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Investigation on Combustion Synthesis of Nanocyrstalline Nickel Ferrite Using Sodium Azide as a Potential Fuel

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Abstract: The present investigation reports the synthesis of highly crystalline single phase nickel ferrite ($NiFe_2O_4$) nanoparticles by combustion route using Ni and Fe nitrate precursors with sodium azide as a potential fuel. Sodium azide becomes valuable fuel because of the highly exothermic character of the decomposition reaction and the low moleculecular weight of the decomposition gas. The physico-chemical and morphological features of the synthesized powders have been assessed using different analytical tools. X-ray diffraction (XRD) study reveals the formation of high degree of crystalline powders of nickel ferrite. The EDAX analysis indicates the correct elemental composition of the ferrite powder. FT-IR spectroscopy shows the stretching and bending vibrations of Ni-O and Fe-O bonds of nickel ferrite. The morphological features of the powder examined by SEM reveals the fine crystalline nature of the powder. The magnetic property of the synthesized poweder is studied with help of VSM.
Keywords- Nickel Ferrite ($NiFe_2O_4$), Combustion synthesis, Sodium azide, Nanocrystalline, XRD, SEM, FTIR and VSM.

1. Introduction

Nanomaterials have gained remarkable scientific interest owing to their interesting and unusual physical and chemical properties that are significantly different from those conventional bulk materials due to their extremely small size or large specific surface area [1] and helping them find a wide variety of applications. In recent years, there is a growing interest in magnetic ferrite nanoparticles because of their applications in permanent magnets, magnetic drug delivery, microwave devices and high density information storage technology[2]. Among the different ferrite materials, spinel ferrites exhibit low eddy current losses in alternating current applications and are particularly useful in the radio frequency range. Hence, they have numerous applications in recording heads, core materials for various transformers, inductors and TV deflection units, and in recording tape [3]. Nickel Ferrite ($NiFe_2O_4$) is one of the most important spinel ferrite which finds applications in the fabrication of soft magnets and low loss materials at high frequencies [4]. It is known that the fascinating electrical and magnetic properties of ferrites depend upon the nature of the ions, charges and their distribution among tetrahedral (A) and octahedral (B) sites [5]. $NiFe_2O_4$ is a typical soft ferromagnetic material crystallizing in a completely inverse spinel structure with all nickel ions located in the B-sites and ferric ions occupying both A-sites and B-sites [6]. The compound, thus can be represented by the formula $(Fe^{3+})_A [Ni^{2+}Fe^{3+}]_B O_4^{2-}$ [7]. The wide and varied applications of nano sized ferrites has led to the development of various synthesis methods, including solid-state [8], co-precipitation [9], sol-gel [10], sonochemical [11], hydrothermal [12] microwave [13], pulsed wire discharge [14], sol-gel auto-combustion [15], combustion

method [16] etc. Among these methods, combustion synthesis is a versatile and promising technique for the preparation of soft ferrites, leading to highly pure, chemically homogeneous, nano scale particles [17]. This process is quite simple and involves an exothermic and self-sustaining chemical reaction between the metal salts and suitable organic fuels like urea [18], glycine [19], citric acid [20], hexamine [21] and DL-alanine [22]. To the best of our knowledge, no attempt has been made to synthesize nickel ferrite using sodium azide as a fuel. The present investigation deals with the synthesis of nanosized single phase nickel ferrite by combustion method using sodium azide as a fuel. XRD, SEM, EDAX , FTIR and VSM were used to characterize the synthesized powders and the results are reported.

2. Experimental

2.1. Synthesis

The analytical grade nickel nitrate ($Ni(NO_3)_2.6H_2O$), ferric nitrate ($Fe(NO_3)_3.9H_2O$) and sodium azide (NaN_3) were used as raw materials. Stoichiometric quantities of nitrate salts and sodium azide were dissolved in de-ionized water to form a solution. The mixed solution was then poured into a quartz bowl and stirred for two hours to obtain a homogeneous gel. The obtained gel was placed in a hot plate and heated to a temperature of 300^oC to initiate a self propagating exothermic reaction. The gel swelled and ignited with an evolution of large amounts of gaseous products and the desired ferrite resulted in the form of a foamy powder. The as-combusted porous brown powder was finely grounded and calcined at 800^oC for 8 hour in air. The flow chart of the synthesizing procedure is illustrated in Fig.1.

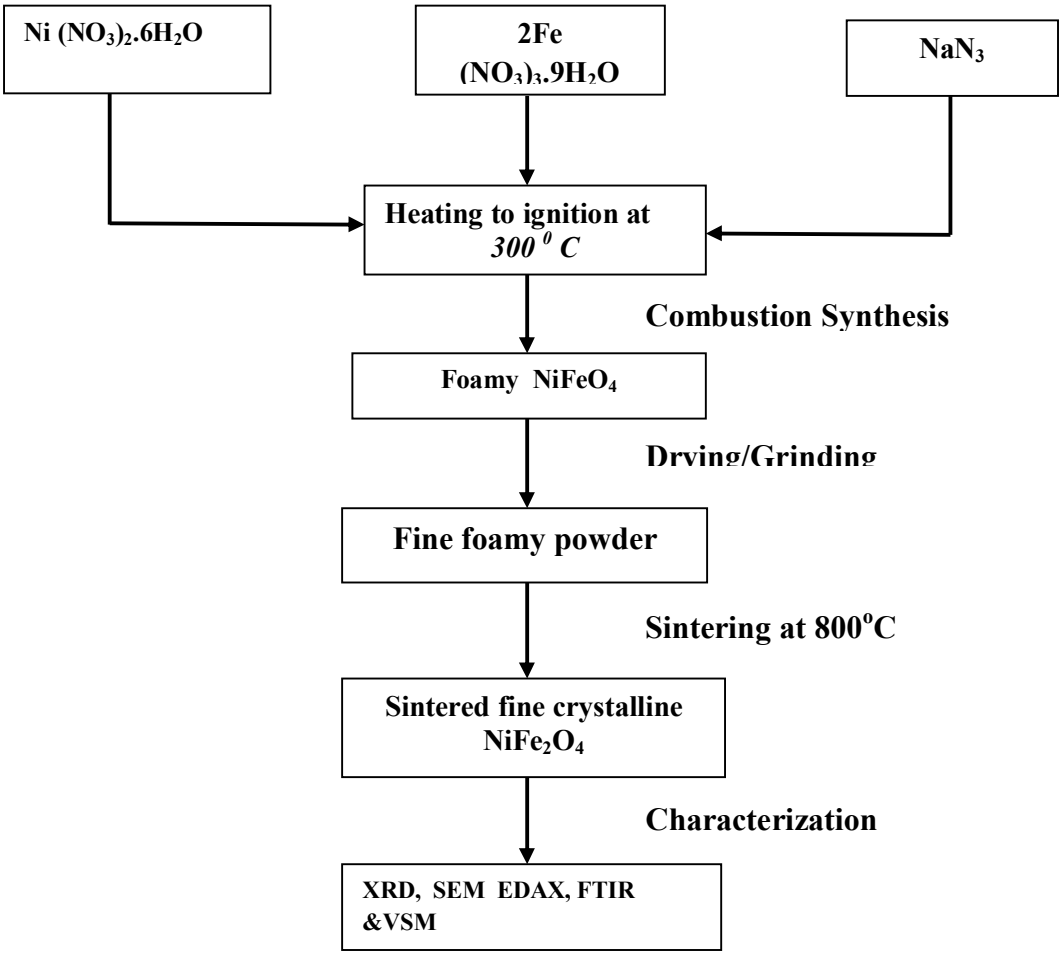

Fig.1. Flow chart of the experimental procedure

2.2. Characterization

Crystalline structure, phase composition and crystallite size of the synthesized powder was identified from XRD data using Cu-Kα radiation (λ=1.541 A^0) with 2θ values ranging from 10^0 to 80^0 by JEOL 8030 X-

ray diffractometer. The FTIR spectra of the sample was recorded in KBr pellets in the wavelength range 400-4000 cm^{-1} using Paragon-500, Perkin Elmer FTIR spectrophotometer. The elemental composition of the ferrite powder was determined using EDAX spectrum. Scanning Electron Microscopy (SEM) images were recorded using a JEOL (JSM-3.5 CF) instrument.

3. Results And Discussion

3.1. Structural analysis

XRD pattern (Fig. 2) of $NiFe_2O_4$ samples obtained by the combustion process exhibiting reflections with *hkl* values of (220), (311), (222), (400), (422), (511) and (440) planes, indicate the cubic spinel structure of nickel ferrite [23]. All the peaks matched with the standard characteristics peaks of the cubic spinel lattice of $NiFe_2O_4$ (JCPDS Card No.10-0325). Also, no secondary peaks were detected in the XRD pattern which ensures the phase purity of the sample.

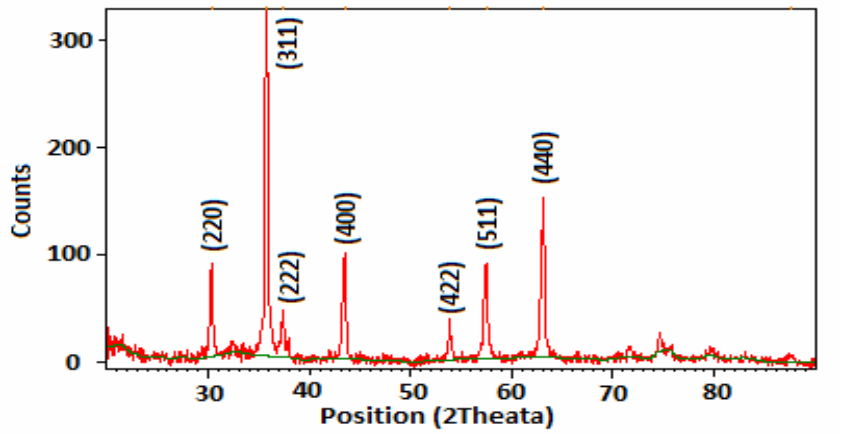

Fig. 2. XRD pattern of $NiFe_2O_4$ nanoparticles powder

The average crystallite size (d) of $NiFe_2O_4$ was calculated from main characteristic peak (311) at full-width at half maximum (FWHM) using the Scherrer's relation [24] given in equation (1)

$$d = \frac{0.9\lambda}{\beta \cos\theta} \qquad (1)$$

where, β is the broadening of diffraction line measured at half maximum intensity (radians) and λ=1.541A^0, the wavelength of Cu-Kα radiation. The average crystallite size of nickel ferrite synthesized using sodium azide as a fuel was found to be 25.303nm. The lattice constant of $NiFe_2O_4$ nanoparticles was determined using the following equation (2) [25].

$$a = d_{hkl}\sqrt{\left(h^2 + k^2 + l^2\right)} \qquad (2)$$

The lattice parameter of the sample calculated to be a=b=c=0.8323236 nm was found to be in good agreement with the earlier reported values of 0.833nm for nano $NiFe_2O_4$ [26] and 0.8339nm for the bulk $NiFe_2O_4$ [8].

3.2. Morphological and compositional analysis

The SEM micrographs with different magnifications are shown in Fig 3 (a) & (b). The SEM picture shows that the synthesized particles have a fine crystalline nature. The agglomeration observed could be mainly due to the magnetic dipole interaction among the particles.

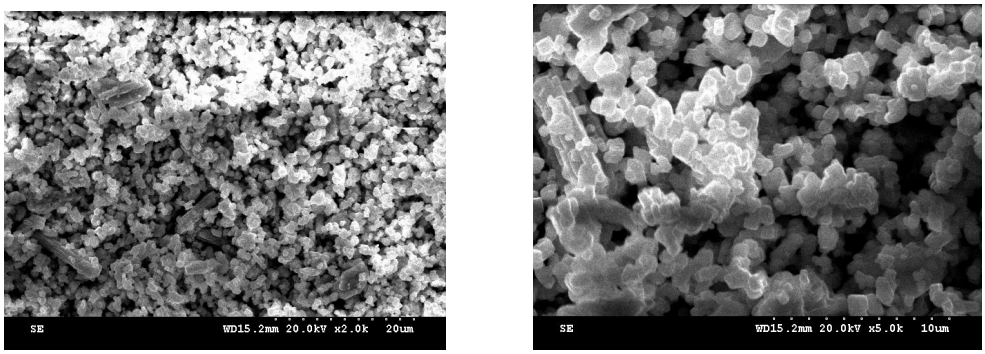

Fig.3 (a) & (b) SEM micrograph of $NiFe_2O_4$

The EDAX spectrum (Fig.4) and the composition data (Table 1) show that the sample contains only Ni, Fe and O elements.

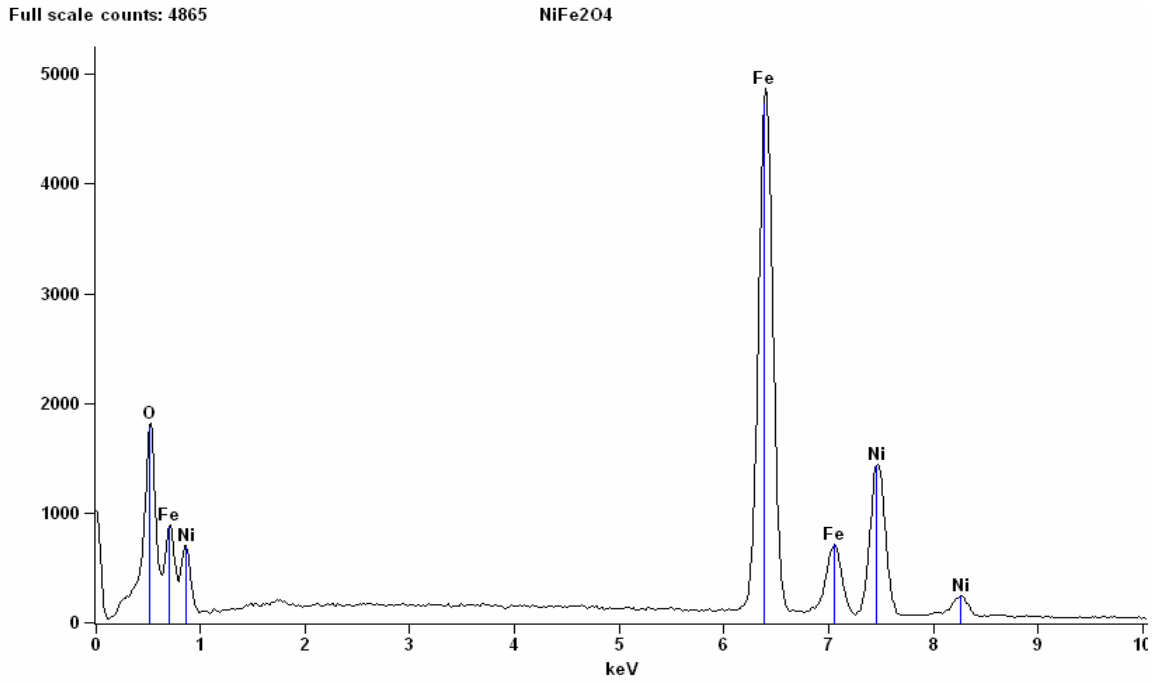

Fig.4. EDAX spectrum of $NiFe_2O_4$.

Table.1. EDAX analytical data of $NiFe_2O_4$

Element	Net Counts	Weight %	Atom %
O	15069	16.23	40.72
Fe	85423	57.12	41.06
Ni	26531	26.65	18.22
Total		100.00	100.00

3.3. FT-IR Analysis

The FT-IR spectrum (Fig. 5) for the nickel ferrite compound was recorded in the wavelength range of 400-4000cm^{-1}. A strong absorption band around the wavenumber 576.54 cm^{-1}, confirms the presence of spinel $NiFe_2O_4$ as reported in literature [27]. The weak and broad absorption band noticed around 1629.29 cm^{-1} is analogous to the presence of minor concentration of residual carbon in the spinel compound [28]. It has been reported that the IR bands of solids are usually assigned to vibration of cations in the crystal lattice [29].

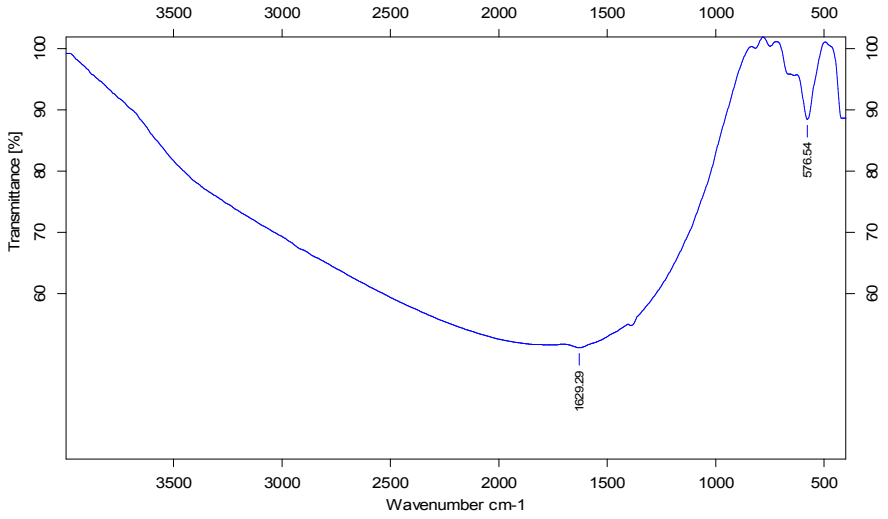

Fig. 5. FT-IR spectrum of $NiFe_2O_4$

3.4. VSM Analysis

Magnetic characterozation of the as-prepared $NiFe_2O_4$ nanopartices was carried out at room temperature using Vibration Sample Magnetometer (VSM) and the hystresis loop of the sample is shown in Fig. 6.

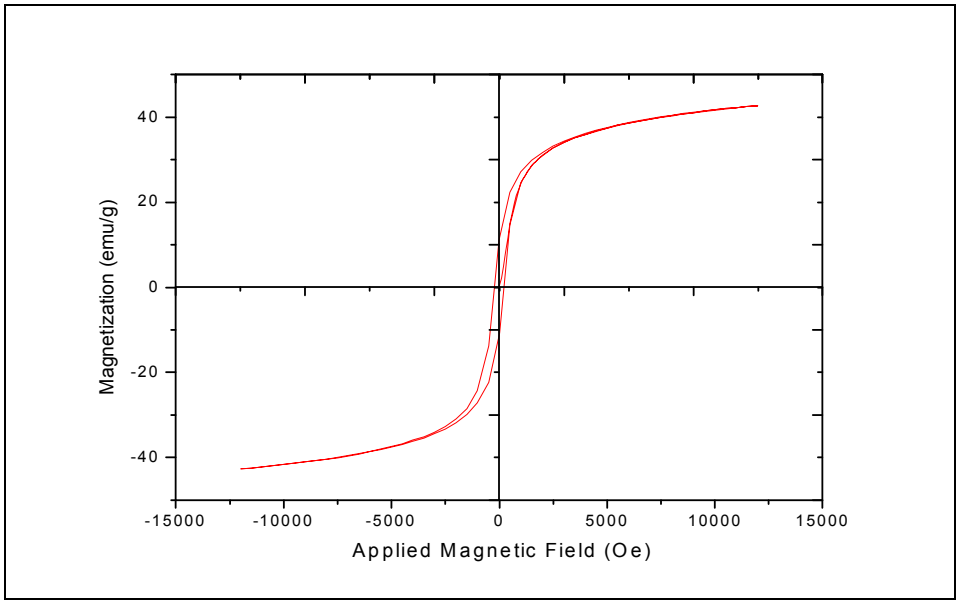

Fig 6. Magnetic hysteresis curve of as-prepared $NiFe_2O_4$ nanoparticles

From the figure, it is observed that the variation of magnetization as a function of applied field shows a narrow cycle and the hystereis loop characterises the behaviour of soft magnetic materials. The saturation magnetization of nickel ferrite is 42.73 emu/g which is higher than that are reported earlier (22,30,31) and the value nearly approached the bulk values of 56emu/g [32]. The lower value of the saturation magnetization of the nanoparticles is due to the combined effect of surface spin disorder, formation of spin glass structure and magneto crystalline anistoropy

4. Conclusion

To summarize, $NiFe_2O_4$ nanparticles have been successfully synthesized by combustion route using Ni and Fe nitrates and sodium azide as a fuel. To the best of our knowledge no report was observed where sodium

azide has been used as a fuel for synthesizing the nanocrystalline $NiFe_2O_4$. The approach was found to be simple and efficient to prepare nanoparticles. The XRD pattern reveals the formation of highly crystalline nanosized $NiFe_2O_4$ powders. The formation of spinel $NiFe_2O_4$ was ascertained by FT-IR spectrum. EDAX analysis confirms the elemental composition which is in appropriate proportion in the synthesized compound. The SEM micrographs exhibit the fine crystalline nature of the powders. The VSM measurement at room temperature shows that the synthesized powder possessed good magnetic properties with high saturation magnetization close to bulk $NiFe_2O_4$. The synthesized $NiFe_2O_4$ can be used in several applications such as low loss materials at high frequencies and magnetic fluids. Thus, sodium azide is found to be one of the potential fuels for the synthesis of nanostructured ferrite materials.

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