Life Science Informatics Publications

Research Journal of Life Sciences, Bioinformatics, Pharmaceutical and Chemical Sciences

Journal Home page http://www.rjlbpcs.com/

RJLBPCS

ISSN 2454-6348

Original Research Article **DOI: 10.26479/2018.0406.59**

FABRICATION OF NICKEL OXIDE NANOPARTICLES FOR ANTIBACTERIAL AND PHOTOCATALYTIC ACTIVITY

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ABSTRACT: In this work, we have developed a catalyst of Nickel oxide nanoparticle from the Hydrolysis precipitation method, it was found that NiO NPs even at very low concentration can greatly enhance the antibacterial and photocatalytic activity and it has been characterized by various Analytical and Spectroscopic techniques like X-Ray diffraction (XRD), Scanning electron microscope (SEM), Fourier–Transform Infrared (FT-IR), and Ultraviolet spectroscopy (UV), Dynamic light scattering (DLS) and Fluorescence spectroscopy (FL). The NiO NPs are against bacterial infections. The Antibacterial activity was evaluated through the bacteria include *Bacillus subtilis*, *E. coli* and *Enterobacter*. The Photocatalytic activity was tested on the degradation of Methylene orange under Heber reactor. The synthesized NiO NPs rapid generation of charge carriers leads to the increase zone formation and the degradation of dye molecules.

KEYWORDS: Hydrolysis precipitation method, Nickel oxide nanoparticle, Antibacterial and Photo catalytic activity.

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1.INTRODUCTION

Nickel oxide is one of the transitional metal oxide nanoparticle. It is also a semiconducting material [1] with diverse applications. It has a high surface area and unique properties [2-4]. Therefore, the researchers significantly concern on this nanoparticle. Nickel oxide nanoparticle contain low toxic [5] and well biocompatibility [6]. It consists of a lot of applications like gas

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sensor, energetic materials, electrochemical supercapacitor, solar cell battery cathodes [7-13] etc. Now days the pathogenic bacteria cause many diseases in human beings [14]. Nickel oxide nanoparticles exhibit a higher antibacterial activity against a gram-positive and gram-negative pathogen. [15] Antibacterial activity destroys a bacterium or suppresses its growth or their ability to reproduce. E. coli induces microbial spoilage in milk, which is present in the digestive tract of the humans, animals, faecal contaminated foods and water. [16,17]. Enterobacter species causes bone illness, nosocomial infections etc [18-20]. Bacillus species which produce subtilin and subtilosin will cause two types of illness like diarrhoea, vomiting. [21-23]. So that we need to use the microbial activity using Nickel oxide nanoparticle to prevent and control these issues. The NiO nanoparticle consists of numerous synthesis method including Sputtering [24-26] evaporation [27], Electrode deposition [28], Co-precipitation [29] and Sol-gel [30]. Here, we synthesise it by Hydrolysis precipitation method. [31,32] Among these methods, a hydrolysis precipitation method is the best way to prepare Nickel oxide nanomaterial due to eco-friendly and low impurities. The Antibacterial activity of silver, gold, copper nanoparticle from plant extract was determined. Metal oxide NPs have been used for the photocatalytic environmental remediation like wastewater decontamination due to high photosensitivity, environmentally friendly, non-toxic and low cost. However, this catalyst involves absorption of photons to produce electrons (e^-) and holes (h^+) pairs in aqueous solution. These charge carriers lead to dye dehydration through redox species. [33] For several decades, research groups have attempted to develop a photocatalytic activity using the Nanoparticles like Titanium oxide, Chitosan, and Zinc oxide. It was developed as a photocatalyst due to High photoreactivity, low toxicity, long-term stability against chemical corrosion and photo-corrosion [34-37]. In this report, Nickel oxide nanoparticle is prepared in rod and hexagonal shape by hydrolysis precipitation method from the solution of Nickel chloride in the aqueous solution as a dissolving agent. Urea acts as a hydrolysis agent. Finally, pH is maintained by the addition of Sodium hydroxide in the preparation of Nickel oxide nanoparticle. The antibacterial and photocatalytic activity was investigated in this paper.

2. MATERIALS AND METHODS

Nickel chloride, Urea, Sodium Hydroxide are used in this experiment were purchased from Sigma-Aldrich, *E. coli*, Bacillus species and *Enterobacter* were purchased from IMTECH at Chandigarh. Methylene orange and Muller Hinton agar were of analytical grade.

Synthesis of Nickel oxide nanoparticle

The Nickel oxide nanoparticle was synthesized by Hydrolysis precipitation method. The 1:1 ratio amount of Nickel chloride and Urea was dissolved in 100 ml of $D.H_20$ and the mixture is maintained under Magnetic stirrer for 20 mins. Then, Sodium hydroxide was added to adjust pH 7 and the solution is heated at 80°C for 30 mins. After the mixture cools down to room temperature, the precipitate solution is washed with ethanol and $D.H_20$, followed by centrifugation. Finally, the

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solution was kept drying at 60 ^{0}C for 12 hrs and calcinate at 400^{o}C. Thus, black colour NiO nanoparticle was obtained.

Assay of Antibacterial activity

Antibacterial activities of the synthesized NiO nanoparticle were performed against pathogenic bacteria. The antibacterial activity was done by the well-diffusion method. The pure culture of bacteria was subculture using Mueller- Hinton broth at 37^{0}C on a rotary shaker at 150 rpm for 12 hrs. After that, 25 ml of Muller-Hinton medium was poured uniformly in the Petri dishes and then smeared using sterile cotton swabs with the bacterial culture of *Bacillus sp, E. coli, Enterobacter*. NiO NP could be distributed in various concentration at 20 µl, 40 µl,40 µl,80 µl. The plates were incubated at 37^{0}C for 24 hrs and resulting zone inhibition was measured.

Photocatalytic experiment

The Photocatalytic experiment was carried out in a Heber reactor. The experiment was carried out in a 50 ml quartz. The suspension was carried out by 350W with high-pressure Mercury lamp. Methylene orange dye was used for the degradation purpose. 2mg of photocatalyst and the concentration of Methylene orange solution is used for further Photocatalytic activity. The degradation of the dye was determined by UV– spectrometer.

Characterization techniques

X-ray Diffraction Spectroscopy (XRD) pattern of the sample was recorded on a Phillips PW1800 diffractometer with Cu kα radiation generated at 40 KV for crystalline phase identification, Optical properties of NiO NPs were studied for Perkin ElmerLS45 Fluorescence spectrometer and Shimadzu Japan UV-spectrometer. Particle sized of synthesized NPs was determined by Dynamic Light Scattering (DLS) analysis using Nano plus Micromeritics. The surface morphology of the sample was obtained by Scanning Electron Microscopy (SEM) with a TESCAN (model WEGA11), operation at an accelerating voltage of 25.0KV. Fourier transform Infrared spectroscopy (FT-IR) analysis of NiO NPs performed in Perkin-Elmer spectrometer spectrum one.

3. RESULTS AND DISCUSSION

X-ray diffraction (XRD) analysis

The diffractions peaks in XRD pattern of Nickel oxide Nanoparticle corresponds to Face Centered Cubic crystal structure (FCC). Five characteristic peaks of NiO NP were found and correspond to Miller indices (111), (200), (220), (311) and (222) as shown in Fig.1. The average crystallite size of the nanoparticles was calculated using Scherrer’s formula [38-39].

$$D ¼ k\lambda = \beta \cos \theta$$

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Where D is a grain size, K is an empirical constant to 0.9 and λ is the wavelength of the CU. K α radiation. β is the full width half maximum value and θ is the angular of the peak position. The average crystallite size is about 41.8nm, which was calculated from (111) plane. With the match of JCPDS card NO :(47-1049).

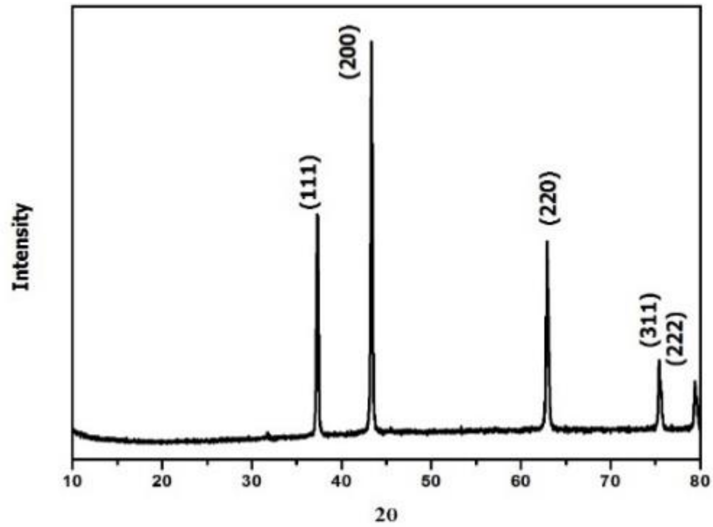

Fig.1. X-ray diffraction (XRD) analysis

Optical properties analysis

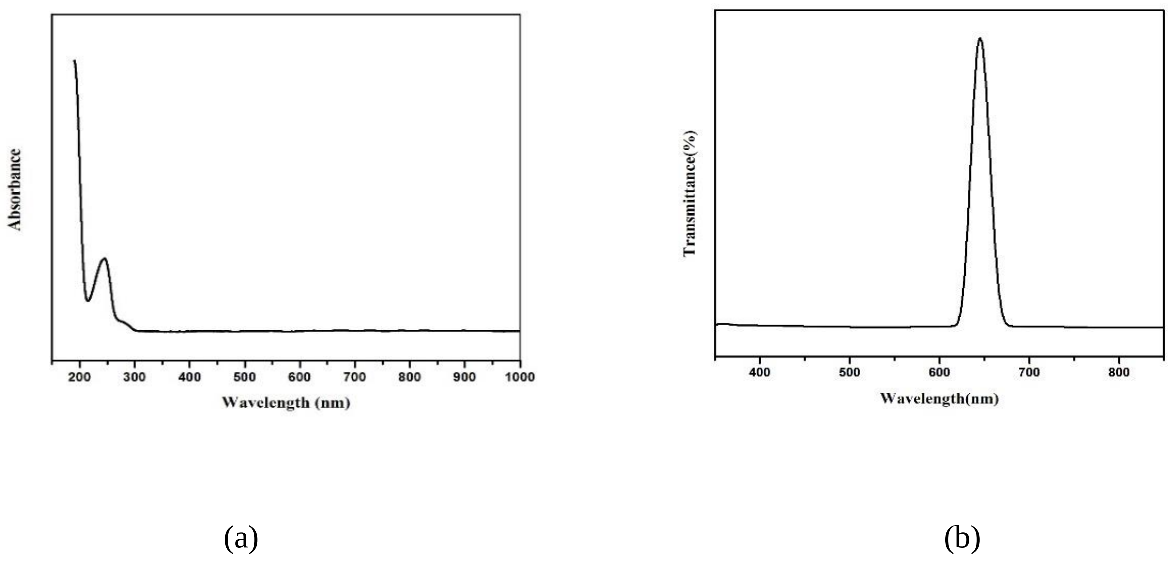

Fig.2. (a) UV- Visible absorption profile of NiO NPs Fig. 5.2. (b) FL Spectra of NiO NPS.

The optical properties of NiO nanoparticle were synthesized by the Chemical precipitation method which was studied by UV-Visible and Fluorescence spectroscopy. Fig. 2 (a) shows UV -Visible spectra. The sample was dispersed in distilled water kept at vortex for 10 minutes. These spectra are found in two characteristics peaks. The first peak is signified at 245 nm, which corresponds to

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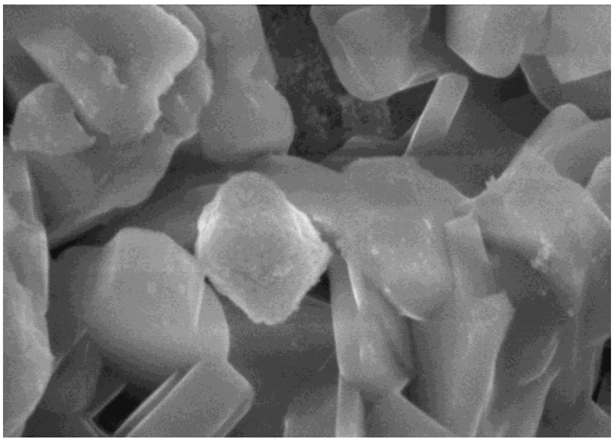
the formation of NiO nanoparticle, while the second peak is attributed at 675 nm. It can correspond to the surfactant. Fig.2 (b) shows the fluorescence properties of NiO NP excitation wavelength which were investigated. The fluorescence higher emission wavelength by redshift was found at 645 nm.

Scanning Electron Microscopy



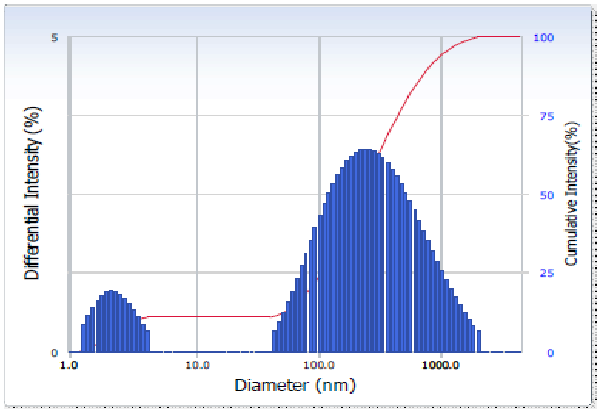


Fig.3 (a). SEM morphology of NiO NPs.   **Fig.3 (b). DLS analysis of NiO NPs.**

The morphology of the Nanoparticle was characterized by SEM. Typical Nickel oxide nanoparticle was synthesized by Hydrolysis precipitation method. SEM image of the NiO NP is shown in Fig.3(a). The result shows some particles are in a rod shape and some are in hexagonal shape. We can find that the particles are aggregated with irregular particles morphology having a diameter of 1µm. The mechanisms of $Ni(OH)_2$ and end product NiO involves two criteria i.e., reaction system and parameters. The Urea act as a hydrolysis agent[40] to generate OH ions along with Ni^{2+} to form $Ni(OH)_2$ based on reaction systems and parameters, which produce crystal growth. The $Ni(OH)_2$ is decomposed into NiO by increasing temperature at 400°C. In Fig.3(b) particles were observed by using Dynamic Light Scattering with wide size distribution. The average particle size was found to be 343nm. The reaction steps are given below.

$$H_2N\text{-}CO\text{-}NH_2 + H_2O \longrightarrow 2NH_3 + CO_2$$

$$NH_3 + H_2O \longrightarrow NH_4^+ + OH^-$$

$$Ni^{2+} + 2OH^- \longrightarrow Ni(OH)_2$$

$$Ni(OH)_2 \xrightarrow{\triangle} NiO$$

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Fourier Transform-Infrared Microscope (FT-IR) analysis

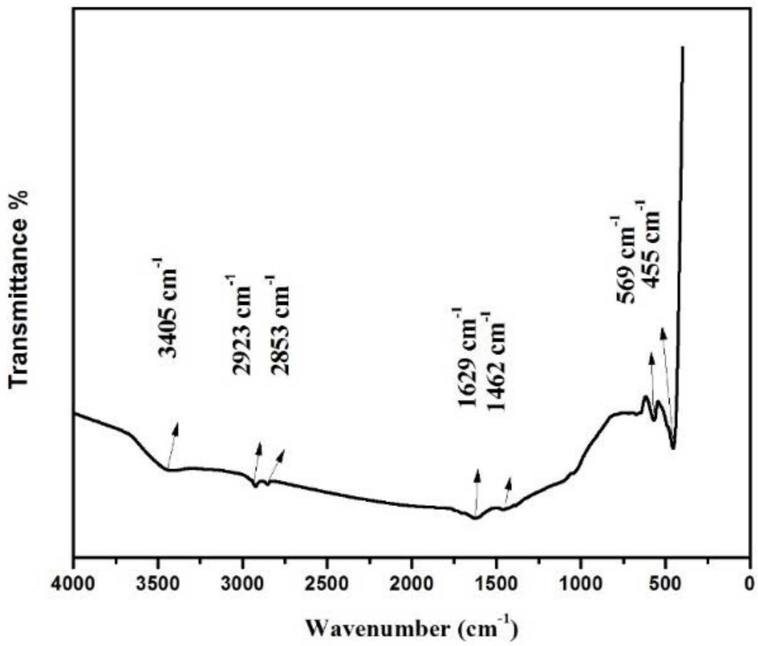

Fig.4. The FTIR spectra of NiO NPs.

The FTIR spectrum of NiO NPs is shown in Fig.4. The prominent band at 3494cm^{-1} is assigned to the O-H stretching mode which is ascribed to the hydroxyl group [41]. The weak band at 2923 cm^{-1} and 2853 cm^{-1} are assigned to the C-H symmetric stretching vibration were presented to the surfactant molecules as like Urea. [42] 1629 cm$^{-1.}$ These peaks indicate the presence of water in the sample [43]. The band at 1462 cm^{-1} confirm the presence of CH_2 bending vibration [44] The spectrum contains one broadband at 569 cm^{-1} is characteristic of the stretching vibration hydroxyl group, hydrogen- bonded to Ni-O [45]. The sharp band appears at 455 is assigned to the stretching vibration of NiO bands [46].

Antibacterial activity

The antibacterial activity of NiO NPs were examined. The NiO nanoparticles were able to attach to the membrane of pathogenic bacteria by electrostatic interaction which produces toxicity ions and then generates the ROS which destroys the growth of bacteria [47-51]. However, these NPs were investigated with different antibiotics against *Escherichia coli*, *Bacillus subtilis* and *Enterobacter*. It was observed that the antibacterial activity of *Bacillus subtilis* was better than *Enterobacter* and *Escherichia coli* and also it was found that only a few activities are present in *Enterobacter,* which is shown in Fig.5 and 6.

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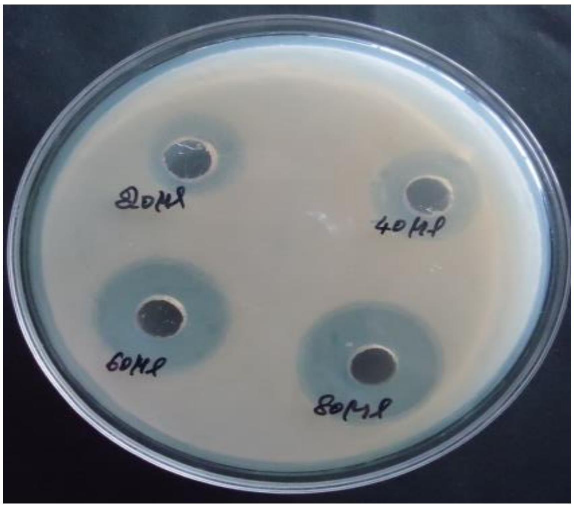
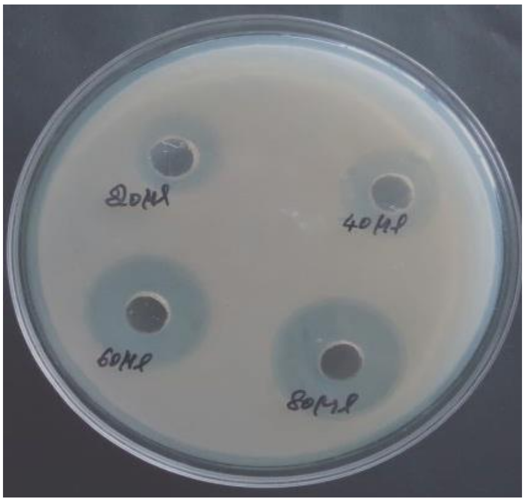
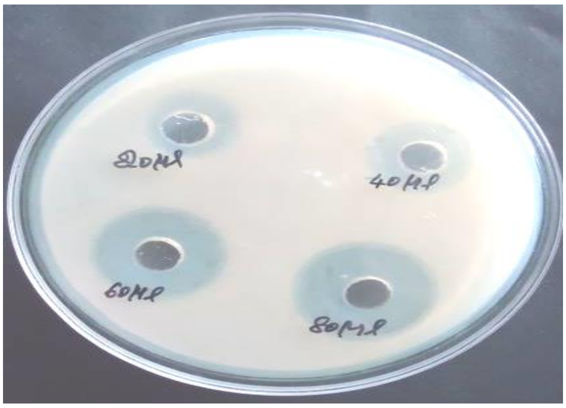


(a)



(b)



(c)

Fig. 5. Zone of inhibition E. coli (a), Bacillus subtilis (b) and Enterobacter(c).

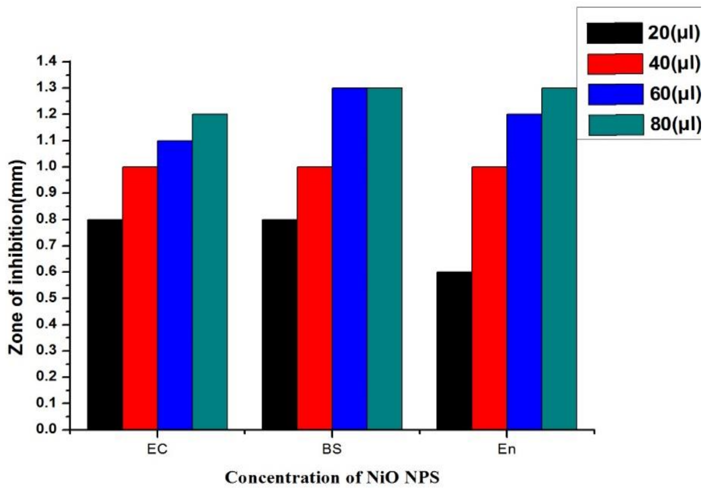


Fig.6. Bar graph of Zone formation using NiO NPs.

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Photo catalytic activity studies

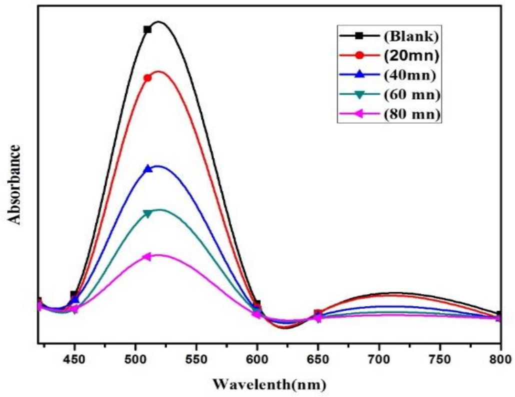

Fig.7. Photodegradation of Methylene orange dye by the NiO NPs using U-V Spectrometer

The photocatalytic activity of Nickel oxide nanoparticles was tested on the degradation of Methylene orange dye under Heber reactor [52].0.2mg of photocatalyst was taken from the stock solution was dispersed in 50 ml of Methylene orange solution under magnetic stirrer. This experiment is carried out on different time periods. This process was carried out from 10 min to 60 min. Methylene orange dye degradation is recorded using UV–Spectrometer. NiO NPs having high surface area can provide more active absorption capacity. Ni ions produce hydroxyl groups by absorbing water in the air. When light falls on the sample, the electrons move from valence band to conduction band and the creation of holes and electron pairs will occur [53] This result shows an increase in the Photocatalytic activity which is shown in Fig .7. The time required for 80% degradation of dye is about 60 min.

4. CONCLUSION

This study investigates photocatalytic and antibacterial activity using NiO NPs. These Nanoparticles were fabricated through hydrolysis precipitation method and the Photocatalytic degradation of MO dye under Heber reactor. In this work, we observed that minimum concentration of NiO NPs is attributed for dehydration of dye and Zone formation of Organisms such as *E. coli*, *Bacillus subtilis* and *Enterobacter.*

CONFLICT OF INTEREST

Authors have no any conflict of interest.

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