

SYNTHESIS, CHARACTERIZATION, AND ANTIBACTERIAL EVALUATION OF NICKEL-CONTAINING BIOGENIC ORGANIC COMPOSITE DERIVED FROM ORANGE PEEL

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Received: 18 November 2025; Revised: 22 April 2026; Accepted: 6 May 2026

ABSTRACT

This study focuses on the synthesis, characterization, and antibacterial activity of a nickel-containing biogenic organic composite (NBOH) material using orange peel extract. Given the limited research on the use of Ni²⁺ ions in the formation of metal–biogenic organic networks, this work aims to expand the understanding of Ni-organic networks produced under mild reaction conditions. The composite was characterized by SEM-EDS, XPS, and XRD, and its antibacterial activity was evaluated using the agar diffusion method. SEM micrographs display irregular, loosely packed flake-like, blocky aggregates with rough surfaces, consistent with a fluffy, dispersed morphology as observed visually. The EDX analysis revealed the presence of C, O, and Ni as principal components with elemental weight percentages of C (31.04%), O (33.25%), and Ni (31.92%). According to XRD analysis, NBOH was predominantly amorphous, as demonstrated by a broad diffraction peak in the 2θ range of 20–45° without sharp reflections. The antibacterial tests revealed that NBOH exhibited significantly enhanced inhibitory activity against *Staphylococcus aureus* (a Gram-positive bacterium) and *Escherichia coli* (a Gram-negative bacterium), with inhibition zones over twice as large as those of the nickel salt solution. Meanwhile, the crude orange peel extract showed no antibacterial activity. These findings underscore the contribution of this synthesis strategy to advancing sustainable bioinorganic materials and highlight the promising biomedical and environmental applications of nickel-based natural organic networks.

Keywords: orange peel, natural extract, antibacterial activity, biogenic organic, composite, nickel

1. INTRODUCTION

Metal–phenolic networks (MPNs) are supramolecular coordination assemblies formed between metal ions and phenolic ligands. These composites exhibit tunable structures and multifunctional properties, making them attractive platforms in materials science and nanoengineering. Recently, MPNs have gained increasing attention in biomedical applications, particularly in antimicrobial systems, due to the synergistic effects between polyphenols, which possess intrinsic antioxidant, anti-inflammatory, and antimicrobial properties, and metal ions, which further enhance the material performance [1-3]. Owing to their dynamic coordination chemistry, structural versatility, and biocompatibility, MPNs can be rationally designed to confer targeted functionalities, such as antibacterial activity, offering substantial promise for medical and environmental applications [4]. Phenolic networks potentially offer antibacterial activity through mechanisms such as disrupting microbial cell membranes, inhibiting key enzymes, and reducing biofilm formation. These characteristics suggest possible applications in areas like antimicrobial coating for medical devices, active layers in food packaging, and additives for water treatment systems.

Metal ions play a crucial role in the self-assembly and functionality of MPNs. Their ability to coordinate with phenolic hydroxyl groups enables the formation of stable and responsive networks. Commonly used metal ions in MPNs include Cu²⁺, Eu³⁺, Zn²⁺, Mg²⁺, and Ag⁺, each contributing unique

properties such as antibacterial activity and structural versatility. The selection of metal ions influences the therapeutic potential and adaptability of MPNs, allowing for controlled release and environmental responsiveness [5].

Nickel oxide-based nanoparticles have demonstrated significant antibacterial activity due to their ability to disrupt microbial membranes and generate reactive oxygen species (ROS), and their broad-spectrum effectiveness makes them suitable for applications such as coatings, wound dressings, and water purification systems [6], [7]. Although MPNs have been widely investigated using various metal ions, the incorporation of Ni²⁺ ions into MPN architectures remains scarce. In this context, integrating nickel species into organic networks to produce Ni-based MPNs can offer a wide range of promising biomedical and environmental applications, particularly for antioxidant and antibacterial effects.

Orange peel is known to be rich in proximate nutrients and bioactive phytochemicals. According to Adewole et al. [8], it contains abundant secondary metabolites, such as tannins, saponins, alkaloids, flavonoids, and phenolic compounds, thereby offering a valuable natural source for the synthesis of functional organic materials. Despite its phytochemical value, orange peel remains an abundant agro-industrial byproduct that requires sustainable and efficient valorization strategies.

The present study reports the formation of a nickel-containing biogenic organic composite (NBOH) material using orange peel extract as a natural source of organic precursors. The synthesized NBOH was characterized using SEM-EDS, XPS, and XRD to investigate its morphology, surface chemistry, and crystalline structure. Furthermore, its antibacterial activity was evaluated and compared with that of the crude orange peel extract and the nickel salt solution to assess the enhancement in antimicrobial performance. The findings of this study not only advance the development of sustainable biogenic materials but also broaden the potential applications of Ni-incorporated biogenic organic networks in related fields.

2. MATERIALS AND METHODS

2.1. Chemicals

Nickel (II) chloride hexahydrate (NiCl₂·6H₂O, 98%) and sodium hydroxide were purchased from XiLong Co. Ltd. (China). All chemicals were used without further purification. Deionized water was employed throughout the experiments.

2.2. Preparation of orange peel extract

Fresh orange peels were thoroughly washed to remove soil particles and other impurities from the surface. Darkened or withered areas on the peel surface were discarded. Next, the cleaned peels were dried in an oven at 60 °C for 8 h, ground into fine powder using an inox grinder, and then sieved through a 60-mesh screen. The dried orange peel powder (5 g) was mixed with 250 mL of deionized water and sonicated at 35 °C for 45 min. The mixture was left to stand overnight, then filtered through Whatman filter paper to obtain the orange peel extract (Figure 1).

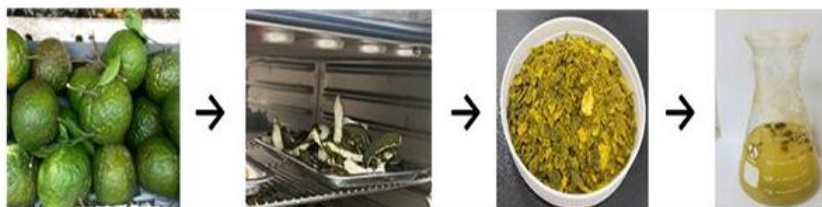


Figure 1. Extraction of orange peel

2.3. Synthesis of nickel-containing biogenic organic composite

Diluted orange peel extract was prepared by mixing the extract with deionized water at a 1:2 v/v ratio. The extract solution was adjusted to pH 8 using 0.1 M NaOH under magnetic stirring at room

temperature, while the 0.1 M nickel (II) chloride solution was stirred and heated to 80 °C. After that, the extract was added dropwise to the salt solution at a 1:2 v/v ratio. After the reaction was completed, the mixture was concentrated on a hot plate. The concentrated solution was then used for the antibacterial assay. The obtained precipitate was dried in an oven at 100 °C for 2 h, and then a nickel-organic composite powder was obtained (Figure 2).

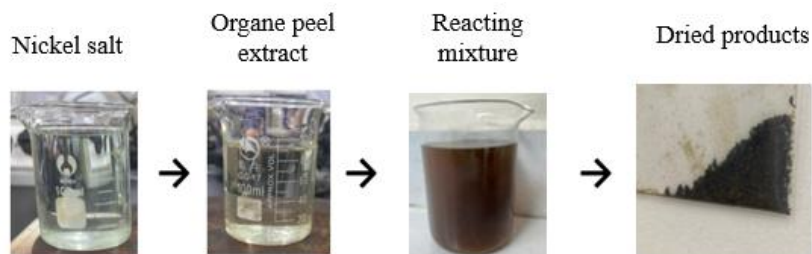


Figure 2. Synthesis of NBOH using the orange peel extract

2.4. Evaluation of antibacterial activity

The antibacterial activity of the orange peel extract, nickel salt, and NBOH solution was tested against *Staphylococcus aureus* ATCC 25923 (Gram-positive bacteria) and *Escherichia coli* ATCC 25922 (Gram-negative bacteria). The agar well diffusion assay was performed following the procedure described by Idris et al. [9], with slight modifications. Bacterial strains were cultured in Mueller–Hinton Broth (MHB), sterilized by autoclaving at 121 °C and 1 atm for 15 min, and incubated for 24 h in a shaking incubator at 200 rpm and 37 °C until reaching a density of approximately 1×10^8 CFU/mL (0.5 McFarland standard). Chloramphenicol (200 ppm) served as the positive control. For inoculation, 0.1 mL of each bacterial suspension was evenly spread on Mueller–Hinton agar (MHA) plates using a sterile cotton swab, rotating the plate by 60° between three passes to ensure uniform coverage. Three wells (6 mm diameter) were bored into the agar and filled with 40 μ L of negative control (solvent), positive control, and the test extract, respectively. Plates were then incubated at 37 °C for 24 h, and inhibition zones were measured after incubation. All experiments were conducted in triplicate, and results are expressed as mean \pm SD.

2.5. Characterization methods

Surface morphology and elemental composition were examined using SEM-EDS with a JMS–IT 200 instrument (JEOL, Japan). The measurements were conducted under an accelerating voltage of 10 kV, with a magnification of $\times 10,000$, image resolution of 512×384 pixels, and a dwell time of 0.20 ms. X-ray diffraction (XRD) analysis was carried out using a D8 Advance diffractometer (Bruker, Germany). The measurements employed Cu–K α radiation with a wavelength of $\lambda = 1.5406$ Å, and the scanning range was set from $2\theta = 5^\circ$ to 80° . X-ray photoelectron spectroscopy (XPS) analysis of the material was performed on an AXIS SUPRA spectrometer (Kratos Analytical, UK). The XPS measurements utilized Al K α radiation (photon energy 1486.6 eV) as the excitation source.

3. RESULTS AND DISCUSSION

3.1. SEM-EDX data

The SEM images (Figure 3A and 3B) demonstrate the angular block-like structures of the synthesized nickel – containing biogenic organic composite. The NBOH surface shows a loosely packed, flaky, and porous texture, indicating a heterogeneous microstructure with voids and interparticle boundaries. Moreover, during the reaction, the formed materials exhibit a fluffy, cotton-like morphology, forming loose aggregates that are visible to the naked eye. Such morphology facilitates active site exposure, which is advantageous for diverse biomedical and catalytic applications.

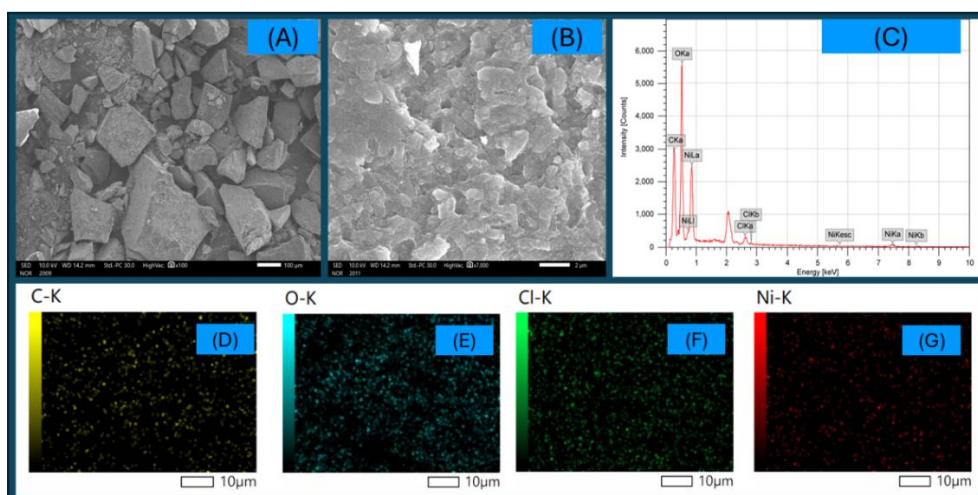


Figure 3. SEM-EDX results of NBOH: SEM images (A, B); EDX spectrum (C); and elemental distribution map for carbon (D), oxygen (E), chlorine (F), and nickel (G)

The EDX spectrum shows the characteristic peaks corresponding to carbon (C), oxygen (O), chlorine (Cl), and nickel (Ni) (Figure 3C), proving that C (31.04%), Ni (31.92%), and O (33.25%) are the major components in NBOH (Table 1). These elements are distributed evenly in the material as indicated by the mapping data (Figures 3D-G). The residual Cl from nickel salt was detected at 3.79%. The presence of Ni and O peaks confirms the successful incorporation of nickel within the composite.

Table 1. Composition of NBOH analyzed by EDX

Element	Mass (%)	Atom (%)
C	31.04	48.64
O	33.25	39.11
Cl	3.79	2.01
Ni	31.92	10.23

3.2. XRD pattern

The XRD pattern presented in Figure 4 exhibits a broad diffraction region centered around $2\theta \approx 20-45^\circ$ without any clearly discernible peaks, indicating the dominance of amorphous carbonaceous components integrated with nickel species. Such an amorphous nature is commonly observed in biogenic or plant-mediated synthesis routes, where organic molecules from the extract act as reducing and stabilizing agents, resulting in incomplete crystallization under mild conditions. The amorphous state of NBOH may benefit in terms of unique physicochemical properties, including high dispersion and potential for strong interaction with bacterial cell membranes.

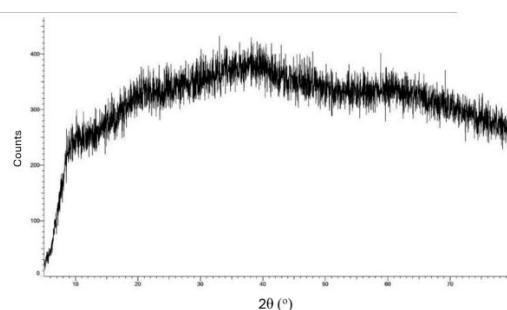


Figure 4. XRD pattern of NBOH

3.3. XPS spectrum

The C1s XPS spectrum of NBOH is presented in Figure 5. Three distinct peak components were identified upon deconvoluting the spectrum using Gaussian functions, indicating the presence of polyphenolic networks derived from the orange peel extract. The dominant peak at approximately 284.8 eV corresponds to C–C/C–H bonds, which represent the aromatic and aliphatic carbon backbone typical of polyphenolic structures. The second peak, centered at 286.5 eV, is attributed to C–O species indicative of hydroxyl and ether groups commonly found in phenolic compounds. The third peak at around 288.5 eV is assigned to C=O functionalities, suggesting carbonyl groups present in flavonoids and other oxidized polyphenols [10, 11]. These chemical signatures confirm that the organic fraction in the nickel–organic composite is derived from plant-based metabolites, which provide abundant functional groups for coordination with nickel ions and contribute to the formation of a composite network.

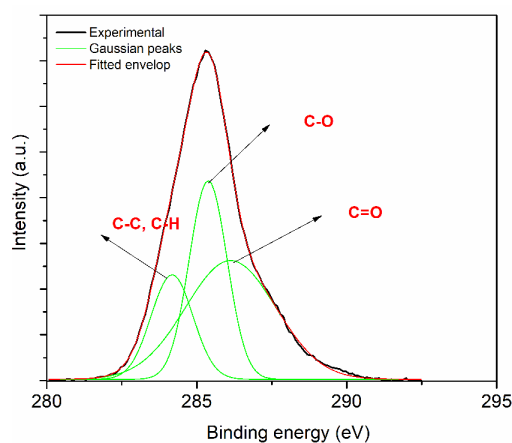


Figure 5. C1s XPS spectrum of NBOH

3.4. Antibacterial activity of metal-organic composite

Antibacterial tests were performed against *Staphylococcus aureus* (Gram-positive bacterium) and *Escherichia coli* (Gram-negative bacterium). The antibacterial activity results (Table 2 and Figure 6) demonstrate the antibacterial potential of the synthesized material. The orange peel extract showed no inhibitory effect, while the nickel salt solution exhibited inhibition zones with diameters of 7–9 mm, with higher activity found against *E. coli*. Notably, NBOH demonstrated significant enhancement in the antibacterial activity, with over 2-fold increase in inhibition diameters compared to the initial orange peel extract, highlighting the synergistic effect of nickel incorporation and carbonaceous components derived from orange peel extract as a typical feature of MPNs. Moreover, NBOH also showed a higher inhibitory effect than NiO nanoparticles synthesized using the onion extract [6] and the ethyl acetate extract of *C. asiatica* reported by Sellathoroe et al. [12].

The antibacterial activity of Ni-based organic networks can be explained through several well-established mechanisms. The release of Ni²⁺ ions can damage the bacterial cell membrane by interacting with phospholipids, increasing permeability and causing leakage of intracellular substances. This membrane-disrupting effect has been clearly demonstrated in studies on the interaction between NiO or Ni-containing materials and bacterial cells, showing changes in membrane permeability, enzyme leakage, and structural instability [13]. In addition, Ni²⁺ ions are capable of catalyzing the formation of reactive oxygen species (ROS), which induce oxidative damage to DNA, proteins, and essential enzymes, ultimately causing cell death [14]. Moreover, Ni²⁺ ions are known to interact with thiol- and carboxyl-containing residues in bacterial enzymes, disrupting biochemical activity and inhibiting metabolic processes, thereby slowing or preventing bacterial growth [15]. Meanwhile, the polyphenolic framework contributes to antibacterial activity by disrupting membrane structure and interacting with bacterial proteins. Polyphenols can also coordinate with metal ions, creating a synergistic effect that enhances antimicrobial efficacy [16].

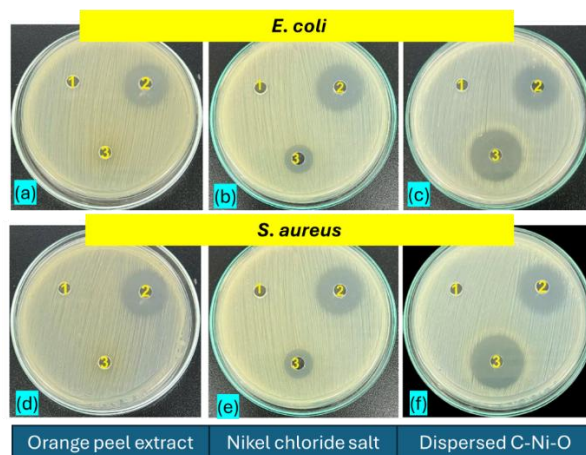


Figure 6. Antibacterial activity of orange peel extract, nickel chloride (NiCl_2), and dispersed C–Ni–O against *E. coli* (a, b, c) and *S. aureus* (d, e, f). In each plate, position (1) represents the negative control, position (2) represents the positive control, and position (3) shows the test sample.

Table 2. Inhibition zone diameters against *S. aureus* and *E. coli*

Material/Extract	Diameter (mm)		Reference
	<i>S. aureus</i>	<i>E. coli</i>	
Orange peel extract	—	—	This study
Nickel chloride salt solution 0.1 M	7.64 ± 0.19^a	8.53 ± 0.31^a	This study
NBOH solution	17.56 ± 0.35^b	17.35 ± 0.69^b	This study
NiO nanoparticles (synthesized using onion extract)	10–14 mm	12–15 mm	[3]
Ethyl acetate extract of <i>C. asiatica</i>	10.07 ± 0.11	8.53 ± 0.15	[9]

(—) No growth inhibition zone; values are presented as means \pm SD; different letters in the same column indicate statistical significance at $p < 0.05$ (t-Test)

Overall, the combination of structural analysis and biological evaluation confirms that the eco-friendly synthesis route yields an amorphous Ni–organic composite from waste orange peel with promising antibacterial property, making it a potential candidate for biomedical and environmental applications.

4. CONCLUSION

In this study, a nickel-containing biogenic organic composite synthesized from orange peel extract was successfully obtained and demonstrated clear antibacterial effects against both *Staphylococcus aureus* and *Escherichia coli*. The composite outperformed its individual precursors, while the original plant extract showed no detectable activity. These findings indicate that the combination of nickel species with biogenic organic components can create a functional material with enhanced antimicrobial performance. Although further investigations are needed to fully assess its biological safety and broad-spectrum efficacy, the promising antibacterial response observed here highlights the potential applicability of this composite in future biomedical, environmental, and antimicrobial technologies.

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