Structural and optical studies of nickel-cobalt-ferric oxides nanocomposite

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Abstract. In this study single salts of nickel oxide, cobalt oxide, ferric oxide and the nanocomposite of nickel-cobalt-ferric oxide was prepared by the well-known co-precipitation method. The samples were annealed at different temperatures and were characterized using SEM, EDAX, TGA, FTIR, XRD, UV and Photo luminescence (PL). UV analysis showed that the nanocomposite can be suitably tuned to a wide band gap material. The Photo Luminescence analysis showed that the nanocomposite can be used as light emitters in visible region.

1. Introduction
During the last few years, synthesis of nano structured oxide materials had attracted considerable attention [1]. The metal oxides are extremely important technological materials for use in electronic and photonic devices, in chemical industries and in medical fields [2]. An expanding trend for the nanomaterials is the fabrication of composite structures and devices with materials capable of enhancing the properties of the composite material. This can be done either by utilizing the size advantage through templating on the nanomaterials or by enhancing the properties to drive new synergetic properties of the two combined materials. It is also found that the magnetic nanoparticles provide unprecedented levels of new functionality for nano medicine. Such particles commonly consist of magnetic elements such as iron, nickel and cobalt and their chemical compounds [3]. After surface modification these materials provide both biocompatibility and functionality. In the present work a nanocomposite of nickel-cobalt-ferric oxide was prepared and their modifications in various known properties have been studied.

2. Materials and Methods
AR grade chemicals obtained from Merck were used for the preparing single salts of nano nickel oxide, cobalt oxide, ferric oxide and then the composite. Individual nano oxides and the composite were prepared by the co-precipitation method. The samples were annealed at 350°C, 700°C and 900°C for three hours.

The surface morphology of the powdered sample was obtained by scanning electron microscope (SEM) [JEOL/EO JSM-6390] and the chemical compositions were found out using the energy dispersive analysis of X-rays (EDAX). FTIR studies were made using Perkin-Elmer FTIR Spectro Photo Meter in the wavenumber range 400 cm⁻¹ and 4000 cm⁻¹ by KBr disc method. XRD study was carried out using XPERT-PRO model powder diffractometer (PAN analytical, Netherlands) employing Cu- Kα radiation (λ = 1.54060Å) operating at 40kV, 30mA. TG analysis was carried out by Perkin-Elmer, diamond TGA/DTA apparatus. UV study was carried out by Schimadzu 160 A...
Spectrophotometer and PL study was carried out by Horiba Jobin Yvon Flurolog 3 modular Spectrofluorimeter.

3. Results and Conclusions
From the SEM analysis it was found that the as prepared sample showed aggregates of uniform, smooth, polished solid like pattern and the sample annealed at 350°C showed aggregates of scaly layers as shown in Figure 1 (a) & (b). Figure 1 (c) shows the EDAX of as prepared sample which confirms the presence of nickel, cobalt, iron and oxygen in the nanocomposite.

![Figure 1](image1)

**Figure 1 (a) & (b) SEM images of nanocomposite in as prepared and annealed at 350°C (c) EDAX of as prepared sample.**

FTIR spectrum of the nanocomposite annealed at different temperatures showed that the precursor exhibits the basic absorption frequency bands between 4000 and 400 cm⁻¹. The prominent bands observed in the spectrum include: 3418 cm⁻¹ due to hydrogen-bonded hydroxyl groups, 1618 cm⁻¹ due to bending vibration of water molecules. The weak bands at 538 cm⁻¹ and 429 cm⁻¹ were attributed to metal oxide vibrations. It was seen that as the annealing temperature increased the bands shifts towards shorter wavelength side.

![Figure 2](image2)

**Figure 2 FTIR spectrum of the nanocomposite**

Figure 3 (a) depicts the typical TGA curve of the nickel oxide [4]. Correspondingly, the TGA curve has two sharp weight losses. The former corresponds to the evaporation of the adsorbed and intercalated water molecules and the latter is associated with the loss of water produced by
dehydroxylation of the hydroxide layers. Figure 3(b) depicts the typical TGA curves of the cobalt oxide. The first peak indicates the departure of physically sorbed water molecules; the next peaks correspond to the loss of water of constitution and decomposition. The final weight loss is due to the decomposition of Co$_3$O$_4$ into CoO [equation (1)]. The theoretical value of the weight loss in this conversion is 4% which is in close agreement with the result.

$$2\text{Co}_3\text{O}_4 \rightarrow 6\text{CoO} + \text{O}_2$$  \hspace{1cm} (1)

Figure 3(c) shows the TG curve of ferric oxide. From the curve it can be concluded that there is no transformation. Figure 3(d) shows the TG curve for the nanocomposite and it confirms the formation of composite.

![Figure 3](image)

**Figure 3** (a) Prepared sample of nickel oxide, (b) Prepared sample of cobalt oxide, (c) Prepared sample of ferric oxide and (d) Prepared sample of nickel-cobalt-ferric oxides nanocomposite.

XRD analyses of all the samples were carried out and except ferric oxide the other two showed crystalline nature. The particle size was calculated using Scherer equation (2)

$$D = \frac{k\lambda}{(\beta_{\text{hkl}})\text{measured} \times \cos\theta_{\text{hkl}}}$$  \hspace{1cm} (2)

The particle size for all the samples are in the range 10-40 nm. The XRD spectrums obtained for nano oxides are in close agreement with the one available in literature, especially the XRD of the prepared sample of nickel oxide is in close agreement with XRD of $\beta$Ni(OH)$_2$ [5]. The XRD’s of the annealed nickel oxide is matching with the JCPDS card no. #895881 and Cobalt oxide with #742120. With respect to these cards the two transition metal oxides are face centred cubic crystals. In the case of composite polycrystalline nature was observed due to the presence of ferric oxide.

![XRD Spectra](image)

**Figure 4** (a) Prepared and annealed sample of nickel oxide, (b) Prepared and annealed sample of cobalt oxide, (c) Prepared sample of ferric oxide. (d) Annealed samples of nickel-cobalt-ferric oxides nanocomposite at 350$^\circ$C, 700$^\circ$C and 900$^\circ$C for three hours.

UV analysis of the nanocomposites annealed at 350$^\circ$C and 700$^\circ$C for three hours was done. The UV spectrums of both the samples are shown in figure 5 (a) & (b). The absorbance peaks for the samples were at 221.15 nm and 301.91 nm range. The Corresponding band gap curve of the sample annealed at
350°c is shown in figure 5 (c). The composite is found to have a band gap of 4.2eV. By proper tuning these materials can be converted into a wide band gap material.

**Figure 5 (a) & (b) UV spectrum of Nickel-cobalt-ferric oxides nanocomposite annealed at 350°C & 700°C (c) Tauc plot of nanocomposite annealed at 350°C**

Photo luminescence study of the nanocomposites was carried out. From the literature it is found that the NiO nanoparticle is a good emitter of 341nm wavelength [6]. The photoluminescence studies are preferred rather than the optical absorption. This is a mechanism where the impurity on absorption of light, gives rise to the bound excited state from which it returns to its ground state abiding in accordance with the colour centre creation mechanism. The room temperature photoluminescence spectra of the nickel-cobalt-ferric oxides nanocomposite annealed at 350°C and 700°C are given in figure 6. The PL spectrums of both the samples were almost same with a slight variation in intensity. From PL analysis it could be concluded that this material can be used as light emitters in the visible range.

**Figure 6 PL spectrum (a) Nickel-cobalt-ferric oxides nanocomposite annealed at 350°C. (b) Nickel-cobalt-ferricmagnesium oxides nanocomposite annealed at 700°C.**

In the present work single salts of nano nickel oxide, cobalt oxide, ferric oxide and their composites were prepared. Using UV analysis the band gap of the nanocomposite was found to be 4.2 eV and if properly tuned the material can be made as a good wide band gap material. The photoluminescence studies showed that the composite can be used as light emitters in the visible region.

**References**

[1] Lee J, Mahendra S and Alvarez P. 2010 ACS Nano 4 3580–3590.
[2] Karimi M. A. et al 2011 Int. Nano Lett. 1 43-51.
[3] Neamtu J and Verga N. 2011 J. of Nanomat. and Biostru. 6 969-978.
[4] Fu G. et al 2009, Int. J. Electrochem. Sci. 4 1052 – 1062.
[5] Cheng M and Hwang B. 2007 Nanoscale. Res. Lett. 2 28–33.
[6] Chakrabarty S and Chatterjee K. 2009 J.Phys.Sci. 13 245-250.