

Microwave synthesis of nickel ferrite: Structure, morphology and bonding

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ABSTRACT

Microwave synthesis of ferrite materials integrates the synthetic technology for information storage materials. Microwave route is adopted for the synthesis of nanosized NiFe_2O_4 using urea as a fuel. Nickel oxalates and iron oxalates were prepared by dissolving nickel and iron salt in oxalic acid solution. These two oxalates are irradiated with microwaves using urea as a fuel to get cubic NiFe_2O_4 particles. The structure of the prepared sample is studied by X-ray diffraction (XRD) which shows crystalline cubic structure, Morphology of the sample is viewed by Scanning electron micrograph (SEM) which shows spherical particles with compact and globular arrangement. Metal-Oxygen and metal-metal bonding is studied by infrared (IR) technique.

Key words: Microwave synthesis, Morphology, NiFe_2O_4 , Oxalate, bonding.

INTRODUCTION

Modern trend in materials science and related fields is the fabrication, characterization and applications of materials at scale particles.¹In recent years several kind of metal oxide particles finding properties and applications in several fields. Metal oxides are of interest to many scientific and technological disciplines. These materials have attracted considerable interest as they exhibit materials properties that differ strongly from those of the bulk phases. Particle size effect enable to tailoring the materials to a wide range of applications including magnetic ferrofluids, electronics, etc. Iron oxides are class of ferrite materials which shows wide applications improves the technology of ferrite materials².

Synthesis of ferrite materials creates the road map for synthetic technology. Microwave synthesis of nickel ferrite has gained great

importance in the recent years because of easy scale up³⁻⁴. Further microwave method is also very clean and non polluting. In order to use the microwave route, it is desirable that at least one of the reactant should be overcome by the use of a secondary suseptor, which is chemically inert with respect to the reactants of interest. Several microwave reactions are now known to occur at lower temperature than the in conventional methods⁵⁻⁶.

Present investigation is a microwave synthesis of cubic NiFe_2O_4 using metal oxalate precursors employing microwave route. Urea is used as a fuel for complete conversion of iron and nickel oxalate in to nickel ferrite particles and possessing interesting morphology. The structure of as prepared nickel ferrite sample was undertaken by employing X-ray diffraction (XRD), Morphology by Scanning Electron Micrograph (SEM) and bonding by Fourier transform infrared (FTIR) techniques.

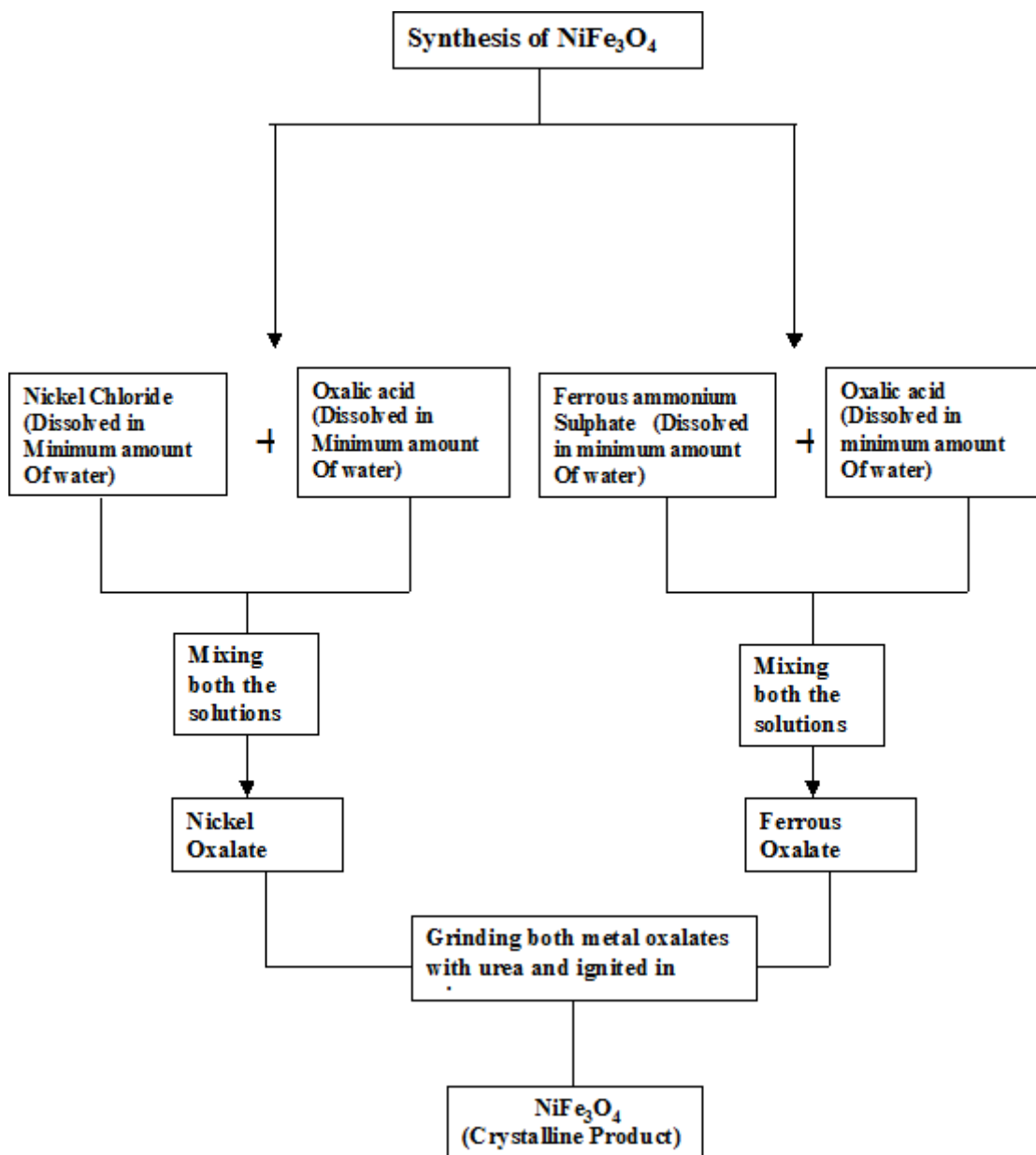
EXPERIMENTAL

Materials and Methods

Nickel Chloride, ferrous ammonium sulphate, oxalic acid and urea used were AR grade. Microwave method is used for the synthesis of NiFe_2O_4 materials. Urea is used as a fuel for the combustion reaction.

Synthesis of NiFe_2O_4

The synthetic step for nickel ferrite initiates by preparation of nickel oxalate and iron oxalate precursors. These precursors were prepared by dissolving equimolar quantities of nickel chloride and ferrous ammonium sulphate with oxalic acid and was stirred well in separate beakers. The precipitates of nickel and iron oxalate obtained were



Schme 1: Synthesis of Nickel Ferrite

filtered through sintered glass crucible and was washed with oxygen-free distilled water till free from chloride ions and oxalic acid, finally with dry acetone and was then dried under vacuum.

The nickel oxalate, iron oxalate and urea were mixed in weight ratio 1:1:5 and ground well in a pestle and mortar. Resultant solid was placed in a crucible and ignited in microwave oven ⁷. The reaction was found to be completed in about ten minutes at high power level and forms a brown crystalline NiFe_2O_4 material is formed. On cooling to room temperature no trace of carbon impurities was observed in the final residue of NiFe_2O_4 . Followings are the reactions taking place in the synthetic process and synthetic scheme is given in the scheme-1

1. $(\text{COOH})_2 + 2\text{NiCl}_2 \rightarrow (\text{COONi})_2 + 2\text{HCl}$
2. $(\text{COOH})_2 + 2\text{Fe}(\text{NH}_4)_2\text{SO}_4 \rightarrow (\text{COOFe})_2 + 2\text{NH}_3 + 2\text{H}_2\text{SO}_4$
3. $(\text{COONi})_2 + (\text{COOFe})_2 \rightarrow 2\text{NiFe}_2\text{O}_4 + 4\text{CO}_2$

Characterization

The powder X-ray diffraction pattern was obtained from GEOL JDX-8P or SEIMEN (Japan) X-ray diffractometer using CuK_α radiation. The

morphology of the maghemite sample was obtained from Leica Cambridge-440 scanning electron microscopy. Bonding in NiFe_2O_4 was obtained from Perkin-Elmer FTIR spectrophotometer (1000).

RESULTS AND DISCUSSION

X-ray diffraction

Fig. 1. shows XRD pattern of as prepared nickel ferrite and the indexed XRD data of the as synthesized NiFe_2O_4 sample is given in Table-1.

Table 1: Indexed XRD data of NiFe_2O_4 sample

hkl	$2\theta_{\text{obs}}$	$2\theta_{\text{lit}}$	I_{obs}
111	18.421	18.392	18
220	30.280	30.293	32
311	35.670	35.699	100
400	43.330	43.362	28
511	57.355	57.357	35
440	62.986	62.916	45
731	90.421	90.385	18

$2\theta_{\text{obs}}$ = Observed 2θ values, $2\theta_{\text{lit}}$ = literature 2θ values
 I_{obs} = Observed intensity

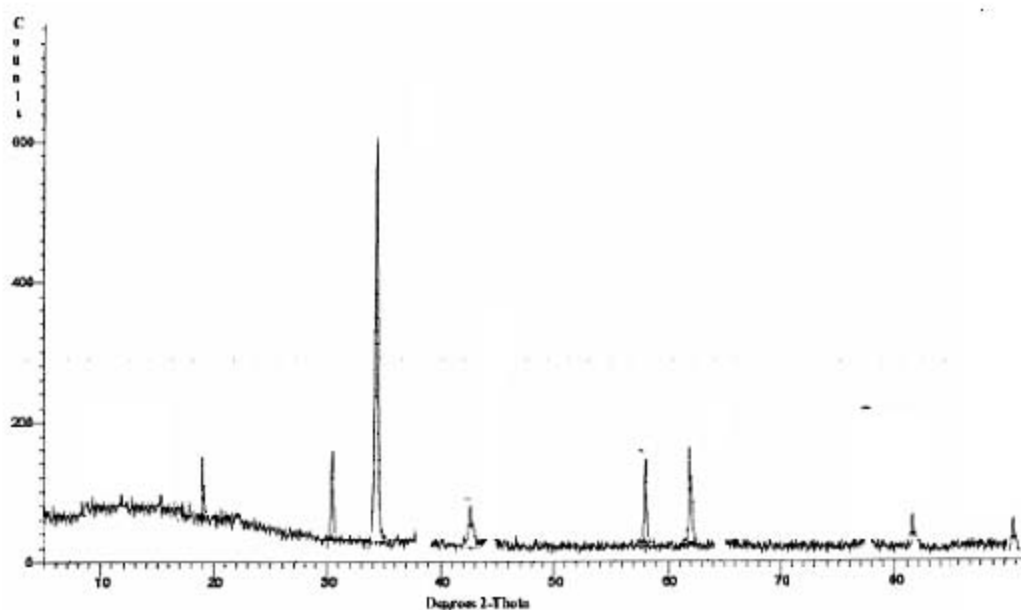


Fig. 1: XRD pattern of as prepared NiFe_2O_4

The table shows the comparison of diffraction angles of prepared sample and literature diffraction angles. 2θ of prepared nickel ferrite sample matches well with 2θ values reported in the literature (JCPDS-10-0325). Unit cell parameters were obtained by least square refinement of the powder XRD data. The XRD study reveals that, the prepared nickel ferrite is monophasic with cubic spinel structure.

Scanning electron micrograph

Fig. 2(a-b) shows SEM image of as prepared NiFe_2O_4 sample at low and high resolution respectively. The particles are spherical in shape with compact arrangement. Some particles are found as agglomerates. Flakes of agglomerates of NiFe_2O_4 particles are also observed. The particles of NiFe_2O_4 form self assembled irregular shaped blocks with globular arrangement.

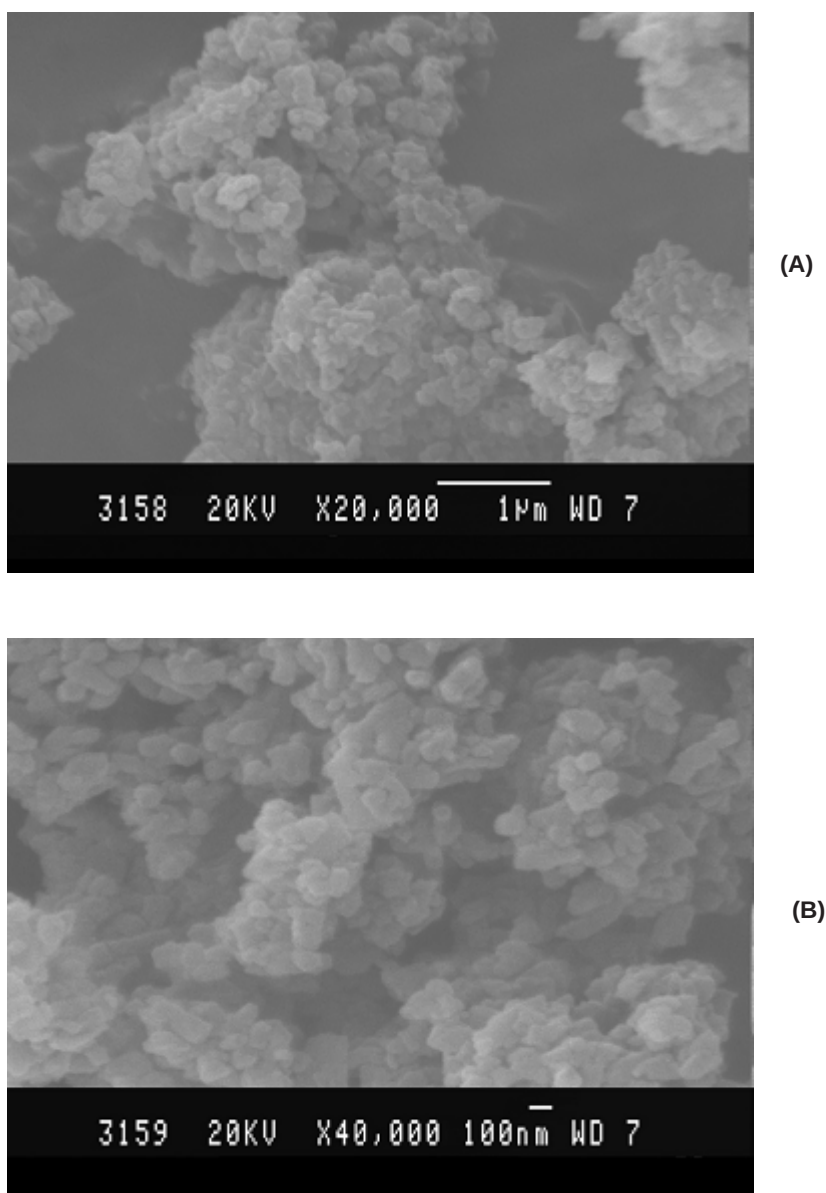


Fig. 2: (a-b): SEM image of as synthesized $\text{NiFe}_{23}\text{O}_4$ at (a) low resolution (b) high resolution

Infrared

The infrared study was performed aiming to ascertain the metal-oxygen and metal-metal bond in the prepared product. Table-2 gives the vibrational frequencies of as synthesized NiFe_2O_4 . The NiFe_2O_4 sample shows the absorption in the region 3050, 1065, 510, 465, 435, 420, 260 and 220 cm^{-1} . The peak at 3050 cm^{-1} corresponds to water of adsorption and the peak at 1065 cm^{-1} is due to the presence of some overtones. The peaks at 510, 465, 435 and 420 cm^{-1} correspond to the metal-oxygen vibrational modes of the spinel compound⁸. The peaks at around 260 and 220 cm^{-1} is observed is due to metal-metal (Ni-Fe) vibration frequency range. This confirms the formation of nickel ferrite materials.

Table 2: Vibrational Frequencies of NiFe_2O_4 sample

Peak No	Vibrational Frequencies(cm^{-1})
1	3050,
2	1065
3	510
4	465
5	435
6	420
7	260
8	220

CONCLUSION

1. Microwave route for NiFe_2O_4 is very simple, energy efficient technique and easy to scale up.
2. The synthetic route may be applied for the synthesis of other layered ceramic materials.
3. Urea is used as a fuel for the reaction and this fuel can be used for synthesis of other oxide materials.

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