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Titanium doped barium ferrite powders (Ba Fe $_{(12-x)}$Ti$_x$ O $_{19}$) , prepared using sol gel method and characterised using XRD, SEM and FTIR techniques

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***Abstract**: Ti-doped barium ferrite powders $BaFe_{(12-x)}Ti_xO_{19}$ for different 'x' valuenanomaterial has been synthesized using sol-gel method is presented in this article. As prepared nanomaterial is heat treated at 950^{o}C temperature and characterised using XRD, FTIR and SEM techniques.*

***Keywords:**Barium ferrite, sol-gel route, Titanium, Nano ferrites, morphology.*

I. Introduction

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 [1 to 4]. The physical properties of ferrites are controlled by the preparation conditions, chemical composition, sintering temperature and time, type and amount of substitutions [5].

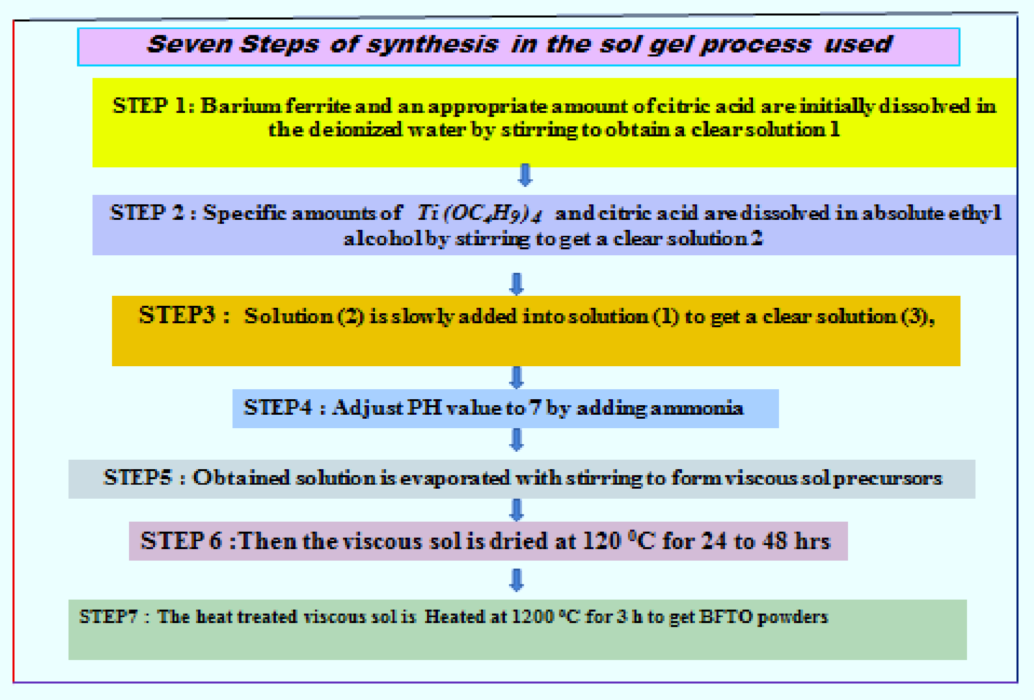
Nano scale magnetic ferrite materials possess a set of unique magnetic and electrical properties and chemical stabilities [6, 7]. The main applications of these materials intend to reduce the human exposure to microwaves by means of absorbing coatings [8-10].

Ti-doped barium ferrite powder is an efficient absorber of electromagnetic waves in the microwave spectrum. In this communication the preparation &characterisation nanomaterial of BFTO was prepared using sol-gel method. As prepared powders are characterized using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR).

Synthesis

The sol–gel combustion method has the unique advantage for the low costs using simple equipment in large-scale high-purity. The effects of milling time and heat treatment temperature on the characteristics of powder mixture has been reported by M.J.Molaeiet,al.,[11(a) and (b)], during his study on Magnetic property enhancement and characterization$BaFe_{12}O_{19}/Fe_3O_4$ and Fe/Fe_3O_4magnetic nano-composites. As synthesised powders were studied by XRD,VSM,TEM and Mossbauer spectroscopy. Phase analysis results showed that Fe_2O_3 in barium ferrite partially reduced to Fe_3O_4 during milling. Hence, the reduced phase and remaining barium ferrite formed a nano-composite of $BaFe_{12}O_{19}/Fe_3O_4$ after 20 h of milling Fe_3O_4. Fe containing nano-compositewas formed due to the heat treatment of the 40 h milled samples at 750–900 °C temperature.

The Flow chart and detailed procedure of the synthesis for the Ti-doped barium ferrite powders $BaFe_{(12-x)}Ti_xO_{19}$ is



II. Raw materials

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($Ti(OC_4H_9)_4$), 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. Synthesis of the samples

According to the composition of $BaFe_{(12-x)}Ti_{(x)}O_{(19)}$ (x = 0.33), 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. 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. Solution (2) was very slowly added into solution(1) continuously 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 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 [13, 14]. 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 80^0C & then dried at 120 ^{0}C, for 24 to 48hrs. Then the viscous sol was heat treated for 3 hrs, at 950^0C. Same procedure is repeated by varying the ‘x’ value at 950^0C. So obtained BFTO powder samples were analysed by various characterization techniques.

Characterisation of the Synthesised Samples

X-ray Diffarctometer (XRD) (Philips: PW1830), at University of Hyderabad,A.P. India and Scanning Electron Microscope (SEM) (SEM Hitachi- S520), at O.U.,Hyderabad ,A.P., INDIAwas used for phase identification and grain distribution of the sintered samples. To ascertain the metal-oxygen and metal-metal bond in the prepared ferrite sample, FTIR (Schimadzu Perkin-Elmer 1310), at SAIF, IITM, India, was used

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 1,2 and 3 shows the XRD patterns of the $BaFe_{(12-x)}Ti_xO_{19}$(for x = 0.33 , x = 0.35 and x = 0.37) powders sintered at 950^0C for 3 h. All samples show single phase tetragonal structure, indicating the doping element has been successfully substituted into the structure. The average crystalline size was found to be in between 15 to 50 nm and was calculated using equation (1).
The Average grain size has been calculated using Debye – Scherrer’s [15] 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

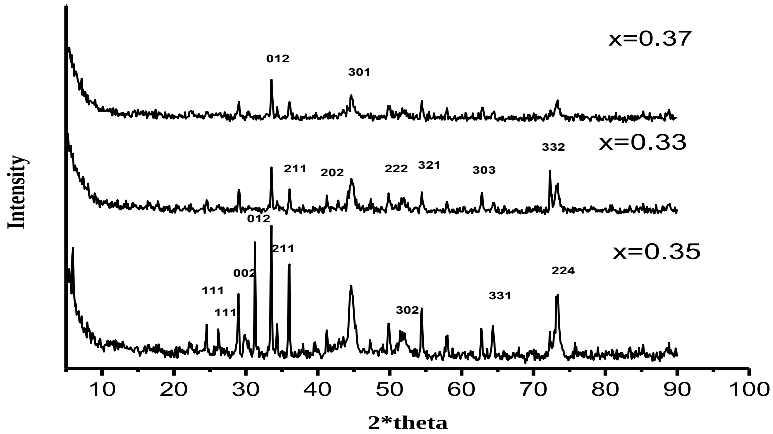


Figure 1,2 and 3: XRD graphs of Ti-doped barim ferrite ('x'=0.35 ,0.33 and 0.37) at 950^{0}C temperatures

x=0.33		x=0.35		x=0.37	
2θ(deg)	D(nm)	2θ(deg)	D(nm)	2θ(deg)	D(nm)
33.55	20.7353	33.55	13.823	33.55	13.8235
36.05	20.8776	36.05	13.9184	36.05	20.8776
44.65	21.4616	44.65	21.4616	44.65	42.9233
54.45	22.3263	54.45	14.8842	54.45	44.6527
73.35	49.5064	73.35	16.5021	73.35	16.5021

Table1: Average grain size D and 2θ values for x = 0.33, 0.35 and 0.37 at 950^{o}c temperatures

By observing figure(1,2 and 3), we can conclude that the formation of nanostate is very nearly complete at x=0.35 value sintered at 950^{0}C as we can seewell developed na.rrow peaks at this 'x' value than the sample (x=0.33 and 0.37) sintered at same temperature.

Scanning Electron Microscope (SEM):

The morphology and size distribution of nanoparticles was confirmed by SEM technique. The obtained SEM images of the synthesised barium ferrite samples are shown, in Figure-4. Figures show that the particles of all samples exhibit plate – like nearly tetragonal shape. Particles show compact arrangement with irregular shape and lies in the range of 10 to 50nm.

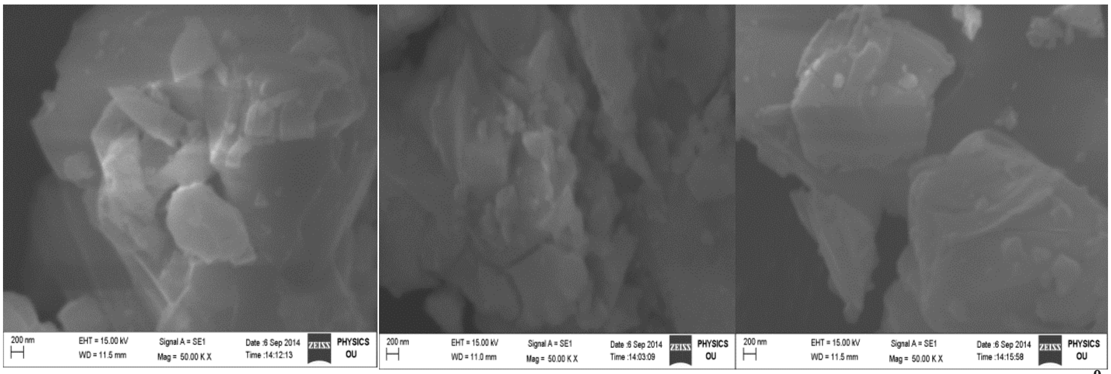
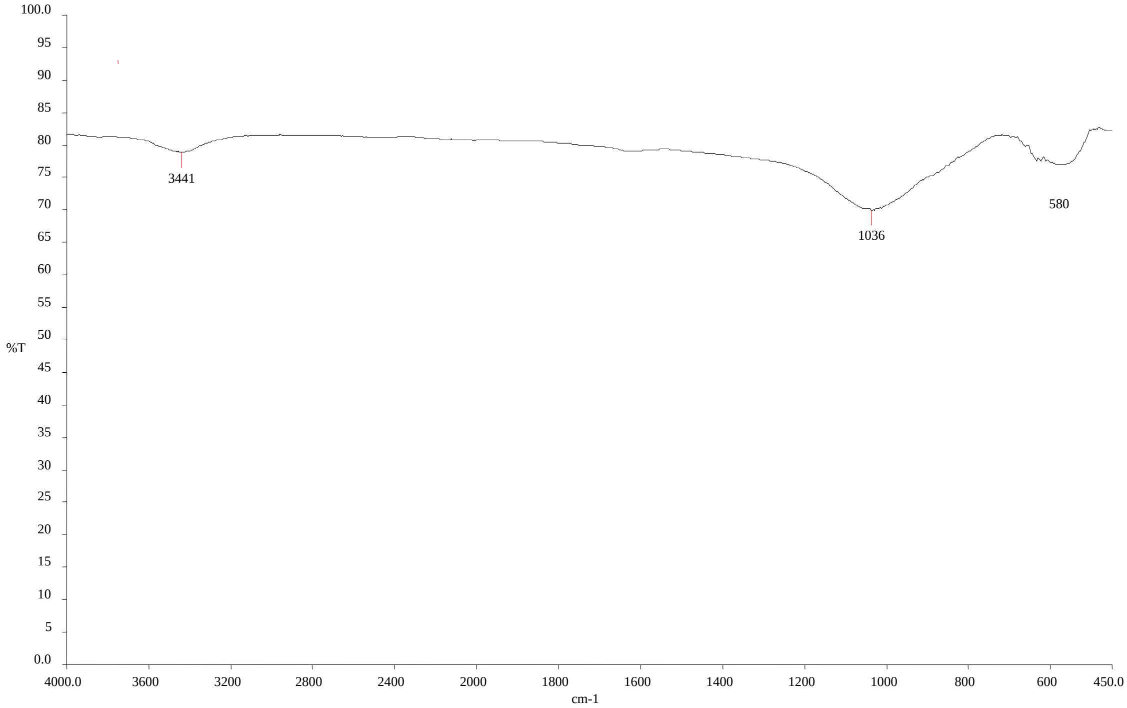


Figure 4: SEM pictures of Ti-doped barium ferrite at 'x'=0.33, 'x'0.35 and 'x' = 0.37 value at 950^0C temperature

Fourier Transform Infra-Red (FTIR):

Schimadzu Perkin-Elmer 1310 FTIR spectropho-tometer with KBr pellets in the range 4000 – 400 cm^{-1} was used to record Fourier Transform Infra-Red (FTIR) spectra. Characteristic peaks in the required region, i.e., 3418.34, 1618.51, 1400.80, 1080 and 543cm^{-1} was showed in the FTIR of the BFTO powder(figure 5 (x=0.35)). The inverted peaks corresponding to 1618.51, 1400.80 and 3441cm^{-1} does not appear at the phase formation where the 'x' value of the BFTO powder ('x'=0.33 and 0.37) in the FTIR spectrum was observed. This is due to the absence of the $-CH_3$ group and C-H band at 'x'=0.33 and 0.37 possibly due to the varied 'x' value during doping of Titanium ion to Barium Ferrite is responsible for this. Reflection of radiation is more,due to the more width of the FTIR inverted peaks. This reveals that in a given cross section more nanoparticles scattered radiation. Hence thenumber of nanoparticles in that cross section is more and the size of the nanoparticle is less. Therefore the broader peaks represent the formation of particles of smaller size.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cm-1
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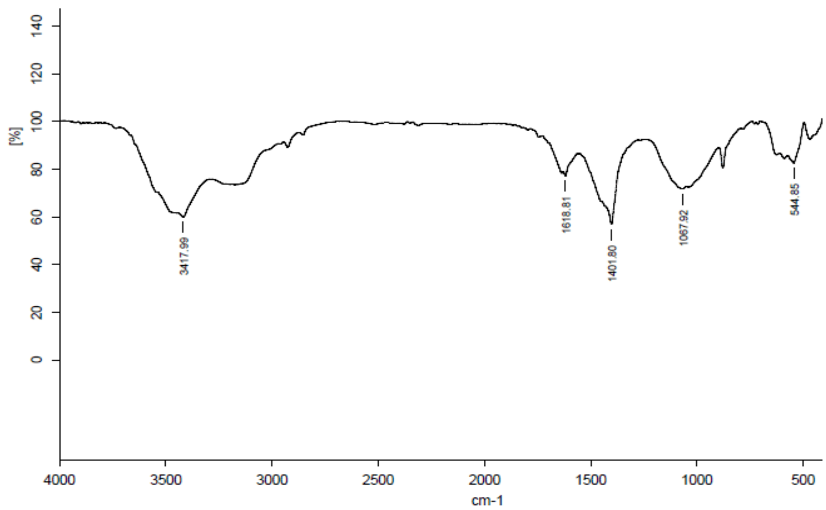
1036
580


[%]
cm-1
3417.99
1618.81
1401.80
1067.92
544.85

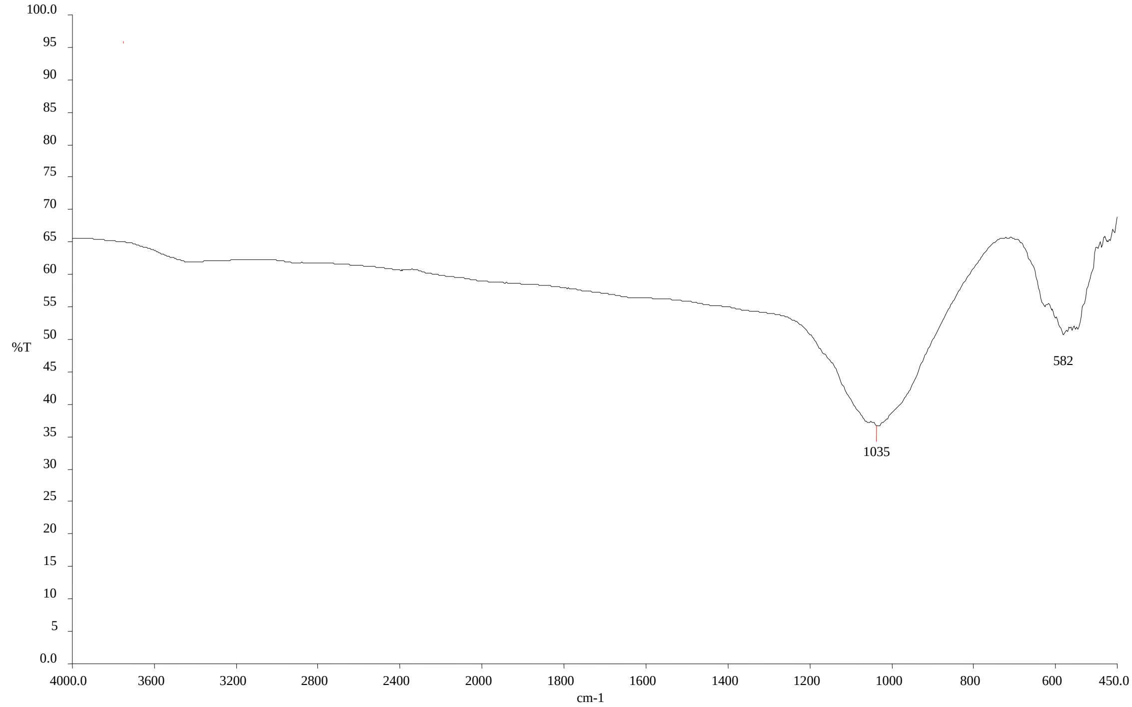


Figure 5: FTIR graphs of Ti-doped barium ferrite at 'x'=0.33 , 'x'0.35 and 'x' = 0.37 value at 950^{0}C temperatures

Existence of the metal-oxygen vibrational modes of the spinel compound is indicated by stretching Peak at 541cm^{-1} , Stretching peak at 1080cm^{-1}indicates C-O, bending peak at 1400 cm^{-1}indicates $-CH_3$ [18], stretching peak at 1618 cm^{-1} indicates remanants of C-H band and stretching peak at 3418 cm^{-1} indicates O-H[17].

V. Conclusion

Ti- doped barium ferrite (x=0.33,0.35 and 0.37) nanopowder has been successfully synthesized by Sol-gel technique. . As prepared powder is in nanometer range. The formation of the same has been confirmed by XRD, SEM and FTIR techniques.

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