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Rapid solution combustion synthesis of NiO Nanostructures: Characterization and Evaluation of antibacterial activity

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Abstract: *Nickel oxide (NiO) NPs were synthesized by solution combustion method using Nickel nitrate as an oxidizer and starch as fuel. The X-Ray Diffraction (XRD) exhibit cubic structure and confirmed the presences of NiO NPs (JCPDS: 65-2901). The morphology of the NiO nanoparticles was investigated by means of SEM and confirms the nanostructure. It is possible to suggest that the organic fuel starch is responsible for the formation of nanostructure due to the easier complex formation. The chemical composition of NiO NPs was investigated by Energy Dispersive Spectroscopy (EDAX). It confirms the presence of Nickel oxide nanoparticles. In the FTIR analysis, Ni-O stretching vibration mode is obtained in the region of 454.02 cm^{-1}. The prepared NiO NPs are very effective to gram positive strains than the gram negative strains. Gram positive and gram negative bacteria have differences in their membrane structure, the most distinctive of which is the thickness of the peptidoglycan layer.*

Key words: Nickel oxide NPs, Solution Combustion method, XRD, SEM & EDAX, UV, FT-IR, Antibacterial activity.

1. INTRODUCTION

Nickel oxide (NiO) is a significant transition metal oxide that has garnered attention as a strong candidate for many fields including super paramagnetic devices, photovoltaic devices, electrochemical super capacitors, magnetic materials, catalysis, smart windows, fuel cell, and photovoltaic devices [1]. These nanostructured particles are regarded as a p-type semiconductor having large exciton binding energy with stable wide band gap (3.6–4.0 eV). Bulk NiO is an antiferromagnetic insulator with a Neel temperature of 523K [2]. They exhibit many unique magnetic, optical, electronic, and chemical properties that are significantly different than those of bulk-sized NiO particles due to their quantum size and surface effects [3]. Some of these techniques suffer from the difficulty in size homogeneity and dispersion of NiO nanoparticles (NPs). Generally, most techniques aim to reduce the costs of chemical synthesis and to produce materials for technological applications [4]. These materials like copper, zinc, nickel, silver present high antibacterial activity, low toxicity, chemical stability, long lasting action period and thermal resistance compared to organic antibacterial agents [5]. In the present study, NiO nanostructures were synthesized by solution combustion method using starch as fuel. The antibacterial activities of the prepared NiO nanostructure were investigated.

2. MATERIALS AND METHODS

2.1 Synthesis of NiO NPs

For the preparation of NiO NPs, nickel nitrate, starch was taken as starting materials. The stoichiometric composition of solution components fuel and oxidizer was calculated according to the principle of chemistry keeping the oxidizer (metal nitrate) to fuel (starch) ratio unity. Stoichiometric amount of Nickel (II) nitrate and starch were dissolved in de-ionized water separately. The solution was mixed vigorously until the homogenous solution was obtained and then the solution was kept the furnace at 300^{0}C. The solution boils and undergoes dehydration, then the solution reaches the point of combustion, it began to burn released a lot of heat as fumes and vaporizing all the solution. As a result, NiO product and gases of H_2O, N_2 can be formed directly from the reaction between fuel and oxidizer without necessary of getting oxygen from outside. The process was completed in 20 minutes and fine black colour powder was obtained as a result.

2.2 Characterizations

The Shimadzu IR affinity-1 Fourier Transform Infrared spectrometer was used to carry out vibrational studies. The X-ray diffraction patterns were recorded on PANalytical X-ray Diffractometer using Cu Kα radiation (λ=0.1542nm) operated at 50kV and 100mA.The experiments were performed in the diffraction angle range of 2θ. Energy Dispersive Spectroscopy was carried out by BRUKER instrument. Scanning Electron Microscopy images were obtained by an instrument VEGA3 LMU.

2.3 Assay for antimicrobial activity of NiO NPs against microorganism

The antibacterial activity of NiO NPs was evaluated against gram positive Bacillus cereus, Entero coccus, and Staph aureus, gram negative bacteria Escherichia coli, Klebsiella pneumonia and Pseudomonas aeruginosa. Exactly 0.2 ml of fresh cultures of each organism was inoculated into 5 ml of serile nutrient broth and incubated for 3-5 h to standardize the culture to McFarland standards (106 CFC/ml). Three replicates of respective microorganism were prepared by spreading 100 µl of the revived culture on MHA (Mueller Hinton Agar-Hi Media) with the help of spreader. The well was made having a diameter of about 7 mm and 50 µl samples of NiO were added in one well and 50 µl of distilled water as control. The petri plates were kept at 37 ^{0}C for 24 h in incubator for bacteria during which its antibacterial activity was evidenced by presence of a zone of inhibition (mm) surrounding the well.

3. RESULTS AND DISCUSSION

3.1 X-ray diffraction spectroscopy (XRD)

XRD is a popular technique for determining phase purity of the materials. The width of the diffraction lines is closely related to the size distribution defects and strain in NPs. Fig.1 shows the XRD spectra of NiO NPs. The purity and crystalline of the synthesized NiO NPs were examined by using powder X-ray diffraction (XRD). The five diffraction peaks of the XRD pattern of NiO NPs were observed at 37.23^o, 43.19^o, 62.72^o, 75.28^o, 79.31^o which correspond to (111), (200), (220), (311), and (222) diffraction planes respectively (JCPDS, NO: 65-2901). All diffraction peaks of NiO correspond to the cubic structure and the volume of the cell was 49.97 $(A^0)^3$. The average particle size of the NiO NPs was calculated using the Debye scherrer equation [6]. The lattice constant value is calculated as 4.194 A^0. The average particle size found to be around 18 nm. Fig.2 shows the FTIR spectra of combustion product of NiO NPs. The FTIR spectrum was observed over the frequency range 400-4000 cm^{-1}.

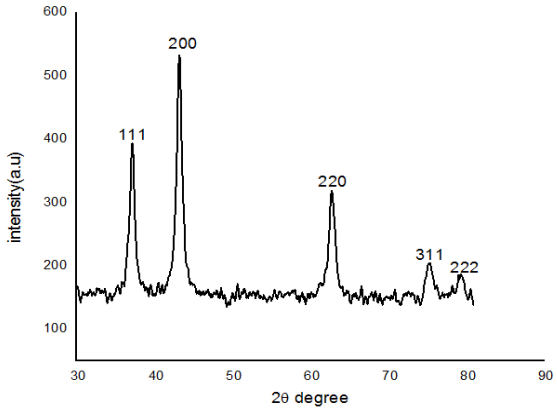

Fig.1 XRD spectra for NiO NPs

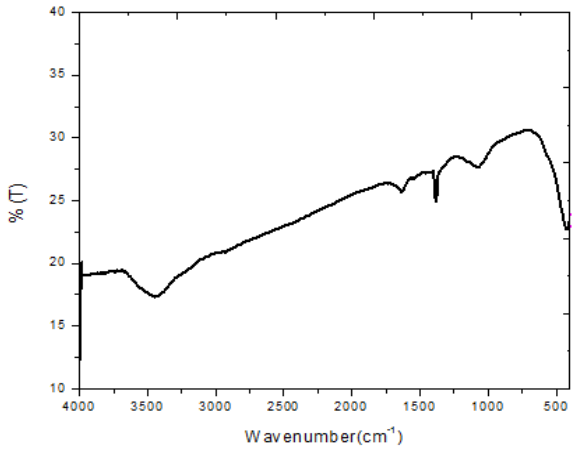

Fig.2. FTIR spectra of NiO NPs

The broad band in the region of 454 cm^{-1} is assigned to Ni-O stretching vibration mode. The FTIR band seen around 3500 cm^{-1} was attributed to O-H stretching of hydrogen bonded water on the surface of NPs and the band at 1634 cm^{-1} is assigned to H-O-H bending vibrations mode presented due to the adsorption of water in air during the preparation of FTIR sample disks in an open air atmosphere. The band at 1385 cm^{-1} is attributed to O-C=O symmetric and asymmetric stretching vibrations. The moderate peak at 1071 cm^{-1} may be due to single C-O band stretching mode [7].

3.2 Scanning electron microscopy (SEM)

Fig.3 shows that the SEM image of NiO NPs. Studying, the growth mechanism of NiO NPs, it is possible to suggest that the organic fuel starch is responsible for the formation of the NiO nanostructure due to the easier complex formation [8]. When starch is employed, the heat released in combustion is more and as a result which is responsible for the growth of the sample and complete combustion reaction with more crystalline phase. So the result indicates that the presence of starch has a significant effect on the morphology of the sample. Fig.4 shows that the chemical composition of NiO NPs which was investigated by Energy Dispersive Spectroscopy (EDAX). It confirms the presence of NiO NPs.

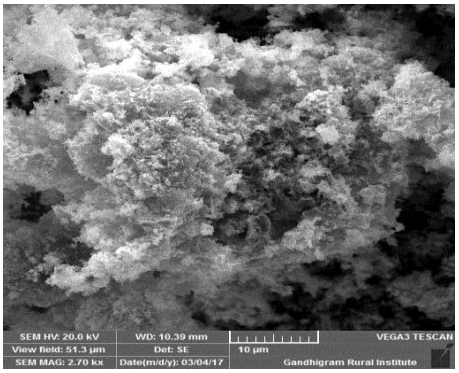

Fig.3. SEM image of NiO NPs

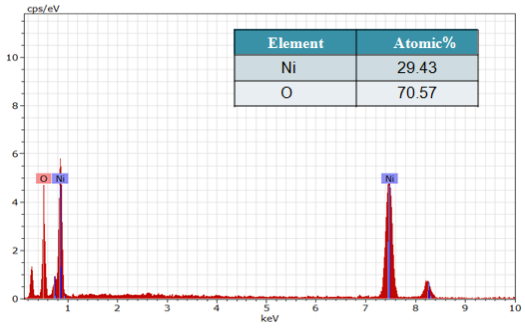

Fig.4. EDAX for NiO NP

3.3 Antibacterial activity

In this study, to evaluate the antibacterial effects against various microorganisms (gram positive and gram negative), Staphylococcus aureus, Entero coccus, Bacillus cereus, E.coli, Klebsilla pneumonia, Pseudomonas aeruginosa were used. Antibacterial activity done for synthesized NiO NPs using Kirby-Bauer method on both Gram-negative and Gram positive bacteria. Fig.5, 6, shows the antibacterial activity of NiO nanostructure on the corresponding microorganisms.

Gram-negative bacteria

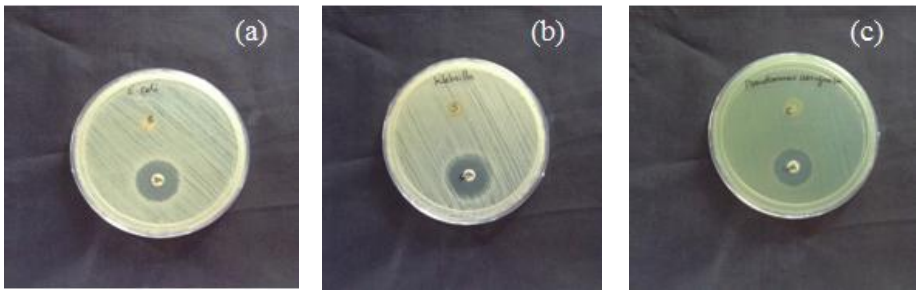

Fig.5. (a), (b), (c) *E.coli, Klesilla pneumonia, Pseudomonas aeruginosa* negative bacteria

Gram-positive bacteria

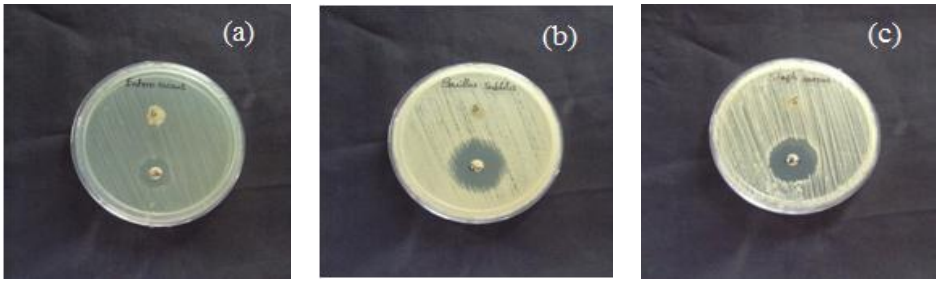

Fig.6. (a), (b), (c) Entero coccus, Bacillus cereus, Staph aureus, positive bacteria

Table1. Antibacterial efficacy results of NiO NpS.

Microorganisms	Zone of inhibition (mm)
E. coli	23
K. pneumonia	22
P. aeruginosa	19
S. aureus	26
E. coccus	17
B. cereus	29

The diameter of inhibition zones (in mm) produced by NiO NpS against theses test strains are shown in Table1. The prepared NiO NPs is effective to Gram positive strains than the Gram negative strains. Gram positive and Gram negative bacteria have differences in their membrane structure, the most distinctive of which is the thickness of the peptidoglycan layer. The lower efficacy of the NiO NPs against *Entero coccus*, *Pseudomonas araginosa* may derive from the difference as a point of membrane structure [9]. The growth inhibition of bacterial cells may be due to distractions of cell membrane by NiO NPs which results in breakdown of cell enzyme.

4. CONCLUSION

The NiO NPs were synthesized by using solution combustion method. The XRD confirms the NiO NPs and the average crystalline size of the NiO NPs were found to be about 18 nm and exhibits cubic structure. The morphology of the NiO nanoparticles was investigated by means of the SEM and confirms the nanostructure. It is possible to suggest that the organic fuel starch is responsible for the formation of nanostructure due to the easier complex formation. The EDAX is confirms the presence of NiO NPs. It confirms the presence of Nickel oxide nanoparticles. In the FTIR analysis, Ni-O stretching vibration mode is obtained in the region of 454.02cm^{-1}. As prepared NiO NPs are very effective to gram positive strains than the gram negative strains. Gram positive and gram negative bacteria have differences in their membrane structure, the most distinctive of which is the thickness of the peptidoglycan layer.

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