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RESEARCH ARTICLE

SYNTHESIS & CHARACTERIZATION OF NICKEL OXIDE NANOPARTICLES BY USING CO-PRECIIPITATION METHOD.

***Pooja Khandagale and Dipali Shinde.**

Department of Environmental Science, New Arts, Comm. & Science College, Ahmednagar 414001.

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Key words:-

Co-Precipitation Method, Nanoparticles, XRD, FTIR, SEM, TEM EDAX etc

Abstract

In the present work Nickel oxide nanoparticles were Synthesized using Co-Precipitation method .It is simple and cost effective method .Nickel chloride hexahydrated was used (as a starting material)& ammonia solution was used for this synthesis. The prepared nanoparticles of metal oxide (Nio) were characterized by using XRD,SEM,TEM,FTIR&EDAX. The average particle size ,crystalline structure were estimated using XRD Analysis. The structural functional groups &optical characters were analyzed by using SEM, FTIR & UV-Visible Techniques. EDAX spectrum that showed the elemental composition of Nickel oxide, FTIR Showed the functional groups present in the synthesized Nickel oxide Nanoparticles.TEM Results confirmed the synthesis of nickel oxide nanoparticles. This simple & cost effective synthesis method will be useful for different industries for the preparation of Nickel oxide nanoparticles. In future this synthesized nanoparticle will be useful for photocatalytic Degradation of toxic dyes.

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Introduction:-

Nanotechnology is the science of production & use of nanosized materials to produce novel products &process.(L.Alan porter et al,2008),Metal oxide Nanomaterial exhibit significantly chemical,mechanical,electronic,thermal,magnetic,catalytic properties & optical properties in comparision with their bulk concentrates & have extensively attracted application.(Q.Li,L-s et al 2007)& (X.Xin,et al 2007).Nickel Oxide (Nio) already has received much attention due to its excellent electrical ,magnetic& catalytic properties.(N.R.Jana,et al 2004).for the preparation of metal oxide nanoparticles many techniques have been used such as Sol-gel method, ultrasonic radiation ,pyrolysis,hydrothermal synthesis precipitation-calcintion method ,leser chemical method ,solid state method ,Co-Precipitation method.(X.Y.Deng & Z.chen et al 2004 , V.R.R.Pulimi & P.Jeevanandam J 2009).

The key applications of nickel oxide nanoparticles are as follows:-

- In preparation of nickel cermet for the anode layer of solid oxide fuel cells
- In lithium nickel oxide cathodes for lithium ion micro batteries
- In electrochromic coatings, plastics and textiles
- In nanowires , nanofibers and specific alloy and catalyst applications
- As a catalyst and as anti-ferromagnetic layers
- In light weight structural components in aerospace

Corresponding Author:-Pooja Khandagale.

Address:-Department of Environmental Science, New Arts, Comm. & Science College,Ahmednagar 414001.

- Adhesive and coloring agents for enamels
- In active optical filters
- In ceramic structures
- In automotive rear-view mirrors with adjustable reflectance
- In cathode materials for alkaline batteries
- Electro chromic materials
- Energy efficient smart windows
- P-type transparent conductive films
- Materials for gas or temperature sensors, such as CO sensor, H₂ sensor, and formaldehyde sensors
- As a counter electrodes [Source: AZo Nano]

Chemical Properties:-

Chemical symbol	NiO
CAS. NO	1313-99-1
Group	Nickel 4 Oxygen16
Electronic configuration	Nickel[Ar]3d ⁸ 4s ² Oxygen[He]2s ² 2p ⁴
CHEMICAL COMPOSITION	
Element	Content [%]
Nickel	78.55
Oxygen	21.40

Physical Properties:-

The physical properties of nickel oxide nanoparticles are given in the following table:

Properties	Metric	Imperial
Density	6.67 g/cm ³	0.240 lb/in ³
Molar mass	74.71 g/mol	

Thermal Properties:-

The thermal properties of nickel oxide nanoparticles are in the following table :

Properties	Metric	Imperial
melting point	1955 ⁰ c	3551 ⁰ c

The Co-Precipitation method used for the synthesis of Nio Nanoparticles is simple & cost effective Method. In the present study is focused on synthesis of Nio nanoparticles using Co-Precipitation method with nickel chloride hexahydrated (as a basic material) & ammonia solution as precipitating material. The samples were characterized by FTIR, XRD, SEM, TEM & EDAX.

Experimental:-

Materials Methods:-

Nickel chloride hexahydrated & Ammonia solution were used for this experiment. All chemical used were of AR Grade brought from qualigens fine chemicals a division of GlaxoSmithKline pharmaceuticals limited Mumbai), & deionised water used for the preparation of solutions.

Synthesis of Nickel Oxide nanoparticle:-

In this synthesis 26.4g of Nickel chloride mix with 100 ml of deionized water, Then NH₄OH solution was injected to the above solution at 25°C under constant stirring, and the resulting mixture was kept at the room temperature for 24hrs for aging.

After the reaction was complete, the resulting light-green solution was filtered, and then washed with deionized water and ethanol for 5–10 times to remove the by-products or impurities, and dried in air at 100°C for 4hr. The as-synthesized material was calcinated at 600°C for 4 hrs and 6 hrs in air to obtain nanoparticles.

Characterization:- XRD, SEM, FTIR, TEM & EDAX.

The size, structure & Morphology of metal oxide nanoparticles were characterized by FTIR. This Spectra of NiO NP's were recorded using (FTIR-180 Shimadzu spectrophotometer) with KBr powder technique in the wavelength range 400-4000 cm^{-1} , XRD (D8 Bruker with CuK radiation= 1.5406 \AA in the range of 2θ degree 20-80), SEM (Scanning electron machine model-JEOL JSM 6360), TEM (Transmission Electron Microscopy was carried out using JEOL-3010) & EDAX (Energy Dispersive x-ray spectroscopy), this spectrum shows the elemental composition of Nickel oxide

Result and Discussion:-

Synthesis of Nickel Oxide nanoparticle:-

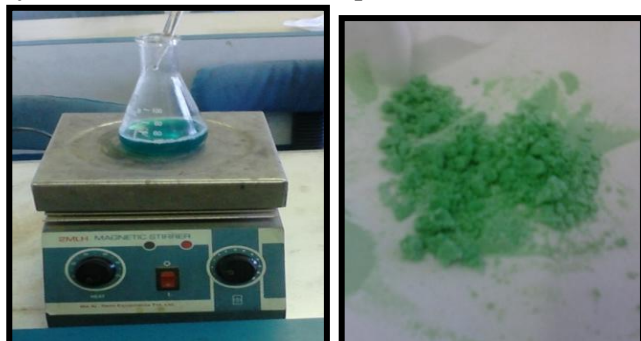


Fig.1:-Synthesis of NiO nanoparticles: **Fig.2:-** NiO nanopowder

From above figure, it observed that addition of ammonia solution in nickel chloride after stirring it becomes homogeneous and formation of green precipitate indicate formation of nickel oxide nanoparticle.

X-ray Diffraction study:-

The prepared nanoparticles were characterized by powder X-ray diffractometer; fig.3 shows that X-Ray diffraction spectrum of NiO samples. The XRD Results showed that prepared powder was Amorphous in nature. The pattern has number of impurity peaks so unable to identify characteristics peak of NiO. Hence unable to calculate the crystalline size.

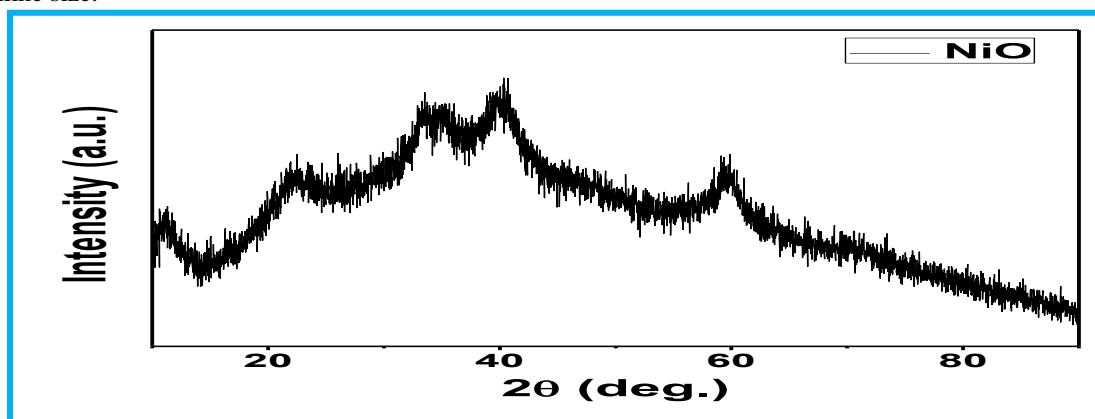


Fig.3:-XRD Analysis Pattern of structure of NiO is Amorphous in nature.

SEM Analysis:-

Scanning electron microscopy is widely used to study the morphological features and surface characteristics of photo catalyst materials. The surface morphological features of synthesized nanoparticles were studied by SEM Technique. Fig 4 shows the SEM image of NiO nanoparticles with magnification 100 Kx & particles was approximately in the range of 100-500 nm. The instrumental Parameter, accelerating voltage, SEM magnification & working diastance are indicated on image. The result indicate that occurrence of particle is Rod shape & highly agglomerated.

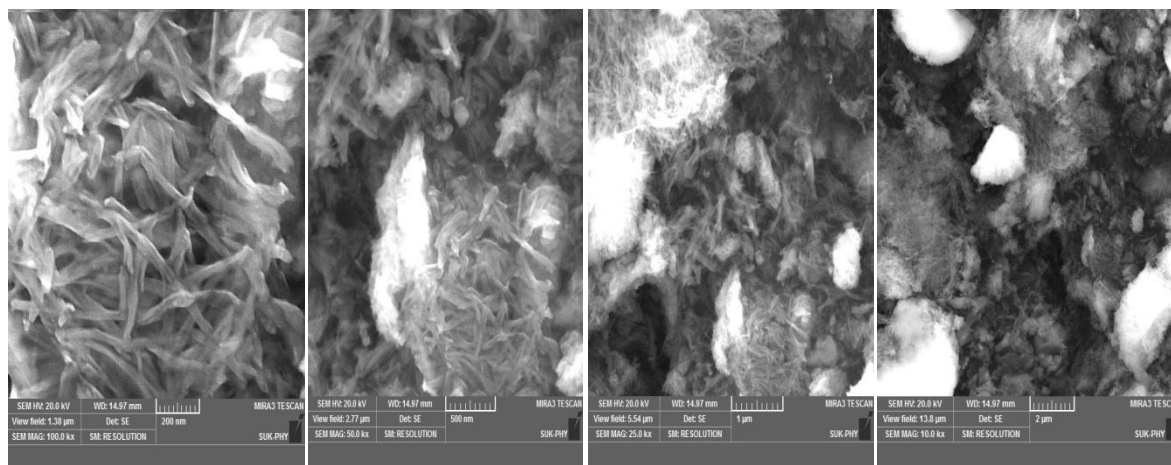


Fig.4:-SEM image of NiO Nanoparticles

TEM Analysis:-

The Transmission Electron microscopic analysis was carried out to confirm the actual size of particles, their growth pattern and distribution of the crystalline. This result showed that the particle size of Nanoparticles were found to be range is **31.87 nm**. TEM Results also confirm the Synthesis of Nio NP's.

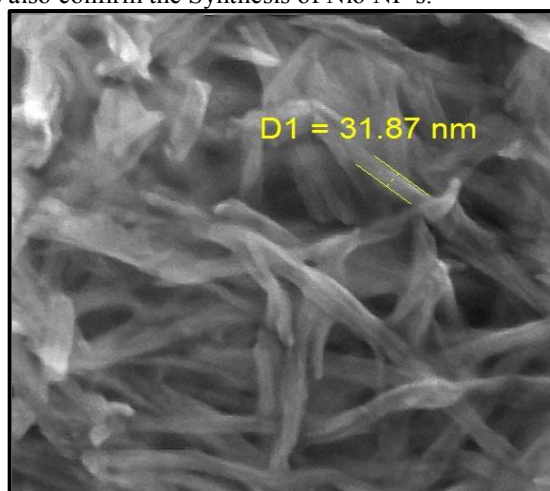


Fig. 5:- TEM Image of the prepared NiO Nanoparticles.

EDAX Analysis:-

The quantitative Compositional analysis of NiO Nanoparticles was carried out using Energy dispersive X-ray (EDAX) spectroscopy measurement .The EDAX analysis also confirm the presence of NiO NP's (Fig.6).The presence of impurities with nickel oxide nanoparticles are also confirmed by EDAX analysis and it was given in Table 1.

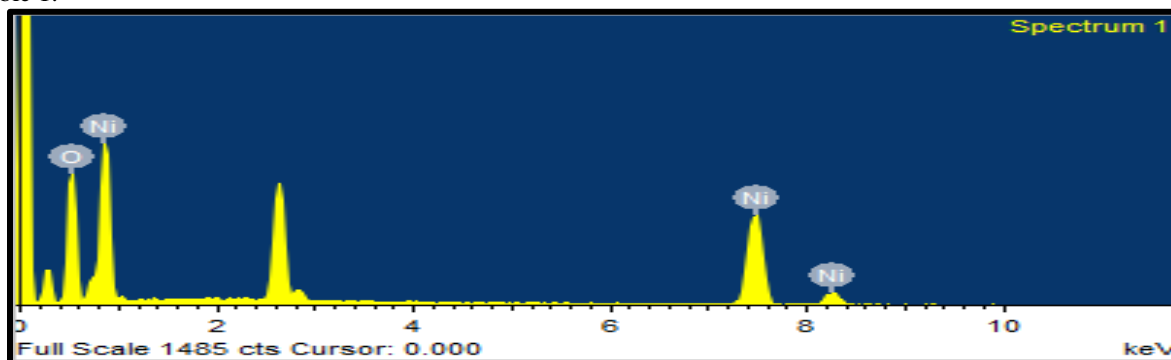


Fig. 6:- EDAX Spectrum of NiO Nanoparticles.

Spectrum processing : Peaks possibly omitted : 2.628, 2.841, 6.105 keV Processing option : All elements analyzed (Normalised) Number of iterations = 3

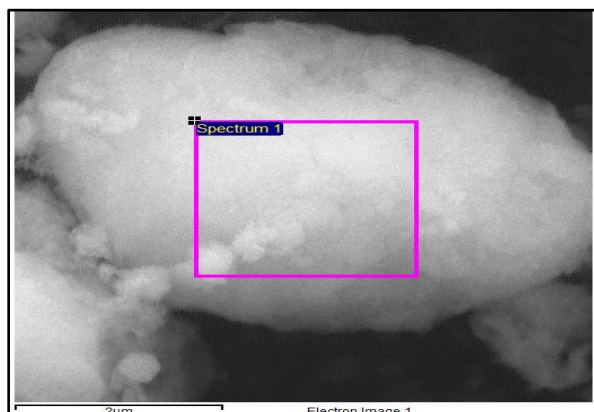


Fig. 7:- EDAX image of NiO Nanoparticles

Element	Weight%	Atomic%
O K	37.45	68.72
Ni K	62.55	31.28
Totals	100.00	

Table 1- EDAX analysis of element NiO

FTIR Analysis:-

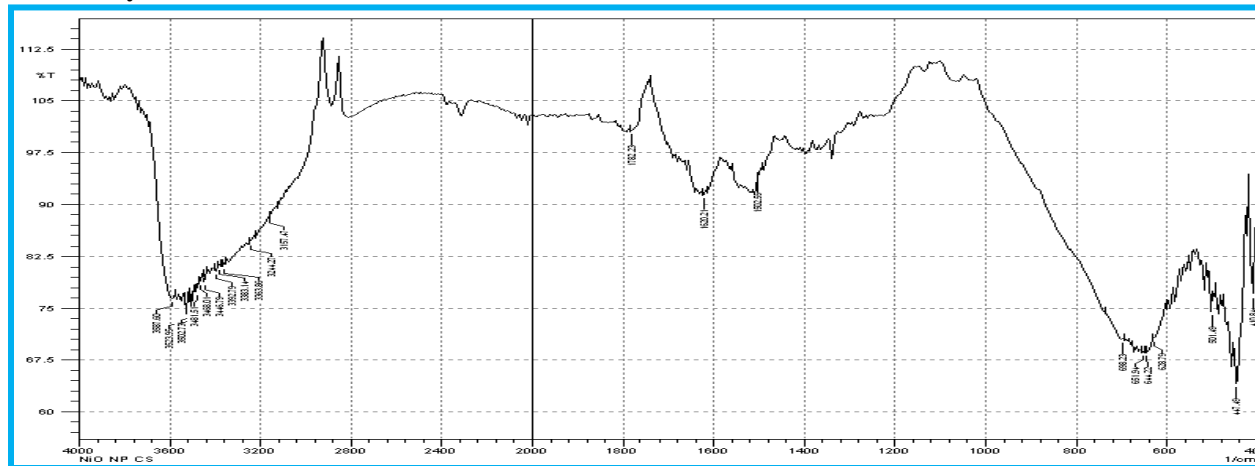


Fig 8 :-FTIR analysis pattern of Nio nanoparticles

Indicates the FTIR spectra of NiO Nanoparticles in the range of 700-400 cm^{-1} , The FTIR spectrum shows the characteristic peak at 410.84 cm^{-1} , 447.49 cm^{-1} , 459.06 cm^{-1} , 482.20 cm^{-1} , 501.49 cm^{-1} , 516.92 cm^{-1} , 547.78 cm^{-1} , 628.79 cm^{-1} , 651.94 cm^{-1} , 698.23 cm^{-1} . FTIR spectrum of NiO nanoparticle showed significant absorption peak at 447.49 cm^{-1} is due to Co-O stretching vibration mode.

Conclusion:-

The present study reported on Nickel oxide nanoparticles have been successfully synthesized by co-precipitation method (using nickel chloride & ammonia). The synthesized nanoparticles were characterized by XRD, SEM, TEM, FTIR & EDAX Analysis. The SEM Result confirmed that synthesized nanoparticles were in rod shape. From TEM result the particle size of Nanoparticles were found to be range is 31.87 nm. TEM Results also confirmed the Synthesis of Nio NP's.

The FTIR Spectrum confirmed the presence of Nio nanoparticles. The elemental nickel & oxide molecules are confirmed by EDAX Analysis.

This simple & cost effective synthesis method will be useful coming for different industries for the preparation of Nickel oxide Nanoparticles. In future this synthesized Nanoparticle will be used for photocatalytic Degradation of toxic dyes.

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