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RESEARCH ARTICLE

STUDY OF STRUCTURAL AND DIELECTRIC PROPERTY OF ($BaFe_{12}O_{19}$ - ZrO_2) NANOCOMPOSITE MATERIAL VIA SOL-GEL METHOD

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ABSTRACT

The nano powders of Bariumhexaferrite and zirconium oxide in different weight ratios had been prepared by sol-gel method. The formation of pure crystallized $BaFe_{12}O_{19}$ and ZrO_2 nanoparticles were occurred, when the precursor were calcined at 800^0C and 1000^0C for 3hrs. The vibrational, structural, morphological, thermal and dielectric properties have been determined by Fourier Transform Infrared Spectroscopy (FTIR), X-ray Diffraction (XRD), Scanning Electron Microscope (SEM), Thermal analysis (TG/DTA), and scalar analyzer. FTIR spectra exhibited band in the range of 473-550 cm^{-1} which confirmed the presence of ($BaFe_{12}O_{19}$ – $ZrO_{2)}$ nanoparticles. Nanoparticles of barium hexaferrite and zirconium oxide had average crystalline size between (32nm- 44nm) respectively. The SEM image revealed that powder is well crystallized and the particles are elongated hexagonal structure. The dielectric studies showed the frequency, dielectric constant and dielectric loss in the range of 11.5GHz, 24.6 and 0.0064.

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INTRODUCTION

The magneto dielectric materials had greater scope in technological developments and wireless communication .this type of material exhibited the properties of both magnetic and dielectric at the same time. Ferrites are the ceramic compounds which have high electrical resistivity than the metallic ferromagnetic materials. Ferrites reduce the eddy current losses and also absorb the electromagnetic field penetration. Ferrites can be divided into three categories namely spinel, hexagonal and garnets according to their crystal lattice structure (Virendra Pratap Singh *et al*., 2014). The ferrites have been the subject of continuous interest as well as the focus of many research studies in the last few decades to be utilized as microwave absorbers in the gigahertz (GHz) range due to its high saturation magnetization, large anisotropy field, excellent chemical stability and good microwave absorption properties.M-type Barium hexaferrite (BaFe12O19) particle size on microwave absorption at X-band (8.2-12.4 GHz) (Wei Chen *et al*., 2012). Many methods, including co-precipitation (Husan-Fu and Yun, 2013), sol–gel (Perdamean Sebayang *et al*., 2016; Xiuna Chen and Guolong Tan, 2010), polymer precursor method (Abedini Khorrami *et al*., 2013), sol-gel method have been developed to produce ferrite nanoparticles.

Zirconiumoxide (ZrO_2) is a material of importance due to its mechanical and electrical properties, high dielectric constant and wide band gap. In this work, Magnetic material (BaM) has combined with dielectric material ZrO_2 in different weight proportion to enhance their property.

MATERIALS AND METHODS

($BaFe_{12}$ O_{19}-ZrO_2) nanoparticles were prepared by using sol-gel method in different weight ratios such as T1&T2 (100:100), T3(90:10), T4(70:30),T5(50:50), T6(30:70), T7(10:90). Appropriate amount of $Ba(NO_3)_2$, $Fe(NO_3)_3.9H_2O$, $ZrOCl_2$ get dissolved into an unionized distilled water separately and mixed all the solution sequentially stirred continuously for an hour, then add glucose which is a fuel and it gives requisite energy to initiate exothermic reaction, add 4ml of ethylene glycol which is act as a reducing agent and finally add the oxalic acid as a gelling agent. The solution mixture is stirred continuously for 2 hours at 80^0C. As water evaporated, the solution become a very viscous brown dried gel, it again dried at 110^0 C for 24hours to get a precursor powder which is used to take FTIR and TG/DTA. Then precursor is again calcined at 800^0C and 1000^0 C for 3hours in a furnace to get a fine powder which is used for other characterization.

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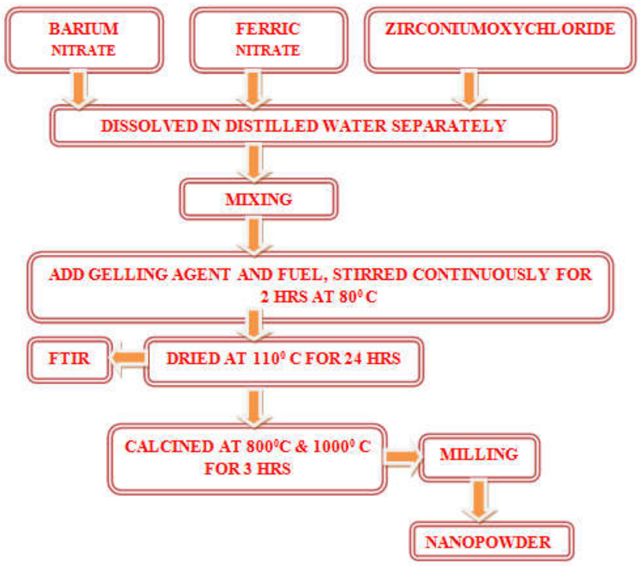

Fig. 1. Flowchart for synthesis of ($BaFe_{12}O_{19}$-ZrO_2) nanoparticles

RESULTS AND DISCUSSION

TG/DTA

The DTA–TG curve of the $BaFe_{12}O_{19}$ powder is shown in Fig. 2. In the temperature range 50–136^{0}C due to desorption of water molecules and solvents. DTA curve shows a broad exothermic peak at 375.51 ^{0}C, a sharp endothermic peak at about 526 ^{0}C and another exothermic peak at 755^{0}C and 835^{0}C. These peaks are due to the decomposition of gel and nitrate molecules. The TG curve of the $BaFe_{12}O_{19}$ shows a gradual weight loss of about 88% in the temperature range 50–152^0 C and a sharp $BaFe_{12}O_{19}$weight loss of about 61% in the temperature range 152–376 ^{0}C show a further weight loss of about 49% in the temperature range of 376- 528 ^{0} C and the final stage of weight loss is about 26% in the temperature range 528–800 ^{0}C. $BaFe_{12}O_{19}$ powder has high crystalline temperature, due to addition of fuel, the crystalline temperature attained above 800^{0}C (Abhishek Kumar *et al.*, 2013).

Fourier Transform Infrared Spectroscopy

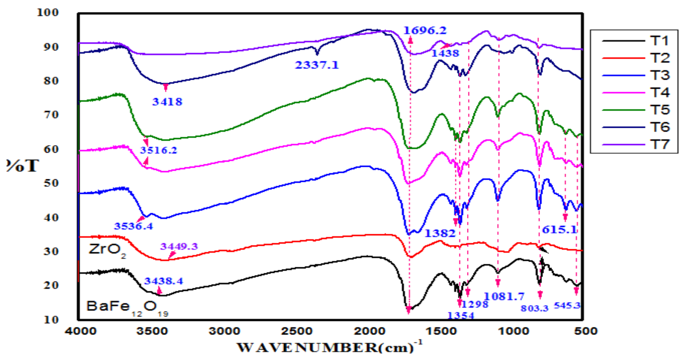

Fig. 3. FTIR spectra of ($BaFe_{12}O_{19}$-ZrO_2) nanoparticles

FTIR spectra of ($BaFe_{12}O_{19}$ –ZrO_2) nanoparticles of different weight ratios obtained in the wave number range of 500-4000 cm^{-1}. The characteristics peaks observed at the wave number of 1382 cm^{-1}, 1354 cm^{-1} corresponding to bending of C-H bond. While the bands located at 1298 cm^{-1}, 1081 cm^{-1} symmetric stretch of nitro groups. The absorption bands at 3516 cm^{-1}, 3536cm^{-1} and 3438 cm^{-1} are attributed to the stretching of water molecules O-H bond,. The strong absorption observed between 545cm^{-1}- 803 cm^{-1} belong to the metal oxide stretching vibration of ($BaFe_{12}O_{19}$ –ZrO_2) nanoparticles (Yaowen *et al.*, 2009; Lim *et al.*, 2013).

XRD

XRD pattern of ($BaFe_{12}O_{19}$-ZrO_2) nanoparticles in different ratios at 800^{0}C and 1000^{0}C are shown in Figure (4,5). The diffraction peaks are appeared in the XRD pattern can be indexed with the standard pattern for $BaFe_{12}O_{19}$ (JCPDS NO: 84-0757) and ZrO_2 (JCPDS NO:65-1024). The average particle sizes of different phases were determined from the line widths of diffraction peaks using scherrer equation.

$D=K\,\lambda/\beta\cos\theta$
D- Grain diameter.
B- Half intensity width of relevant diffraction.

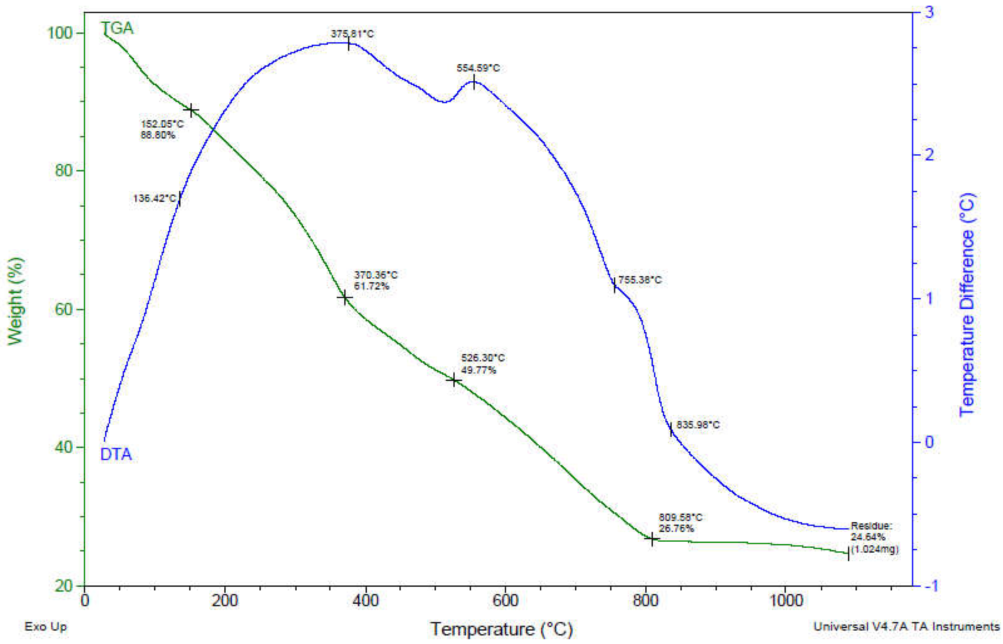

Fig. 2. TG/DTA curve for ($BaFe_{12}O_{19}$)

λ- X-ray wavelength.
θ- Diffraction angle.

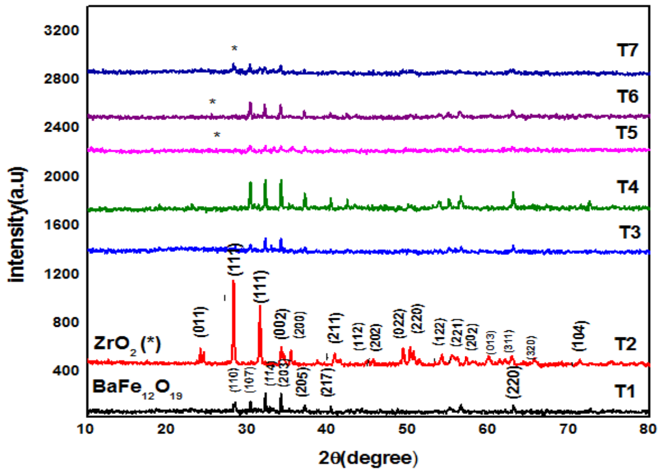

Fig. 4. XRD spectra of ($BaFe_{12}O_{19}$-ZrO_2) at 800⁰C

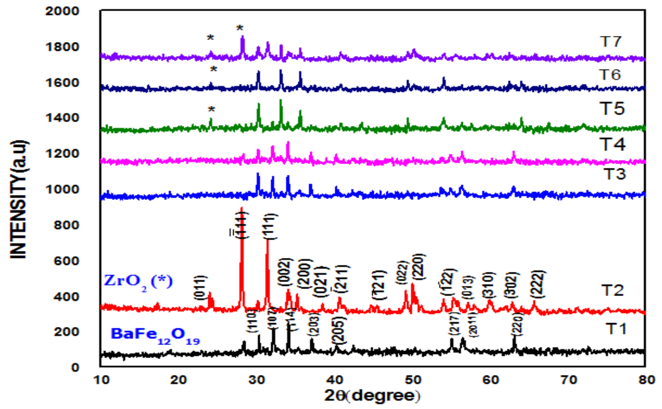

Fig. 5. XRD spectra of ($BaFe_{12}O_{19}$-ZrO_2) at1000⁰C

The average crystallite size for the resulting nanopowder are tabulated below

Table 1. Crystallite size of ($BaFe_{12}O_{19}$-ZrO_2) nanoparticles in different weight ratios at 800^{0}C

Sample	Weight ratios (%)	Crystallite size (nm)
T1($BaFe_{12}O_{19}$)	100:100	32
T2(ZrO_2)	100:100	39.54
T3($BaFe_{12}O_{19}$-ZrO_2)	90:10	33.2
T4	70:30	34
T5	50:50	35
T6	30:70	37
T7	10:90	38.14

Table 2. Crystallite size of ($BaFe_{12}O_{19}$-ZrO_2) nanoparticles in different weight ratios at 1000^{0}C

Samples	Weight ratios (%)	Crystallite size(nm)
T1($BaFe_{12}O_{19}$)	100:100	34
T2(ZrO_2)	100:100	42.41
T3($BaFe_{12}O_{19}$-ZrO_2)	90:10	38.19
T4	70:30	39.60
T5	50:50	40
T6	30:70	42.26
T7	10:90	44.19

The average crystallite sizes for the resulting nanopowder are in the range of (32 - 44) nm. By comparing the XRD pattern and tabulation of these two temperatures suggests that, when the calcinations temperature increased, the particle size also increased. The XRD measurement revealed the diphase in the samples (T5, T6, T7) and indicated the presence of both ($BaFe_{12}O_{19}$-ZrO_2). Hence, it acted as a nanocomposite material (Meng *et al.*, 2014; Wei Chen *et al.*, 2012).

Scanning Electron Microscope (SEM)

The Morphologies observed by SEM for the powders produced in different weight ratios at calcined temperature of 1000^{0}C are shown in fig6. The particles are well-crystallized.

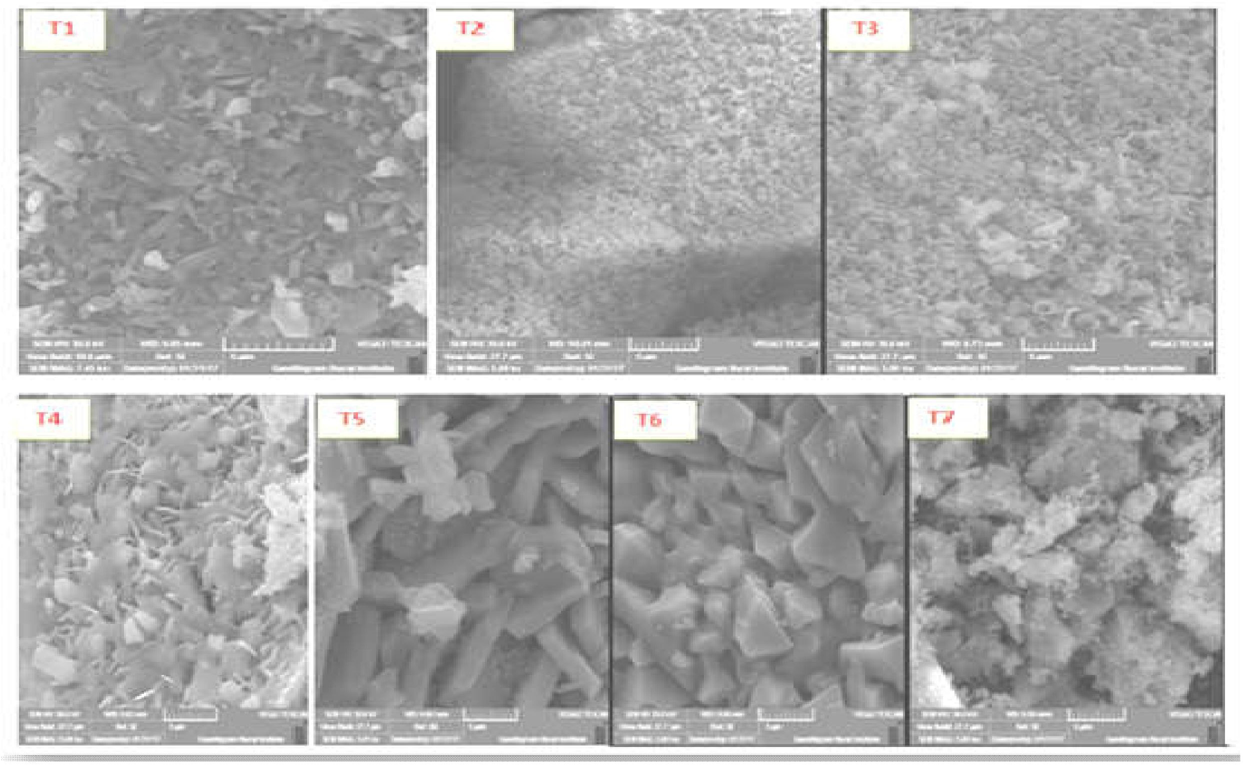

Fig. 6. SEM images of ($BaFe_{12}O_{19}$-ZrO_2) nanoparticles in different weight ratios at 1000^{0}C

Table 6. Dielectric property of ($BaFe_{12}O_{19}$ - ZrO_2) nanoparticles

Sample Name	Frequency (GHz)	ε_r	Tanδ (10^{-3})
T1	11.47800	20.84	1.176
T3	11.48375	24.61	6.453
T4	11.4571875	21.31	4.688
Note: T2, T5, T6, T7 didn't get any resonance due to non-sinterability.			

These particles are tightly compacted with each other, the shape of the sample T1 ($BaFe_{12}O_{19}$) is hexagonal, for the sample T2&T3 (ZrO_2, $BaFe_{12}O_{19}$-ZrO_2) are in spherical shape, where as hexagonal with needle shape structure are found in the sample T4 ($BaFe_{12}O_{19}$-ZrO_2) .the shape of the samples T5&T6 ($BaFe_{12}O_{19}$-ZrO_2) are elongated hexagonal structure. The sampleT7 (($BaFe_{12}O_{19}$-ZrO_2) exhibited the shape of hexagonal along with tiny spherical particles, which confirmed the presence of nano-composite material. The result of XRD and SEM are matched with each other (Janasi *et al*., 2002; Madhusudhana *et al*., 2014).

Dielectric property

There are three characteristics of dielectric properties to be evaluated are frequency, dielectric constant (ε_r) and dielectric loss (tanδ) have been identified by using scalar analyser (Aeroflex IFR6823). The result of the samples (T1,T3,T4) are tabulated below

From the tabulation, the material acted in maximum frequency and dielectric constant attained at sample T3 is 11.5 (GHz) and 24.61. Dielectric loss decreases with increase in frequency so, it has low dielectric loss in the range of 0.0064 (Chen *et al*., 2004; Kajfez, 2001; Jarvis *et al*., 2001).

Conclusion

Barium hexaferrite and zirconium oxide nanopowder were synthesized by sol-gel method.TG/DTA curve showed the crystalline temperature of barium hexaferrite is above 800^0C. FTIR spectra exhibit band in the range of (500-800 $cm^{-1)}$ which confirmed the presence of ($BaFe_{12}O_{19}$ - ZrO_2) nanoparticles. The XRD pattern revealed the average crystallite size to be (32nm - 44nm) for ($BaFe_{12}O_{19}$ - ZrO_2) nanoparticles respectively. The SEM image revealed that powder is well crystallized and the particles are elongated hexagonal structure. The dielectric studies showed the frequency, dielectric constant and dielectric loss in the range of 11.5GHz, 24.6 and 0.0064.

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