proceedings

Proceedings

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 †

Reza Peymanfar *, Mitra Rahmanisaghieh, Arezoo Ghaffari and Yousef Yassi

Department of Chemical Engineering, Energy Institute of Higher Education, 39177-67746Saveh, Iran; mitra94rahmani@gmail.com (M.R.); arezoghafari@yahoo.com (A.G.); yousefthegreat@yahoo.com.uk (Y.Y.)

* Correspondence: reza_peymanfar@cmps2.iust.ac.ir

† Presented at the 3rd International Electronic Conference on Materials Sciences, 14–28 May 2018. Available online: https://sciforum.net/conference/ecms2018.

Published: 18 May 2018

Abstract: In the last decade, spinel structures have been widely explored due to widespread applications in antibacterial nanocomposites, memory devices, catalysts, photocatalysts, high-frequency devices, and electromagnetic absorbing materials. In this study, $BaFe_2O_4$ spinel structures were synthesized through the sol–gel method using a low sintering temperature and were identified by vibrating sample magnetometer (VSM), X-ray powder diffraction (XRD), Fourier transform infrared (FTIR) spectroscopy, field emission scanning electron microscopy (FE-SEM), and vector network analyzer (VNA) analysis. Results showed that uniform and pure crystal structures of $BaFe_2O_4$ nanoparticles were prepared based on the sol–gel method. Finally, $BaFe_2O_4$ nanoparticles were blended by silicone rubber to characterize the microwave absorption properties of the nanocomposite at the ku-band frequency. According to the VNA results, the $BaFe_2O_4$/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.

Keywords: $BaFe_2O_4$; Silicone rubber; microwave absorption

1. Introduction

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

2. Experimental

2.1. Materials and Instruments

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

Tescan Mira2 (Brno, Czech Republic)presented SEM micrographs of the nanoparticles. The crystal structure of the nanostructures was investigated using a Philips X'Pert MPD (Amsterdam, Netherlands) instrument operating on 40 mA and 40 kV current with a Co tube and a wave length of λ = 1.78897 Å. Shimadzu 8400 S FTIR (Kyoto, Japan) revealed the chemical structure of the sample. The magnetic hysteresis loop was obtained using IRI Kashan VSM. Microwave absorption properties were investigated by Agilent technologies (Santa Clara, CA, USA), E8364A.

2.2. 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 the pH of the solution was raised to an alkaline medium by the ammonia solution. Finally, the solution was dried and calcined at 450 or 650 °C for 4 h to compare the results.

2.3. Preparation of $BaFe_2O_4$/Silicone Rubber Nanocomposite

The $BaFe_2O_4$ nanoparticles were blended with silicone resin and then a hardener was added with 20 wt% to mold a $BaFe_2O_4$/silicone rubber nanocomposite and study the 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 the JCPDS number [00-046-0113]. The XRD patterns indicate that by increasing the calcination temperature from 450 to 650 °C, the $BaCO_3$ (JCPDS: [00-005-0378]) crystalline phases disappeared and a pure phase of $BaFe_2O_4$ nanoparticles was synthesized. The size of the $BaFe_2O_4$ nanoparticles was 10.2 nm based on the Scherrer equation.

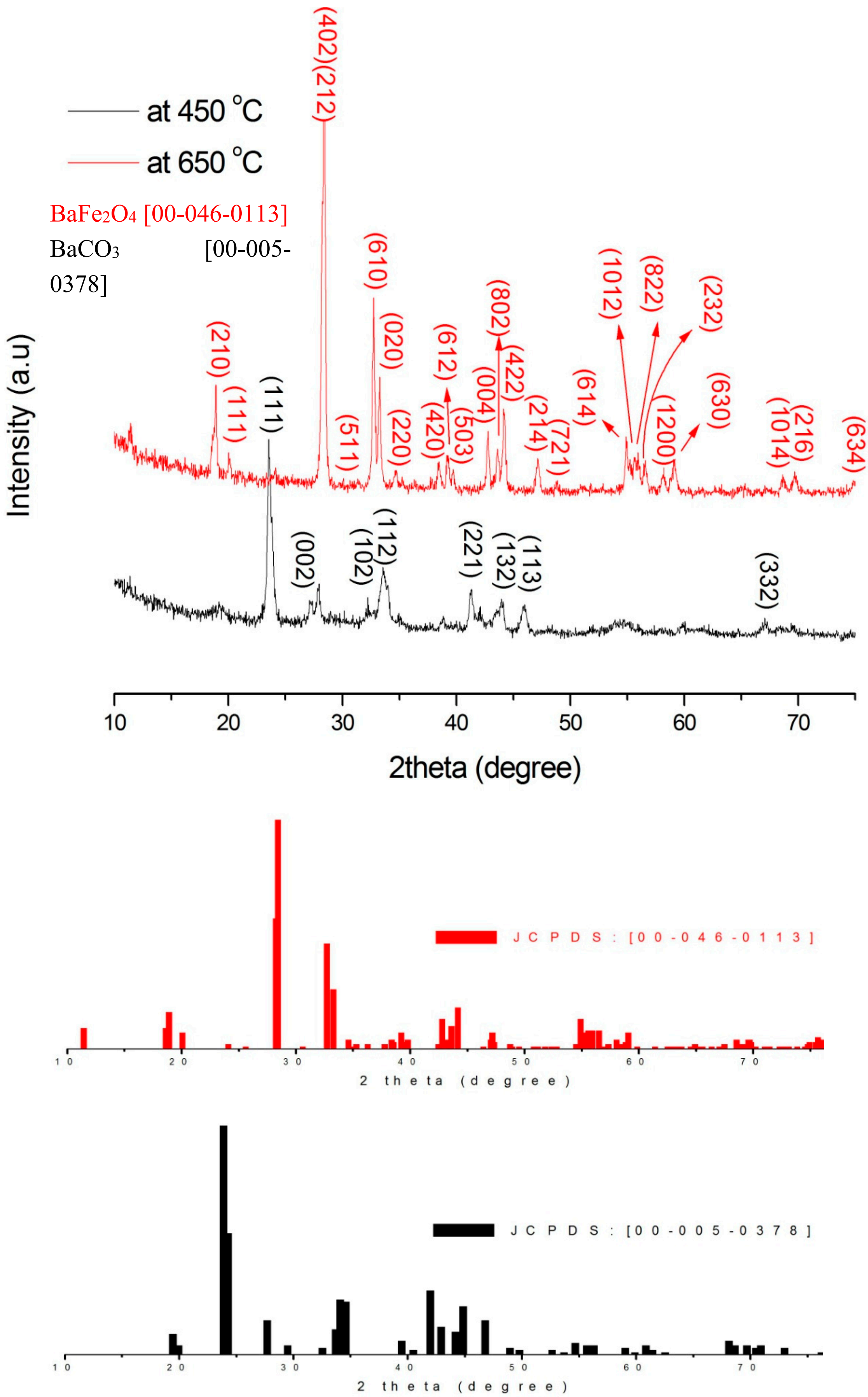

Figure 1. XRD patterns of $BaFe_2O_4$ nanoparticles calcined at 450 or 650 °C.

3.2. FE-SEM Morphology

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

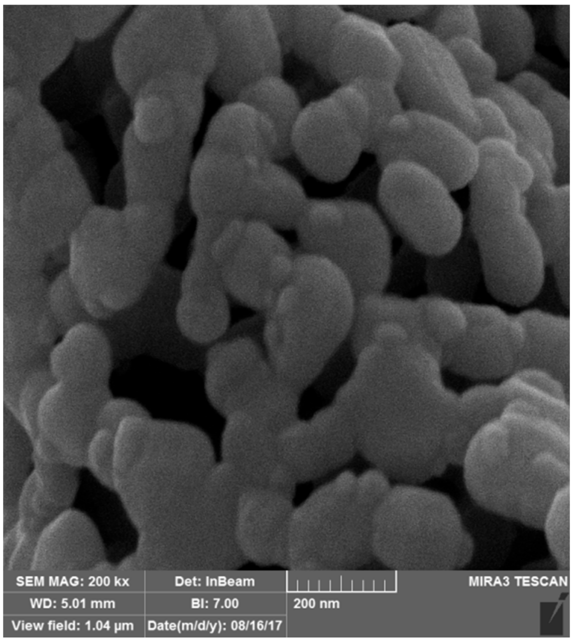

Figure 2. FE-SEM micrograph of $BaFe_2O_4$ nanoparticles.

3.3. FTIR Spectroscopy

The FTIR analysis was used to determine the structure and measurement of chemical species. According to the results shown 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, and the peaks at 1053.63 and 1111.98 cm^{-1} are associated with vibrations of M–O–M (M = Ba^{2+} or Fe^{3+}) in the finger print region corresponding to the orthorhombic crystalline structure of the prepared $BaFe_2O_4$ nanoparticles [2,7,8]. The peak at 1630.34 cm^{-1} and broadband absorption at 3434.51 cm^{-1} are attributed to the bending and stretching vibration of the O–H bond associated with adsorbed water as well as the remaining hydroxyl functional groups on the surface of the nanoparticles [5,6].

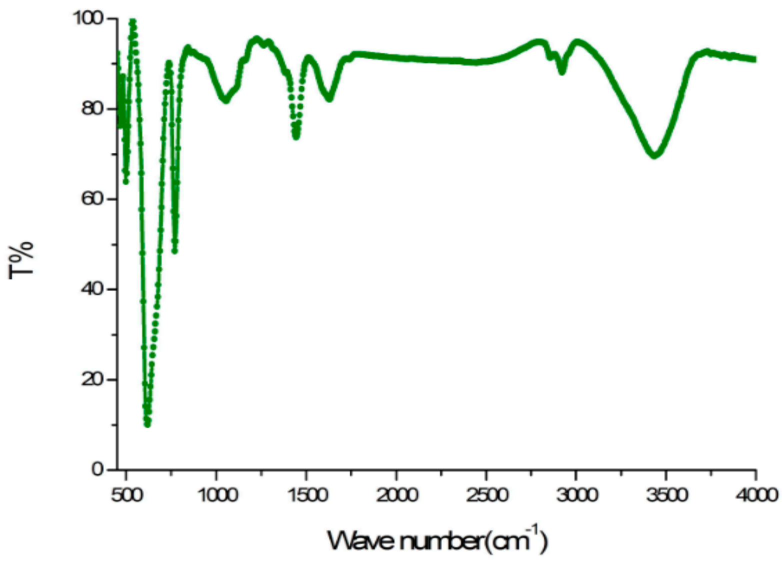

Figure 3. FTIR spectrum of $BaFe_2O_4$ calcined at 650 °C.

3.4. Magnetic Properties

The magnetic properties of the $BaFe_2O_4$ nanoparticles were explored using VSM operated at a 25-Hz frequency, −15 < kO_e < 15 field, and room temperature. The 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 studies have investigated the magnetic parameters of M-type $BaFe_{12}O_{19}$ nanoparticles, exhibiting Ms = 41, 54.97, 75.54 emu/g, Hc = 5450, 4964.5, and 2800 Oe [9–12], which show more paramagnetic properties in comparison to synthesized $BaFe_2O_4$ nanoparticles.

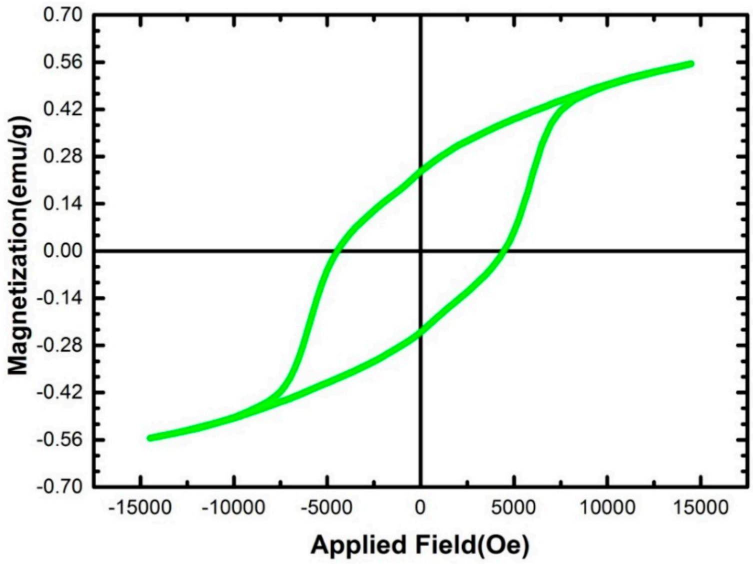

Figure 4. The hysterisis loop of $BaFe_2O_4$ calcined at 650 °C.

3.5. Microwave Absorption Properties

The transmission line theory equation indicates that the microwave absorption properties of the materials are generally related to the permittivity and permeability of the absorbers [13–16]. According to the results, the $BaFe_2O_4$/silicone rubber nanocomposite with 1.75 mm thickness absorbed more than 94.38% of microwave irradiation at 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 (Figure 5). Table 1 compares the results of this study with some previously published data. The broadband and intense microwave absorption of the $BaFe_2O_4$/silicone rubber nanocomposite originated from proper impedance matching, multiple scattering, and interfacial polarization, which led to more microwave attenuation [17–20].

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

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

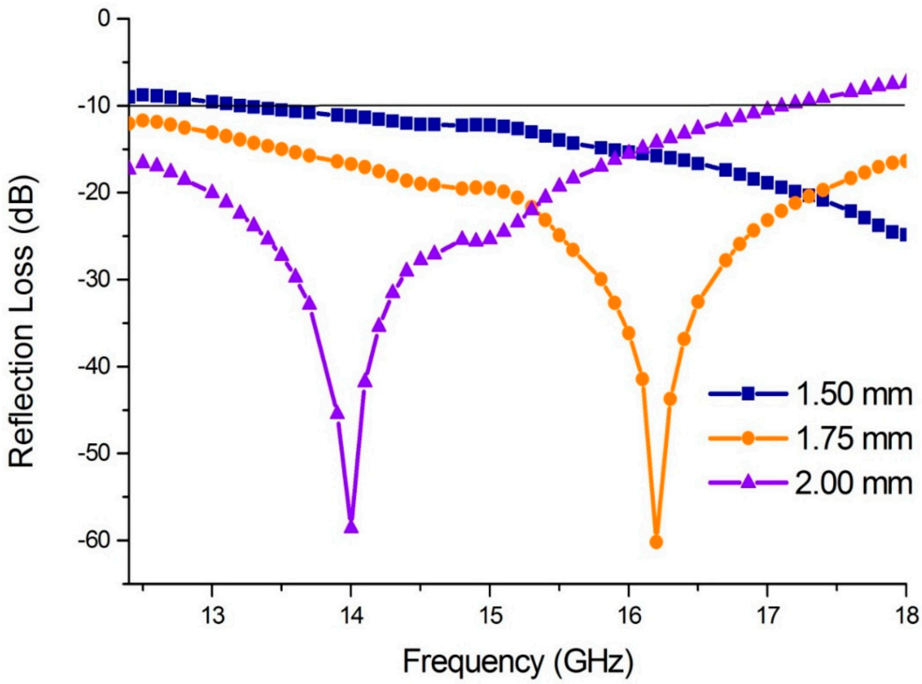

Figure 5. The reflection losses of $BaFe_2O_4$/silicone rubber nanocomposite at various thicknesses.

4. Conclusions

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

References

1. Galvão, W.S.; Neto, D.; Freire, R.M.; Fechine, P. Super-paramagnetic nanoparticles with spinel structure: A review of synthesis and biomedical applications. In *Solid State Phenomena*; Trans Tech Publications: Zürich, Switzerland, 2016; pp. 139–176.
2. Mandizadeh, S.; Salavati-Niasari, M.; Sadri, M. Hydrothermal synthesis, characterization and magnetic properties of $BaFe_2O_4$ nanostructure as a photocatalytic oxidative desulfurization of dibenzothiophene. *Sep. Purif. Technol.* **2017**, *175*, 399–405.
3. Lemine, O.; Bououdina, M.; Sajieddine, M.; Al-Saie, A.; Shafi, M.; Khatab, A.; Al-Hilali, M.; Henini, M. Synthesis, structural, magnetic and optical properties of nanocrystalline $ZnFe_2O_4$. *Phys. B Condens. Matter* **2011**, *406*, 1989–1994.
4. Peymanfar, R.; Javanshir, S. Preparation and characterization of $Ba_{0.2}Sr_{0.2}La_{0.6}MnO_3$ nanoparticles and investigation of size & shape effect on microwave absorption. *J. Magn. Magn. Mater.* **2017**, *432*, 444–449.
5. Saravani, H.; Esmaeilzaei, M.R.; Ghahfarokhi, M.T. Synthesis and Characterization of Ferromagnetic $BaFe_2O_4$ Nanocrystals Using Novel Ionic Precursor Complex $[Fe(opd)_3]_2[Ba(CN)_8]$. *J. Inorg. Organomet. Polym. Mater.* **2016**, *26*, 353–358.

6. Liu, X.; Zhang, T.; Xu, D.; Zhang, L., Microwave-Assisted Catalytic Degradation of Crystal Violet with Barium Ferrite Nanomaterial. *Ind. Eng. Chem. Res.* **2016**, *55*, 11869–11877.
7. Shen, P.; Luo, J.; Zuo, Y.; Yan, Z.; Zhang, K. Effect of La-Ni substitution on structural, magnetic and microwave absorption properties of barium ferrite. *Ceram. Int.* **2017**, *43*, 4846–4851.
8. Ali, K.; Iqbal, J.; Jana, T.; Ahmad, N.; Ahmad, I.; Wan, D., Enhancement of microwaves absorption properties of $CuFe_2O_4$ magnetic nanoparticles embedded in MgO matrix. *J. Alloys Compd*. **2017**, *696*, 711–717.
9. Bahadur, A.; Saeed, A.; Iqbal, S.; Shoaib, M.; Ahmad, I.; ur Rahman, M.S.; Bashir, M.I.; Yaseen, M.; Hussain, W. Morphological and magnetic properties of $BaFe_{12}O_{19}$ nanoferrite: A promising microwave absorbing material. *Ceram. Int.* **2017**, *43*, 7346–7350.
10. Wang, M.; An, K.; Fang, Y.; Wei, G.; Yang, J.; Sheng, L.; Yu, L.; Zhao, X. The microwave absorbing properties of $CoFe_2$ attached single walled carbon nanotube/$BaFe_{12}O_{19}$ nanocomposites. *J. Mater. Sci. Mater. Electron.* **2017**, *28*, 12475–12483.
11. Afzali, A.; Mottaghitalab, V.; Afghahi, S.S.; Jafarian, M. The coating of composite nanoparticles of $BaFe_{12}O_{19}$/multi-walled carbon nanotubes using silicon matrix on nonwoven substrate for radar absorption in X and Ku bands. *J. Ind. Text.* **2018**, *47*, 1867–1886.
12. Feng, H.; Bai, D.; Tan, L.; Chen, N.; Wang, Y. Preparation and microwave-absorbing property of EP/$BaFe_{12}O_{19}$/PANI composites. *J. Magn. Magn. Mater.* **2017**, *433*, 1–7.
13. Afghahi, S.S.S.; Peymanfar, R.; Javanshir, S.; Atassi, Y.; Jafarian, M. Synthesis, characterization and microwave characteristics of ternary nanocomposite of MWCNTs/doped Sr-hexaferrite/PANI. *J. Magn. Magn. Mater.* **2017**, *423*, 152–157.
14. Peymanfar, R.; Javidan, A.; Javanshir, S. Preparation and investigation of structural, magnetic, and microwave absorption properties of aluminum-doped strontium ferrite/MWCNT/polyaniline nanocomposite at KU-band frequency. *J. Appl. Polym. Sci.* **2017**, *134*, doi:10.1002/app.45135.
15. Ma, Z.; Mang, C.; Weng, X.; Zhang, Q.; Si, L.; Zhao, H. The Influence of Different Metal Ions on the Absorption Properties of Nano-Nickel Zinc Ferrite. *Materials* **2018**, *11*, 590.
16. Li, Y.; Li, D.; Yang, J.; Luo, H.; Chen, F.; Wang, X.; Gong, R. Enhanced Microwave Absorption and Surface Wave Attenuation Properties of $Co_{0.5}Ni_{0.5}Fe_2O_4$ Fibers/Reduced Graphene Oxide Composites. *Materials* **2018**, *11*, 508.
17. Peymanfar, R.; Javanshir, S.; Naimi-Jamal, M.R. Preparation and characterization of MWCNT/$Zn_{0.25}Co_{0.75}Fe_2O_4$ nanocomposite and investigation of its microwave absorption properties at x-band by silicone rubber polymeric matrix. In Proceedings of the 21st International Electronic Conference on Synthetic Organic Chemistry, Santiago, Spain, 1–30 November 2017.
18. Seyed, A.S.S.; Peymanfar, R.; Javanshir, S.; Javidan, A. Preparation and investigation of structural, magnetic and microwave absorption properties of $Ba_{0.2}Sr_{0.2}La_{0.6}MnO_3$/MWCNT nanocomposite in comparison with $Ba_{0.2}Sr_{0.2}La_{0.6}MnO_3$ in x-band region. *Nanoscale* **2015**, *2*, 73–80.
19. Cao, W.-Q.; Wang, X.-X.; Yuan, J.; Wang, W.-Z.; Cao, M.-S. Temperature dependent microwave absorption of ultrathin graphene composites. *J. Mater. Chem. C* **2015**, *3*, 10017–10022.
20. Dalal, M.; Greneche, J.-M.; Satpati, B.; Ghzaiel, T.B.; Mazaleyrat, F.; Ningthoujam, R.S.; Chakrabarti, P.K. Microwave Absorption and the Magnetic Hyperthermia Applications of $Li_{0.3}Zn_{0.3}Co_{0.1}Fe_{2.3}O_4$ Nanoparticles in Multiwalled Carbon Nanotube Matrix. *ACS Appl. Mater. Interfaces* **2017**, *9*, 40831–40845.
21. Lin, Y.; Liu, Y.; Dai, J.; Wang, L.; Yang, H. Synthesis and microwave absorption properties of plate-like $BaFe_{12}O_{19}$@Fe_3O_4 core-shell composite. *J. Alloys Compd.* **2018**, *739*, 202–210.
22. Feng, J.; Wang, Y.; Hou, Y.; Li, J.; Li, L. Synthesis and microwave absorption properties of coiled carbon nanotubes/$CoFe_2O_4$ composites. *Ceram. Int.* **2016**, *42*, 17814–17821.
23. Yang, H.; Ye, T.; Lin, Y.; Liu, M. Preparation and microwave absorption property of graphene/$BaFe_{12}O_{19}$/$CoFe_2O_4$ nanocomposite. *Appl. Surface Sci.* **2015**, *357*, 1289–1293.
24. Tang, X.; Wei, G.; Zhu, T.; Sheng, L.; An, K.; Yu, L.; Liu, Y.; Zhao, X. Microwave absorption performance enhanced by high-crystalline graphene and $BaFe_{12}O_{19}$ nanocomposites. *J. Appl. Phys.* **2016**, *119*, 204301.

25. Nikmanesh, H.; Moradi, M.; Bordbar, G.H.; Alam, R.S. Synthesis of multi-walled carbon nanotube/doped barium hexaferrite nanocomposites: An investigation of structural, magnetic and microwave absorption properties. *Ceram. Int.* **2016**, *42*, 14342–14349.
26. Ezzati, S.N.; Rabbani, M.; Leblanc, R.M.; Asadi, E.; Ezzati, S.M.H.; Rahimi, R.; Azodi-Deilami, S. Conducting, magnetic polyaniline/$Ba_{0.25}Sr_{0.75}Fe_{11}(Ni_{0.5}Mn_{0.5})O_{19}$ nanocomposite: Fabrication, characterization and application. *J. Alloys Compd.* **2015**, *646*, 1157–1164.