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Synthesis and Characterization of Nickel Oxide Nanoparticles Synthesized via Chemical Precipitation Method

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Abstract:
The Nickel oxide Nanoparticles were synthesized from Nickel Nitrate Hexahydrate aqueous solution under the chemical method at 90°C. The average crystallite size was calculated from De-Bye Scherrer's equation. FESEM, EDX, XRD were used to characterize the structural features of the product. FTIR spectra confirmed the adsorption of the Nickel oxide nanoparticles. In addition, UV-visible absorption spectra were employed to estimate the band gap energy of the Nickel oxide nanoparticles. This method may be suitable for large scale production of Nickel oxide nanoparticles for practical applications. The effect of Nickel oxide nanoparticles is screened in vitro for antimicrobial activity by Disc diffusion method. The bacterial organisms used in this study are E.coli, Bascillus Subtilis and also fungi Aspergillus Niger. The observed inhibition zones for these nanoparticles are in the range of 8mm for E.coli and 7mm for Bascillus Subtilis and 7mm for fungi Aspergillus Niger. The cytotoxicity activities of Nickel oxide nanoparticles screened by MTT assay. We have screened for one type of cancer cell-line i.e MCF-7(Breast Cancer), Nickel oxide nanoparticles obtained IC_{50} values in the range of 32.59ug/ml for MCF-7 cell line.

Keywords*: Nickel Oxide Nanoparticles, SEM, EDX, XRD,FTIR,UV-Vis, Disc diffusion method, Cytotoxicity.*

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I. Introduction:

Nanoparticles are being synthesized globallyowing to various exciting and unique propertieswhich facilitates its ther exploitations in completely unrelated fields such as nanomedicine, photocatalysis etc.The main use of inorganic oxides for nanoparticles are their stability, robustness, and long shelf life.

Producing nanoparticles involves breaking up a bulk material into atoms or ions and then allowing those atoms or ions to condense into nanoparticles. Ni nanoparticles are having applications as catalyst, conducting and magnetic materials.Ni nanoparticles in particular being cheap, need mild reaction conditions for high yields of products in short reaction times as compared to the traditional raney-Nickel.NiO is an anti-ferromagnetic Oxide semiconductor with P-type conductivity due to its wide band gap energy range from 3.6-4.0evIt also having applications as smart windows, spin valves, giant magneto resistance(GMR) sensor, solar cell etc.

II. Materials:

All of chemicals used in experiment are of analytical grade and used as purchased without any purification. Nickel nitrate hexahydrate Ni $(No_3)_2{\cdot}6H_2O$), of 98% purity is used. De-ionized water used as a solvents. Sodium hydroxide (NaOH) is used to adjust the pH.

Synthesis of nickel oxide Nanoparticles:

Nickel nitrate hexahydrate $Ni(No_3)_2.6H_2O$ and sodium hydroxide NaOH were each dissolved separately in deionized water to form the liquid media of the desired concentrations of 0.05M (4.575g/500mL)and 0.1M (2g/500mL) for sample A and B respectively the ratio of the concentrations was 1:1 ($Ni(No_3)_2.6H_2O$: NaOH). The nickel nitrate hexahydrate was slowly added drop- wise to NaOH solutions under vigorous stirring at room temperature, forming transparent white solutions, then inserted into an electrical oven at 90°C for 2 hours. These solutions were reacted to produce nickel oxide precipitates. Following the precipitation, the solution was centrifuged at 3000 rpm for 30 minutes. The supernatant was then removed, and the precipitation which contains nickel oxide was obtained. Finally, cnickel oxide was grinded with mortar to be shaped into powder

UV Absorption and Reflection Spectra

The optical property is one of the vital properties for the assessment of photo-catalytic-activity of the light-green NiO NPs. Due to the optical adsorption of radiant energy, the spectrum including band-gap energy of the metal oxide could be achieved. The UV/Vis spectra of the NiO NPs solution were recorded in the visible range (400–800 nm) and a broad absorption band at around ~299.0 nm were found respectively (Fig. 1a). On the basis of the maximum level band absorption, the band-gap energies of the NiO NPs were calculated using Tauc's equation (1).

$(\alpha h\upsilon)^n = A(h\upsilon - E_g)$…… (1)

where, α is the absorption coefficient, A is the constant related to the effective mass of the electrons, r = 0.5 (Direct transition), h = Plank's constant, v = Frequency, and Ebg = Band-gap energy.

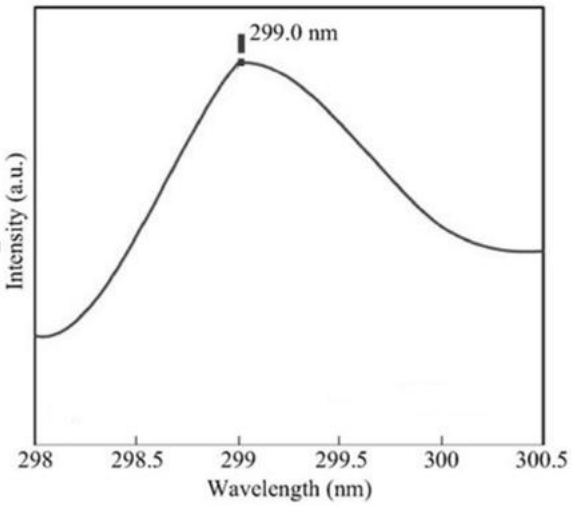


Fig.1. UV-visible of synthesized ZnO nanoparticles

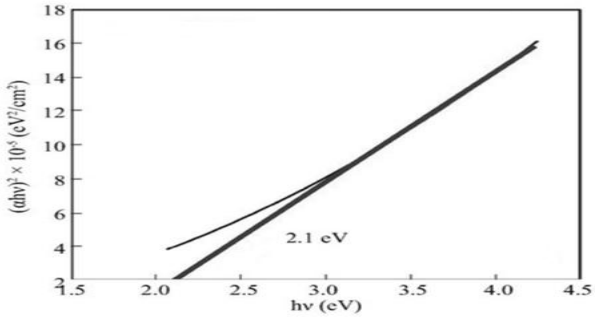


Fig.2. Tauc plot for determination of band gap

According to the direct band-gap rule, (αhv) 2 = A(hv − Ebg), curve of (αhv) 2 vs hv were plotted and then extrapolated to the axis. From the extrapolated curve, the band-gap energies for NiO NPs were calculated as 2.1eV respectively (Fig. 1b). No additional peaks associated with impurities and structural defects were observed in the spectra that indicated the crystalline characteristics of the prepared NiO nanoparticles.

FTIR Spectrum of nickel oxide Nanoparticles

FTIR spectroscopy uses Michelson interferometer to produce an interferogram and the spectrum of CuO is shown in the Fig.3. It has been shown that as particle size decreases, increase in frequency for the bond is observed in nanoparticles. Bands at 416.35 cm^{-1} are assigned to the stretching vibrations of Cu-O. The stretching frequency of bulk CuO is 424 cm^{-1}. Here a blue shift is observed in that frequency i.e., that frequency due to quantum confinement Three intense bands were centered at 1384.34 cm^{-1}, 1041.54 cm^{-1} and 1556.58 cm-1 and are attributed to the stretching vibrations of C = O, C = C and C-H groups in acetate species, which suggests its presents as absorbed species in the surface of nanoparticles. The broad absorption peak centered at 3423.61 cm-1 and 1626.40 cm1 corresponds to O-H stretching and bending frequencies of H_2O, indicating the existence of water in the surface of nanoparticles [32].Variations of the peak positions of CuO [33-35] are presented in the Table 1.

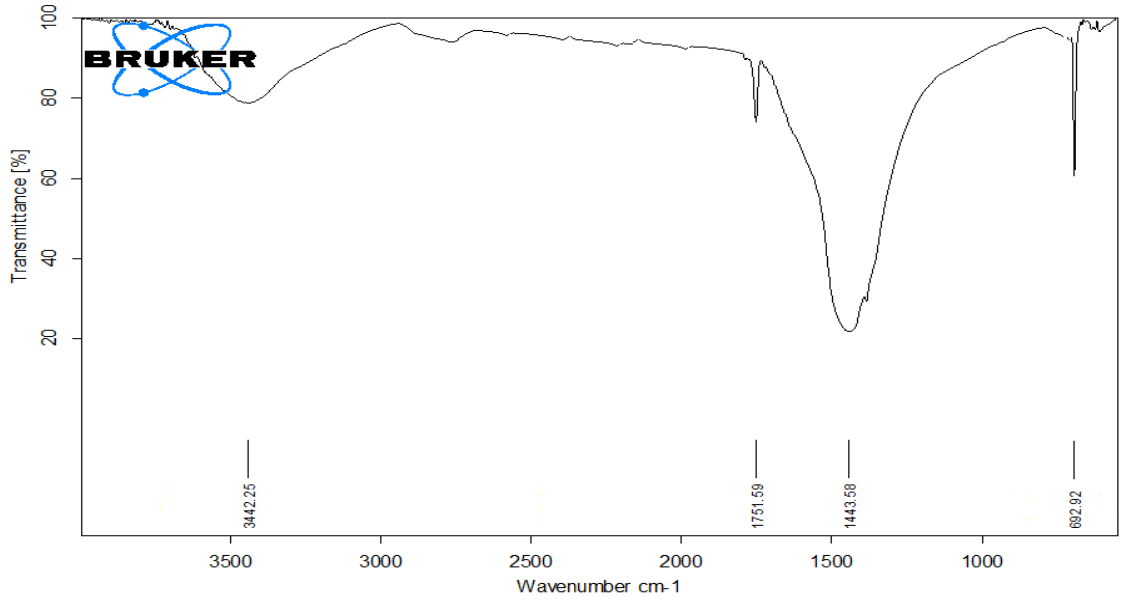


Fig.3 FTIR Spectra of nickel oxide nanoparticles

The peak observed at 622.92 cm-1 is presence of nickel oxide nanoparticle. The broad absorption peak centered at 3423.01cm-1 corresponds to O-H stretching of water indicating the existence of water in the surface of nanoparticles.

S.No	**CuO (cm^{-1})**	**Vibrational modes**
1	3423.01	OH
2	1482.84	OH deformation
3	622.92	Stretching of CuO
4	1781.01	C-H

Table.1. Comparison of Vibrational modes of nickel oxide nanoparticles

Powder XRD analysis

Fig.4 shows the crystal structure and purity of the synthesized NiO nanoparticles were determined by powder XRD. Fig.4 depicts the xrd pattern of NiO nanoparticles. The prominent diffraction peaks at 2θ values 30.89°, 33.92°, 35.65°, 56.32°, 62.53°, 66.72°, 68.87°, 76.07° are associated with [110],

[111], [022], [202], [113], [020], [220], [222] planes respectively. The evolved diffraction peaks could be indexed to a monoclinic phase of CuO with lattice parameters a= 4.683 Å, b= 3.421 Å, c= 5.129 Å and corresponds to the JCPDS file no 80-1268. The peak intensities and width of the spectrum indicates the presence of nanoscale crystallites. The absence peaks corresponding to by products such as indicate the purity of NiO nanoparticles.

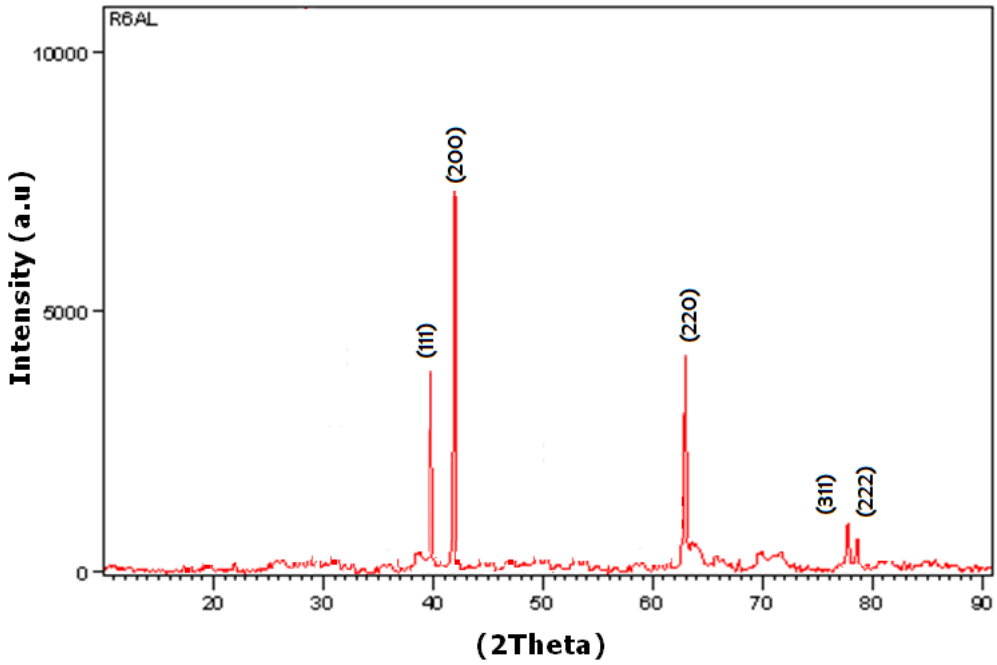


Fig.4. XRD spectra of Nickel oxide nanoparticles

Inter planar d-spacing was calculated using Bragg's Law equation (Table 2):

$2d\sin\theta = n\lambda$ ……. (2)

where, θ is Bragg's angle of diffraction, λ is X-ray wavelength, i.e. 1.5406 A˚ and n = 1. Further, particle size was calculated from the intense peak corresponding to (101) plane using Scherrer formula. The crystallite size is calculated using Debye Scherrer equation

$d= 0.9\ \lambda/ \beta\cos\theta$…….. (3)

Where d is the average crystallite size (nm), K is the grain shape factor (0.9), λ is the X-ray wavelength (nm), is the line broadening at half the maximum intensity in radians, and Θ is the Bragg diffraction angle of the 2Θ peak. The average crystallite size was estimated to be 20-50nm.

Field emission Scanning Electron Microscope (FESEM)

Fig.5 shows the surface morphology and particle size of the synthesized nickel oxide nanoparticles. It is clear from the images that the size of the nickel oxide nanoparticles is ranging from 20-50 nm. The obtained products are composed of nearly flower shaped morphology with average size in the range of 50 nm.

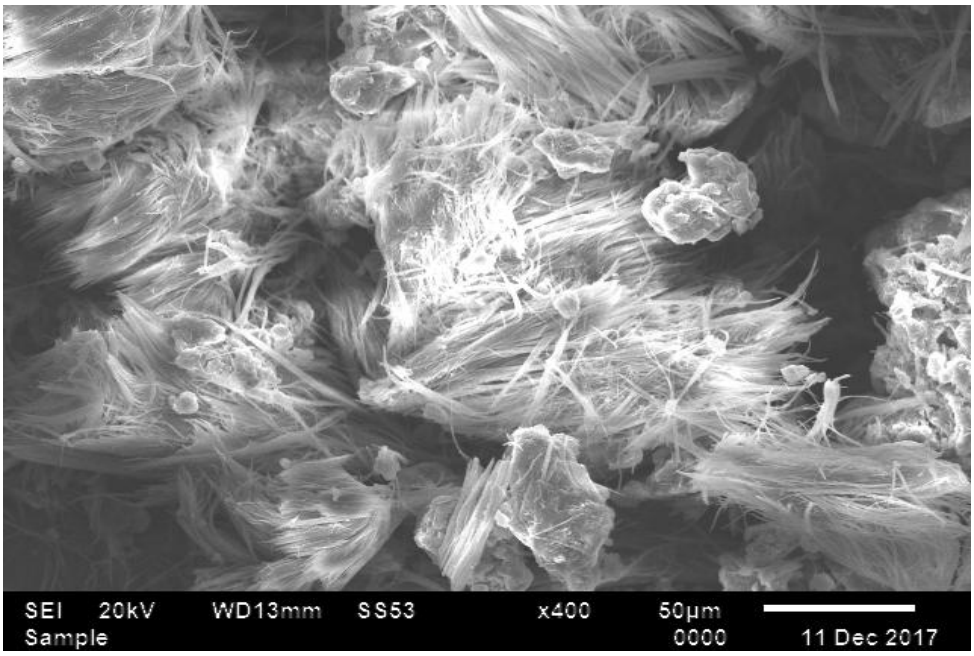
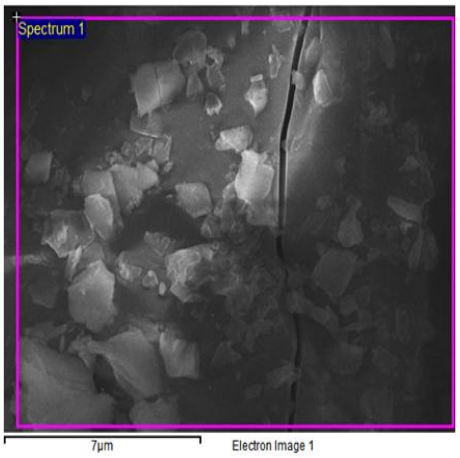


Fig.5. FESEM image of nickel oxide nanoparticles

Energy Dispersive X-ray Spectroscopy (EDX):

EDX spectrum, Figure.6, plot not only identifies the elements corresponding to each of its peaks, but the type of X-ray to which it corresponds as well. The higher a peak in a spectrum, the more concentrated the element is in the spectrum. Spherical shaped morphology is observed in the micrograph of nickel oxide nanoparticles.

Element	Weight%	Atomic%
O K	52.66	80.90
Ni K	47.34	19.10
Totals	100.00	



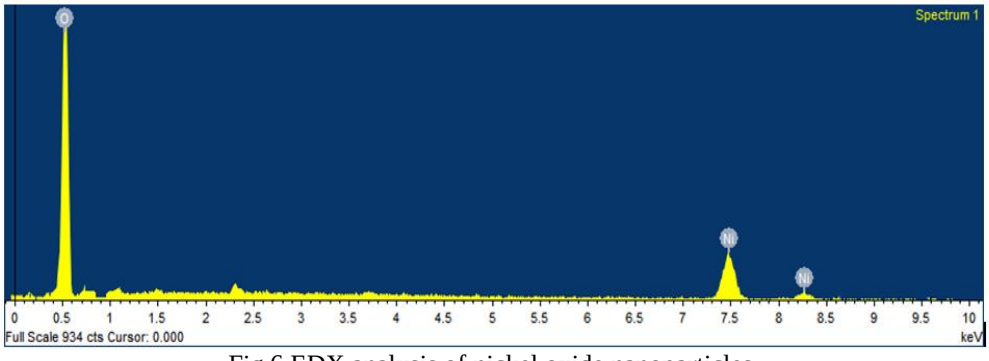


Fig.6.EDX analysis of nickel oxide nanoparticles

The dried powder of the sample was analyzed on Energy Dispersive X-ray Analysis (EDX) technique. The peaks have confirmed the presence of nickel, oxygen. The average atomic weight percentage ratio of Cu, O. CuO nanoparticle was 58.22: 41.78. The energy ratio (in keV) 25.97: 74.03. The data's are presented in the Fig 6. The presence of doped rare earth element in nickel oxide nanoparticles was confirmed by the analysis.

Antimicrobial Screening of nickel oxide nanoparticles

The nickel oxide nanoparticles in vitro for antibacterial activity against E.coli, B.subtilis and antifungal activity against A. niger by Agar-well diffusion method. The antibacterial and antifungal activities of nickel oxide nanoparticles are listed in table 2.

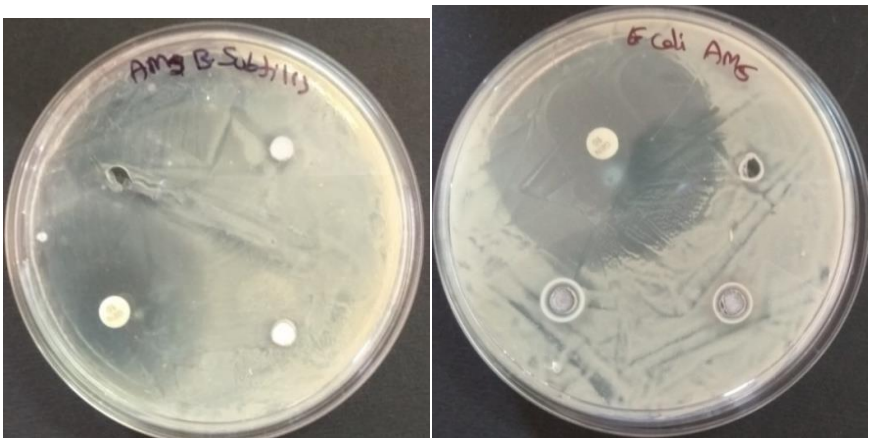


Fig.3. Inhibition zones for nickel oxide nanoparticles against. B. SubtilisE.coli

Fig.4. Inhibition zones for nickel oxide nanoparticles against A. niger.

Bacteria	**Inhibition zone (mm)**
E. coli	08
B. subtilis	07
Fungi	**Inhibition zone (mm)**
A. Niger	07

Table.2. Anti-microbial activities of nickel oxide nanoparticles

The nickel oxide nanoparticles showed good antibacterial activity against E.coli and B.subtilis andAnti-fungal activity against A.niger

Cytotoxic studies of nickel oxide NPs

The synthesized complex is screened for their cytotoxicity (MCF-7, cell lines).[27-58]from the data, it is observed that the complex displayed their cytotoxic activities as IC_{50} (μg/mL) against breast cancer MCF-7,The IC_{50}values of the complex are listed in table

Conc(µg/ml)	% cell survival	% cell inhibition
0.1	91.40216955	8.597830454
1	94.45560466	5.544395339
10	63.47930896	36.52069104
100	10.6870229	89.3129771

Table.3 Dose response of complex on MCF-7 cell line

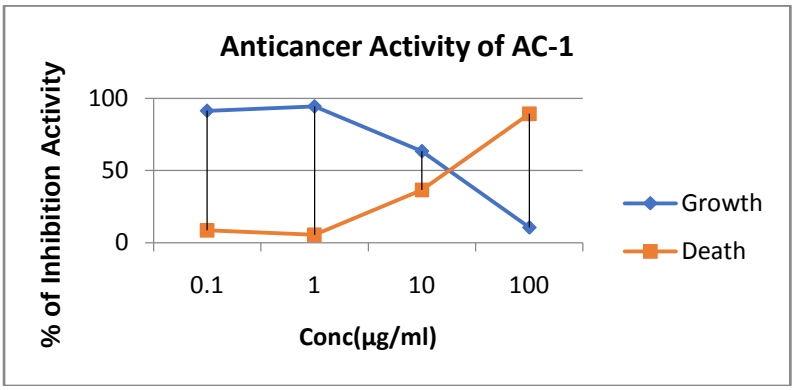


Fig.5. Effect of complex on MCF-7 Cell Viability for 24 h Incubation Time.

IC50	32.59µg/mL

III. Conclusion:

In the present study on synthesis of Nickel oxide nanoparticles using chemical method and their antimicrobial activity, Nickel oxide nanoparticles were synthesized using Nickel nitrate hexahydrate. Synthesis conditions were optimized and resultant nanopowder was characterized using UV-Visible spectroscopy, XRD, FESEM. Morphological analysis report particle size range of 50 nm and also revealed that the nanoparticles are present in the form of aggregates. While studying the effect of nanoparticles for their antifungal potential, these showed activity against 2 bacterial pathogens and 1 fungal pathogen. It could be utilized for developing antifungal agents for commercial use in the field of agriculture. This study conclusively reports a synthesis of zinc oxide nanoparticles. Such studies have the potential for developing good fungicidal formulations having nanoparticles. The cytotoxicity activities of Nickeloxide nanoparticles screened by MTT assay. We have screened for one type of cancer cell line, viz., MCF-7 (breast cancer), zinc oxide obtained IC_{50} values in the range of 32.59 µg/mL for MCF-7 cell line most of these nanoparticles are in cytotoxic activity.

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