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Preparation and Characterization of PPY/$BaFe_{(12-x)}Ti_xO_{19}$ Nanocomposite

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***Abstract*— The preparation of hybrid composite in two stages is being presented in this paper. There are two stages of synthesis. In first stage, Ti-doped barium ferrite powders $BaFe_{(12-x)}Ti_xO_{19}$ (for x = 0.33 and 0.37) nanomaterial using sol-gel route were synthesized. In second stage, Ti-doped barium ferrite powders $BaFe_{(12-x)}Ti_xO_{19}$ nanomaterial synthesized in the first stage is incorporated into the Polypyrrole through solution processing method. The phase structure and morphology were analyzed by standard XRD, SEM, EDS and FTIR techniques.TEM and SAED were done on Nanocomposite samples to know the dispersion of PPY with BFTO nanopowder. *Scifinder software couldn't trace* any earlier communication involving this nanomaterial in literature.**

Keywords*— Barium ferrite, sol-gel route, Titanium, Nano ferrite, impregnation technique)

I. INTRODUCTION

Barium ferrite (BaFe12O19) is a permanent magnetic material, a ferrimagnetic material better chemical stability, High saturation magnetization, great coercivity and it is a low cost material.

It has a very wide range of application,very useful in Microwave communication, useful in microwave dark room, absorber for electromagnetic wave radiation (microwave absorbing materials reduce the human exposure to microwaves by means of absorbing coatings) [1-3]

Barium ferrite has hexagonal structure. It belongs to magneto-plumbite ceramic oxide group and can be classified as hard magnetic material. It is one of a ferromagnetic oxide that has both dielectric and magnetic properties when applied with high frequency (microwave region), hence is ideal for microwave applications. The Fe^{3+} ions occupy the sub lattice at different sites that lead to different magnetic properties and change its sub lattice and magnetic properties on addition of nonmagnetic materials [4-6].

Ti-doped barium hexaferrite (Ti-doped BHF) powder is an efficient absorber of electromagnetic waves in the microwave spectrum increase in the saturation magnetization (M_S) and a decrease in the coercivity ($_JH_C$) are required for optimization of the absorption. These properties depend on the localization of the Ti_{4+} in the barium hexaferrite structure. The Ti_{4+} ions preferentially occupy the octahedral 4f2 sites of the BHF structure when the Ti-doped BHF is synthesized using the sol-gel route, especially at low doping rates. Also Since the magnetic moment of the Ti_{4+} ions is zero and the spin direction of 4f2 site in the BHF structure is down. M_S must increase with the doping concentration of titanium, while H_C must decrease, which corresponds our expectations of lower magnetocrystalline anisotropy. However with higher doping rates of Ti_{4+}, M_S decreases , because Ti_{4+} ions could be located also in other lattice sites[7-12].

Both polyaniline (PANI) and polypyrrole (PPy) are Probably the most widely studied conducting polymers due to their good stability in air, high conductivity, and reversible process between oxidation and reduction state. Polypyrrole can be dispersed into metallic fillers like Ferrites by easy methods [13-25].

II. EXPERIMENTAL METHOD

Ti doped barium ferrite was prepared by Sol-Gel method. $BaFe_{11.67}Ti_{0.33}O_{19}$ and $BaFe_{11.63}Ti_{0.37}O_{19}$ at 950^0C.The synthesis of Nanoferrite composites was done by Impregnation Technique. The Nanoparticle impregnation process involves dissolving an appropriate metallic precursor in an polymer and then exposing the substrate to the solution. And reduced by variety of methods resulting in films, powders. This technique has allowed both highly dispersed and uniformly distributed metal nanoparticles and formation of agglomerated clusters of nanoparticles. Substances impregnated into Polymers are generally Dyes, Fragrances, Metal nanoparticles etc.

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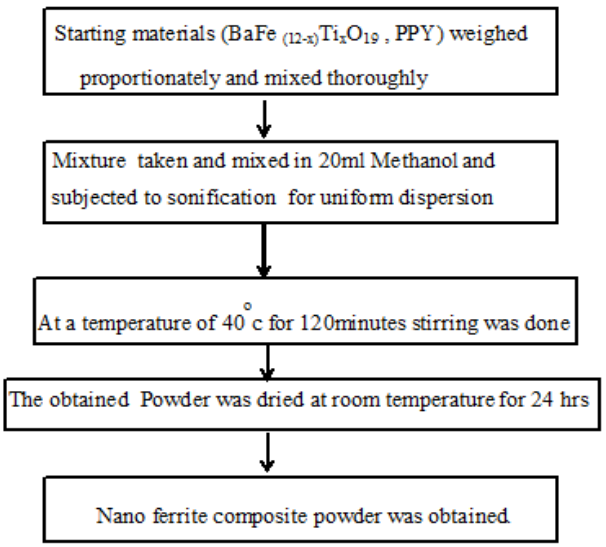

Fig 1.Flow chart for nanoferrite composite preparation

III. RESULTS AND DISCUSSION

A. *XRD (X-ray Diffractometer)*

XRD was performed at room temperature using CuKα radiation of wavelength 1.5406Å to confirm crystallographic phase formation of nanocomposite material.

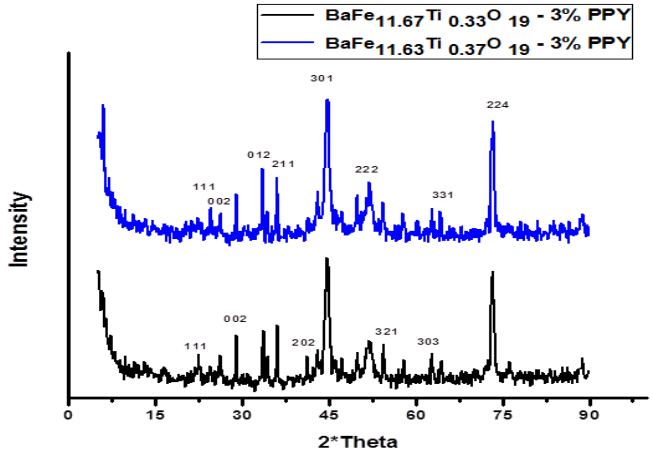

Fig 2. XRD graphs of PPy/Ti-doped barim ferrite for x=0.33 and 0.37 at 950 ºC temperatures.

The substituted samples are completely be dissolved in the magneto plumbite lattice and no intermediate phases in the hexagonal plane of magneto plumbite structure are observed. Analyzing the effect of varying the 'x' value, we can observe from figure 2 that the sample 2 shows well developed narrow peaks than the sample 1 which indicates that formation of nanoparticles are good at higher 'x' value.

Table1. XRD parameters of Nanoferrite Composites at 'x' =0.33 and 0.37

S.no	Name of Sample	Crystallite Size "D"	Parameter "a" Å	Interplanar Spacing "d" Å	Volume of unit cell gm/cm^3	Density of Unit Cell
1	$BaFe_{11.67}Ti_{0.33}O_{19}$-3% PPY	(11.91) nm	6.2965	2.01894	249.919	7.7809
2	$BaFe_{11.63}Ti_{0.37}O_{19}$ -3% PPY	(13.168)nm	6.32899	2.4471	254.364	7.78052

The values of the lattice constants, crystallite size,, miller planes determining the type of lattice formed, we found that the prepared doped nanoferrite is a cubic lattice. To know the structural formation of Nanocomposites, XRD technique is done on the samples. We know that PPY is amorphous in nature. By relative comparison of two samples, we can see in the case of sample 1 the interaction of PPY with $BaFe_{12-x}Ti_xO_{19}$ decreased the average interparticle distance leading to the reduction in the size of Nanoparticles. Varied doping concentration may be the reason for this.

B. *SEM(Scanning electron microscopy) and EDAX(Energy dispersive analysis)*

The surface morphology of the composite sample was examined using SEM.

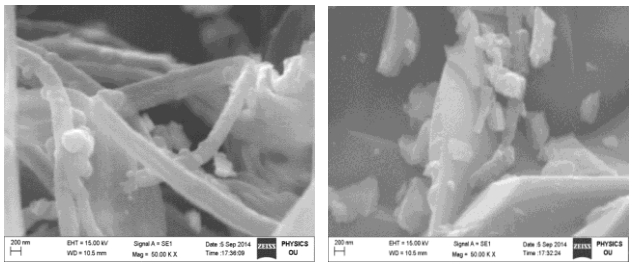

Fig. 3. SEM micrographof PPy/Ti-doped barim ferrite for x=0.33 and 0.37 at 950 ºC temperatures

SEM micrograph reveals that in sample 1 long and continuous nanorods are more prominent when compared with sample 2, here and there some cubic structure are also present. Whereas in sample 2 short nanorods and cubic structures are present.

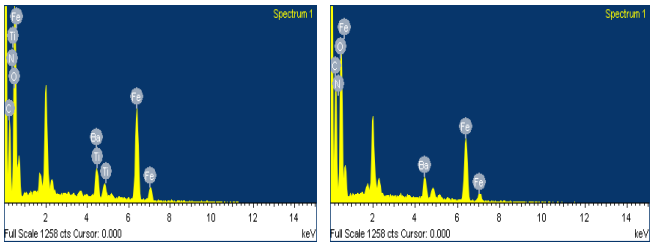

Fig 4.EDAX plotsof PPy/Ti-doped barim ferrite for x=0.33 and 0.37 at 950 ºC temperatures

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EDAX characterization was done in order to ratify the purity and surety of chemical composition. It is a supplementary technique to TEM. The energy dispersive x-ray analysis pattern reveals that these nanoparticles are well crystallized.

In sample 1ions are present in higher and lower energy levels. This confirms that A-O-B spinel sub-lattice formation. In sample 2Ti peak at 5Kev energy value are absent, indicating that they were knocked out from lower energy spinel state. Whereas Fe peak at 6Kev has reduced indicating that number of nanoparticles are reduced at that particular energy level. This sample therefore cannot provide absorbing ions for the EMI radiation. Hence their ability for EMI absorption is less. However the above absorption of the Ti ion are necessarily to be confirmed by XPS and mausbaur studies.

C. FTIR (Fourier Transform Infra-Red)

FTIR spectra of the obtained powder were recorded using FTIR spectrometer in the wavenumber range 4000-400cm^{-1} using kbr pellet to ratify the structure of sample. The identities, surrounding environments and concentration of chemical bonds that are present can be determined.

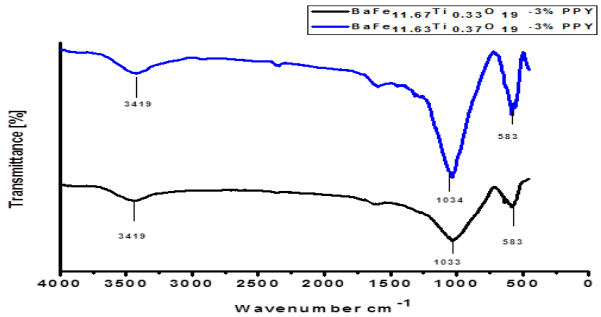

Fig 5.FT-IR spectraof PPy/Ti-doped barim ferrite for x=0.33 and 0.37 at 950 ºC temperatures

Dips indicate the formation of groups. The bands around 500cm^{-1} correspond to metal-oxygen vibration.The bands around 1033,1034 cm^{-1} correspond to =C-H band in plane vibration.The peaks around 3000 cm^{-1} assigned to N-H stretching vibration from pyrrole.Charge carrier existence is confirmed by several peaks indicating bi-polaron band formation. This indicates the formation of PPY in oxidised state.

D. TEM (Transmission electron microscope) AND Histographic analysis

TEM and SAED were done on Nanocomposite samples to know the dispersion of PPY with BaFe Ti O
12-x x 19

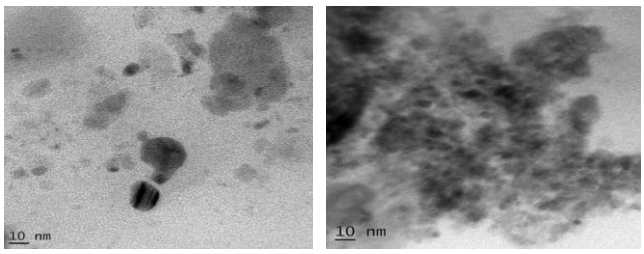

Fig 6.TEM microgramof PPy/Ti-doped barim ferrite for x=0.33 and 0.37 at 950 ºC temperatures

In TEM images of $BaFe_{12-x}Ti_xO_{19}$ for all X-Values studied, the particles appear to be dark.Since ferrite is magnetic in nature, it absorbs more elctrons than Ti. Hence appear darker. In $BaFe_{12-x}Ti_xO_{19}$/PPY ,for different percentages of 'x' value the $BaFe_{12-x}Ti_xO_{19}$particles are surrounded by PPY confirming the coating of the former by later. In microgram polymer grains are white in colour and Ferrite grains are black in colour. This clearly indicates the formation of core-shell structure with doped ferrite as core and PPY as shell. It is observed that PPY was coated on $BaFe_{12-x}Ti_xO_{19}$. But for some samples the coating shell obstructed the contact between the rest of PPY and $BaFe_{12-x}Ti_xO_{19}$.Rest of PPY assembled as oxidant.

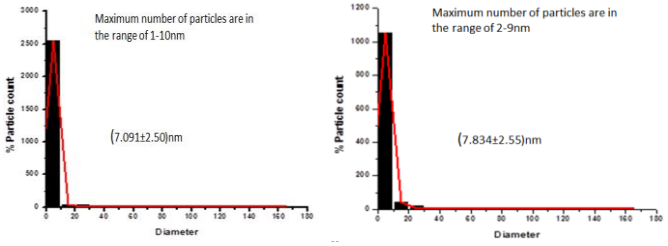

Fig7. Histogramsof PPy/Ti-doped barim ferrite for x=0.33 and 0.37 at 950 ºC temperatures

Using image j software with SEM and TEM images number of particles distributed size wise are determined. The data is presented as histograms, Gaussian distribution curve is also superimposed to analyses the data. The particle diameter data for the different values of 'X' (calculated using image j software) registered a vivid contrast with similar data from XRD. In general majority of the values of the cryatallite parameter D showed reasonably good concurrent with the corresponding parameter of xrd calculations with large values of beta in debye-schherr equation. The data in (a) TEM and EDX as well as (b) Debye–Scherrer equation from XRD.show reasonably good agreement .

E. SAED (Selected area electron diffraction)

Electron diffraction analysis was used considerably to gain structural information of the sample. Often both electron microscopy (information in real space) images and diffraction patterns (information in reciprocal space) are obtained for the same reason. Electron diffraction can be performed on a single nanoparticle or on an area consisting of multiple nanoparticle, by inserting the relevant selected area aperature. Hence selected area electron diffraction(SAED).

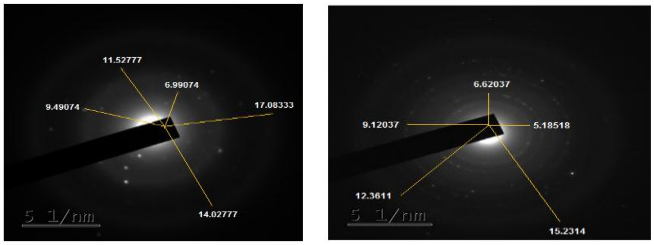

Fig 8. SAED patternof PPy/Ti-doped barim ferrite for x=0.33 and 0.37 at 950 ºC temperatures

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Central part is called nose. It is a source of electrons causing diffraction. Bright spots indicate crystalline structure and rings indicate polycrystalline structure. Each bright spot reveals the location of an atom in the crystal. Rings are formed when an atom in a crystal are systematically arranged in different miller plane. If multiple rings are formed more number of miller planes. Such a crystal gives interesting information about interactions with magnetic field, electric field, optical field etc. Sample 2 shows well developed distinct rings indicating polycrystalline structure. Diffraction rings and also Bright spots (in good number) are seen indicating perfect formation of Nanostate .

CONCLUSION

we have successfully incorporated Ti- doped barium ferrite (x=0.33 and 0.37) nanopowder into polypyrolle nanocomposite. The formation of PPy/Titanium doped Nano ferrites has been con-firmed by XRD, SEM studies. FT-IR, EDAX, TEM and SAED studies on the same are also reported.

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