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Synthesis, Characterization And Photocatalytic Activity Of Zn Doped Nio Nanoparticles

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Abstract

The photo-degradation of newly synthesized of Zn doped and NiO nanoparticles were done by simple novel sol gel method, this method is low cost method and simple. Effect of different wt% of Zn dopant in NiO was studied on photocatalytic degradation of Toluidine Blue. The study suggested the increased photocatalytic degradation with increased time duration. The synthesized samples were analyzed by surface electron microscopy (SEM) and X-ray diffraction studies.

Keywords: NiO nanoparticles, doping, sol-gel, photocatalytic activity

Introduction

Nanoscience **and Na**notechnology refer to the science and technology of matter , manipulated at the atomic level. Nanoscience made up of two words – Nano and Science. Where nano is a prefix as in nanometer referring to 10^{-9}cm.Therefore the nanoscience is the science of matter in the size regime of nanometer (10^{-9}cm). **Nanoparticles** are particles that size between 1 and 100 nanometers. In nanotechnology, a particle is defined as a matter or object in the size regime of nanometers that behaves as a whole unit with respect to its transport and properties. Particles are further classified on the basis of its diameter and size . Ultrafine particles are the particles that size between 1 and 100 nanometers , fine and small particles are the particles that size between 100 and 2,500 nanometers, and coarse particles cover a range between 2,500 and 10,000 nanometers. Scientific research on nanoparticles is intense as they have many potential applications in medical devices, pharmaceuticals, environmental remediation ,optics, and electronics.

The study of the synthesis , characterization, photocatalytic activity and application of materials in the nanometer size is called nanochemistry. These materials include organic molecule, inorganic molecule, metallic or semiconductor particles. The synthesis of nanoparticles is important because their small size with unusual structure and optical properties that may find application in electro-optical devices and catalysis. The physical and chemical properties of matter depend on the size of matter and chemical composition. The variation of properties depend on the size occurs in different materials differently. Nanoparticles may have better chemical reactivity than bulk material(macromaterial) because on the nanoscale particles have increased surface area of matter. The chemical reactivity of the matter depends on the surface area .

Nanochemistry deals with generating and altering the chemical system ,which develop special and new effect in nanoworld..

Nanotechnology deals with synthesis, characterization and various application of nanomaterials. Nanoparticles may be synthesized by various synthetic procedure for example sol-gel method, Hydrothermal synthesis, Solid state synthesis, microwave method etc. Nanoparticles have enhanced physical properties like optical, electronic ,mechanical etc.[1]

Recently, nanomaterial have been studied by scientists due to its potential CdS application.nanoparticles may be used in catalysis ,batteries, gas sensors, electrochromic films and magnetic material.[2-3]

Nanoparticles of semiconductor materials are a crucial material that can be used in wide range of application such as catalyst, sensors, photoelectron devices, Solar cell ,capacitor ,rechargeable lithium ion batteries.[4,5]

The important parameter that affect the size and morphology of nanoparticles are calcination temp. , pH values of the solution must be controlled to produce pure nanoparticles.[6]

Dyes and other organic material are widely used in industry and daily life and were released in waste water resulting arises various problems to microorganism ,aquatic environments and human beings. These dyes are chemical stable and cannot be destroyed by traditional method. Recently heterogeneous photo catalysis is widely used to decompose targeted pollutant including dye and other organic compound.[7]

Sythesis:-

1. (A) Synthetic Protocol for preparation of NiO and Zn doped NiO nanoparticles

Preparation of undoped, doped and co-doped NiO nanoparticles is done by using sol-gel method.

1.1. Preparation of Pure NiO nanoparticles

A typical synthetic protocol for the preparation of pure NiO nanoparticles have been developed by dissolving 0.1 M Nickel Nitrate in 20 ml n-butanol and 20 ml of polyethylene glycol and mixture was stirred with magnetic stirrer for 24 hrs, then solution of ammonium hydroxide was added until solution reaches pH 11. In this mixture, triton was added to avoid particle agglomeration. The solution was gradually heated at 80^0C until gel was formed. The gel was dried at 200^0C. Pure Nickel Oxide was prepared powder form. The structural elucidation of final product is done by its characterization.

1.2. Preparation of Zn-doped NiO nanoparticles

The typical synthesis procedure for the preparation of Zn-doped NiO nanoparticles with different metal (Zinc Nitrate) loading amount of 0.1, 0.5, 1.0, 1.5, 2.0 and 2.5 mol% was synthesized by the sol-gel method is as follows. Initially, 0.1 M nickel nitrate dissolved in 20 ml n-butanol and add different ratios (0.1, 0.5, 1.0, 1.5, 2.0 and 2.5 mol%) of Zinc Nitrate for doping add 20 ml polyethylene glycol . Solution were stirred with magnetic stirrer for 24 hrs. The solution of ammonium hydroxide was added until solution reaches pH 11 . Triton was added to avoid particle agglomeration. The solution was gradually heated at 80^0C until gel was formed. The Zn doped NiO nanoparticles were obtained after calcination of the product in air at 200°C for 1h and subsequently the product was crushed into fine powder for further characterization.

Results and Discussion:

1.1.1 FTIR spectral analysis:

The study of spectrum gives information of various peak present at different wavelengths. The broad absorption bands due to H-O-H or and N-H stretching is observed at 2921-3323cm^{-1} and is because presence of water on the NiO surface. The absortion band at 1376cm^{-1} is due to presence of bending vibration of alcohol used in this process. The broadness of bands reveals that NiO powder is nanocrystals and crystilized. The peaks in the range of 757-450cm^{-1} is due to Ni-O stretching vibration mode. The absorption bands observed at 1500-1400cm^{-1} are due to presence of finger print regions of bending vibrations of the molecules.

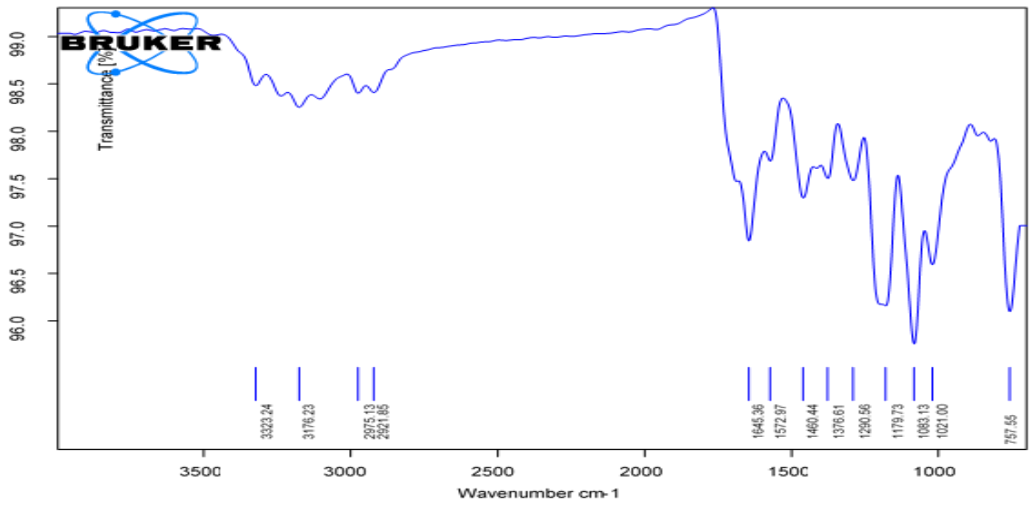

Figure 1: IR spectrum of undoped NiO nanoparticles

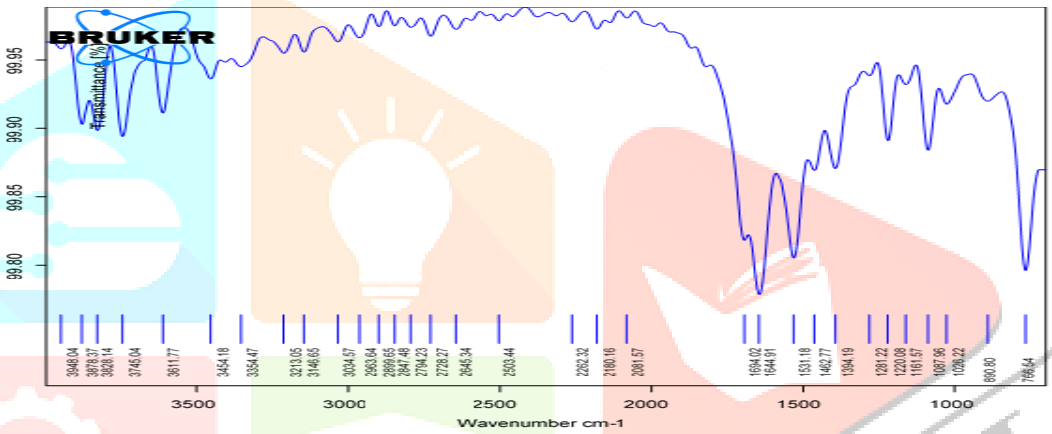

Figure 2: IR spectrum of Zn doped NiO nanoparticles

XRD Study

Powder X-ray diffraction (XRD) pattern of NiO nanoparticles provides concrete knowledge about crystal structure, orientation, average size and phase of various nanoparticles. As predicted by figures (3,4), it has been observed that nanoparticles doped and codoped with variable concentration of % TM metal ions, show different diffraction curves are compared with undoped NiO nanoparticle synthesized under the similar conditions and samples are pre-heated at heat-treated at 400 °C, 500 °C, and 600 °C . The diffraction peaks observed at (111), (200), (220), (311) and (222) planes at the reflacted angles (2Q) = 38.25°, 41.31°, 63.80°, 74.47° and 80.13° indicates phase pure Face centered structure of NiO. The high intensity of (101) peak indicates that the growth of nanoparticles gave birth by the process of crystallization of NiO nanoparticles. Many sharp and intense peaks observed in NiO NPs indicates that the samples are purely crystalline and have polycrystalline face centered cubic structure with space group of Fm-3m and fit well with JCPDS Card no. 78-0429. There is no additional peaks corresponding to the secondary phases of nickel oxides, this indicates that the cubic structure is not disturbed by the doping .

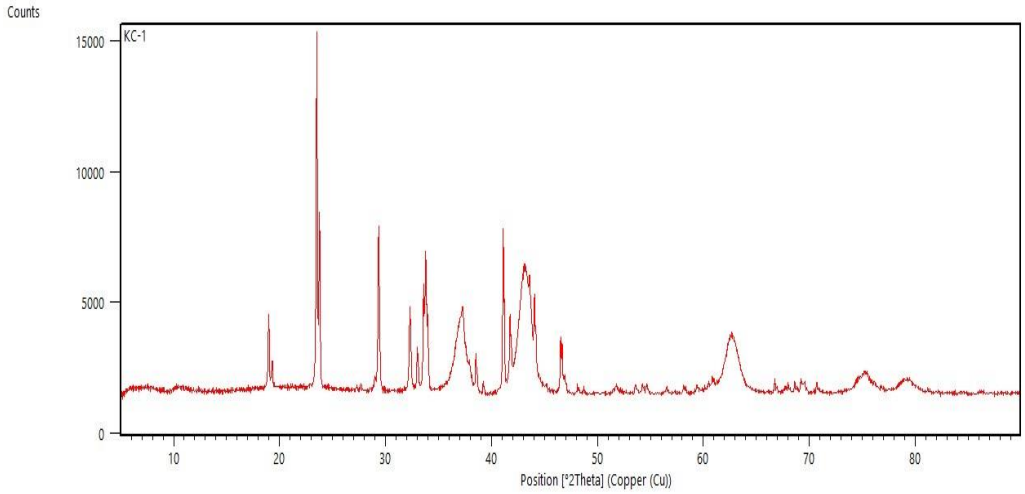

Figure 3: XRD image of pure NiO nanoparticles

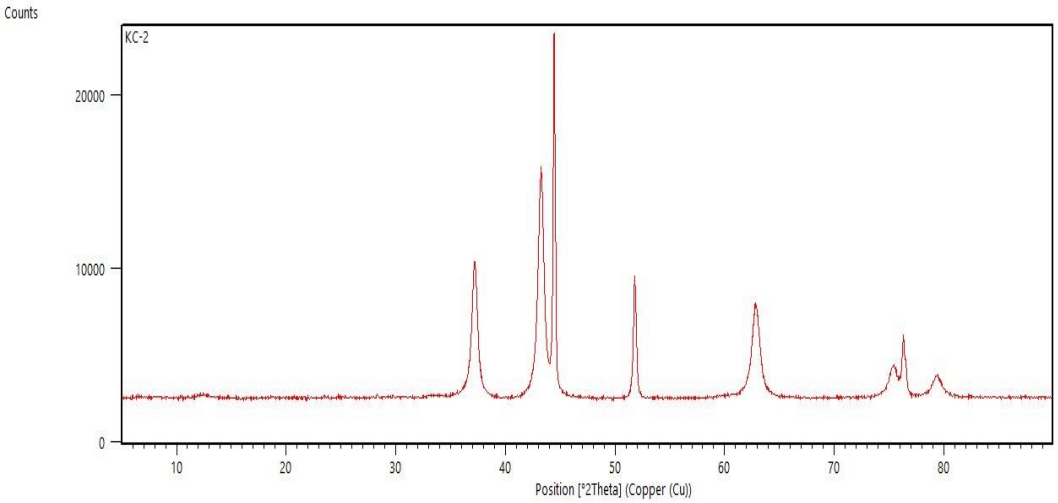

Figure 4: XRD image of Zn doped NiO nanoparticles

SEM Analysis

The topological, morphological measurement and compositional information of the samples was investigated by SEM. Figure 5 showed the SEM micrographs of Zn doped NiO nanoparticles calcined at 400^{o}C and revealed that these nanoparticles were randomly agglomerated with variable size from nanometer to micrometer and it can be clearly observed in the pictures.

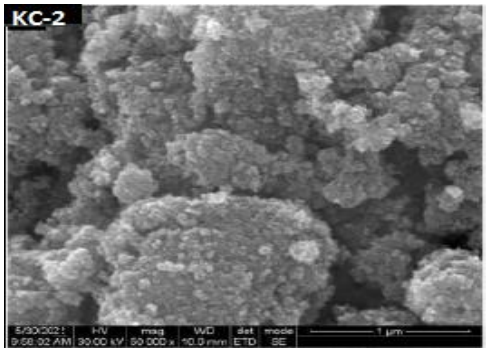

Fig: SEM images of Zn doped NiO Nanoparticles

Photocatalytic Activity:

Nickel Oxide is an important photocatalyst due to its small size, non-toxicity, chemical stability, strong oxidizing power and good photocatalytic activity against various organic pollutants.For this process, 20 ml of dye solution (selected conc.) was taken for experiment and exposed to sun light and during light irradiation the solution was stirred for different time intervals mentioned in table on magnetic stirrer and is valuated by UV visible spectrophotometer to study the photodegradation of organic dyes such as Toluidine Blue (TB) .

Table .Absorption studies carried out at 285 nm to study the photocatalytic activity of Pure and Zn doped NiO nanoparticles on toluidine Blue dye:-

S.No.	**Time interval**	**Toluidine Blue**	**NiO/ Toluidine Blue**	**Zn/NiO/ Toluidine Blue**
1	10 min.	0.943	0.911	0.838
2	20 min.	0.922	0.854	0.712
3	30 min.	0.885	0.789	0.612
4	40 min.	0.768	0.654	0.421

Conclusion:

The present study provides information regarding better and simple methods developed for synthesis of NiO nanoparticles doped with different % Zn dopant and investigated photocatalytic degradation of these compounds on Toluidine Blue . Addition of doping and codoping has enormously increased the photodegredation of dyes . Synthetic formation of these compounds is confirmed by IR, SEM and XRD.

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