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ORIGINAL ARTICLE

Electrochemical Synthesis, Characterization, and Antibacterial Activity of Nickel Nanowires

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ABSTRACT

A major challenge in the treatment of infectious illnesses is multi-drug resistance, which can possibly be addressed using nanomaterials. The study investigates the synthesis of nickel nanowires followed by their characterization through various analytical methods including scanning electron microscopy, X-ray diffraction spectroscopy, and energy-dispersive X-ray spectroscopy. Additionally, the antibacterial properties of nickel nanowires were evaluated against Gram-positive (*Streptococcus pyogenes*) and Gram-negative (*Pseudomonas aeruginosa*, *Proteus mirabilis*, *Escherichia coli*, *Enterobacter*) bacterial species. Nickel nanowires depicted significant antibacterial properties against *Enterobacter, Proteus mirabilis*, and *Pseudomonas aeruginosa* activity than selected antibiotics called Levofloxacin. This revealed their effectiveness in combating resistance prevalent among bacteria across the world.

Keywords
Nickel Nanowires, Electrochemical, Synthesis, Characterization, Antibacterial.

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INTRODUCTION

In recent years, much scientific attention has been directed towards the development and application of nanomaterials in various fields. The unique properties of nanomaterials as compared to their bulk counterparts, establish them as prime candidates for future research[1]. Nanomaterials provide a range of interesting properties to experiment with and test for discovering novel applications and benefits. Physically, nanomaterials provide various benefits as compared to bulk materials such as a higher surface-to-volume ratio, high tensile strength, sustainability and ease of production, environmentally friendly synthesis and applications, conservation of finite natural resources, and their applicability to a large host of purposes [2,11].

The past decade has witnessed a notable increase in nanotechnology products and an increase in understanding and capabilities of the associated technology. Recent advancements in nanotechnology have made possible the miniaturization of electronic circuits and devices and have helped tackle age-old problems in the scientific community[3,12,13]. In the field of biotechnology, nanomaterials such as nanoparticles, nanofilms, and nanowires have opened new avenues for the treatment and cure of various ailments and diseases[4,17]. Nanomaterials have formed the basis of a new generation of biosensors due to their applicability in the synthesis of thin flexible films whereas nanoparticles have emerged as prime candidates in the field of targeted medicine, cancer treatment, and other such fields where traditional technologies have failed to produce results[5]. Keeping in mind these advancements, we can explore previously unexplored facets of applicability and experimentation about nanomaterials and nanoparticles.

One such pressing and concerning problem faced by the medical community in recent years is the growing resistance of bacteria and other microorganisms to antibiotics and other such anti-microbial agents. This has led to an increase in the severity of common diseases with a resultant increase in the threat of global epidemics arising due to the immunity of microorganisms to traditional medical agents such as antibiotics[5,14,15].

In the face of this challenge, the possibility of the use of nanomaterials for the targeted extermination of microorganisms through various mechanisms has arisen as a promising avenue of research and analysis. The use of nanomaterials for the treatment of cancerous cells has also been an object of intensive research in the past decade and has shown promising results [6,16]. The growing need for alternative methods of microbial control has led to the recent interest in the synthesis, characteristics, and application of nanomaterials such as nanoparticles and nanowires[7,16].

Nickel is a transition metal that has long been in use traditionally as a coinage metal due to its non-corrosive and lustrous nature. It is one of the four such elements, which are ferromagnetic at room temperature. Furthermore, nickel also serves as an essential nutrient in plants and animals that have nickel active sites in their enzymes [7]. Nickel is also highly conductive to heat, and electricity, and nickel nanomaterials display a very high compressive strength as compared to its bulk material[8,9]. Due to its ferromagnetic structure, the nanomaterials of nickel provide the option to be controllable and site-directed by localization using magnets or magnetic fields when applied to an experimental setting regarding micro-organisms where a controllable and regulated action is required[10,11]. Nickel is a highly biologically active element as it forms a part of many plant and animal enzymes and thus is a crucial part of various biological systems. Nickel also displays high biological toxicity and has also been found to cause cancer in cases where high exposure has been observed[8].

In the form of nanowires, nickel induces the lysis of biological cells such as bacteria or cancerous tissue through the process of internal entanglement with cellular structures. This property of nickel as a cytotoxic agent paired with the ability to control and confine its activity to a limited area using its ferromagnetic properties has created a niche for the use of nickel nanowires and potent antimicrobial and anticancer agent[9,12]. This study aimed at utilizing the most efficient methodology for the synthesis of nickel nanowires, followed by their physical characterization through a number of analytical methods and the application of these nanowires as antibacterial agents[17].

MATERIALS AND METHODS

Materials

The perchloric acid **(**$HClO_4$**),** ethanol (C_2H_5OH), phosphoric acid (H_3PO_4), aluminum oxide (Al_2O_3), boric acid (H_3BO_3), chromic acid (H_2CrO_4), nickel(II)sulfate hexahydrate ($NiSO_4.6H_2O$) and sodium hydroxide (NaOH) were used in this study. Aluminum sheets and electrodes are used to synthesize nanowires.

Bacterial Strains

Gram-positive (*Streptococcus pyogenes*) and Gram-negative (*Pseudomonas aeruginosa*, *Proteus mirabilis*, *Escherichia coli*, *Enterobacter*) bacterial strains were used in this study.

Synthesis of Nanowires

Aluminum sheet (Whatman, 99.9% pure) cut down 6cm x 6cm in size. Six strips made each 1 cm wide. The aluminum sheet was then washed with detergent to remove dirt on it. Then rinsed with distilled water and placed to dry at room temperature. Electro-polishing was performed in a solution of $HClO_4$ and C_2H_5OH prepared by mixing $HClO_4$ 20% and C_2H_5OH 80% (1:4 by volume). Before performing the polishing reaction, the solution was placed in a deep freezer to make the solution cool. The Al template is fixed in the electrode, acting as an anode. The reaction was performed by applying 9 volts DC voltage for 3-4 min. During this reaction, the surface of the aluminum sheet was made smooth and shiny. At the end of the reaction, the Al template was removed from the solution, rinsed with distilled water immediately, and placed to dry at room temperature. After getting the polished aluminum, then treated with a solution of phosphoric acid (5 mL) in 95 mL of distilled water. The solution was first placed in the freezer to cool for better performance. The aluminum template was fitted in the electrode and the template dipped in a chilled anodizing solution. The beaker was then placed in a stirrer plate, which was placed in the

refrigerator. The battery is connected in such a way that the positive terminal of the battery is connected to the Al template and the negative terminal of the battery is connected to the electrode. The first anodization was performed for 5 h under a constant DC voltage of 60 V at a current of 0.06 A. The Al_2O_3 membrane with vertical pores formed on the aluminum surface during the first anodization. The regular pores are made in the second anodization reaction. After 5 h, the connections were removed. The template was removed from the solution. Rinsed with distilled water and kept it to dry. To remove the irregular pores that were made during the first anodization the template was treated with a heating solution containing chromic acid and phosphoric acid. The template was placed in a clean petri dish then, solution was poured on it to dip the template. Then place in oven at 60°C for 3 h. After 3 h, the template was removed from the solution and then rinsed with distilled water properly. Kept template in a supporting material to dry. During the second anodization, the reaction was carried out for 20 h. The voltage and current were the same as in the first anodization reaction. This reaction gave regular and uniform size pores. The regularity of the pores depends upon the duration of the reaction. The anodizing solution was the same as was used in the first anodization reaction i.e. 5 mL H_3PO_4 in 95 mL of distilled water. After the second anodization, the barrier of the anodic aluminum oxide (AAO) template was removed. For this, an Al strip dipped in the solution was used for the anodizing reaction. The reaction was the same as anodizing at the start such as the voltage was 60 V and the current kept below 0.06 A, but after 1 h, the voltage stepped down after a specific interval of time i.e. every three minutes. During this process, the barrier layer is completely removed from the bottom of the templates. The Al strips were then removed from the solution and apparatus, rinsed with distilled water, and kept to dry. Now the template is ready for the electrodeposition of nanowires. The aluminum strips were then fixed in the electrode in the same way as done for the anodization process. This apparatus is dipped in the 0.1 M solution of $NiSO_4.6H_2O$. The ingredients of the solution were $NiSO_4.6H_2O$ (2.62 g), distilled water (100 mL), and boric acid (0.5 g) used as a buffer. An AC voltage of 15 volts was applied for 1 minute. Nickel is deposited in the pores. The electrodeposited nickel nanowires were then etched in sodium hydroxide (NaOH). Two molar solutions of NaOH were prepared. For this, 8 g of NaOH was added to 100 mL of distilled water. The small pieces of aluminum templates containing nickel dipped in their pores in the NaOH solution. Nickle nanowires were removed from the pores in the solution. A magnet was placed below the beaker; the magnet attracted the nickel nanowires towards itself. The solution of NaOH was then removed with the help of a dropper. These nanowires were washed 5 times with distilled water to remove NaOH if present. The nickel nanowires were then put in the Eppendorf tube and placed to dry to get them in the powdered form.

Characterization of Nanowires

Scanning Electron Microscopy (SEM)

Scanning electron microscopy is an advanced imaging technique used to identify the morphological structures of Nano scale materials and scanning electron micrographs of nickel nanowires were taken.

X-Ray Diffraction (XRD) Spectroscopy

This method of characterization provided a spectrum of absorption related to the energy absorbed by nanowires when exposed to different wavelengths of X-rays. This absorption spectrum is then compared to a standard to obtain data regarding constituent materials of the target based on the absorption behavior of nanowires and is utilized to give an idea of the crystalline morphology of nanowires.

Energy Dispersive X-ray (EDX) Spectroscopy

EDX was performed on the sample to confirm the presence of nickel nanowires in the Anodic Aluminium Oxide (AAO) sample and to study the structure of the nickel nanowires based on crystal structure.

Biological Testing of Nanowires

The nanowires, after physical characterization found to be of the required specification, and, the next step of an experiment was conducted by application of these nickel nanowires as antibacterial agents. Selected bacteria mentioned above, were cultured through the streaking method on a petri dish having nutrient media composed of peptone (5 g), beef extract (3 g) sodium chloride (5 g), and agar (15 g). The solution of nanowires was prepared by diluting 5 mg of nanowires in 1 mL distilled water after

which the solution was sonicated for 30 min to obtain uniform dispersion of nanowires throughout the solution. The agar well diffusion method was used to assess the antibacterial potential of these nanowires. Levofloxacin is used as a reference antibiotic (AB). The prepared plates were then incubated for 24 h at 37°C to obtain the growth of each selected bacterium. After 24 h of incubation, the zone of inhibition was taken via ruler.

RESULTS

Characterization Studies

SEM Analysis

The scanning electron microscopy depicted the honeycomb like morphology of nickel nanowires (Figure **1 A-B**).

XRD Analysis

A consistent presence of nickel deposited onto the template along with the presence of the AAO template was evident in Figure **2**. The Ball and stick model of nickel nanowires is given in Figure **3**. The size of the nickel crystals deposited onto the template was in nano range and constitute a greater part of the sample (Table **1**).

EDX Spectroscopy

The EDS analysis (Figure **4**) determines the presence of nickel crystals in the sample where weightage is less due to the presence of the AAO template nevertheless the nickel nanowires deposited onto the template are confirmed by the results mentioned in Figure **4**.

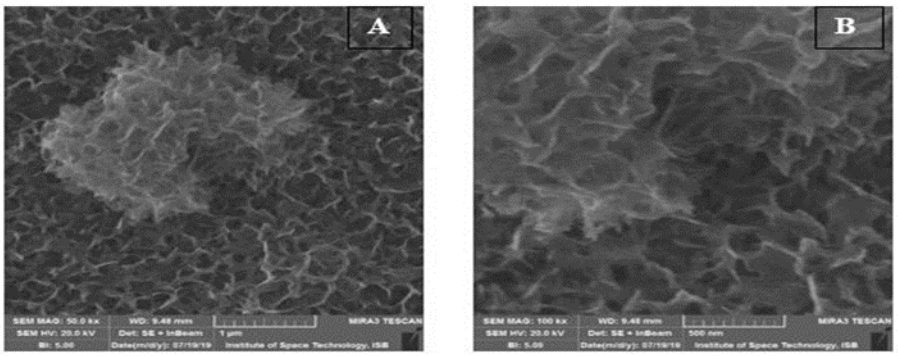

Figure 1. Scanning electron micrographs of nickel nanowires at 1 µm (A) and 500 nm (B).

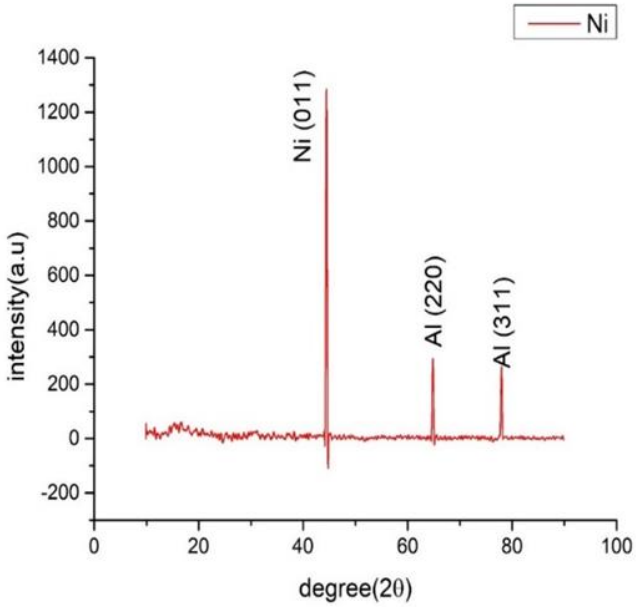

Figure 2. XRD pattern of nickel sample.

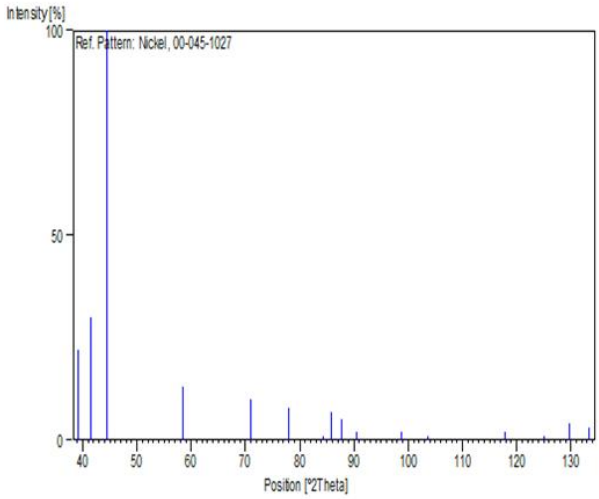

Figure 3. Ball and stick pattern of nickel nanowires made via XRD analysis.

Table 1. An Overview of Hkl Planes of Nickel Nanowires.

H	k	l	d[A]	2 Theta[deg]	I [%]
0	1	0	2.30189	39.101	22
0	0	2	2.17264	41.531	30
0	1	1	2.03342	44.521	100
0	1	2	1.57840	58.422	13
1	1	0	1.32679	70.982	10
1	0	3	1.22399	78.002	8
2	0	0	1.14850	84.242	1
1	1	2	1.13167	85.793	7
2	0	1	1.11037	87.853	5
0	0	4	1.08434	90.533	2
2	0	2	1.01503	98.734	2
0	1	4	0.98038	103.574	1
2	0	3	0.89897	117.935	2
2	1	0	0.86777	125.166	1
2	1	1	0.85088	129.726	4
1	1	4	0.83899	133.307	3

Biological Application

The resultant antimicrobial activity of the nanowires on the selected bacteria was studied based on the size of the zone of inhibition created by the bacteria in each of the bacterial samples. The results demonstrated clear antibacterial activities against the selected bacteria being comparable in strength to the reference antibiotic and in a few cases even surpassing the effects of the reference antibiotic having a much greater antibiotic effect on the selected bacteria (Figure **6 A-E**).

An overview of the antibacterial activities of nickel nanowires in terms of zones of inhibition against different bacterial strains was done (Figure **7**). Thus, results exhibited the promising effectiveness of nanowires solution in combating the bacteria present in each sample. The most promising result was observed in the case of *Enterobacter* in terms of higher zones of inhibition as compared to other tested bacterial strains including *Streptococcus pyogenes*, *Pseudomonas aeruginosa*, *Proteus mirabilis*, and *Escherichia coli*.

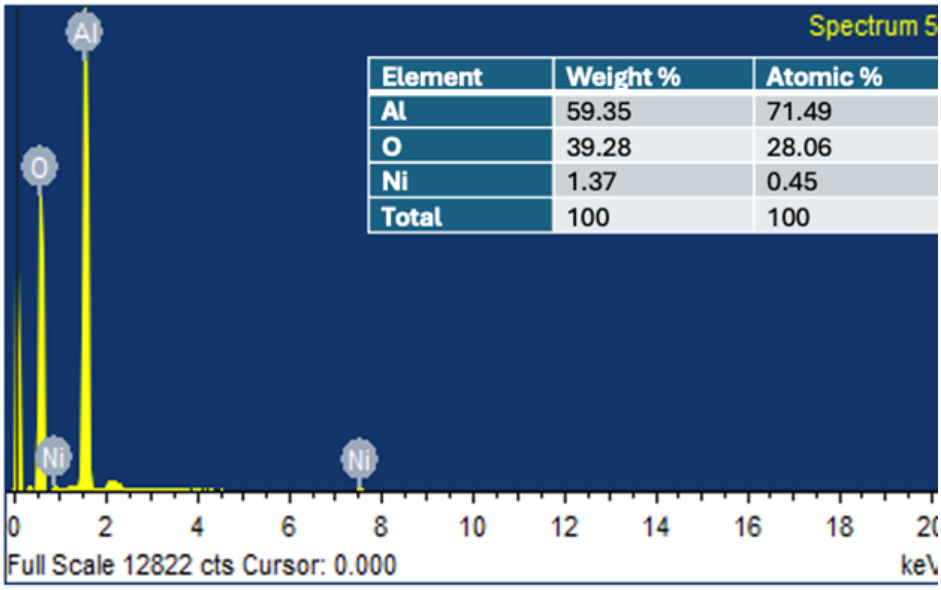

Figure 5. EDX analysis of nickel nanowires.

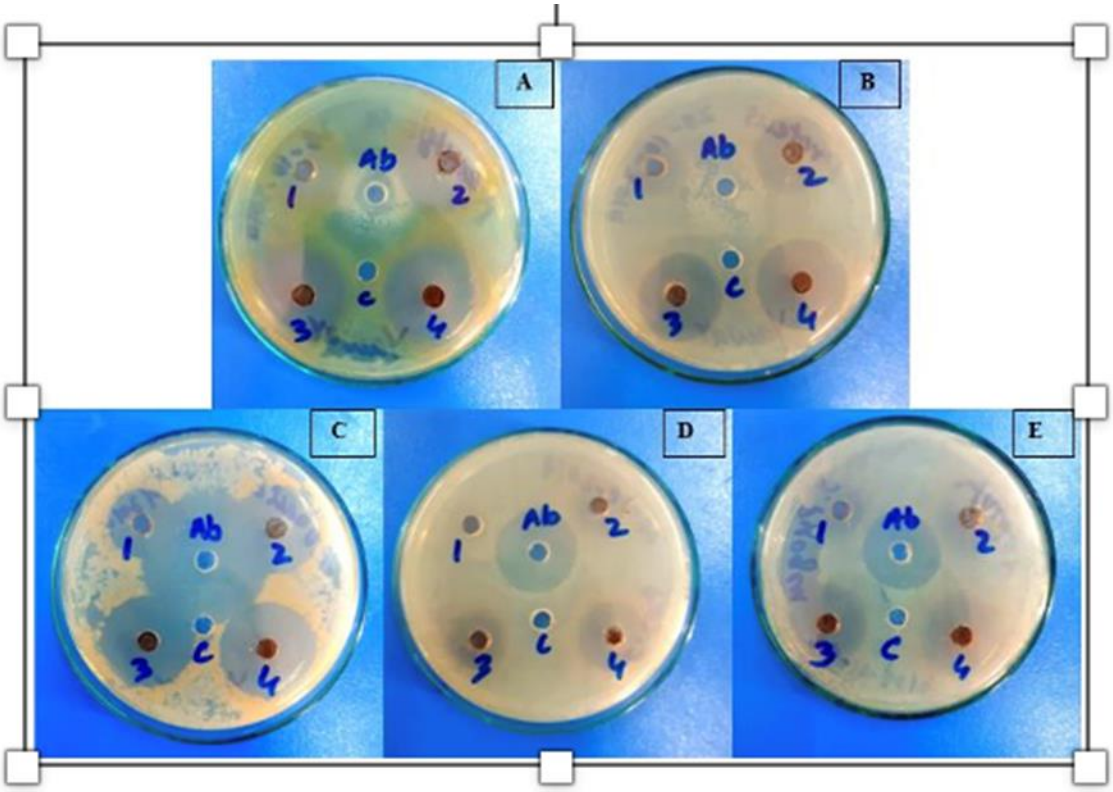

Figure 6. Antibacterial activities of nickel nanowires against *P. aeruginosa* (A), *E. coli* (B), *Enterobacter* (C), *P. mirabilis* (D), *S. pyogenes* (E).

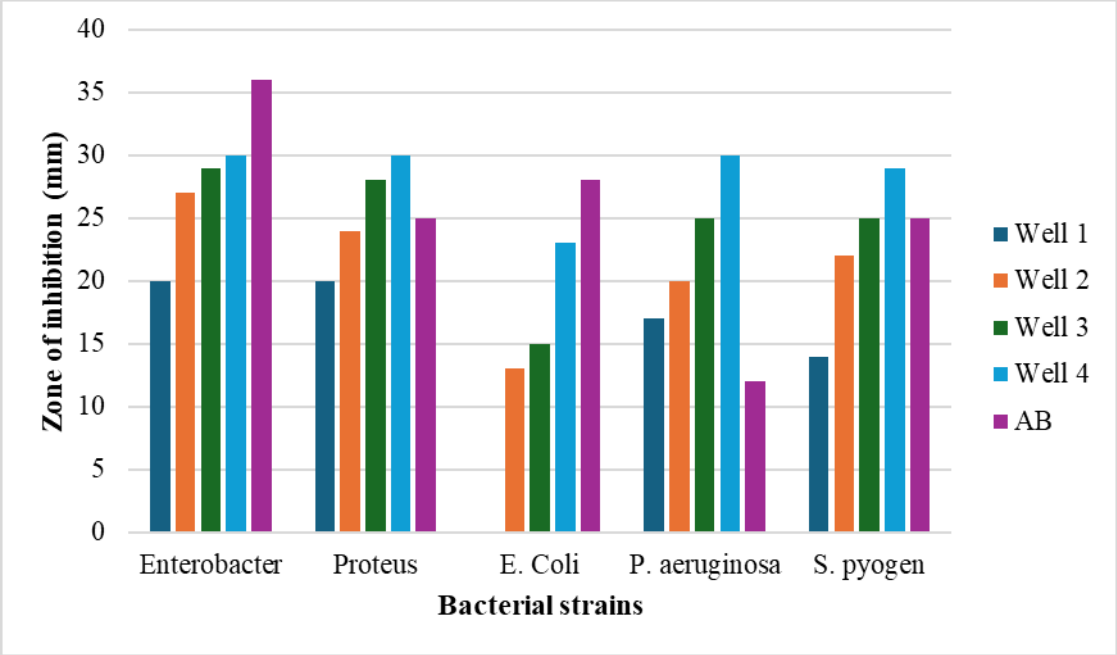

Figure 7. Comparison of antibacterial activities of nickel nanowires against different bacterial strains. [Concentration of nickel nanowires in wells (1-4) was 40 mg/mL, 20 mg/mL, 10 mg/mL, 5mg/mL and antibiotic (AB) was 5 mg/mL].

DISCUSSION

The results obtained from the characterization of the nickel nanowire samples provided proof of the synthesis of clear and unbranched nickel nanowires deposited onto the pores of the AAO template[10]. The SEM imaging displayed clear-deposited nickel nanowires onto the template and results from the spectroscopic analysis of the samples on the other hand provided clear quantitative proof of the presence of the nickel nanowires on the sample and insight into the crystal structure of the nickel nanowires[18].

In the experiment, the nickel nanowire solution displayed extremely promising effectiveness against the selected bacterial species. Where in most cases the zone created by the nanowires was greater than the zone created by the antibiotic, in other cases the zone was comparable to that of the antibiotic and in only a few cases the activity of the antibiotic was greater than the nanowires[19]. This effectiveness of the nanowires displays their great potential to be used as antibiotic agents against a wide range of gram-negative and gram-positive bacteria as their inhibitory effects on the growth of the bacteria are incredibly promising and comparable to or even surpassing the effectiveness of the commonly used antibiotic solutions present for use today[20].

In this light, the use of the nickel nanowires synthesized through the template-assisted approach holds great promise for various applications in nanotechnology and materials science. The electrodeposition method shows not only the possibility of creating a functioning and high-quality nanowires in one dimension but also their application to various biological agents[21]. This revealed their effectiveness for the combating of antimicrobial resistance prevalent among bacteria across the world by providing a physical methodology for the lysis and control of bacterial cells, and populations without having the risk of creating a possibility for resistance in the target bacteria[22,23].

CONCLUSION

The study investigated the synthesis of nickel nanowires followed by their characterization through various analytical methods i.e. XRD, SEM, and EDX analysis. Moreover, the effectiveness of nickel nanowires as an antibacterial agent against Gram-positive and Gram-negative disease-causing bacterial species was checked. The results showed greater effectiveness as compared to the accessible antibiotic against the selected bacterial species.

CONFLICT OF INTEREST

The authors declare no conflict of interest with any organization, party, or individual.

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