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GREEN SYNTHESIS OF NICKEL NANOPARTICLES USING *PEUMUS BOLDUS* KOCH. EXTRACT AND ANTIBACTERIAL ACTIVITY

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Abstract

Original scientific paper

Today, nano-systems, especially multi-functional inorganic nanoparticles, attract a lot of attention in the biomedical field. Nickel nanoparticles, known as inorganic nanoparticles in nanotechnology and known as metallic nanoparticles with an important place, show antibacterial properties. The fact that nickel nanoparticles find use in areas requiring human contact leads to nanoparticle synthesis with biological systems. Thus, more environmentally friendly structures emerge. In this study, nickel nanoparticles were synthesized for the first time by biosynthesis using *Peumus boldus* extract. Structural, chemical, and morphological properties were examined by UV-vis spectrophotometer (UV-vis), fourier transform infrared spectroscopy (FTIR), X-ray diffractometer (XRD), and scanning electron microscopy (SEM) methods. Extract obtained from *Peumus boldus* was used as reducing agent in the synthesis of nickel nanoparticles. In addition, the antibacterial activities of the obtained nickel nanoparticles were investigated against *E. coli* and *S. aureus.*

Keywords: *Nickel nanoparticle, peumus boldus, antibacterial activity*

PEUMUS BOLDUS KOCH ÖZÜTÜ KULLANILARAK NİKEL NANOPARTİKÜLLERİN YEŞİL SENTEZİ VE ANTİBAKTERİYEL AKTİVİTESİ

Özet

Orjinal bilimsel makale

Günümüzde nano sistemler, özellikle çok fonksiyonlu inorganik nanopartiküller, biyomedikal alanında büyük ilgi görmektedir. Nanoteknolojide inorganik ve metalik nanopartiküller olarak bilinen ve önemli bir yeri olan nikel nanopartiküller antibakteriyel özellik göstermektedir. Nikel nanoparçacıklarının insan teması gerektiren alanlarda kullanım bulması biyolojik sistemlerle nanoparçacık sentezine yol açmaktadır. Böylece daha çevreci yapılar ortaya çıkmaktadır. Bu çalışmada, nikel nanoparçacıkları, *Peumus boldus* özütü kullanılarak biyosentez yoluyla ilk kez sentezlenmiştir. Yapısal, kimyasal ve morfolojik özellikler UV-vis spektrofotometresi (UV-vis), fourier transform kızılötesi spektroskopisi (FTIR), X-ışını difraktometresi (XRD) ve taramalı elektron mikroskobu (SEM) yöntemleri ile incelenmiştir. Nikel nanoparçacıkların sentezinde indirgeyici ajan olarak *Peumus boldus*'tan elde edilen ekstrakt kullanılmıştır. Ayrıca elde edilen nikel nanoparçacıkların *E. coli* ve *S. aureus*'a karşı antibakteriyel aktiviteleri araştırıldı.

Anahtar Kelimeler: *Nikel nanopartikül, peumus boldus, antibakteriyel aktivite*

1 Introduction

Nanotechnology covers the fields of study dealing with the synthesis, characterization, and applications of substances generally between 1-100 nm sizes. In this size range, all properties (chemical, physical, and biological) of individual atoms / molecules vary. New applications of these nanoparticles used in nanotechnology can be developed depending on the particle diameter, homogeneous distribution, and morphology, and thanks to these properties, it is growing rapidly. NPs are widely used as antibacterial agents in the healthcare industry, food storage, textile coatings, and numerous environmental applications. As antibacterial agents, NPs have been used in a wide range of applications, from disinfecting medical devices and white goods to water treatment. In addition to these, NPs are used in fabrics in the textile industry. [1, 2].

Among the nanoparticles used for all the purposes mentioned above, metallic nanoparticles show promise. Because metal ions contain important antibacterial properties due to their large surface area. Thus, they attract the attention of researchers due to the increased microbial resistance against antibiotics and the development of these resistant strains [3, 4]. Nano metals have attracted great interest in branches such as physics, technology,

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chemistry, and bioengineering. Production and characterization of nickel nanoparticles are very important in terms of nanotechnology.

Among all metal nanoparticles, nickel nanoparticles attract unlimited attention in the field of nanotechnology due to their unique properties such as chemical stability, good conductivity, catalytic, and most importantly antibacterial, antiviral, antifungal [5, 6].

Generally, nickel nanoparticles are synthesized using various techniques to obtain different shapes and sizes for use in various applications. Various chemical and physical preparation methods are used to produce nanoparticles, including radiation, chemical precipitation, photochemical methods, electrochemical, and Langmuir-Blodgett techniques [7, 8]. The use of chemical and physical method in the synthesis of nanoparticles is very expensive and cumbersome and leads to the presence of some toxic chemicals that are absorbed on the surface and may have adverse effects on applications, so there is an increasing need to develop environmentally benign nanoparticles [9, 10]. Biosynthetic methods using microorganisms or plant extracts have emerged as a simple and viable alternative to chemical synthetic procedures and physical methods [11, 12].

Green synthesis of nanoparticles using plant extracts, due to their easy accessibility, is emerging as an important research topic in the field of bionanotechnology today [13]. The synthesis of nanoparticles by means of plants is simpler and easier compared to typical physical and chemical methods as it can be studied without any special operating conditions. Synthesized products of the process, including waste products, are derived from natural plant extracts and thus this technique is highly environmentally friendly [14]. With the bio-based nanoparticle synthesis protocol, higher reproducibility of the process and higher stability of the synthesized nanoparticles can be achieved. Therefore, the bio-based production of nanoparticles is suitable for large-scale production that is more effective cost investment, environmentally friendly and safe for human therapeutic use [15].

The main purpose of this study is to produce and characterize nickel nanoparticles with green synthesis method. For this purpose, the synthesis of nickel nanoparticles with the reducing properties of molecules such as flavonoids and terpenoids found in the extract of *Peumus boldus* was carried out. The synthesized nickel nanoparticles were characterized by UV-Vis, FTIR, and powder XRD techniques. At the same time, the antimicrobial activity of nickel nanoparticles synthesized by the green synthesis method was investigated. The results showed that with *Peumus boldus* extract, nickel nanoparticles can be produced by green synthesis.

2 Material and Method

The *Peumus boldus* plant was brought from Chile. Aqueous extracts were prepared by putting 10 g of the homogenized dried leaves and 100 mL of distilled water in a Soxhlet extractor for 4–5 h.

Biosynthesis was carried out by mixing *Peumus boldus* extracts with aqueous solution of $NiNO_3.6H_2O$ (Sigma-Aldrich), and the solution was homogenized by stirring at room temperature. For the synthesis of NiNP, 0.1 M 100 ml nickel (II) nitrate $NiNO_3.6H_2O$ solution was prepared. Nickel (II) nitrate (10 ml) solution was slowly added to the resulting *Peumus boldus* extract (100 ml) at a rate of 10:1 (Fig.1).

The optical properties of NiNPs were characterized by taking UV-Vis region spectra (THERMO, Model Multiscaner spectrophotometer) between 200-800 nm. FTIR (Shimadzu Iraffinity-1) was used for functional group analysis. Particle morphologies and sizes were measured by SEM (TESCAN, MAIA3 XM). The crystal structures and particle sizes of NiNPs were calculated using XRD analysis. XRD analysis was performed using a powder X-ray diffractometer (Rigaku, Smartlab).

Antibacterial activity of NiNPs was assessed using Broth Micro-dilution Assay [16]. For this, Gram-positive bacterium *Staphylococcus aureus* and Gram-negative bacterium *Escherichia coli* cultures were grown on LB Broth (24 h, 37ºC). Following this, new cultures were prepared and grown until 0.5 McFarland Unit. 20 µL of bacterial cultures were added to 180 µL of Nutrient Broth containing various concentrations of NiNPs in microtiter plate wells. Negative controls were prepared using the medium without bacteria. OD_{600} of microtiter plates were read before and after incubation at 37ºC for 24 hours.

Figure 1. Schematic representation of NiNP synthesis.

3 Results and Discussion

UV-Visible spectra of nickel nanoparticle synthesized from *Peumus boldus* by green synthesis shown in Figure 2. NiNp gives a strong absorbance peak at 656 nm. When the UV-Visible spectrum is examined, it shows that NiNps are homogeneous. The maximum band in the UV-visible spectrum is due to the oscillation of the nickel conduction band electron. In the UV-Visible spectrum of the nickel nanoparticle solution dispersed in water, the maximum absorbance was observed at 656 nm wavelength.

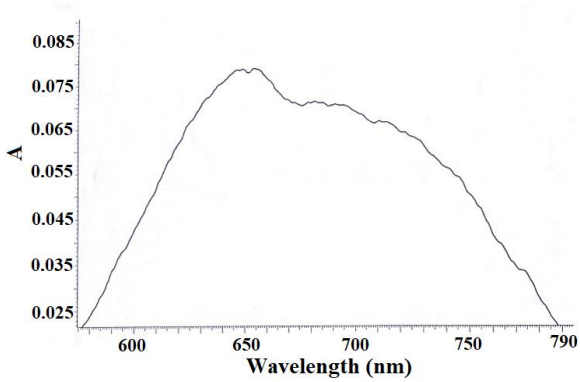

Figure 2. UV-visible spectrum of NiNPs synthesised at *Peumus boldus* extract

The FTIR spectrum of biologically synthesized nickel nanoparticles is shown in Figure 3. When the FTIR spectrum of nickel nanoparticles was examined, it showed a transmission peak at 3370, 2967, 2889, 1587, 1376, and 1047 cm^{-1}. The peak at 1047 is thought to be due to saturated alkanes, while the peak at 1376 is due to alcohol and phenol. The peak at 1587 indicates amide, the peak at 3500 indicates the hydrogen bonded alcohol and phenol.

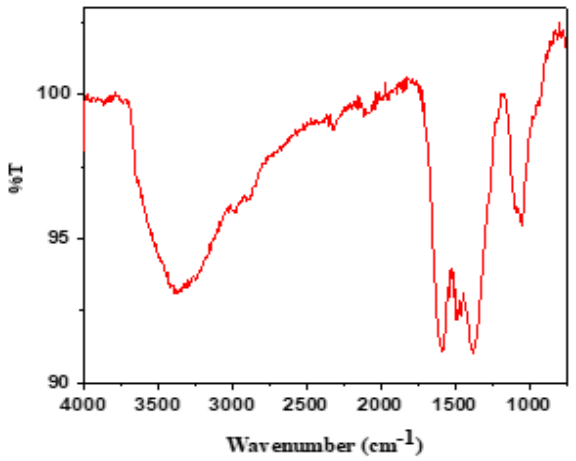

Figure 3. FTIR spectra of synthesized Ni nanoparticles by *Peumus boldus* extract

In order to study the morphology and size of the biosynthesized NiNPs, SEM images were recorded at different magnifications (Fig. 4). The formation of NiNPs as well as their morphological dimensions through the SEM study demonstrated that the average size was around 15-20 nm with the shape of spherical nature. The SEM image further confirms the production of a high density of NiNPs synthesized through the *Peumus boldus* extract.

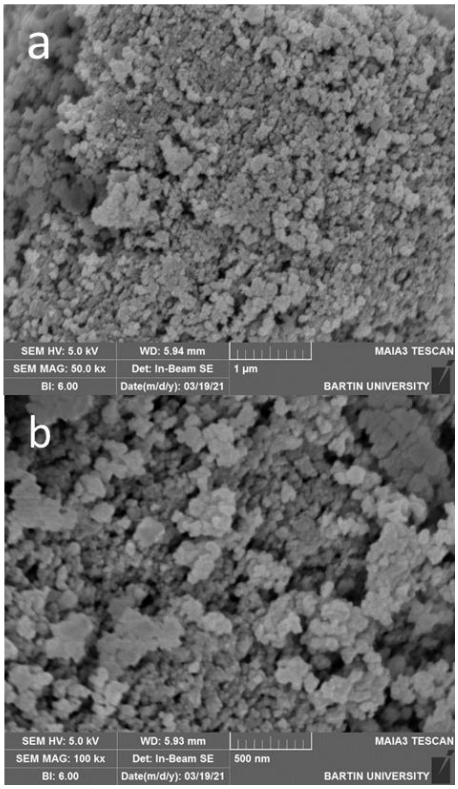

Figure 4. SEM of NiNPs biosynthesized using the *Peumus boldus* aqueous extract

The crystalline structures of the NiNPs were characterized by XRD. The major diffraction peaks appear at 2θ, 36.85, 42.90, and 62.49, which can be indexed as (111), (200), and (220) planes. The crystalline structure of NiO NPs is in accordance with JCPDS card No. 04-0835. No impurities were observed which suggests the high purity of monophasic Ni nanoparticles

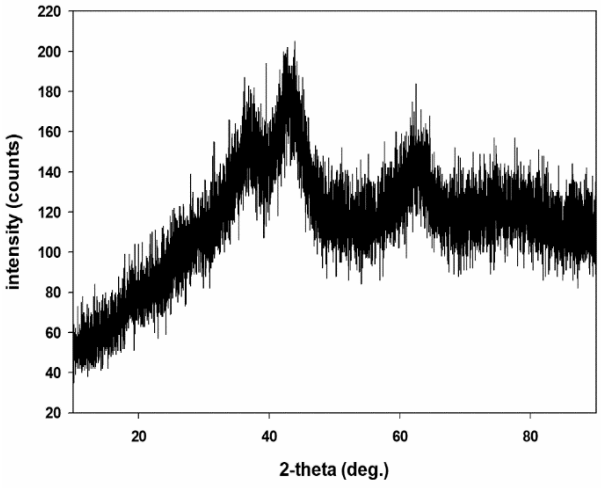

Figure 5. X-ray diffractogram of *Peumus boldus* extract – reduced NiNPs.

NiNPs were used to assess their antibacterial properties against very common bacteria, *E. coli* and *S. aureus.* The results showed that these nanoparticles are very effective against Gram-negative bacterium *E. coli* (Figure 6A), especially at the highest concentration (800 µg/mL) the bacterial viability was found as around 1.7% as compared to control group (0 µg/mL). On the other hand, these nanoparticles are also effective against Gram-positive bacterium *S. aureus* but this effect is lesser (Figure 6B).

Previous studies also showed that Nickel nanoparticles have antibacterial activities on these bacteria [17]. These nanoparticles can alter the bacterial cell membrane permeability, thus transport through membranes are improper, leading to cell death [18]. Furthermore, they can penetrate into the cell and release Ni ions, which damage the biomolecules that contain phosphorous and sulfur, such as DNA [19].

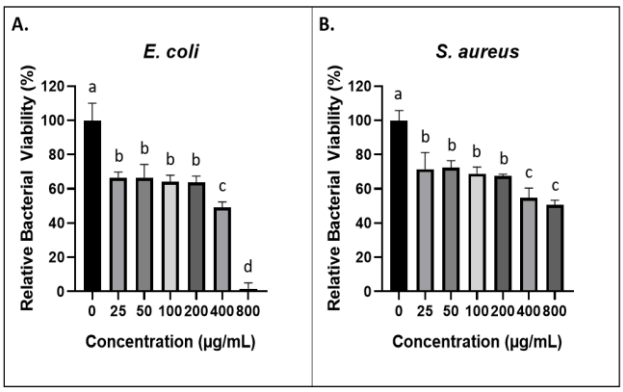

Figure 6. Antibacterial activities of NiNPs against (A.) *E. coli* and (B.) *S. Aureus.* Different lower cases indicate that the mean values are statistically different according to One-way ANOVA ($p<0.05$)

4 Conclusion

Nanotechnology is an important area of research that is a pioneer of different technologies and fundamental innovations, and it is expected to inspire many other important innovations in the future. Today, one of the most important outputs of these innovations is the production of nickel nanoparticles with the green synthesis method. The NiNPs were successfully fabricated via green route using *Peumus boldus* extract, which were confirmed by UV–vis, XRD, FTIR, and SEM techniques. SEM images of the synthesized silver nanoparticles show that the particles have a spherical structure and an average diameter of 15-20 nm. FTIR results, which help to understand the relationship of functional groups between metal nanoparticles and biomolecules, at 3370 cm^{-1} peak value confirm the presence of -NH and -OH groups in the *Peumus boldus* extract rich in flavonoids and terpenoids. With this result, it was confirmed that $NiNO_3$ was reduced due to the presence of -OH group. In UV-Vis spectrum analysis, the presence of a strong and wide absorption band with a peak located at 656 nm and the observation of its change from transparent to brown hues during synthesis are proof of the success of nickel nanoparticle synthesis.

In the present study, antibacterial properties of NiNPs were studied and the results showed that these nanoparticles have significant effects on both *E. coli* and *S. aureus*, but the effect on *E. coli* was found higher than the effect on *S. aureus.*

Due to the effective biological, pharmacological, and antioxidant properties of silver nanoparticles obtained by the green synthesis method from *Peumus boldus*, a more comprehensive in vitro and in vivo study is needed together with phytochemical studies in order to expand its daily use.

Acknowledgements

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Declaration

Ethics committee approval is not required for this study.

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