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Radiation losses in microwave K_u region by conducting pyrrole/barium titanate and barium hexaferrite based nanocomposites

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ABSTRACT

Nanocomposites of substituted barium hexaferrite and barium titanate embedded in a polymer were synthesized via emulsion polymerization. The study was performed by using X-ray diffraction, Fourier transform infrared spectroscopy, transmission electron microscopy, electron spin resonance spectroscopy, a vibrating sample magnetometer and a vector network analyzer. It is found that maximum radiation loss occur at 16.09 GHz (−14.23 dB) frequency owing to the combined effect of conducting polymer, suitable dielectric and magnetic material. This suggests that prepared material is suitable for radiation losses. Micro structural study reveals the presence of all the phases of the compounds comprises composite. Benzene ring absorption band (at 1183 cm^{-1}) in FT-IR spectra illustrates the presence of polymer. Surface morphology reveals the presence of array of particles encapsulated by the polymer.

1. Introduction

The radiation absorbing nano-composites have attracted much attention due to their applications in Wi-Fi systems and other communication systems [1] , microwave devices and radar absorbing materials [2]. The intensive use of wireless systems in microwave region with advancement of communication systems has also originate the electromagnetic interference and environmental pollution and become hazardous for biological tissues [3,4]. The radiation absorbing materials can help in reducing the unwanted electromagnetic signals. Earlier, magnetic material, metal flakes, carbon nanostructures were used as absorbent and extensively studied for the same property, but the bulk size could not meet the technical requirement like easy synthesis, broad bandwidth, impedance matching [5]. The magnetic materials tend to decrease their permeability at Gigahertz frequency range, so a conjugated polymer can be used to damp the eddy currents [6]. Absorption in ferrites depends on the magnetic resonance phenomena because of the anisotropy field [7].

Polymer has good stability in air and have good conductivity to promote the charge carrier drift. The magnetic relaxation oscillation of hexaferrite can be affected with the structural disorder like pores, defects etc. The embedding of magnetic material in polymer can induced disorder in structure because of polymer and material matrix. It makes the system more complex resulting in changes of internal magnetic fields. This cause shifting of the resonance frequency. The permeability ($\mu = \mu' + j\mu''$) and permittivity ($\varepsilon = \varepsilon' + j\varepsilon''$) contributes effectively to absorb or dissipate the radiation. Real parts of both parameters contributes to the energy storage and imaginary parts contributes to the losses in a material. Theoretical study suggest that the composites with both magnetic losses and dielectric losses can absorb the wide frequency range radiation at effective level. So, a composite with magnetic material, dielectric material and a conducting polymer possessing π-conjugated system has been synthesized to absorb the radiation. The radiations falling on such composites suffer multiple reflections inside the composites due to their interactions with magnetic and dielectric materials resulting in dissipation of radiation energy (Fig. 1).The role of barium titanate, a dielectric material in the composites is to increase the polarization which increases the real part of permittivity causing dissipation of radiation energy. The pyrrole, a conducting polymer has attracted much attention because of its high electrical conductivity [8–10]. So, the researchers have turned their attention towards polymers embedded with dielectric and magnetic materials [11–23].

There are many methods available to develop **magneto-electric nanocomposites** but emulsion polymerization, an easy and reliable method, has been used in the present research work [9]. The structural, magnetic and radiation absorbance properties of synthesized composites have been investigated.

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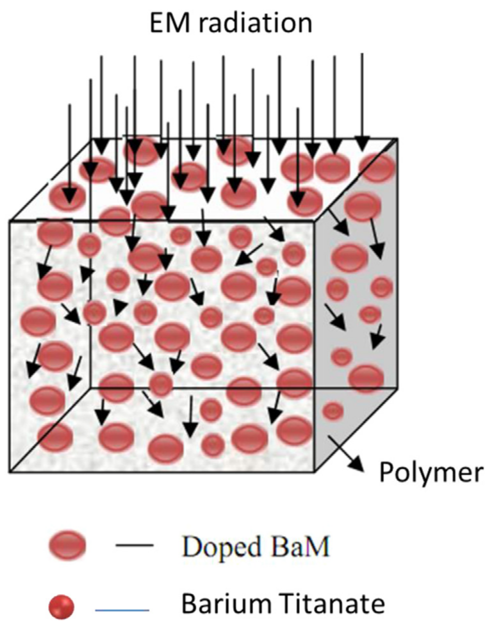

Fig. 1. Mechanism for radiation absorbance.

2. Experimental procedure

2.1. Synthesis of substituted Barium hexaferrite

The synthesis of Gd-Co and Nd-Co substituted barium hexaferrite was carried out via sol gel method using AR grade chemicals [24,25]. The stoichiometric amount of citric acid and other salts were dissolved and mixed together to make a clear solution. The molar ratio of cations to citric acid was taken as 1:1.5. To maintain 6.8 pH value of the solution , ammonium hydroxide (NH_4OH) solution was added drop wise. After that, a continuous stirring at a temperature range 80 °C–85 °C for 4–6 h was provided to the solution. The solution was converted into a homogenous brown colored gel. The gel was dried over hot plate at 280–300 °C for 3 h to obtain precursor powder. The resultant precursor was heat treated at 500 °C for 2 h to remove impurities. The heat treated precursor material was calcined at 900 °C for 5 h at the rate of 23 °C/min to obtain hexaferrite.

2.2. For synthesis of barium titanate

Barium nitrate, TiO_2 and oxalic acid were used as a starting materials for the synthesis of barium titanate. An aqueous solution of barium nitrate (0.12 M) was prepared using distilled water. Required amount (Ba/Ti=1:1) of TiO_2 powder was poured to the aqueous solution of barium nitrate with constant stirring. To avoid the agglomeration of titanium oxide article, the mixture was ultrasonicated for 10 min. Oxalic acid (0.4 M) was added to the mixture drop wise with vigorous stirring to get precipitates. Ammonia solution was used to maintain the pH of the solution 6.8. The precipitates were washed repeatedly with distilled water followed by drying at 40 °C for 2 days to obtain precursor. Further, the precursor was calcined at 900 °C to get barium titanate.

2.3. Preparation of Composites

The surfactant dodecyl benzene sulfonic acid (DBSA) was used as surfactant. 0.3 M solution of DBSA was added in barium hexaferrite and barium titanate to form an emulsion.After that, the pyrrole was added in the solution. For uniform dispersion, the solution was ultrasonicated before and after the addition of pyrrole monomer. An initiator, ammonium persulphate was used to initiate the polymerization at 2 °C. Obtained solution was kept on stirring for 24 h. The final product was filtered with suction pump and demulsified with isopropyl alcohol. After that, product was washed with distilled water. The composites were placed in an oven and dried at 80 °C for 24 h [9].

3. Characterization techniques

Structural properties have been investigated using X-ray diffraction powder patterns obtained from Bruker AXS D8 Advance X-ray diffractometer in the range 20–80° using Cu-k_α radiation operating at 40 kV and 35 mA having step size of 0.02°. Attached functional groups have been analyzed with Fourier transform infrared spectra (FT-IR interferometer IR prestige-21 FT-IR (model-8400S)) in the range of 400–4000 cm^{-1} by making pallets with KBr in weight ratio of 1:10 (sample to KBr ratio). EPR measurements were performed at room temperature using an X-band JEOL JES- ME spectrometer. EPR spectra are recorded under following experimental conditions: magnetic field sweep rate of 50 mT/min, modulation width of 0.35 mT, modulation frequency of 100 kHz, and microwave power of ~10 mW (9.5 GHZ). Magnetic properties have been studied with a vibrating sample magnetometer (Lakeshore 7410) at room temperature. Microwave studies have been carried out with Vector network analyzer (Agilent 8722ES) by pressing the powder with palette making machine and making palettes of 2 mm thickness (15.8 mm × 7.9 mm). Readings of rectangular palettes have been taken by inserting sample in sample holder having dimension 15.8 × 7.9 mm^2. Transmission electron microscope (TEM) images of samples have been recorded using JEOL JEM 2100 Japan instrument.

4. Results and discussions

4.1. Phase identification

For the phase identification, the X-ray diffraction patterns of the nanocomposites have been presented in Fig. 2. All samples show crystalline phase. The presence of apparent peaks at angles 23.006° (006), 30.318° (110), 32.236° (107), 34.214° (114), 56.512°

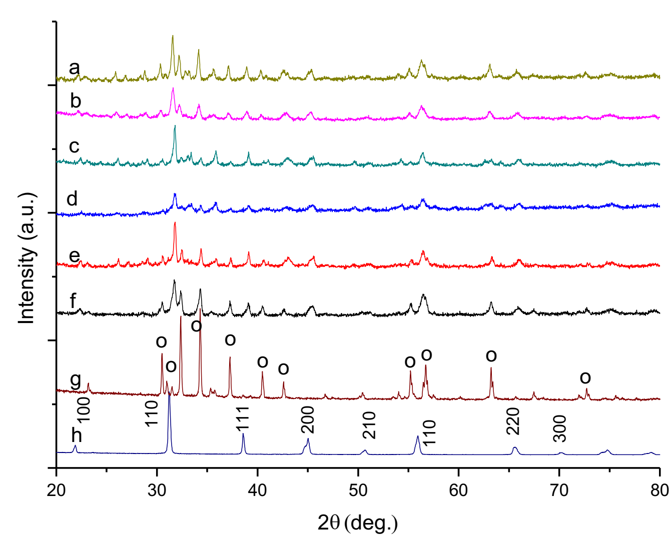

Fig. 2. X-ray diffraction patterns for $Ba_{1-x}Gd_xCo_xFe_{12-x}O_{19}$ (or $Ba_{1-y}Nd_yCo_yFe_{12-y}O_{19}$)/$BaTiO_3$/Polypyrrole composites at (a) $y=0.4$, (b) $y=0.25$, (c) $y=0.1$, (d) $x=0.5$, (e) $x=0.2$, (f) $x=0.0$ whereas (g) Pure barium hexaferrite and (h) Pure barium titanate.