



## Original article

Equilibrium synthesis and magnetic properties of BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> nanocomposite prepared by co precipitation methodC. Thirupathy<sup>a</sup>, S. Cathrin Lims<sup>a</sup>, S. John Sundaram<sup>a,\*</sup>, Ahmed Hossam Mahmoud<sup>b</sup>, K. Kaviyarasu<sup>c,d</sup><sup>a</sup> Department of Physics, Sacred Heart College (Autonomous), Tirupattur - 635601, Tirupattur District, Tamil Nadu, India<sup>b</sup> Department of Zoology, College of Science, King Saud University, Riyadh, Saudi Arabia<sup>c</sup> UNESCO-UNISA Africa Chair in Nanosciences/Nanotechnology Laboratories, College of Graduate Studies, University of South Africa (UNISA), Muckleneuk Ridge, PO Box 392, Pretoria, South Africa<sup>d</sup> Nanosciences African Network (NANOAFNET), Materials Research Group (MRG), iThemba LABS-National Research Foundation (NRF), 1 Old Faure Road, 7129, PO Box 722, Somerset West, Western Cape Province, South Africa

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## ABSTRACT

The hard and soft magnetic nanocomposites (BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub>) at the ratio 1:1 were prepared by simple mixing and heat treatment. XRD peak indicates the formation of good crystalline quality of the prepared sample. The FTIR spectra reveals the bands for metal oxygen stretching which ranges from 584 cm<sup>-1</sup> to 540 cm<sup>-1</sup> forms the tetrahedral shape in nature. However, the hexaferrite site ranges from 580 cm<sup>-1</sup> to 440 cm<sup>-1</sup> and this bands assign to Ba-O and Fe-O bonds respectively. Furthermore, the UV-visible range, the observed absorption peak indicates that the prepared sample color is congo red, the characteristic absorption peaks at ~739 nm for BaFe<sub>12</sub>O<sub>19</sub>, 698 nm for NiFe<sub>2</sub>O<sub>4</sub> and 780 nm for BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub>. Overall, the ferrite powder seems agglomerated, but the microstructure shows its respective structure. The particle size of BaFe<sub>12</sub>O<sub>19</sub> (hexagonal) is 150 nm, again the particle size of NiFe<sub>2</sub>O<sub>4</sub> (cubic) is 89 nm apparently. Finally, the structure of composite (BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub>) material seems to achieve intermediate and its particle size is 120 nm correspondingly.

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## 1. Introduction

Ferrites have numerous industrial applications with excellent electric and magnetic properties (Zhidong et al., 2006), which shows promising properties in radar absorption, microwave absorption, magneto optic or perpendicular recording media, magnetic device, transformers and spintronics (Remya et al., 2016). Therefore, the magnetic nanocomposites have obvious advantage over nonmagnetic materials, these nanocomposite materials consist of hard and soft magnetic phase (Prakash et al., 2016). Which make them suitable for many different applications which include permanent magnet too (Kadi and Mohamed, 2014). The magnetic fillers of ferrite materials are spinal ferrite and hexaferrite it shows

a loss nearer to dipole relaxation and ferromagnetic resonance (Couture et al., 2017). In addition, spinal ferrites can be used in EMR absorption upto 3 GHz frequency range, but hexagonal ferrite use up to the frequency range of (2–18 GHz) because of uniaxial anisotropic property (Sun et al., 2017). Hexaferrites are sub classified in different categories M,U,W,X,Y and Z with general formula like BaFe<sub>12</sub>O<sub>19</sub>; Ba<sub>4</sub>Me<sub>2</sub>Fe<sub>36</sub>O<sub>60</sub>; BaMe<sub>2</sub>Fe<sub>16</sub>O<sub>22</sub>; Ba<sub>2</sub>Me<sub>2</sub>Fe<sub>28</sub>O<sub>46</sub>; BaMe<sub>2</sub>Fe<sub>12</sub>O<sub>22</sub> and Ba<sub>2</sub>Me<sub>2</sub>Fe<sub>2</sub>O<sub>41</sub>. Where Me- divalent cation of first transition series like Ni, Co, Zn and Mg. Among different system these combining hexagonal ferrite with spinal/inverse spinal ferrite have obtained noticeable attention by researcher (Bashir et al., 2019a). In order to get high crystalline mono-domain particle barium hexaferrite and nickel ferrite different synthesis techniques were adapted they are sol-gel (Bashir et al., 2019b), co-precipitation (Williams et al., 2018), solvothermal method (Tyagi et al., 2018), organic acid precursor method (Ali et al., 2017) etc., In this work the present investigation deals with barium and nickel ferrite nanocomposite followed by co-precipitation method. Due to this method is low cost and easy technique to mass production compared with other applications (Kennedy et al., 2016). Therefore, the established nanocomposites were characterized by X-ray diffraction (XRD) pattern will reveal the crystalline nature

\* Corresponding author.

E-mail address: [johnsundaram@shcpt.edu](mailto:johnsundaram@shcpt.edu) (S. John Sundaram).  
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of nanocomposites. FTIR spectrum shows the presence of functional group and vibrational bands at the surface of BaFe<sub>12</sub>O<sub>19</sub> / NiFe<sub>2</sub>O<sub>4</sub> nanocomposite. Finally, scanning electron microscopy (SEM) shows the morphology growth of the samples with EDAX will explain to confirm the elemental composition which is present in the compound. Lastly, VSM will show the magnetic property of the entire samples were reported in detail.

## 2. Experimental procedure

### 2.1. Synthesis of BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> and BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> nanocomposites

In the present investigation barium nitrate (Ba(NO<sub>3</sub>)<sub>2</sub>), ferric nitrate monohydrate (FeH<sub>18</sub>N<sub>3</sub>O<sub>18</sub>)<sub>12</sub>; nickel nitrate (Ni(NO<sub>3</sub>)<sub>2</sub>) and ammonia solution is used for synthesizing BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> and BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> nanocomposite by co-precipitation method. 1% of barium nitrate and 12% of ferric nitrate monohydrate of molecular weight is taken (I). The, 1% nickel nitrate and 2% of ferric nitrate monohydrate of molecular weight taken (II) separately. Add the two mixture separately in a 50 ml of water, let these mixtures stir for 2 h continuously until the compound's mixtures and dissolves completely. In that condition, after 2 h ammonia solution was added drop by drop until the precipitate forms then allowed to stir for an hour, finally, the brown precipitate deposits at the bottom and the crystal-clear liquid will deposit on the brown precipitate. Then the solution is completely washed with water and ethanol for several times, the washed nanoparticles were kept in hot air oven for 24 h at 120 °C. The dried, brown rocky like nanomaterial forms. By using the mortar, the brown rocky material was grained well. The grinded particles were put into a silicon crucible kept in a muffled furnace for 2 h up to 800 °C. Therefore, the prepared BaFe<sub>12</sub>O<sub>19</sub> and NiFe<sub>2</sub>O<sub>4</sub> nanoparticles has

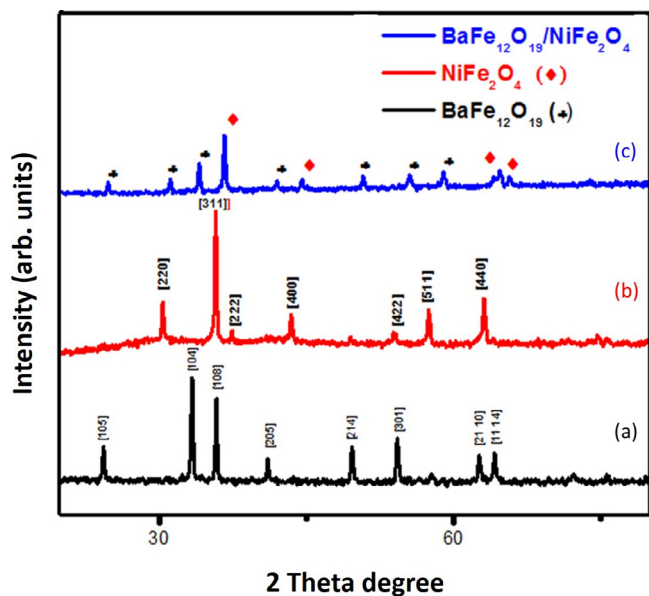


Fig. 1. XRD pattern of (a) BaFe<sub>12</sub>O<sub>19</sub>; (b) NiFe<sub>2</sub>O<sub>4</sub> and (c) BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub>.

been done by mixing the hard and soft ferrite in weight ratio of 1:1 then the sample was grained and heated for 100 °C respectively.

## 3. Results and discussion

### 3.1. X-ray powder diffraction (XRD)

The XRD pattern of barium hexaferrite (BaFe<sub>12</sub>O<sub>19</sub>) and nickel ferrite (NiFe<sub>2</sub>O<sub>4</sub>) nanopowders were calcinated under 800 °C for 2 h as shown in Fig. 1. The hard and soft magnetic nanocomposites (BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub>) at the ratio 1:1 were prepared by simple mixing and heat treatment. The well-defined sharp peak indicates the formation of good crystalline quality of the prepared sample. The peaks in BaFe<sub>12</sub>O<sub>19</sub> corresponds to hexagonal structure ( $a = b \neq c$ ) according to the (JCPDS file number 84–0757) and NiFe<sub>2</sub>O<sub>4</sub> corresponds to cubic structure ( $a = b = c$ ) according to (JCPDS file number 86–2267). All these diffraction peaks for BaFe<sub>12</sub>O<sub>19</sub> (♣) and NiFe<sub>2</sub>O<sub>4</sub> (◆) and no other impurity peaks has been observed even after simple mixing heat treatment. Therefore, the XRD peak positions of those composites BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> has both hard and soft magnetic ferrite phase and this confirms the presence of both BaFe<sub>12</sub>O<sub>19</sub> and NiFe<sub>2</sub>O<sub>4</sub> exist in nature (Kaviyarasu et al., 2012). The average grain size of powder sample was estimated from full width at half maximum (FWHM) by using Debye Scherer formula to determine the crystallite size, dislocation, lattice strain and inter planner distance were listed in Table 1.

### 3.2. Fourier transform infra-red spectroscopy (FTIR)

The FTIR spectra were recorded in mid-range 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> for barium hexaferrite (BaFe<sub>12</sub>O<sub>19</sub>) and nickel ferrite (NiFe<sub>2</sub>O<sub>4</sub>) and the composite of BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> with the ratio 1:1 as shown in the Fig. 2. The FTIR spectra reveals the bands for metal oxygen stretching which ranges from 584 cm<sup>-1</sup> to

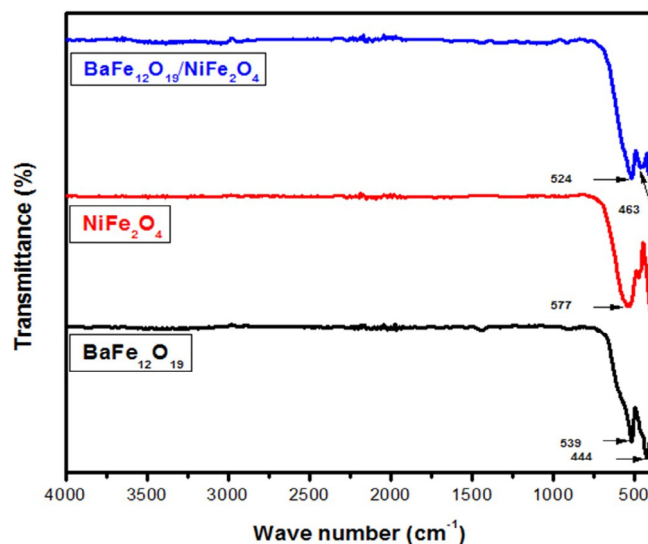
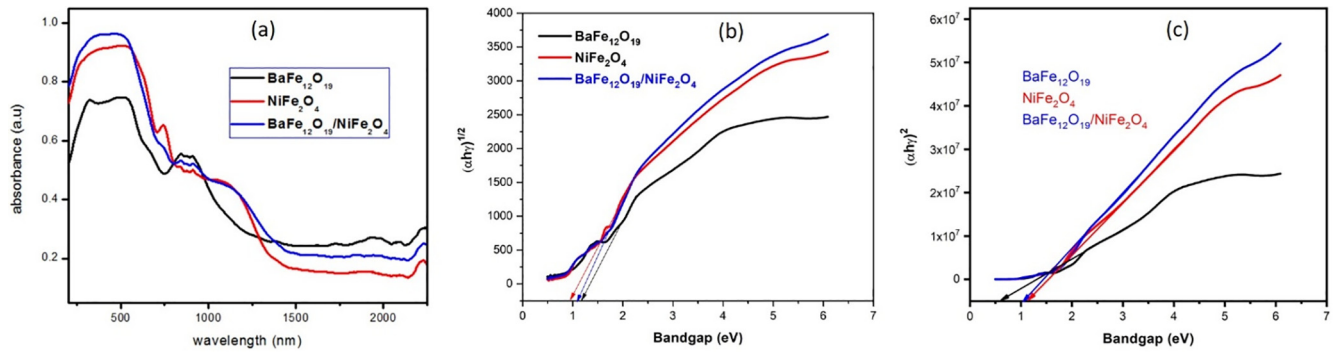


Fig. 2. FTIR spectrum of BaFe<sub>12</sub>O<sub>19</sub>; NiFe<sub>2</sub>O<sub>4</sub> and BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub>.

Table 1

Powder XRD pattern of (a) BaFe<sub>12</sub>O<sub>19</sub>, (b) NiFe<sub>2</sub>O<sub>4</sub> and (c) BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub>.

Sample	BaFe <sub>12</sub> O <sub>19</sub>	NiFe <sub>2</sub> O <sub>4</sub>	BaFe <sub>12</sub> O <sub>19</sub> /NiFe <sub>2</sub> O <sub>4</sub>
Crystallite size	41 nm	40 nm	31 nm
Dislocation ( $\delta$ ) = $\frac{1}{L^2}$	$0.493 \times 10^{15}$	$0.625 \times 10^{15}$	$1.040 \times 10^{15}$
Lattice train ( $\Sigma$ ) = $\frac{4\cos\theta}{\lambda}$	$0.9371 \times 10^{-3}$	$0.9935 \times 10^{-3}$	$1.0891 \times 10^{-3}$
Inter planner distance (d)	$0.952 \times 10^{10}$	$0.921 \times 10^{10}$	$2.0033 \times 10^{10}$



**Fig. 3.** (a) UV-visible spectrum of BaFe<sub>12</sub>O<sub>19</sub>, NiFe<sub>2</sub>O<sub>4</sub>, BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> nanocomposites, (b) Tauc plot for indirect transition, (c) direct transition.

**Table 2**

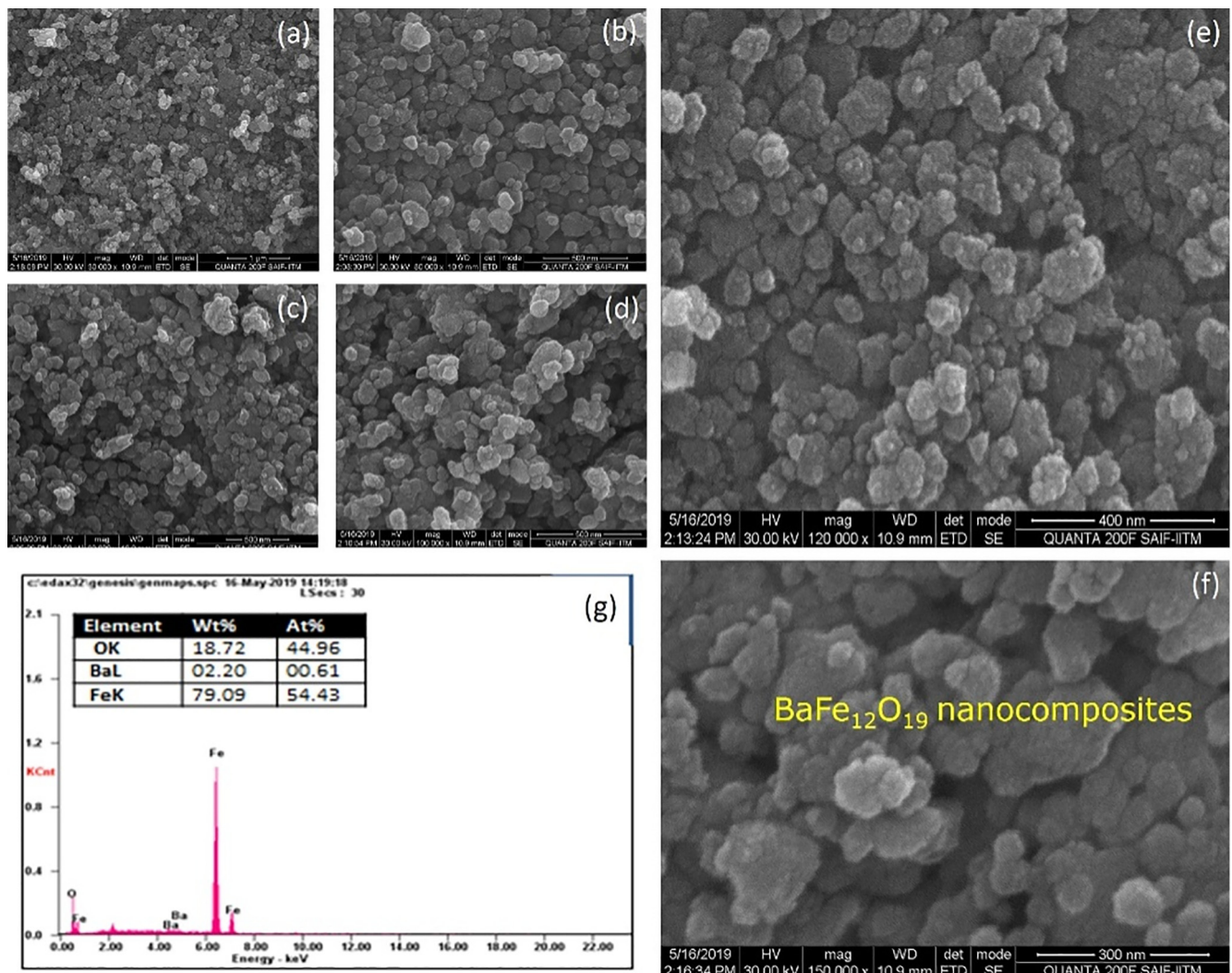
Tauc plot of BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> and BaFe<sub>12</sub>O<sub>19</sub>/NiFe<sub>2</sub>O<sub>4</sub> nanocomposites for (a) direct transition, (b) indirect transition.

Sample	Direct bandgap energy (eV)	Indirect bandgap energy (eV)
BaFe <sub>12</sub> O <sub>19</sub>	1.72	1.41
NiFe <sub>2</sub> O <sub>4</sub>	1.40	1.16
BaFe <sub>12</sub> O <sub>19</sub> /NiFe <sub>2</sub> O <sub>4</sub>	1.57	1.05

540 cm<sup>-1</sup> forms the tetrahedral shape. The hexaferrite site ranges from 580 cm<sup>-1</sup> to 440 cm<sup>-1</sup> and this bands assign to Ba-O and Fe-O bonds. The other bands like CO<sub>2</sub>-, -OH peaks are completely vanished under calcination at 800 °C for 2 h.

### 3.3. UV-visible spectrometer

The optical properties are closely related to the atomic structure, electronic bond patterns. The optical properties of nano



**Fig. 4.** (a-g). SEM with EDAX image for BaFe<sub>12</sub>O<sub>19</sub> nanocomposites.

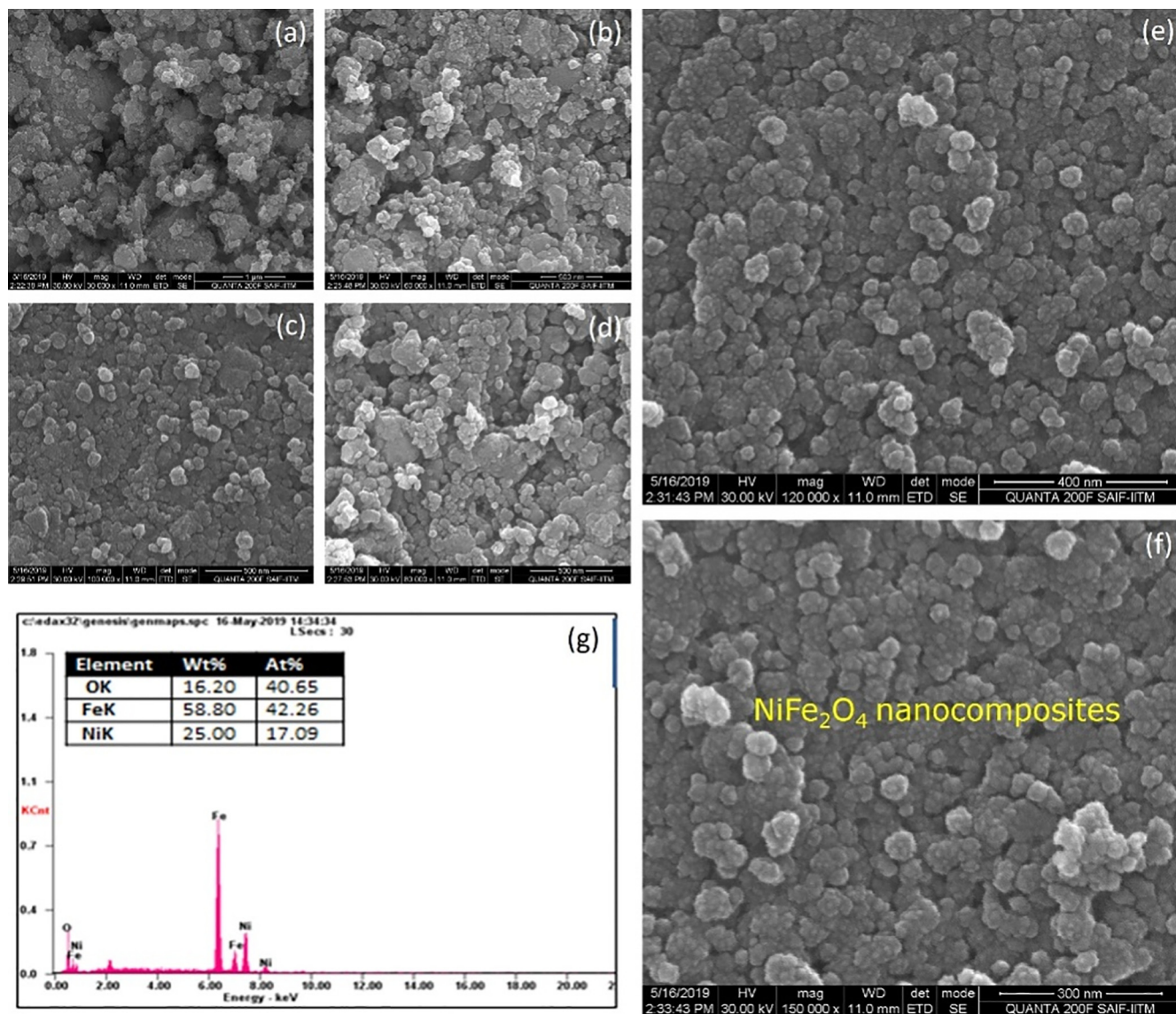


Fig. 5. (a-g). SEM with EDAX image for  $\text{NiFe}_2\text{O}_4$  nanocomposites.

composites were analyzed from wavelength ranges from 200 to 2500 nm as shown in Fig. 3(a). At UV-visible range the observed absorption peak indicates that the prepared sample color is congo red, the characteristic absorption peaks at  $\sim 739$  nm for  $\text{BaFe}_{12}\text{O}_{19}$ ; 698 nm for  $\text{NiFe}_2\text{O}_4$  and 780 nm for  $\text{BaFe}_{12}\text{O}_{19}/\text{NiFe}_2\text{O}_4$  (MobeenAmanulla et al., 2018). Therefore, the energy gap can be obtained from *Tauc plot*,  $\alpha h\nu = A(h\nu - E_g)^M$ ; where,  $\alpha$  - is the absorption co-efficient;  $h\nu$  - is photon energy;  $E_g$  - is the optical energy band gap; A - is a constant; M - is the characteristics of transition. At near infrared range transmittance occurred so the material can be used in radar absorption or microwave absorption applications (Kaviyarasu et al., 2017). The energy gap can be obtained from *Tauc plot* as shown in Fig. 3(b, c). The *Tauc plots* were plotted for direct and indirect transition and the band gap were listed in the Table 2.

#### 3.4. Scanning electron microscope & EDAX

The scanning electron microscopic image  $\text{BaFe}_{12}\text{O}_{19}$ ,  $\text{NiFe}_2\text{O}_4$ -ferrite nanoparticles as well as hard and soft magnetic phase mixed ( $\text{BaFe}_{12}\text{O}_{19}/\text{NiFe}_2\text{O}_4$ ) in the ratio of 1:1 as shown in the Figs. 4–6,

the image says that the ferrites and composites are not well dispersed because of its unfavorable condition (Kaviyarasu et al., 2013). Ferrite powder seems agglomerated but the micro structure shows its respective structure (Sazelee et al., 2018). The particle size of  $\text{BaFe}_{12}\text{O}_{19}$  (hexagonal) is 150 nm as shown in Fig. 4(a-g), the particle size of  $\text{NiFe}_2\text{O}_4$  (cubic) is 89 nm as shown in Fig. 5(a-g) and the structure of composite ( $\text{BaFe}_{12}\text{O}_{19}/\text{NiFe}_2\text{O}_4$ ) material seems to achieve intermediate and its particle size of is 120 nm Fig. 6(a-g). From the image the elongated shape says that the composition is insufficient to the formation of nanoparticles (Salwa et al., 2014). The spherical particles will also disappear by increasing its temperature (Jesudoss et al., 2017). The data which is generated by energy dispersive X-ray microanalysis has some peaks corresponds to different elements that are present in the sample and each element has a specific peak of unique energy, all comprehensively documented (Ming and Liang Gao, 2012). The EDAX spectra of  $\text{BaFe}_{12}\text{O}_{19}$ ;  $\text{NiFe}_2\text{O}_4$  and  $\text{BaFe}_{12}\text{O}_{19}/\text{NiFe}_2\text{O}_4$  as shown in Fig. 6, the peak corresponding to the elements Ba, Ni, Fe and O were observed in ferrite nano particles and the peaks of the elements Ba, Ni, Fe and O were observed in composite (Virik et al., 2011). The observed percentage of Ba, Ni, Fe and O match with

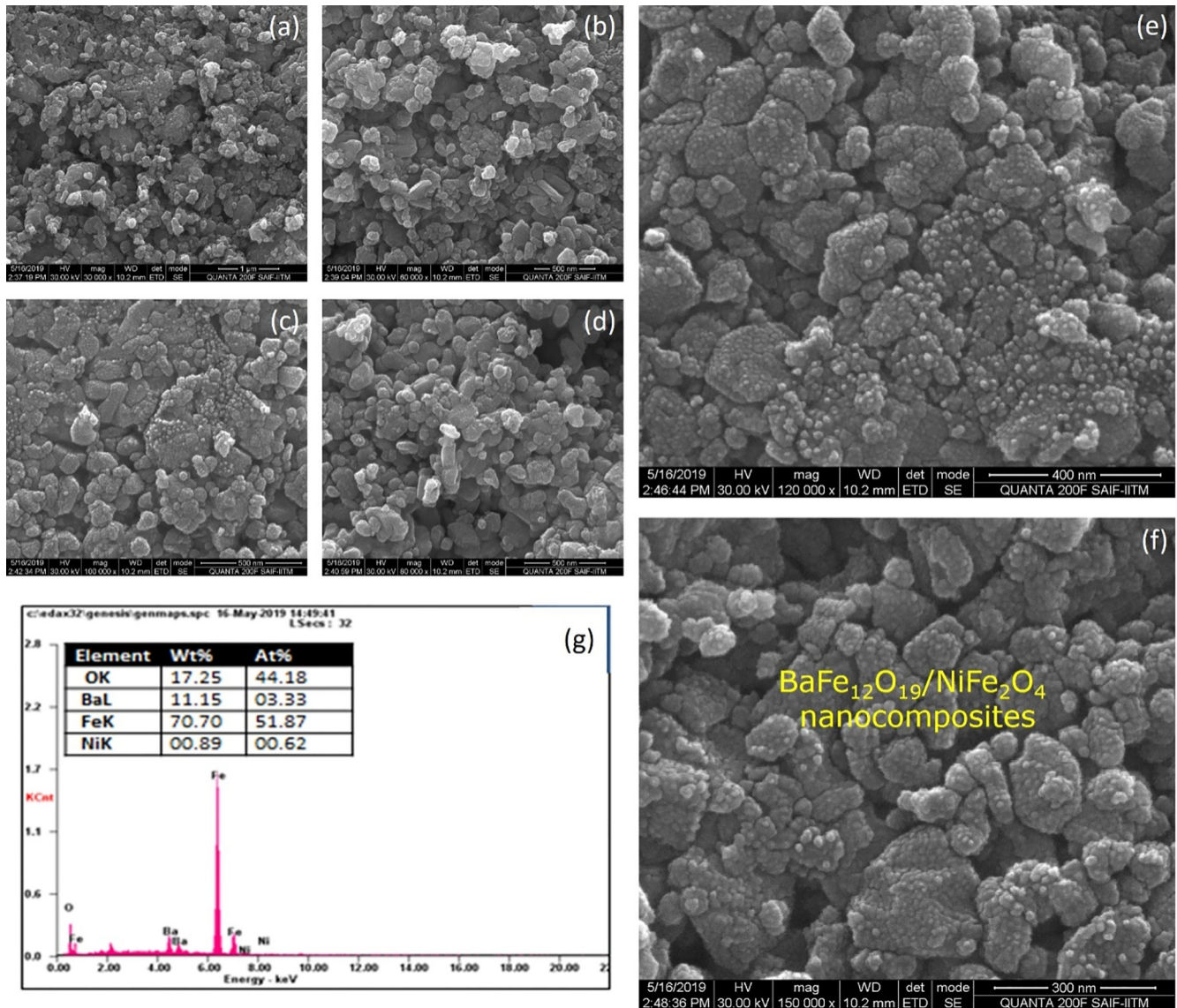


Fig. 6. (a-g). SEM with EDAX image for  $\text{BaFe}_{12}\text{O}_{19}/\text{NiFe}_2\text{O}_4$  nanocomposites.

Table 3

VSM image of  $\text{BaFe}_{12}\text{O}_{19}$ ,  $\text{NiFe}_2\text{O}_4$  and  $\text{BaFe}_{12}\text{O}_{19}/\text{NiFe}_2\text{O}_4$  nanocomposites.

S. No	Properties	$\text{BaFe}_{12}\text{O}_{19}$	$\text{NiFe}_2\text{O}_4$	$\text{Ba}_2\text{Ni}_2\text{Fe}_{12}\text{O}_{22}$
1	Coercivity (Hci)	6246.3 G	190.93 G	824.04 G
2	Saturation Magnetization (Ms)	0.41713 emu	0.21146 emu	0.38348 emu
3	Retentivity (Mr)	0.19876 emu	$48.071\text{E}^{-3}$ emu	0.12533 emu

the amount of respective precursor without any characteristic peak (Gurbuz et al., 2012). The atomic percentage and weight percentage were also listed.

### 3.5. Vibrating sample magnetometer (VSM)

The magnetic properties were studied using vibrating sample magnetometer at room temperature and various magnetic properties were listed using the curve below in the Table 3 (Sivakumara et al., 2012). Hysteresis loop are important in the construction of several electrical devices as shown in Fig. 7(a-d).

## 4. Conclusion

In summary, the structure of nanocomposites  $\text{BaFe}_{12}\text{O}_{19}/\text{NiFe}_2\text{O}_4$  seems to achieve intermediate and its particle size of is 120 nm. From the XRD peak positions have confirmed the presence of both  $\text{BaFe}_{12}\text{O}_{19}$  and  $\text{NiFe}_2\text{O}_4$  exist in the composites. The FTIR spectra reveals the bands for metal oxygen stretching which ranges from  $584\text{ cm}^{-1}$  to  $540\text{ cm}^{-1}$  and the other bands like  $\text{CO}_2^-$ ,  $-\text{OH}$  peaks were completely vanished under calcination at  $800\text{ }^\circ\text{C}$  for 2 h. SEM image were elongated shapes that the composition is insufficient to the formation of nanoparticles. Therefore, the spherical particles have disappeared by increasing the temperature.

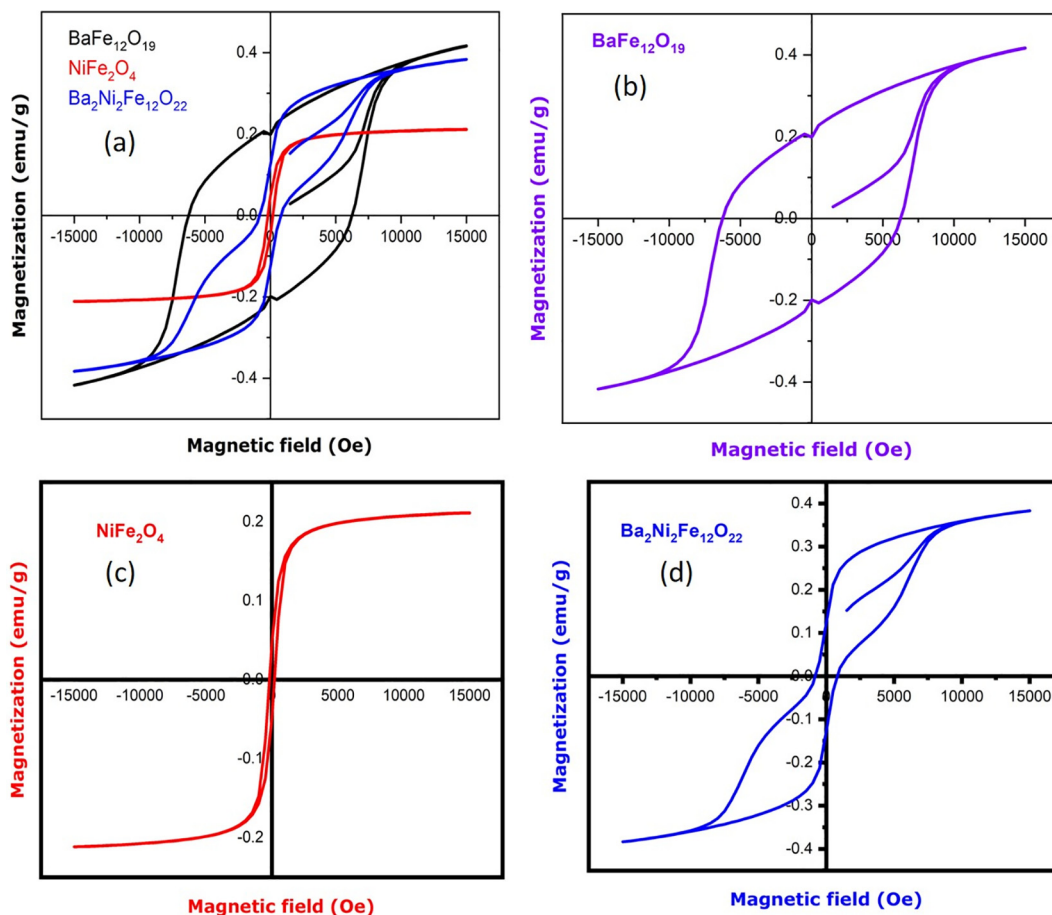


Fig. 7. (a) VSM image of different nanocomposites; (b)  $\text{BaFe}_{12}\text{O}_{19}$ ; (c)  $\text{NiFe}_2\text{O}_4$ ; (d)  $\text{BaFe}_{12}\text{O}_{19}/\text{NiFe}_2\text{O}_4$  nanostructures.

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