ISSN (Online): 0975-8232,
ISSN (Print): 2320-5148

INTERNATIONAL JOURNAL OF
PHARMACEUTICAL SCIENCES AND RESEARCH

An International Journal published monthly

An Official Publication of Society of Pharmaceutical Sciences and Research

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ELECTROCHEMICAL SYNTHESIS, CHARACTERIZATION AND ANTIMICROBIAL SCREENING OF NICKEL OXIDE NANOCLUSTERS

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ABSTRACT: The nanoclusters are having different properties than their bulk material. Hence, the synthesis and applications of nanoclusters of metal oxides have received more attention. Out of many available methods for the synthesis of metal oxide nanoclusters, the electro-chemical reduction method is simple, cheap, and provides pure material hence preferred. The present study describes the synthesis of nickel oxide nanoclusters by electrochemical reduction method wherein the nickel-metal sheet was used as a sacrificial anode while platinum was used as an inert cathode. The electrolysis was carried out at varied current density for 2 h in aqueous solutions using tetrabutylphosphonium bromide (TBPB) as a capping agent. The nanostructure of the synthesized nanoclusters has been analyzed using X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transforms infrared spectrometer (FT-IR), and UV-visspectro-photometer. The nickel oxide nanoclusters were screened for their antimicrobial potential against gram-positive and gram-negative bacterial strains of *Staphylococcus aureus* and *Escherichia coli* and found to have a broad spectrum of activity.

Keywords:

Electrochemical reduction, Nickel oxide, Nanoclusters, Tetrabutylphosphonium bromide, Antimicrobial activity

INTRODUCTION: Nanocluslers received consi-derable attention due to their unique properties entirely differing from bulk material [1]. The nanomaterials are known for their unique mechanical, chemical, physical, thermal, electrical, optical, magnetic, biological, and also specific surface area properties, which in turn define them as nanostructures, nanoelectronics, nanophotonics, nanobiomaterials, nanobioactivators, and nano-biolabels.

In the last decade, a large variety of nanomaterials and devices with new capabilities have been generated by employing nanoparticles based on metals, metal oxides, ceramics (both oxide and non-oxide), silicates, organics, and polymers [2]. There are several methods reported in the literature for the synthesis of nickel nanoparticles, including ultrasonic spray pyrolysis [3], liquid-control-precipitation [4], electrodeposition [5], chemical vapor deposition [6], the sol-gel route [7], reduction of metallic salts followed by oxidation of metallic species [8,] and microemuslion method [9].

The present work aims at synthesis of nickel oxide (NiO) nanoclusters by electrochemical reduction method, the analysis of synthesized nanoclusters by X-ray diffraction (XRD), scanning electron microscopy (SEM), Fourier transforms infrared spectrometer (FT-IR), UV-visspectro-photometer and antimicrobial activity screening.

MATERIALS AND METHODS:

Electrochemical Synthesis: In the preparation of nickel oxide nanoclusters, electrolysis cell vessel of volume capacity 20-50 ml and two electrode systems consisting of the bulk nickel sheet (1 × 1 cm) and the same inert cathode (1 × 1 cm platinum sheet). Both electrodes were immersed in 0.01 N aqueous solution of tetrabutylphosphonium bromide (TBPB). The tetrabutylphosphonium bromide serves not only as the supporting electrolyte but also as the stabilizer for nanoparticles to prevent their further, growth. During the synthesis, the bulk nickel metal is oxidized into nickel ion which reduced at inert cathode to form nickel oxide nanoparticles most probably at the interfacial region of the cathodic surface and within the electrolytic solution.

A controlled current electrolysis was carried out for 2 hrs and current density of 5, 10, 15 and 20 mA/cm^2. During the course of electrolysis, the solution became light green and the whitish green precipitate formed. After two hours electrolysis was stopped. The solution was transformed into bottle and after some time the solid particles were separated from the solution by a simple decantation process and washed for 3 to 4 times with water to remove unreacted tetrabutylphosphonium bromide.

Characterization of Nickel Oxide Nanoclusters: The nickel oxide nanoclusters were characterized by high end X-ray diffractometer, scanning electron microscopy (SEM), fourier transform infrared spectrometer (IR Affinity 1 Shimadzu) and UV-visible spectrophotometer (UV 1800 Shimadzu).

Antimicrobial Studies: *In-vitro* antibacterial activity of synthesized nickel oxide nanoclusters were tested against gram positive and gram negative bacterial strains of *Staphylococcus aureus* (NCIM-2079) and *Escherichia coli* (NCIM-2109) using the agar well diffusion assay method [10].

Approximately, 25 ml of molten and cooled nutrient agar media were poured in the sterilized petri dishes. The plates were left over night at room temperature to check for any contamination to appear. The bacterial test organisms were grown in nutrient broth for 24 h at 37 °C. A 100 µl nutrient broth culture of each bacterial organism was used to prepare bacterial lawns. Agar wells were prepared with the help of a sterilized stainless steel cork borer. The wells in each plate were loaded with 50 and 100 µl of 100 µg/ml of nickel oxide nanoclusters, and zone of inhibitions was measured in comparison with standard antibiotic ampicillin.

RESULTS AND DISCUSSION:

Electrochemical Synthesis: The compounds a tod were synthesized by an electrochemical method with appreciable yields. Samples were withdrawn after 15 min of electrolysis for UV-visible spectral analysis to check the growth of particle size.

UV-visible Studies: The compounds a tod were scanned from 200 nm to 800 nm using a double beam UV-visible spectrophotometer. The absorption maxima were observed to be in the UV region; hence all the compounds were scanned from 200 nm to 400 nm.

The overlain spectra with absorption maxima and absorbance of compounds a tod after 15 min of electrolysis are shown in Fig. 1. From the UV-visible spectral analysis, blue shift is observed in the nickel oxide nanoclusters when current density is increased during electrochemical synthesis.

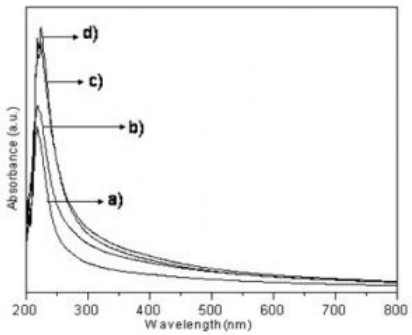

FIG. 1: UV-VISIBLE SPECTRA OF AS PREPARED SAMPLE OF NIO NANOCLUSTERS SYNTHESIZED USING TBPB AS CAPPING AGENT (0.01M) DISPERSED IN WATER AT A: 5, B: 10, C: 15, D:20 MA/CM^2

FT-IR Studies: The compounds a tod were calcinated by heating at 500 °C in muffle furnace. The FTIR spectra of nickel oxide nanoclusters (compound a tod) before and after calcination were recorded.

The FTIR spectral analysis reveals that after calcination the capping agent tetra-butylphos-phonium bromide has been completely removed from the nickel oxide nanoclusters. The absence of band at 2960.50 and 3641.40 cm^{-1} after calcination which was appeared in the FTIR spectra of the compound a before calcination Fig. 2 confirms that tetrabutylphosphonium bromide has been completely removed during calcination. Similar changes are observed in the FTIR spectra of compound b, c, and d also.

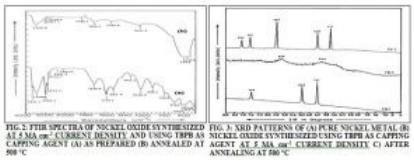

FIG. 2: FTIR SPECTRA OF NICKEL OXIDE SYNTHESIZED AT 5 MA cm^{-2} CURRENT DENSITY AND USING TBPB AS CAPPING AGENT (A) AS PREPARED (B) ANNEALED AT 500 °C

FIG. 3: XRD PATTERNS OF (A) PURE NICKEL METAL (B) NICKEL OXIDE SYNTHESIZED USING TBPB AS CAPPING AGENT AT 5 MA cm^{-2} CURRENT DENSITY C) AFTER ANNEALING AT 500 °C

XRD Studies: XRD patterns of nickel oxide samples prepared at 5 mA, 10 mA, 15 mA and 20 mA current density, heated at 500 ºC were obtained and are shown in Fig. 3.

The as-prepared samples are amorphous except the sample prepared at 5 mA. XRD pattern of as-prepared NiO at 5 mA shows that it is not completely in the amorphous phase.

Analysis of XRD patterns of annealed samples shows that there is no change in peak position corresponding to different d values indicating stable crystal structure.

The d values were determined by using Bragg's equation and compared with JCPDS data. The comparison shows that nickel oxide nanoparticles formed in the electrochemical reduction process were in the cubic phase (NiO).

SEM-EDS Studies: The EDS analysis of various regions confirmed the presence of nickel energy bands centered at 7.5 and 8.3 Kev (K lines) and 0.8 Kev (L lines). The oxygen detected is an oxide of nickel in nickel oxide nanoclusters with a percent composition of O K 23.96 weight %, 61.48 atomic %, and Ni K 55.08 weight %, 38.52 atomic %.

The weight and atomic percentage of oxygen and nickel clearly reveal the formation of nickel oxide nanoclusters. The SEM image of as-prepared nickel oxide nanoclusters capped with TBPB (0.01M) at various current densities is shown in Fig. 4 and 5, whereas the EDS spectrum in Fig. 6.

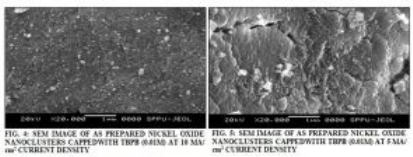

FIG. 4: SEM IMAGE OF AS PREPARED NICKEL OXIDE NANOCLUSTERS CAPPEDWITH TBPB (0.01M) AT 10 MA/ cm^2 CURRENT DENSITY

FIG. 5: SEM IMAGE OF AS PREPARED NICKEL OXIDE NANOCLUSTERS CAPPEDWITH TBPB (0.01M) AT 5 MA/ cm^2 CURRENT DENSITY

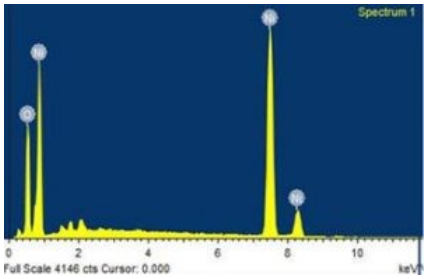

FIG. 6: EDS SPECTRUM OF AS PREPARED NICKEL OXIDE NANOCLUSTERS

Antimicrobial Studies: The antibacterial activity of synthesized nickel oxide nanoclusters in terms of zone of inhibition against gram-positive and gram-negative bacterial strains of *Staphylococcus aureus* and *Escherichia coli* is shown in Table 1. The antibacterial activity data reveals that nickel oxide nanoclusters show comparable antibacterial potential as that of ampicillin. The nickel oxide nanoclusters are observed to be more active against gram-positive than gram-negative bacterial strains.

TABLE 1: *IN-VITRO* ANTIBACTERIAL ACTIVITY OF SYNTHESIZED NICKEL OXIDE NANOCLUSTERS

Compound	Quantity	Antibacterial Activity (Zone of Inhibition in mm)	
		Staphylococcus aureus	*Escherichia coli*
NiO nanoclusters	50 µl	16	12
	100 µl	20	15
Ampicillin	50 µl	19	15
	100 µl	25	18

CONCLUSION: The nickel oxide nanoclusters were successfully synthesized with appreciable yields by an electrochemical method using tetra-butylphosphonium bromide as a capping agent. The characterization studies confirmed the formation of nanoclusters. The nickel oxide nanoclusters show broad-spectrum antimicrobial activity.

ACKNOWLEDGEMENT: Authors acknowledge the support of the Department of Physics, Savitribai Phule Pune University, Pune, for SEM and XRD analysis.

CONFLICTS OF INTEREST: Authors have no conflicts of interest.

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How to cite this article:

Sawant MR, Gaikwad ST and Rajbhoj AS: Electrochemical synthesis, characterization and antimicrobial screening of nickel oxide nanoclusters. Int J Pharm Sci & Res 2021; 12(1): 487-90. doi: 10.13040/IJPSR.0975-8232.12(1).487-90.

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Received: 13 January 2020

Revised: 10 April 2020

Accepted: 15 April 2020

DOI: 10.13040/IJPSR.0975-8232.12(1).487-90

Published: 01 January 2021

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