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COMBUSTION SYNTHESIS OF NICKEL FERRITE

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ABSTRACT

New synthesis route using metal carboxylates precursor method was adopted for the synthesis of $NiFe_2O_3$ is reported. X-ray density, Tap density and powder densities of as prepared $NiFe_2O_3$ are calculated. The as synthesized NiO, Fe_2O_3 and $NiFe_2O_3$ are characterized by X-ray diffraction (XRD) technique. Infrared spectroscopic (IR) technique was used to know the formation of M-O and M-M bonds in prepared oxide materials.

Keywords: $NiFe_2O_3$, XRD and IR density.

INTRODUCTION

Synthesis of nanosized materials through a Self-propagation route employing a carboxylate precursor. This synthetic route can be considered interesting for its simplicity, reproducibility and easy scale up. It produces a homogeneous precursor with a controlled stoichiometry and also does not require expensive chemicals. It is a low energy reaction and can be carried out in a china dish in an open atmosphere. In this self-propagation combustion reaction, a suitable fuel for our study was found to be Poly (ethylene glycol).

In recent developments, techniques such as Sol-Gel and other soft chemicals (Chimie douce)[1,2] method have led to the preparation of engineered and advanced materials.

Use of microwave for the synthesis of inorganic compounds has gained great importance in recent years[3-4]. The method offers several advantages over the conventional routes, the most important of them being the very short time and energy economy. Several microwave reactions are now known to occur at lower temperature than in conventional methods. The rapidity of the reactions offers excellent conditions for retention of metastable phases. This novel method has been found to result in better reaction yields and better structural uniformity of products than conventional ceramic methods.

Metal oxide materials prepared by Sol-Gel chemistry are high surface area and high porosity materials are attractive in applications such as insulators and catalytic supports. The versatility of sol-gel chemistry provides a mean of controlling the shape. Morphology and textual properties of the final materials[5-6]. Sol-gel chemistry also provides a means of preparing mixing oxide phases can be controlled on both the molecular and nanoscale[7].

In the present investigation we report the self-propagating combustion synthetic route using a new precursor (Nickel oxalate and ferrous oxalate) for the synthesis of nanosized $NiFe_2O_3$. Poly (ethylene glycol) is used as a fuel for the conversion of metal salts to its respective oxides. Various methods are adopted to calculate density of $NiFe_2O_3$.

EXPERIMENTAL

Material and Methods

All the chemical used were AR grade. Poly (ethylene glycol) of molecular weight 4000 was obtained commercially. Cold oxygen free distilled water is used in the present work.

Self-propagating combustion synthetic route was adopted for the synthesis of $NiFe_2O_3$ particles. The polyethylene glycol is used as a fuel for the combustion reaction.

Preparation of metal oxalate precursor (Nickel oxalate & ferrous oxalate)

This metal oxalate precursors is prepared by dissolving equimolar quantity of metal salts (Nickel chloride and ferrous ammonium sulphate) and oxalic acid was stirred well. The precipitate of metal oxalates obtained was filtered through sintered glass crucible and was washed with oxygen-free distilled water till free from chloride ion, sulphate ions and oxalic acid, finally with dry acetone and was then dried under vacuum.

Synthesis of NiO, Fe_2O_3 and $NiFe_2O_3$

The metal oxalates was mixed with poly (ethylene glycol) in the weight ratio 1:5[8] and ground well in a separate pestle and morar. Resultant solid was placed in a crucible and heated in air. It was observed that initially poly (ethylene glycol) melted, then frothed and finally ignites to form NiO and Fe_2O_3 from respective metal oxalates. On cooling to room temperature no trace of carbon impurities was observed in the final residue of NiO and Fe_2O_3. These oxides were mixed together and heated at 1000°C of about four hours. the resultant crystalline powder of $NiFe_2O_3$ is formed in the crucible.

Density measurement

Density evaluation from X-ray data

The X-ray density of the samples have been computed from the values of lattice parameters using the formula[9]

$$d: = 8 \frac{M}{N a^3}$$

where 8 represents the number of molecules in a unit cell of a spinal lattice

M = Molecular weight of the sample

N = Avagardo's number

a = Lattice parameter of the sample

The lattice constant for the cubic was calculated using the equation

$$d = \frac{a}{(h^2 + k^2 + l^2)^{1/2}}$$

Tap density

The as prepared oxides samples crushed in agate mortar using a pestle and mortar. A known amount of this powder was filled into a graduated cylinder of 25ml capacity. The cylinder was tapped until the powder level remains unchanged. The volume occupied by the powder was noted. The ratio between the weight of the substance and the volume gave tap density[10].

Powder density

The powder densitites were measured using Archimedes principle[11] with a pycnometer and xylene as a liquid medium. The pycnometer of volume 25 ml was used. The following weights were taken and used in the density calculation.

Weight of the bottle = W_{1g}
Weight of the bottle + Substance = W_{2g}
Weight of the bottle + Substance + Xylene = W_{3g}
Weight of the bottle + Xylene = W_{4g}
Density of Xylene = ρ_{sol}
Density of sample = ρ_{sample}

$$\rho_{sample} = \frac{(w_2 - w_1)\,\rho_{sol}}{(w_4 - w_3) + (w_2 - w_1)}$$

Characterization

The X-ray diffraction patterns were obtained employing a Geol JDX-8p spectrometer using CuKα radiation. The X-rays generator was operated at 30kV and 20mA. The scanning range, $2\theta/\theta$ was selected. The scanning speed = 1° min^{-1} were employed for precise lattice parameter determination. High purity silicon powder was used as an internal standard. The shape, size and distribution of the powder, as prepared metal oxide samples, microstructure of the sample have examined using a Leica-440 Cambridge Stereoscan, scanning electron microscope image.

The infrared spectra of metal oxides were recorded on a Perkin-Elmer FTIR spectrophotometer (Model 1000) in the range 300 cm^{-1} to 4000 cm^{-1}.

RESULTS AND DISCUSSION

X-ray diffraction study

Table - 1 shows the observed 2θ values of as prepared NiO, Fe_2O_3 and $NiFe_2O_3$ as well as literature 2θ values of respective metal oxides. The observed 2θ values of metal oxides are matchs well with the literature 2θ values of respective metal oxides. It confirms the formation metal oxides.

Table -1 : Comparison of 2θ values of as prepared samples with Standard JCPDS files

S. No.	2θ values					
	NiO		Fe_2O_3		$NiFe_2O_3$	
	$2\theta_{obs}$	$2\theta_{lit}$ JCPDS (47-1049)	$2\theta_{obs}$	$2\theta_{lit}$ JCPDS (39-1346)	$2\theta_{obs}$	$2\theta_{lit}$ JCPDS (10-0325)
1.	37.32	37.24	14.98	14.96	18.40	18.39
2.	42.96	43.27	18.40	18.39	30.30	30.29
3.	62.12	62.87	23.88	23.79	38.33	37.31
4.	75.42	75.41	30.33	30.26	48.33	47.36
5.	79.88	79.40	32.00	32.15	57.44	57.35
6.	95.22	95.05	33.79	33.91	62.92	62.91
7.	106.22	106.98	35.44	35.65	74.66	74.63
8.	112.20	111.11	43.96	43.32	79.60	79.58
9.	-	-	53.44	53.77	87.45	87.44
10.	-	-	58.00	57.32	90.40	90.38
11.	-	-	63.96	62.98	106.67	106.23
12.	-	-	-	-	114.66	114.63

$2\theta_{obs}$ = Observed 2θ values, $2\theta_{lit}$ = Literature 2θ values

These oxides reveals a good crystalline cubic [NiO(fm3m), Fe_2O_3($p_4$32), $NiFe_2O_3$ (fd3m)] structure.

IR studies

Infrared technique is used to know the metal-oxygen bond in prepared metal oxides and metal-metal bond in $NiFe_2O_3$. Table -2 shows the vibrational frequencies of as synthesized NiO, Fe_2O_3 and $NiFe_2O_3$ respectively. In the Table the characteristic lines below 1000 cm^{-1}, which shows the metal-oxygen vibrational frequency range[12]. This shows the formation of NiO, Fe_2O_3 and $NiFe_2O_3$ particles. The peak at around 220 cm^{-1} is observed in the $NiFe_2O_3$ spectrum, which shows the metal-metal (Ni-Fe) vibration frequency range. This confirms the formation of nickel ferrite.

Density measurements

Density evaluation from X-ray data, Tap density and Powder density of as prepared nickel ferrite is calculated and are given in Table -3. The density values evaluated from different methods are approximately same.

SEM Studies

The particle morphology of nickel ferrite was studied by using scanning electron micrograph images. Fig. -1 shows the particle morphology of as prepared $NiFe_2O_3$ sample. The particles are mostly irregular shape with a nano meter range.

Table -2 : Various vibrational bands of as prepared metal oxide samples

S. No.	Sample	Frequencies of samples
1.	NiO	550, 478
2.	Fe_2O_3	555, 465, 460
3.	$NiFe_2O_3$	650, 560, 550, 220

• all values are in cm^{-1}

Table -3 : Density of as prepared metal oxides from different methods

Sample	Density from XRD	Tap density	Power density
NiO	4830	4980	3930
Fe_2O_3	4980	4999	4866
$NiFe_2O_3$	5030	4995	4630

• All the values are in kg/m^3

Some particles are found as agglomerates. Flakes of agllomerates of $NiFe_2O_3$ particles are also observed. The particles of $NiFe_2O_3$ form self assembled irregular shaped blocks of sub micro dimensions.

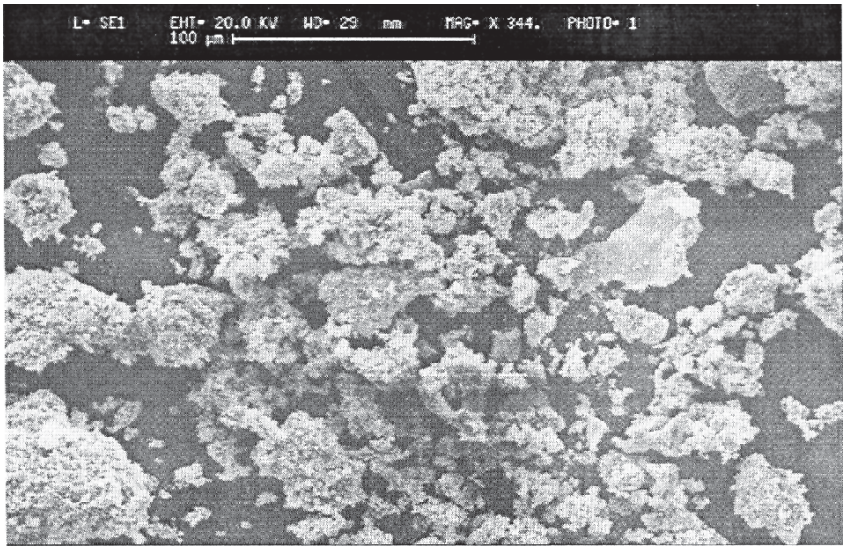

Fig. - 1 : SEM image of $NiFe_2O_3$

Conclusion

Following are the conclusions made from the above study

- This self propagating combustion synthesis method may use for the synthesis other oxide materials
- The XRD data shows the formation of crystalline $NiFe_2O_3$
- Polyethylene glycol acts a fuel in the synthetic reaction. Other polymers may be used as a fuel in the other synthetic reaction.

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