



### Short Communication

## Chemical synthesis of cobalt oxide (Co<sub>3</sub>O<sub>4</sub>) nanoparticles using Co-precipitation method

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### Abstract

In the present work, Cobalt oxide (Co<sub>3</sub>O<sub>4</sub>) nanoparticles were synthesized by co-precipitation method. The structural purity of as-prepared nanoparticles was confirmed by using X-Ray diffraction (XRD) analysis. The average particle size of amorphous structure of Co<sub>3</sub>O<sub>4</sub> nanoparticles was found to be 25.62 nm and lattice parameter 7.97Å. The optical band gap of Co<sub>3</sub>O<sub>4</sub> nanoparticles was estimated by using ultraviolet-visible (UV-VIS) spectroscopy. The direct band gap value was found to be 4.07eV. Photoluminescence (PL) spectroscopy was investigated two broad emission peaks at wavelength 396nm, 467nm of Co<sub>3</sub>O<sub>4</sub> nanoparticles.

**Keywords:** Cobalt oxide, Co-precipitation method, Optical properties.

### Introduction

The transition metal oxide have greatly attracted towards wide range of application in durable solar absorber<sup>1</sup>, glucose sensor<sup>2</sup>, lithium-ion battery as electrode material<sup>3</sup>, supercapacitor<sup>4,5</sup>, gas sensor<sup>6</sup>. Among the transition metal groups, cobalt oxide (Co<sub>3</sub>O<sub>4</sub>) is one of the most important materials because of its fascinating properties and thermal stability<sup>7</sup>. The Co<sub>3</sub>O<sub>4</sub> is spinal structure of p-type semiconducting materials with chemical stability at high temperature, high mechanical strength and direct band gap (1.48-2.19eV)<sup>8</sup>. The Co<sub>3</sub>O<sub>4</sub> nanoparticles show good conductivity due to the existence of Co<sup>3+</sup> ions<sup>9</sup>. Nandapure et al.<sup>10</sup> reported synthesis of Co<sub>3</sub>O<sub>4</sub> nanoparticles at 750°C by using sol-gel method. Yarestani et al.<sup>11</sup> spherical Co<sub>3</sub>O<sub>4</sub> nanoparticles was prepared in aqueous KOH solution with stabilizing agents (Triton X-100) by hydrothermal method. Makhlof et al.<sup>12</sup> have been studied electrical conductivity of cobalt oxide at different temperature. The co-precipitation method is a simple process for the formation of nanoparticle and size of nanoparticles controlled by using this method. The composites of different metal oxide can be prepared using this method and modified particle surface by doping other nanoparticles.

In the present article, the stable phase of cobalt oxide (Co<sub>3</sub>O<sub>4</sub>) powder was prepared by co-precipitation method without any capping agent. The as-prepared cobalt oxide (Co<sub>3</sub>O<sub>4</sub>) was analyzed through X-ray diffraction pattern (XRD) (Rigaku miniflex-II diffractometer, CuK $\alpha$  radiation ( $\lambda=1.54\text{\AA}$ ), Ultraviolet-Visible (UV-Vis) Spectrometer (Model: Lamda25 Perkin Elmer) and PL Spectrophotometer (Model: HITACHI, F-7000).

### Materials and methods

The cobalt oxide (Co<sub>3</sub>O<sub>4</sub>) nanoparticles were synthesized without any capping agents by co-precipitation method. In a typical procedure, 1: 2 M ratio of Co(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O, and NaOH were dissolved in 20 mL distilled water under constant magnetic stirring. In this procedure, 2M NaOH solution was dropped in 1 M Co(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O solution under constant magnetic stirrer for 2 hours at room temperature. Then it was permitted to hold on over night at room temperature. The cobalt hydroxide settles down and the excess solutions found on top discarded very carefully.

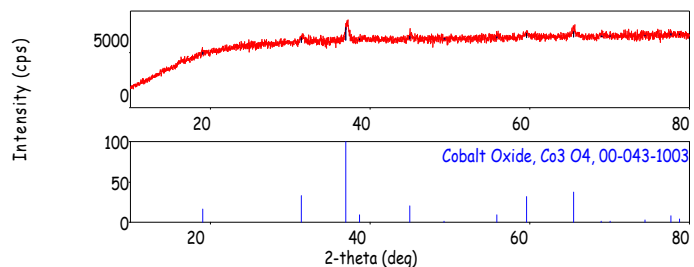
The stock solution of precipitates were separated using centrifuge machine. Finally Co<sub>3</sub>O<sub>4</sub> was obtained in black colour from their original one when it was heated at 353K for 5 hours.

### Results and discussion

The typical X-ray diffraction (XRD) spectrum of cobalt oxide (Co<sub>3</sub>O<sub>4</sub>) nanoparticles is shown in Figure-1. The intensity peaks and scattering angle of synthesized Co<sub>3</sub>O<sub>4</sub> nanoparticles is well consistent with cobalt oxide, Co<sub>3</sub>O<sub>4</sub> (PDF card No.00-043-1003). In the XRD spectra of Co<sub>3</sub>O<sub>4</sub> nanoparticles, the absence of higher intense peak inside Figure 1 indicates the cobalt oxide is in amorphous phase. The seven peak appears at  $2\theta = 19.29^\circ$ ,  $31.60^\circ$ ,  $37.15^\circ$ ,  $55.86^\circ$ ,  $59.65^\circ$ ,  $65.47^\circ$ ,  $68.91^\circ$  related to the plane of (1, 1, 1), (2, 2, 0), (3, 1, 1), (4, 2, 2), (5, 1, 1), (4, 4, 0), (5, 3, 1) respectively. The average particle size of material was determined using Debye-Scherrer formula:

$$D = K\lambda/\beta\cos\theta \quad (1)$$

Where:  $\beta$  is the full width at half maximum in radian,  $\theta$  is the scattering angle,  $\lambda$  is the X-ray wavelength of radiation with  $1.54\text{\AA}$ ,  $K$  is the correction factor (0.89) and  $D$  is the particle size of material in nm. The substituting the values in equation (1), the average size of  $\text{Co}_3\text{O}_4$  particles was estimated as  $25.62\text{nm}^{13}$ . The lattice parameter ( $a_0$ ) determined according to the relation  $a_0 = d_{hkl} (h^2 + k^2 + l^2)^{1/2}$ <sup>14</sup>. The lattice parameter ( $a_0$ ) of  $\text{Co}_3\text{O}_4$  nanoparticles was calculated as  $7.97\text{\AA}$  from the major (3, 1, 1) plane which is approximately described in et al. tuwirqi<sup>15</sup>. The determined average micro strain ( $\epsilon$ ) produced in  $\text{Co}_3\text{O}_4$  nanoparticles was found to be  $0.3206 \text{ lines}^{-2}/\text{m}^4$ <sup>16</sup>.

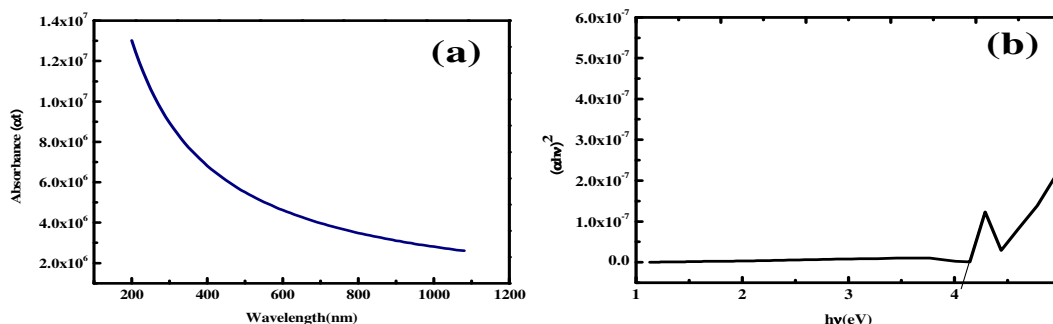


**Figure-1:** XRD spectra of as-synthesized  $\text{Co}_3\text{O}_4$  nanoparticles.

Table-1 displays structural parameters such as  $2\theta$  position, Full width at half maxima (FWHM), miller indices (h, k, l), particle size (D) nm and average particle size (D) nm.

**Table-1:** Structural parameters of as prepared  $\text{Co}_3\text{O}_4$  nanoparticles.

Sr.No	$2\theta$ (deg)	FWHM	h,k,l	D(nm)	Strain( $\epsilon$ ) $\text{lines}^{-2}/\text{m}^4$
1	19.29	0.55	1,1,1	14.69	0.8093
2	31.60	0.51	2,2,0	16.23	0.4506
3	37.15	0.49	3,1,1	17.14	0.3646
4	55.86	0.18	4,2,2	50.08	0.0849
5	59.65	0.63	5,1,1	14.57	0.2748
6	65.47	0.20	4,4,0	47.57	0.0778
7	68.91	0.5	5,3,1	19.32	0.1822



**Figure-2:** (a) UV-Vis spectra (b) Band gap of as-synthesized  $\text{Co}_3\text{O}_4$  nanoparticles.

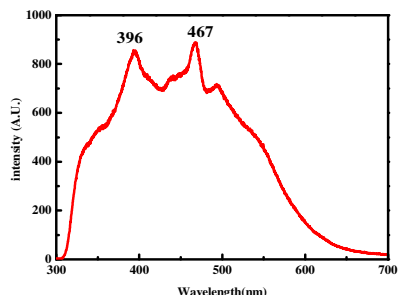
**UV-Vis Spectroscopy:** Figure-2 shows ultraviolet-Visible (UV-Vis) spectra of as-prepared sample in range between 200-1100 nm. The  $\text{Co}_3\text{O}_4$  sample was deposited on clean glass substrate using screen printing technique. The Figure-2 shows optical absorbance spectrum of as prepared film at  $100^\circ\text{C}$ . From UV-Vis spectra of  $\text{Co}_3\text{O}_4$  sample, it reveals that the optical absorbance of  $\text{Co}_3\text{O}_4$  nanoparticles was gradually decreases with an increasing wavelength within region 200-1100nm<sup>17</sup>.

The band gap of materials was estimated from absorption coefficient ( $\alpha$ ) and photon energy ( $h\nu$ ) using the following relation<sup>18</sup>.

$$(\alpha h\nu) = A (h\nu - E_g)^n \quad (2)$$

Where:  $\alpha$  is the absorption coefficient,  $h\nu$  is the incident photon energy,  $A$  is constant and the value of  $n$  is 2 for a direct transition and  $1/2$  for an indirect transition. In this case,  $n=2$  for the determination of optical band gap of  $\text{Co}_3\text{O}_4$  nanoparticles. Figure-3(b) shows the plot of  $(\alpha h\nu)^2$  versus photon energy ( $h\nu$ ). The value of band gap of  $\text{Co}_3\text{O}_4$  nanoparticles was measured by extrapolating the intercept line on the photon energy ( $h\nu$ ) axis gives band gap ( $E_g$ ),  $4.07\text{eV}$ .

**Photoluminescence (PL):** Figure-3 show PL emission spectra of cobalt oxide ( $\text{Co}_3\text{O}_4$ ) powder under UV excitation at 240 nm. Figure-3 observed that emission spectra consist broad band in range 300-700nm and two small sharp peaks were appeared at 396nm and 467nm.



**Figure-3:** PL emission spectrum of  $\text{Co}_3\text{O}_4$  nanoparticles under UV excitation.

## Conclusion

In the summary of present work,  $\text{Co}_3\text{O}_4$  nanoparticles were successfully achieved by co-precipitation method. The XRD analysis suggests that as-prepared  $\text{Co}_3\text{O}_4$  nanoparticles have some amorphous content. The average particle size of  $\text{Co}_3\text{O}_4$  nanoparticles was found to be 25.62nm. The values of mean strain ( $\epsilon$ ) and lattice parameter ( $a_0$ ) of cobalt oxide powder determined as 0.3206 lines<sup>-2</sup>/m<sup>4</sup> and 7.97Å. The optical band gap of cobalt oxide powder was found to be 4.07eV. In the PL spectra, there are two small sharp peaks were appeared at 396nm and 467nm.

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