

Study of NiO Nanoparticles Synthesis by Sol Gel Method, Its Characterization and Biological Application

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ABSTRACT

The present research work focuses on synthesis of Nickel oxide nanoparticles by sol-gel method, their characterization and the antibacterial effect of NiO on *E. coli* (NCIM 2065) and *Bacillus subtilis* (NCIM2063). Sol-gel technique is one of the simple, cost effective, environmentally safe technique for synthesis of Nickel oxide nanoparticles by optimizing reaction parameters to provide the good biological properties and applications. The green color Nickel oxide nanoparticles was synthesized, washed with distilled water, dried and calcinated at 500°C for 5 hours was investigated by Fourier transform infrared spectroscopy, The X-ray powder diffraction, Scanning Electron Microscopy, Energy dispersive X-Ray spectroscopy and to study the antibacterial effect of NiO on *E. coli* (NCIM 2065) and *Bacillus subtilis* (NCIM2063).

Keywords: Sol-gel method, Nanoparticles

I. INTRODUCTION

Nanoparticles are tiny particles with at least one dimension less than 100nm. For this, these NPs are very attractive materials to handle in biological system. Nanoparticles are found to be very suitable in sensing and detection of biological structures and systems [1]. Metal nanoparticles appear in different shapes such as nano powder or nanocluster or nanocrystal and different sizes ranging from two nm to 1000 nm. Nanoparticles are of great scientific interest as they are effectively a bridge between bulk

materials and atomic or molecular structures [2]. Numerous methods have been developed for the synthesis of NiO nanoparticle such as pyrolysis method, hydrothermal method, precipitation method, electrochemical reduction, chemical reduction and sol-gel method. [3,4] The synthesis of NiO nanoparticles includes the dry and wet methods. The sol gel method is wet chemical method for fabrication of NiO nanoparticles. The use of Sol-gel method has various advantages such as it is simple, cost-effective method to synthesized homogenous, good crystallinity, uniform and sharp size of

nanoparticles.[3] The interactions between microorganisms and metals have been well documented and the ability of microorganisms to extract and/or accumulate metals is already employed in biotechnological processes such as bioleaching and bioremediation.[5] The microbial property of the nanoparticles has significant practical relevance control of microbial, bacterial and fungal organisms. Water treatment and water purification and are some of the applications of the nanoparticles [6]. Microorganism are foundation of aquatic ecosystems and provide key environmental services ranging from primary productivity to nutrient cycling and waste decomposition so they are of great environmental importance. The research focuses on inhibition of growth of *E. coli* by rapid removal of RhBand organic pollutant from water [7]. Microorganism grow rapidly and are inexpensive to culture so they are used as test model. They are microscopic and have a high surface-to-volume ratio, which, make them sensitive to low concentrations of toxic substances; and facilitate studies at many levels ranging from a single biochemical reaction in bacteria to complex ecosystems containing a diversity of microorganisms. In this study synthesis of NiO using sol gel method. The prepared nanoparticles were characterized using FTIR, XRD, SEM and EDS techniques. The present study aimed to the synthesized of NiO nanoparticles by Sol gel method and to study the antibacterial effect of NiO nanoparticles on *E. coli* (NCIM 2065) and *Bacillus subtilis* (NCIM2063).

II. MATERIAL AND METHOD

2.1. Synthesis of NiO nanoparticles:

Nickel Oxide nanoparticles was fabricated via Sol-gel method. 2.5 g of Ni (NO₃)₂·6H₂O and 15ml of distilled water mixed with each other to form the solution which is titrated against 1N NaOH solution dropwise from burette with constant stirring. The solution formed is of alkaline pH. After the complete reaction,

green color precipitate of NiO nanoparticles was formed, kept for aging for 24 hours. Then formed green color precipitate of nanomaterial was filtered, washed with distilled water, dried and calcinated at 500°C for 5 hours. The synthesized Nickel Oxide nanoparticles was investigated by X-ray powder diffraction method, Fourier transform infrared spectroscopy, Scanning Electron Microscopy, Energy dispersive X-Ray spectroscopy.

2.2. Antimicrobial activity test

Antimicrobial activity test was performed by Kirby-Bauer Method and PDA medium, respectively. Antimicrobial activity of Ni was evaluated by disc diffusion assay against the Gram-negative bacteria (*E. Coli* - NCIM 2065) and Gram-positive bacteria (*B. subtilis*-NCIM2063), which is designed to determine the smallest amount of the antibiotic needed to inhibit the growth of microorganisms. One loop of mother culture inoculated in Sterile 15 ml NA broth and for 24hrs it is incubated at 37 °C. Sterile cotton swabs is taken and dip it into a culture of Test organism suspension and incubated the entire agar surface of each plate first in horizontal and then in vertical direction to ensure the even distribution of the organism over the agar surface using the swab. The agar surface dried for 5 min. a cork borer sterilized by disinfect it by rinsing in alcohol followed by sterile water or by autoclaving. Sample solution was put in A Mueller –Hinton agar plates (8mm) the underside of the Petri is marked to label the wells, using a wax pencil. With the help of a micropipette a test solution in the well is added, procedure is repeated for all wells. All plates are incubated at 37°C for 24-48 hrs. in an incubator. All the plates were examined for the clear zone of inhibition surrounding the discs / well, the diameter of zone of inhibition is measured in mm using a ruler the zone size is recorded on the underside of the plate and by comparing the zone obtained with the known concentration versus the zone of inhibition of std. antibiotic a graph is prepared.

III.RESULTS AND DISCUSSION

The formed dried green color nanomaterial calcinated at 500°C for 5 hours were investigated by Fourier transform infrared (FTIR) spectroscopy, The X-ray powder diffraction (XRD), Scanning Electron Microscopy (SEM). Energy dispersive X-Ray spectroscopy (EDX).

3.1. Fourier transform infrared (FTIR) spectroscopy.

In this research paper, Fourier transform infrared (FTIR) spectroscopy was performed to investigate the formation of the NiO nanoparticles which was fabricated by sol-gel technique and calcinated at 500°C for 5 hours. The FTIR spectra shows that the peak around 800 cm^{-1} is due to the Ni-O stretching vibration. The peak at 2400 cm^{-1} and 1340 cm^{-1} is due to the antisymmetric stretching and symmetric stretching mode of vibrations of CO_2 molecule adsorbed from air. The O-H stretching vibration due to moisture indicate the absorption peak at 3600 cm^{-1} . [9]

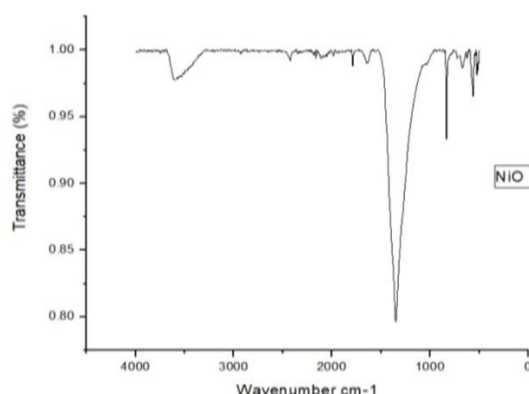


Fig-3.1 FTIR Spectrum of NiO nanoparticles

3.2. The X-ray powder diffraction. (XRD)

The X-ray powder diffraction spectra is the most useful technique for phase identification, structure and crystallinity of nanoparticles.[9] The X-ray powder diffraction pattern shows various intense peaks which indicate that the highly crystalline nature of the NiO nanoparticles. The Scherrer equation $D = K \lambda / \beta \cos \theta$ is used to determine average crystal sizes D of nanoparticles like NiO where D is average crystallite size of nanoparticles, K is equal to 0.9 which is

constant, λ is the wavelength of the X-ray and β is the full width at half- maximum (FWHM), θ is the Bragg diffraction angle. (10, 11)

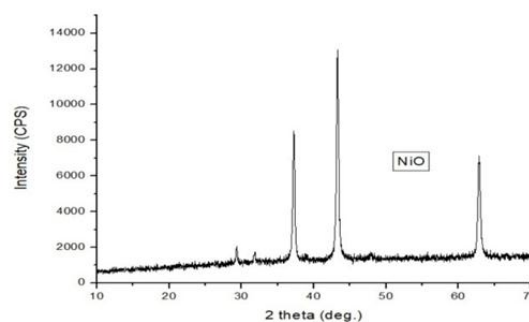


Fig-3.2 The XRD patterns of NiO nanoparticles

3.3. FESEM Analysis

The surface morphology, shape and size of the synthesized NiO nanoparticles has been studied by Scanning Electron Microscopy FESEM. [10,12] The NiO nanoparticles were fabricated by a simple sol-gel method using nickel nitrate $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ as precursor. The FESEM images of NiO nanoparticles show that the NiO nanoparticles in Figure 3 are agglomerate and porous. The FESEM photo shows the sizes of the particles, which range from 18.89 to 35.07 nm.

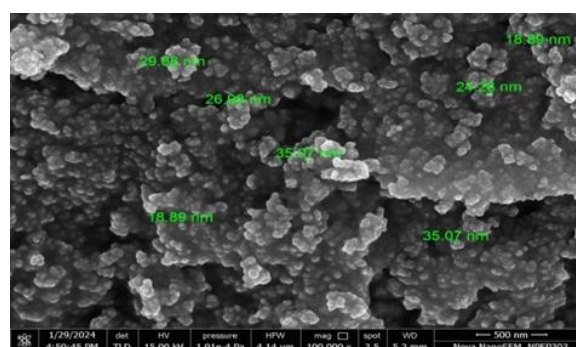


Fig-3.3 The SEM image of NiO nanoparticles

3.4. Energy dispersive X-Ray spectroscopy (EDX)

The elemental composition of synthesized Nickel oxide nanoparticles is determined by Energy dispersive X-Ray spectroscopy (EDX) [10]. The quantitative analysis of the NiO nanomaterial shows that elements found in the nanomaterial are 44.44 % Ni content and 17.29% O content and other trace of materials.

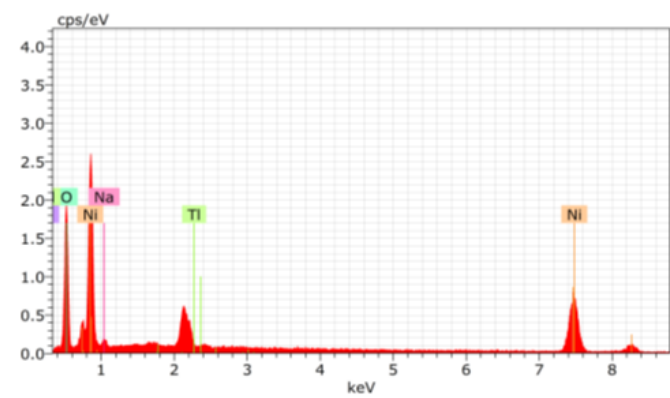


Fig-3.4 EDX Spectrum of NiO NPs

3.5. Antimicrobial activity

In disc diffusion assay, the suppression of bacterial growth was determined in Petri plates loaded with 1mg /mL NiO NP after 24 h at 37°C. In this procedure, the inhibition zone was measured in millimetres, and gentamycin and streptomycin were used as negative control. Minimal zone of inhibition was observed in control plates loaded with negative control. NiO NP shows zone of inhibition of diameter ranged from

13mm and 12.3 mm, in *E. Coli* and *B. subtilis*, respectively, while in streptomycin and Gentamycin it ranges from 31.6 mm 36mm, 23mm and 26.3mm respectively (Table No-1).The presence of inhibition zone in Figure 3.5confirmed the antibacterial activity of NiO.

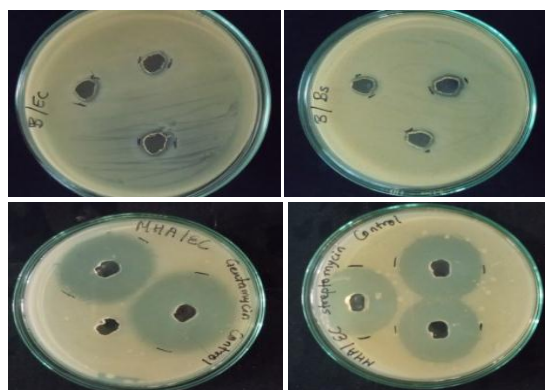


Fig-3.5 The antibacterial activity of NiO shown by the presence of inhibition zone Table No-1

Sample Name	Zone of Inhibition (mm)(Values are mean of Triplicates)					
	Microorganism studied (sample in DMSO) (Cons mg/ml)					
	mg sample	1ml DMSO	Eco (mm)	Mean (mm)	Bs (mm)	Mean (mm)
B-NiO Np	1	1	13,12,14	13	11,12,14	12.3
DMSO Negative Control	-	1	9,9,9	9	-	-
Streptomycin	10	1	31,31,33	31.6	23,23,23	23
Gentamycin	10	1	35,37	36	27,27,25	26.3

Ec - *E. Coli*, Bs-*B. subtilis*

IV.CONCLUSION

For NiO nanoparticles the maximum zone of inhibition was found against *E. coli* (13 mm). Table - 1 indicate the lowest degree of antibacterial activity against *B. subtilis* (12.3 mm) The results above clearly showed that the produced nickel nanoparticles have bacterial inhibitory capabilities. The mechanism of

the antibacterial action of NiO nanoparticles can be explained in several ways. In aqueous suspensions of NiO-NPs, the released Niionscan interfere with the thiol group of essential bacterial enzymes, leading to inactivation and cell death. Additionally, the radicals present in the aqueous of NiO nanoparticles can interact with the negatively charged bacterial cell surface, disrupting vital life processes such as cell

replication, respiration ultimately causing inactivation and cell death [7].

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VI. REFERENCES

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