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Synthesis and Structural, Morphological & FTIR Studies on Ferrite Powders Bafe $_{(12-X)}$ Ti $_X$o $_{19}$, Using Sol-Gel Method

Aparna.A.R.[1], Dr.Brahmajirao.V[2], Dr.Kartikeyan.T.V[3] and Dr.Moneesha Fernandes[4]

[1]*Ph.D. Research Scholar, Department of Nanoscience and Technology, JNTU, Hyderabad, India*
[2]*(Previously) Matrix Institute of technology, Cheekatimamidi (V), Bommalaramaram (M),[Jawaharlal Nehru Technological University,] HYDERABAD , Pin Code – 508116, &(at present) MGNIRSA, A Unit of D. Swami Nathan Research Foundation, Hyderabad-500029, A.P. INDIA .*
[3]*Scientist 'F', ASL, DRDO, Hyderabad, India,*
[4]*Scientist, Department of Organic chemistry, National Chemical Laboratory, Pune,India.*

Abstract : *This article presents the preparation of Ti-doped barium ferrite powders* $\boldsymbol{BaFe_{(12-x)}Ti_xO_{19}}$*for (x = 0.35) nanomaterial using sol-gel route, followed by the thermal insulation process and heat-treatment, recently reported by Wangchang Li et.al.,[1]. The pH of the medium and annealing temperatures were the aspects of concentration of our study in this communication. Nanomaterial is synthesized for the value of(x = 0.35) at three different temperatures. The phase structure and morphology were analysed by standard XRD, FTIR and SEM techniques.*

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

I. Introduction

Nano scale magnetic ferrite materials possess a set of unique magnetic and electrical properties and chemical stabilities [2, 3]. Ferrites might be a promising candidate as the microwave absorbing materials because of their high specific resistance, fascinating magnetic and electromagnetic properties. Also Polypyrrole (PPy) has aroused more and more attention for practical applications on the basis of its unparalleled architectural diversity and flexibility, excellent environmental stability, high conductivity, relatively low densities, and easy of preparation [4 to 6] . These properties are significant not only from a fundamental point of view, for example, blocking behaviour, nanoscale confinement, and nanomagnetism [7 to 9] , but also for their potential applications, such as high-density data storage, spin-electronics, bio separation, magnetic resonance imaging, and magnetically guided drug delivery systems [10 to 14].In recent years development of novel Nano materials to be used as absorbing coatings [15 to 18], with excellent microwave absorption properties, that are strongly dependent on material properties, including complex permeability, complex permittivity and resistivity, is assuming technological importance. 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 [19]. Kim et al obtained PET fabric / PPy composites and the electromagnetic interference shielding effectiveness of composites increased with the high electrical conductivity [20]. 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 [21(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 [22 to 25]. 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[26 to28].

Barium Hexaferrite ($BaFe_{12}O_{19}$) 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].

Venugopalan Anbarasuet.al. synthesized[40], magnetically ordered barium hexaferrite powders and the adjustment of magnetic properties for perpendicular magnetic recording media are realized through substitution of divalent cation (Ca) in the $BaFe_{12}O_{19}$ system The Ca2+ substituted $Ba_{1-x}Ca_xFe12O19$ (where x = 0.05, 0.1, 0.15 and 0.2) compounds have been prepared through solid state reaction technique. The powder X-ray diffraction analysis reveals that all the prepared compounds crystallized in magnetoplumbite hexagonal structure and the flat hexagonal platelet morphology of the crystallites was identified through scanning electron microscopy. The formation of magnetoplumbite structured $Ba_{1-x}Ca_xFe_{12}O_{\mathbf{19}}$ system due to mechanical activation was supported by micro-Raman measurements. From the room temperature magnetization studies, it was observed that the saturation magnetization (MS) and remnant magnetization (MR) values drastically decreases for the $Ba_{0.95}Ca_{0.05}Fe_{12}O_{19}$ compound which may be due to the existence of spin canting effect and leads to the reduction of super exchange fields.

Xin Tang et.al. [41], prepared a composite of polyaniline (PANI)-coated M-type hexagonal barium ferrite (M-Ba-ferrite) powder by an in situ polymerization of an aniline monomer in the presence of M-Ba-ferrite particles. They characterized the obtained composite by Fourier transform infrared spectra (FT-IR), X-ray diffraction (XRD) and transmission electron microscopy (TEM), to investigate the structure and microwave response properties. They observed that a continuous coverage of polyaniline has been produced on the platelet M-Ba-ferrite particle surface which has prominent influence on microwave response of M-Ba-ferrite particles and changed the character of frequency dispersion of microwave absorption .They concluded that a core–shell structure has been formed. They found the existence of an interaction at the interface of polyaniline macromolecule and barium ferrite particle, which influences the physical and chemical properties of the composite. The interaction and interfacial polarization are pointed out to be important factors contributing to the influence on microwave response of the PANI-coated ferrite composite powders.

In the present communication the preparation &characterisation nanopowder of BFTO was prepared using sol-gel method. The prepared powders were characterized using X-ray Diffraction (XRD), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIR).

II. Experimental Procedure

2.1 Why Solgel Method ?

Hexagonal ferrites are prepared by using various synthesis routes like: Hydrothermal synthesis (Duong et al.[42]; Drofenik et al. [43],). Reverse micelle-based method (Xu et al. 44]),Chemical precipitation (Pankov et al. [45]), Ammonium nitrate melts (Topal et al. [46]) , Precipitation in alcohol (Lisjak and Drofenik [47,48]), Mechanical alloying synthesis (Sharma et al.[49]), Citrate precursor synthesis (Sankaranarayanan et al. [50]), Low-temperature combustion synthesis (Huang et al. [51]), etc.

The sol–gel combustion method has the unique advantage for the low costs using simple equipment in large-scale high-purity. Martirosyan et al. [52, 53] reported vivid contrasts in between Solution Combustion Synthesis (SCS) and carbon combustion synthesis of oxides (CCSO) in the synthesis of nano ferrites. The sol–gel combustion synthesis of hexagonal barium ferrite was reported to be especially conspicuous in the process of converting $Fe_{\mathbf{2}}O_{\mathbf{3}}$ into barium ferrite.

The reaction mechanism of the precursor gel under the programmed thermal heating was determined by Wangchang Li et.al [1], by plotting TG/Differential Thermal Analysis (DTA) curve. According to them, the mechanism involved in the synthesis of Barium ferrite can be approximately detailed in the following manner. First, an endothermic reaction with a weight loss of 2.4% is observed at around 100 $^{\mathbf{0}}$C, corresponding to the evaporation of absorption water in the samples. An exothermic peak in the TG/DTA curve of the dried sol. around 200–220 $^{\mathbf{0}}$C is associated with a large weight loss of (60.01%). This indicated the self-propagation of the dried gel following the formation of Carbon dioxide, the Ferrite and BaO.

Huangs' [54] also reported exactly similar result. Citrate acts as reductant and Nitrate acts as oxidant, respectively in their self-propagating process, which synthesised the Barium ferrite. They attributed the observed further weight loss between 200$^{\mathbf{0}}$ C and 350$^{\mathbf{0}}$C in this typical mechanism to the dehydrogenation of the residual groups. Since we are using the ferrite procured directly from the manufacturer, we attempted our synthesis at three different calcination temperatures, adopted from the literature & the findings from the observations on the synthesis of Hexaferrite Powders by a Sol-Gel Auto-combustion of S. Alamolhodaet.al., [53],K Sadhana et.al, by microwave-hydrothermal method, [55]

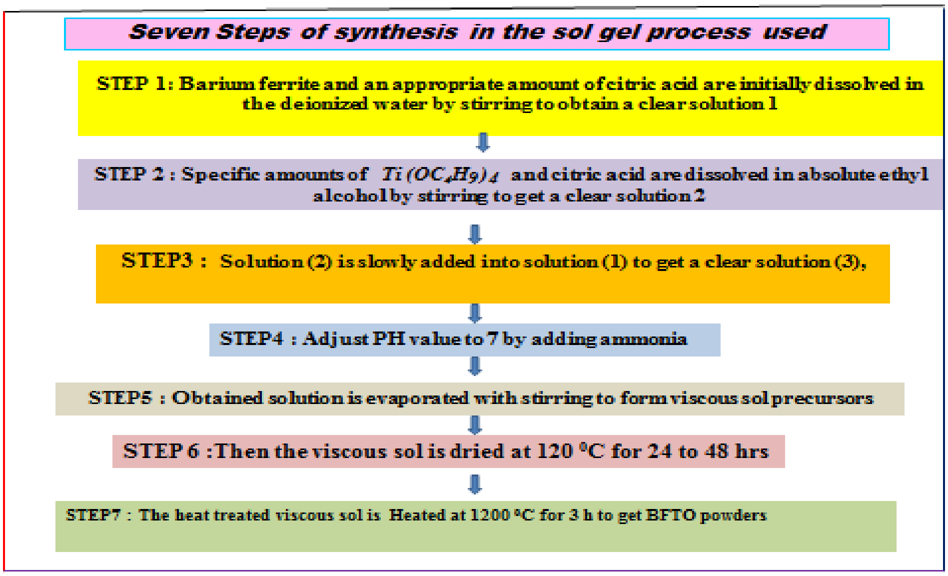
M.J.Molaei[56(a) and (b)], in his study on Magnetic property enhancement and characterization$BaFe_{12}O_{19}/Fe_3O_4$ and Fe/Fe_3O_4magnetic nano-composites, reported the effects of milling time and heat treatment temperature on the characteristics of powder mixture . The powders were studied by X-ray diffraction analysis, vibrating sample magnetometry, transmission electron microscopy 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. Heat treatment of the 40 h milled samples at 750–900 °C resulted in formation of Fe containing nano-composite

2.2 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 $Ti(OCH_2CH_2CH_2CH_3)_4$] 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.

2.3 Synthesis Of The Samples

The flow chart adopted and the photos of the equipment used are shown below



`Figure1 Flow chart used for the synthesis of BaFe $_{(12-x)}$Ti$_{(x)}$O $_{(19)}$ (x = 0.35),at NCL, Pune ,(India)

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 [52] 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 [57, 58]. 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** were chosen for the study of the phase formation by heat treatment. This was studied in 3 different samples of the BFTO powders so obtained.

Figure 2 Oil bath &magnetic stirrer cum heater ,with precise temperature control and provision for monitoring the number of revolutions per minute .The outer trough contains the oil whose temperature is maintained . the inner beaker contains the Nano material being synthesised

Figure 3 The cylindrical furnace ,with precise temperature control N.C.L.,Pune ,India ,The axial placement of the sample facilitates uniform regulation of the temperature

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 I.I.C.T.,Hyderabad ,A.P., INDIA .The FT-IR (Schimadzu Perkin-Elmer 1310), at I.I.C.T.,Hyderabad ,A.P.,India, was used to ascertain the metal-oxygen and metal-metal bond in the prepared ferrite sample.

III. Results And Discussion

4.1 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 the $BaFe_{(12-x)}Ti_xO_{19}$(x = 0.35) powders sintered at 850^0C, 900^0C and 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 20 to 50 nm and was calculated using equation (1).

The Average grain size has been calculated using Debye – Scherrer's [64] equation (1) as shown below

$$D = \left[\frac{0.9\lambda}{\beta_{\frac{1}{2}}\cos\theta}\right] \text{--------------------} (1)$$

Where λ = wave length of the x- ray beam

$B_{1/2}$ = Angular width at the half max intensity

θ = Braggs angle

Table1

850^0C		900^0C		950^0C			
2θ(deg)	D(nm)	2θ(deg)	D(nm)	2θ(deg)	D(nm)	a(Å)	c(Å)
33.550	28.716	33.55	27.66	33.55	39.04	4.622	4.6239
35.950	29.171	35.95	28.34	36.05	32.68	4.992	4.9921
54.450	26.75	54.45	22.204	54.45	28.75	4.762	4.76257

Table1 Average grain size D,2θand lattice parameter 'a' and 'c' values at chosen three temperatures

Analysing the effect of the tempering temperature, we can observe from figure(1) that for the sample sintered at 850^0C and 900^0C less number of peaks are formed whereas at 950^0C well developed narrow peaks are seen which indicates that formation of nanoparticles are good at higher temperatures.

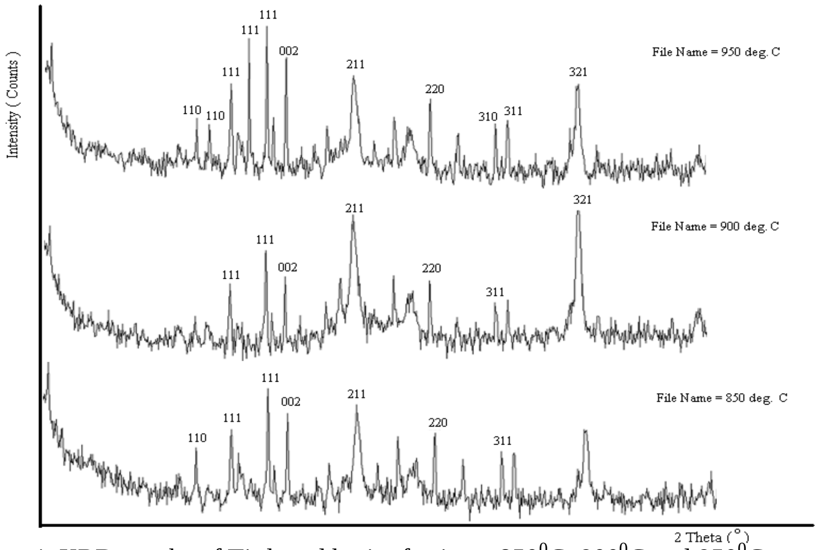


Figure 4 XRD graphs of Ti-doped barim ferrite at 850^0C, 900^0C and 950^0C temperatures

4.2 Scanning Electron Micrograph (Sem)

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

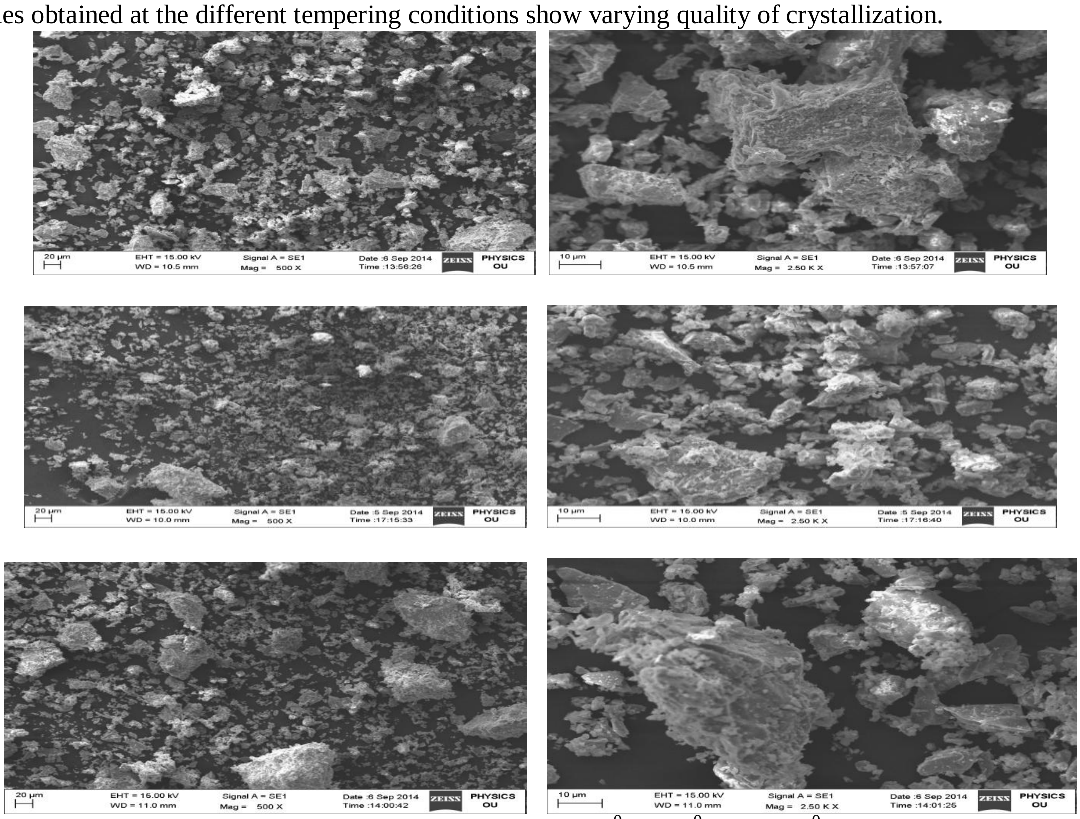


Figure5 SEM pictures of Ti-doped barium ferrite at 850^0C, 900^0C and 950^0C temperatures (at two magnifications)

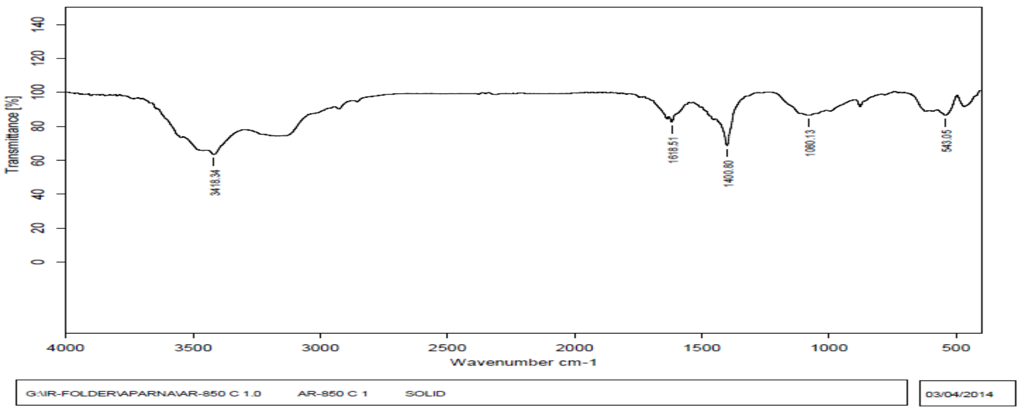
4.3 Fourier Transform Infra Red Studies (FT-IR)

FTIR Technique is extremely competent for the charecterization of organic or inorganic materials in the form of fingerprints obtained in a Transparency (or Reflectance) intensity profile of the IR radiation plotted against wavenumber. The vibrational spectrum of a molecule is considered to be a unique physical property and is characteristic of the molecule. As such, the infrared spectrum can be used as a fingerprint for identification by the comparison of the spectrum from an ‘‘unknown’’ with previously recorded reference spectra. [59] This is the basis of computer-based spectral searching. In the absence of a suitable reference database, it is possible to effect a basic interpretation of the spectrum from first principles, leading to characterization, and possibly even identification of an unknown sample It provides qualitative compound Identification [60]in the form of band stretching, binding ,out of plane bending ,band shortening [61,62] (at a specific wavenumber) etc.,

Fourier Transform Infra-Red (FT-IR) spectra have been recorded using Schimadzu 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., 3418.34, 1618.51, 1400.80, 1080 and 543cm^{-1}.It is observed that a peak corresponding to 543cm^{-1} does not appear at the phase formation temperature of 900^{0}C,in the FTIR spectrum. Even in the XRD plots corresponding minor peak formation around ($2\theta = 73.5^0$)is missing ,and distinct formation of the corresponding peaks is observed at 950^{0}C. This is attributed to the absence of the BFTO,at 900^0 C .possibly due to the doping of Titanium in to Barium Ferrite is responsible for this. Other details of reasoning are furnished with our Magnetic and impedence measurement data, being communicated elsewhere [65].

Figure 6 F.T.I.R. Sample analysis

Figure 7 pellet preparation for F.T.I.R.



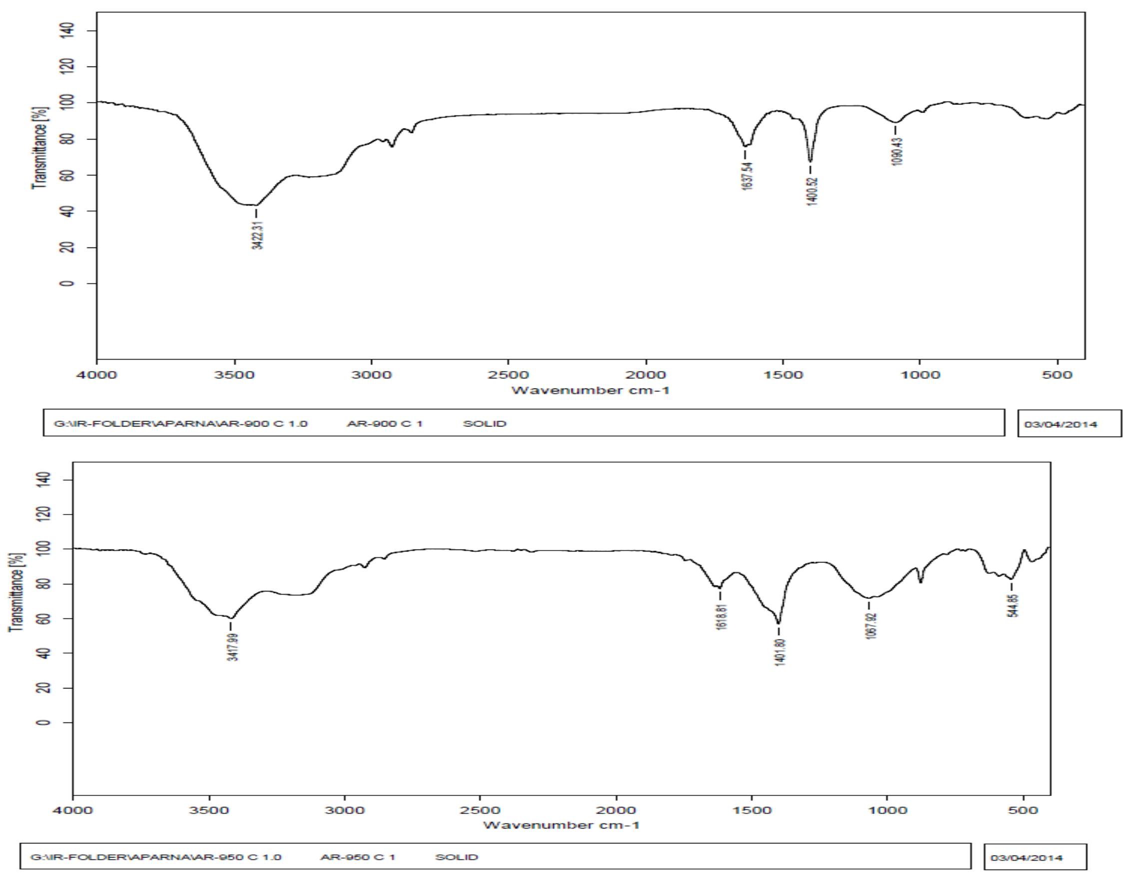


Figure 8 FT-IR graphs of Ti-doped barium ferrite at 850^0C, 900^0C and 950^0C temperatures

Stretching Peak at 541cm^{-1}indicates existence of the metal-oxygen vibrational modes of the spinel compound, Stretching peak at 1080cm^{-1}indicates C-O, bending peak at 1400 cm^{-1}indicates $-CH_3$ [63], stretching peak at 1618 cm^{-1} indicates remanants of C-H band and stretching peak at 3418 cm^{-1} indicates O-H[1].

IV. Conclusion

In summary, we have successfully synthesized Ti- doped barium ferrite (x=0.35) nanopowder by using Sol-gel technique. The formation of Titanium doped Nano ferrites has been con-firmed by XRD,SEM studies. FT-IR studies on the same are also reported. The crystal-lite size is found to be in the range 20-50 nm.

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