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Synthesis of Ag-ZnO by Seed Mediated Hydrothermal Method and its Characterization.

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ABSTRACT

Metal nano composite finds key position in many bioengineering and biomedical field. Metal nano composite also shows good catalytic efficiency, optical, thermal and electrical properties than pure nano crystal. In this regard the current paper deals with controlled synthesis and characterization of Ag-ZnO. The synthesis of Metal nano composite Ag-ZnO based on Wet Chemical Method (seed-mediated hydrothermal method) and size was found to be 14 nm and characterization were carried by XRD, TEM, PLE, absorption spectra by using UV-Vis spectrophotometer. The catalytic activity of metal nano composite have discussed as supporting explanation.

Keywords: Ag-ZnO, Catalytic activity, Seed-mediated hydrothermal method, Metal nano composite, nano crystal, Wet Chemical Method

I. INTRODUCTION

Metal nano composites gain much interest in today scientific, research and industrial era. Nano compositeis a combination or matrix, in which different materials combine to develop new properties of the materials ensuring that one of the materials have size in range of 1-100nm[1-2]. Nano composite can be prepared from any combination of materials that can be categorized into three basic building blocks i.e. metals, ceramics and polymers)[1, 3]. The nano composite hence can have a combination or have markedly different mechanical, electrochemical, electrical, catalytic, thermal and optical properties from the component materials [1, 4-7]. These NC have gained the attention of scientists, researchers and engineers, which had led to the sudden raise in number of publications related to these materials. Further, these NC have number of technological and business breakthroughs in all the sectors of life. This paper regarding synthesis of Metal nano composite namely Ag-ZnO using Wet Chemical Method basically Seed-mediated hydrothermal method and study of its structure using XRD ,TEM ,PLE and characteristic optical absorption using UV-Vis spectrometer.

II. MATERIALS AND METHODS

The synthetic methods are frequently classified in to three classes i.e. solution based synthesis, vapor phase synthesis and gas phase synthesis [8]. wet chemical methods give uniformity in size of nano composites as

controlled particle size can be achieved easily. There are different wet methods for synthesis of metal nano particles and nano composites but co-precipitation, sol gel and hydrothermal methods are cost effective as compared to other methods. We use hydrothermal method for preparation of Ag-ZnO

2.1 Seed-mediated hydrothermal Method

The Seed-mediated hydrothermal method involves the heterogeneous chemical reaction in a solvent (aqueous or non-aqueous) occurring above room temperature and at pressure more than 1atm in a closed system [9]. To modify the size and properties the use of surfactants, capping agents, mineralizer is a common practice [10-13] The new trend is to use this technique in combination with microwave [14], sol-gel [15] that can not only vary the physiochemical and structural properties of the materials but in addition to that can result in formation of single phased materials with enhanced stability [15]. Further just by altering the temperature, time and pressure of the reaction particle size, phase changes morphology and properties as presented in Figure 12.4 [13, 16].

2.2 Characterization of Metal Nano composites

The Metal nano composites are characterized using different techniques to get insight into the morphology, particle size, phase, and composition, optical, magnetic, electrical and thermal properties.

2.2 (a) X-ray Diffraction (XRD)

X-ray diffraction, used to study the structure, composition, and physical properties of materials. Powder X-ray Diffraction (XRD) is used for phase determination and unit cell information of the nanocomposites under investigation [17].

2.2(b) Microscopic techniques – TEM

TEM technique has gained much attention as compared to SEM because of its better resolution i.e. 0.1-0.2nm and wealth of information that can be extracted from it and especially from high resolution TEM (HRTEM) [18, 19] The core-shell structure of the nano composites [20] doping effects on morphology [21], layering in structure [22], particle size [23] gelling agents impact [24], surface roughness [25], nano particle dispersion [26] etc. can be determined. In this regard use of dark field and bright field images can help in getting more information about the structural changes. The main limitation of the technique is in sample preparation which requires low film thickness.

2.2 (c) Absorption techniques (UV-Vis Spectrophotometer)

Absorption spectroscopy refers to spectroscopic techniques that measure the absorption of radiation, as a function of frequency or wavelength, due to its interaction with a sample. The sample absorbs energy, i.e., photons, from the radiating field. The intensity of the absorption varies as a function of frequency, and this variation is the absorption spectrum. Absorption spectroscopy is performed across the electromagnetic spectrum.

Absorption spectroscopy is employed as an analytical chemistry tool to determine the presence of a particular substance in a sample and, in many cases, to quantify the amount of the substance present. Infrared and ultraviolet-visible spectroscopy are particularly common in analytical applications. Absorption spectroscopy is also employed in studies of molecular and atomic physics, astronomical spectroscopy and remote sensing.

There are a wide range of experimental approaches for measuring absorption spectra. The most common arrangement is to direct a generated beam of radiation at a sample and detect the intensity of the radiation that passes through it. The transmitted energy can be used to calculate the absorption. The source, sample arrangement and detection technique vary

significantly depending on the frequency range and the purpose of the experiment.

2.2 (d) Photoluminescence excitation (abbreviated PLE)

Photoluminescence excitation (abbreviated PLE) is a specific type of photoluminescence and concerns the interaction between electromagnetic radiation and matter. It is used in spectroscopic measurements where the frequency of the excitation light is varied, and the luminescence is monitored at the typical emission frequency of the material being studied. Peaks in the PLE spectra often represent absorption lines of the material. PLE spectroscopy is a useful method to investigate the electronic level structure of materials with low absorption due to the superior signal-to-noise ratio of the method compared to absorption measurements.

III. RESULTS

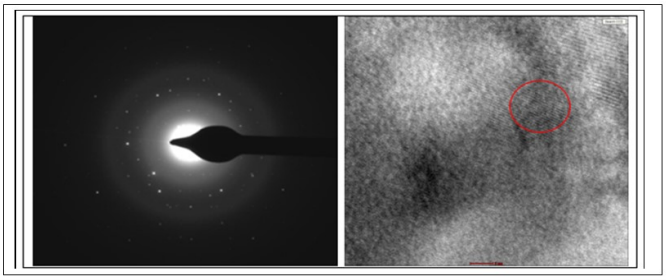

Figure 1. TEM images of ZnO The selected area diffraction (SAED) pattern shows diffraction rings with separate dots, which indicates good crystallinity and growth of ZnO nanoparticles

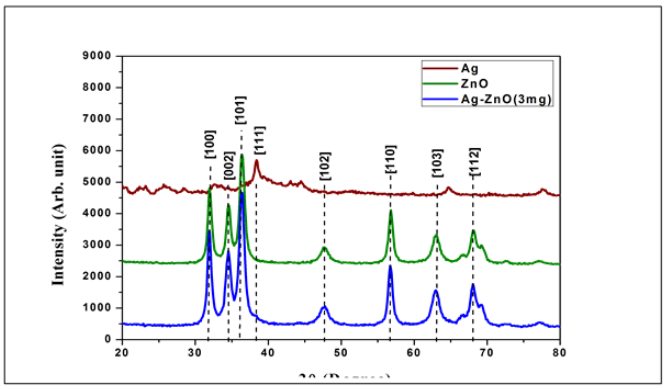

Figure 2. XRD Ag, ZnO &Ag-ZnO (3mg)

Figure 1. shows the TEM images of ZnO. The size of Ag-ZnO was found to be 14nm. Figure 2. shows the XRD of Ag, ZnO and Ag-ZnO at 3mg conc. Figure 3 and 4 shows absorption spectra of Ag at two different conc. 300µL and 400µL respectively. While figure 5 shows the absorption spectra of ZnO , Ag-ZnO at 2mg conc and at 3mg conc and figure 6 shows Photoluminescence excitation (PLE) of ZnO , Ag-ZnO at 2mg conc and at 3mg conc respectively..

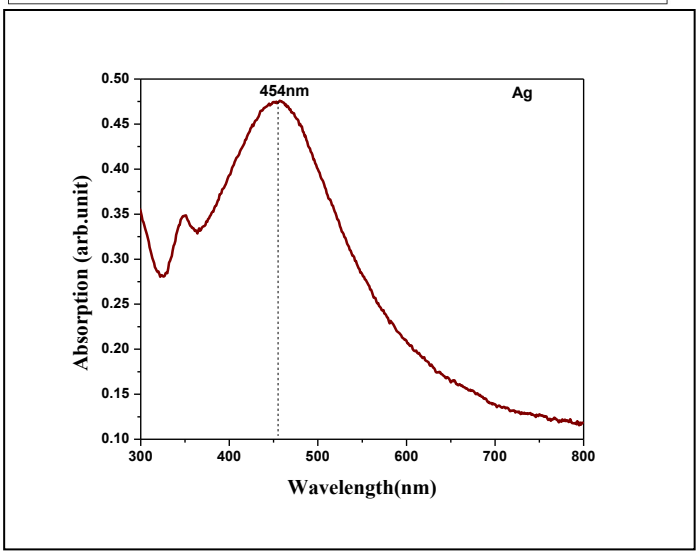

Figure 3. Absorption spectra of Ag (Conc. 300µl)

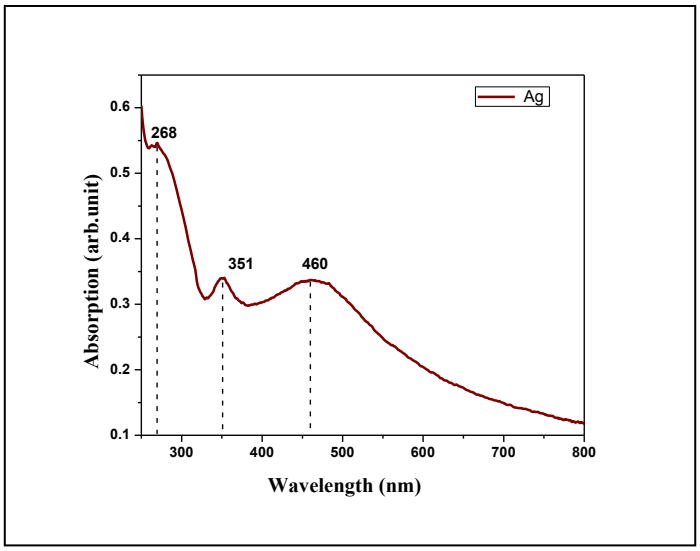

Figure 4. Absorption spectra of Ag (Conc. 400µl)

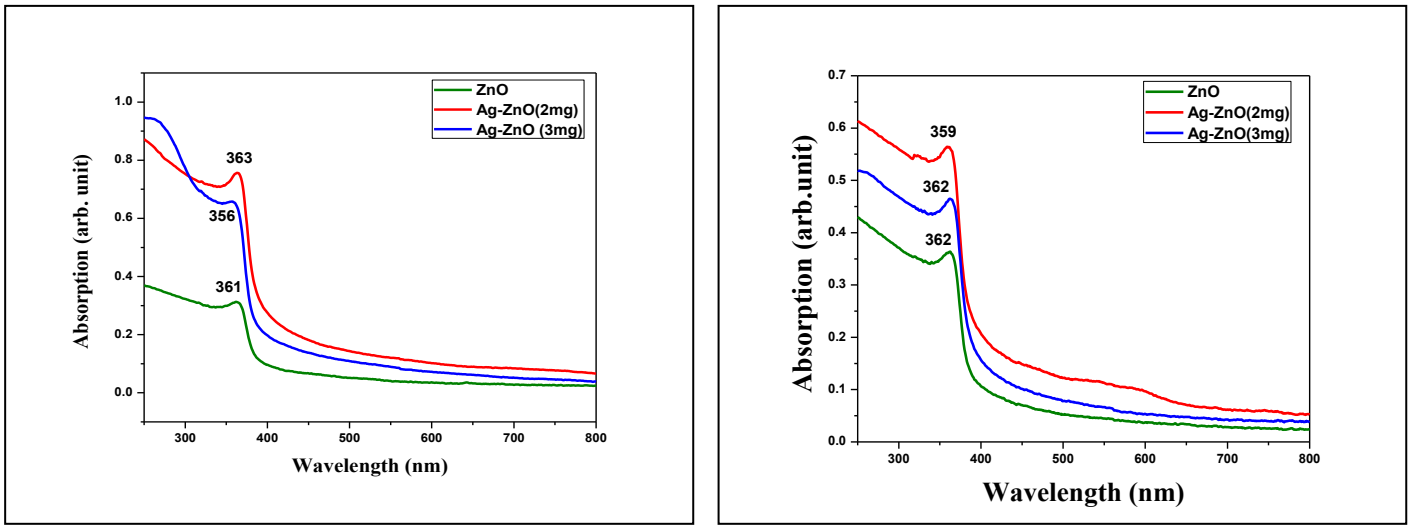

Figure 5. Absorption Spectra ZnO, Ag-ZnO (2mg) & Ag-ZnO (3mg)

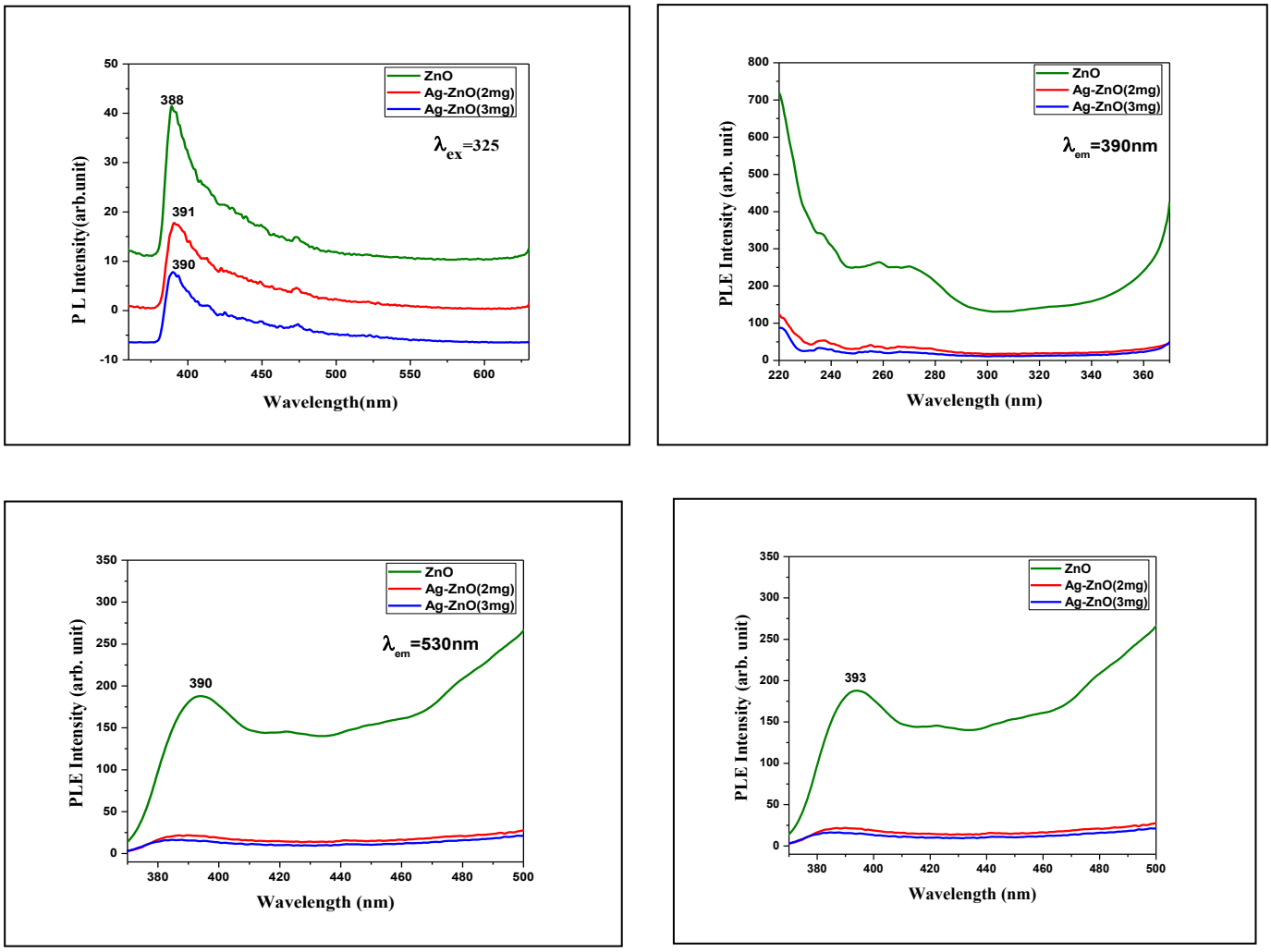

Figure 6. PLE spectra of ZnO, Ag–ZnO (2mg) & Ag-ZnO (3mg)

From the Figure 2 it shows that X ray diffraction pattern for Ag is different than that of ZnO and Ag-ZnO while the X-ray diffraction pattern for ZnO and Ag-ZnO is nearly same. This is because in principle A nano composite is a multiphase solid material, and hence the XRD pattern should show diffraction peaks assignable to (at least) two crystalline solids and A doped material should show only the diffraction peaks corresponding to the major component. Fig 2 also shows that doping of Ag with ZnO does not change the composition of ZnO hence this property of Ag can be used as a catalyst in many chemical reactions. Also observe that in case of nano particles the base of XRD peaks are broad owing to their small size because material is a solid solution, but the crystal lattice has a high concentration of defects. Then the effects of broadening can "mask" the peak's shift, so you won't be able to discern exactly whether you have pure compound or solid solution and material is a composite, but the particles of the second phase are either extremely small (for example, atomic clusters in mechanochemically synthesized pseudo-alloys) or are in a small number (less than 2-5%). XRD won't usually be able to detect such phases. Hence use TEM.

In case of doping the XRD pattern will shift for the host, (without any extra pattern from dopant material) but in case of composite or alloy you will get XRD pattern for all the material in that sample. Alloy or composite just mixture of materials, so you will see xrd pattern for all the materials.Doping is replacement of atoms in original matrix, so you will see alteration in XRD pattern of matrix material. There will be no doped material's pattern. A nanocomposite is a mixture of different material with different phases, and doping is also a mixture of differential material but single phase, hence the XRD pattern should show diffraction peaks related to all the phases for nano-composities but in case of doping peak shift is observed from the host material. Further, peak broadening for both the cases is mostly depends upon particle size (some other reason also possible).

Figure 3 and Figure 4 indicate that that absorption peak by pure Ag at particular wavelength changes with respect to concentration. We have seen from Fig 3 and Fig 4 that absorption peak shifted towards lower wavelength as the concentration increase. Similarly Figure 5 shows that absorption peak occur for nano composite (ZnO) and Metal nano composite (Ag-ZnO) at different wavelength. Figure 5 also shows that wavelength at which peak observed shifted to lower wavelength as the concentration of Ag-ZnO increases. This shifting with concentration can be used for calibration purpose in many chemical reactions. This variation in absorptions shows that formation of Metal nano composite (Ag-ZnO) by our methods hence give validation of hydrothermal method. Similarly the PLE graphs Figure 6 also show the formation of metal nano composite using hydrothermal method.

IV. CONCLUSION

With above all discussion we found that the using Wet Chemical Method i.e. Seed mediated hydrothermal method is one of good method for synthesis of Metal nano composite and all the methods use for characterization supports that by above method Metal nano composite have formed.

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