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Microwave Synthesis of $CoFe_2O_4$: Structure, Morphology, Bonding, and Thermal Study

KEYWORDS: Nanomaterials , Microwave, Thermal, Electrical Bonding, Structure, Morphology

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ABSTRACT *Synthesis of ferrite materials at nano dimension using microwave route enhances the synthetic chemistry of nanomaterials. It is an energy efficient method, less time consuming method and also known for its simplicity. Bimetallic oxide nanomaterial preparation using microwave route finds much importance in the field of synthetic technology. Microwave route is used for the preparation of cobalt ferrite using polymer as a fuel in the present study. As prepared cobalt ferrite sample was characterized for its structure by employing powder X-ray diffraction (XRD) tool. The morphology of as prepared oxide material was studied by Scanning Electron Micrograph (SEM) tool. Fourier Transform infrared (FTIR) spectral study was undertaken to know the bonding in the prepared oxide sample. Thermal behavior of the sample was well studied.*

1. INTRODUCTION

The researchers have given more concentration towards the ferrite materials especially at nano scale due to many technological applications [1-2]. Ferrite materials are known for its magnetic and electrical properties which enhances the world's science and technology. The most significant and popular usage of ferrites are in optics, electronics, mechanics [3-4]. Research on nano-sized ferrite has been considerably increased due to their superior properties and applications in new fields like magnetic, drug delivery, catalyst, sensor etc. Among most of the ferrites, cobalt ferrite with inverse spinal structure is promising magnetic materials because of their moderate saturation magnetizastion, high electrical properties, high magneto-crystalline anisotropy, good mechanical properties and chemical stability[5-6]. Many researchers have synthesized ferrite materials by various methods[7-9] Among these methods, combustion route is widely used for the synthesis of ferrite nanomaterials[10-11].

The synthesis of Cobalt ferrite nanomaterials employing microwave route using polyvinyl alcohol as a fuel is reported. Combustion treatment is given for the complete conversion of salts in to bimetallic oxide nanomaterials. As prepared Cobalt ferrite sample was well characterized for its structure by X-ray diffraction (XRD), morphology by Scanning Electron Microscope (SEM) and bonding by Fourier Transform Infrared study (FT-IR) techniques. Thermal stability of the sample was well studied.

2. EXPERIMENTAL

2.1. Synthesis of Cobalt Ferrite

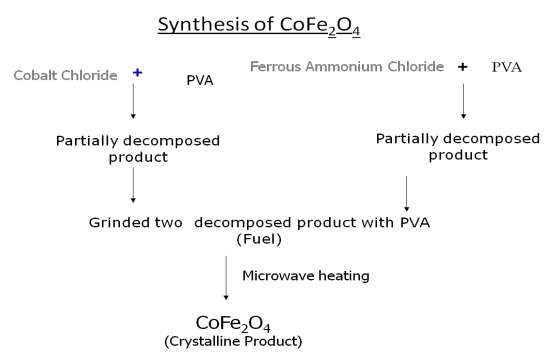
2.1.1 Materials and methods

Cobalt Chloride, Ferrous ammonium sulphate, polyvinyl alcohol chemicals are used in the present study are of AR (Analytical Reagent) grade. Polyvinyl alcohol is used as fuel for the combustion process and Microwave method is adopted for the synthesis of cobalt ferrite nanomaterials

2.1.2. Preparation of cobalt ferrite nanomaterials

One gram of cobalt chloride and ferrous ammonium chloride was ignited with polyvinyl alcohol fuel in a separate container on electrical oven to get partially decomposed product. After complete evolution of the fumes, both samples were again grinded with polyvinyl alcohol in weight ratio 1:1:4. The partially decomposed product formed may be attributed to the low temperature of the reaction giving rise to insufficient energy needed for complete conversion. Hence, the sample was under microwave irradiation in domestic microwave woven having frequency 2.45GHz for about ten minutes at highest power level. The solid burns by producing different coloured light and leaving behind cobalt ferrite nano product. The preparation scheme is given in the scheme 1. [12]

Scheme-1



2.1.3. Characterization

The structures of as prepared cobalt ferrite were studied by X – ray diffraction using Phillips X – ray diffractometer (PW3710) with Cu Kα as source of radiation. Morphology and bonding of the above oxide was studied by Phillips XL 30 ESEM and Perkin–Elmer 1600 spectrophotometer in KBr medium tools respectively. The thermal studies were undertaken using Mettler Toledo Star instrument.

3. RESULTS AND DISCUSSION

3.1. X-ray diffraction

Figure-1 shows XRD pattern of as prepared $CoFe_2O_4$ sample. The pattern shows the presence some Bragg's reflec-

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tions confirm the formation of crystalline product. The d-spacing values of the sample matches well with standard 22-1086 JCPDS file. Unit cell parameters ware obtained by least –square refinement of the powder XRD data. This study reveals that the sample is monophasic cobalt ferrite with cubic spinal structure having nanosized particles.

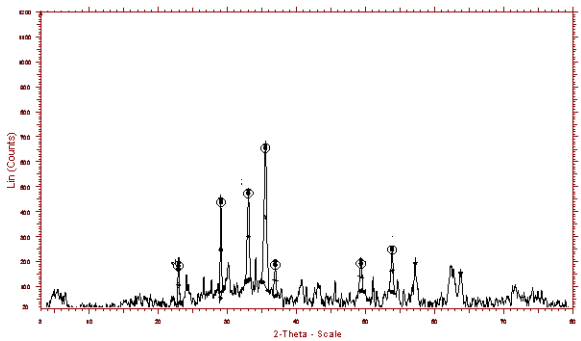

Figure-1: XRD pattern of $CoFe_2O_4$ sample

3.2. Scanning Electron Microscopy (SEM)

Figure-2 shows SEM image of as synthesized cobalt ferrite sample. This image shows the most of the particle are irregular shape with globular arrangement. Some particles are spherical in shape with compact arrangement. In addition to this a very small needle particle are with freely assembled each other is also observed.

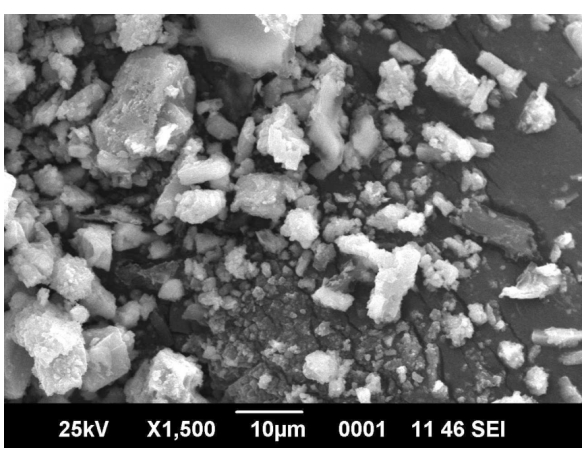

Figure-2: SEM image of $CoFe_2O_4$ sample

3.3. Infrared Study

The metal- oxygen bonding and nature of the synthesized Cobalt ferrite sample was carried out by infrared tool. Metal oxides generally give absorption bands below 1000cm^{-1} arising from inter-atomic vibrations [13]. The peak 3250 cm^{-1} corresponds to water of absorption and the peak at 1150 and 1180 cm^{-1} may be due to the presence of some overtones. The peaks below 1000 cm^{-1} corresponds to Metal-oxygen (M-O) vibrational modes of the sample. This conform the formation of cobalt ferrite sample.

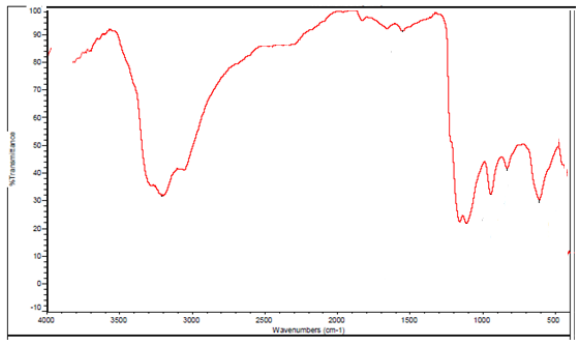

Figure-3: FT-IR spectra of $CoFe_2O_3$ sample

3.4. Thermal Study

Figure-4 shows TGA trace of cobalt ferrite sample. The trace shows two step weight losses, the first weight loss is due to the loss of adsorbed moisture present in the sample. A second loss from 140 to 250 ° C indicates the weight loss due to decomposition of the sample. The weight loss is slow process and is multistep one.

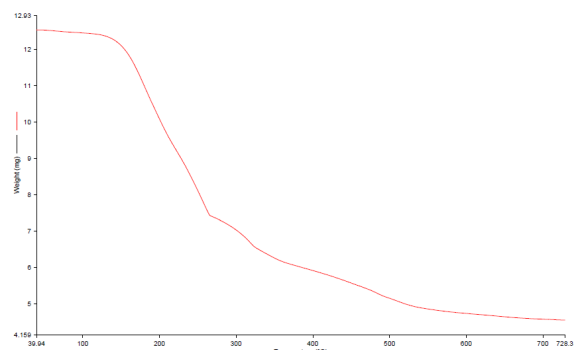

Figure-4: TGA trace of Cobalt ferrite sample

CONCLUSIONS

1) Synthesis of cobalt ferrite by microwave method using polyvinyl alcohol as a fuel
2) This method finds its importance because of its simplicity and less time consuming.
3) Nanosized crystalline materials are prepared by this method
4) . Crystalline materials formed with simple way with applicable morphology.

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