Section A-Research paper
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Synthesis and Properties of Pure and Calcium Doped Zinc Oxide Nano Particles

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Abstract: Zinc oxide is a wide band gap semiconductor of the II-VI semiconductor group which are used in emerging applications for transparent electrodes in liquid crystal displays in energy saving or heat protecting windows. Solvothermal synthesis, which has been well developed in the last decade, is a versatile method for controlling the morphology of the well-defined nanostructure materials (e.g., metals, metal oxides, and MOFs). In this aspect, we have an attempt to prepare pure ZnO and Ca doped nanocrystals by solvothermal method. The structural property of the prepared sample was analysed by power X-ray diffraction. The results were reported and discussed here. The prepared samples were optically characterized by UV –vis spectral analysis. The results were reported and discussed here.

1.Introduction

Zinc oxide is an inorganic compound with the formula ZnO. ZnO Crystallizes in two main forms, hexagonal wurtzite and cubic Zinc blend. Calcium nano particles, nanodots or nano powders are spherical high surface area particles. Nano scale calcium chloride particles are typically 10- 100(nm) with specific surface area (SSA) in the 10-75 m^2 /g range. These particles are available in passivated and ultra-high purity and coated and dispersed forms.[15] In earlier study Rochman et al.., (2017) explained the synthesis of ZnO Nano particles made by sol-gel method. Research reveals the greater the PH of the sol-gel will increase the agglomeration of particle and vice-versa. Karam et al.., (2018) conducted an experiment on TiO2 coated ZnO Nano crystals and came to a conclusion that the change of the total active surface area is uniform and single way for PS 5 microns. But, in the PS 1 microns, the uniformity is broken due to the active surface depends on how much quantity we are adding TiO2 NPs.we can still say that the active surface increases by increasing the number of layers or increasing the TiO2 NPs layer thickness.

Datta et al.., (2017) research conducted to check the potential of parthenium hysterophorus leaf extracts for the extraction of zinc oxide Nano particles for their anti-microbial properties. McLaren et al.., (2009) study illustrated the photolytic activity of ZnO crystals. Kumari et al.., (2015) studied influence of nitrogen doping on structural and optical properties of ZnO nano particles. Sutra Dhar et al.., (2016) study used green synthesis of ZnO by thermal method and under microwave irradiation using the aqueous extract tomatoes. A.R. Butt reported that, synthesis CaO nanoparticle by coprecipitation method the X-ray diffraction reveal that the particles size of calcium oxide as 11 nm the absorption spectrum of nanoparticles shows the absorption in UV region with the larger band gap of calcium oxide. Zahra mirghiasi showed that CaO nanoparticle can be produced by direct thermal-deposition of Ca (OH)2 as precursor, at 65080 c for one hour.

In the present study, we have an attempt to prepare pure ZnO and Ca doped nanocrystals by solvothermal method. Pure ZnO and Calcium doped zinc oxide Nanoparticles were prepared by a simple domestic microwave assisted solvothermal route by using ethylene glycol as a solvent, zinc acetate, urea, calcium acetate as a precursor. The prepared samples were annealed at 500 ^{0}c. The structural property of the prepared samples was analysed by power X-ray diffraction and the prepared samples were optically characterized by UV –vis spectral analysis. The results were reported and discussed here.

2. Experimental Details

In this present study we provide the simple methods available for the preparation of nanomaterials in general, along with the preparation of ZnO nanoparticles and ZnO-Cacl2 using microwave assisted solvothermal method. The solvothermal method is analogous to the hydrothermal method, except those organic solvents, instead of water, are used in the synthetic procedure. The synthesis by applying solvothermal method is performed in sealed containers where the temperature can exceed the boiling point of the solvents by the increase of the auto genous pressures, while the route using the water as solvent is also called hydrothermal synthesis. Increase of temperature and pressure in the solvothermal process promotes the solubility and the reactivity of the reagents, which might lead to unexpected reactions as compared with the classical routes [17].

The preparation of pure ZnO Nano particles 1:3 molecular ratio of Zinc acetate and urea were taken and dissolved separately in 150 ml Ethylene Glycol. The stringing condition of zinc acetate, urea solution was added drop wise and the resultant solution were kept in a microwave oven. calcium chloride is used for a dopant at a weight percentage of 5%.the preparation of Ca (5%) doped ZnO Nano particles, zinc acetate and urea 20 was taken in 1:3 molecular ratio along with the amount of dopent. The method of preparation as the same as that used for preparation of ZnO Nano particles. This resultant solution was kept in a microwave oven. The microwave oven irradiation was carried out until the solvent gets evaporated completely and the colloidal precipitate were formed. This colloidal precipitate was washed several times with double distilled water and then with acetone to remove any organic impurities if present the washed samples were dried in atmospheric air and collected as the yield. The collected samples were annealed at 500□ for one hour. The yield percentage was calculated by using the formula.

Yield percentage = ($product\ of\ mass / sum\ of\ the\ masses\ of\ reactants$) ×100

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The annealed samples were used for characterization studies. The required amount of the substances was estimated by using the formula.

A=$M\ X\ V$ /1000

Where, M is the molecular weight of the particular substance X is the concentration in molar units V is the volume of the solvent.

Annealing in metallurgy and materials science is a heat treatment that alters metal causing changes in properties such as hardness and ductility. It involves heating material to above its critical temperature and then cooling. Annealing can induce ductility, soften material, relieve internal stressed, refine the structure by making it homogeneous and improve cold wording properties.

3.Result and discussion

The sample name, colour in before and after annealing, particle size of the 2 samples were noted and presented on table.

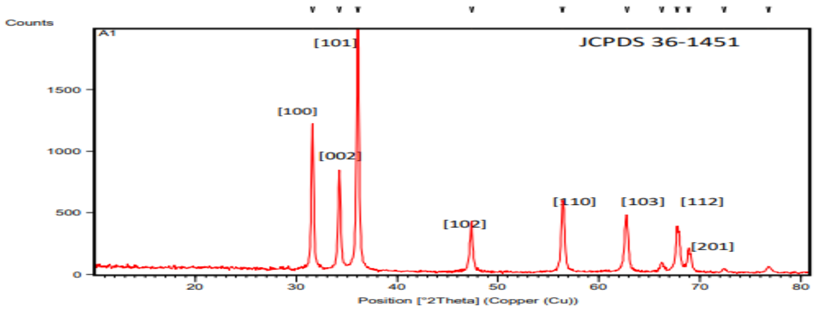
Name of the Samples	Before annealing	After annealing	Annealing time (hr)
Pure ZnO	White	Pure white	2
Ca doped ZnO	Yellowish White	Dark ash	2

Name of the Samples	Oven time (min)	Particle size (nm)
ZnO	45	35.8692
Ca doped ZnO	38	33.7949

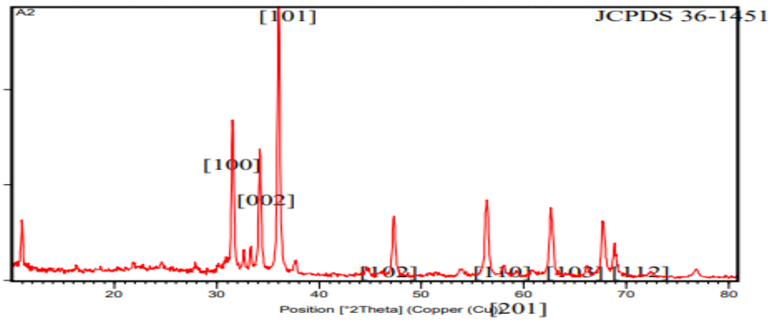
before and after annealing for ZnO nano particles

before and after annealing for ZnO and Ca doped nanoparticles

PXRD pattern recorded in the present study for pure and Ca doped zinc oxide nanoparticles were shown in fig.The observed PXRD data were indexed by matching with the data available for ZnO in the literature JCPDS file (36-1451). The broad peaks observed in doped sample indicate the reduced crystalline size of prepared sample. The particle size of the given sample is shown in table.

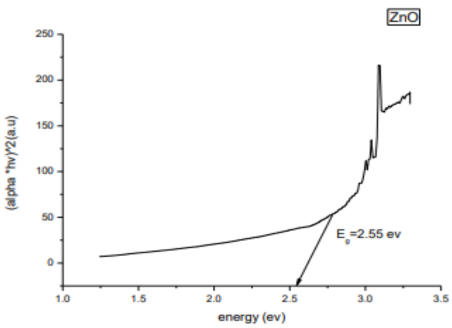


PXRD spectrum of ZnO nanoparticles

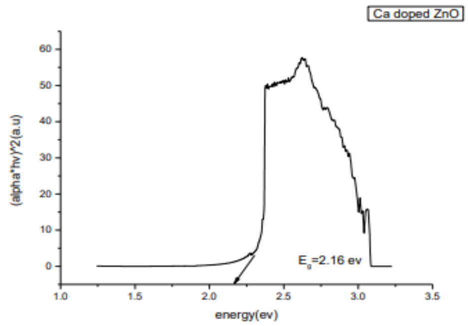


PXRD spectrum of ZnO and Ca doped nanoparticle

UV-Vis spectral analysis has been widely used to characterize semiconductor nanoparticles. The UV-Vis absorption spectrum and energy band gap plot for ZnO and Ca doped ZnO are shown in fig. The optical band gap value of pure and doped nano particles found from tauc plot was 2.55 ev and 2.16 respectively. The optical band gap value for pure nanoparticle is 2.55 ev and also observed that the optical band gap for bulk modulus for zinc oxide is 3.35 ev.it have been reduced.



Tauc plot of annealed ZnO nanoparticles



Tauc plot of annealed ZnO and Ca doped nanoparticles

4.Conclusion

In recent years, there has been considerable interest generated in the study of compound semiconductor with dimensions in the nanometre range. Zinc Oxide nanoparticles as pure and Ca doped nanoparticles as doped were successfully synthesised, using a solvothermal method. Then the sample were characterized by PXRD, UV-Vis spectral analysis. The PXRD results shows that the XRD peaks with the indices using JCPDS 36-1451 file and grain size values are obtained. The optical band gap energy values were evaluated using tauc plot. The optical band gap values of ZnO and Ca doped nanoparticles found from the tauc plot was 2.55 ev and 2.16 ev respectively.

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