2O_4$ nanoparticles as a promising microwave absorbing material.

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