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Synthesis and Characterization of NiO Nanopowder by Sol-Gel Method

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Abstract: ***Nanoparticles of nickel Oxide has been synthesized by Sol-Gel method. In the first step Nanosols were prepared by dissolving metal salt in suitable Solvent. On Condensation Nanosols were converted into Nanostructured Gel . Prepared sample was characterized by FTIR, XRD, TGA/DTA Technique. The Size of Nickle Oxide nanoparticles was found to be 8.78 nm calculated by Debye-Scherrer Equation.***

Keywords: Nanosols , Semiconductor , Metal Oxide ,XRD.

1. Introduction

Nanotechnology deals with the Preparation,Characterization, Exploration and exploitation of Nanomaterials. Nanopatriclescan be used prepared by various synthetic techniques for example Solid state Synthesis, Vapour phase synthesis, Hydrothermal synthesis , and microwave method etc. Nano composites are more complex structures which are synthesized by compatibilization of inorganic nanoparticles.[2] These nanostructures have enhanced physical properties like mechanical, electrical and optical etc. methods of preparation are selected on the basis of their applications. Nano materials are mostly compositions. These are also regarded as polymers. The basic principle in the preparation of these materials is to blend particles of a high aspect ratio into polymer matrix.[3]

2. SOL-GEL Method

It is most commonly used technique for the preparation of bulk nanomaterial of Metal Oxides. The method is useful to perform completely controlled reaction by the diffusion of Ionic species or Atomic Species of reactants & products. It requires high temperature and small particle size to bring the reaction partners close sufficiently .Harsh reaction conditions result in thermodynamically stable phase which enable the development of structural properties of the compounds. If this method of synthesis is compared with Organic chemistry where highly sophisticated synthetic routes are required, This method is crude in nature. Liquid phase routes provide most promising methods alternative to Solid state processes. By the help of Sol-gel procedures bulk metal oxide nanoparticles for example (Fibers, films, ceramics, &glasses) can be easily prepared.[4]

Chemistry

It can be defined as conversion of a precursor solution into Inorganic Solid by Inorganic polymerization reactions with the help of water. Precursor may be metal salt (Chloride, Nitrite or sulfate etc.) or an Alkoxide metal organic compound. It involves two main reaction. First is Hydrolysis and other is Condensation. In hydrolysis, alkoxide groups are replaced by Oxygen atoms of water molecules and metal hydroxide isformed. In condensation two hydroxylated species react to M-O-M bonds under release of water molecules (oxolation). Finally reaction between hydroxide and Alkoxide produces M-O-M bonds with release of alcohol molecule.

$M\text{-}O\text{-}R + H_2O$ _____ M-OH+ROH

Condensation:"

M-OH+ H-O-M _____ $M\text{-}O\text{-}M + H_2O$ (oxolation)

M-OR + M-OH _____ M-O-M + R-OH

These Metal Oxide nanoparticles can be used to optimize UV absorption and to increase Stiffness, Toughness and other qualities of polymeric materials. They have been used to enhance the appearance and durability of Poly meric Products. Another example is Photocatalytic reaction. It is attracting a great attention from the view point of Fundamental Science and its Applications.

3. Literature Survey

N. Shahruz&M.M.Hossain from Iran synthesized NanocrystallineTitania powders at room temperature (22^0C). Precursor $TiCl_4$ was used with ethanol solution and argon gas environment without any other materials .Results showed that Anatase was only phase in titanium oxide powders up to 500^0C. The size was controlled and average grain size of anatase particle was obtained in the range of 4nm to 32nm. [1]

In Feb.2012, Nanoparticales of Silica by Sol-gel method were prepared by Ismail A.b .Rehman and Vejayakumaran. They also developed Silica Polymer composites and reported their applications.[2]

Raksha Sharma and co-workers studied effects of particle size and different composition of metal oxide/Poly Aniline Nano composites in 2008. Sizes of nanoparticles found to be 10-20 nm by X – ray diffraction and TEM. Thermal stability and amount of polymer present was detected by (TGA-DTA) analysis.[6]

Jae-Hon Yang, synthesized phospho tungstate titina Nano composites from aqueous media and characterization was carried out by TEM and XRD in 2007. [3]

Applications of nanoparticles in Environmental remediation such as water purification was carried out by Bootharaju & Pradeep T. in 2012 .They explained pathway for the degradation of Pesticides by noble metal nanocomposites. [20]

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A detailed description of milling processes was published by Koch in 2003 by using high temperature zone reaction conditions. The products of the reaction included oxide powder and hydrochloric acid. These Nano powders have large surface area.[15]

Glieter in 1999 introduced a modified process of preparing nanoparticles of normal size distribution.[16]

4. Experimental: Synthesis of NiO Nanopowder:

4.1 Chemicals Required:

- $NiCl_2.6H_2O$
- Absolute Alcohol
- NaOH (Pellets)
- Distilled Water
- Ethanol

4.2 Procedure

First of all,1.5gm of $NiCl_2.6H_2O$ was transferred to 250 ml round bottom flask at room temperature . Then 70 ml absolute alcohol was added in the flask.. The Solution was subjected to continuous stirring. 0.5 gmNaOH was dissolved in 100 ml absolute alcohol in another beaker. This Solution was added in $NiCl_2.6H_2O$ solution dropwise .It was stirred for 2hours. Light green coloured Gel was formed after two hours. Gel was kept for three hours. Filtered and washed with water and Ethanol. Light green coloured precipitate formed. The ppt. was Oven dried at 100^0C for 2 hours. Fine green powder was subjected to Calcination at 290^0C for 30 minutes. Black colourednanopowder of NiO was prepared.

4.3 Characterization

The synthesized compound was characterized by UV-VIS, FTIR, XRD ,TGA/ DTA technique

5. Results and Discussions of NiONanopowder

The Analysis results pertaining to Nano crystalline NiO are discussed separately.

5.1 UV-VIS

UV Spectrum was recorded with Sol of NiO . Sol was diluted with water. The spectrum shows absorbance at 381nm. Fig :1 exhibits absorbance pattern of sample. Another small peak is seen at 510nm.

5.2 FTIR

The FTIR spectrum was recorded with Sol solution and with powder sample after

Table 1: FTIR results of NiONanopowder

	BAND POSITION	RESULT
1	3449 cm^{-1}	O-H Stretching Vibrations
2	1622.8 cm^{-1}	Bending of water molecules
3	672.7 cm^{-1}	Ni-O Bond Stretching
4	1384 cm^{-1}	Presence of CO_3^{2-} anions
5	2897.8 -2975.7 cm^{-1}	Due to organic impurities

heating. Fig :2 exhibits absorption bands. The broad bands at 3449cm^{-1} are due to O-H Stretching Vibrations. The Band at 1622cm^{-1} can be attributed to bending vibrations of water molecules adsorbed physically. The absorption band at 1384 cm^{-1} is due to presence of CO_3^{2-} anions. After heat treatment the band at 672.7 cm^{-1} is due to Ni-O bond. The observed weak bands at 2897.8 -2975.7 cm^{-1} might be due to stretching vibrations of organic impurity.

5.3 XRD

The XRD results recorded for NiO Nanoparticles after heating are shown in Fig:3 . First sample formed by Sol-Gel method was amorphous. After strong heating crystalline nanoparticles were prepared and analyzed by XRD technique. Observed peaks at 31.744 , 45.50 , 56.48, and 62.51 show crystalline nature of NiO Nanoparticles. The grain size was calculated by Debye-Scherrer Equation. It was found to be 8.78 nm & 7.37 nm.

Table 2: XRD results of NiONanopowder

	2Ø	D
1	31,744	2,8165
2	45,501	1,9918
3	56,488	1,6277
4	62,516	1,4845

5.4 TGA/DSC/DTA

Thermal Analysis was done by TGA DSC SDT TA USA at 100-1000^{0}C . This study reveals that first wt. loss occurs at about 90^{0}C with 1% wt. loss due to dehydration. The second wt. loss occurs at 300^{0}C . About 6.5% wt. loss takes place. Reaction is first endothermic then becomeshighly exothermic. At 800^{0}C compound decomposes. Total wt. loss is 23% . At 840^{0}C complete decomposition occurs with heat evolution. Graph is shown Fig:4

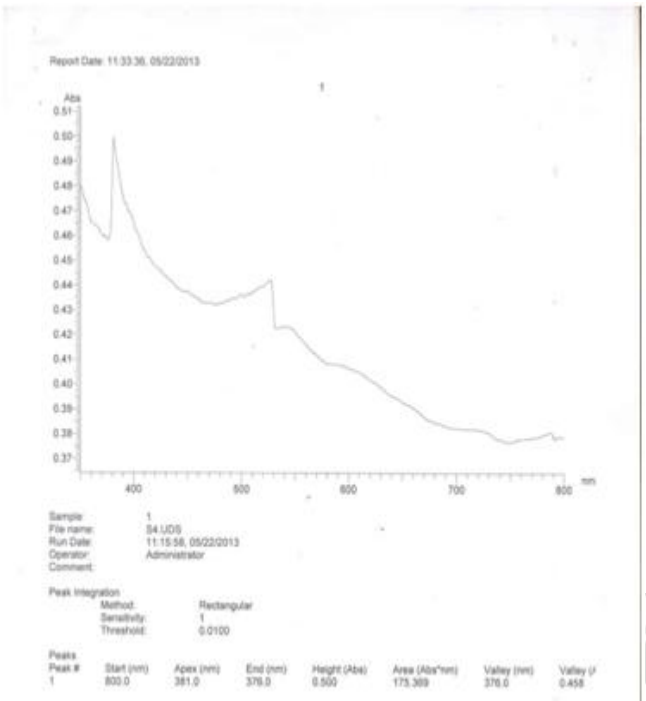

Figure 1

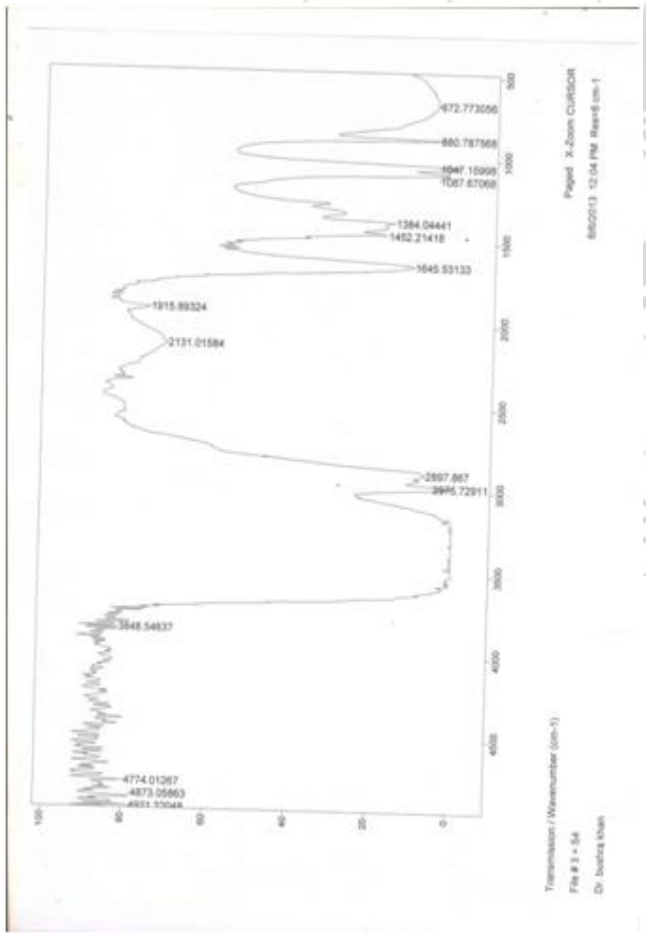

Figure 2

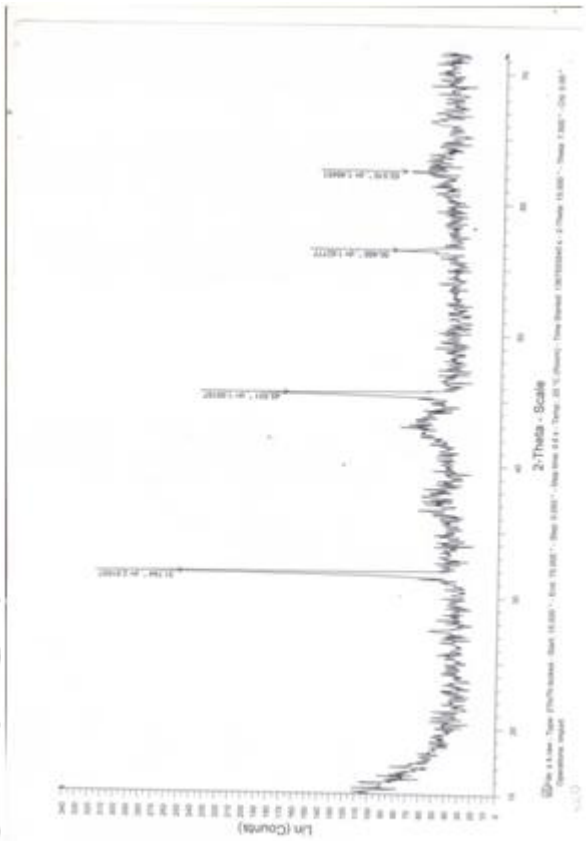

Figure 3

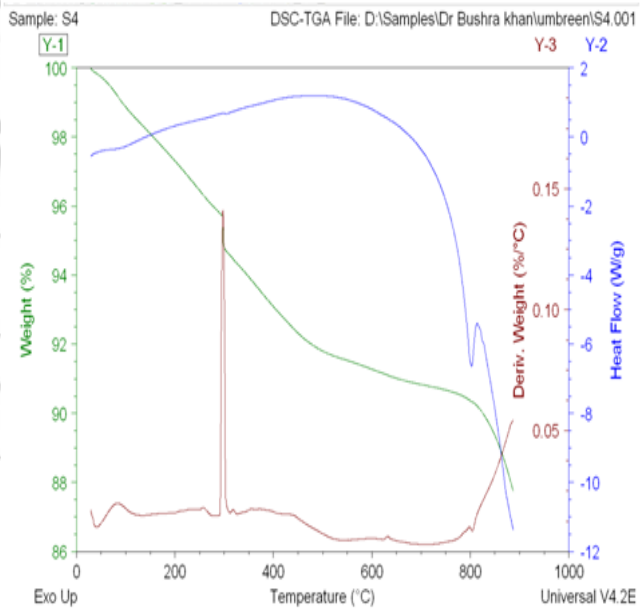

Figure 4

6. Conclusion

Nanoparticles of NiO were successfully prepared by Sol-Gel method and prepared sample was characterized by FTIR, XRD, TGA/DTA techniques. Results have shown that prepared sample contains expected chemical composition. Particle size of sample was in Nano Scale range.

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