

SOLUTION COMBUSTION SYNTHESIS OF CO₃O₄ NANOPARTICLES

Bony K. John

Assistant Professor (on contract), Department of Chemistry, Deva Matha College
Kuravilangad P.O., Kottayam Dist. Kerala

(Affiliated to Mahatma Gandhi University, Kottayam, Kerala)

ABSTRACT-The main aim of this work is rapid synthesis of Co₃O₄ nanoparticles by solution combustion synthesis and its characterization by IR and PXRD. Co₃O₄ is one of the most thoroughly investigated metal oxide nanoparticles. The reagents used for this work are Co(NO₃)₂.6H₂O (Merck) and Urea (BDH chemicals) of analytical grade. Through this method Co₃O₄ with different morphologies such as nanospheres, nanocube, nanofiber, and mesoporous structures have been prepared. Rapid and Easy synthesis of cubic spinel phase of Co₃O₄ nanoparticles of Crystallite size 27.02 nm portrays the efficiency of the method. The properties of newly synthesized Co₃O₄ strongly depend on their morphologies and structures, including crystal sizes, orientations etc. From this work it is clear that Cobalt Oxide nanoparticles can exhibit unique physical and chemical properties due to their limited size and a high density of corner or edge surface sites. As an important magnetic p-type semiconductor, Co₃O₄ is of special interest due to its potential applications as sensors, heterogeneous catalysts, electrochromical devices, nano submarines and magnetic materials. Co₃O₄ Nanomaterials having wide range of applications in the field of electronics, agriculture, food industry, and medicines, etc.

Keywords: Nanospheres, solution combustion synthesis, Mesoporous structures, Nanoparticles

INTRODUCTION

Solution combustion synthesis (SCS) is an effective method for the synthesis of nanoscale materials and has been used in the production of various ceramic powders for a variety of advanced applications. This is a method based on the principle that once a reaction is initiated under heating, an exothermic reaction occurs that becomes self-sustaining within a certain time interval, resulting in a powder as final product. This process is used directly in the production of high purity, homogeneous ceramic oxide powders widely. The foundation for the combustion synthesis technique lies in the thermodynamic concepts used in the field of propellants and explosives, and its extrapolation to the combustion synthesis of ceramic oxides and its thermodynamic interpretation are discussed extensively by several researchers. The success of this process is closely linked to the mix of constituents of a suitable fuel or complexing agent (e.g., citric acid, urea, and glycine) in water and an exothermic redox reaction between the fuel and the oxidant (e.g., nitrates). The solution combustion synthesis method has proven to be a great technique to obtain various types of oxides at the nanometer scale and is used for a variety of technological applications. This wide range of oxides is prepared with an eye on their magnetic, mechanical, dielectric, catalytic, optical and luminescent properties. Solution combustion synthesis (SCS) takes distinct advantages over other routine methods to synthesize nanomaterials such as coprecipitation, mechanochemical synthesis, microwave assisted synthesis, sol-gel methods etc. Compared with the above methods, there are some obvious advantages in our experiments:

- (1) They can be easily repeated, and the reaction is safe and quick
- (2) They are very convenient and low cost because they do not require expensive reagents, or complicated setups.
- (3) They are environment-friendly since the byproducts are often CO₂, N₂ and H₂O
- (4) It saves time and energy.

Objectives of the study

1. Rapid synthesis of Co₃O₄ nanoparticles by solution combustion synthesis
2. Characterization of synthesized Co₃O₄ nanoparticles by IR and XRD

REAGENTS AND SOLUTIONS

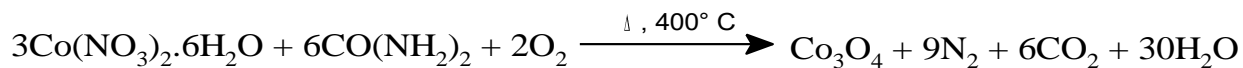
Co(NO₃)₂.6H₂O (Merck) and Urea (BDH chemicals) were of analytical grade and used as received. All aqueous solutions were prepared in double distilled water.

SYNTHESIS OF CO₃O₄ NANOPARTICLES

Co₃O₄ nanoparticles have been prepared by solution combustion synthesis (SCS) as reported elsewhere. 10.0 × 10⁻³ M Co(NO₃)₂.6H₂O and 20.0 × 10⁻³ M urea were prepared separately in 100 mL of double distilled water. 30.0 mL of both the solutions were mixed nicely and transferred into the crucibles. The solutions were appeared to be pink in crucibles. Then, the crucibles were kept in preheated (400° C) muffle furnace for 15.0 minutes. The solution boiled within a few minutes, and was ignited to produce a self-propagating combustion, yielding fluffy loose products which were allowed to cool down naturally in atmosphere. The obtained powder is called Co₃O₄ nanoparticles which appeared to be in brown color. The prepared Co₃O₄ nanoparticles were characterized using IR spectroscopy and PXRD.

RESULTS AND DISCUSSIONS

Mechanism of Formation of Co_3O_4 Nanoparticles



A typical SCS relies on an exothermic reaction between an oxidizer, typically cobalt nitrates, and an organic fuel such as urea. The driving force for lattice formations derives from the internal chemical energy. The temperature input for SCS is not high, which is required just to trigger the combustion reaction, rather than to provide a continuous energy input to form lattices as the case in conventional material fabrication routes. Various water-soluble organics containing large quantities of C and H are utilized as fuels, which facilitates the liberation of heat by combustion. The mixture of oxidizer and fuel was autoignited at 400°C in muffle furnace at the ignition temperature. This self sustainable exothermic reaction leads to the formation of Co_3O_4 nanoparticles .

Characterization of Co_3O_4 nanoparticles using IR spectroscopy

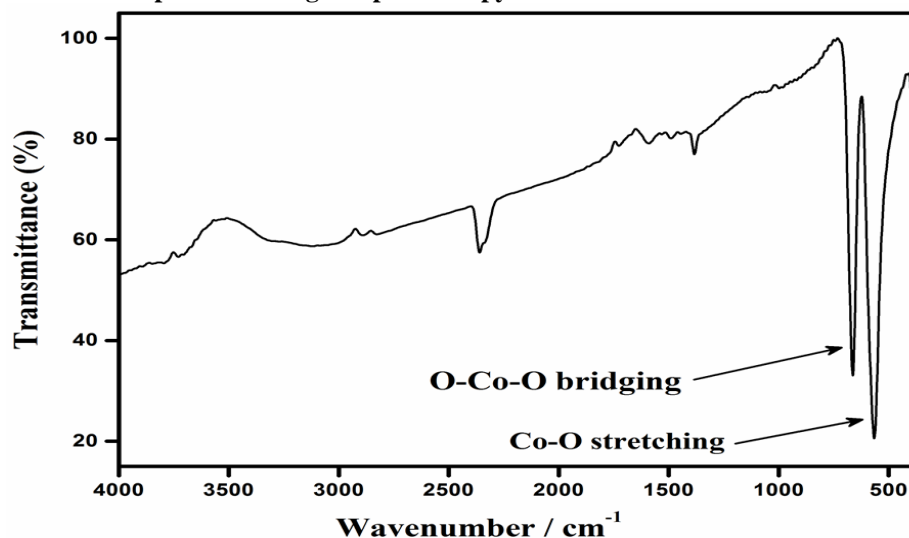


Figure 1: FT-IR spectrum of Co_3O_4 nanoparticles

The IR spectrum of Co_3O_4 was recorded and shown in Fig.1. The lower wave number regions are characteristic of Metal–Oxygen stretching .The characteristic Co–O stretching vibrations of Co_3O_4 nanoparticles occur at 562 cm^{-1} . The absorption band at 660 cm^{-1} was due to the O–Co–O stretching vibration. These stretching vibrations confirm the formation of spinel Co_3O_4 nanoparticles .

Characterization of Co_3O_4 Nanoparticles Using PXRD

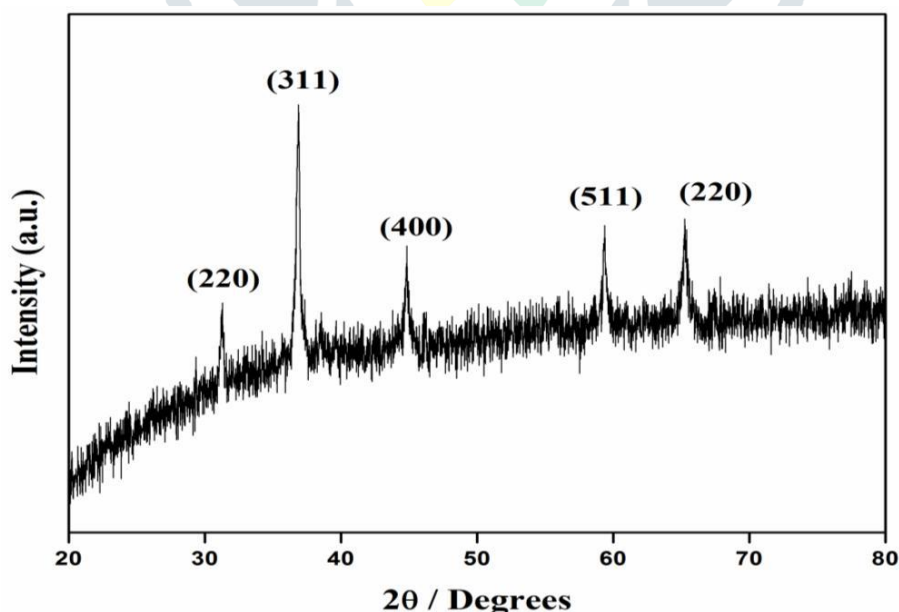


Figure 2: PXRD pattern of Co_3O_4 nanoparticles.

The prepared Co_3O_4 nanoparticles were characterized using X-Ray diffraction technique. The resulted PXRD pattern of Co_3O_4 nanoparticles is shown in Fig. 2. This data clearly shows distinct peaks at 2θ of 31.18° , 36.86° , 44.9° , 59.41° and 65.32° correspond to (220), (311), (400), (511) and (220) Bragg reflections of Co_3O_4 . The observed reflections are clearly seen and

closely match the reference pattern for Co_3O_4 nanoparticles maintained by International Centre for Diffraction Data (ICDD or JCPDS card No. 43-1003). The diffraction peaks can be clearly indexed to a cubic spinel Co_3O_4 phase with the strongest peak being the (311) reflection [47,55]. The sharp peaks indicate the crystalline nature of the prepared Co_3O_4 nanoparticles. Besides, no other secondary or amorphous phase was found from the PXRD pattern indicates pure crystalline phase of Co_3O_4 nanoparticles. The prominent peaks of Co_3O_4 have been utilized to calculate the particle (crystallite) or grain size of Co_3O_4 nanoparticles. The Debye-Scherrer equation was exploited for the calculation of crystallite size. The Debye-Scherrer equation can be written as follows:

$$D = \frac{K\lambda}{\beta \cos\theta}$$

where,

D → Average size of crystallite

K → Constant with value 0.9

λ → Wave length of X-Ray used (0.154056 nm)

β → Full width at half maximum (FWHM in radians)

θ → Angle of diffraction (Bragg's angle in degrees)

$$D = \frac{0.9 \times 0.154}{\beta \cos 18.43} = \frac{0.1386}{0.00541 \times \cos(18.43)} = \frac{0.1386}{0.00513} = 27.02 \text{ nm}$$

The significance of present synthesis route lies in the rapid synthesis of homogeneous high quality cubic spinel Co_3O_4 nanoparticles of 27.02 nm size which could be used for the various applications in science and technology.

CONCLUSIONS

Rapid and Easy synthesis of cubic spinel phase of Co_3O_4 nanoparticles were carried out using solution combustion synthesis. This method is very suitable for the synthesis of Co_3O_4 nanoparticles with Crystallite size of 27.02 nm. The prepared Co_3O_4 nanoparticles were characterized using IR spectroscopy and X-ray diffraction which ascertain the formation of Co_3O_4 nanoparticles. The stretching vibrations of Co–O of Co_3O_4 nanoparticles occur at 562 cm^{-1} and the absorption band at 660 cm^{-1} due to the O-Co-O stretching vibration confirm the formation of spinel Co_3O_4 nanoparticles. The diffraction peaks can be clearly indexed to a cubic spinel Co_3O_4 phase with the strongest peak being the (311) reflection. The sharp peaks indicate the crystalline nature of the prepared Co_3O_4 nanoparticles. Besides, no other secondary or amorphous phase was found from the PXRD pattern indicates pure crystalline phase of Co_3O_4 nanoparticles. The prominent peaks of Co_3O_4 have been utilized to calculate the particle (crystallite) or grain size of Co_3O_4 nanoparticles. Further studies reveals wide range of applications of prepared Co_3O_4 nanoparticles in various fields of science and technology such as in heterogeneous catalysts, solid-state sensors, electrochromical devices, and magnetic materials.

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