

# Structural, morphological, optical and magnetic properties of $\text{Co}_3\text{O}_4$ nanoparticles prepared by conventional method



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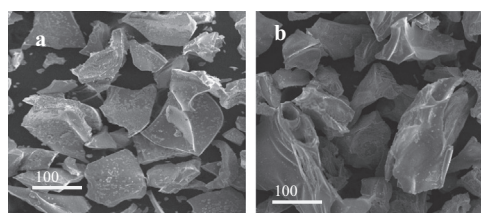
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## HIGHLIGHTS

- Simple route for the preparation of cobalt oxides.
- Conventional method is a useful and attractive technique.
- It provides higher % of yields with good purity.

## GRAPHICAL ABSTRACT

HR-SEM images of (a) sample A- $\text{Co}_3\text{O}_4$  prepared by the CM (starch) (b) sample B-  $\text{Co}_3\text{O}_4$  prepared by the CM (aqueous ammonia solution).



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## ABSTRACT

Cobalt oxide ( $\text{Co}_3\text{O}_4$ ) is one of the favorable nanoparticles (NPs) that possesses many remarkable properties so that it can be used in medicine, chemistry, environment, energy, information, industry, and so on. In this study, the crystalline  $\text{Co}_3\text{O}_4$  nanoparticles (NPs) were successfully prepared by an efficient conventional method technique from an using different fuels. In the present paper, pure phase and well-dispersed  $\text{Co}_3\text{O}_4$  were synthesized via the starch and aqueous ammonia solution in the stoichiometric fuel compositions. The structure and morphology of by way of organized  $\text{Co}_3\text{O}_4$  nanoparticles were characterized by the structural analysis, electron microscopy studies, and optical properties studies. Magnetic properties exposed that the  $\text{Co}_3\text{O}_4$  nanoparticles had ferromagnetic performance at room temperature with saturation magnetization of 71.09 emu/g. The results revealed that the changing the precursor led to great effects on the crystal size, emission peaks, and the reaction time of preparing the  $\text{Co}_3\text{O}_4$  NPs. The significant feature of this manuscript is that the effects of different precursors on the structural magnetic and optical properties of  $\text{Co}_3\text{O}_4$  NPs were investigated for the first time. The average particle size of samples (A and B) 23.6 and 22.2 nm, respectively.

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## 1. Introduction

Small size and the large surface-to-volume ratio of nanoparticles can lead to the different physical and chemical properties

which are altered from those of their bulk counterparts. Recently, there has been increasing interest in the synthesis of nanocrystalline mixed metal oxides. Metal oxide spinels comprise a very large group of structurally related compounds many of which are of considerable technological or environmental significance. Spinel structure materials exhibit a wide range of electronic, optical and magnetic properties, including superconductivity in cobalt oxide [1].

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Attention was first drawn to their optical properties, since  $\text{Co}_3\text{O}_4$  has been a historic blue pigment for a long time till today, for instance as a contrast-enhancing luminescent pigment. These materials show us the interesting enhanced optical properties, with potential applications in the design of optoelectronic materials and as efficient  $\text{Co}_3\text{O}_4$  materials for flat-panel displays. This synthetic approach might be extended to prepare other kinds of nano and mesostructures having excellent properties and various application areas [2,3].

One of the greatest important advantages of nanosemiconductors is their tunable band gap i.e., the absorbed or emitted radiation wavelength can be adjusted by the changing the conditions of their preparation. Additional advantage is that in contrast to traditional semiconductor materials that they are crystalline or rigid, they can be designed in a change of different forms like nanostructures or three-dimensional arrays [4–6]. It is well recognized that chemical, structural and the magnetic properties of spinel cobalt oxide are strongly influenced by their composition, nano and microstructures, which are sensitive to the preparation methods [7,8].

Spinel-type cobalt oxide ( $\text{Co}_3\text{O}_4$ ) are commonly prepared by the ceramic technique that involves, such as, combustion method, microwave irradiation, hydrothermal/solvothermal method, chemical spray pyrolysis, sonochemical method and modified sol–gel method [9–13]. Compared with other techniques, the conventional method is a useful and attractive technique for the preparation of  $\text{Co}_3\text{O}_4$  because of the fact that pure and the ultrafine powders can be formed on relatively low temperatures.

In this work,  $\text{Co}_3\text{O}_4$  were prepared by the modified sol–gel method, due to the simplicity, low temperature and cost effective. The starch and aqueous ammonia solution prepared by the conventional method nanocrystalline  $\text{Co}_3\text{O}_4$  was achieved and the effect of different factors on the controlling crystalline phase, size and electron state of  $\text{Co}_3\text{O}_4$  was investigated. The glycine and calcination temperature were chosen as controllable input factors. The structure, morphology, optical and the magnetic properties of the as-prepared  $\text{Co}_3\text{O}_4$  remained studied by the XRD, HR-SEM, HR-TEM, PL, and VSM spectra, respectively. This approach provides a one-step, simple, general, and the inexpensive method for the preparation of the  $\text{Co}_3\text{O}_4$  nanoparticles.

## 2. Materials and methods

### 2.1. Preparation of $\text{Co}_3\text{O}_4$

All the substances used were analar grade attained from Merck, India and were used as received without further purification. conventional method depend on the benefit of the propellant chemistry in making the redox mixtures, in which cobalt nitrate acts as an oxidizing reactant and fuel (starch and aqueous ammonia solution) as a reducing reactant. In the first step, stoichiometric amounts of cobalt nitrate (0.810 g), starch (0.45 g) and aqueous ammonia solution 15 ml (25 wt%) were dissolved separately in 25 ml of de-ionized water and the poured into a silica crucible and stirred for 15 min to obtain clear solution. Second stage, successive 60 °C/2 h constant heating with incessant stirring let the ions react within totally subsequent in a see-through colourless solution and the continued until the solution transformed into a viscous light green and red gel. All precursors were ground into powders and calcined in a furnace at 300 °C for 3 h to obtain the  $\text{Co}_3\text{O}_4$  powder. The resulting powders are represented as sample A(starch) and sample B (aqueous ammonia solution).

### 2.2. Characterizations $\text{Co}_3\text{O}_4$

The structural studies were carried out using a Philips X' pert diffractometer for  $2\theta$  values ranging from 10° to 80° using Cu K $\alpha$  radiation at  $\lambda=0.154$  nm. Philips PW3040 with Cu K $\alpha$  radiation ( $k=0.15406$  nm) Holland] was used to identify phase and structure of the powder. Morphological studies and energy dispersive X-ray analysis of the nanomaterials have been performed using a Joel JSM6360 high resolution scanning electron microscope. The samples were coated with gold by a gold sputtering device for the better visibility of the surface morphology. Photo luminescent (PL) analysis was conducted on a Hitachi F-4500 spectrophotometer with Xe lamp at room temperature. The magnetic behavior at room temperature was studied by the vibrating samples magnetometry (VSM 7403, Lakeshore, USA) in a field up to  $\pm 10$  kOe.

## 3. Results and discussion

### 3.1. XRD studies

The purity and crystallinity of the  $\text{Co}_3\text{O}_4$  were observed via using powder X-ray diffraction (XRD), as shown in Fig. 1. It can be seen in Fig. 1(a, b) that the diffraction peaks are broadened markedly due to the small size effect of the particles.  $\text{Co}_3\text{O}_4$  powder correspond to the representative peaks of the cubic spinel-phase  $\text{Co}_3\text{O}_4$ , specifically, the peaks at  $2\theta=31.9$ , 36.1, 44.7, 55.2, 59.8 and 65.4 are associated with (220), (311), (400) (422), (511), and (440) plane, respectively. These planes were then associated with D-spacing values of 2.85, 2.42, 2.01, 1.65, 1.54, and 1.42 Å, respectively, which could be readily assigned to a cubic phase of  $\text{Co}_3\text{O}_4$  (space groupFd3m). Further observation revealed that the sample A and sample B have sharp peaks, indicating good crystallinity, but the diffraction peaks for sample B are slightly broadened, due to the smaller crystallite size [14,15].

These XRD patterns are in agreement with the JCPDS card (JCPDS No. 76–1802) and also specify that the produce is a single-phase and the high purity material.  $\text{Co}_3\text{O}_4$  samples, which were prepared chelating agents (starch and aqueous ammonia solution) in the stoichiometric fuel compositions. The results also exposed that the using starch and aqueous ammonia solution in their fuel mixture, reason broadening and the characteristic peaks, due to its consequence on reducing reaction. This occurrence showed to be valuable for procurement well crystallized yields, which include easy agglomerate with small crystallite size [16]. The mathematical convolution of the peaks by a lorentz function permitted a

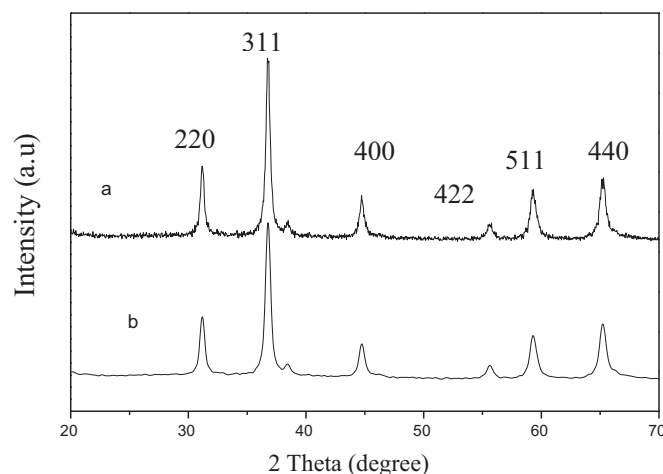


Fig. 1. XRD pattern of  $\text{Co}_3\text{O}_4$  samples (A and B).

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