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Magnetic, Optical and CHN Analysis of Mixed ZnO- NiO Nanoparticles

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Abstract: ***The synthesis of semiconductor nanoparicles is a growing research area due to the prospective application for the development of novel technologies. I have been studied about the structural, elemental analysis, surface morphology and, magnetic properties of mixed zinc oxide and nickel oxide nanoparticles. A novel microwave assisted solvo thermal synthesis method of defect free nanoparticles has been approached to prepare ZnO- NiO nanoparticles. The prepared nanoparticles were characterized by X-ray diffraction, UV Vis and SEM which carried out to characterize the structural, optical and morphological properties of synthesized samples respectively. VSM analysis is carried out to characterize the magnetic properties of the sample. CHN analysis is carried out to observe the least probability of the presence of carbon, hydrogen, nitrogen in the synthesized sample.***

Keywords: Nanostructures, oxides, solvo thermal method, X-ray diffraction, properties.

I. INTRODUCTION

Nanotechnology can be defined as the design, synthesis, and application of materials and devices whose size and shape have been engineered at the nanoscale. It exploits unique chemical, physical, electrical, and mechanical properties that emerge when matter is structured at the nanoscale. Nanotechnology is the creation and use of materials or devices at extremely small scales. Nanostructured metal oxides are the materials which exhibit significant electronic, magnetic, thermal and optical properties in comparison with their bulk counterparts[1,2]. Transition metal oxide nanomaterials have gained much attention due to their small size effect, high surface to volume ratio which lead to many advantages in applications like gas sensors, catalysts, battery electrodes, photoelectronic devices, magnetic materials, electrochemical devices[3-7]. In past decade, there has been an increasing interest in the synthesis of nanosized crystalline metal oxides because of their large surface areas to volume ratio, high chemical reaction rate, unusual absorptive, properties, surface defects, and fast diffusivities[8]. In many of them, the main objective is to reduce the costs of synthesis and to produce high phase purity materials for technological applications[9].

Based on the higher capacities and better safety nanoscale transition metal oxides have been widely investigated as electrode materials for lithium-ion batteries which become increasingly imperative for high-power applications such as hybrid electrode vehicles and future electric vehicles. NiO and ZnO have attracted considerable attention as anode materials NiO is of high theoretical capacity low cost, environmental friendliness and natural abundance, making it widely studied since it was reported by transcon's group. ZnO has been proven to be useful for solar cells, semiconductor lasers, and other devices. So nano structured NiO-ZnO powders have been investigated as anode materials

II. EXPERIMENTAL

A. General

X-ray diffraction is an ideal technique for the determination of crystallite size of the powder samples. The basic principle for such a determination involves precise quantification of the broadening of the diffraction peaks. The average crystallite size (D) has been calculated from the line broadening using Scherrer's relation $D = K\lambda / \beta\cos\theta$, where the constant K is taken to be 0.94, λ is the wavelength of X-ray used which is CuKα radiation (λ= 1.5406 Å) and β is the full width at half maximum of the diffraction peak corresponding to 2θ.The powder XRD pattern of the prepared nanoparticles is recorded with automated X-ray diffractometer (X-PERT PRO Philips System) operating CuK_α at wavelength 1.54056 Å. The morphology of the powder samples was characterized by scanning electron microscope (SEM) JEOL/EO JSM-6390. The UV spectrum was obtained using LAMBDA-35 UV visible spectro photometer. UV-Visible absorption spectrum of synthesized nanoparticles shows the band edge-absorption peak is found to be at 350 nm. The magnetic nature of nanoparticles are studied using Lake Shore: Model: 7404 vibrating sample magnetometer (VSM). Perkin Elmer, Diamond TG/DTA is used for thermo gravimetric analysis. CHNS elemental anlysers provide a means for the rapid determination of carbon, hydrogen, nitrogen, sulphur in organic matrices and othe type of materials.

B. Procedure

The mixed ZnO-NiO nanoparticles were synthesized using microwave assisted solvo thermal process. nickel acetate, zinc acetate, urea were taken as solute in the molecular ratio 1:3 and dissolved in 50ml ethylene glycol as individually with the help of magnetic stirrer around 15 minutes. Then the three solutions were mixed together stirrered again for 10 minutes. The colour was noted as light green, the solution was kept in microwave oven operated with 2.45GHz and power 800W. Microwave irradiation was carried out for about 10 minutes till the solvent was evaporated completely. The colloidal precipitates obtained was light green colored, then this precipitate was washed three times with distilled water and then washed with acetone two times to remove the organic compounds present, if any. Then the sample was dried in atmospheric air and collected as the yield. The synthesized NPs have been characterized by using X-ray Diffraction (XRD), Scanning Electron Microscopy with EDAX (SEM-EDAX), UV-visible spectroscopy (UV-Vis), Fourier Transform Infrared Spectroscopy (FTIR), Thermal analysis (TG/DTA), Vibrating Sample Magnetometer (VSM) and CHNS elemental analyses.

The formula used to estimate the required amount if substance was,

required substance=MXV/100(in gram unit)

COLOUR AND YIELD PERCENTAGE (%):

Color of the sample was noted. The yield percentage was calculated using the following relation:

Sample	Reaction time(min)	Yield (%)	Colour
Zno-Nio	35minutes	24.14	Light green colour.

III. RESULTS AND DISCUSSION

The powder XRD pattern of the prepared ZnO: NiO nanoparticles is recorded with automated X-ray diffractometers operating Cukα at wavelength 1.54056 A^o. The sample is scanned over 2θ range 20-90^o. X-ray diffraction is an ideal technique for the determination of crystallite size of the powder samples. The basic principle for such a determination involves precise quantification of the broadening of the diffraction peaks. The obtained XRD pattern is shown in fig.

The PXRD pattern is compared well with that available in the literature, which indicates that the material of the samples, prepared in the present study is basically ZnO-NiO. The diffraction peaks at 2θ values of 33.64, 59.80, corresponds to -1, 1, 1, 110 miller planes were selected for calculating the crystalline size for NiO phase. On the other hand the diffraction peaks at 33-58 compounds to 101, 110 miller planes for ZnO. In Fig, the diffraction peaks are considerably broadend that is attributed to the small crystalline size. Small crystalline has relatively few crystal planes that contribute to the diffraction peaks. The spectrum shows the diffraction peaks are considerably small enough to have the quantum confinement.

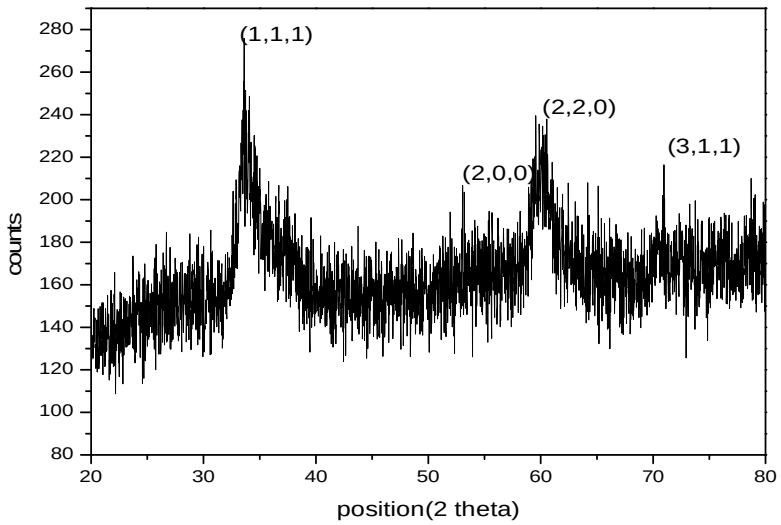

Fig: 1, PXRD graph for mixed ZnO- NiO nanoparticles

The crystalline size can be estimated using D=kλ/ pcosθ where k is a constant (k=0.96)
Using the above relations the crystalline sizes are found to be range of nanometer. The obtained average particle size ranges from 5-7 nm which shows the crystalline size is significantly small. From the JCPDS file numbers 750273, 750272, 750271, 750270 confirms that the prepared particle is cubic monoclinic phase with a, b, c values are 4.219(a=b=c) and the hkl planes.
The morphology of the prepared ZnO- NiO nanoparticles were studied using SEM. SEM picture is shown in Fig. SEM image clearly shows the surface features, which indicates that NiO nanoparticle was successfully prepared. It can be understood that the crystallines prepared are of nearly spherical shape. So that the grain sizes determined by using scherrer formula can be considered as valid. The crystalline grains of the prepared nanoparticles do not directly contact each other and are completely surrounded by a thin layer of material such as amorphous intergranular phase. It forms a kind of continous foam- like network, where the amorphous intergranular phase amount could be increased by the decrease in grain size. We can improve the ordering by further annealing process. The particle size of prepared sample is analyzed using SEM image is also in the range of nanometer. Which indicates the method of solvothermal synthesis is successful one to prepare mixed nanoparticles also.

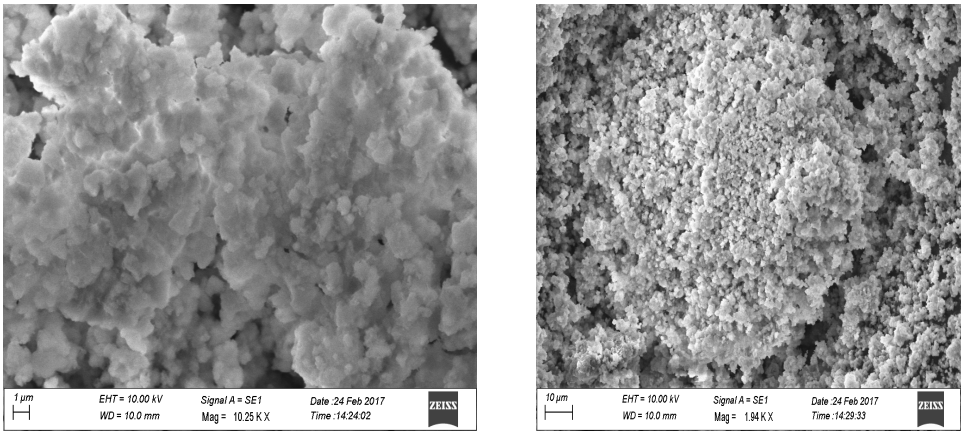

Fig 2 : Sem Image For Mixed Zno- Nio Nanoparticles

The UV-Vis spectrum was obtained using a LAMBDA-35 UV-visible spectro photometer. The absorption and bandgap spectra of prepared nanocrystals obtained in the present study are presented in fig. From the UV-Vis spectra, bandgap (Eg) energies were calculated. The calculated bandgaps corresponding to grain sizes of the samples are 3.2eV.
The absorption of electromagnetic radiation in the ultraviolet and visible regions of the spectrum by the substances results in the electronic structure of ions and molecules through the excitations of bonded and non- bonded electrons. UV- Visible spectrophotometer can be used for both qualitative and quantitative investigations of the samples. UV-Vis spectroscopy is routinely used for the quantitative determination of different analyses, such as transition metal ions, highly conjugated organic compounds, and biological macromolecules and for the surface Plasmon resonance absorption of metal nanoparticles. The wavelength at the maximum of the absorption band will give information about the structure of the molecule or ion and the extent of the absorption is proportional to the amount of the species absorbing the light.
In UV- Vis, high energy electromagnetic radiation in the wavelength range of 100-700 nm is utilized to promote electrons to higher energy orbital's. Since orbital's have quantized energy, only certain transitions can occur in the UV-Vis energy range. The differences in the incident and transmitted beam give information about the frequencies which are absorbed by the sample molecules. Based on absorbance data, the sample chemical structure can be analyzed. The best linear relationship is obtained by plotting (αhv) 2 against hv indicating that the optical band gap of the prepared nanoparticles is due to a indirect allowed transition. The direct bandgap is determined from the intercept of the straight line at a=0, which is found to be 3.2eV for my prepared nanoparticles. Thus, it can be inferred that ZnO- NiO can be activated by visible light. Bandgap value is calculated from Tauc plots which show the variation of bandgap and crystallite size with temperature. Our results indicate a slight blue shift in the direct band edge as the particle size is reduced. Such a blue shift has also been reported in the literature for quantum dots, where the blue shift has been attributed to the quantum confinement effects of nanoparticles which have been attributed to the quantum confinement effect of nanoparticles.

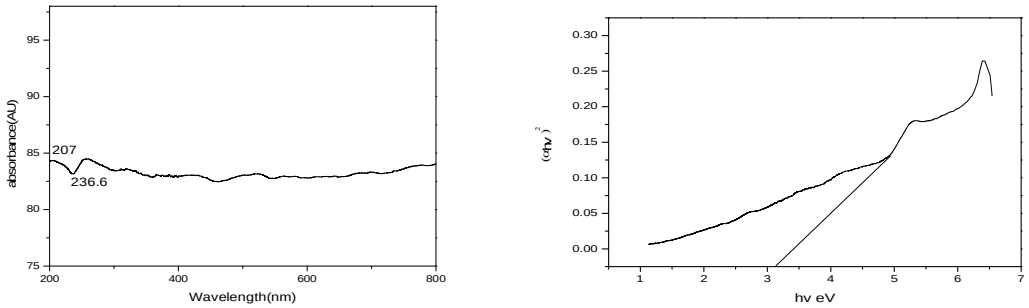

FIG 3 : UV-Vis and TAUC PLOT FOR MIXED ZnO- NiO NANOPARTICLES

The magnetization versus magnetic field(M-H) curves for the ZnO- NiO nanoparticles recorded at 300k are shown in fig and coercivities, saturated magnetization, mass and retentivity values are shown in table. Coercivities, saturated magnetizations, mass. The magnetizations of the samples increase with increasing magnetic field strength. It is also observed that at smaller magnetic fields the magnetizations increase with the magnetic field nonlinearly whereas at relatively higher magnetic fields the magnetizations increase with the magnetic field almost linearly. These are characteristics of ferromagnetic materials. There is no sign of saturation of magnetizations for the samples because ferromagnetic materials usually require very high magnetic field to saturate. Magnetic susceptibility is defined as the ratio of magnetization to the applied magnetic field. For this the linear portion of the magnetization versus magnetic field curve is used. The fraction of spins lying on the surface of antiferromagnetic particles increases with decreasing particle size. Magnetic measurements performed on my prepared nanoparticles revealed that the energy barrier prohibiting spins rotation could overcome by thermal energy.

Co-ercivity	sensitivity	Retentivity(Mr)	Magnetization(Ms)
39.410G	-2.6000emu	10.366E-6emu	2.6761E-3emu

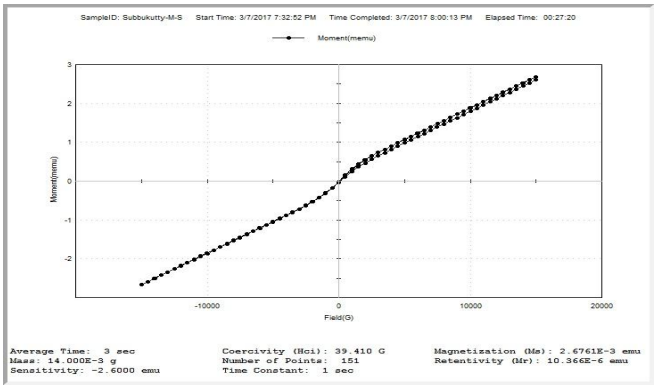

Fig4: VSM graph for mixed ZnO- NiO nanoparticles.

As we know that energy dispersion X- ray spectroscopy is use for the elemental analysis or chemical characterization of all the samples considered. The excess energy of the electron that migrates to an inner shell to fill the newly created hole can do more than emit an X-ray. EDS spectrum will indicate the presence of all kind of elements in a crystal matrix.
But CHNS analysis is the analysis of the presence of carbon, hydrogen, nitrogen and sulphur in the prepared nanoparticles.
In my study I have chosen CHNS analysis in order to analyze the presence of % of existence of least probability of impurities.
The table which shows the presence of % of compounds in the prepared ZnO-NiO nanoparticles.

Carbon	hydrogen	Nitrogen	sulphur
3.014	1.188	0.225	0.0292

IV. CONCLUSIONS

This century has witnessed a escalation in the field of science and technology, for which the contribuition of nanotechnology is much substantial. In the past decade, nanoscale research has opened revolutionary opportunities for a wide number of technological applications due to their special optical, magnetic, electrical and catalytic properties.

In the present work combination with monoclinc structure has been prepared with nickel acetate as a precursor by an efficient method called microwave assisted solvothermal method. The yield of sample was appreciable. And the prepared sample was characterized by XRD, SEM, UV, VSM, CHN. From this, it confirms the as- prepared sample was nanostructures.

According to XRD analysis, the peaks are indexed to be monoclinic structure with lattice parameters a=b=c=4.219A^{o} which agrees well with JCPDS(Joint committee on powder diffraction standards values (750273), (750272). The morphology of the prepared ZnO- NiO nanoparticles were studied using SEM. SEM image clearly shows the surface features, which indicates that ZnO- NiO nanoparticle was successfully prepared. It can be understood that the crystallines prepared are of nearly spherical shape. From the UV analysis, observed absorbtion range is 207nm and 236.6nm and from the Tauc plot observed bandgap is 3.2eV. The magnetization versus magnetic field (M-H) curves shows that the measured co-ercivities, structured magnetization(Ms), sensitivity and retentivity(Mr) values of combination of ZnO- NiO nanoparticles 39.410G, 2.6761E-3 emu, -2.6000emu, 10.366E-6emu respectively. From the CHN analysis table shows the presence of CHN in prepared nanoparticles. CHN analysis strictly shows the purity level of prepared nanoparticles.

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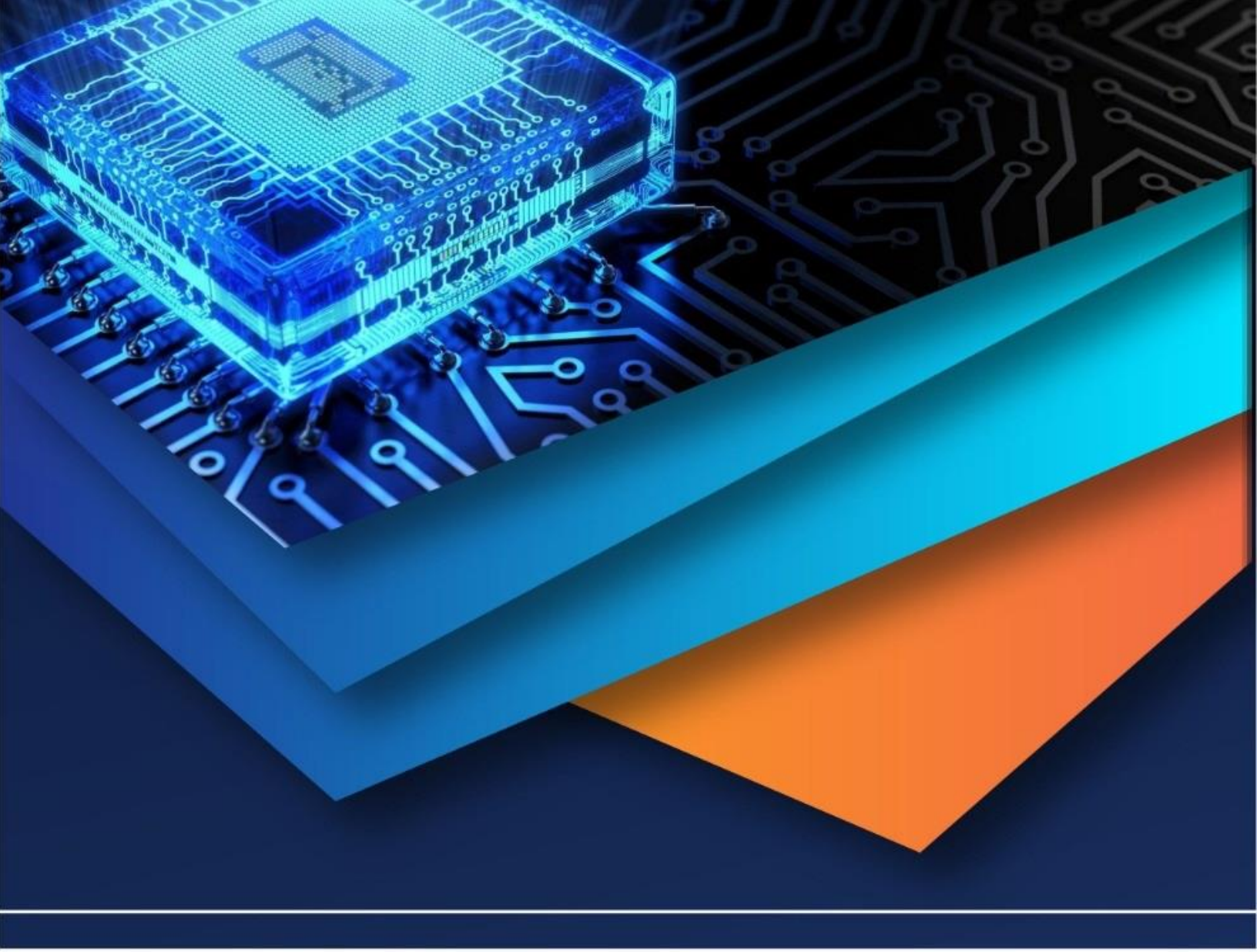

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