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# STRUCTURAL, OPTICAL AND SEM STUDIES OF NICKEL DOPED TINOXIDE ($SnO_2$) NANOPARTICLES SYNTHESIZED Via CO-PRECIPITATION METHODS

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**Abstract:** Pure and Ni–doped Tin Oxide ($SnO_2$) nanoparticles were synthesized by simple co-precipitation method. The structural, morphological and optical properties of these nanoparticles were investigated by using X-ray diffraction, UV-Vis spectroscopy and SEM Studies. The X-ray diffraction revealed that all samples are pure tetragonal rutile- structure with crystalline space ($P4_2/mnm$) and the nickel doping did not change the tetragonal structure of tin oxide. The optical band energies of pure and Ni doped $SnO_2$ nanoparticle were evaluated from UV-Vis-NIR studied and found to be ~2.16 eV and ~1.75 eV respectively. The surface morphology of the pure and Ni doped $SnO_2$ nanoparticles studied by SEM.

**Key Words:** Tin Oxide, X-ray Diffraction, UV-Absorption, SEM Studies

## 1. INTRODUCTION

Tin oxide is a widely used, a stable and intensively studied n-type semiconductor with many potential ap-plications in various nanodevices. The success in many of its applications depends on crystalline SnO2 with a uniform nano-size pore structure [1]. It is a well-known fact that materials at the nanoscale behave differently than their bulk counterparts because of large surface to volume ratio. Efforts toward the development of tin oxide nanomaterials with high sensitivity, excellent selectivity, quick response, and recovery behavior has been done over the years by using different synthesis techniques and various dopants. Tin oxide is used widely to control air pollution and to detect toxic or smelling gases at low levels in the air and in the field of domestic and industrial applications [2]. Tin oxide ($SnO_2$) is one of the most intriguing materials to be investigated today, This is because tin dioxide is a well-known n-type semiconductor with a wide band gap of 3.6-3.8 eV [3-5], and for its potential application in transparent conductive electrode for solar cells a gas sensing material for gas sensors devices, transparent conducting electrodes, photochemical and photoconductive devices in liquid crystal display, gas discharge display, lithium-ion batteries, etc., [6-12]. Co-precipitation [13] is a suitable chemical method in a nanoparticles synthesis because it does not require high pressure and temperature and impure materials are eliminated by filtration and washing. In present study tin oxide nanoparticles were synthesized by co-precipitation method and ultrasonic waves irradiation was used to homogenize nanoparticles.

## 2. EXPERIMENTAL PROCEDURE

For preparation of Ni doped $SnO_2$, the appropriate amounts (1M of Ni ($Ni(NO_3)2.9H_2O$ and 99M of Tin ($SnCl_2.2H_2O$) of two precursors of Tin chloride (Tin ($SnCl_2.2H_2O$) and Nickel nitrate (Ni ($Ni(NO_3)2.9H_2O$) were dissolved in de-ionized water, stirred for 2 hours hot using plate with a magnetic stirrer. Then, Ammonium Hydroxide ($NH_4OH$) was added into the solution (drop by drop), with stirring, until the white precipitates were obtained. After 30 minutes of stirring the resultant mixtures were rinsed, several times, with de-ionized water to remove chlorine and other ionic impurities, which may formed during the synthesis process. Then, washed precipitates were dried in air at $40^0$C for 20 hours followed by natural cooling up to the room temperature and then final powder products were collected carefully. These precursors were ground in an agate mortar pestle for 30 minutes to obtain fine powder. These powders placed in a $Al_2O_3$ crucible for sintering in furnace at 450 °C for 5 hrs followed by furnace cooling at room temperature. To get the annealed powdered sample of Nickel doped $SnO_2$.

## 3. RESULTS AND DISCUSSION

Fig.1(a) shows the XRD patterns of pure and nickel doped tin oxide nanoparticles synthesized by co-precipitation method.

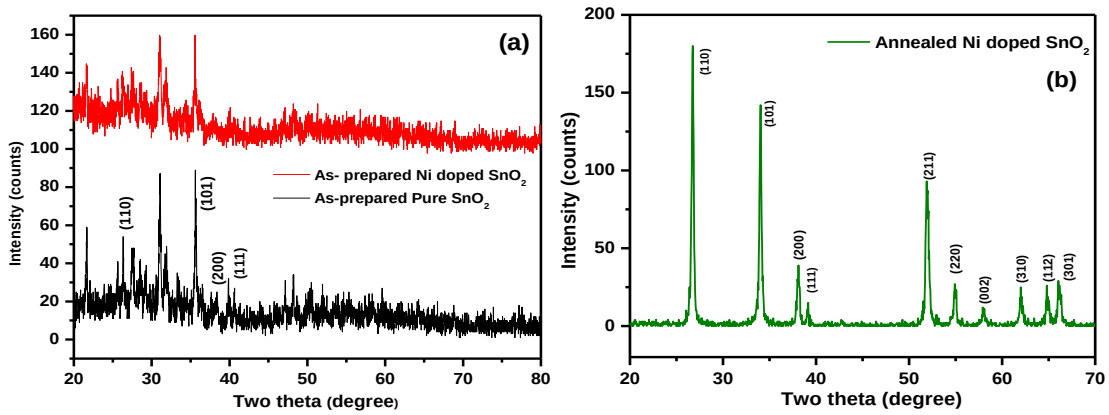



Fig.1 Shows the XRD patterns of (a) as-prepared pure $SnO_2$ and Ni doped $SnO_2$, (b) shows the Ni doped $SnO_2$ annealed for $400^0$C for 2h sample

Fig.1(b) shows the XRD spectrum of Ni doped $SnO_2$ nanoparticles annealed at 400°C for 2 hr which showing well defined characteristics peaks. The observed values of 2θ and d-spacing's are in good agreement with the standard values of the JCPDS file no. 21-1250. The diffraction angle 2θ with corresponding diffraction planes (h k l) was observed at 26.81° (1 1 0), 34.04° (1 0 1), 38.07° (2 0 0), 39.13° (1 1 1), 51.93° (2 1 1), 54.90° (2 2 0), 57.98° (0 0 2), 62.01° (3 1 0), 64.82° (1 1 2) and 66.18° (3 0 1) correspond to tetragonal $SnO_2$ rutile structure with crystalline space group ($P4_2/mnm$). Ni doped $SnO_2$ samples structure also a comparable similar pattern of pure-$SnO_2$ was observed except that the diffraction peak shifts slightly to the lower angle. Ni doping does not affect the original tetragonal unit cell of $SnO_2$. The diffraction planes are broadened. The peak position also shifts towards lower angle indicating the expansion of lattice. The crystalline size of the Ni doped $SnO_2$ samples were calculated by the Debye-Scherrer's equation which is given by $d = k\lambda/\beta \cos\theta$, where 'k' is the shape factor, ' λ' is the wavelength of x-rays used, β is the full width half maximum of the peak and θ is the glancing angle. The annealed sample of Ni doped $SnO_2$ determined the Crystalline size is about 23 nm of lattice constant a=4.738 and c=3.188 (Tetragonal).

**UV-VISIBLE SPECTROSCOPY STUDIES: Ni doped $SnO_2$**

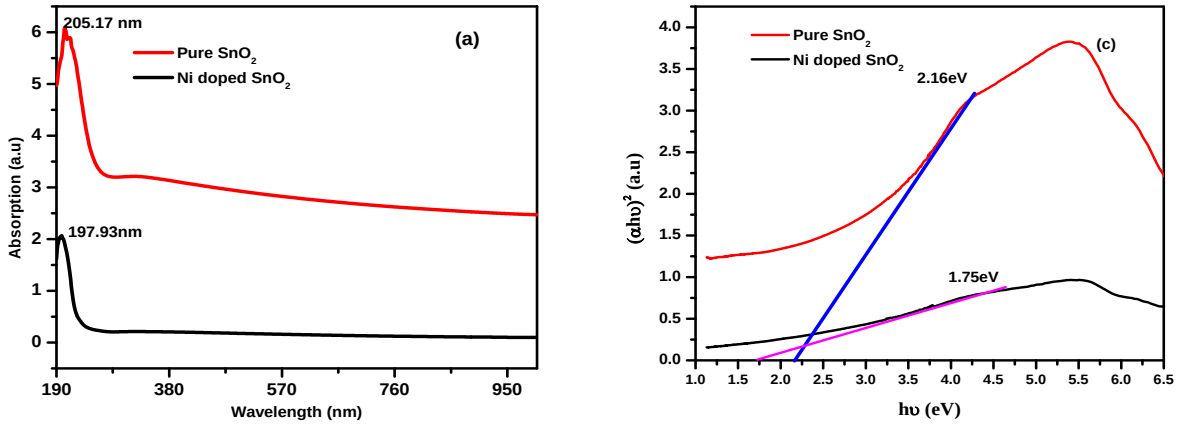
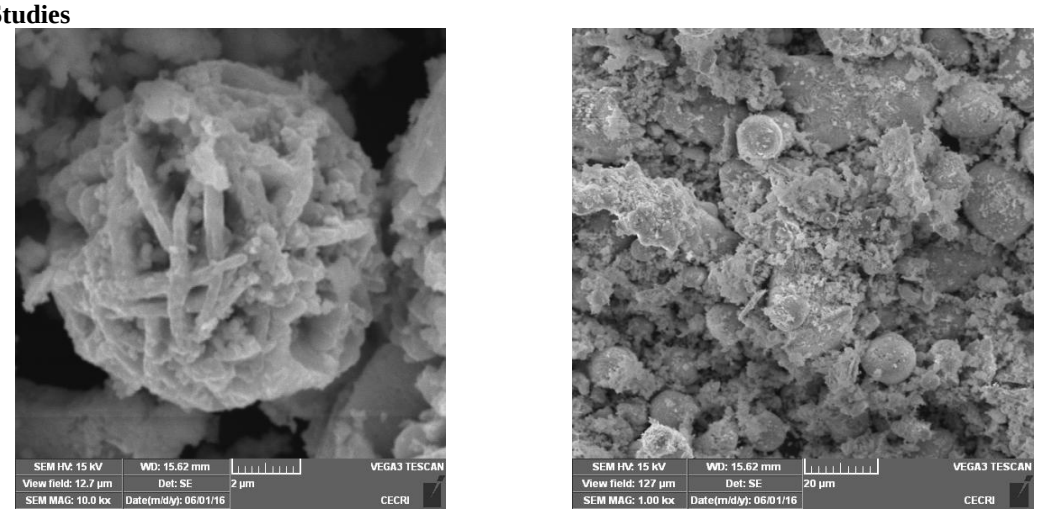



Figure 2. shows the (a) absorption spectrum of annealed Pure and Ni doped $SnO_2$ (b) energy band gap of annealed Pure and Ni doped $SnO_2$

Absorption spectra shows an ultraviolet cut-off around 190–250 nm Fig. 2(a) which can be attributed to the photo-excitation of electrons from valence band to conduction band. Generally, high absorption value is observed in the UV region and it becomes low at visible region. Optical transmittance spectra of pure $SnO_2$ and Ni-doped $SnO_2$ nanoparticles at annealed samples from 200 to 1000 nm are shown in Fig. 2(b). It is observed

that the values of transmittance are high in the visible region and minimum at wavelength ~200 nm. The inset of the figure shows 2(c) the Tauc's plot for determining the band gap energy of nanoparticles. The estimated band gap energy of un‐doped $SnO_2$ is ~2.16 eV, while, the band gap energy of the Ni doped compound found to almost same and is ~1.75 eV. The observed band gap energy of un‐doped $SnO_2$ nanoparticles is quite higher than the band gap energy of bulk $SnO_2$ (3.6 eV). The decrease in the band gap energy may be due to the accumulation of donor energy levels of TM ions in the actual band gap of $SnO_2$.

**SEM Studies**




(a) (b)

Fig. 3 – SEM morphology of (a) as-prepared and (b) Ni- doped $SnO_2$ nanoparticles

The surface morphology of the pure and Ni doped $SnO_2$ nanoparticles was studied by SEM. Figure 3a and 3b shows the SEM images of Pure and nickel doped tin oxide nanoparticles. Pure $SnO_2$ nanoparticles shows that mushroom pattern but in the case Ni doped $SnO_2$ nanoparticles mushroom pattern was absent. Particle size and spreading of nanoparticles primarily depend upon the relative rates of nucleation and growth processes, as well as the extent of agglomeration [14].

## 4. CONCLUSION

Pure and Ni-doped Tin Oxide nanoparticles have been synthesized by using co-precipitation method. The nanoparticles were synthesized without any requirements of special atmosphere and high pressure. Structural study reveals that the prepared samples have tetragonal structure. The band gap energy of doped $SnO_2$ nanoparticles was found to be lesser than the pure. This may due to the accumulation of donor energy levels of Ni ions in the bandgap of tin oxide. Surface morphology pure and Ni doped $SnO_2$ nanoparticles were studied by SEM technique.

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