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# Selective microwave absorption in Nd<sup>3+</sup> substituted barium ferrite composites<sup>☆</sup>

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## ABSTRACT

Microwave (MW) frequency based wireless communications and electronic devices became prospective due to several ramifications. To meet this need, a series of neodymium ions (Nd<sup>3+</sup>) substituted barium ferrite composites with composition (20)BaO:(80-x)Fe<sub>2</sub>O<sub>3</sub>:(x)Nd<sub>2</sub>O<sub>3</sub> (0 ≤ x ≤ 3 mol%) was prepared at 1100 °C using solid-state reaction method. We evaluated the effect of various Nd<sup>3+</sup> ions contents on the surface morphology, structure, and magnetic properties of the as-synthesized barium ferrite composites. Meanwhile, microwave reflection loss, complex permittivity and permeability were determined using the transmission/reflection line method in the X-band (8–12 GHz). SEM image of the composites shows that the surface morphology consists of rough and porous microstructures. XRD patterns of the un-doped composites reveal the existence of BaFe<sub>12</sub>O<sub>19</sub> (hexagonal) and Fe<sub>21.333</sub>O<sub>32</sub> (tetragonal) crystalline phases. Furthermore, a new hexagonal crystalline phase of Ba<sub>6</sub>Nd<sub>2</sub>Fe<sub>4</sub>O<sub>15</sub> with the crystallite sizes between 15 and 67 nm is observed due to Nd<sup>3+</sup> ions substitution in the composite. The saturation magnetization of the composite containing 2 mol% of Nd<sup>3+</sup> does not exhibit any significant alteration compared to the one devoid of Nd<sup>3+</sup>. The complex relative permittivity and permeability of the achieved composites enriched in Ba<sub>6</sub>Nd<sub>2</sub>Fe<sub>4</sub>O<sub>15</sub> and BaFe<sub>2</sub>O<sub>4</sub> phases disclose significant MW frequency dependence. The composites also display selective MW absorption in the X-band which could be useful for diverse applications.

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## 1. Introduction

In recent times, wireless communications and electronic devices based on the microwave (MW) frequency in the range of 8–12 GHz (X-band) received focused attention, wherein dedicated efforts have been made to achieve the emission of such frequencies from a transmitter and radar. Materials required for such purposes must be capable in attenuating and dissipating the excess amount of electromagnetic energy in the form of thermal energy via magnetic and dielectric loss mechanisms.<sup>1</sup> Thus, novel magnetic materials (ferrites based) appropriate for absorbing the electromagnetic energy (MW) have continually been explored.<sup>2–10</sup> In this regard, barium hexaferrite (BHF) owing to its extremely high uniaxial

magnetic anisotropy showed an emergent MW absorbing material.<sup>3–5,11,12</sup> Thus, it became essential to optimize the MW absorption capacity of these ferrites in the X-band (8–12 GHz).

It was demonstrated that the natural resonance-frequencies, saturation magnetization, permeability, permittivity and magnetic field anisotropy of such ferrites could be altered by incorporating divalent or trivalent ions into the crystalline structures.<sup>13–15</sup> Consequently, the effects of electromagnetic interferences in these ferrites could be improved. Meanwhile, outstanding relaxation attributes of rare earth ions (REIs) as dopants inside the BHF have also been exploited to alter its magnetic and MW absorption characteristics.<sup>3,13,16–19</sup> Besides, BHF doped with REIs revealed weak coercive field ( $H_c$ ) and low saturation magnetization ( $M_s$ ) which could be useful for the enhancement of MW absorption capacity.<sup>6,13,20,21</sup> Inspired by these innovations, we took an attempt to diminish the values of  $H_c$  and  $M_s$  of the prepared barium ferrite composites (BFCs), there by enhancing the selective absorption of MW frequencies.

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This communication reports the production and characterizations of new BFCs (hereafter referred as NdBFCs) wherein the  $\text{Fe}^{3+}$  ions in pure BHF were replaced by  $\text{Nd}^{3+}$  ions (at various concentrations). Trivalent  $\text{Nd}^{3+}$  due to their compatible hexagonal lattice structure and excellent relaxational attributes were selected as doping agents (so-called substituent ions). Two types of composites (undoped and doped with  $\text{Nd}^{3+}$  ions) were synthesized via tailored solid-state reaction method. As-prepared composites were characterized using different analytical tools to determine the influence of  $\text{Nd}^{3+}$  ions on the microstructures, surface morphologies, magnetic characteristics, and MW reflection loss in the X-band. Achieved  $\text{Ba}_6\text{Nd}_2\text{Fe}_4\text{O}_{15}$  and  $\text{BaFe}_2\text{O}_4$  phases of the proposed BFCs were shown to be beneficial for sundry applications.

## 2. Experimental

Few BFCs (one without and three with  $\text{Nd}^{3+}$  ions doping) with the composition of  $(20)\text{BaO}:(80-x)\text{Fe}_2\text{O}_3:(x)\text{Nd}_2\text{O}_3$ , where  $0 \leq x \leq 3$  mol% were prepared using modified solid-state reaction strategy. High purity powders (analytical grade) of  $\text{BaCO}_3$  (Merck, 99%),  $\text{Nd}_2\text{O}_3$  (Sigma Aldrich, 99%) and  $\text{Fe}_2\text{O}_3$  (Sigma Aldrich, 99%) were used as preparatory materials to synthesize such BFCs. Furthermore, the  $\text{BaCO}_3$  powder was calcined at  $300^\circ\text{C}$  for an hour to remove the existed carbon element. Later,  $\text{BaO}$  powder was mixed gradually with  $\text{Fe}_2\text{O}_3$  via annealing and sintering processes. Afterward, the  $\text{Nd}_2\text{O}_3$  powder was added to the mixture and thoroughly mixed using a magnetic stirrer at the revolution speed of 700 r/min (rotation per minute) for 2 h. Polyvinyl alcohol (PVA) was mixed and stirred continually until a sol was achieved. After that, the composite powder was compacted to produce pellets with the respective thickness and diameter of 1 mm and 1 cm.<sup>13</sup> Besides, some pellets of rectangular shape with a dimension of  $2.5\text{ cm} \times 1.5\text{ cm} \times 0.2\text{ cm}$  were obtained for additional characterizations. These pellets were further annealed at  $800^\circ\text{C}$  for 1 h and sintered at  $1100^\circ\text{C}$  for 5 h<sup>22</sup> before being cooled down naturally to

room temperature. The achieved pellets were named as NdBF0, NdBF1, NdBF2 and NdBF3 based on their corresponding  $\text{Nd}^{3+}$  ions content of 0 mol%, 1 mol%, 2 mol% and 3 mol%. Finally, the prepared composites were characterized at room temperature using different analytical techniques to determine the feasibility of achieving their selective MW absorption effectiveness in the X-band.

A scanning electron microscope (SEM, Hitachi SU 3500) equipped with energy dispersive X-ray (EDX, Horiba) was utilized to determine the surface morphologies, microstructures and elemental compositions of the synthesized NdBFCs.<sup>13,21,23</sup> The presence of crystalline phases in the studied NdBFCs was verified using X-ray diffraction measurement (XRD, SmartLab 3 kW) equipped with  $\text{Cu K}\alpha$  radiation of wavelength ( $\lambda$ ) 0.1541874 nm. A vibrating sample magnetometer (VSM, Oxford 1.2H) was employed to examine the magnetic behavior of the studied composites. The scattering (S) parameters of the proposed composites were measured using a Vector network analyzer (VNA, Advantest R3770) operated in the frequency range of 8–12 GHz. Additionally, the transmission/reflection line method was exploited to determine the MW reflection loss ( $R_L$ ), the complex relative permittivity and permeability.

## 3. Results and discussion

### 3.1. Surface morphology and elemental traces

Fig. 1 displays the SEM images of all synthesized composites, wherein the surface morphology consisted of porous microstructures with roughness. Overall, the composites surface morphology and the distribution of particles size were influenced due to the  $\text{Nd}^{3+}$  ions substitution into the crystalline lattices. At higher  $\text{Nd}^{3+}$  concentrations, the microstructure manifested two phases including the hexagonal grains of barium ferrite (dark color) and Nd-rich (bright color) regions. The microstructure of NdBF2 was

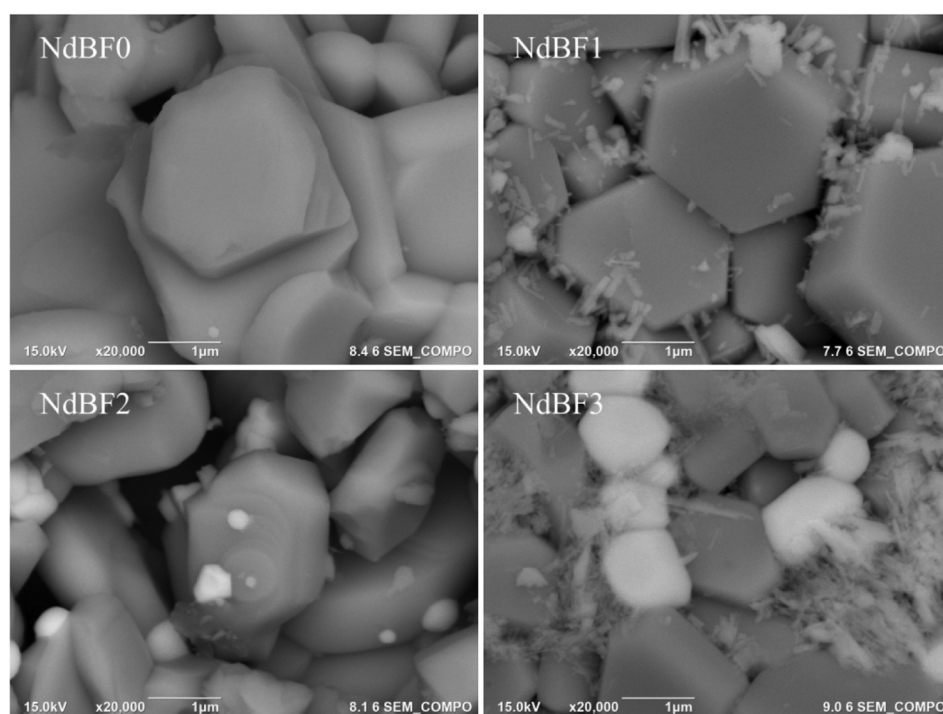


Fig. 1. SEM micrographs of the as-synthesized NdBFCs.

more porous than the other samples. The mean grain size BFCs was shrunk from 2.5 to 1  $\mu\text{m}$  with the increase in the  $\text{Nd}^{3+}$  ions content from 0 to 3 mol% accompanied by porosity enhancement. This enrichment in the porosity led to an improvement in the magnetic characteristics and MW-reflection loss.<sup>21</sup> Fig. 2 depicts the EDX spectra of all BFCs, confirming the appropriate elemental traces of O, Fe, Ba and Nd where in the inclusion of Nd in the composite caused an adjustment in the level of other elements (table in the inset). Sample NdBF2 revealed the minimum traces of Nd (0.1 at%), and NdBF3 exhibited the highest level of Nd (0.3 at%).

### 3.2. Crystalline structure and sizes

Fig. 3 illustrates the XRD patterns of the synthesized composites which comprised of several sharp peaks characteristics of different crystalline lattices. For the sample without  $\text{Nd}^{3+}$  substitution (NdBF0), all the observed peaks were assigned to the dominant hexagonal crystalline structure of  $\text{BaFe}_{12}\text{O}_{19}$  which matched with the ICDD number 00-027-1029 with crystallographic parameters of  $a = b = 0.5892 \text{ nm}$ ,  $c = 2.3198 \text{ nm}$ ,  $\alpha = \beta = 90^\circ$  and  $\gamma = 120^\circ$ . However, the peak that appeared at  $28.36^\circ$  was allocated to the tetragonal crystal structure of  $\text{Fe}_{21.333}\text{O}_{32}$  which was tallied with the ICDD numbers 01-076-1470. Furthermore, substitution of  $\text{Fe}^{3+}$  by  $\text{Nd}^{3+}$  ions (at 1 mol% of  $\text{Nd}_2\text{O}_3$ ) in the barium ferrite lattice could produce a significant change in the crystal structure as verified by the XRD pattern of NdBF1. The emergence of two new peaks in NdBF1 corresponding to a primary phase of  $\text{Ba}_6\text{Nd}_2\text{Fe}_4\text{O}_{15}$  and a secondary phase of  $\text{BaFe}_{18}\text{O}_{27}$  were clearly disclosed. The occurrences of sharp XRD peaks were allotted to the hexagonal lattice of  $\text{Ba}_6\text{Nd}_2\text{Fe}_4\text{O}_{15}$  (matched with the ICDD number 01-079-2141) with  $a = b = 1.1815 \text{ nm}$ ,  $c = 0.7078 \text{ nm}$ ,  $\alpha = \beta = 90^\circ$  and  $\gamma = 120^\circ$ . Moreover, the peak centered at  $32.17^\circ$  was endorsed to the hexagonal crystalline lattice of  $\text{BaFe}_{18}\text{O}_{27}$  (matched with the ICDD number 01-075-0406). The inclusion of 2 mol% of  $\text{Nd}_2\text{O}_3$  (sample

NdBF2) into the BFCs could transform the crystalline phase from hexagonal  $\text{BaFe}_{18}\text{O}_{27}$  to orthorhombic  $\text{BaFe}_2\text{O}_4$  structure (matched with the ICDD number 01-077-2337). Meanwhile, further addition of  $\text{Nd}_2\text{O}_3$  up to 3 mol% in the BFCs (sample NdBF3) led to an alteration in the XRD peak position of  $\text{BaFe}_2\text{O}_4$  without significant variations in the phase and structure. It was affirmed that the replacement of  $\text{Fe}^{3+}$  by  $\text{Nd}^{3+}$  ions in the studied BFCs could generate a new primary hexagonal crystalline phase of  $\text{Ba}_6\text{Nd}_2\text{Fe}_4\text{O}_{15}$ . Widening of the diffraction peaks for all as-synthesized NdBFCs is credited to the nano-crystalline size and lattice strain.<sup>24,25</sup> The crystallite sizes of the studied  $\text{Ba}_6\text{Nd}_2\text{Fe}_4\text{O}_{15}$  (calculated using the Debye-Scherrer's equation) were ranged from 15 to 67 nm.

### 3.3. Magnetic properties

Fig. 4 depicts the magnetization ( $M$ ) concerning the applied field ( $H$ ) of all the studied barium ferrites without and with  $\text{Nd}^{3+}$  ions substitution. The composite without containing  $\text{Nd}^{3+}$  ions (NdBF0) revealed that the saturation magnetization ( $M_s$ ), remanence magnetization ( $M_r$ ), and coercivity field ( $H_c$ ) were 16.36 emu/g, 11.82 emu/g and 0.24 T, respectively. The overall magnetic behavior of the composite (NdBF1), except for the  $M_s$  did not significantly change due to addition of 1 mol% of  $\text{Nd}_2\text{O}_3$ . The inclusion of 2 mol%  $\text{Nd}_2\text{O}_3$  into the composite (NdBF2) did not affect the value of  $M_s$ . However, incorporation of 3 mol%  $\text{Nd}_2\text{O}_3$  into the composite (NdBF3) could lead to an increase in the value of  $M_s$  comparable to NdBF1, which was ascribed to the enhanced porosity of the barium ferrite surface. It was argued that the occurrence of such excessive porosity could isolate the magnetic domain walls, leading to natural polarization of the unpaired magnetic spins in the presence of an externally applied magnetic field. The magnetic hysteresis loop of NdBF0 coincided with NdBF2. Similarly, the magnetic hysteresis loop of NdBF1 was overlapped with NdBF3. Thus, it was established

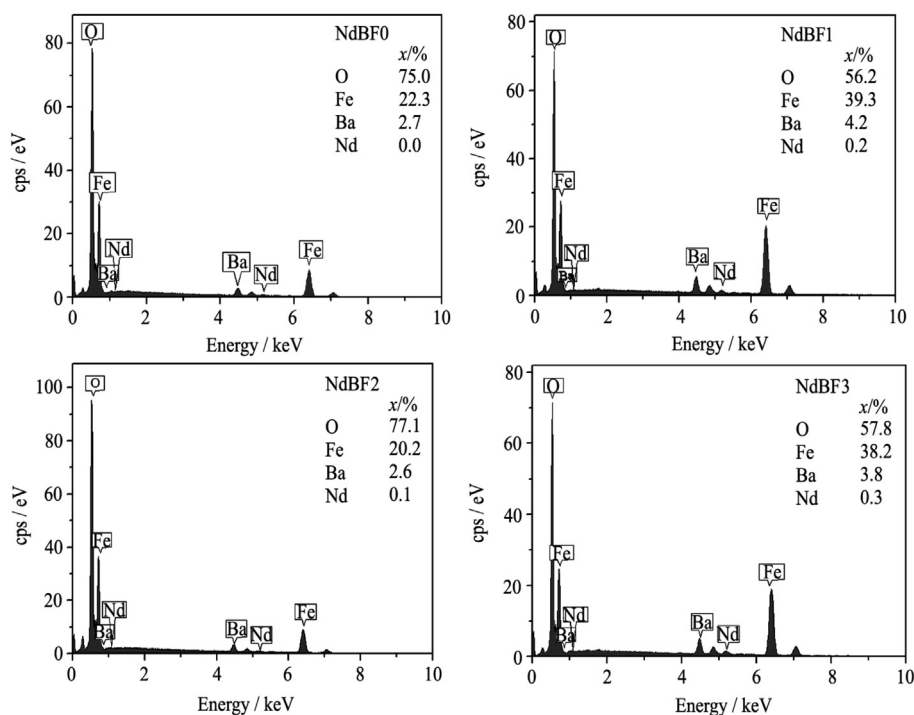


Fig. 2. EDX spectra of the as-synthesized NdBFCs.

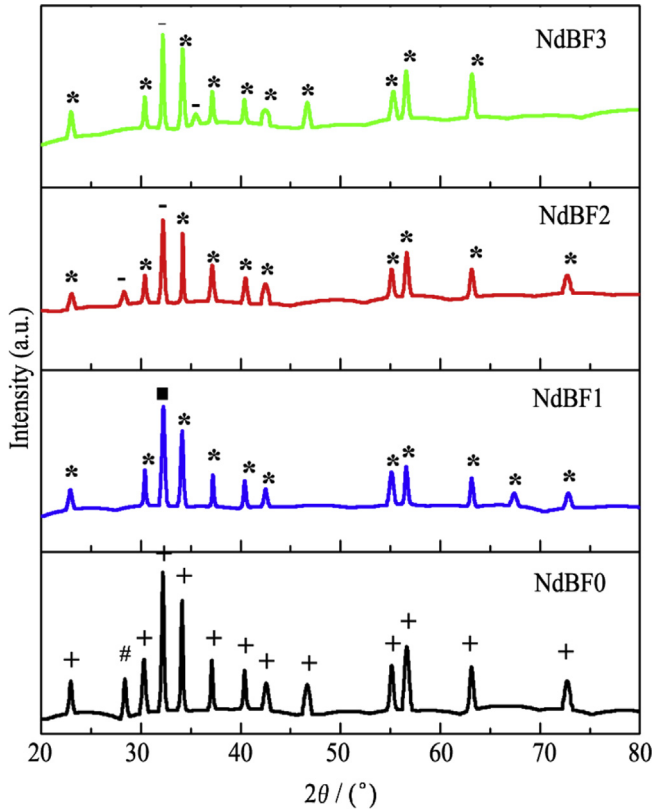


Fig. 3. XRD patterns of all as-synthesized NdBFCs. \*  $\text{Ba}_6\text{Nd}_2\text{Fe}_4\text{O}_{15}$ ; +  $\text{BaFe}_{12}\text{O}_{19}$ ; ■  $\text{BaFe}_{18}\text{O}_{27}$ ; -  $\text{BaFe}_2\text{O}_4$ ; #  $\text{Fe}_{21.333}\text{O}_{32}$ .

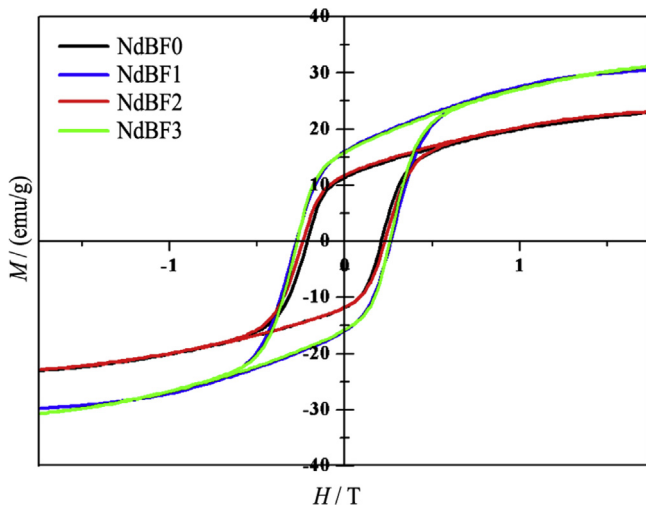


Fig. 4. Hysteresis loops of the studied NdBFCs

that such pair ferrites (NdBF0 and NdBF2 as well as NdBF1 and NdBF3) had almost identical magnetic properties. Disclosure of such narrow hysteresis loops (loop-gap area) indeed confirmed the soft magnetic character of the proposed BFCs. On top, the studied composites failed to display the complete saturation magnetization because of the presence of antimagnetic materials such as  $\text{BaFe}_2\text{O}_4$  and  $\text{Fe}_{21.333}\text{O}_{32}$ . The underlying development of the non-magnetic

phase because of a sintering procedure at high temperature is likewise in charge of altering  $M_s$ .<sup>25,26</sup>

### 3.4. Complex relative permittivity and permeability

Fig. 5(a) shows the frequency-dependent complex relative permittivity ( $\epsilon_r$ ) for all composites in the X-band. The real ( $\epsilon'$ ) and imaginary ( $\epsilon''$ ) components of the permittivity of the prepared composites aroused from the intrinsic electric dipole polarization strongly depended on the MW frequencies.<sup>27</sup> Actually, the real part of the permittivity signified the energy stored in the material from an external electric field.<sup>28</sup> The real part of the permittivity revealed three prominent resonance peaks around 8.7, 10.0 and 11.8 GHz, indicating the storage of energy in the composites from the electric field. Nevertheless, the lack of stored energy could be observed at other MW frequencies. Meanwhile, the imaginary part of the permittivity signified the dielectric loss factor, denoting the energy dissipation capacity of the material. The appearance of the resonance peaks in the  $\epsilon'$  spectrum was trailed by the disappearance of the resonance peaks in the  $\epsilon''$  spectra.<sup>13</sup> It was affirmed that the proposed composites presented the best energy storage performance around 11.6 GHz, wherein the absorbed energy was consumed for the magnetic domain wall motion.

Fig. 5(b) illustrates the frequency dependent complex relative permeability ( $\mu'$ ) for all composites in the X-band. The real part of the permeability ( $\mu'$ ) exhibited three significant resonance peaks around 8.5, 9.3 and 11.0 GHz, demonstrating the magnetic energy storage capacity of the composites. Alternatively, the imaginary part of the permeability ( $\mu''$ ) revealed the magnetic loss factor around 8.2, 9.0, and 10.5 GHz, which was allocated to the natural resonance frequency  $f_r$ .<sup>13</sup> Besides, the magnetic loss factor was increased at the resonance frequency. It was observed that the substitution of  $\text{Fe}^{3+}$  in the BFCs by  $\text{Nd}^{3+}$  at varied concentrations caused the frequency shift. This disclosure was attributed to the emergence of different phases and porosity in the composites. The imaginary part of the permeability plays an important role in the microwave absorbing property of hexaferrites<sup>29</sup> that are related to the relaxation of domain wall resonance.<sup>30</sup>

### 3.5. Microwave reflection loss

Fig. 6 represents the variation in the reflection loss ( $R_L$ ) of all prepared BFCs as a function of applied MW frequency ( $f$ ). The NdBF0 and NdBF3 composites revealed a similar trend in the  $R_L$  pattern which comprised of two weak absorption bands with values above  $-10$  dB. Meanwhile, the NdBF1 specimen exhibited  $R_L$  value below  $-10$  dB at frequencies range of 9.14–10.18 GHz. Besides, NdBF2 composite disclosed two prominent absorption bands centered around 9.94 and 11.64 GHz with  $R_L$  values of  $-22.94$  and  $-23.82$  dB, respectively. All the observed spectral bands showed a significant shift with varying contents of  $\text{Nd}^{3+}$  ions with the bandwidth of less than 1 dB. In short, the microwave absorption capacity of the proposed BFCs was improved due to  $\text{Nd}^{3+}$  ions substitution assisted enhanced porosity in the structure that in turn could confine the MW. Consequently, the  $\text{Nd}^{3+}$  ions substitution into the BF could change the magnetocrystalline anisotropy direction of the composites, leading to an alteration of the natural resonance frequency. This alteration could finally cause a shift of the MW reflection loss to high-frequency zone as revealed by NdBF2. Interestingly enough, the doped composites enriched in  $\text{Ba}_6\text{Nd}_2\text{Fe}_4\text{O}_{15}$  and  $\text{BaFe}_2\text{O}_4$  phases could absorb microwaves selectively in the X-band.

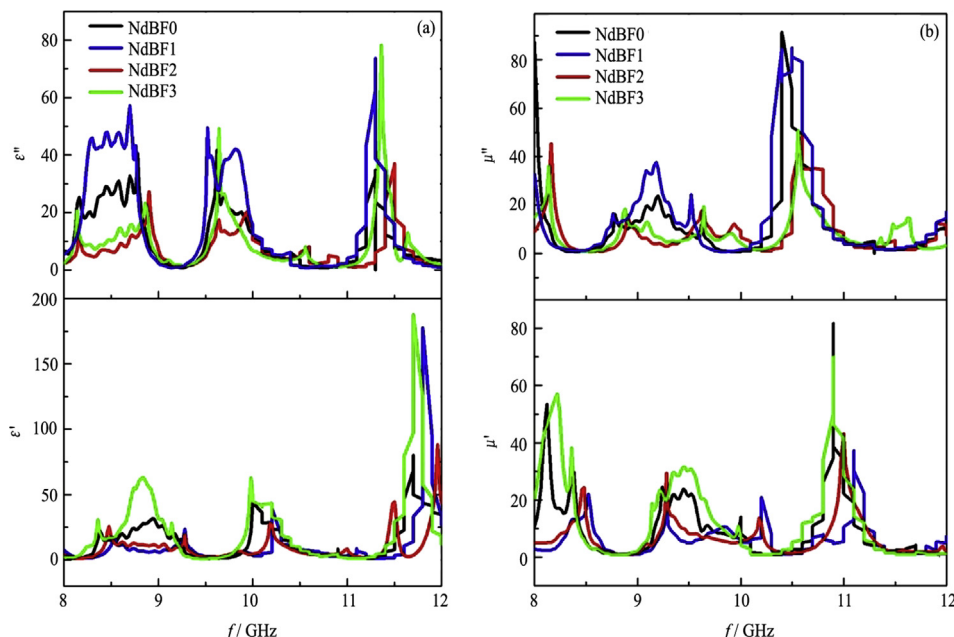


Fig. 5. Frequency-dependent complex relative permittivity (a) and permeability (b) for all NdBFs.

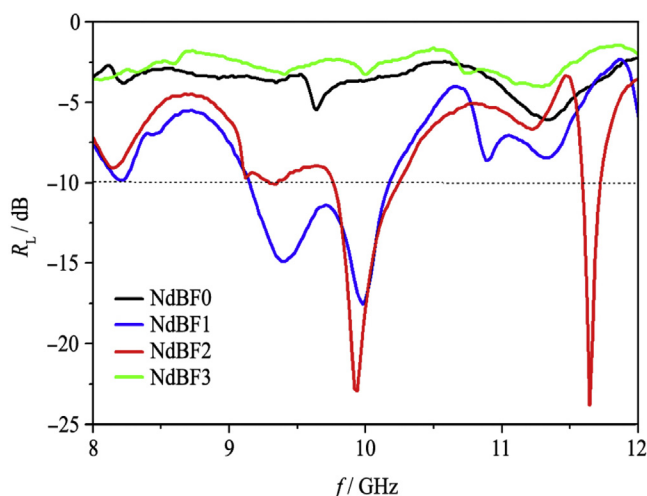


Fig. 6. Frequency-dependent MW reflection loss of all the proposed NdBFs

#### 4. Conclusions

Synthesis and room temperature characterization of some new hexagonal barium ferrite composites with  $\text{Nd}^{3+}$  ions substitution were reported. An appropriate amount of chemical constituents were gradually mixed to prepare these composites following solid-state reaction route. The proposed BFCs reveal excellent capacity of MW absorption in the X-band. EDX spectra reveal the right elemental compositions of the prepared BFCs and presence of Nd in the structures. The surface morphology, structure, magnetic properties and MW absorption ability of the prepared BFCs are strongly influenced because of the inclusion of  $\text{Nd}^{3+}$  ions into the crystalline lattice. The XRD pattern discloses the formation of new crystalline phases of  $\text{Ba}_6\text{Nd}_2\text{Fe}_4\text{O}_{15}$  and  $\text{BaFe}_2\text{O}_4$  due to the incorporation of  $\text{Nd}^{3+}$  ions into the BFCs structure. SEM images of the studied BFCs exhibit highly rough and porous surface morphology. At 2 mol% of  $\text{Nd}^{3+}$  ions, the saturation magnetization of the BFCs

displays insignificant alteration. The complex relative permittivity and permeability of the studied BFCs are found to depend appreciably on the MW frequencies. Furthermore, the achieved low reflection loss at 9.94 and 11.64 GHz frequencies with selective MW absorption shown by the proposed BFCs may be advantageous for diverse device applications in the X-band.

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