Review of: "Synthesis of Nickel Nanoparticles Using Ionic Liquid-Based Extract from Amaranthus viridis and Their Antibacterial Activity"

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Potential competing interests: No potential competing interests to declare.

Review report

This study presents a novel method for synthesizing nickel nanoparticles (Ni NPs) using an environmentally friendly approach. Habib Ullah and his associates utilize an ionic liquid-based extract from *Amaranthus viridis*, combined with microwave assistance, to produce Ni-NPs. The significance of this work lies in its potential to offer a more sustainable and efficient alternative to traditional nanoparticle synthesis methods, which often involve harsh chemicals and energy-intensive processes.

The use of plant extracts for nanoparticle synthesis (green synthesis) has gained attention in recent years due to its eco-friendly nature. However, the innovation in this study is the combination of ionic liquids and microwave assistance, which could potentially enhance the extraction efficiency and provide better control over nanoparticle formation.

Following are the major strengths of the study:

- Novel synthesis approach combining ionic liquids, plant extract, and microwave assistance.
- Comprehensive characterization of the nanoparticles using multiple techniques.
- Demonstration of antibacterial activity, suggesting potential practical applications.
- Use of a green synthesis method, contributing to more sustainable nanotechnology practices.

However, the authors are suggested to improve the manuscript based on the following observations:

Suggested Methodology Improvements

Control experiments:

Include a control group using conventional Ni NP synthesis methods for direct comparison.

Conduct experiments with just the ionic liquid and just the plant extract to isolate their individual effects.

Optimization studies:

Discuss the systematic optimization of synthesis parameters (temperature, time, concentration, microwave power) using a

design of experiments (DOE) approach.

Mechanism elucidation:

Authors are suggested to highlight the phytochemical composition of the Amaranthus viridis extract to discuss the possible role of specific plant metabolites in the reduction and stabilization of Ni NPs.

Expanded characterization:

It is suggested to include X-ray photoelectron spectroscopy (XPS) analysis to confirm the oxidation state of nickel.

Biological studies:

Authors are suggested to determine minimum inhibitory concentrations (MICs) for each bacterial strain.

More quantitative antibacterial tests and comparisons with standard antibiotics would provide better context for the efficacy of nanoparticles.

It is suggested to include cytotoxicity studies on mammalian cell lines to assess biocompatibility.

Also, the manuscript will be improved if the authors are able to investigate the mechanism of antibacterial action (e.g., membrane damage, ROS generation).

Statistical analysis:

Authors are suggested to include appropriate statistical tests for all quantitative data.

Also, include p-values and confidence intervals where relevant.

Figures:

Improve the quality and resolution of all figures, especially FESEM images.

It is also suggested to add scale bars to all microscopy images.

If possible, use color-coding consistently across different characterization techniques for easier comparison.

Comparative analysis:

Include a table comparing the properties of the synthesized Ni NPs with those reported in the literature for other green synthesis methods.

Size distribution analysis:

Present a histogram of nanoparticle size distribution from FESEM or TEM analysis, not just DLS.

Antibacterial activity:

Use a bar graph to clearly show the zones of inhibition for different concentrations and bacterial strains.

Include error bars to represent the variability in measurements.

Correlation analysis:

Investigate and present any correlations between nanoparticle characteristics (size, zeta potential) and antibacterial efficacy.

In the introduction section, please include a more comprehensive overview of green synthesis methods for metal nanoparticles.

Please include a discussion section at the end of the results section by considering the following points:

1. Include a critical comparison of ionic liquid-based methods with other green synthesis approaches.
2. The authors are suggested to discuss whether the synthesis conditions (e.g., microwave power, time, ionic liquid concentration) were optimized.
3. A comparison of this method with traditional Ni NP synthesis techniques or other green synthesis methods would strengthen the paper.
4. Propose and discuss possible mechanisms for the formation of Ni NPs using this method, based on current understanding in the field.
5. Expand on potential applications beyond antibacterial use, such as catalysis or magnetic applications.
6. Discuss how the properties of these Ni NPs might be advantageous or limiting for various applications.
7. Clearly state the limitations of the current study.
8. Propose specific future experiments to address these limitations and further advance the field.
9. Discuss the environmental impact of this synthesis method compared to conventional methods.
10. Address any potential concerns about using ionic liquids in green synthesis.
11. Discuss the potential for scaling up this synthesis method for industrial applications.
12. Provide a brief cost analysis comparing this method to conventional Ni NP synthesis.

Authors are also requested to rectify the following typographical errors in the manuscript:

1. In the Introduction section: "It employs ionic-liquids to decrease the metal particles to nanoparticle size." The hyphen in "ionic-liquids" is unnecessary.
2. In section 2.2 (Synthesis of nickel nanoparticle): " Thermogravimetric analysis of Ni NPs was performed using Mettle Toledo SDTA 10000, TG, at a heating rate of 10oC/min and a temperature range of 50 to 900^{o}C." There's an inconsistency in the use of the degree symbol.
3. In section 2.3 (Characterization): "Zeiss Supra 55VP FESEM equipment operating at 5 kV was utilized to determine the surface morphology of Ni NPs at different magnifications. Prior to picture observation, the nanoparticles were sufficiently coated with gold water to ensure conductivity." "gold water" should be "gold layer".
4. In section 2.4 (Anti-bacterial activity): " Synthetic nanoparticles were tested against Aeromonas hydrophilia,

Staphylococcus aureus, and Escherichia coli to determine their antibacterial activity. --------- After being incubated for 24 hours at 37°C, E. coli, S. aureus, and A. hydrophilia were injected into nutritional broth." There are several issues with this section: a. the scientific names of the bacterial species should be written in italics. There are spelling mistakes in the species name of the bacteria *Aeromonas hydrophila* throughout the manuscript. Instead of injected, please use the term ‘inoculated’.

5. In section 3.1 (UV-Vis analysis of Ni NPs): The entire first paragraph of this section is a repetition of section 2.4, which seems to be a copy-paste error. Please rectify this section. Write in detail about the UV-Vis analysis of Ni NPs).
6. In section 3.2 (FTIR analysis): "These peaks show that the production of Ni NPs and the decrease of nickel ion may be caused by secondary metabolites of the plant." "decrease" should be "reduction".
7. In section 3.4 (Thermogravimetric analysis): " The remaining mass doesn't change beyond 530oC." Inconsistent use of the degree symbol (oC instead of °C)
8. In section 3.6 (Zeta size and zeta potential): "Ni NPs have a zeta potential of -41 mV on average. The remarkable stability of the nanoparticles is indicated by this valve." "valve" should be "value".
9. In section 3.7 (Anti-bacterial activity): "Three distinct gram positive and gram negative bacterial strains were evaluated using the produced Ni NPs ionic liquids-based extract from Amaranthus viridus." "Amaranthus viridus" should be "Amaranthus viridis" (consistent with the title and earlier mentions)

Overall, this research contributes valuable insights to the field of green nanotechnology and opens up avenues for further research into sustainable nanoparticle synthesis and their applications in antimicrobial treatments.