

Structural and Dielectric Characterization of Nickel-Cobalt Oxide Nanocomposite

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Abstract

In the present work nickel-cobalt oxide nanocomposite were prepared using well known co-precipitation method. For comparison nano nickel oxide and nano cobalt oxide was also prepared. The samples were annealed at 500°C and characterized using SEM, TG, FTIR, XRD and UV. The dielectric studies at various frequencies of nickel-cobalt oxide nanocomposite were also done. The results showed that the capacitance of the as prepared and the annealed samples varied from milli farad to pico farad range. From this study it can be concluded that the present material by proper tuning can be used as a supercapacitor.

Keywords: Nanostructure; Nanocomposite; Chemical co-precipitation; Capping agent

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 nano particles provide unprecedented levels of new functionality for nano medicine. The magnetic elements like iron, nickel, cobalt and their chemical compounds are commonly present in magnetic nano particles [3]. After surface modification these materials provide both biocompatibility and functionality.

The transition metal oxides have various manufacturing possibilities based on their different properties such as anodic electrochromasim, excellent durability, electrochemical stability and large spin optical density. The conventional capacitors such as electrostatic and electrolytic capacitors cannot fulfil the needs of today's computer world as their utility is limited to certain specific applications. The next generation electrochemical double layer capacitor has been developed using transition metal oxide as one of the electrodes along with carbon. This capacitor is expected to deliver both the desirable power and energy densities [4].

Different methods have been reported for the synthesis of these nanoparticles and nanocomposite such as evaporation, magnetron sputtering, and sol-gel. In the present work a nanocomposite of nickel-cobalt oxide was prepared and their modification in various known properties has been studied. Among the various methods, here we have used the famous co-precipitation method for the synthesis of nickel-cobalt oxide nanocomposite.

Materials and Methods

AR grade chemicals obtained from Merck were used for the preparation of the nano particles of nickel oxide, cobalt oxide and their composites. Both the individual nano oxides and the composites were prepared by the co- precipitation method. Nickel nitrate hexahydrate,

cobalt nitrate hexahydrate, and sodium hydroxide were used as starting materials. Citric acid was used as stabilizer. Aqueous solutions of 0.1 M nickel nitrate, 0.1 M of cobalt nitrate and 1 M of sodium hydroxide were slowly mixed drop wise into a beaker containing aqueous solution of citric acid and stirred well using a magnetic stirrer for two hours. The nickel-cobalt hydroxide precipitate formed was washed several times in distilled water to free it from ions and other impurities. The wet precipitate obtained was dried at room temperature and thoroughly grounded using an agate motor to obtain the nickel-cobalt hydroxide precursor in the form of fine powder. The powder so obtained was annealed at 500°C for 3 hours in muffle furnace to obtain nanocomposite of nickel-cobalt oxide. For comparing the properties of nanocomposite with its counterpart's individual nano oxides of nickel oxide and cobalt oxide were also prepared using the same procedure.

The surface morphology of the powdered sample was obtained by Scanning Electron Microscope (SEM) [JEOL/EO JSM-6390]. FTIR studies were made using Perkin-Elmer FTIR Spectro Photo Meter in the wave number 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- K_α radiation (λ=1.54060 Å) operating at 40 kV, 30 mA. TG analysis was carried out by Perkin-Elmer, diamond TGA/DTA apparatus. UV study was carried out by Shimadzu 160 A Spectrophotometer and PL study was carried out by Horiba Jobin Yvon Flurolog 3 modular Spectrofluorometer. AC conductivity studies were done using Gain Phase Impedance Analyzer, MODEL HP, 4294 A, 40 Hz-110 MHz.

Results and Discussions

The samples were annealed at different temperatures and characterized using SEM, TG, FTIR, XRD, UV and PL. The dielectric

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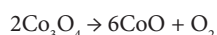
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studies at various frequencies of nickel-cobalt oxide nanocomposite were also done.

SEM analysis of nickel oxide, cobalt oxide and the nanocomposite are shown in figure 1. SEM image of nickel oxide showed aggregates of smooth solids of different size and shape. Cobalt oxide also showed an aggregation of non uniform solids. The morphology of nickel-cobalt oxide is shown in figure 1c. From SEM image it is clear that particles had undergone agglomeration.

Figure 2 depicts the typical TGA curves of the nickel oxide, cobalt oxide and nickel-cobalt oxide nanocomposite. For Nickel oxide [5] 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. In the case of cobalt oxide [6], 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 the decomposition of Co_3O_4 into CoO corresponds to the equation given below.



The TG curve for the nanocomposite shows that the material exists as a single unit at higher temperature. The weight loss due to decomposition of Co_3O_4 into CoO is not seen.

Figure 3 depicts the FTIR spectrum of the nickel oxide, cobalt oxide and nickel-cobalt oxide nanocomposite. The prominent bands observed in the spectrum for nickel oxide includes: 3421 cm^{-1} due to

hydrogen-bonded hydroxyl groups, 1631 cm^{-1} due to bending vibration of water molecules. The weak bands at 421 cm^{-1} were attributed to metal oxide vibrations. In the case of cobalt oxide, the corresponding peaks were observed at peak positions 3432 cm^{-1} , 1634 cm^{-1} and 863 cm^{-1} to 430 cm^{-1} respectively. For the nanocomposite the corresponding peak positions were at 3426 cm^{-1} , 1627 cm^{-1} and 865 cm^{-1} to 565 cm^{-1} .

Figure 4 shows the XRD spectrum of all the three samples. The phase purity of all the three samples was established by comparison of the X-ray diffraction pattern with JCPDS International data value. Nickel oxide was in matching with JCPDS Card no #78-0423, Cobalt oxide with #74-2120 and the nanocomposite was matched with #73-1704. From the JCPDS Card both nickel oxide and the nanocomposite had cubic system with FCC lattice. Cobalt oxide also had cubic system. The lattice constants for all the three samples were calculated and were in agreement with JCPDS Card values. From JCPDS for nickel oxide $a=4.179$, for cobalt oxide $a=8.084$ and for nickel-cobalt oxide, $a=9.387$.

From the XRD analyses the particle size were calculated using Scherrer equation (1).

$$D = k\lambda / (\beta_{hkl})_{\text{measured}} * \cos\theta_{hkl} \quad (1)$$

Here, D is the average crystallite size normal to the reflecting planes, k – is the shape factor which lies between 0.95 and 1.15 depending on the shape of the grains ($k=1$ for spherical crystallites), λ -is the wavelength of x-ray used and $(\beta_{hkl})_{\text{measured}}$ is the FWHM of the diffraction line in radians and θ_{hkl} is the Bragg angle corresponding to the diffraction line arising from the planes designated by Miller indices (hkl). From the crystallite size calculation the average size of nickel

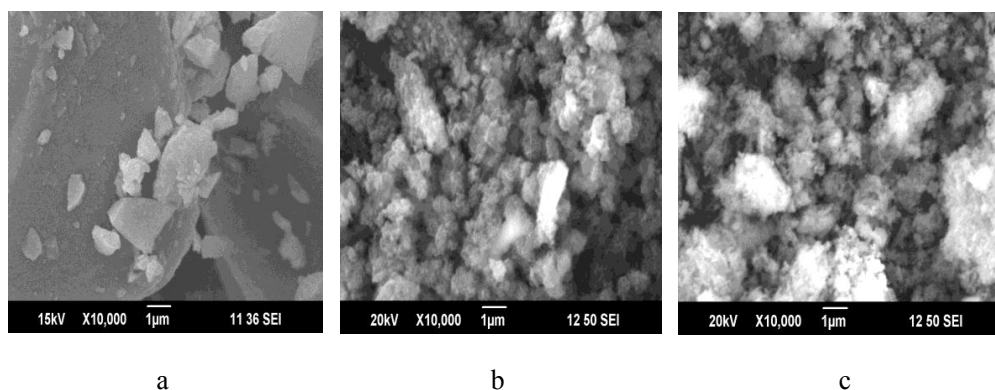


Figure 1: SEM image of nickel oxide (a), cobalt oxide (b) and nickel-cobalt oxide (c).

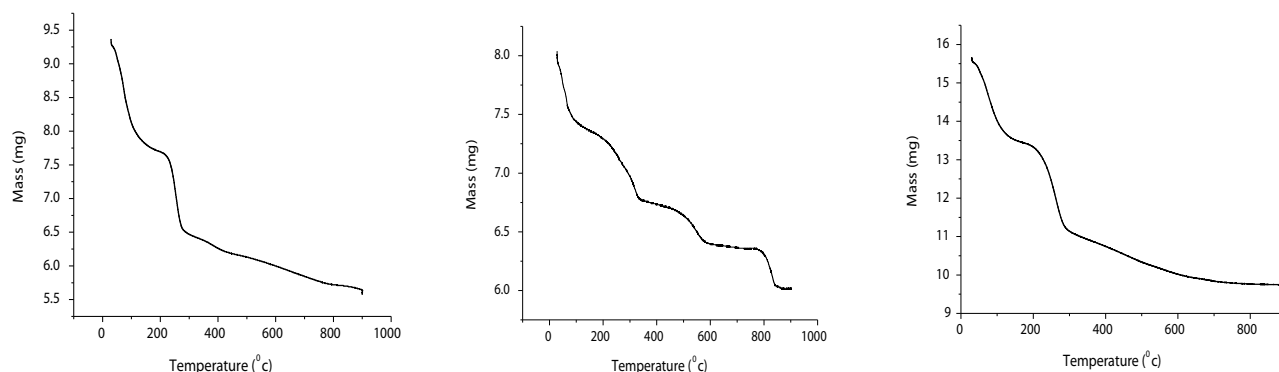


Figure 2: TG Curve of nickel oxide (a), cobalt oxide (b) and nickel-cobalt oxide (c).

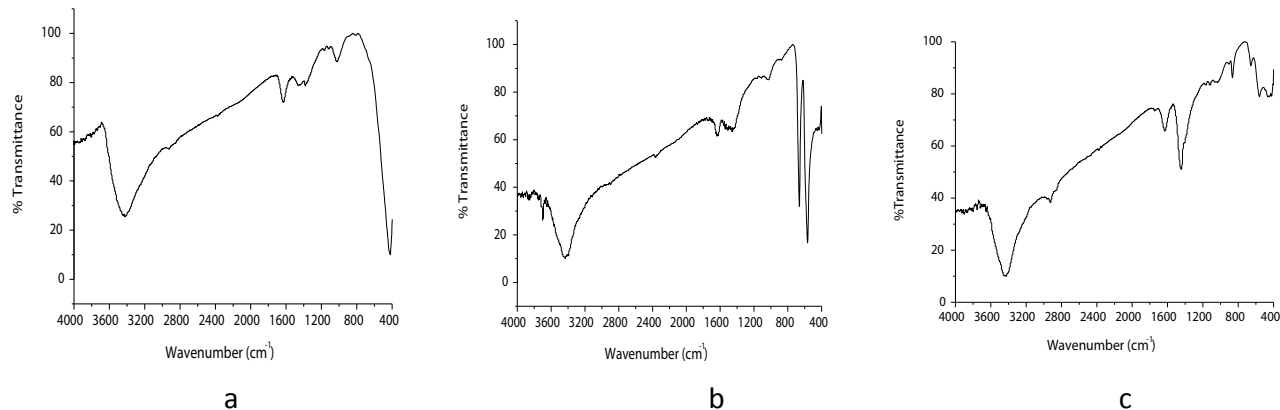


Figure 3: FTIR spectrum of nickel oxide (a), cobalt oxide (b) and nickel-cobalt oxide (c).

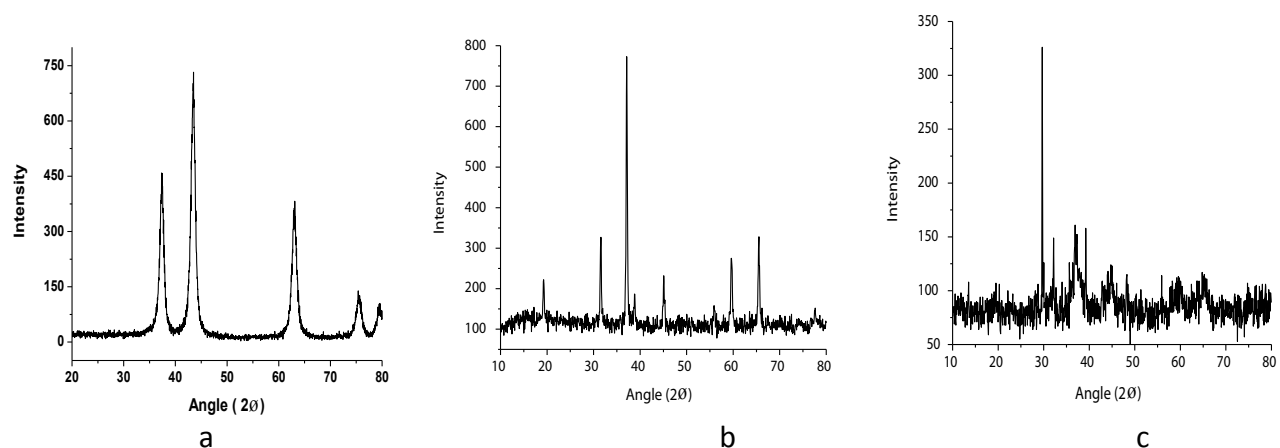


Figure 4: XRD spectra of nickel oxide (a), cobalt oxide (b) and nickel-cobalt oxide (c).

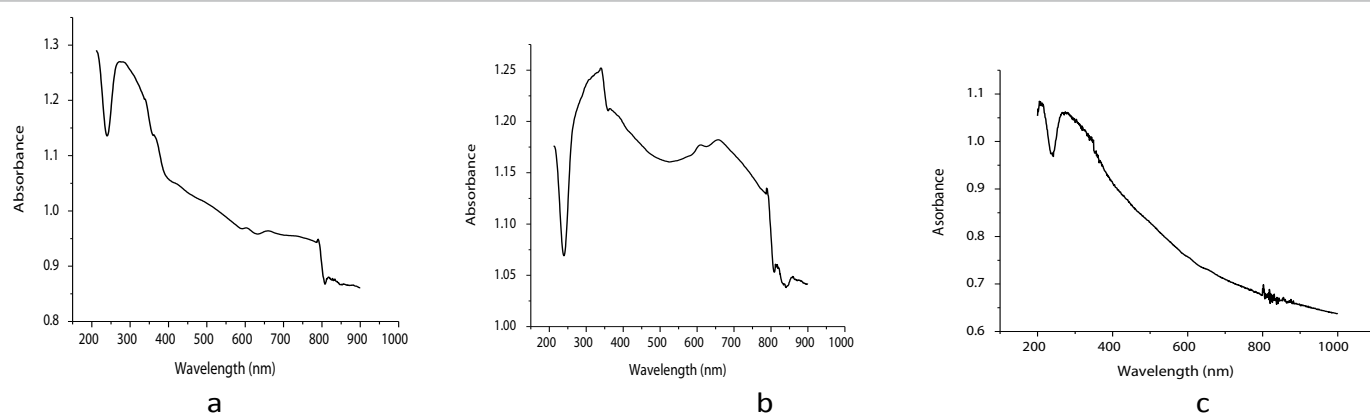
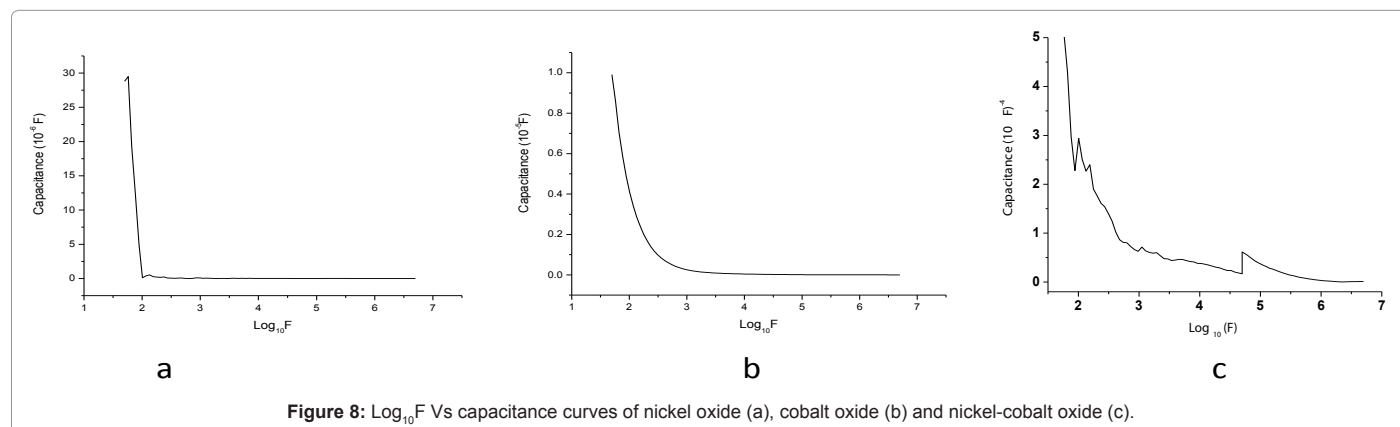
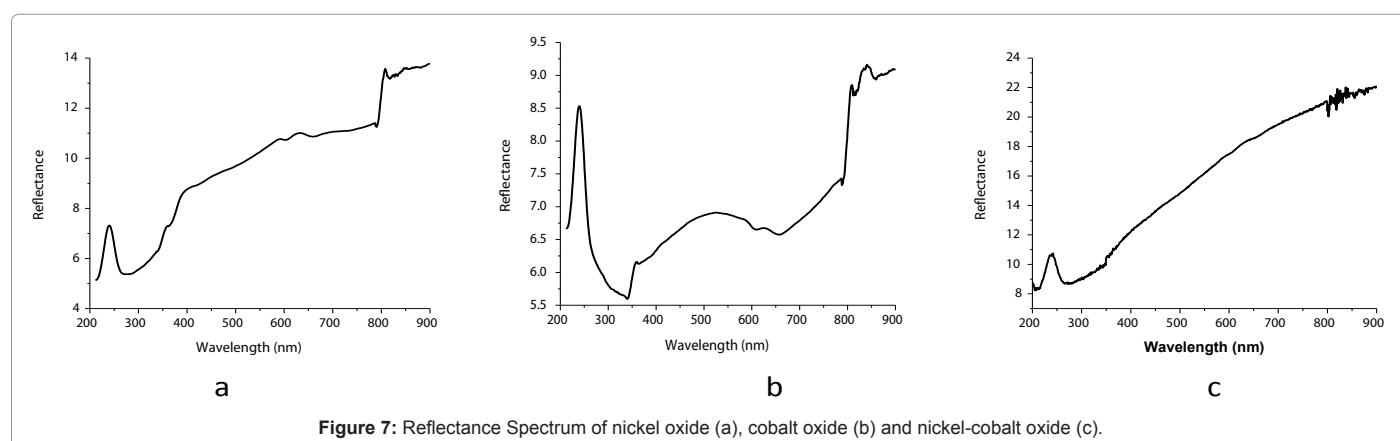
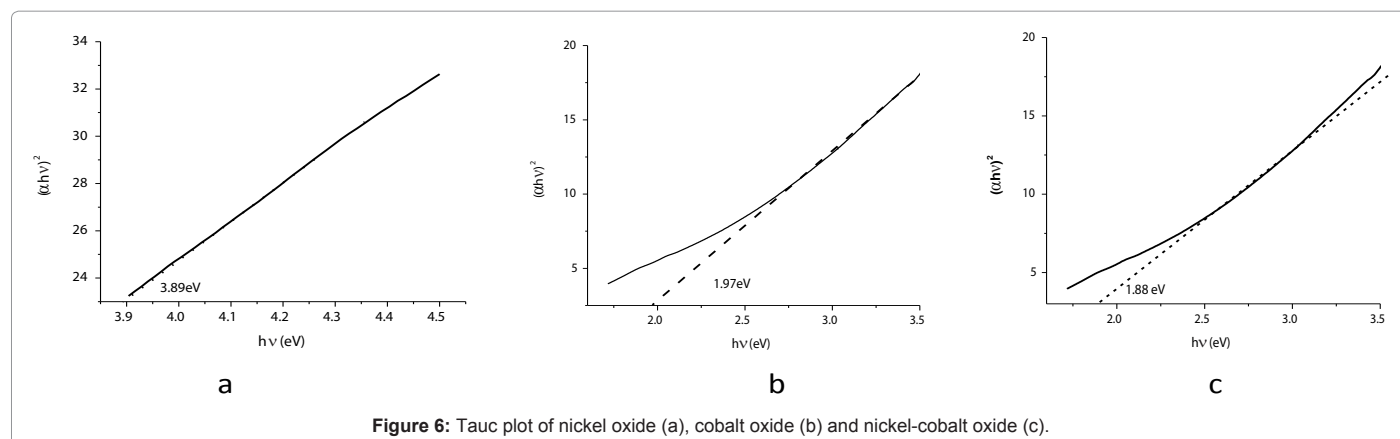


Figure 5: UV Spectrum of nickel oxide (a), cobalt oxide (b) and nickel-cobalt oxide (c).

oxide was 24 nm, cobalt oxide was 20 nm and that of nickel – cobalt oxide was 23 nm respectively.

UV analysis of the oxides and nanocomposite were done. The UV spectrum of nickel oxide, cobalt oxide and the nanocomposite in the wavelength range 200nm - 900nm are shown in the figure 5. The energy band of the material is related to the absorption coefficient α by the Tauc relation, $\alpha = A(h\nu - E_g)^n$, where A is a constant, $h\nu$ is the photon

energy ($\nu = c/\lambda$), E_g is the band gap and n is either 2 for an indirect transition or $1/2$ for a direct transition. The $(\alpha h\nu)^2$ vs $h\nu$ for all the three samples is plotted and are shown in figure 6. The extrapolation of the linear portion to $h\nu$ axis gave value of energy gap nickel oxide as 3.89 eV, cobalt oxide as 1.97 eV and the nanocomposite as 1.88 eV. From the value of the band gap of the composite it could be concluded that material can be suitably used to get wide band gap materials.



The reflectance spectrum of all the three samples was also studied. The reflectance spectra of all the three samples are shown in figure 7 respectively.

The AC conductivity studies of the samples were carried out. For this the samples annealed at 500°C for three hours was pelletized in presence of PVA which was then sintered at 1200°C for three hours and then frequency versus capacitance measurement was carried out. The results showed that the composite in as prepared form had a capacitance ranging from millifarad to picofarad in the frequency range 100Hz to 13MHz. From the result it is understood that, if properly tuned this composite can be used as a super capacitor. Conductivity also showed the corresponding variation. Figure 8 shows the frequency

versus capacitance plot for annealed samples in the frequency range 100 Hz to 5MHz.

Conclusion

From the crystallite size calculation using XRD spectrums, the average size of nickel oxide was 24 nm, cobalt oxide was 20 nm and that of nickel-cobalt oxide was 23 nm respectively. From the value of the band gap of the composite it could be concluded that material can be suitably used to get wide band gap materials. From the AC conductivity study it can be concluded that the present material by proper tuning can be used as a super capacitor. Also nickel-cobalt oxide nanocomposite may be a very good magnetic nanocomposite since nickel and cobalt

in their individual state shows magnetic properties. Further studies on these materials and similar nanocomposites are needed.

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