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Synthesis and Characterization Study of Polypyrrole Composite of Titanium Doped Barium Ferrite ($Bafe_{(12-X)}Ti_xo_{19}$)

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Abstract:*This article presents the preparation of hybrid composite in two stages. In First Stage,Ti-doped barium ferrite powders $BaFe_{(12-x)}Ti_xO_{19}$(for x = 0.35) nanomaterial using sol-gel route, at three different temperatures were synthesised. In second stage , Polypyrrole is incorporated into the Ti-doped barium ferrite powders $BaFe_{(12-x)}Ti_xO_{19}$for (x = 0.35) nanomaterial synthesised in the first stage through impregnation technique. The phase structure and morphology were analysed by standard XRD, SEM and FTIR techniques.*

I. INTRODUCTION

Electromagnetic interference (EMI) is an undesirable and uncontrolled off-shoot of explosive growth of electronics and widespread use of transient power sources. It's a novel kind of pollution which not only tries to disturb the normal operation of appliances but may also adversely affect human health. Hence, continued efforts have been made over past two decades to reduce EMI using a number of strategies and variety of materials including metals, carbon based materials, conducting polymers, dielectric/magnetic materials [1-9]. Based on results of various studies, it can be concluded that no single material can take care of all the aspects of shield e.g. strength, weight, cost, processability, level of shielding and stability under wide variety of chemical, thermal and radiative environment. Therefore, several attempts have also been made to utilize the good properties of above materials by using them in various combinations e.g. alloys, blends, composites, ceramics or layered/sandwiched structures [10-17]. The discovery of various nanomaterial and unique set of properties possessed by them has led to extensive research activities to identify the best materials for shielding. In particular, Nanocomposites have attracted enormous world-wide attention both due to promising properties as well as critical issues associated with them [18-21]

Conducting polymers have emerged as a new class of materials in the last three decades. Because of their high conductivity, intriguing electrical properties, and ease of production, potential applications such as microwave absorbers were seriously considered soon after the discovery of these materials [22].Conducting polymers composites i.e. polyaniline, polypyrrole and polythiophene etc. has been investigated as suitable for EMI shielding application due to their tunable conductivity, corrosion resistance, light weight, easy processability, good environmental stability and tailor-able permittivity[23-26].

It is well known that conducting polymers can effectively shield electromagnetic waves generated from an electric source, whereas electromagnetic waves from a magnetic source can be effectively shielded only by magnetic materials. Thus, the incorporation of magnetic constituents and conducting polymeric materials into multifunctional composites opens new possibilities for the achievement of good shielding effectiveness for various electromagnetic sources [27]. The development of microwave absorbing materials continues to attract much attention. Among the candidates for such application, ferrites present an interesting material and polypyrrole (PPy) is one of the most promising conducting polymers due to unique properties and excellent environmental stability [28].

BariumHexaferrite ($BaFe_{12}O_{19}$) is a member of the ferrite family in which simultaneous occurrence of big Ferro electricity and strong ferromagnetism has been observed [29]with significant material qualities such as high Curie temperature, large magnetization, large magneto crystalline anisotropy, high coactivity, and excellent chemical stability [30]. It has been widely adopted as a traditional permanent magnet and also recently used as high-density magnetic and magneto optical recording media and microwave filters.[31 to 34]. They are applied as permanent magnets, in microwave devices or in perpendicular magnetic recording. Another application is in catalysis area [35 to 39].

Recently, Xu and co-workers have synthesized barium ferrite/PPy composites by a conventional in situ chemical oxide polymerization and found that the composites have more excellent reflection loss properties

[40]. Kim et al obtained PET fabric / PPy composites and the electromagnetic interference shielding effectiveness of composites increased with the high electrical conductivity [41]. Novel nano-structured composites based on modified anionic and cationic metal oxide nanoparticles and modified with different transition metals are subject matter of current interest [42(a), (b) and (c)]. Strong microwave absorption and broad bandwidth are the growing requirements for the future materials for EMI Shielding. They reduce the human exposure to microwaves with the frequency range between 26.5 and 40 GHz(i.e., has the characteristics of both centimetre waves and millimeter waves). Materials with microwave absorption properties can work not only as all-weather materials but also as high-resolution probes. Today there are many kinds of radars with the 26.5–40 GHz wave band being widely applied. Ferrites exhibit outstanding microwave absorption properties and are widely employed in Defence and allied fields due to their high resistivity and strong EM energy attenuation, especially near the natural resonance frequency of magnetic moments [43 to 46].

It has been reported that barium and strontium ferrites can be heat treated in presence of nitrogen, hydrogen or carbon containing gasses to achieve high saturation magnetization and low coericivity values which makes these materials suitable for using in recording media such as hard disks, cassette and video tapes[47 to 49].

This article presents incorporation of Ti doped barium ferrite (BFTO) powder into polypyrrole nanocomposite to form hybrid composite. The prepared powders were characterized using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR).

II. RAW MATERIALS

The Synthesis of the chosen Nanomaterial for the study was done at National Chemical Laboratories, Pune, INDIA. Ti-doped barium ferrite powders were synthesized by the sol–gel method from the starting raw materials. Barium ferrite ($BaFe_{12}O_{19}$) and Titanium(IV) butoxide($Ti(OC_4H_9)_4$), [complete chemical formula being $Ti(OCH_2CH_2CH_2CH_3)_4$] and polypyrrole obtained from Sigma Aldrich. Citric acid, Ammonia, Absolute Ethyl alcohol and Deionized water were used as ancillary raw materials. These were procured from E-Merck and were eventually purified using prescribed standard chemical procedure

III. EXPERIMENTAL PROCEDURE.

There are two stages of synthesis

Stage 1: Preparation of ferrite

According to the composition of $BaFe_{(12-x)}Ti_{(x)}O_{(19)}$ (x= 0.35), three solutions were prepared. Solution (1) is prepared by dissolving pre estimated amount of metal ferrite and an appropriate amount of citric acid in the deionized water by stirring for 30 minutes to obtain the clear solution(1). Solution (2) is prepared by dissolving specific pre estimated amounts of $Ti(OC_4H_9)_4$ and citric acid in absolute ethyl alcohol by stirring for 30 minutes to get a clear solution(2). Solution (2) was very slowly added into solution(1) carefully by keeping the mixture continuously stirred for three hours. This gave the clear Solution (3). Then ammonia was added drop by drop to Solution (3), until the pH value was adjusted to 7.0. The system [50] should be acidic to maintain a clear solution as well as to prevent unwanted precipitation of either one or both the reactants before the gel formation and before combustion actually starts. The pH was determined using a precise pH meter. The pH is an important parameter that governs the characteristics of the Nano material. It is reported that as the pH of the solution increases the particle size also increases [52, 53]. Also as the pH increases, the weight losses are found to be small according to the literature. The obtained solution was evaporated with continuous stirring to form viscous sol precursors at 80^{0}C& then dried at 120 ^{0}C, for 24 to 48hrs. Then the viscous sol was heat treated for 3 hrs, after dividing into three parts. Three different temperatures 850^{0}C, 900^{0}C and 950^{0}C.

Stage 2: Preparation of composite

BFTO/PPy nanocomposite were prepared by adding the synthesized nano powders of $BaFe_{(12-x)}Ti_{(x)}O_{(19)}$ (x= 0.35) and polypyrrole at different weight percent (BFTO powder=97% and polypyrolle=3%) with 20ml of methanol. Mixture was stirred for 2 hours and evaporated for 30 min. then the mixture is dried overnight.

Characterisation of the Synthesised Samples

The phase identification and grain distribution of the sintered samples were identified using XRD X-ray Diffarctometer (XRD) (Philips: PW1830), at University of Hyderabad,A.P. India and Scanning Electron Microscope (SEM) (SEM Hitachi- S520), at Osmania University.,Hyderabad ,A.P., INDIA .The FT-IR (Schimadzu Perkin-Elmer 1310), at SAIF, IITM, Chennai, India, was used to ascertain the metal-oxygen and metal-metal bond in the prepared sample.

IV. RESULTS AND DISCUSSION

X-ray diffraction (XRD) studies

In the utilised X-ray powder diffraction (XRD) method,Cu K-alpha radiation (wavelength 1.54178 Å), is used for the scattering experiments. Figure 1shows the XRD patterns of thePolypyrroleincorporatedTi-doped barium ferrite powders $BaFe_{(12-x)}Ti_xO_{19}$for (x = 0.35) nanomaterial. All samples show cubical like structure, indicating the doping element has been successfully substituted into the structure. The average crystalline size was found to be in between 20 to 50 nm and was calculated using equation (1).

The Average grain size has been calculated using Debye – Scherrer's [54] equation (1) as shown below

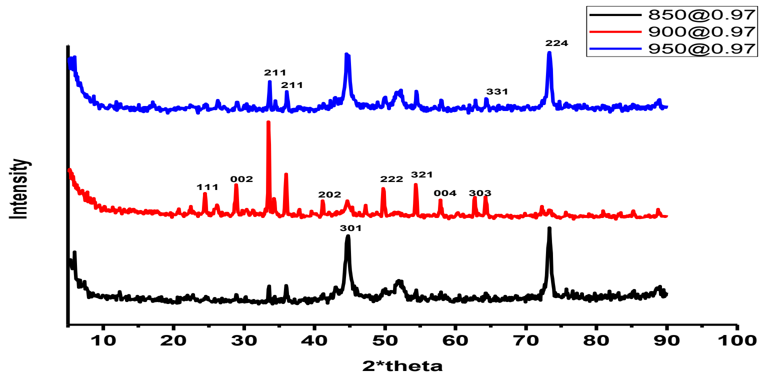
$$D = [0.9\lambda / \beta_{½}\cos\theta]\text{------------}(1)$$

Where

λ = wave length of the x- ray beam

$\beta_{½}$= Angular width at the half max intensity

θ= Braggs angle



XRD graphs of PPy/Ti-doped barim ferrite at 850^{0}C, 900^{0}C and 950^{0}C temperatures

850^{o}c		900^{o}c		950^{o}c	
2θ(deg)	D(nm)	2θ(deg)	D(nm)	2θ(deg)	D(nm)
33.55	13.823	33.55	10.367	33.55	13.82
35.95	13.914	35.95	13.914	36.05	13.918
44.65	8.41	44.65	8.58	44.65	21.46
64.35	23.455	64.35	11.620	64.35	15.63

Table1:PPy/BFTO nanocomposite average grain size D and 2θfor x=0.35 at 850^{o}c,900^{o}c and 950^{o}c temperatures

we can observe from figure(1) that for the PPY/$BaFe_{(12-x)}Ti_xO_{19}$(x = 0.35) sample sintered at 850^{0}C and 900^{0}C less number of peaks are formed whereas at 950^{0}C well developed narrow peaks are seen which indicates that formation of PPY/$BaFe_{(12-x)}Ti_xO_{19}$ nanocomposite are good at this particular temperatures. And also size of the particle is small at this temperature range. Formation of nanostate is complete at 950^{0}C temperature range.

Scanning Electron Micrograph (SEM)

The SEM technique is used to characterize the morphology and size distribution of nanoparticles. The obtained SEM images of the synthesisedpolypyrrole incorporated barium ferrite samples are shown, in Figure-2. It is to be noticed that the particles of all samples exhibit plate – like nearly cubicalshape.The particles are irregular in shape with compact arrangement and lies in the range of 40nm. In some particles flakes of agglomerates are also observed. The samples obtained at the different tempering conditions show varying quality of crystallization.

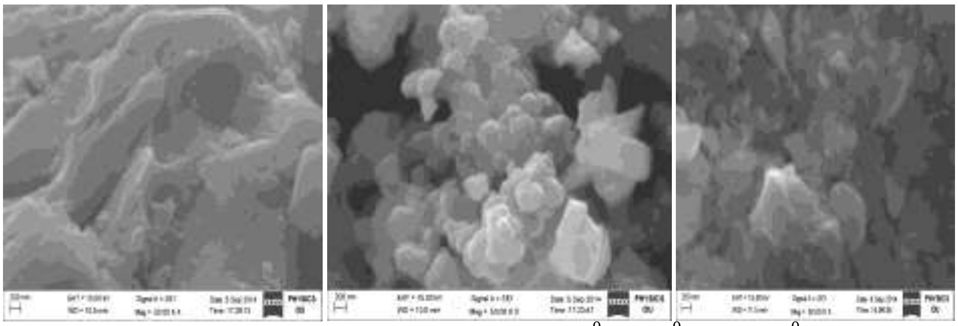


Fig 2 SEM images of PPy/Ti-doped barim ferrite at 850^{0}C, 900^{0}C and 950^{0}C temperatures

Fourier Transform infra red studies (FT-IR)

Fourier Transform Infra-Red (FT-IR) spectra have been recorded usingSchimadzu Perkin-Elmer 1310 FT-IR spectropho-tometer with KBr pellets in the range 4000 – 400 cm^{-1}. The FTIR of the BFTO powder (fig 3) shows characteristic peaks in the required region, i.e.,3441,1444,1029,880 and 545cm^{-1}.

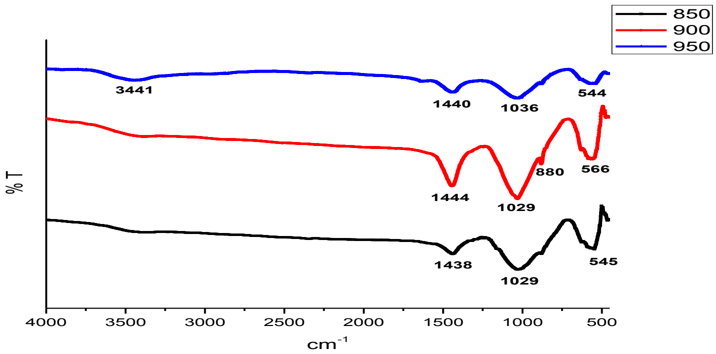


Fig 3 FT-IR images of PPy/Ti-doped barim ferrite at 850^{0}C, 900^{0}C and 950^{0}C temperatures

Stretching Peak at 541 cm^{-1} and 880 cm^{-1}indicates existence of the metal-oxygen vibrational modes of the spinel compound, Stretching peak at 1036cm^{-1}indicates C-O, bending peak at 1400 cm^{-1}indicates –CH_3 [55] and stretching peak at 3441cm^{-1} indicates O-H[56].

V. CONCLUSION

In summary, we have successfully incorporated Ti- doped barium ferrite (x=0.35) nanopowder in polypyrolle nanocomposite. The formation of PPy/Titanium doped Nano ferrites has been con-firmed by XRD,SEM studies. FT-IR studies on the same are also reported. The crystallite size is found to be in the range 15-40 nm.

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