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SYNTHESIS AND INFRARED CHARACTERIZATIONS OF COBALT SPINEL FERRITE NANOPARTICLES

Bhaskar S. Munde
Department of Physics, K. K. M. College, Manwat, 431505, Parbhani (M.S.), India

ABSTRACT

This paper reports facile synthesis and Infrared spectral evaluation of cobalt spinel ferrite nanoparticles. Cobalt ferrite nanoparticles were prepared by sol-gel auto combustion technique using citric acid as a fuel. The fuel to metal nitrate ratio was derived as 1:3 using propellant chemistry. Different Synthesis parameters such as fuel to nitrate ratio, pH, synthesis temperature, stirring speed and annealing temperature were optimized and the same are listed. Further, the prepared nanoparticles were characterized by Infrared spectroscopy (IR) for chemical identification. The room temperature (300 K) IR spectra revealed the presence of two characteristics absorption bands near 400 cm^{-1} and 600 cm^{-1} confirming the formation of cubic spinel structure of prepared nanoparticles.

Keywords: *Cobalt ferrite, Sol-gel auto combustion, Infrared.*

I. INTRODUCTION

Nanocrystalline materials are the subject of intense research because of their prospective applications and fascinating properties. Nanostructured materials are considered very striking as compared to their bulk counterpart as they demonstrate advanced physical and chemical properties because of the quantum confinement, smaller size (nanometer dimension), high surface to volume ratio etc [1-2]. The synthesis of newer materials at nanoscale level is attracting the great attention of scientist and technologist in recent years. The size, shape and purity of the nanostructured materials are more important to modify the properties for desired applications [3-4].

The magnetic properties of spinel ferrite nanoparticles have been of great concern in recent years due to the nanosize constituent particles or crystallites. The magnetic properties of the ferrite nanoparticles are found to undergo changes due to superparamagnetism, surface screen effects and also with their cation distribution which depend on synthesis method. Much attention has been devoted on investigations of nanosized spinel ferrite particles because of their potential applications in several areas of microwave devices, recording media, magneto fluids, catalysis, magneto refrigeration systems and gas sensors [5-7]. Magnetic nanoparticles of spinel ferrite are found to be thermally and chemically stable, that's why they deserved their applicability in special kind of applications.

Spinel ferrites recognized by the formula MFe_2O_4 (where M is a divalent cation like Co, Ni, Mn, Mg etc.) are one of the most interesting magnetic nanoparticles due to their applications in diverse fields. The unit cell of a spinel ferrite crystal structure consists of a cubic closed pack arrangement of oxygen ions with 64 tetrahedral (A-site) and 16 of octahedral [B-site]. Out of this, 8 of tetrahedral (A-sites) and 16 of octahedral [B-site] sites are occupied by the metal cations. The properties of ferrites are strongly influenced by the material composition, microstructure (which is sensitive to preparation method), type and amount of dopant etc. Many synthesis processes have been illustrated in the literature for the synthesis of spinel ferrite nanoparticles. The synthesis methods include ball milling, sol-gel, coprecipitation, hydrothermal etc [8-10].

In the range of different methods of synthesis of spinel ferrite nanoparticles, sol gel auto-combustion technique is fairly simple and cost effective involving both chemical and physical processes [11]. The Sol-gel auto combustion method is based on gelling and subsequent auto combustion of an aqueous solution containing nitrates/chloride/sulphates, fuel/chelating agent, giving a voluminous and fluffy powder with larger surface area and

better crystallinity. Oxidizing metal salts, such as metal nitrates/chlorides/sulphates, and a combustion agent/chelating agent (fuel), such as citric acid, polyacrylic acid or urea are used as starting materials. Due to the good competence of chelating metallic ions and to low decomposition temperatures, citric acid is appropriate for obtaining precursors of transition metal oxides. The organic fuel/chelating agent plays an important role in synthesis process. It is the fuel for the combustion reaction; which forms complexes with metal ions preventing the precipitation of hydroxylated compounds [12-13].

Cobalt ferrite ($CoFe_2O_4$) with high magnetocrystalline anisotropy, high saturation magnetization, high electrical resistivity, high coercivity, is one of the best candidate among the other spinel ferrite for various applications [14-15]. The cubic spinel structured cobalt ferrite, $CoFe_2O_4$, represents a well-known and important class of iron oxide materials. The O^{2-} ions form f.c.c. close packing, and the Co^{2+} and Fe^{3+} occupy either tetrahedral (A) or octahedral (B) interstitial sites. Cobalt ferrites, typically inverse spinel ferrites, have been extensively used in electronic devices because of their large permeability at high frequency, remarkably high electrical resistivity and high saturation magnetization [16-17]. Thus, herein we report the facile synthesis and Infrared spectral evaluation of cobalt spinel ferrite nanoparticles prepared by sol-gel auto combustion method.

II. EXPERIMENTAL METHOD

Materials

Cobalt nitrate (Co $(NO_3)_2 \cdot 6H_2O$), ferric nitrate (Fe $(NO_3)_3 \cdot 9H_2O$) and citric acid were used as a raw materials for sol-gel auto combustion synthesis of $CoFe_2O_4$ spinel ferrite nanoparticles. All the reagents used for the synthesis were of analytical grade (AR) and used as received without further purification.

Preparation

$CoFe_2O_4$ spinel ferrite nanoparticles were synthesized by sol-gel auto combustion method using citric acid as a fuel. The stoichiometric proportions of metal nitrates to fuel (citric acid) ratio as 1:3 were taken into separate glass beakers. The mixed solution was stirred for 20 - 25 minutes to dissolve completely into distilled water. After complete dissolution they were mixed together. Ammonia was added drop-wise into the solution to adjust pH value at 7 and stabilize the nitrate-citric acid solution. Then the neutralized solution was constantly magnetically stirred and heated at 90°C - 100°C for 6 h on a hot plate. On the formation of sol-gel, very viscous gel the temperature was further raised up to 110°C so that the auto combustion of the dried gel started and finally powder was obtained. The reaction mechanism of $CoFe_2O_4$ is depicted in fig. 1. The as prepared loose cobalt ferrite powder was grinded for 40 minutes and annealed at 600°C for 6 h in muffle furnace. The flowchart of the sol-auto combustion synthesis is shown in fig. 2.

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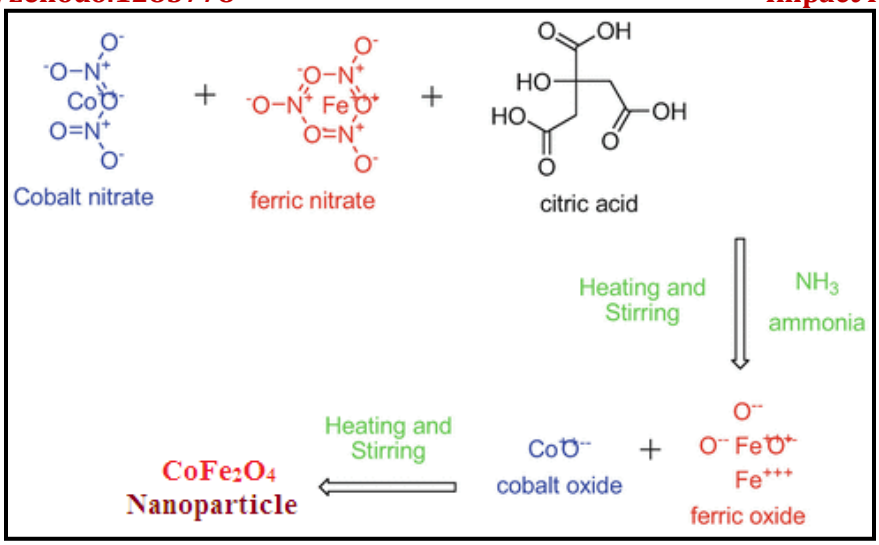

Fig. 1 Reaction mechanism of $CoFe_2O_4$ nanoparticles

Table 1 Various synthesis parameters optimized during sol-gel auto combustion synthesis of $CoFe_2O_4$ nanoparticles

Purity of raw materials	**99.9 %**
Fuel	**Citric acid**
Fuel to nitrate ratio	**1 : 3**
Mixing	**Uniform**
pH	**7**
Temperature	**90 °C – 100 °C**
Stirring speed	**800 rpm**
Annealing temperature and time	**600 °C for 6 h**

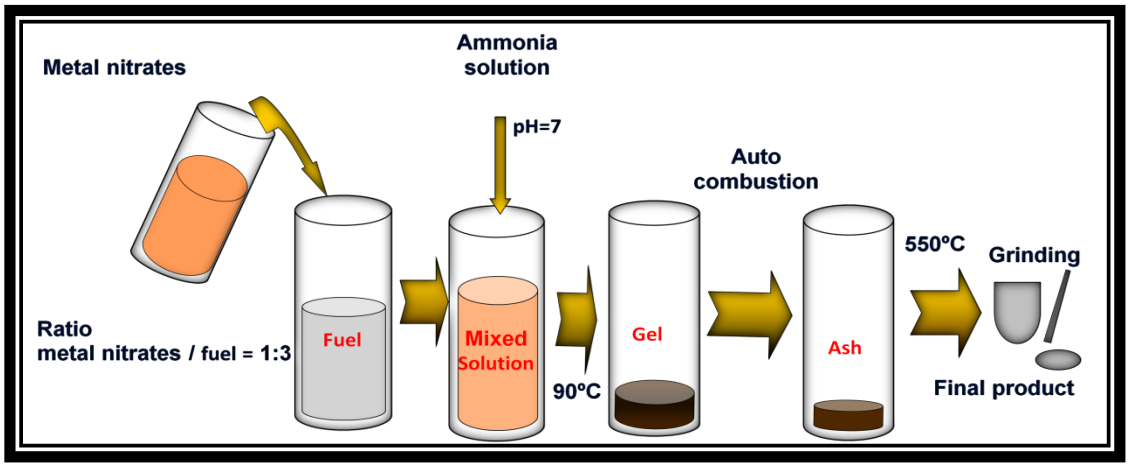

Fig. 2 Flowchart of Sol-gel auto combustion method

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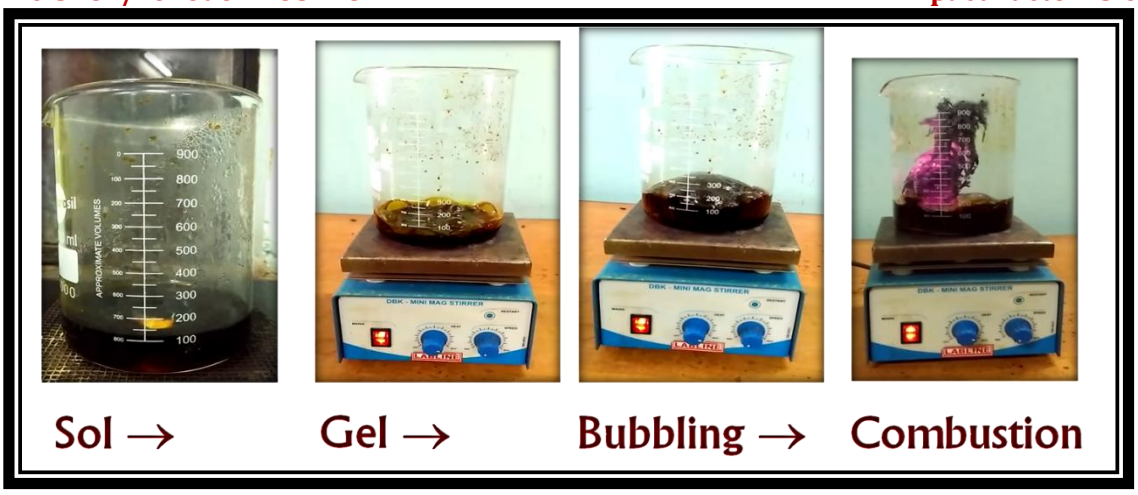

Fig. 3 Step by step Process of Sol-gel auto combustion synthesis

Characterizations

The prepared nanoparticle of cobalt ferrite was characterized by Infrared Spectroscopy (IR) technique. The room temperature IR spectrum was recorded in the range of 400 cm^{-1} to 1000 cm^{-1} using infrared spectrophotometer. For recording IR spectra, powders were mixed with KBr in the ratio 1:100 by weight to ensure uniform dispersion in the KBr pellet. The mixed powders were then pressed in a cylindrical die to obtain clear disc.

III. RESULTS AND DISCUSSION

The Infrared (IR) spectroscopy is one of the most influential analytical techniques, which offers the possibility of chemical identification. The technique is based upon the simple fact that a chemical substance shows marked selective absorption in the infrared region. After the absorption of IR radiation the molecules of chemical substance vibrate at many rates of vibrations, giving rise to closed-packed absorption bands, called IR absorption spectrum, which may extend over a wide wavelength range. Various bands present in IR spectra are corresponding to the characteristic functional groups and bonds present in chemical substance. Thus, an IR spectrum of a chemical substance is the finger print of its identification at least for organic compounds. Infrared absorption spectroscopy has been used to study the occurrence of various absorption bands in the spectra and these have been analyzed on the basis of different cations present at the (A) and [B] sites of the spinel lattice.

Thus, we have employed IR spectroscopy technique in the present study. The infrared (IR) spectrum of $CoFe_2O_4$ nanoparticles was recorded in the range 400 cm^{-1} – 1000 cm^{-1}. The room temperature (300 K) recorded spectrum is depicted in fig 4. It the spectrum two absorption bands at 530.30 cm^{-1} and 360.40 cm^{-1} are observed. No absorption bands were observed above 800 cm^{-1}. Normally, the absorption modes within 1000 cm^{-1} range are attributed to the bands between inorganic elements. The higher frequency band ν_1 and lower frequency band ν_2 were observed in the range of 600 – 500 cm^{-1} and 450 – 360 cm^{-1} and was assigned to tetrahedral (A) and octahedral [B] metal stretching, which are consider to be the typical bands of spinel structure. The difference in the band position (υ_1 and υ_2) is expected because of the difference in the Fe^{3+}- O^{2-} distances for the octahedral and tetrahedral complexes [18]. The values of absorption frequency ν_1, ν_2, K_t and K_O are listed in table 2.

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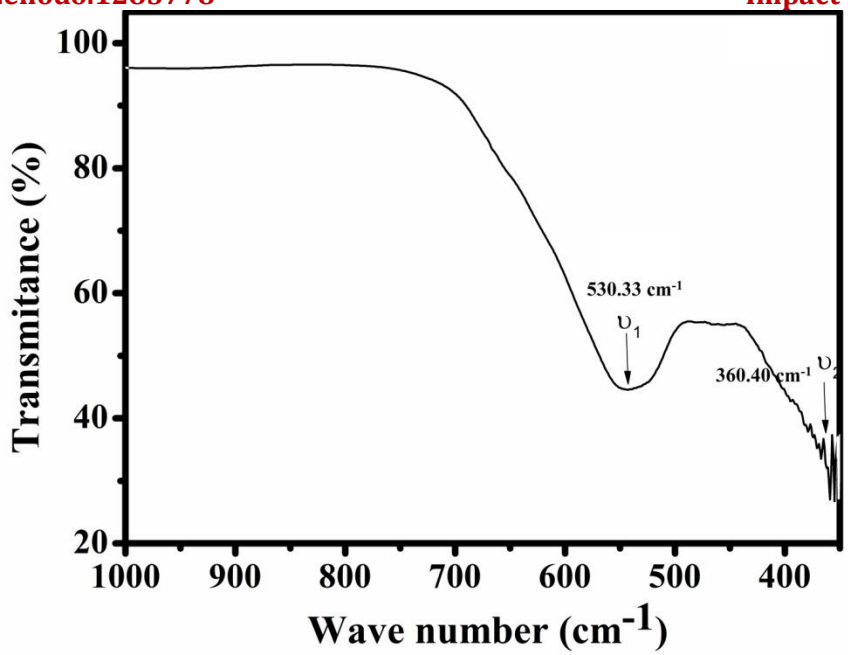

Fig. 4 IR spectrum of $CoFe_2O_4$ nanoparticles

Table 2 Values of infrared absorption frequency band positions (v_1 and v_2) and force constants (K_t and K_O) for $CoFe_2O_4$ nanoparticles

Parameters	$CoFe_2O_4$
ν_1 (cm^{-1})	530.33
ν_2 (cm^{-1})	360.40
K_t (N/m)	117.98
K_O (N/m)	63.28

IV. CONCLUSION

Cobalt ferrite nanoparticles were successfully prepared by sol-gel auto combustion technique using citric acid as a fuel. Different Synthesis parameters such as fuel to nitrate ratio, pH, synthesis temperature, stirring speed and annealing temperature were optimized. The room temperature (300 K) IR spectra revealed the presence of two characteristics absorption bands near 400 cm^{-1} and 600 cm^{-1} confirming the formation cubic spinel structure of prepared nanoparticles. The difference in the band position (υ_1 and υ_2) was attributed to the difference in the Fe^{3+}-O^{2-} distances for the octahedral and tetrahedral complexes.

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