NOVEL DESIGNED PROCESSING METHOD FOR THE SYNTHESIS OF SILICA SUPPORTED MANGANESE FERRITE NANOPARTICLES

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A B S T R A C T

Newly designed silica supported manganese spinel ferrite nanoparticles & their processing technique has been successfully developed at room temperature. The synthesized nanoparticles are crystalline in nature & their other properties such as optical properties, particle size, conductivity etc. have been studied. The optical properties of synthesized nanomaterial were studied using UV–Visible spectrometer with the maximum wavelength absorptivity at 313 nm. Particle size of the sample was experimentally calculated by using TEM & X–ray Diffraction technique and found to be 42 nm and was in agreement with the theoretical values. Other thermal stability properties also havebeen studied using TGA & DTA

Keywords:Spinel manganese ferrite nanoparticles, Optical activity, Particle size, Magnetic Properties etc.

1. Introduction

In the literature, several methods were reported for the synthesis of Nanometric for their extra-ordinary physical and chemical properties. Silica supported manganese ferrite nanoparticles areof particular interest due to their enhanced magnetic, optical, electrical properties and its different properties depend on their particle size. Manganese spinel ferrite present in AB_2O_4 crystal structure consists of twocation sites: 8 tetrahedral A sites and 16 octahedral B sites, while oxygen anion sites are represented by O.[1] The nanosized spinel ferrites possess superior controlled properties than the bulk material.The properties of manganese ferrites highly depend on the morphology, particle size and composition. The spinel manganese ferrite shows magnetic, electrical,optical applications. These are also used in applications such asdrug delivery,[2] Media recording devices,[3] biosensor,[4] ferrofluids,[5] MRI technology.[6] In the previous reports, synthesisof manganese ferrite nanoparticles make the use of tedious methods such as thermal treatment method,[7]sol–gel method,[8] hydrothermal method,[9] reverse micelles method.[10]

In the continuation of our research interest, we herein reportto design a novel method for the synthesis of silica supported manganese spinel ferrite nanoparticles using chemical co–precipitation method. In comparison to the previous attempts, our chemical co–precipitation method is excellent due to use of economical chemicals, eco-friendly route with no hazardous effect on environment. These newly designed silica supported manganese spinel ferrite nanoparticles have been studied at higher temperatures also for their controlled morphology as size & shape. It is characterized by using different techniques and corresponding afford was excellent in electrical conductivity, maximum absorption of UV Visible spectrum for band gap.

2. Experimental

2.1 MATERIALS

$Mn(NO_3)_3.4H_2O$, $Fe(NO_3)_3.9H_2O$, polyvinylpyrrolidone as capping agent & hydrazine hydrate were procured from HiMediaand used without any purification.

2.2 METHODS

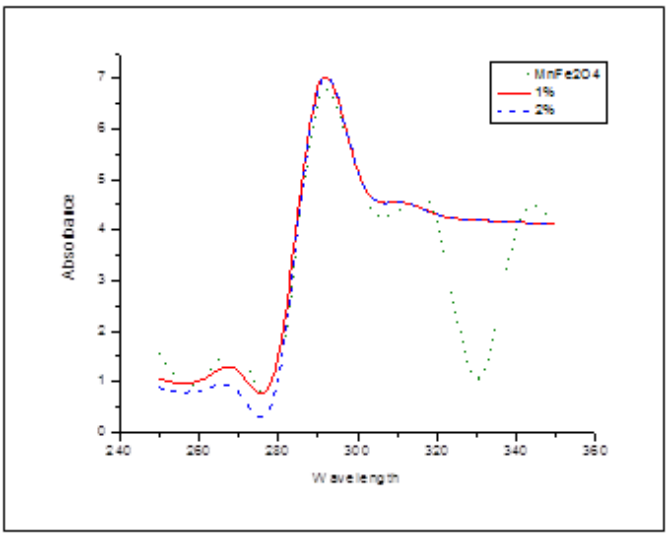
The silica supported manganese ferrite nanoparticles were synthesized by using chemical co-precipitation method. In a typically experimental procedure,a mixture of $Mn(NO_3)_3.4H_2O$, $Fe(NO_3)_3.9H_2O$ and tetraethyl orthosilicate silane (TEOS) were taken in 1.2:0.5 proportion and dissolved in double deionized water and the reaction mixture was heated at 40^{o}C followed by vigorous stirring and the capping agent PVP (0.01M) was added in stoichiometric amount upto 10minutes and then the reaction contents were cooled. Continue the stirring and 0.1 M hydrazine hydrate was added drop wise into the reaction mixture to maintain the pH = 09 to get stability of precipitate within one hour at room temperature. Then the precipitate was separated out by filtration and washed with mild hot distilled water. The resulting precipitate was dried at 30^{o}Ctemperature. The dried product was calcinated at 30^{o}C for 2 hours to recover $MnFe_2O_4@SiO_2$ powder.

3. Results & Discussion

We have successfully synthesized $MnFe_2O_4@SiO_2$at room temperature. Absorption peak and band gap of $MnFe_2O_4@SiO_2$ is characterized by using UV–Visible Spectrophotometer. The particle size and crystalline structure was confirmed by using X–Ray Diffractometer (XRD) technique from the scanning angle of 10^o to 80^o. The FT-IR gives information about functional group and surface composition. The thermal decomposition of $MnFe_2O_4@SiO_2$ have been studied using temperature range of 30 ℃ to 900 ℃ at TGA and DSC i.e. thermo-analytical technique used to measure temperature required for sample. In continuation of our study, TEM technique was used to study the morphology of $MnFe_2O_4@SiO_2$ nanoparticle and for calculating the granular particle size of $MnFe_2O_4@SiO_2$.

3.1 UV ABSORPTION SPECTRA

The absorption spectrum was used to study optical properties of the synthesized silica supported manganese spinel ferrite nanoparticles. In the experimental result, it has been slightly soluble in 0.003 M Hydrochloric acid for analyzing the UV- Visible spectrum. From this the band gap and the molecule experiences,the electronic transitions were determined. In the UV absorption spectrum, the charge transition band was observed in the maximum wavelength absorptivity at 313 nm and corresponding optical band gap was 2.6 eV,see Fig.1(A) and (B).



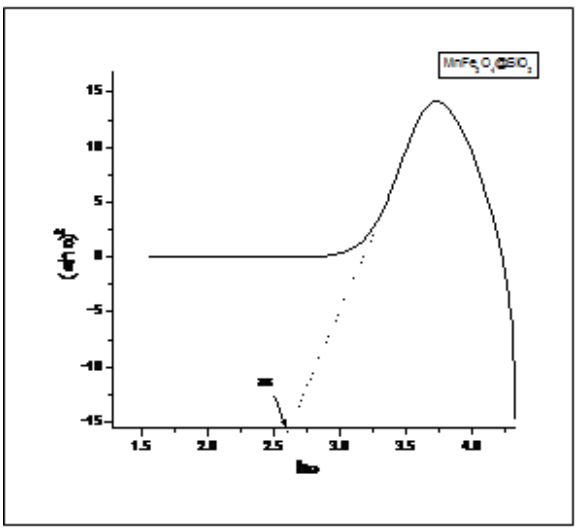


Figure 1:(A) UV spectrum of $MnFe_2O_4@SiO_2$ nanoparticles **(B) Optical band gap of $MnFe_2O_4@SiO_2$ nanoparticles**

3.2 XRD SPECTRA

XRD pattern, (see Figure 2) consist of sharp intense peaks of Silica supported manganese ferrite nanoparticles which confirmed the excellent crystalline nature of peaks, (110) (111) (120) (311) matches with those of spinel

ferrite crystalline structure (JCPDS card no. 10-0319)and particle size was calculated using Debye – Sheerer equation. The XRD technique is widely used for particle size and structural determination of silica supported Manganese ferrite nanoparticles. From the data of XRD pattern the crystalline structure was identified to be FCC and no extra heights of peaks are observed due to any type of impurity. Herein Debye –Sheerer equation is

$$d = \frac{0.9\lambda}{\beta \, Cos \, \theta} (1)$$

Using this equation (1) the particle size of silica supported manganese ferrite nanoparticle was calculated as 45 nm.

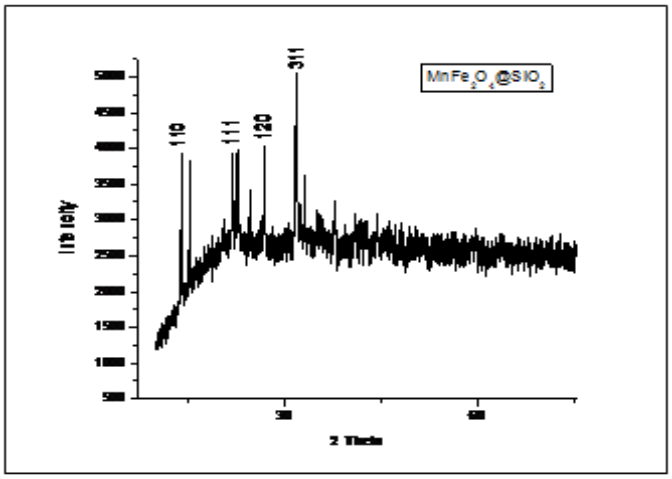


Figure 2: XRD of $MnFe_2O_4@SiO_2$ nanoparticles

3.3 FTIR Spectra

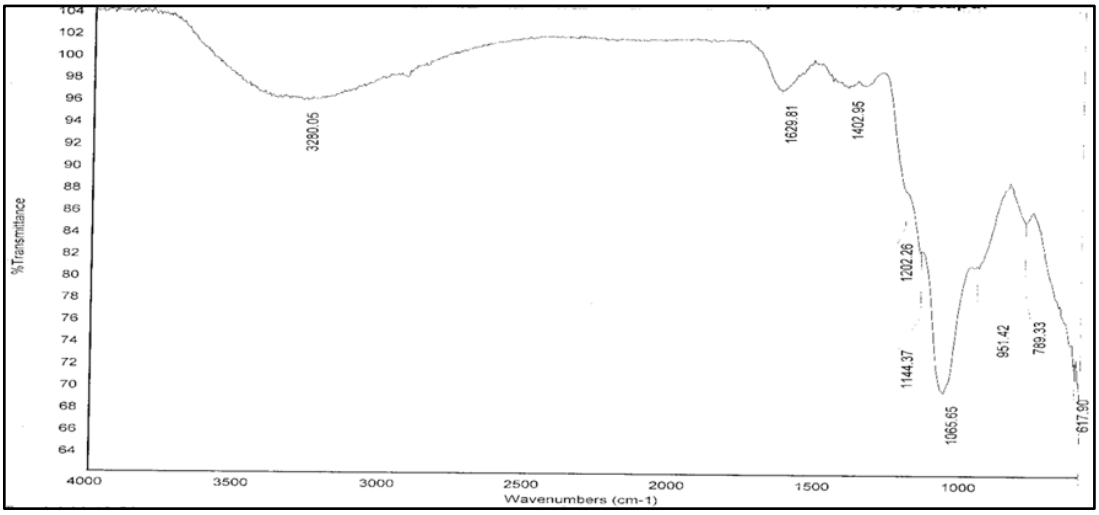


Figure 3: IR spectra of $MnFe_2O_4@SiO_2$ nanoparticles

FTIR spectra of silica supported manganese ferrite nanoparticles (Figure 3). The broad band at 3280.05 cm^{-1} corresponds to O-H group stretching while the band at 1629.81 $cm^{-1}$was attributed to the vibration of O-H group present on the surface of sample. In addition, the absorption band at 1144.34 $cm^{-1}$was the characteristic peak of anti-symmetric stretching vibrational mode of Si-O-Si siloxane bridge. The absorption peak at 951.42cm^{-1} was due to the Si-O-H stretching vibration, while the band at 789.33cm^{-1} was due to SiO_4 ring vibration. The band at 617.90 cm^{-1} corresponds to the Fe-O stretching in the Fe-O-Si bond. FTIR spectrum confirmed the bonding of silica to surface of the manganese ferrite nanoparticles.

3.4 THERMOGRAVIMETRIC ANALYSIS

In this method, loss of mass occurs due to any other functional group via O-H on the surface of the nanomaterial. This mass of nanomaterial was lostat the range of temperature from 30 ℃ –900 ℃. The initially loss of mass occurring from 200℃ upto 800℃ to get stable oxides of silica supported manganese ferrite nanoparticles. Later on these are decomposed above 900℃ (see Fig. 4)

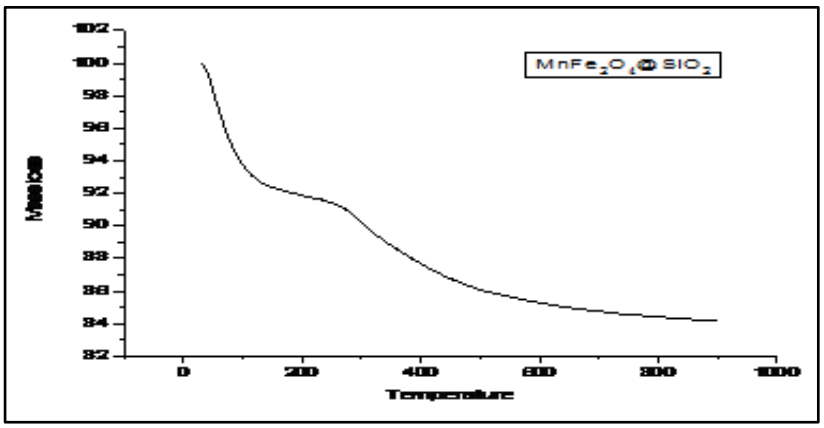


Figure 4: TGA Curve of $MnFe_2O_4$@SiO_2 nanoparticles

3.5 DIFFERENTIAL THERMAL ANALYSIS

This thermoanalytical technique used to measure temperature required for silica supported manganese ferrite nanoparticles. The endothermic peak observed at 55 ℃ temperatures and exothermic peak observed at 305℃temperature.

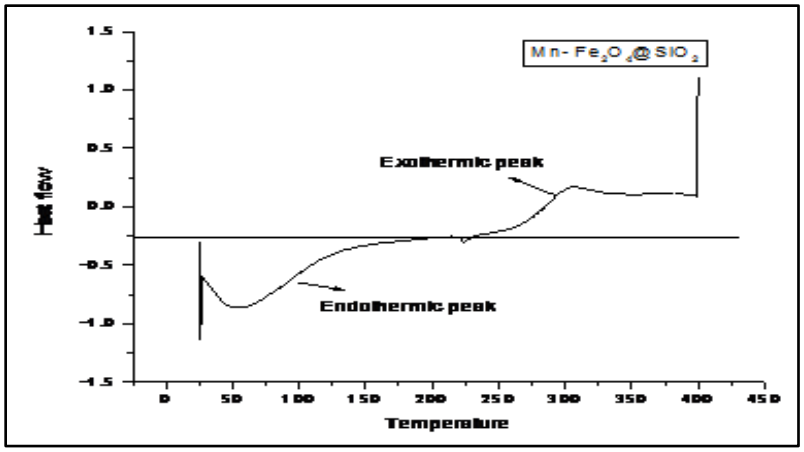


Figure 5: DTA curve of $MnFe_2O_4$@SiO_2 nanoparticles

3.6 TRANSMITTED ELECTRON MICROSCOPY

The TEM image shows the shape, size and distribution of particles of silica supported manganese ferrite nanoparticles which was chemical stabile when sample was calcinated at 300 ℃ within 2 hours. In the observation of the image, the particle size of nanoparticle should be uniformally distributed and their resulting particle size is 42 nm (Figure 6)

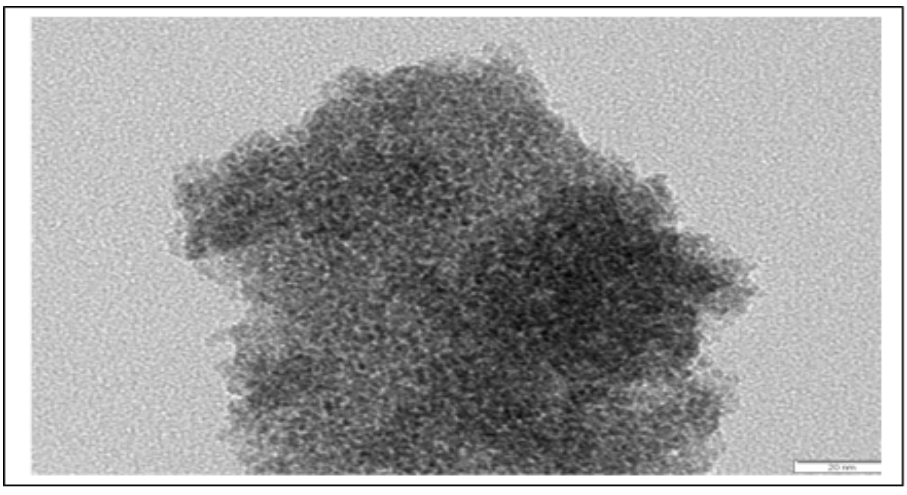


Figure 6: TEM of $MnFe_2O_4$@SiO_2 nanoparticles

4. Conclusion

In summary, we have described the novel method for the synthesis of silica supported manganese spinel ferrite nanoparticles by using atom economical, cost effective and eco-friendly chemical co– precipitation procedure. The synthesized nanoparticles were confirmed by using various analytical techniques such as UV-Visible, XRD, IR, TGA, DSC, TEM etc. The maximum absorption peak was obtained at 313 nm and band gap was 2.6 eV. XRD pattern showed the particle size 45 nm and FCC crystalline structure.IR showed their vibrating stretching of Si-O-Si and SiO_4 groups. In addition when sample was heated at particular range of temperature then there was loss of mass at higher temperature using TGA but the samplewas found to be stable after calcination at 300 ℃ and DTA curve shows the exothermic and endothermic peaks. Finally, TEM image confirms the particle size to be 42 nm which result from the strongmagnetic behavior.

5. Acknowledgement

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6. References

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