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# Synthesis and Electro Chemical Application of $Ni_xCo_{(1-X)}O$ Nanocomposites

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## ABSTRACT

The electrode material of $Ni_xCo_{(1-x)}O$ nanocomposites were prepared by using microwave assisted solvo-thermal method .The $Ni_xCo_{(1-x)}O$ composites were prepared for different ratios as follows (X=0, 0.25, 0.5, 0.75, 1.0).The prepared samples were characterized by Powder X-ray Diffraction spectra (P-XRD),Fourier transform infrared spectroscopy (FT-IR),UV-Visible spectroscopy and Scanning electron microscope (SEM). The electrical conductivity of pelletised samples were measured for different temperatures ranging from 40-150oC at various frequencies viz.,100Hz, 1kHz,10kHz,100kHz and 1MHz.

**Key words :** $Ni_xCo_{(1-x)}O$ nanocomposites, Scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FT-IR)

## 1 INTRODUCTION

The transition metal compounds like Nickel, Copper, Manganese and Cobalt etc are used as electrode materials in several of electro chemical process. Metal oxides nanoparticles are participate in different fields such as physical, chemical, electronic and biology, this is because of their electrical, optical and photo-electrochemical properties. The properties of the metal nanoparticles are dependence on their sizes, shapes and composition [1].Metal oxide nanoparticles have variety of applications by their own. So many research works are going on to improve their uses, application and properties; From earlier research we identify that bi-metallic compounds are one of the types which have higher electrode application than metal oxides nanoparticles. The bi-metallic compounds are used as industrial catalyst, electrode materials etc. For example: Used as catalyst in Fischer-Tropsch process [2].The mixed oxide of transition metal compounds have a mutual interactions lead to the complex structure formation. Various transitional metal oxides including W, V, Mg, Co and Ni has been studied extensively as hosts for proton insertion ($Li^+$, $Na^+$, and $H^+$)[3]. The catalytic properties of the mixed oxide compounds are higher than that of the simple metal oxide compounds [4].Nickel is used as a dopant with various transition metals like Zn, Co, Cu etc. to produce better optical, electrical and catalytic materials [5].And the Nickel dispersed or doped compounds are many hydro related reactions, such as the hydrogenation, hydrogenolysis, and hydrotreating reactions [6].Modified electrode prepared with cobalt and its complex has intensely been reported for the detection and sensing of several analysts such as glucose, cysteine, hydrogen peroxide, hydroquinone, thiol-based nerve agents, epinephrine and nitrite .The nitrogen is a important element in environment and the detection of nitrogen can be detected by the electrode made by Co/ $Co_3O$ nanoparticles [7].The compounds formed by the Ni and cobalt oxide crystals are used as transistor, resistor and capacitors this is because of the electrical properties of the Ni and Cobalt oxide nanocrystalline compounds [8]. The Nickel and cobalt nano powders are used in batteries, hard alloy, magnetic, catalyst and electrical materials etc [9, 10]. Kulkarni[ 58] has reviewed the results reported on the doped II-VI compounds semiconductor nano particles. It has been demonstrated that the Pl yield can be greatly enhanced by forming a two compound nanocomposites. A similar approach with NiO-CoO is expected to bring fruitful results. In our present study for the first time, we have reported the novel microwave assisted solvothermal method for the preparation of $Ni_xCo_{(1-x)}O$ nanocomposites .And the results are discussed below.

## 2 MATERIALS AND METHODS

### 2.1 Materials Used

Analytical reagent (AR) grade Nickel chloride, Cobalt chloride and urea along with ethylene glycol were used for the preparation of NiO, $Ni_{0.75}Co_{0.25}O$, $Ni_{0.5}Co_{0.5}O$, $Ni_{0.25}Co_{0.75}O$, $Co_3O_4$, nano powders. Double distilled water and AR grade acetone were used for washing purpose.

$$CoCI_2.6H_2O + NH_2CONH_2 \longrightarrow Co(CI_2)(NH_2CONH_2) \longrightarrow CO_3O_4 \quad (1)$$

$$NiCI_2.6H_2O + NH_2CONH_2 \longrightarrow Ni(CI_2)(NH_2CONH_2) \longrightarrow NiO \quad (2)$$

$$CoCI_2.6H_2O + NiCI_2 6H_2O \xrightarrow{NH_2CONH_2} Ni_xCo_{(1-x)}O \quad (3)$$

To prepare NiO nanopowder, Nickel chloride and urea in 1: 3 molar ratios were mixed (as per the required proportionality) and dissolved in 100 ml of ethylene glycol. The solution was kept in a domestic microwave oven operated at a frequency of 2.45 GHz and 840 W powers. Microwave irradiation was carried out until the solvent evaporated completely. The colloidal precipitate obtained was cooled and washed several times with distilled water and then with

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acetone to remove if any organic impurities are present. The sample was then dried in open atmosphere and collected as the yield. The same procedure was followed for the other composite samples.

The required amount of the substance (A) was estimated by using the formula

$$A = \frac{M x X x V}{1000} (in\ gram\ units)$$

Where, M is the molecular weight of the (particular) substance, X is the concentration in molar units (1 or 3 or fraction as per the requirement). V is the required volume of the solvent (100 ml in the present work).

The reactions were found to be fast (within 18 min) and highly yielding with microwave. The yield percentage was calculated using the relation

$$Yield\ \% \frac{\text{Product mass}}{\text{Sum of masses of reactants}}$$

## 3 Result and Discussions

### 3.1 Powder X-ray Diffraction Spectra (P-XRD)

PXRD patterns of the prepared sample are shown in Fig. 1. The existence of sharp peak in the PXRD pattern of NiO indicates the presence of big crystallites embedded in the nano phase of the prepared samples. Moreover, instead of forming the mixed system, Co entered as a dopant in Ni lattice. Addition of Co reduces the intensity of NiO peaks. Also Co addition increases the broadening of peaks which in turn reduces the size of Nano composites, which makes strong quantum confinement. The size of the synthesized samples were calculated using the scherer formula

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

Where, D is the mean dimension of the crystallites, β the broadening at half band width, λ the wavelength, K is a constant approximately equal to unity. The calculated grain size of the samples are given in Table 1.

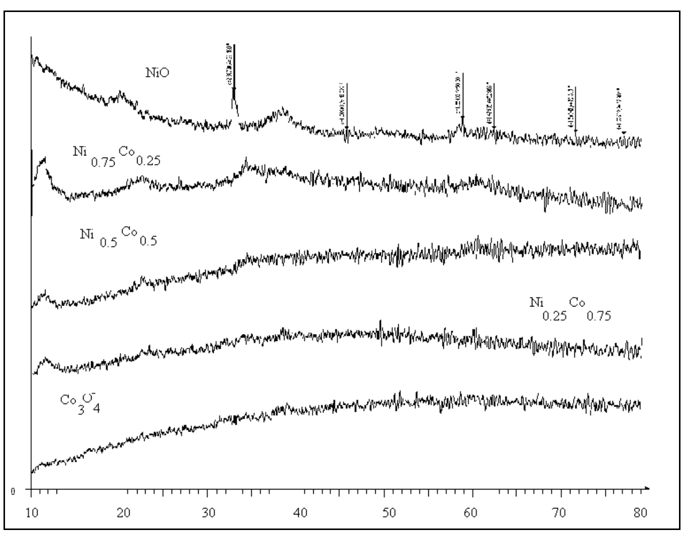



Fig .1. XRD image for the $Ni_x Co_{(1-x)}O$ composites

| S. No | Sample Name | Grain Size (nm) |
|---|---|---|
| 1 | NiO | 2.5 |
| 2. | $Ni_{0.75}Co_{0.25}O$ | 2.6 |
| 3. | $Ni_{0.5}Co_{0.5}O$ | 2.72 |
| 4 | $Ni_{0.25}Co_{0.75}O$ | 2.92 |
| 5 | CoO | 2.8 |

### 3.2 Fourier Transforms Infrared Spectroscopy (FT-IR)

The FTIR spectra of $Ni_x$ Co $_{(1-x)}$ O compounds for different ratios are given in Fig. 2.The following FT-IR data will explain that the doping of different ratio of Cobalt to the Nickel oxide matrix. The FTIR is taken for the sample at mid FTIR region (4000 $Cm^{-1}$ to 400 $Cm^{-1}$).The Broad absorption band in the region around 3450$Cm^{-1}$ is due to the presence of co-ordinated water. The absorption band at 1680 $Cm^{-1}$ to 1200 $Cm^{-1}$ is corresponding to carboxylate ion. The absorption band around 650-700 $Cm^{-1}$ corresponds to bending modes of vibration of cobalt and the absorption band around 550 $Cm^{-1}$ is corresponding to bending modes of vibration of Nickel oxides.

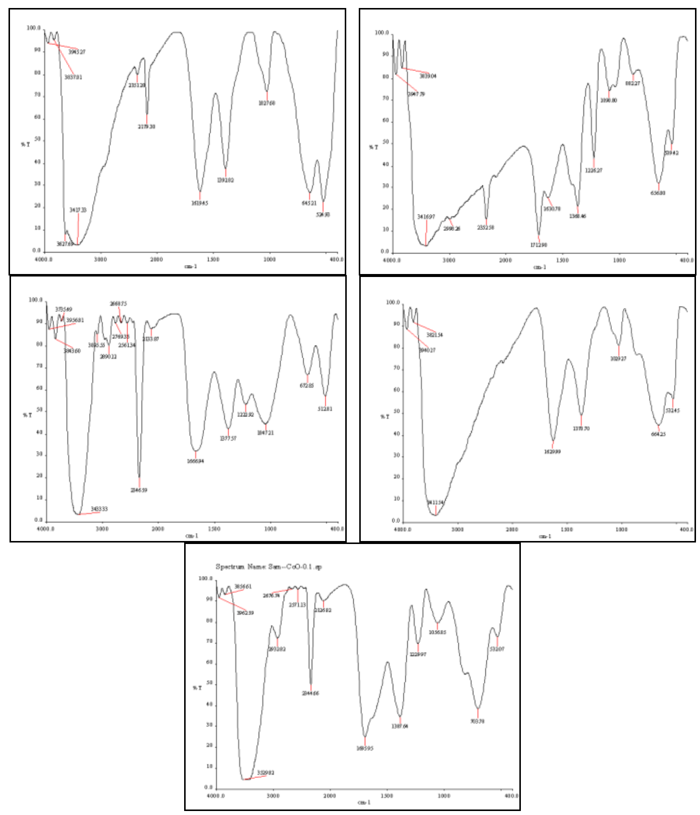


Fig .2. FTIR spectrum for a) NiO b) $Ni_{0.75}Co_{0.25}O$ c) $Ni_{0.5}Co_{0.5}O$ d) $Ni_{0.25}Co_{0.75}O$ e) $Co_3O_4$ nano composites

### 3.3 UV-Visible Spectroscopy

UV-Vis absorption spectra were recorded for the entire 5 synthesized sample. The recorded spectra are shown in fig. 3. The substitution of Co in Ni sites continuously leads to red shift and results in very low band gap energy. The broadening of peak nearly at 275-422 nm is conforming the doping of cobalt oxide in nickel oxide lattice

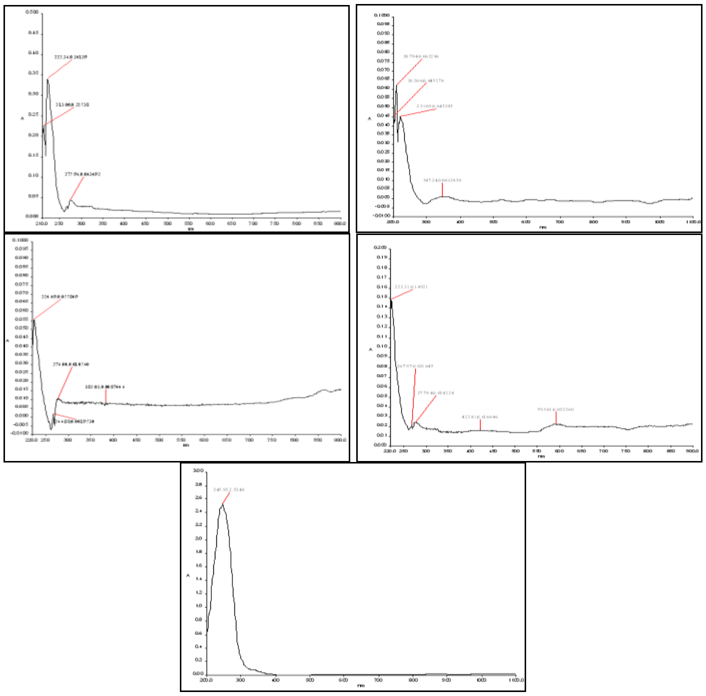
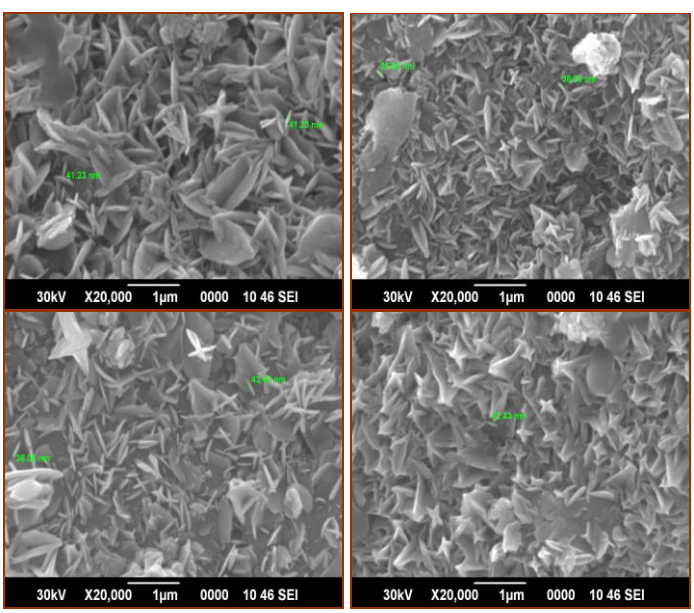


Fig. 3. UV-Visible spectrum for a) NiO b) $Ni_{0.75}Co_{0.25}O$ c) $Ni_{0.5}Co_{0.5}O$ d) $Ni_{0.25}Co_{0.75}O$ e) $Co_3O_4$ nano composites.

### 3.4 Scanning Electron Microscope (SEM)

The SEM image for the $Ni_xCo_{(1-x)}$ is shown in fig.4 . particle size from SEM studies were higher than the particle size calculated from XRD measurements. This is due to agglomeration of particles. Also all the synthesized samples have flower like morphology.



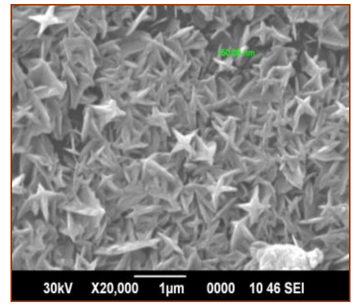




Fig .4. The SEM image for $Ni_x Co_{(1-x)} O$ compounds

### 3.5 Dielectric and Conductivity Measurements

The following Fig.5-7 shows the temperature dependence of dielectric properties of $Ni_x Co_{(1-x)} O$ composites. The $\varepsilon_r$ obtained for the end members (NiO- and CoO-) are very small when compared with bulk crystals of the same. The electrical parameters, viz. $\varepsilon_r$, tanδ and $\sigma_{ac}$ observed in the present study are increased with increase in temperature for all the five $Ni_x Co_{(1-x)} O$ nano crystals considered in the present study. Decrease in dielectric properties in the prepared samples can be explained as follows: Nano particles lie between the infinite solid state and molecules. When one reduces the dimensions of the solids to a nanometer size, the size of the exciton becomes comparable or even larger than the particle. This results in splitting of the energy bands into discrete quantized levels, and the band gap starts opening. The electrical resistivity of nano crystalline material is higher than that of both conventional coarse grained polycrystalline material and alloys. The magnitude of electrical resistivity and hence electrical conductivity in composites can be altered by altering the size.

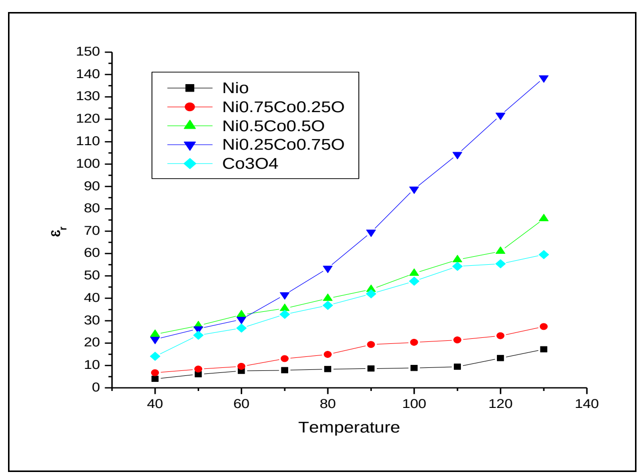



Fig. 5 .Dielectric constant for various Temperature and constant frequency 1Kz for all Composites

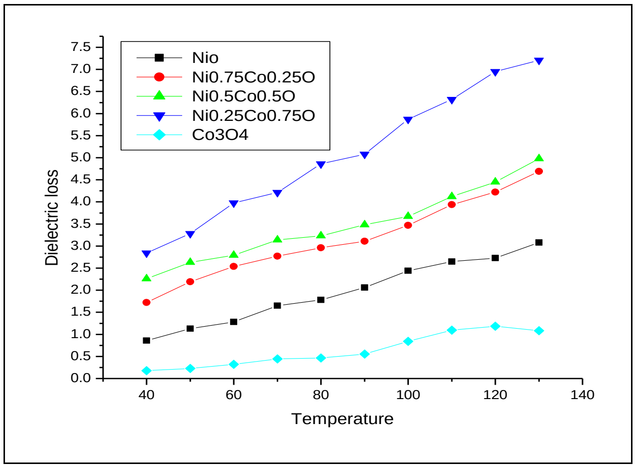



Fig.6. Dielectric Loss for various Temperature and constant frequency 1Kz for all Composites

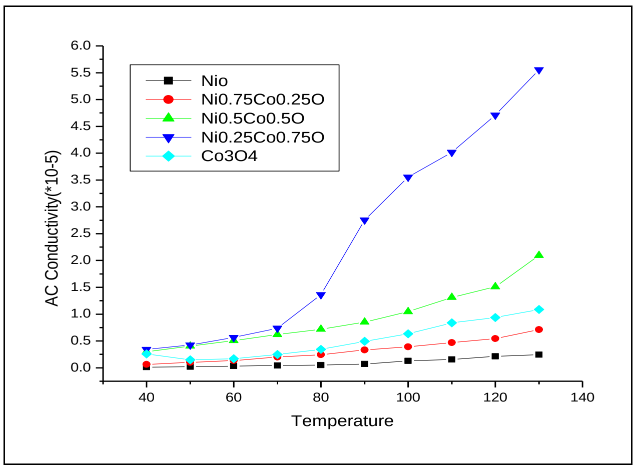



Fig. 7. AC conductivity for various Temperature and constant frequency 1Kz for all Composites

## CONCLUSION

The results obtained in the present syudy indicate the possibility to prepare $Ni_x$ $Co_{(1-x)}$ O nano crystals by using simple microwave assisted solovothermal method. Also the method is found to be fast and highly yielding. The unit cell volume expansion is found to increasing with the increasing compositions, reveals the formation of nano composites. The results obtained in the present electrical studies indicate the occurrence of nano confined states which may substantially contribute it's electro-chemical applications.

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