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RESEARCH ARTICLE

Synthesis of Ag Doped ZnO Nanowire by Hydrothermal Method and its Characterization

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Abstract

This research is mainly focused over synthesizing zinc oxide (ZnO) nanowire (NW) and silver doped zinc oxide nanowires (Ag/ZnO) using two step hydrothermal method which comprises of seed layer deposition and hydrothermal nanowire growth step. Hydrothermally grown nanowires are of great significance. The synthesized nanowires were characterized by UV-Vis Spectroscopy (UV-Vis), X-ray Diffraction Spectroscopy (XRD), Energy Dispersive Spectroscopy (EDS), Field Emission Scanning Electron Microscopy (FE-SEM) and High-Resolution Transmission Electron Spectroscopy (HR-TEM). XRD pattern revealed as-synthesized materials are of crystalline nature having crystallite size of 166.27 nm and 42.32 nm for ZnO nanowire and Ag doped nanowire, respectively. Further confirmation was done by EDS, SEM and TEM. The microscopic images of SEM and TEM evidenced for successful fabrication of nanowires.

Keywords: Hydrothermal; Nanowire; Seed layer; Silver doped; Zinc Oxide

Introduction

Nanotechnology, an emerging field of science, holds significant importance from both industrial and academic perspectives, involving the production, manipulation, and utilization of materials at the subatomic level to create innovative products and processes [1]. Fabrication of nanomaterials of various morphologies like nanoparticles, nanotubes, nanowires and nanosheets etc. has been especially alluring in recent years [2]. Great significance owed by nanomaterials is directly related to the high aspect ratio. Based on literatures, various physicochemical properties such as electronic, optical, thermal conducting, magnetic, medicinal, gas sensing and many more have turned these existing nanomaterials as the main root for advancement of material science [3], [4]. Beside these, nano structures have been used as photocatalyst to deactivate bacteria and also to accelerate the degradation of environment pollutants like dyes, pesticides and volatile organic compounds [5]. Among various types of metal and metal oxide nanomaterials, ZnO NW is one of the potential materials for advancement of broad range of fields such as gas sensor [6], biosensor [7], optoelectronics [8], UV light absorption [9], nanogenerator [10], photocatalyst [11], waste water treatment [12] etc. Since last few decades,

ZnO nanomaterials have been greatly employed in solar cell panel for its efficient light absorption capacity and electron transport efficiency owing to the higher number of grain boundaries [13]. Additionally, ZnO nanowires have been extensively used for sensing various kinds of gases such as ethanol (C_2H_5OH), ammonia (NH_3), carbon monoxide (CO), hydrogen (H_2), HCHO etc. [14]. In recent years, nanomaterials have been simply modified using different kinds of dopants such as P, N, Al, As, Li, Sb Ag, etc. so as to improve their various physical and chemical properties such as conducting, electrical, sensing and many more [15], [16].

In the context of synthesizing nanomaterials, various methods such as sol-gel method [17], chemical vapour deposition method [18], ultrasonic method [19], hydrothermal method [20] etc. have been found to be the most convenient and common methods all over the world. Comparative to other synthetic methods, hydrothermal synthesis has gained much more these days for its low cost, scalability, convenient to handle. The solution phase synthesis occurs in mild condition (less than 200 °C). The process is nontoxic and of great importance as nanowire of length up to 500 μm is easily synthesized under aqueous condition without using any organic solvents [21]. Concurrently, one dimensional nano-materials have been realized to be the material of choice for advancement of material mainly because of its superior crystallinity and ultra-high stability [22], [23], [24], [25]. Literatures related to ZnO of one-dimensional type is rarely available, thus main target of this research is to fabricate one dimensional ZnO nanowire along with Ag doped ZnO nanowire to enhance their functional properties using simple and cost-effective hydrothermal method. The most common dopants for the modification of nanomaterials are being the noble metal, hence silver is chosen in the research. Additionally, high stability, strong absorbing, electrical and conducting properties of silver subjected to prefer it as the dopant for this research [26].

Materials and Methods

Materials

Acetone (AR grade, Merck Company), methanol (AR grade, Merck Company), distilled water and silver nitrate (AR grade, Qualigens company), Zinc acetate dihydrate (AR grade), Zinc nitrate hexahydrate (AR grade, Qualigens company), Hexamethylenetramine (HMTA, AR grade, Qualigens company), Diethanolamine (DEA, Merck Company) were used.

Methods

The synthesis of nanowires of ZnO and Ag modified ZnO nanowires were actually accomplished in two steps:

Seed layer deposition

Figure 1: *Homemade spincoater*

The first step involved deposition of a ZnO seed layer over the pre-cleaned glass substrate. Initially, a precursor solution was prepared by dissolving 1 mM of zinc acetate and DEA in acetone, which was then continuously stirred using a magnetic stirrer at room temperature [27], [28]. Next, the glass substrate

was spin-coated with the prepared zinc acetate solution using a homemade spin coater at 3000 rpm for 30 seconds. The picture of locally made homemade spin coater is presented in **Fig. 1**. Finally, to achieve the thermal decomposition of zinc acetate, the substrate was annealed in air at 350 °C for 20 minutes. In another set, silver doped ZnO seed layer was synthesized using the similar process. At first glass slide were sequentially cleaned by acetone, methanol and distilled water and then dried. The Ag doped ZnO seed layer solution was prepared by dissolving 1mM of zinc acetate, 4% $AgNO_3$ and DEA in acetone using magnetic stirrer without external heating [28]. Then the substrates were subjected for drop coating using homemade spin coater at the speed of 3000 rpm for 30s. Finally, the substrates were annealed in air at 350° for 20 min. Then, the substrates were allowed to hydrothermal growth. Seed layer seemed to be deposited involving following reactions as mentioned here in [29]:

$Zn(CH_3COO)_2.2H_2O \longrightarrow Zn^{2+} + 2CH_3COO^- + 2H_2O$ ... (1)

$CH_3COO^- + H_2O \longrightarrow CH_3COOH + OH^-$..... (2)

$2OH^- + Zn^{2+} \longrightarrow Zn(OH)_2$ ..... (3)

$Zn(OH)_2 \longrightarrow ZnO + H_2O$..... (4)

Zinc acetate is decomposed to give Zinc and acetate ion. The acetate ion reacts with water to give hydroxyl ion. The formed zinc ion and hydroxyl ion combines to give zinc hydroxide which further decomposes to give zinc oxide (seed layer).

Hydrothermal growth of nanowire

Secondly, hydrothermal growth of ZnO nanowire was accomplished using solution (25 mM) Zinc nitrate hexahydrate and hexamethylenetetramine in distilled water [28]. The substrate with seed layer was dipped in the aqueous solution of Zinc nitrate hexahydrate. Then the beaker along with substrate was heated over water bath maintaining temperature (95 °C) continuously for two hours. Afterwards, at the end of growth; the solution was cooled down to room temperature. The substrate was soon removed from beaker and rinsed with distilled water and finally annealed in air at 350 °C for 30 min to remove the residual organics [20]. Similarly, Ag doped ZnO nanowire was grown hydrothermally using aqueous solution containing 25 mM Zinc nitrate hexahydrate and hexamethylenetetramine [28] in a beaker. The solution mixture along with the substrate layer was heated in water bath at 95 °C. Growth of nanowires assumed to take place with following chemical reactions [29]:

$Zn(NO_3)_2.6H_2O \longrightarrow Zn^{2+} + 2NO_3^- + 6H_2O$ (5)

$C_6H_{12}N_4 + 6H_2O \longrightarrow 6HCHO + 4NH_3$ (6)

$NH_3 + H_2O \longrightarrow NH_4^+ + OH^-$ (7)

$2OH^- + Zn^{2+} \longrightarrow Zn(OH)_2$.. (8)

$Zn(OH)_2 \longrightarrow ZnO + H_2O$ (9)

Here, Zinc nitrate hexahydrate supply Zn^{2+} for the formation of nano wire. HMTA hydrolyses in water yielding formaldehyde and ammonia. The formed ammonia dissolves in water to give ammonium ion and hydroxyl ion. Hydroxyl ion reacts with Zn^{2+} producing Zinc hydroxide. Finally, Zinc hydroxide decomposes to Zinc oxide and water.

Characterization

ZnO nanowire and Ag-doped ZnO nanowire prepared by hydrothermal methods were characterized by UV-vis spectrophotometer in the range of wavelength 300 nm – 700 nm (UV-HR40000V NIR ocean optics, Amrit Campus, Physics department). The phase structure of materials (ZnO and Ag doped ZnO nanostructure) was studied using the powder X-ray diffractometer (Model: D/Max 2500 V/PC;

Rigaku Co., Japan). Morphological characteristics of the prepared undoped ZnO and Ag doped nanostructure were investigated by field emission scanning electron microscopy (FE-SEM, Hitachi SD-8230, carried in Jeonbuk National University, Korea). The size of the diameter of nanostructure was determined using HR-TEM microscopy (HR-TEM, JEM-2010, JEOL, Japan). Hydrothermally grown ZnO and Ag doped ZnO nano structures were subjected to EDS (Hitachi SD-8230, Korea) for determination of elemental composition.

Results and Discussion

Preliminarily, the formation of ZnO nanowire was confirmed from optical absorption spectrum obtained from the UV-vis spectroscopy. From the **Fig. 2**, average of 95% light is observed to be transmitted by thin film of ZnO nanowire within the visible light range 300 nm to 1000 nm. At the same time, the sharp absorption band edge is clearly seen at 360 nm which is quite near to that reported in the literature [28]. Here, the shift of sharp absorption band may be because of the thin film of nanomaterials of different dimensionalities i.e., ZnO nanowire. Furthermore, the transmittance *vs* wavelength plot of the hydrothermally grown Ag doped ZnO nanowire shows an average of 90% transmittance in the visible range (**Fig. 3**). Comparatively, the transmittance shown by 4% doped sample is found slightly lesser than that of the undoped ZnO. The formation of Ag doped ZnO was also supported with red shift of the absorption band edge to 370 nm [24], [30], [31]. Optical band gap is the minimum energy required to excite an electron that is stuck in its bound state into a free state where it can participate in conduction. An excitation is state of electron and hole, which are bound by the columbic force. An excitation occurs when the semiconductor absorbs a photon, so band gap is the threshold for photon to be absorbed [32]. The band gap energy of ZnO and Ag doped ZnO nanowires were obtained from Tauc's relation. The plot of $(\alpha h\nu)^2$ vs $h\nu$ of ZnO nanowire and Ag doped nanowire grown using low cost, hydrothermal method is shown in **Fig. 4(a) and Fig. 4(b)**, respectively.

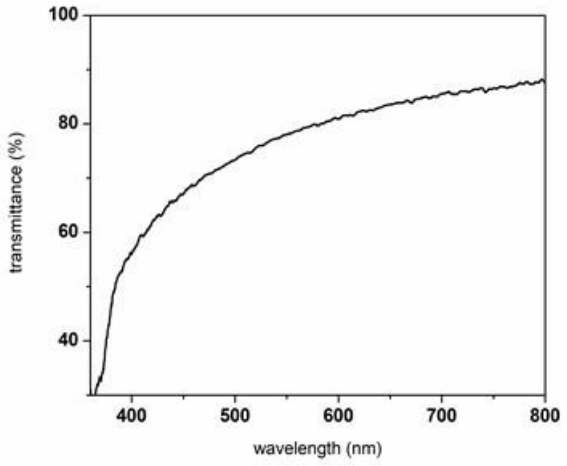

Figure 2: *UV-Vis spectrum of hydrothermally grown ZnO nanowire*

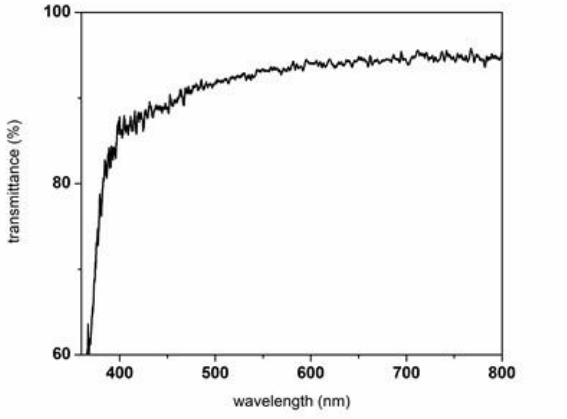

Figure 3: *UV-Vis spectrum of hydrothermally grown Ag doped ZnO nanowire*

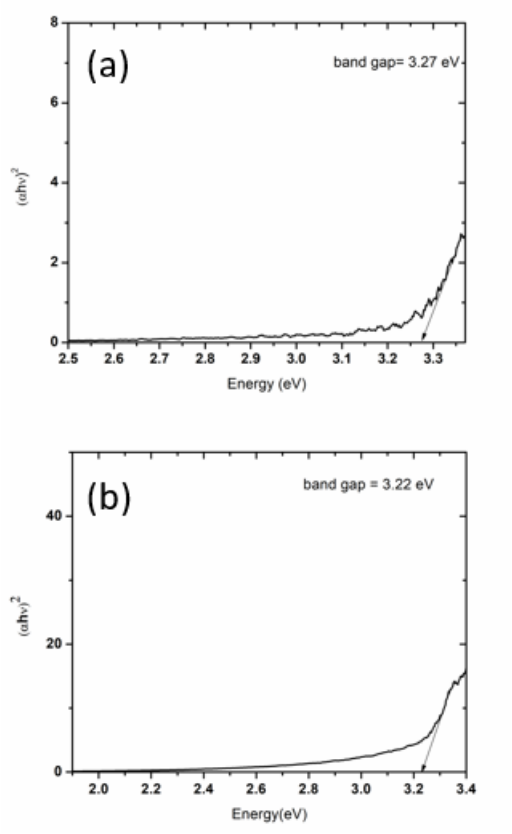

Figure 4: *Band gap of nanowires: a) ZnO nanowire, b) Ag doped ZnO nanowire*

The band gap energy of undoped ZnO was calculated to be 3.37 eV which is equivalent to the reported value in the literature [33]. Similarly, band gap energy of as-synthesized Ag doped ZnO nanomaterial was estimated to be 3.22 eV which is lesser than reported band gap energy of undoped ZnO nanomaterials. Thus, decreased value of band gap energy observed after modification of ZnO supported for the formation of Ag doped ZnO nanowire [24], [34], [35]. Hence, UV-vis spectra support for slight doping with silver ions over thin film of ZnO nanowire [36].

Then after, the as-synthesized materials were subjected to X-ray diffraction spectroscopy for exploration of detail of the phase morphologies. Results obtained from the XRD spectroscopy of hydrothermally grown undoped ZnO heating over water bath at 90 °C for 2 hours and Ag doped ZnO are presented in **Fig. 5**.

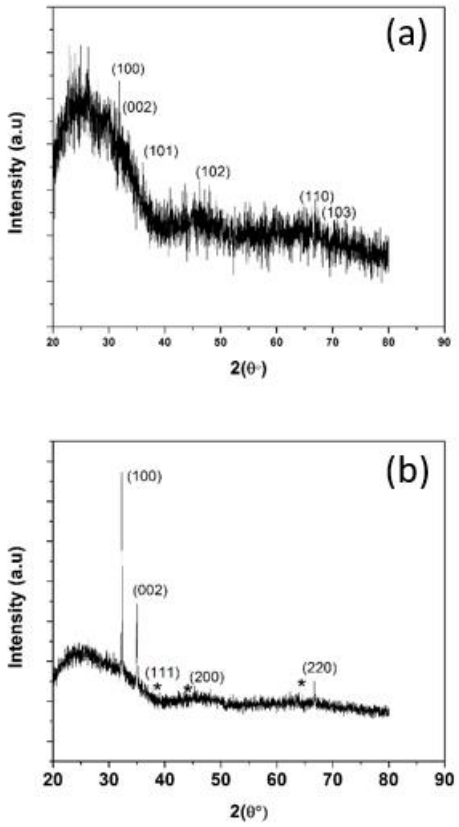

Figure 5: *X-ray Diffraction pattern of: (a) Undoped ZnO (b) Ag doped ZnO*

The **Fig. 5(a)** reveals sharp peaks at various diffraction angles (2θ) 31.72°, 34.17°, 36.26° corresponding to crystal planes (100), (002) and (101), respectively following JCPDS file no. 36-1451 which well supports for the formation of crystalline ZnO having wurtzite crystals as reported in literature. Similarly, the plot shows some weak peaks at 46.16° (102), 56.2° (110) and 66.6° (103) here quite match with the literature [36]. The phase morphology of nanoparticles are found to be dependent on temperature and time duration [36]. In general, sharp peaks of ZnO nanomaterials are obtained when the materials are annealed at higher temperature for more than 12 hours [37]. However, the average calculated crystallite size by using the Debye Scherrer equation as 166.27 nm. Similarly, diffraction pattern of (4 wt.%)

Silver doped ZnO nanowires (**Fig. 5b**) comprises of three sharp peaks. Among the three, peak located at diffraction angles (2θ) 31.72°, 34.17° corresponds to (100), (002) planes of ZnO nanomaterial and new weak peak observed at 38°, 44° and 64.42° corresponds to (111), (200) and (220) plane of the face centered cubic silver which was confirmed from the JCPDS file -04-0783 [38]. The result is found to be supported by the literature [39] which has revealed similar results as presented herein. The crystallite size of Ag doped ZnO nanowire was calculated to be 42.32 nm using the FWHM values. Thus, the result of X-ray diffraction (XRD) shows the formation of ZnO and Ag doped ZnO wires of nano size.It is very crucial to study the surface morphology of so prepared materials in the research; hence the materials were subjected to field emission scanning electron microscopy (FE-SEM) at resolution 100 μm. The FE-SEM of ZnO nanowires and Ag doped ZnO nanowires are shown in **Fig. 6**.

The microscopic image (FE-SEM; **Fig. 6(a)** clearly shows the formation of ZnO wires exist in the form of network like structure. However, the microscopic image do not comprised of nanowires of well aligned nature which would be possible only at controlled growth parameters such as; temperature, time, concentration, precursors only [40]. Similarly, **Fig. 6b**, reveals the existence of network of wire like materials as shown in the Figure 6a which confirmed for formation of modified ZnO. Upon closer view, the materials *i.e.,* ZnO nano wires (**Fig. 6b**) are found to exhibit shining and smooth surface marked by square further evidenced for the silver doping on ZnO nanowire. The surface morphology of as prepared nanowires was further characterized *via* high resolution transmission electron microscopy (HR TEM, JEM-2010, JEOL, and Japan). The transmission electron micrograph presented black colored somewhat rod like structured ZnO nanowires (**Fig.7a**) and the Ag doped ZnO nanowires are found to exhibit bright patches of silver ions (**Fig. 7b**). Thus, the result confirmed for the formation of ZnO and Ag doped ZnO nanowires. The diameter of ZnO wire and Ag doped ZnO wire were calculated using image J software and the corresponding values are found to be 180 nm and 169 nm, respectively.

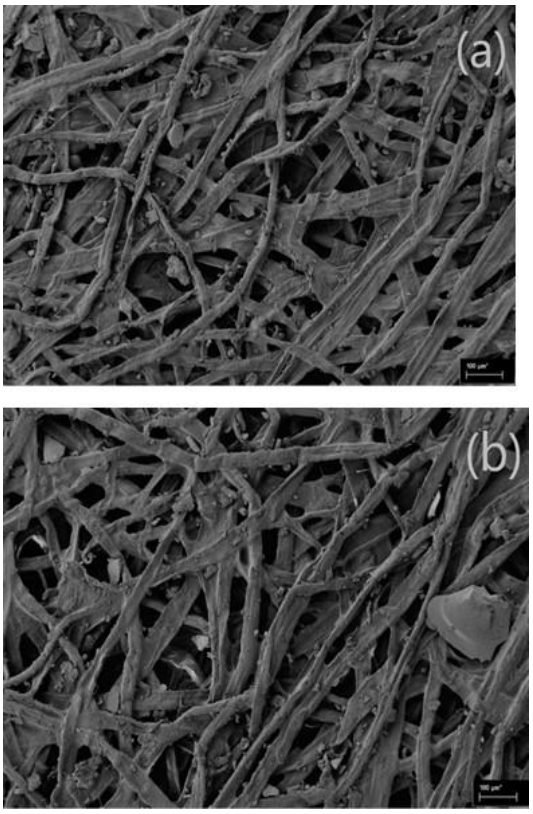

Figure 6: *FE-SEM of nanowire (a) ZnO nano wire (b) Ag doped ZnO*

Likely, energy dispersive X-ray spectroscopic spectrum of ZnO nanowire consists of well-defined peaks of constituting elements such as Zn, O at 1 eV, and 0.4 eV correspondingly in the **Fig. 8(a)**. In the same context, **Fig. 8(b)** shows peak with small intensity of Ag at 2.5 eV which may be due to use of less wt.% of silver during fabrication of the material.

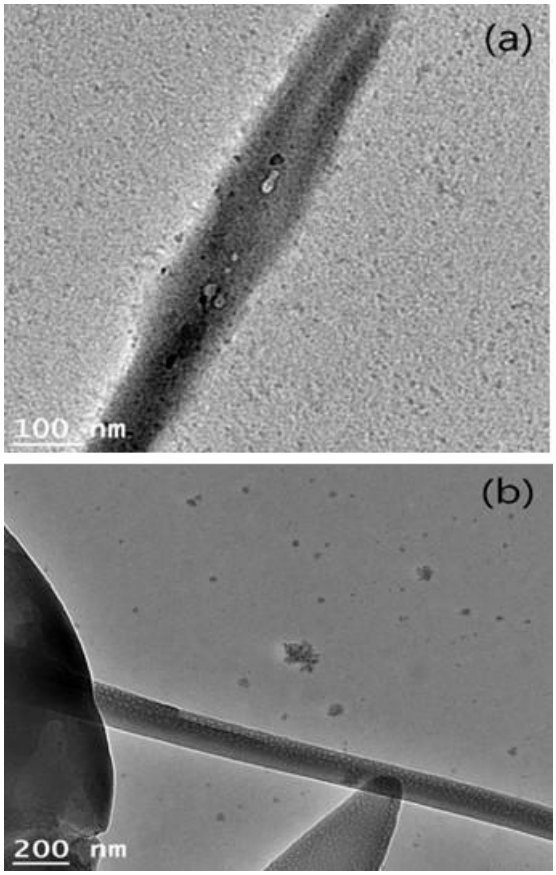

Figure 7: *Transmission Electron micrographs of hydrothermally grown nanowire (a)ZnO nanowire b) Ag doped ZnO nanowire*

Table 1: Elemental composition of ZnO and Ag doped ZnO nanowire achieved from EDS analysis

	ZnO nanowire		**Ag doped nanowire**	
Composition	**Wt.%**	**At.%**	**Wt.%**	**At.%**
Zn	64.33	70.24	63.23	69.93
O	23.82	17.46	11.15	13.27
C	11.84	12.30	24.37	16.22
Ag	-	-	1.24	0.58
Cu	0.00	0.00	0.00	0.00
Si	0.01	0.00	0.01	0.00

The **Table 1** shows the composition by at. % and wt. % of ZnO nanowire and Ag doped ZnO nanowire. Some elements like C, Cu & Si are also appeared which may arise from sampling process. Thus, the EDS spectra comprising of sharp peaks, especially of Zn, O and Ag provide us good evidence for confirmation of ZnO and Ag doped ZnO nanowire.

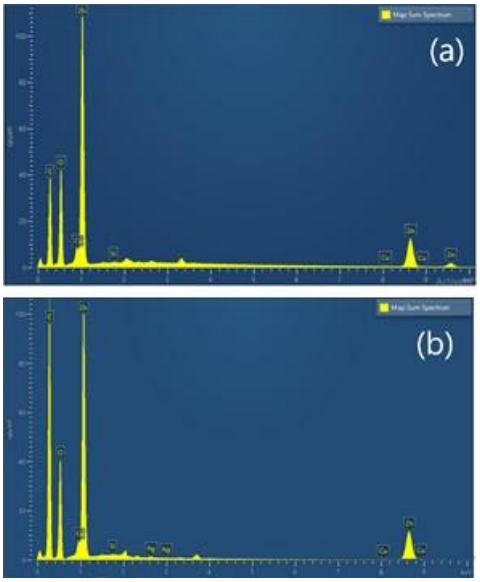

Figure 8: *Energy Dispersive Spectra of hydrothermally grown: (a) ZnO nanowire (b) Ag doped ZnO nanowire*

Conclusions

This research successfully fabricated ZnO nanowires and silver doped ZnO nanowires based on eco-friendly and cost-effective hydrothermal method in two steps. The process constitutes seed layer development in the first step while the second step consists of nanowire development. Various type sophisticated instruments were used for confirmation of as synthesized nanomaterials. The characteristic optical band edge appeared at 380 nm and 370 nm in UV-vis spectrum indicated the formation of ZnO and Ag doped ZnO nanomaterials. Further confirmation of the as-synthesized materials was done using XRD, SEM, TEM and EDS. The result of EDS evidenced for presence of desired elements Zn, O and Ag. Likely, SEM and TEM confirmed the formation of ZnO nanowire and silver doped nanowires having diameter of 180 nm and 169 nm which were calculated using the image J software. Exploration of photocatalytic dye degradation and gas sensing properties of as-synthesized nanowires could be interesting topic for future.

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Author's Contribution Statement

Utsab Luitel: Methodology, Investigation, Formal analysis, Data curation, Writing-original draft preparation, **Kamal Prasad Sapkota:** Formal analysis, Writing-review and editing **Santu Shrestha:** Formal analysis, Writing-review and editing **Dasu Ram Paudel:** Formal analysis, Writing-review and editing**, Sharmila Pradhan:** Conceptualization, Resources, Funding acquisition, Writing-review and Editing, supervision

Conflict of Interest

The authors do not have any conflict of interest throughout this research work.

Data Availability Statement

The data supporting this study's findings are available from the corresponding authors upon reasonable request.

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