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Structural Characterization of Co_3O_4 Nanoparticles Synthesized By a Sol-Gel Method

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Abstract: ***Cobalt oxide (Co_3O_4) nanoparticles were prepared by the simple approach of sol–gel process using starch as a capping agent and cobalt chloride as a precursor. The structure and morphology of as-prepared cobalt-oxide nanoparticles were characterized by Fourier transform infrared (FT-IR) spectroscopy, X-ray diffraction (XRD) and transmission electron microscopy (TEM). FT-IR and XRD results indicated that the product was highly pure well-crystallized cubic phase of cobalt oxide nanoparticles. The TEM images showed that the product powder consisted of dispersive quasi-spherical particles with an average size around 50 nm.***

Keywords: Nanostructure cobalt oxide; sol-gel method; XRD.

1. Introduction

Nanotechnology has become one of the most interesting disciplines in science and technology today. The intense interest in nanotechnology is being driven by various interesting fields and is leading to a new industrial revolution. Nano-materials such as nanoparticles, carbon nanotubes or nanocomposite connected with biomolecules are being used for several bioanalytical applications. In recent years, synthesis of transition metal oxide nanoparticles has attracted much attention because of their outstanding multifunctional physical-chemical properties for their use in different fields. The actual challenge that depends on how to optimize a cost-effective synthetic methodology via soft chemical approach that gives technological grade nanomaterials with specific structural-morphological functional properties remains a challenge to synthetic chemists. Cobalt oxide nanoparticles are widely used in many fields such as magnetic [1-4], gas sensor, lithium ion batteries, catalysis, and electrochemical depending on the size, structure, shape, and phase homogeneity and with surface morphologies. Many approaches were made for the successful synthesis of cobalt oxide nanoparticles in past one decade by using different synthetic approaches, such as thermal method, precipitation methods, pyrolysis process, and sonochemical method [5-8]. However, all these methods have a limited control in particle functional properties with low yield. Therefore, it is necessary to find alternative method for the synthesis of nanopowder that should be cost-effective and environmental friendly. The sol-gel approach is the best synthetic method which helps to synthesize of cobalt oxide nanopowder.

2. Experimental

2.1 Materials and methods

Cobalt chloride (99%) was purchased from Merck. Other supplement chemicals were of AR grade and used as received except aniline which was distilled prior to use. FTIR spectra was performed on Shimatzu FTIR-8101A Spectrophotometer in the wavelength range of 400–4000cm^{-1}. X-ray diffraction (XRD) pattern was obtained using Philips PW1710 automatic X-ray diffractometer with Cu-Ka radiation (λ=1.5404A°) and scanning speed of 10°min^{-1}. Transmission electron microscopy image was obtained using PHILIPS-CM200 TEM with resolution 2.4A°.

2.2 Synthesis of Co_3O_4 nanoparticles

Cobalt oxide (Co_3O_4) nanoparticles were synthesized by the sol–gel process [9] in which 0.1M of cobalt chloride (precursor) was added in 100ml starch solution and then stirring the solution for 30 min. Prepared solution was hydrolyzed by NaOH under constant stirring at room temperature for 2 h. The solution was kept overnight and then filtered using membrane filtration assembly, washed using deionized water and ethanol to remove the impurities present in prepared sample and then dried at 80°C in hot air oven. Dried sample was treated at different temperatures in order to maintain the stability of compound. The color of the sample was changed from faint green at 100°C to black at 750°C.

3. Results and Discussion

3.1 IR Analysis

FTIR spectra of Co_3O_4 nanoparticles are shown in Figure 1. In the IR spectra of Co_3O_4 nanoparticles, two bands were observed in the 700 to 400 cm^{-1} region corresponding to the stretching vibration of metal oxygen bond. The band at 577 cm^{-1} corresponds to stretching band of Co-O in the tetrahedral system (Td) and the band 668 cm^{-1} corresponds to stretching bond of metal Co-O in the octahedral system [10]. A stretching frequency at 3457 cm^{-1} and a weak asymmetric band at 1633 cm^{-1} support the presence of OH^- group due to the absorption of water by nanoparticles during sample preparation.

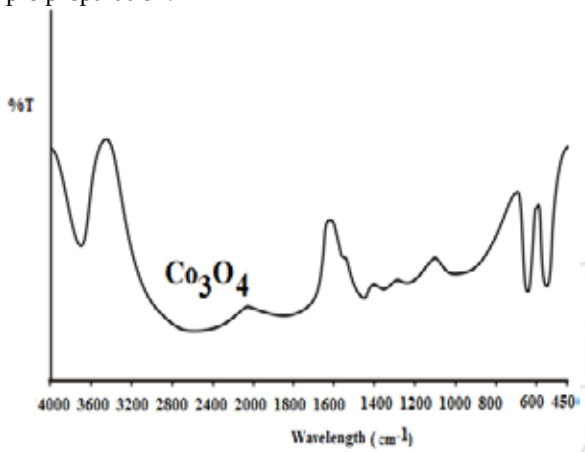

Figure 1: FTIR spectra of Co_3O_4

3.2 XRD Analysis

The XRD patterns of cobalt oxide nanoparticles are shown in Fig. 2. According to standard Co_3O_4 XRD pattern (JCPDS card no. 80–1537). All the peaks of cobalt oxide can be indexed to cubic phase. According to Scherer equation the crystallite size of Co_3O_4 resulted from sol gel method was 35nm. Co_3O_4 nanoparticles with cubic structure showed four broad peaks at about 31.67°, 37.26°, 59.75° and 65.56° for its nanostructure. It reveals that Co_3O_4 synthesized by the sol–gel method is nanocrystalline [11].

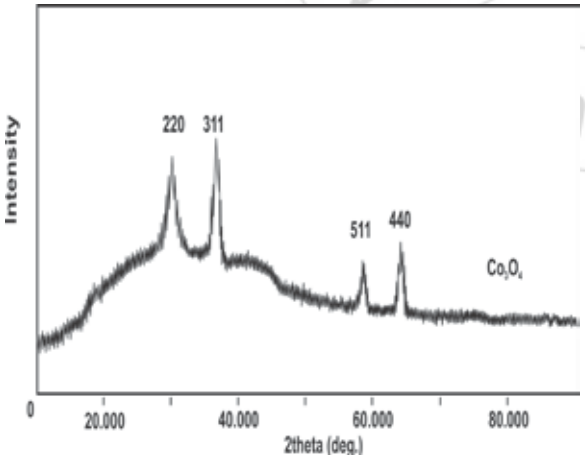

Figure 2: XRD pattern of Co_3O_4

3.3 TEM Analysis

The morphology of Co_3O_4 was studied by TEM. From the TEM image of Co_3O_4, it reveals that the Co_3O_4 nanoparticles were formed from extremely fine particles with the size 50 nm as shown by figure 3. It is evident that the particles have uniform size, a homogeneous sphere-like morphology and a narrow size distribution. From the TEM image of Co_3O_4, it could be concluded that this preparation method is appropriate to obtain the Co_3O_4 nanoparticles with very small size [12].

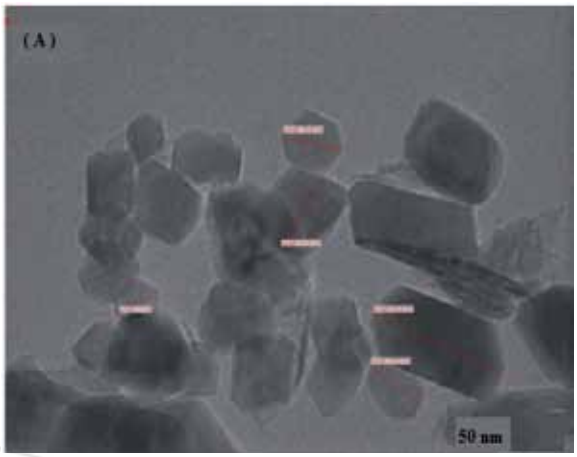

Figure 3: TEM image of Co_3O_4

4. Conclusions

Co_3O_4 nanoparticles with an average particle size of 35nm were successfully synthesized by the sol gel method. IR spectroscopy gives information about the phase composition and the way in which oxygen is bound to the cobalt metal ions. TEM image of Co_3O_4 indicated that the prepared Co_3O_4 nanoparticles have spherical morphology and average particle size of 50nm.

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