SYNTHESIS AND CHARACTERIZATION OF SM-DOPED
ZINC OXIDE NANO-CRYSTALLINE POWDERS

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ABSTRACT

Samarium doped zinc oxide nanocrystals have been synthesized adopting a facile aqueous solution approach. The characterization of these samples has further been accomplished identifying the diffraction peaks using X-ray studies and the size morphology through electron microscopy. The chemical characterization has struck peaks depicting the ubiquity of Sm in the crystalline material along with zinc and oxygen. Aqueous solutions of zinc Carbonate, Samarium Chloride and Urea have been lessened for the synthesis of Sm-ZnO nanocrystals which were found to be dimensionally between 100-120nm.

KEYWORDS: Zinc Oxide, Samarium Doped, Nanoparticles, Synthesis, Characterization

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INTRODUCTION

The attributes that ZnO (BG -3.37 eV) carries with itself, such as, its wurtzite hexagonal close packing alternately composed of O²⁻ and Zn²⁺ ions, the ions bearing a tetrahedral coordination forging these structures to be non-centro symmetric, consequentially exhibiting piezoelectricity [1] and creating a dipole moment and a state of instant polarization that results in the diversification of its surface energy.

The potential of the nano devices has brought about a surge of interest in the research community and the quest to understand and synthesize nanostructures through bio-convivial, viable and uncomplicated methods that can be easily adoptable by the industry for production at a large scale has seen extraordinary efforts of researchers. The contrasting difference of the same characteristic, as the particles are splintered into a nano-scale through multifarious techniques, has been a major area of scientific pursuit. Contemplating on the current subject matter, the past decade witnessed the growth of multiple methods [2-5] for the synthesis of 1-D ultra-fine zinc oxide structures that possessed unique applications. There had been rigorous approaches to gain control over various aspects of synthesis (viz. composition, surface phenomenon, phase, dimensions and most importantly the crystal properties).

Hydro-thermal, PVD, non-hydrolytic route, refluxing technique and wet chemical method etc., have been used by researchers for the synthesis. With vast applications in multiple fields such as transducers, sensors and catalysts due to their excellent electrical, magnetic, optical and thermal properties the RE doped zinc oxide nanocrystals has turned out to be an important area of quest.

Vast applications have been attributed to ZnO nanostructures and extensive pursuit to realize the desired output has been on various aspects such as piezoelectric-gated diode [6], sensors for gas detection [7], cells for storage of non-conventional energy forms [8], LED’s [9], FET’s [10]. The pure form of the synthesis of these...
nanostructures has been insipid. Addition of impurity atoms has been a widely adopted method, and a doping material was found to enhance the possibility of controlling the morphology and properties to obtain the desired electro-optic and magnetic properties. The task of doping has been un-assailable due to the easy formation of a metal-aqua complex and its inability to merge into the ZnO crystal lattice [11]. The radiative efficiency of the impurity-induced emission increases [12] when a rare earth(RE) dopant is introduced into the crystal lattice of ZnO structure, enhancing its photo-luminescence phenomenon.

The aqueous solution method has been tried out [13,14] with possible utility in fabrication of Opto-electronic devices and light emitting phosphors for displays and has been found to be suitable for doping with RE materials where the RE$^{3+}$ ions substitute some of the host ions with little modification of the Wurtzite structure.

The wide band gap of the ZnO increases the possibility of not only the excitation of the ions of the RE dopant but also in controlling its conductivity [15]. Various methods have been reported for the synthesis [16-22].

Compared to physical doping methods, solution method has been found to be advantageous due to low reaction temperature, low cost, minimum equipment requirement, atmospheric pressure and product homogeneity [22, 23].

Aqueous solution method has been found to be beneficial over other methods due to myriad reasons explained in this article. Synthesis of Sm doped ZnO nanocrystals and characterizing the sample by identifying the diffraction peaks using X-radiation studies and the size morphology through electron microscopy has been reported in this article along with a statistical account of the size of the particles and the potential difference (\(\phi\)-potential) between points at the interfacial layer and the bulk of the colloidal sample.

**MATERIALS AND METHODS:**

The synthesis has been carried out as depicted in the Schematic Diagram (Figure 1) with AR-Grade reagents of zinc carbonate (ZnCO$_3$), urea (NH$_2$COH$_2$), samarium(III) chloride hexahydrate (SmCl$_3$.6H$_2$O) and ethylene glycol. DI water has been used during synthesis.

![Figure 1](image_url)
The X-radiation studies were carried out with an X-ray powder diffractometer (CuKα - \( \lambda = 1.54056\text{Å} \)) and the data was compared for sample identification. The grain size has been estimated through the use of the Scherrer relation [24]. The size morphology has been scanned by electron microscopy. EDX analysis was carried out for confirmation of the sample composition.

The electro-kinetic or \( \zeta \)-potential Figure 7 has been estimated by the analyzer through the statistical evaluation of the net electrical charge contained within the region bounded by the slipping plane, which is dependent on the location of that plane and would be different at different locations. It is an indicator of the colloidal stability. The higher value of the \( \zeta \)-potential (±) obtained indicates that the synthesized Sm-ZnO sample is highly stable [25-29]. The statistical distribution to estimate the particle size and the \( \zeta \)-potential have been obtained using Horiba Series Nanoparticle Analyzer.

RESULTS AND DISCUSSIONS

X-Ray Diffraction Studies

The dominant peaks (Figure 2) and the average grain size that has been estimated was found to be between 24 to 35 nm with the evaluated Miller indices indicating that the structure was crystalline with aligned wurtzite hexagonal closing packing structure that matches with the Zinc Oxide (JCPDS Card No:36-1451).

![Figure 2: XRD of Sm-Doped ZnO Nanocrystals.](image)

The average crystallite size (D), line broadening(\( \beta \)) lattice strain (\( \varepsilon \)), and the lattice spacing(d) and \([h,k,l]\) parameters [30, 31] have been evaluated in the Table 1.

<table>
<thead>
<tr>
<th>No.</th>
<th>(&lt; I &gt;)</th>
<th>(&lt; 2\text{Theta} &gt;)</th>
<th>(&lt; \text{FWHM} &gt;)</th>
<th>( D = \frac{K \lambda}{\beta \cos \theta} )</th>
<th>Lattice Strain (( \varepsilon ))</th>
<th>(&lt; D &gt; )</th>
<th>N(\lambda=2d\sin\theta)</th>
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### Table 1: Contd.,

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Estimated: \( a=b \rightarrow 3.287 \, \text{Å}, \ c \rightarrow 5.13 \, \text{Å} \)

Reported: \( a=b \rightarrow 3.2-3.4 \, \text{Å}, \ c \rightarrow 5.2-5.4 \, \text{Å} \).

### SEM Analysis

Approximate Nano-crystallite size ranging within 100-150 nm has been observed surfing through the top as the scanning process dug through to the bottom layers at about 60KX magnification in the 200 nm scale (figure 3)

![Figure 3: SEM Images of Sm Doped ZnO Nanocrystals](image)

The Result also included the conformity of the elemental composition of the synthesized Sm-ZnO sample through EDX analysis (Figure 4)

![Figure 4: EDX Analysis of Sm Doped ZnO Nanocrystals](image)

### Particle Size and Zeta Potential Analyses

The particle analyzer has categorized the actual presence of nano-sized particles through a statistical fit as shown...
in the Figure 6. The size of the particles has been found to be around 110 nm which was in confirmation with the Scanning results through the electron microscopy. The zeta potential was estimated using Horiba Zeta Potential analyzer through the addition of 0.1 g of ZnO nanoparticles in 10 ml of water (neutral pH value). The zeta potential for Sm–ZnO was $|41.3\, \text{mV}|$. The analysis showed that the charge on the surface of the nanoparticles does not change due to samarium doping [32-35].

**CONCLUSIONS**

In summary, Samarium-doped ZnO nanoparticles with particle sizes in the nanometer range have been successfully synthesized through aqueous solution method. The process has been very simple and could be repeated with ease. As there have been no parameters involved that would change the characteristic of the experiment with increase in the volume of the sample amount, the method is easily adoptable for a large scale production of the nanocrystals.

The crystal sizes as witnessed through the SEM were of sizes ranging within 100-150 nm and elemental composition has been found to be in conformity of the sample amounts used for the synthesis, which again reiterates the possibility of the use of this method for large scale production of the sample.
X-Ray Diffraction studies indicated an average crystallite size (D) between 24-35 nm, lattice strain (ε) 0.0018-0.0054, and the lattice spacing (d) varied between 1.36 to 2.84 nm. The crystalline form of the structure is evident from the distinctness of the peaks. The truancy of the peaks related to the RE dopant reveal the possibility of Sm$^{3+}$ ions replacing some of the Zn$^{2+}$ in the crystal lattice. A shift towards a lower value of $2\theta$ of the diffraction peaks that resulted in the increase of the c-value of the lattice parameter could be attributed to the presence of the dopant as the ionic radius of the Sm$^{3+}$ has a higher value in comparison to the Zn$^{2+}$ ($r_{\text{ion - Samarium}} = 0.964 \ \text{Å}, \ r_{\text{ion - Zinc}} = 0.74 \ \text{Å}$).

The statistical fit of the size estimation has resulted in a peak at 113 nm evincing the particle sizes to be around 110 nm. The higher value of the ζ-potential classifies the synthesized sample into a good stability region.[28, 29]

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