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Research Article

SYNTHESIS AND CHARACTERIZATION OF $SrFe_{12}O_{19}$ DERIVED FROM IRON ORE TAILINGS

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ABSTRACT

In this work, we report an experimental study on nano-crystalline $SrFe_{12}O_{19}$ powder derived from the iron ore tailings. Iron ore tailings is a mineral waste contains 15- 20% Fe_2O_3 remaining mostly SiO_2. Iron from its tailings extracted out in the form of $FeCl_3$ and mixed with appropriated amount of $SrCl_2$. Then the mixed aqueous solution containing $FeCl_3$ and $SrCl_2$ was precipitated with citric acid to form corresponding citrate complex. The formed citrate complex precipitate separated out through filtration, washed, oven dried and finally calcined at 800 ^oC for 3 hours to obtain $SrFe_{12}O_{19}$ nano-crystalline powder. The same product was again obtained through Urea hydrolysis. Several advanced techniques have been employed to characterize the prepared $SrFe_{12}O_{19}$ samples. XRD reveals the formation of $SrFe_{12}O_{19}$ as major phase and trace amount of Fe_2O_3 and Strontium oxides were found as minor phases. UV-Visible spectrum of samples shows strong absorbance in the UV and visible region (650 & 500nm). TG curve demonstrates that oven dried sample shows weight loss up to 400 ^oC indicating for the dehydration followed by decomposition of citrate complex to form metal oxide composite. The FTIR result exhibits the bands for M-O bond (M= Sr, Fe) vibration. VSM curve suggests that $SrFe_{12}O_{19}$ obtained by co-precipitation method shows more magnetization than the sample prepared by urea hydrolysis method. Thus, the present work showed that iron ore tailings could be used as one of the raw material for synthesis of rare earth ferrite for its potential application in various field.

INTRODUCTION

Rare earth ferrites have found potential applications in the area of solid-oxide fuel shells, environmental catalyst, chemical sensors, magnetic materials, electrode materials, etc, because they exhibit good electrical, optical, structural and magnetic properties [1-3]. These ferrites provide the best available combination of electrical insulators and magnetic materials with remarkable flexibility in controlling magnetic, conductive properties and also the crystal lattice parameters [4-5]. The enhanced property observed for ferrites at high frequencies leads to a wide variety of microwave phenomena. The traditional ferrites, such as spinels & garnets, are used in microwave range whereas self-biased hexa-ferrites are being used at very high frequencies. They can efficiently replace a large external bias magnet. Ferrites are extensively used in radar, audio-video and digital recording, bubble devices, memory cores of computers, satellite communication and have a wide range of applications from microwave to radio frequencies such as antenna cores in radio receivers, fly back transformer in TV-picture tubes, broadband transformers, mechanical filter, ultrasonic generators, moderators, phase shift isolators, modern day telephone exchange, computers and control equipment [6-7]. Ferrites, in comparison to pure metals, are regarded as better magnetic materials because of their high resistivity, lower cost, superior magnetization and easy to manufacture [2]. Although traditional applications of ferrite in discrete components is known for long time, there are several problems to overcome in order to ensure the application of ferrites in low-cost, mass produced systems, as mobile communication, automotive sensors, consumer circuits [6-9]. It has been reported that hexaferrite has general formula $MeFe_{12}O_{19}$, where Me is a divalent ion of a large ionic radius, such as Ba^{2+}, Sr^{2+}, or Pb^{2+}.They are very stable and have high electrical resistivity [10-11]. $SrFe_{12}O_{19}$ is one among them shows an excellent ferrimagnetic property [12-13]. Due to its magnetic properties, it can be used as permanent magnets, and as a component in microwave and high frequency devices due to its high intrinsic coercivity, large crystal anisotropy and low cost [14-16]. The high coercivity of materials makes it resistant to becoming demagnetized and this is an essential characteristic for a permanent magnet. The hexaferrite, $SrFe_{12}O_{19}$ also possesses a high magnetic permeability [17].

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In order to prepare inexpensive ferrite, it is better to use easily available low cost starting materials [18]. The mine tailings are generated as the wastes worldwide as a result of exploration, excavation, blasting, beneficiation and extraction of mineral ores. The extensive mining activities and increasing low grade ores are resulted into the generation of mine tailings in large quantities, which could lead to environmental and disposal problems [19-20]. Iron ore tailings, which are a major waste in steel industries, have not been explored extensively so far for the value addition to the waste. Therefore, in this study, we have made an attempt to use more abundant mineral waste such as iron ore tailings as one of the starting material to synthesize $SrFe_{12}O_{19}$. The present experimental work illustrates the detailed synthesis and characterization of $SrFe_{12}O_{19}$ derived from iron ore tailings.

MATERIALS AND METHODS

Preparation of $SrFe_{12}O_{19}$

In the present work, co-precipitation method (CPP) and urea hydrolysis method (UR) have been adopted to prepare hexaferrites , i.e., $SrFe_{12}O_{19}$ from iron ore-tailings. Iron in the form of $FeCl_3$ was extracted out from the iron ore tailings by HCl digestion at 90 °C followed by filtration to separate out the acid insoluble SiO_2 and Al_2O_3. Then, the required quantity of $SrCl_2$ was dissolved in distilled water and mixed with appropriated quantity of of aq. $FeCl_3$ solution .The molar ratio of Sr and Fe were maintained at 1:12 ratio. In CPP method, the mixed solution was heated at 90 °C under stirring condition and few drops of conc. HCl was added to get a clear solution. To this solution required amount of citric acid was added as chelating reagent to form corresponding citrate complex. Then the pH was maintained at 9 by addition of KOH solution drop wise to obtain the complex precipitate. The precipitate was aged in its mother liquor overnight. Then it was retrieved by centrifugation and washed with hot water repeatedly to remove Cl ion from it. The chlorine free precipitate was subsequently dried in an oven at 150 °C for 12 hours and was ground to form fine powder. The prepared sample was then calcined at 800 °C for 3 hours. The sample prepared via this route is denoted as $SrFe_{12}O_{19}$-CPP.

The same compound was again obtained through Urea hydrolysis method. In this approach, the mixed aqueous solution of $SrCl_2$ and $FeCl_3$ were taken in a round bottom flask and refluxed with excess amount of urea dissolved in distilled water at 200 °C for 4-5 hrs. At the beginning of hydrolysis, the color was dark brown which gradually turned into a muddy-mustard color. The obtained precipitate was cooled down to room temperature and left overnight to settle down. The precipitation was separated out by centrifugation and washed thoroughly with hot distilled water. The precipitate was dried at 100 ^oC for 12 h and finally calcined at 800 °C for 3 hours. The sample so obtained was referred as $SrFe_{12}O_{19}$-UR.

Characterization

Before calcination at 800 °C, the oven dried nano-crystallite $SrFe_{12}O_{19}$ powder samples prepared from the above two methods were characterized by thermo- gravimetric analysis (TGA, Universal V 4.5A TA instrument) at a heating rate of 10^oC/min in atmospheric environment from room temperature to 800 °C. After calcination, the structural, morphological, magnetic characterizations of the samples were performed by using several advanced experimental techniques. For Phase identification, X-ray powder diffraction patterns were recorded at room temperature, using X-ray powder diffractometer, Panalytical instrument which has equipped with Cu K_α as target. Fourier transform infrared spectroscopy measurement was carried out by using Spectru GX Model, Perkin Elmer India Pvt Ltd. Spectrometer. Laser Raman-Spectra were obtained with help of Reinshaw Raman spectrometer. Diffuse Reflectance Spectra of the samples were obtained by using UV-Visible spectrophotometer (Cary instrument) attached with reflectance attachment. And the magnetic properties of the samples such as intrinsic coercivity and saturation magnetization were studied at room temperature using Vibrating sample magnetometer (PAR 155).

RESULT AND DISCUSSION

The thermo-gravimetric curves (weight % versus temperature) for $SrFe_{12}O_{19}$ powder prepared by CPP and UR methods are given in figure 1 (a) and in figure 1 (b) respectively. Both the samples showed the multistage decomposition processes as reported by V. Harikrishnan *et. al* [15]. The sample from CPP method displayed two stages of weight loss. One at room temperature to 100 °C and it is due to the loss of absorbed moisture. The second stage weight loss is from 100 °C to 350 °C and is attributed to conversion of hydroxide to oxide form. However, the sample prepared by UR method showed the first stage weight loss from room temperature to 250 °C and it is due to the loss of moisture and decomposition of any residual urea present in the sample.

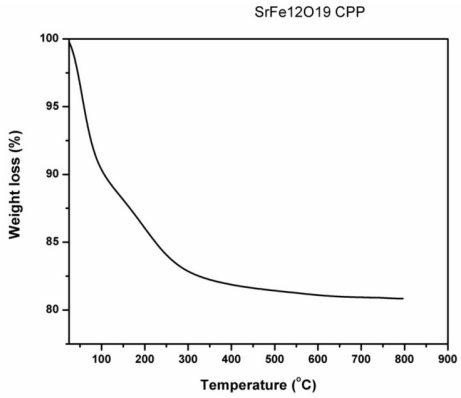

Figure 1 (a) The weight % versus temperature curve of oven dried $SrFe_{12}O_{19}$ nano-crystalline powder prepared by CPP Method

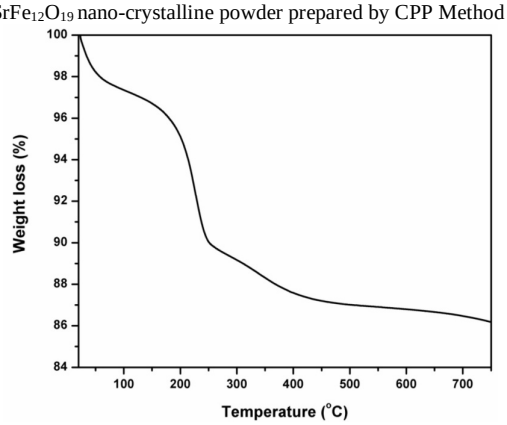

Figure 1 (b) The weight % versus temperature curve of oven dried $SrFe_{12}O_{19}$ nano-crystalline powder prepared by UR Method

The second stage weight loss in sample made from UR is 250 °C to 400 °C and it is due to the hydration of hydroxide to oxide form. Above 400 °C, no significant weight loss is observed from the TGA curves. Looking into the TGA curves it is found that the weight loss % is almost flat to temperature axis at about 800 °C and thus, the calcination temperature is decided at 800 °C for both the samples.

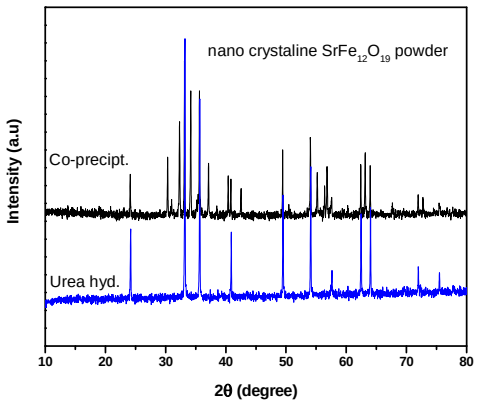

Figure 2 X-ray diffraction pattern of $SrFe_{12}O_{19}$ powder prepared by CPP and UR methods

The XRD patterns of $SrFe_{12}O_{19}$ prepared by both CPP & UR methods are shown in figure 2. Both the samples exhibit prominent diffraction peaks of $SrFe_{12}O_{19}$. Diffraction peaks of both samples have well matched with standard JCPDS file of $SrFe_{12}O_{19}$ (File—00-33- 1340). More number of diffraction peaks is observed for CPP sample as compared to UR-hydrolysis sample. The average crystallite size of both the samples is obtained from the FWHM of diffraction peaks using Scherer equation. It is found that $SrFe_{12}O_{19}$ nano-crystalline powder prepared by UR method has an average crystallite size of 89 nm whereas prepared by CPP method has an average crystallite size of 97 nm. Thus, $SrFe_{12}O_{19}$-UR sample has a lower crystallite size than the sample prepared by CPP method. This difference might be due to the two different approaches adopted for preparation.

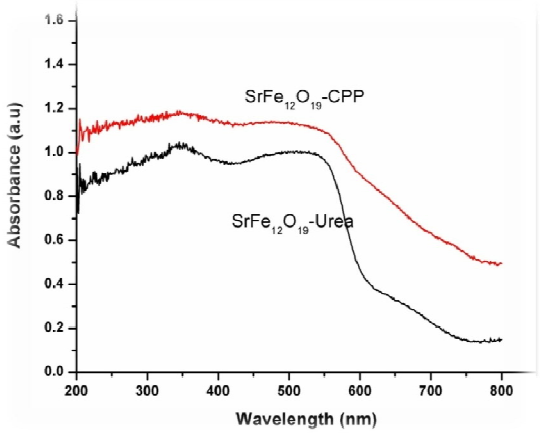

Figure 3 Absorbance (α) of both the samples prepared by CPP and UR methods and calcined at 800 °C for 3 hours

Figure 3 shows the absorbance spectra of both samples in the range 200 nm to 800 nm by transforming the diffuse reflectance spectra to absorbance by the use of the Kubelka-Munk [20] function for diffused reflectance. This function is valid when the particle size is comparable to or smaller than the incident wavelength of light. So it is applicable to our samples. Both the samples show adsorption at 370, 590, 685 nm of wavelengths. Among all these adsorption of wavelength, the absorption at 590 nm is observed to be the most prominent one.The band gap of samples are calculated by plotting $(\alpha h\nu)^2$ versus incident photon energy and linearly regressing the linear portion of the $(\alpha h\nu)^2$ to zero. The point where the line meets the energy axis represents the band gap energy. Figure 4 shows the $(\alpha h\nu)^2$ versus incident photon energy variation from which the band gap for both the samples are extracted. The band gaps are found to be 1.85 eV and 2.05 eV for samples made from CPP and UR methods respectively.

Figure 5 shows the FTIR spectra of both the samples prepared by two different methods. The spectra of samples exhibit peak positions at same wavenumbers. The FTIR spectra reveals characteristic absorption bands at 3780, 3341, 2959, 2952, 2855, 1733, 1593, 1456, 1022, 603, 550,471 $cm^{-1.}$

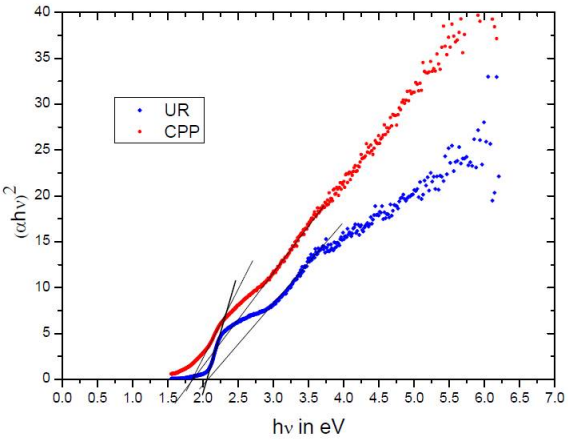

Figure 4 The dependence of $(\alpha h\nu)^2$ on the incident photon energy (hν) for both the samples.

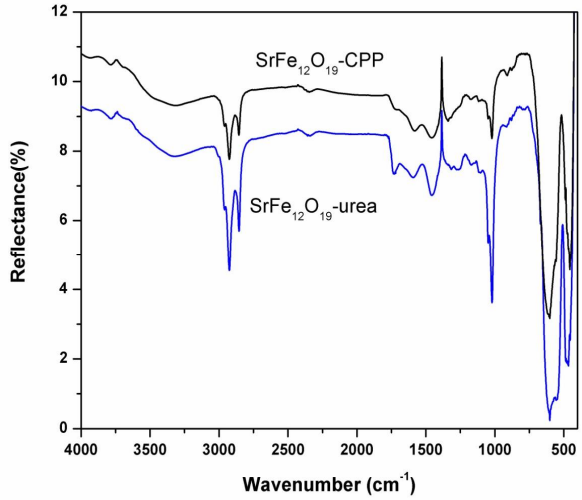

Figure 5 FTIR spectra of both the samples prepared by two different methods.

The absorption bands at 3780, 3341 cm^{-1} are corresponding to the stretching and bending bands of surface hydroxyl group (-OH) acquired from absorbed water molecules. The absorption bands at 2959, 2925 and 2855 cm^{-1} can be ascribed to the atmospheric CO_2. The absorption bands at 1456 and 1022 cm^{-1} can be ascribed to the stretching modes of and Fe-OH-Fe groups. The absorption band seen at 603, 550, and 471 cm-1 are due to the metal-oxygen (Fe-O) stretching vibrations of hexaferrites.

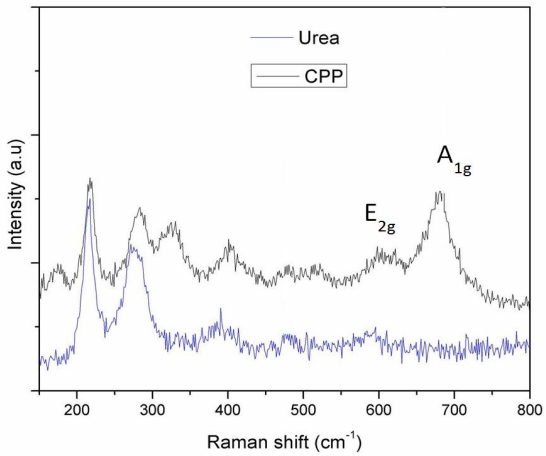

Figure 6 Raman spectra of both the samples prepared by CPP and UR methods and oxidized at 800 °C for 3 hours

The Raman spectra of the $SrFe_{12}O_{19}$ samples was recorded at room temperature in the spectral range of 150-800 cm^{-1} are shown in figure 6. The vibrational band at 697cm^{-1} is present in Urea hydrolysis sample and confirms the presence of trigonal bipyramidal site. The peak observed at 623 cm^{-1} is assigned to E_{2g} vibration mode due to stretching vibration of Fe-O at the $4f_2$ octahedral sites, while peak at 697 cm^{-1} is assigned to A_{1g} vibration mode dominated by motions of the Fe-O bonds at the bipyramidal 2b site.

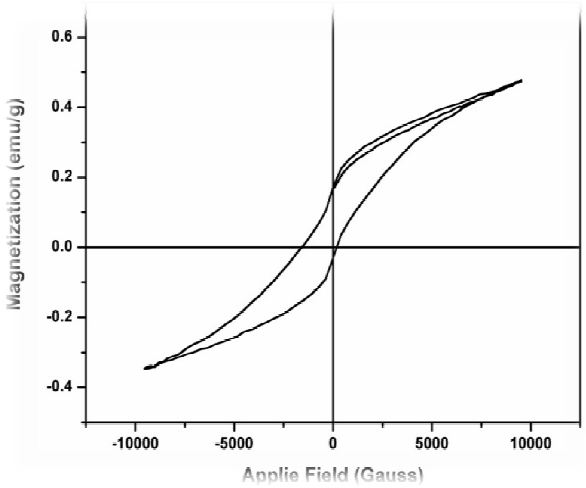

Figure 7 (a) Room-temperature magnetic hysteresis loops of $SrFe_{12}O_{19}$ prepared by UR

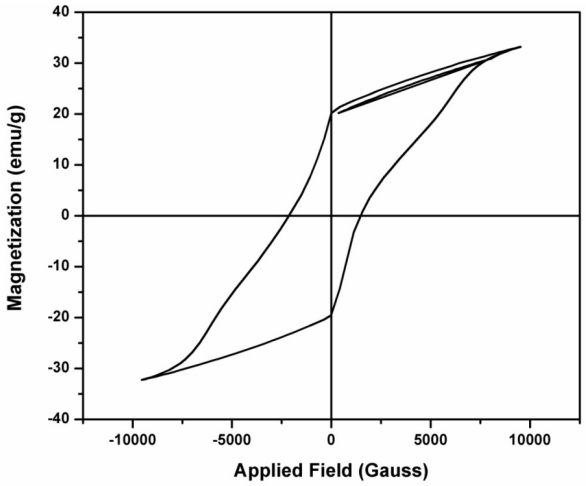

Figure 7 b Room-temperature magnetic hysteresis loops of $SrFe_{12}O_{19}$ prepared by CPP

The Vibrating sample magnetometry (VSM) of $SrFe_{12}O_{19}$-CPP is shown in figure 7 (a) & that of $SrFe_{12}O_{19}$-UR is shown in figure 7 (b). Both the samples exhibit ferrimagnetism. But the sample prepared by CPP method shows greater magnetization and more intrinsic corecivity than the sample prepared by urea-hydrolysis method. This may be attributed to the particle size difference of samples prepared by two different methods.

CONCLUSION

$SrFe_{12}O_{19}$ powder is prepared successfully by chemical co-precipitation and urea-hydrolysis methods from iron ore-tailings. The average crystallite sizes extracted from x-ray diffraction data for the samples prepared by co-precipitation and urea-hydrolysis methods are found to be 97 nm and 85 nm, respectively. The sample obtained by co-precipitation method shows high greater magnetization, high particle size, more intrinsic corecivity but lower band gap as compared to the sample prepared by urea-hydrolysis. Both the samples exhibit ferrimagnetism. In summary, value addition to waste is successfully employed in this investigation. Hence, thought can be given to prepare more materials by using this mineral waste.

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