Ethylene Glycol Mediated Synthesis of Nano - Crystalline $NiFe_2O_4$ Spinel Powder

S. Vivekanandhan*, M. Venkateswarlu**, N. Satyanarayana*

* Department of Physics, Pondicherry University, Pondicherry- 605 014, India, nallanis2000@yahoo.com
** Department of Chemical Engineering, NTUST, Taipei, Taiwan, vralu@yahoo.com

ABSTRACT

Nano - crystalline nickel ferrite ($NiFe_2O_4$) powders was been synthesized by ethylene glycol mediated process using metal nitrates for Ni and Fe ion sources and ethylene glycol. Synthesis of nano-crystalline $NiFe_2O_4$ powder was investigated through TG/DTA and FTIR analysis. Magnetic property of the synthesized nano crystalline $NiFe_2O_4$ powder was identified through VSM measurement.

Key words: Nano - Crystalline $NiFe_2O_4$, Ethylene Glycol, Polyol, TG/DTA, FTIR, VSM.

1 INTRODUCTION

Nickel ferrite ($NiFe_2O_4$) with spinel structure is well known magnetic material, which exhibits wide range of applications such as high-density magnetic recording, magnetic fluids, sensors, etc [1-3]. In recent years synthesis of ultrafine $NiFe_2O_4$ particles has been investigated because $NiFe_2O_4$ powders in nano size exhibits unusual physical and chemical properties because of their small size or large specific surface area [4-6]. Synthesis process is an important tool for the synthesis of nano crystalline powders with desire properties. Solid state reaction method has been widely used for the synthesis of nickel ferrites, which involves the mixing of metal oxides or carbonates and the phase formation occur in very high temperatures. Oxides powders synthesized by the solid state reaction method exhibits disadvantages such as higher operating temperature, in homogeneity, poor stoichiometry, higher crystallite size etc, which affect its magnetic properties [7, 8]. To overcome the above disadvantages, wide range of wet chemical routs, such as sol-gel, combustion, Pechini, polyol, etc are investigated for the synthesis of nano crystalline multi component oxide powders including $NiFe_2O_4$ powders [9-12].

Among them, ethylene glycol mediated process which is also known as polyol process, is found to be a versatile for the synthesis of metal oxides with controlled morphologies. This process is successfully used by X. Jiang et. al., for the synthesis of TiO_2, ZnO nano structures with different shapes [13]. Hence, in the present work, nano - crystalline spinel $NiFe_2O_4$ powders were prepared by ethylene glycol mediated process. The synthesis process was investigated through FTIR, TG/DTA and VSM analysis.

2 EXPERIMENTAL

2.1 Synthesis of nanocrystalline $NiFe_2O_4$ powders by ethylene glycol mediated route

Schematic diagram of the synthesis of nano crystalline $NiFe_2O_4$ Powders by ethylene glycol mediated route is shown in fig. 1. Required amounts of nickel nitrate (AR grade Merck) and ferric nitrate (AR grade Merck) solutions were mixed with the ethylene glycol (SQ grade Qualigencs). Total metal ions to ethylene glycol was kept as 1:20. The resulting brownish red coloured clear solution was heated at 170°C under constant stirring for 2 hours. The evaporation lead to the formation of black coloured particles (glycolate intermediates) in the solution and it was separated by centrifuging. Collected particles were washed with water for the removal of existing organic impurities and further calcined at 300°C to obtain the nano crystalline $NiFe_2O_4$ powders. The complete process for the synthesis of nanocrystalline $NiFe_2O_4$ powder was monitored and investigated using FTIR, TG/DTA and VSM measurements.

2.2 Experimentla techniques

Thermal behavior of the centrifuged precipitate (glycolate intermediates) was studied by simultaneous thermogravimetic and differential thermal analyzer (TG/ DTA). Approximately 4 mg of glycolate intermediate was heated at the rate of 10°C from room temperature to 600°C in a flowing air ambient and recorded the TG / DTA curves using SETRAM TG-DTA system. The FTIR spectra were recorded using shimadzu FTIR - 8000 spectrometer. All samples were examined between 400 and 4000 $cm^{-1}$and KBr was used as diluter. Magnetic property of the obtained nano crystalline $NiFe_2O_4$ powder was investigated through Lake Shore's vibrating sample magneto meter. 5mg of nanocrystalline $NiFe_2O_4$ powder was taken in the quartz tube and measured the magnetic properties.

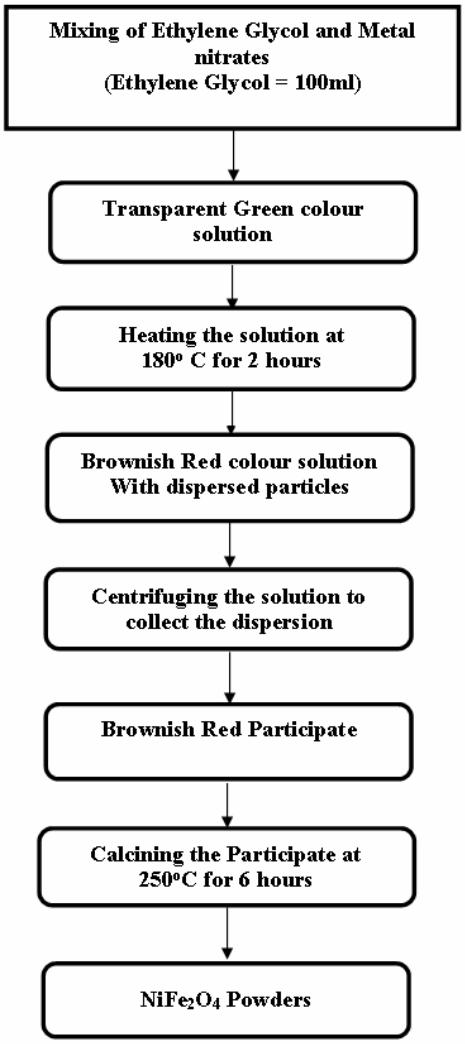

Fig. 1. Schematic diagram of the synthesis of nano crystalline $NiFe_2O_4$ Powder by Polyol method

3 RESULTS AND DISCUSSIONS

Simultaneous TG/DTA thermogram of the intermediate particles (glycolates) is shown in fig 2. From fig 2, the observed exothermic peak between 150°C and 250°C for intermediate particles indicates the decomposition of organic residuals. The respective TGA curve shows the gradual weight loss up to 150°C and the major weight loss occurs between 150°C and 175°C. TG/DTA thermogram clearly shows that the decomposition reaction is ignited at 150°C and completed at 300°C. No more weight loss was observed above 350°C, which indicates the formation of $NiFe_2O_4$ structure, further it was confirmed by FTIR analysis.

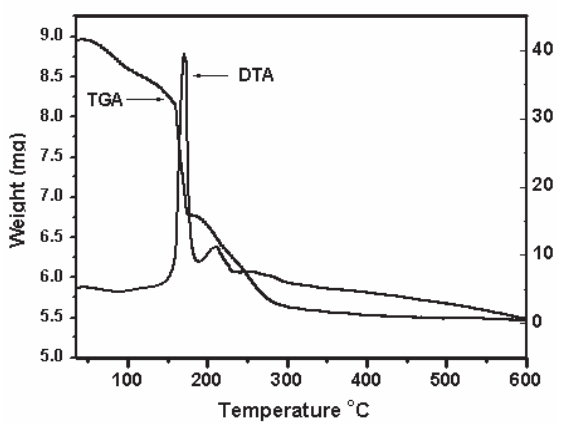

Fig. 2 TG/DTA thermogram of the intermediates

Fig 3 shows the FTIR spectra of the collected intermediate from the ethylene glycol mediated process for the synthesis of nano crystalline $NiFe_2O_4$ powders. From fig. 3, the observed IR peaks for the as collected intermediate exhibits the peak at 3392cm^{-1} and it is assigned to the vibrations of OH groups in the glycolate derivatives [14]. The FTIR peaks observed at 2293 and 2995cm^{-1} are attributed to the symmetric and asymmetric vibrations of $-CH_2-$ groups. The strong peak observed at 1650cm^{-1} and 1180cm^{-1} confirm the formation of the metal carbolylates [15]. The low intense peak observed at 1255cm^{-1} indicates the formation of small quantity of ether. The organic derivatives were disappeared when the intermediates was heated at 300°C and above. The observed new peak at 604 cm^{-1} is due to the presence of Fe_2O_4 groups, which confirms the formation of $NiFe_2O_4$ structure [14].

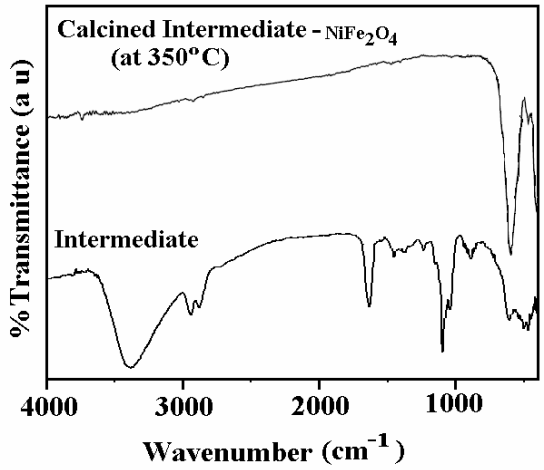

Fig. 3 FTIR spectra of as obtained as well as calcined intermediate

Fig.4 shows the hysteresis loop of $NiFe_2O_4$ sample calcined at 300°C for 12 hours. The observed hysteresis loop indicates the superparamagnetic property of the calcined powder. The observed superparamagnetic property of the synthesized $NiFe_2O_4$ particles confirmed the formation of nano crystalline phase. Also, SEM and TEM analysis are under progress.

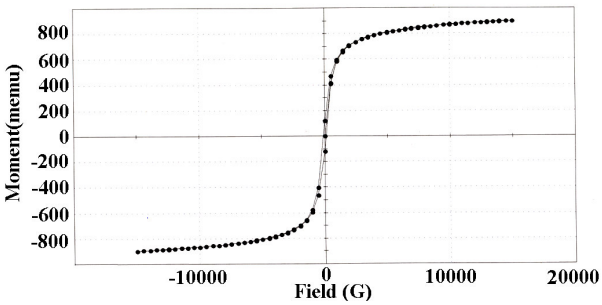

Fig. 4. Hysteresis loop of nanocrystalline $NiFe_2O_4$ powders calcined at 300°C for 6 hours

4 CONCLUSIONS

Nano crystalline $NiFe_2O_4$ powder was successfully synthesized by ethylene glycol mediated polyol route relatively at lower temperature at 300°C. FTIR anaysis confirmed the formation of $NiFe_2O_4$ structure. Also, the VSM measurement of synthesized $NiFe_2O_4$ powder exhibits the superparamagnetic property, which confines the formation nano structured $NiFe_2O_4$.

5 ACKNOWLEDGEMENTS

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