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Preparation and magnetic properties of nano size Zirconium ferrite particles using Coprecipitation method

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Abstract: Zirconium Ferrite ($ZrFe_2O_5$) was prepared by coprecipitation method at temperature as low as 80°C. The prepared samples were calcined at different temperatures like 400°C and 800°C. Synthesized nanoparticles were characterized by using X-ray diffraction (XRD), Scanning Electron Microscope (SEM), vibrating sample magnetometer (VSM) and transmission electron microscopy (TEM). We found that the size of nanoparticles increased with the increase in calcinations temperature. TEM was used to characterize the microstructure of the samples and particle size determination, which exhibited the formation of spherical nanoparticles.
Keywords: Nanostructured materials, Precipitation, Magnetization, Scanning electron microscopy (SEM), Transmission electron microscopy (TEM).

1. Introduction:

Nanoparticles have received increasing attention because they exhibit unusual physical and chemical properties significantly different from those of relatively larger particles of the same materials due to their extremely small size and large specific surface area[1-5]. Precipitation is one of the simplest techniques to prepare particles of numerous materials. Hence co-precipitation method was used for the synthesis of this nano particle-Zirconium Ferrite. However, it often is not easy to synthesize mono-dispersed nanoparticles using this technique due to the coagulation of particles. Hence polyethylene glycol was used as a protective agent and stabilizer to overcome this problem [6].

2. Experimental Procedure:

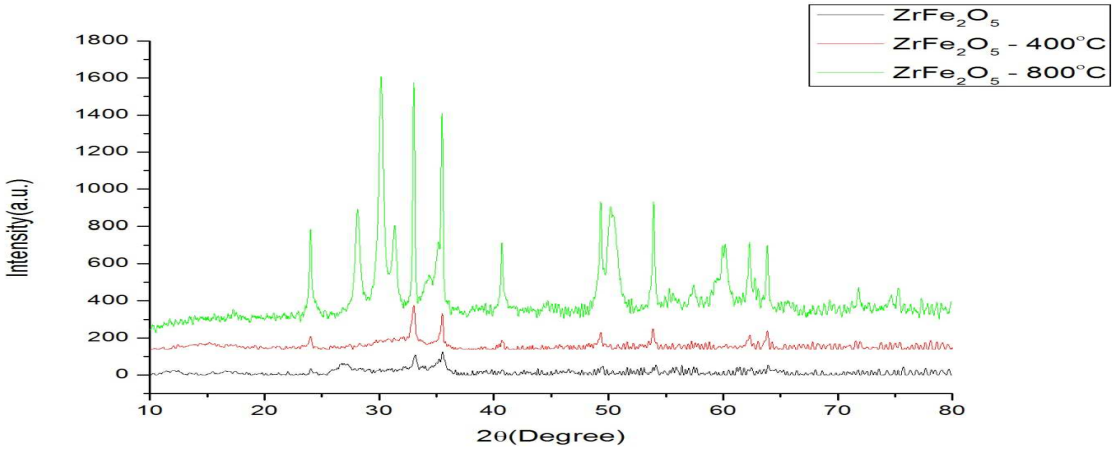
The starting materials used were Ferric chloride anhydrous ($FeCl_3$), zirconium (III) chloride ($Zrcl_3$) of 99.999% purity and sodium hydroxide (NaOH), all from Alfa Aesar and were of Analytical Grade. Polyethylene glycol–400(PEG -400) was used as a surfactant. The molarity of the coprecipitation agent (NaOH) was 3mol/l. The solution of $FeCl_3$, $Zrcl_3$, in their stoichiometry (50 ml of 1.4M $FeCl_3$, 50 ml of 0.6M $ZrCl_3$,) were mixed in double distilled de – ionized water. The salt solutions were mixed together with continues stirring. The neutralization is carried out with NaOH solution, and the pH is maintained around 12.Few drops of PEG – 400 were added to the precipitate and heated at 80°C using a hot plate with continues stirring for 2 hrs. The resultant precipitate was then cooled to room temperature. To remove the sodium and chloride compounds, the precipitate was washed and filtered several times with deionized distilled water. The precipitate was then dried at 100°C for overnight. The dried sample was fluffy mass in appearance which is grinded for 2

hrs, and the resulting powder was sintered for 5 hrs at 500°C. After sintering the sample was again grinded using the ball milling apparatus and the resulting powder sample was subjected to XRD, SEM and TEM characterization.

3. Results and Discussion

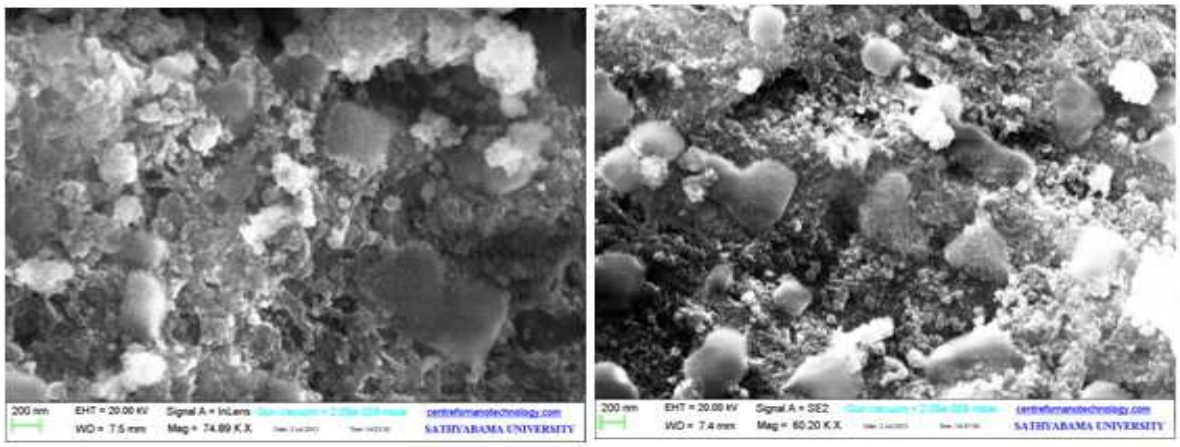
3.1. XRD Analysis:

Powder X-ray diffraction measurements were carried out at room temperature with a diffract meter system equipped with a monochromator in the diffracted beam. Scherrer equation was used to determine the crystallite size. The crystallite size was found to be 38 nm.



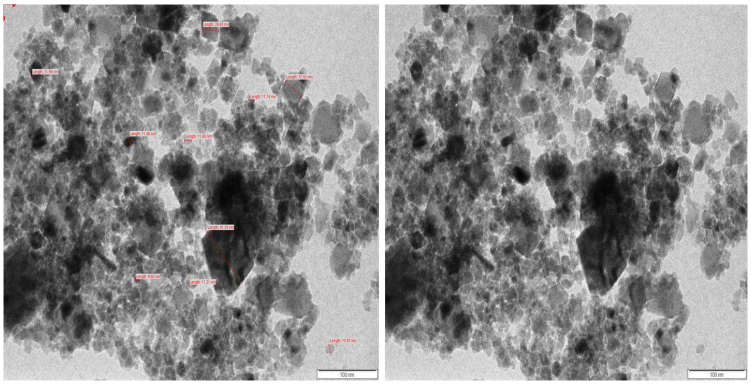
3.2. SEM:

The morphology was examined by Field Emission Scanning Electron Microscope (FE-SEM) using SUPRA 55-Carl ZEISS instrument. Fig. shows SEM micrographs of the powders. SEM images reveal that the sample surfaces exhibit well defined crystalline nanoparticles of spherical shapes with soft agglomeration. The crystalline size is around 34 nm which is nearer to the size obtained in XRD analysis.



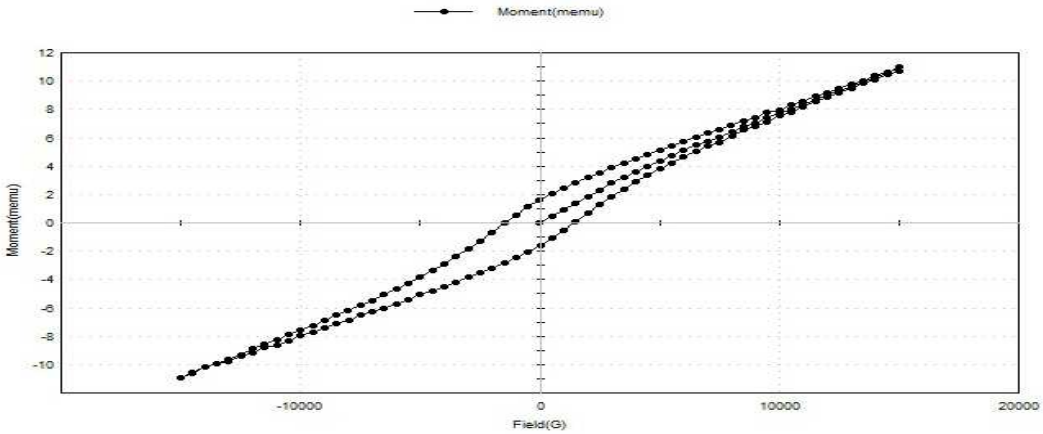
3.3. TEM:

The zirconium ferrite nanocrystals were morphologically characterized by means of transmission electron microscopy. A Zeiss EM 910 with a LaB6 cathode transmission electron microscope was used in the current study. TEM images were taken at an acceleration voltage of 100kW. Representative TEM patterns of different magnifications are depicted in the given figure for the synthesized zirconium ferrite nanocrystals. The influence of calcinations temperature was also investigated by carrying out TEM analysis under different curing stages. From the TEM image the size of the $ZrFe_2O_5$ is found to be 34nm.



3.4. VSM:

The magnetic properties of the synthesized material at 800°C were investigated using VSM. The magnetic hysteresis loops measured at room temperature are shown in fig (6) with a maximum applied field upto 10 Oe. The observed coercivity (H_c), remanent magnetization (M_r), saturation magnetization (M_s), depends on factors such as density, porosity grain size and ions superexchange interaction [16]. The value of remanent magnetization (M_r) is 1.632 emu/g, saturation magnetization (M_s) is 10.99 emu/g.



4. Conclusion:

Zirconium Ferrite are prepared by co-precipitation method and annealed at 400 and 800°C. The XRD spectra reveal the average crystallite size to be in the range of 35 - 38nm.The morphology and the particle size was determined using FESEM and TEM. The remanent magnetization (M_r), saturation magnetization (M_s) of the Zirconium ferrite decreases with the increase in calcinations temperature. The saturation magnetization decreases, whereas there is increase in the coercivity which is attributed to the decrease in grain size and increase in porosity.

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5. References

1. C. Hayashi, Phys. Today 40 (1987) 44.
2. H. Gleiter, Progr. Mater. Sci. 33 (1989) 223.
3. J.H. Fendler, Chem. Rev. 87 (1987) 877.
4. Y.K. Sun, I.H. Oh, K.Y. Kim, Ind. Eng. Chem. Res. 36 (1997)4839.
5. S. Ayyappan, R.S. Gopalan, C.N. Subbanna, C.N.R. Rao, J. Mater. Res. 12 (1997) 398.
6. H.Hirai, J. Macromol. Sci. Chem. A13 (1979) 633.

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