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RESEARCH ARTICLE

PREPARATION AND CHARACTERIZATION OF Cu DOPED NiO NANOPARTICLES: EFFECT OF pH

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Abstract

Abstract-In this paper we discussed a facile chemical co-precipitation method for preparing water soluble Cu doped NiO nanoparticles and studied the pH influence on the structural, morphological, compositional and photo luminescence properties using X-ray diffraction (XRD), scanning electron microscopy (SEM), energy dispersive analysis of X-rays (EDAX) and PL Fluorimeter. Broadened XRD peaks confirmed the formation of nanosized NiO: Cu nanoparticles with face centered cubic (FCC) structure at different pH values. SEM and EDAX analysis shows surface morphology and effective elemental composition of prepared nanoparticles. PL peaks of Cu doped NiO nanoparticles at different pH values ware observed around 434 nm and an increase in PL intensity was observed with increase in pH value. .

INTRODUCTION

Nano-sized materials with uniform morphology have received great interest due to their importance in basic scientific research and potential knowledge applications. The transition metal oxides are extremely popular technological materials for use in electronic and photonic devices. Excellent electronic, optical and catalytic properties recommend NiO nano-crystalline powder for gas sensing units, pollutant clean-up catalysts, alkaline battery cathodes, dyesensitized solar cells, lithium ion batteries, photocatalysts, electrochemical capacitors, electrochromic and optical coatings [1 - 4]. Many researchers begin to investigate doping NiO with metal ions like Al, Si, V, Y, Zr, Nb, Ag etc to enhance optical properties and durability. Due to their small size, nanoparticles exhibit novel material properties that are significantly different from those of their bulk counterparts. Several techniques, such as hydrothermal [5], sol–gel [6], solid-state reaction [7], electrochemistry [8], microemulsions [9], hard template [10], spray pyrolysis [11], reverse micellar route [12] and precipitation [13] methods, have been developed to synthesize NiO nanostructures.

In this study, a simple and cost effective chemical co-precipitation method was used to prepare copper doped NiO nanoparticles at different pH values. The influence of pH on structure, surface morphology, composition and photoluminescence properties were investigated in detail.

EXPERIMENTAL AND CHARACTERIZATION DETAILS

All chemicals were of analytical reagent grade and were used without further purification. Cu doped NiO nanoparticles were prepared by chemical precipitation method and the reactants were $NiCl_2$. 6H $_2$O and $CuSO_4$. 5H $_2$O. Ultrapure de-ionized water was used as the reaction medium in all the synthesis steps. In a typical synthesis, $NiCl_2$. $6H_2O$ (96 at.%) and $CuSO_4$. $5H_2O$ (4 at.%) each in 100 ml were dissolved in ultrapure de-ionized water one after another and stirred for 30 minutes, NaOH solution was drop wisely added to the solution to adjust the pH value 8. Stirring was continued for four hours to get fine precipitation. The obtained precipitate was washed with de-ionized water for several times. Finally, the powders were vacuum dried for 3 hours at 80^0C to obtain Cu doped NiO nanoparticles. The above procedure is reiterated by varying the pH = 9, 10 and 11.

The X-ray diffraction patterns of the samples were collected on a Rigaku D X-ray diffractometer with the Cu-Kα radiation (λ=1.5406A°). Morphology and elemental composition of the prepared samples were analyzed through EDAX using Oxford Inca Penta FeTX3 EDS instrument attached to Carl Zeiss EVO MA 15 scanning electron microscope. Photoluminescence spectra were recorded in the wavelength range of 400–600 nm using PTI (Photon Technology International) Fluorimeter with a Xe-arc lamp of power 60 W and an excitation wavelength of 320 nm was used.

RESULTS AND DISCUSSION

Structural analysis

The XRD patterns for the prepared Cu doped nanoparticles at different pH values are shown in Fig. 1.

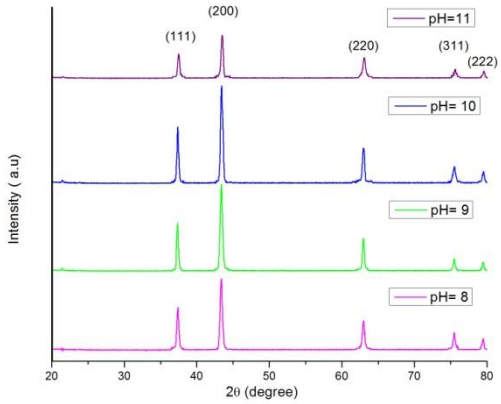

Figure 1: XRD patterns of Cu doped NiO nanoparticles synthesized at different pH conditions (pH - 8, 9,10, 11)

From the figure it is obvious that the peaks are indexed as (111), (200), (220), (311) and (222) planes at 2θ values 37.39°, 43.45°, 62.98°, 75.5° and 79.49° that correspond to face centered cubic structure of NiO nanoparticles which are in consistent with the JCPDS (No. 47-1049) data. The average sizes of Cu doped NiO nanoparticles prepared at different pH values (8, 9, 10 and 11) were of 72, 66, 52 and 58 nm, respectively. The decrease in average particle size is because of the nucleation rate increases on increasing the pH of the reaction mixture.

Morphological and Compositional analysis

Fig. 2 (a), (b), (c) and (d) shows the SEM images of Cu (4.at %) doped NiO nanoparticles at pH = 8, 9, 10 and 11 respectively. The EDAX profile of Cu (4.at %) doped NiO nanoparticles at pH= 10 is shown in Fig. 2(c).

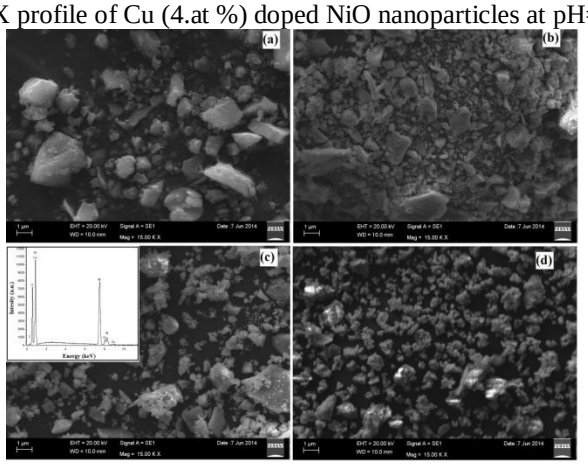

Figure 2: SEM image of NiO: Cu nanoparticles prepared at (a) pH = 8, (b) pH = 9, (c) pH = 10 (Representative EDAX spectra) and (d) pH = 11.

It is evident from the EDAX Spectra that no other elemental peaks other than Ni, O and Cu are observed except that only the elemental carbon arises due to adhesion of carbon tape on to the stud used in the analysis. The results confirm the effective doping of Cu into NiO and the SEM images show slight decrement in agglomeration of particles with increasing pH value.

- **Photo Luminescence**

Fig. 3 shows the PL spectra of Cu doped NiO nanoparticles excited at 320 nm. As seen in Fig. 3, all the samples showed PL emission band at 2.86 eV (434 nm) resulting from the surface oxygen vacancies of NiO samples [14].

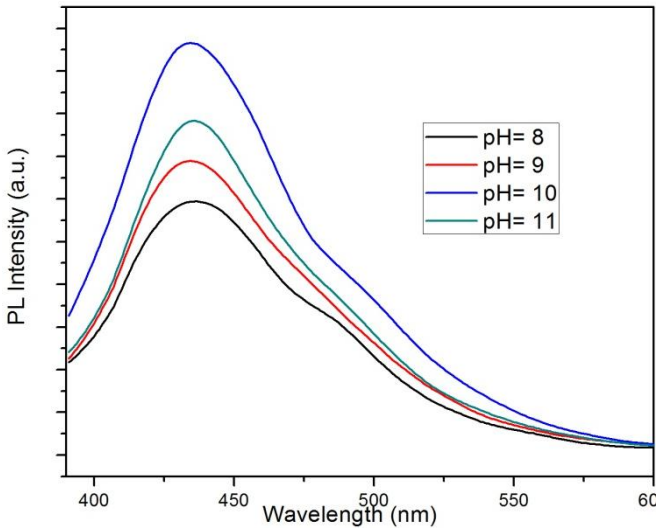

Figure 3: PL spectra of NiO: Cu nanoparticles at pH= 8, 9, 10 and 11.

PL spectrum with a strong band centered at 434 nm corresponding to blue emissions confirmed the presence Cu doped NiO nanoparticles and no impurity peaks were observed [15]. PL intensity is enhanced with increasing pH value (8, 9, 10) and decreased for pH = 11, because of the increment in the agglomeration and hence particle size.

CONCLUSIONS

Cu doped NiO nanoparticles were prepared by chemical precipitation method at room temperature. Size of the NiO: Cu nanoparticles can be tunable by simply changing pH of the reaction solutions. The influence of pH on Structural, compositional, morphological and Luminescence properties of the prepared samples was studied. XRD studies confirmed the fcc structure of the prepared samples. SEM images show the slight agglomeration of Cu doped NiO nanoparticles and EDAX spectra showed the effective doping of copper into NiO. PL peaks of the samples were observed at 434 nm and an enhancement in the PL intensity was observed with increasing pH and prominent intensity was observed at pH = 10.

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