

Characterization of Co and Al Doped with ZnO using Urea



Presely Rajakumar, Bhuvaneshwari Balachander

Abstract: *The Proposed work deals with the synthesis and the characterization of transition metal doped ZnO composite for energy application. Synthesis of composite is done by combustion method and characterization is performed to identify the size of the particle, composition, morphology, electronic, optoelectronic and magnetic properties. The expected outcome of this research is to identify a Nobel composite material with higher power conversion efficiency and can be used as an optical spacer in a photovoltaic device. It covers the synthesis demonstration and the various characterizations done to come to a conclusion on how the composite mixture can be utilized*

Keywords: *Characterization, metal doped ZnO, Power conversion, synthesis, composite material, conversion efficiency.*

I. INTRODUCTION

Semiconductors a type Nano material has been the latest technological trend for higher efficiency due to its unique properties when compared with other bulk materials. Hence semiconductor particles are seen as the future for many technological applications. One such semiconductor nano particle is Zinc Oxide (ZnO), in nano sized form is most frequently used due to its high potential over other materials and hence ZnO is chosen for this research. As already known, Zinc Oxide has various advantages and applications since it is an efficient semiconducting material, with a gap of 3.3eV between the two bands at moderate thermal conditions [9]. Although wide range of nano materials are used ZnO also have the best optical characteristics [1][7]. The transition element mixtures used are a combination of 9.1g of Zinc Oxide, 0.38g of Cobalt and 0.09g of aluminum elements. Aluminum (Al) nanopowders are being widely investigated and utilized, mostly due to its higher reactivity than standard micro sized particles. This mixture was synthesized by the method called combustion which makes the mixture suitable for characterization [3]. Since it is well known that adding impurities into a wide gap semiconductor such as ZnO increases a great deal in optical, magnetic and electrical properties. Other materials like the copper, Lead have also

been tested with resulting with various properties. The optical properties of copper have lesser characteristic value than that of Cobalt which has a better "absorption coefficient" value. The Structure and composition of the Zn(Al,Co)O nano powders are analyzed using "Scanning Electron Microscopy" (SEM) and "X-ray Diffraction" (XRD). To understand various absorption and magnetic field properties of this mixture was analyzed through UV-vis Spectrograph. Hence the overall property and characteristics of this mixture is discussed by the characterization methods.

II. LITERATURE SURVEY (BASE PAPERS)

S. Muthu Kumar et al (2012) stated that pure Aluminum and Copper doped Zinc Oxide "nano-powders" are produced by co-precipitation method. TEM analysis had revealed the morphological structure of Zinc Oxide Nano dust to be partly spherical. Powdered Characterization studies had displayed the doping of Copper in the Zinc Oxide structure upto 10.0% of Copper. In conclusion, there was separation of an alternate phase with respect to the formation of Copper Oxide.

N. Mohammad Basith et al (2014) proposed that ZnO nano crystals "doped with Cobalt" and another "not doped", with various mass ratios were produced by combustion method using urea as a fuel assisted with micro waves. The synthesized product where further processed by characterization by the "Energy dispersive X-ray analysis (EDX), "X-ray diffraction" (XRD), scanning electron microscopy" (HR-SEM), "photoluminescence" (PL) and "vibrating sample magnetometer" (VSM). X-Ray diffraction pattern showed that Co-doped Zinc Oxide has a single intrinsic phase.

Pamela Alex et al (2011) demonstrated the manufacture of Nano form of cobalt in quantities of large amount. The outcome of the analysis with characterization techniques indicates the emergence of portions with higher cobalt ion content in mixture and a high ratio of the same in moderate thermal reduction process. This elevates the plenty amount of cobalt dominative features or qualities into the solution made **Alexander et al (2008)** proposed a quick methodology enabling a very efficient and powerful production of materials in nano form. This method is a unfueled mixture in same resolution of various oxidizing agents and fuels like urea.

III. PROPOSED METHODOLOGY

A. Synthesis

The process used is called combustion synthesis.

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This is the best synthesizing method of substances to convert them into nanoized powdered form of the element for verity of advance application which in this case is to make the element suitable or compatible for the process of characterization. The difference between combustion properties and micro-nano structure is detailed. The outputs are significant in all aspects and not only the versatility stand-point, but significantly lead to progressive advantages.

This self-propagating thermal synthesis is used in this project since it is highly effective and costs low with respect to synthesis of the given elements.

B. Characterization Method

XRD:

X-ray diffraction technique is a crucial or significant tool to check the crystalline or lattice structure, “physical properties chemical composition” of mixtures and powders. It may be measured with varied composition characteristics of those crystalline levels.

UV:

“UV-vis spectroscopy” is built on the parameters of electronic transition in molecules on absorbing certain amount of energy from an incident light that enables the excitation from a lower state to excited energy level. To understand the absorption ability of the material UV is used

SEM (Scanning electron microscope):

A scanning electron microscope is a variant type of microscope that processes images of a mixture by “scanning” with electron beams in focus. The electrons act within the mixture, making various signals that attain facts of the topology and composition of the mixture. It is also used to obtain high magnification images, with a good depth, and can also analyze individual crystals or other relevant features. It is used wide in scientific researches, geology, biology and medicine, to name a few.

IV. DISCUSSION AND IMPLICATIONS

ZnO has applications in electro photochemical hydrogen production and heterogeneous photo catalysis. It makes the photo catalyst capable to work with only visible light. Irradiation and will eliminate the need of certain necessities [1]. ZnO Nano particles when doped with various other materials each have its own significant properties. They improvise in catalytically, optical, electrical and absorbability properties. Of it all the Zn-oxide doped with Aluminum has the best advantage with significant transparent and conductive properties in the visible spectrum within the spectrum. This can be used well in the conductive transparent pastes and relevant application. The various doping amount on the ZnO has effect on the band gap in the optical point of view. This customization can come with various optical properties and conductivity characteristics. This may have a very broad aspect on applications stand point. . This is the best synthesizing method of substances to convert them into nanoized powdered form of the element for verity of advance application which in this case is to make the element suitable or compatible for the process of characterization. The outputs are significant in all aspects and not only the versatility stand-point, but significantly lead to progressive advantages

V. SYNTHESIS & CHARACTERISATION PROCESS

The synthesis process is done in nanotechnology lab using the muffle furnace in Saveetha School of engineering. The process used is combustion synthesis which is the best synthesizing method of substances to convert them into nanoized powdered form of the element for variety of advance application which in this case is to make the element suitable or compatible for the process of characterization [3]. The difference among the “combustion parameters” and “product microstructure” is highlighted. This high thermal synthesis is used in this project since it is highly effective, low cost method in respect to synthesis of the given elements [3]. 9.1g of zinc oxide, 0.38g of Cobalt, 0.09g of aluminum elements is mixed and kept it in plaster of Paris. Urea is added as fuel which helps in combustion and then the mixture is kept in a beaker. Before keeping the mixture in a beaker, preheat it and we get the element in the solid state. The color of the compound results in pink color. Keep it close for 1 or 2 hours. The next day put in inside the muffle furnace, a device in which the samples can be tested at high-temperature. The corners or sides of the device heat the content which is placed inside the temperature radiantly so that the material does not come in direct contact with the flame. Muffle furnace is an instrument which is widely used in test laboratories with a compact means to create a high-temperature say up to 1200 degree Celsius to test the different types of characteristics of the material for highly accurate test results. Preheat the muffle furnace to 500 degrees Celsius and put the content inside. After this let it cool and the nest day take it out and we get the element in powder form which is suitable to go for various characterization.

A. UV vis spectroscopy

After the synthesis process, the various types of characterization was done and studied. The UV characterization was done in nanotechnology department, SRM institute of science and technology, Kattankulathur, Chennai. UV- is a technique that measures the absorbance of a solution in the “ultraviolet and visible regions across the light spectrum”; hence the name is UV- Vis. We used the Agilent 8453 spectrophotometer and the chem software. The spectrophotometer has two lamps, deuterium lamp which generates light across the UV region which is between 190.0 and 400.0 nm and a tungsten lamp which emits light on the “visible and near IR- region” of 400 and 1100 nm in wavelength. Switch ON the instrument and let it heat for 45 minutes to make sure that the lamps are nice and hot and the measurement is of a good quality. Different types of cuvettes are used, some of them are like quartz and has a frosted side and the others are plastic types that are regularly used and are disposable. Blank the cuvette and add a ml of water and wait till we get a black which is nice and flat and put the cuvette in the sample holder. The results can be seen in the screen as shown in the fig 1.

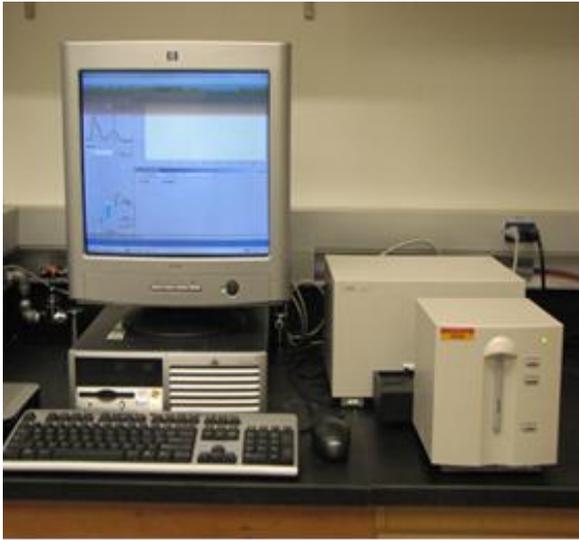


Figure 1: Display showing the UV-vis graph with plot points with the apparatus connected

B. X-ray Diffraction-(XRD)

Powder “x-ray diffraction” is a method that allows the determination of crystallographic density and hence the crystal structure of an unknown crystalline solid. X-ray diffraction technique is a crucial or significant tool to check the crystalline or lattice structure, “physical properties chemical composition” of mixtures and powders. It may be measured with varied composition characteristics of those crystalline levels. In the initial stage, Ensure your sample is dry and start by grinding it in a pestle and mortar to a fine powder of evenly sized particles then spread it uniformly across the frosted well on the glass plate. Take care while spreading it to ensure that the fragile glass is not damaged. Unlock the door of the instrument. To unlock the door, press the door lock button, an alarm will go on until the door is locked again. The “powder” x-ray diffractometer contains three main components and they are x-ray generator, sample holder and detector. The detector is similar to a typical digital camera except that it detects x-rays instead of visible region and inside the x-ray generator electrons are accelerated across a potential difference towards a metal target, as the electron hit the target some will collide and eject the core shell electron from the metal leaving the vacant site. An electron from a higher energy orbital, relaxes into the vacant orbital and a x-ray photon will be emitted. Most of the x-ray produced passes straight through the sample though a small proportion are diffracted by the sample and measured by the detector. This is the mechanism which happens inside the instrument.. Once your sample is prepared, open the door and slide the sample into clips to hold it in place. Lock the door once the sample is in place and beeping will stop. After keeping the sample inside the instrument, open the mini flex software ensuring the diffractometer is on. To collect the pattern click the yellow general measurement button which brings up a new window in which the name and directory of your data is to be saved. After choosing name and directory select the stop x-ray option and press set measurement conditions. Press and run and then start the experiment. After you run and start the experiment the analysis of powder is shown in the screen.

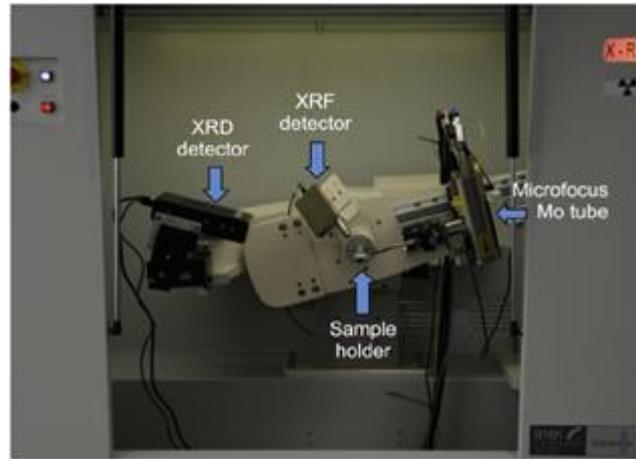


Figure 2: XRD apparatus

C. Scanning Electron Microscopy

“SEM” is also known as “scanning electron microscope”. The SEM is a microscope that uses electrons as a substitute of sunshine to write an image. There are advantages over traditional microscopes. It (SEM) has a giant depth of area, which enables more of a particle to be in focal point at one time. The microscope additionally has so much larger decision, so particles near to one another can also be magnified at much bigger phases.

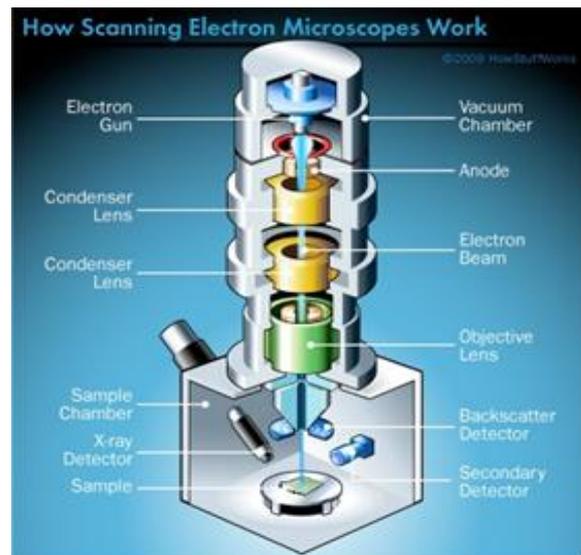


Figure 3: demonstrates the apparatus where An electron gun produces a flow of electrons therefore it comes out with high intensity. This takes place at the head of the microscopic device, where it travels through a “vacuum” medium containing lens

Due to the fact the SEM does not make use of micro-lenses or any type of lenses and the fact that they use electrons to magnify gives it a greater advantage on magnification and provides more detailed control on the “Degree of Magnification” (DOM). This apparatus uses the electrons to create a highly magnified image. An electron gun produces a flow of electrons therefore it comes out with high intensity. This takes place at the head of the microscopic device, where it travels through a “vacuum” medium containing lenses.

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The sample is kept at the other end with respect to the electron gun where, when the electron beam reaches and come in contact with the sample it reflects back "X-Ray" which is caught by the x-ray detectors.[16] The image is created by the detectors which detects the primary and secondary Rays from the mixture, providing a depth field image . This produces the final snaps. This Characterization gives an evident image of the sample with distinctive grayscale contrast displaying the depth of field of which the electron reflects back and gets detected writing an image purely based on the deflected point of the signal. The electrons act within the mixture, making various signals that attain facts of the topology and composition of the mixture and also analyze individual crystals or other relevant features.



Figure 4: SEM instrument with display monitors showing the microscopic image

D. Muffle Furnace

Muffle furnace is an instrument which is widely used in test laboratories with a compact means to make a igh-temperature say up to 1200 degree Celsius to test the different types of characteristics of the material for highly accurate test results. Preheating the muffle furnace to 500 degrees Celsius and put the content inside. After this let it cool and the nest day take it out and we get the element in powder form which is suitable to go for various characterization.

VI. RESULTS

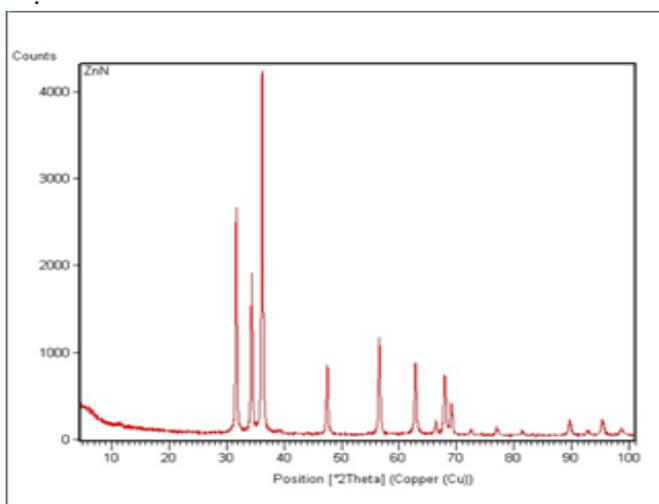


Figure 5: XRD Result for ZnO doped with Al and Co

XRD patterns of synthesized for ZnO doped with Al and Co are shown in "Fig.5" where there are certain high peak. The peak position in the above graph explains about the translational size and shape of the unit cell whereas the peak intensities give details about the electron density in the unit cell.[16] The synthesized nanoparticles are crystalline in nature, because the positions of the peaks in the powder pattern are determined by the size, symmetry and shape of the unit cell and the peak intensities are determined by the pattern of atoms within the cell, the powder pattern is a characteristic "pattern identity" of a period of time.

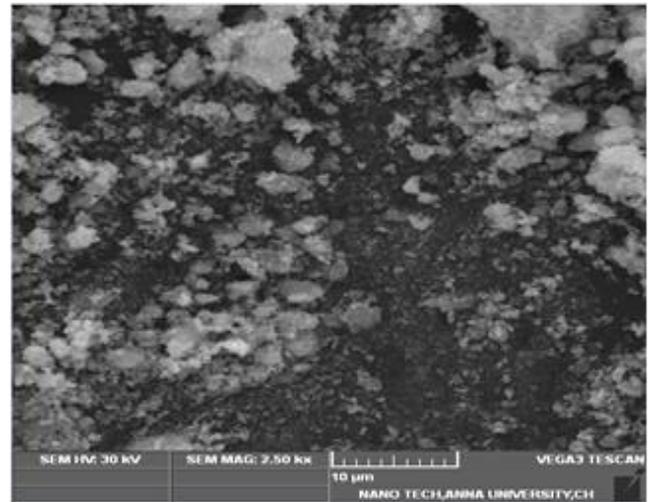


Figure 6: SEM Results for ZnO Doped with Al and Co

This SEM image is a 2D intensity display map in the digital domain. Each pixel on the display corresponds to a point on the sample or mixture. This is proportional to the signal intensity captured by the detector at every specific point and place. The image is zoomed in to a 10 micrometer scale. The dark area of the image cannot be used in retrieving much information .This image can be utilized for morphology study of the particle for further applications.

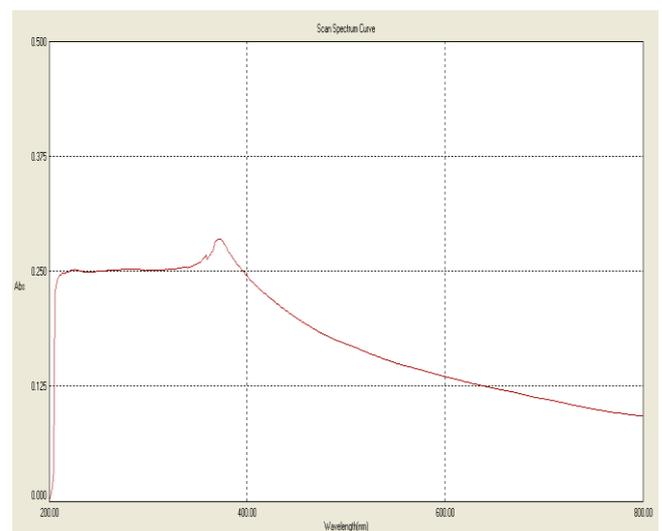


Figure 7(a): Graph obtained by UV-Vis spectrography

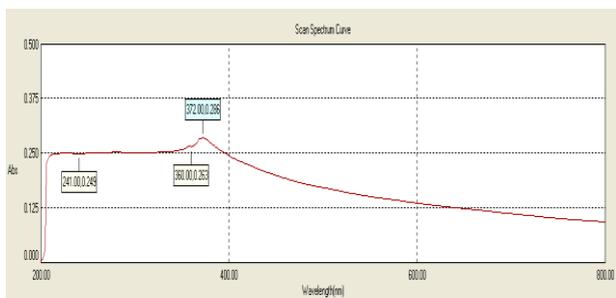


Figure 7(b): Graph with plot points of peak / valley

No.	P/V	Wavelength(nm)	Abs
1	Peak	372.00	0.286
1	Valley	360.00	0.263
2	Valley	241.00	0.249

The outcome graph demonstrates the UV absorption rate with respect to wavelength. Here, the peak value gives the maximum absorption coefficient value of 0.286 at 372 nm wavelength value. This gives an advantage over other element combinations such as the carbon fiber – polymer or aluminum 7075 which gives higher absorption rate on higher wavelength.

VII. CONCLUSION

The proposed research work deals with detailed study of “synthesis and characterization of ZnO doped - Al and Co”. Synthesis of the ZnO doped with Al and Co carried out using combustion method which is also called as top down approach to convert the element into the powder form. Characterization techniques like XRD, SEM and UV VIS SPECTROSCOPY were done to analyze the synthesized nanoparticle. The results of both synthesis and characterization have been discussed in detail. With the results and analysis obtained, this mixture can be used for manufacturing UV-guns, also in the coating of carbon fiber materials used in aircraft’s airframe due to its amazing UV-vis results. Furthermore this mixture can be used in various experiments based on the other results obtained by characterizations.

VIII. FUTURE SCOPE

Semiconducting materials in nanostructures form remain an extensively investigated nanotechnology, subject materials in science current and solid Physics state (nanoscience, physics) and Chemistry. Most of the promising technological applications deal with size dependent properties. As already known, Zinc Oxide has various advantages and applications since it is an efficient semiconducting material, with a gap of 3.3eV between the two bands at moderate thermal conditions [9]. Although wide range of Nano materials are used ZnO also have the best optical characteristics. This can open a huge opportunities to bigger research. So it is important to design and to control better in the fabrication or making of devices, sensors, LED’s, LASER’s, because this research deals with newer devices which can sustain these technologies.

IX. FINANCING AND ETHICAL DISCLOSURE

- Funding:** This study was not funded by any organization or businesses.
- Conflict of Interest:** The authors declare that they have no conflict of interest

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Presely Rajakumar, is a student of Saveetha School Of Engineering (SIMATS) and is doing her bachelors in Electronics and Communication Engineering . He has done researches and projects on CFD aircraft analysis and nano coating airframes . He has done his high schooling in Indian Embassy School , Riyadh . His interest and research include Aviation , airframe designing , material analysis , VLSI

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