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벤질 클로라이드 4급화 키토산 기반 니켈 복합체 형성 및 FTIR/XRD 구조 분석

Nigar Shikhverdiyeva[†], Ulviyya Mammadova, Hajar Aslanova, Nargiz Rahimli,
Chichak Seyidova, and Nizami Zeynalov
Laboratory of Nanostructured metal-polymer catalysts, Ministry of Science and Education,
Republic of Azerbaijan, Institute of Chemistry

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Benzyl Chloride–Quaternized Chitosan as a Tailored Support for Nickel Complex Formation: Structural Insights from FTIR and XRD

**Nigar Shikhverdiyeva[†], Ulviyya Mammadova, Hajar Aslanova, Nargiz Rahimli,
Chichak Seyidova, and Nizami Zeynalov**
*Laboratory of Nanostructured metal-polymer catalysts, Ministry of Science and Education, Republic of Azerbaijan,
Institute of Chemistry, 113 H. Javid Street, Baku AZ1143, Azerbaijan Republic*

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Abstract: The rational design of chemically modified biopolymer supports is critical for tailoring metal coordination environments in functional materials. Chitosan was selectively quaternized with benzyl chloride to form a polymeric matrix that facilitates nickel complex formation. The modified biopolymer was coordinated with Ni^{2+} ions from $NiCl_2 \cdot 6H_2O$, yielding a benzyl-quaternized chitosan–nickel composite. Structural changes were investigated using FTIR and XRD. FTIR confirmed successful incorporation of benzyl quaternary ammonium and revealed spectral shifts indicative of coordination interactions between nickel ions and chitosan. XRD patterns showed new diffraction features in the nickel-loaded composite compared to pristine chitosan, indicating the formation of nickel-containing domains and polymer microstructure rearrangement upon quaternization and metal coordination. These findings demonstrate that benzyl chloride quaternization effectively modulates chitosan's structure, creating a favorable coordination environment for nickel complexes. This strategy offers a versatile platform for designing functional metal–biopolymer hybrid materials with potential applications in advanced materials.

Keywords: quaternized chitosan, benzyl chloride, nickel complexes, biopolymer support.

Introduction

Chitosan, a linear polysaccharide obtained through the alkaline deacetylation of chitin, has emerged as one of the most versatile biopolymers for applications in materials science, coordination chemistry, and functional composite design.[1–3] Its molecular structure, characterized by abundant primary amino groups at the C-2 position and hydroxyl groups at the C-3 and C-6 positions of glucosamine units, provides a rich platform for chemical modification and metal ion coordination. These electron-donating functionalities enable chitosan to act as an effective chelating agent, facilitating the formation of stable metal–polymer complexes. However, the practical utilization of pristine chitosan is often restricted by its limited solubility in neutral and alkaline aqueous environments, which originates from its semicrystalline nature and extensive inter- and intramolecular hydrogen bonding network.[4–7] Moreover, the protonation-dependent availability of amino groups leads to diminished coordination efficiency under neutral conditions, thereby constraining its broader application in functional material systems.

To overcome these intrinsic limitations, a wide range of chemical modification strategies has been developed, among which quaternization has attracted particular attention owing to its ability to permanently alter the charge distribution and physicochemical properties of chitosan.[8–10] Quaternization involves the covalent introduction of alkyl or aryl substituents onto the

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[†] To whom correspondence should be addressed.

nigar.shikverdiyeva@kqki.science.az, ORCID 0009-0007-6863-9715

amino groups, resulting in the formation of quaternary ammonium functionalities that retain positive charges across a wide pH range. This structural transformation significantly enhances aqueous solubility, disrupts the original hydrogen bonding network, and increases the accessibility of coordination sites, thereby creating a favorable coordination environment through the combined contributions of quaternary nitrogen atoms, residual primary amines, and hydroxyl groups.

Among various quaternizing agents, benzyl chloride offers distinct advantages due to its moderate reactivity and the incorporation of bulky aromatic moieties into the polymer backbone.[11] The nucleophilic substitution reaction between benzyl chloride and the primary amino groups of chitosan introduces benzyl quaternary ammonium groups, which not only improve solubility but also induce pronounced structural rearrangements within the polymer matrix. The presence of aromatic benzyl units is expected to disrupt crystalline packing, weaken intermolecular hydrogen bonding, and generate a more open polymer architecture, thereby facilitating enhanced interaction with metal ions. Despite the recognized potential of benzyl-based quaternization for tuning chitosan structure, its implications for metal complex formation and coordination environment engineering remain insufficiently explored.

In parallel, immobilization of transition metal ions onto polymeric supports has emerged as an effective strategy for constructing hybrid organic–inorganic materials with tailored structural and functional characteristics. Nickel-based complexes, in particular, have received considerable attention due to their variable coordination chemistry, favorable electronic properties, and economic advantages over noble metals.[12–14] When anchored within polymeric matrices, nickel ions benefit from improved dispersion, enhanced stability, and reduced leaching, leading to hybrid systems that combine the mechanical robustness of the polymer with the functional versatility of metal centers.[15,16] Chitosan and its derivatives have proven to be promising hosts for such systems; however, most reported studies have focused on native or conventionally modified chitosan supports, while the influence of benzyl-based quaternization on nickel coordination behavior has remained largely unexplored.

Notably, although quaternized chitosan derivatives and chitosan–nickel complexes have been independently investigated, systematic studies addressing the formation of nickel complexes on benzyl chloride–quaternized chitosan matrices are extremely limited. In particular, the structural consequences of benzyl quaternization on polymer crystallinity, hydrogen bonding organization, and metal coordination pathways have not yet been thoroughly elucidated. This knowledge gap restricts a deeper understanding of how aromatic quaternary substituents can be exploited to engineer polymer coordination environments for advanced metal–biopolymer hybrid materials.

In this context, the present work aims to synthesize benzyl chloride–quaternized chitosan and to systematically investigate its structural evolution and nickel complex formation behavior using Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD). Emphasis is placed on elucidating the molecular-level interactions responsible for quaternization-induced structural rearrangement and subsequent metal coordination. The obtained insights provide a structural basis for future studies targeting catalytic and advanced material applications.[17,18]

Experimental

Materials. Chitosan (medium molecular weight, high degree of deacetylation), benzyl chloride (reagent grade), N,N′-methylenebisacrylamide (MBAA, ≥99%), and nickel(II) chloride hexahydrate ($NiCl_2 \cdot 6H_2O$, ACS reagent grade) were purchased from commercial suppliers and used without further purification. Glacial acetic acid, sodium hydroxide, ethanol, and deionized water were employed as solvents, pH-regulating agents, and washing media throughout the experimental procedures. All reagents were of analytical grade and were purchased from Sigma-Aldrich.

Synthesis of Benzyl Chloride–Quaternized Chitosan. Chitosan powder (1.0 g) was dissolved in 0.1 N aqueous acetic acid solution (50 mL) under continuous magnetic stirring at room temperature (25 °C) for 2 h until a homogeneous and transparent solution was obtained. Benzyl chloride (0.27 mL) was then added dropwise to the chitosan solution under vigorous stirring at 60 °C.[11,19] The reaction mixture was maintained at 60 °C for 6 h. The pH was continuously monitored with a calibrated pH meter and adjusted to pH 8-9 by dropwise addition of 1 M NaOH solution to promote quaternary ammonium formation and induce precipitation of the modified polymer. Upon completion, the precipitate was filtered, washed three times with deionized water (3×50 mL) and twice with ethanol (2×30 mL). The purified product was subsequently dried under ambient conditions to obtain the final quaternized chitosan as a pale-colored powder. The yield of the quaternized product was approximately 25%. A schematic representation of the quaternization pathway is illustrated in Figure 1.

Formation of Nickel(II) Complexes. Aqueous nickel(II) chloride hexahydrate $NiCl_2 \cdot 6H_2O$ solution (0.1 M, 50 mL) was

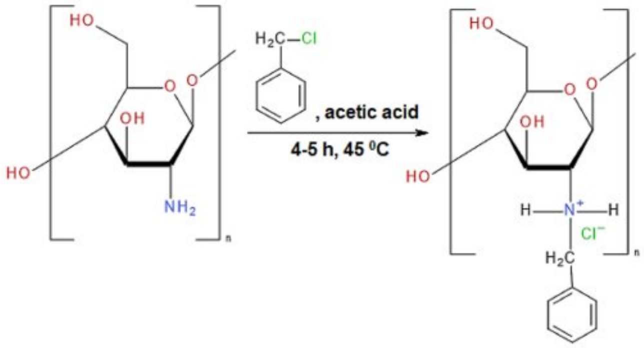

Figure 1. Schematic illustration of the quaternization of chitosan with benzyl chloride.

gradually introduced into a dispersion of benzyl chloride–quaternized chitosan (0.5 g) in deionized water (50 mL) under continuous magnetic stirring at room temperature (25 °C).[20] The pH of the mixture was maintained between 4.5 and 5 using dilute NaOH solution (0.1 M) to ensure effective Ni^{2+} coordination while avoiding bulk $Ni(OH)_2$ precipitation. The mixture was stirred for 12 h. The resulting nickel-loaded polymer was separated by filtration, washed with deionized water (3 × 50 mL), and dried at 50 °C for 24 h (Figure 2).[21-23]

Crosslinking of Nickel-Loaded Quaternized Chitosan. The nickel-loaded quaternized chitosan was dispersed in deionized water to form a homogeneous suspension, after which MBAA was added at a mass ratio of 10% relative to the polymer (0.1 g MBAA per 1.0 g polymer) under continuous stirring.[26] The crosslinking reaction was conducted for 4 h, allowing covalent bridges to form between polymer chains and creating a three-dimensional network that stabilizes incorporated nickel species. After completion, the crosslinked composite was washed with deionized water (3 × 30 mL) and ethanol (2 × 20 mL) to remove unreacted crosslinker, and dried at 50 °C for 24 h. This step improves mechanical robustness and structural stability while minimizing metal leaching, without altering the fundamental coordination chemistry of the Ni^{2+} ions (Figure 3).[27]

Characterization Techniques. The structural and chemical properties of pristine chitosan, benzyl chloride–quaternized chitosan, and nickel-loaded composites were systematically investigated using XRD and FTIR spectroscopy.

Crystallinity and phase composition were analyzed using a Rigaku Miniflex 600 diffractometer equipped with Cu K_α radiation ($\lambda = 1.54$ Å). Diffractograms were recorded to monitor structural changes in the polymer after quaternization and to detect any Ni-related crystalline features.

Spectroscopic analysis was performed with a Nicolet iS10

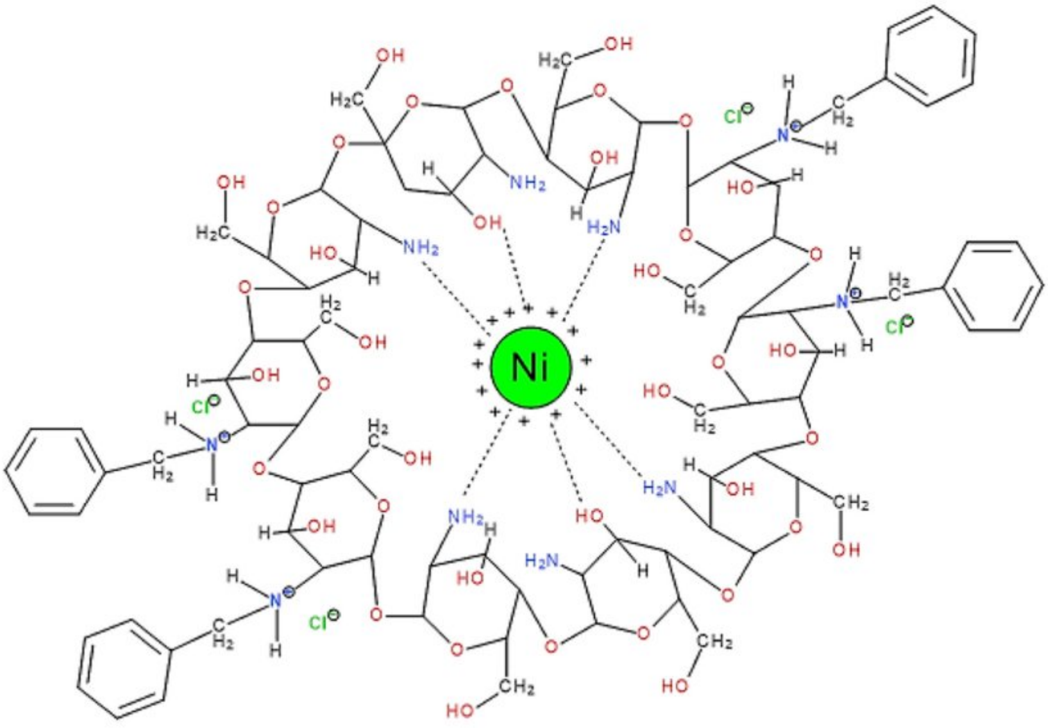

Figure 2. Proposed coordination model illustrating the interaction of Ni^{2+} ions with benzyl chloride–quaternized chitosan through amino, hydroxyl, and quaternary ammonium functionalities, forming a structurally stabilized polymer–metal complex (adapted and modified from Refs. 24 and 25).

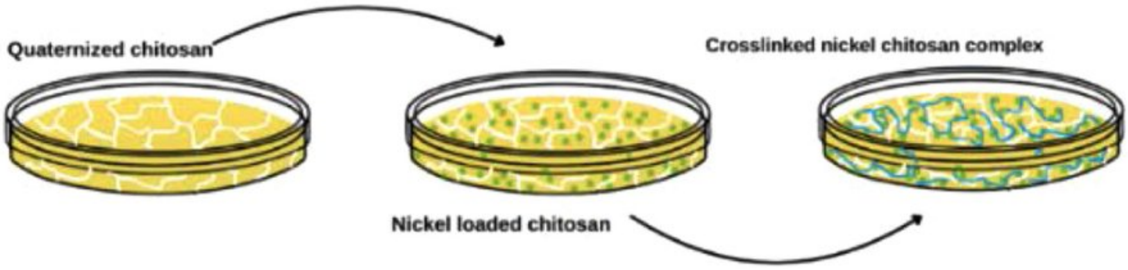

Figure 3. Schematic illustration of the formation of quaternized chitosan-nickel complex.

FTIR spectrometer in the 4000–400 cm^{-1} range. FTIR was employed to confirm successful benzyl quaternization and to identify characteristic spectral shifts corresponding to coordination interactions between Ni^{2+} ions and the functional groups of chitosan. Particular attention was paid to aromatic signals introduced by benzyl modification, as well as to shifts in NH_2, OH, and other functional group bands indicative of metal binding.

Results and Discussion

The structural evolution of chitosan during benzyl chloride quaternization and subsequent nickel(II) incorporation was systematically investigated using XRD (Figure 4) and FTIR (Figure 5) spectroscopy. Pristine chitosan exhibited two characteristic reflections at $2\theta \approx 10°$ and 20°, corresponding to the (020) and (110) planes, respectively,[28-32] indicative of its semicrystalline structure wherein ordered crystalline domains coexist with amorphous regions stabilized by extensive hydrogen bonding among hydroxyl and amino functionalities. Following quaternization, these reflections became broader and less intense, indicating partial disruption of the original hydrogen-bonding network and reduced crystallinity due to the introduction of bulky benzyl quaternary ammonium groups. This structural reorganization enhances the accessibility of functional groups, preparing the polymer matrix for effective metal coordination.[33-37] Upon immobilization of Ni^{2+} ions, additional broad diffraction features appeared around $2\theta \approx 37°$ and 44°, consistent with highly dispersed nickel species, including weakly crystalline $Ni(OH)_2$, residual $NiCl_2$, or molecularly coordinated nickel complexes. The broad and low-intensity nature of these peaks indicates that Ni^{2+} ions are predominantly present as highly dispersed molecular complexes or small clusters rather than as bulk crystalline aggregates, which is advantageous for applications requiring high surface area and uniform metal distribution (Table 1).

FTIR spectroscopy further confirmed the successful chemical modification and metal incorporation. The pristine chitosan spectrum displayed characteristic bands at ~3446 cm^{-1} (O–H/N–H stretching), 2920 cm^{-1} (C–H stretching), 1646 cm^{-1} (amide I, C=O stretching), and 1579 cm^{-1} (N–H bending), corresponding to native functional groups available for coordination.[38] Following quaternization, new bands emerged at 3000–3080 cm^{-1} (aromatic C–H) and 1642 cm^{-1} (C–N$^+$ stretching), confirming

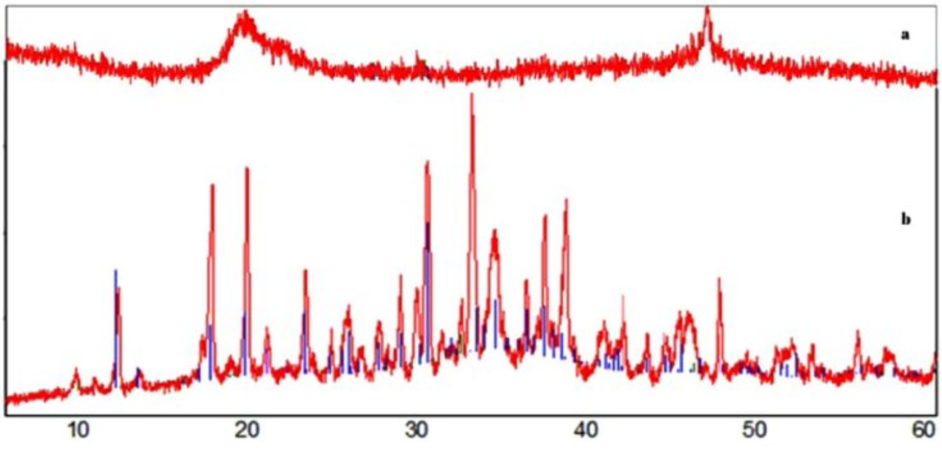

Figure 4. X-ray diffraction patterns of (a) pure chitosan; (b) nickel-loaded benzyl chloride-quaternized chitosan composite.

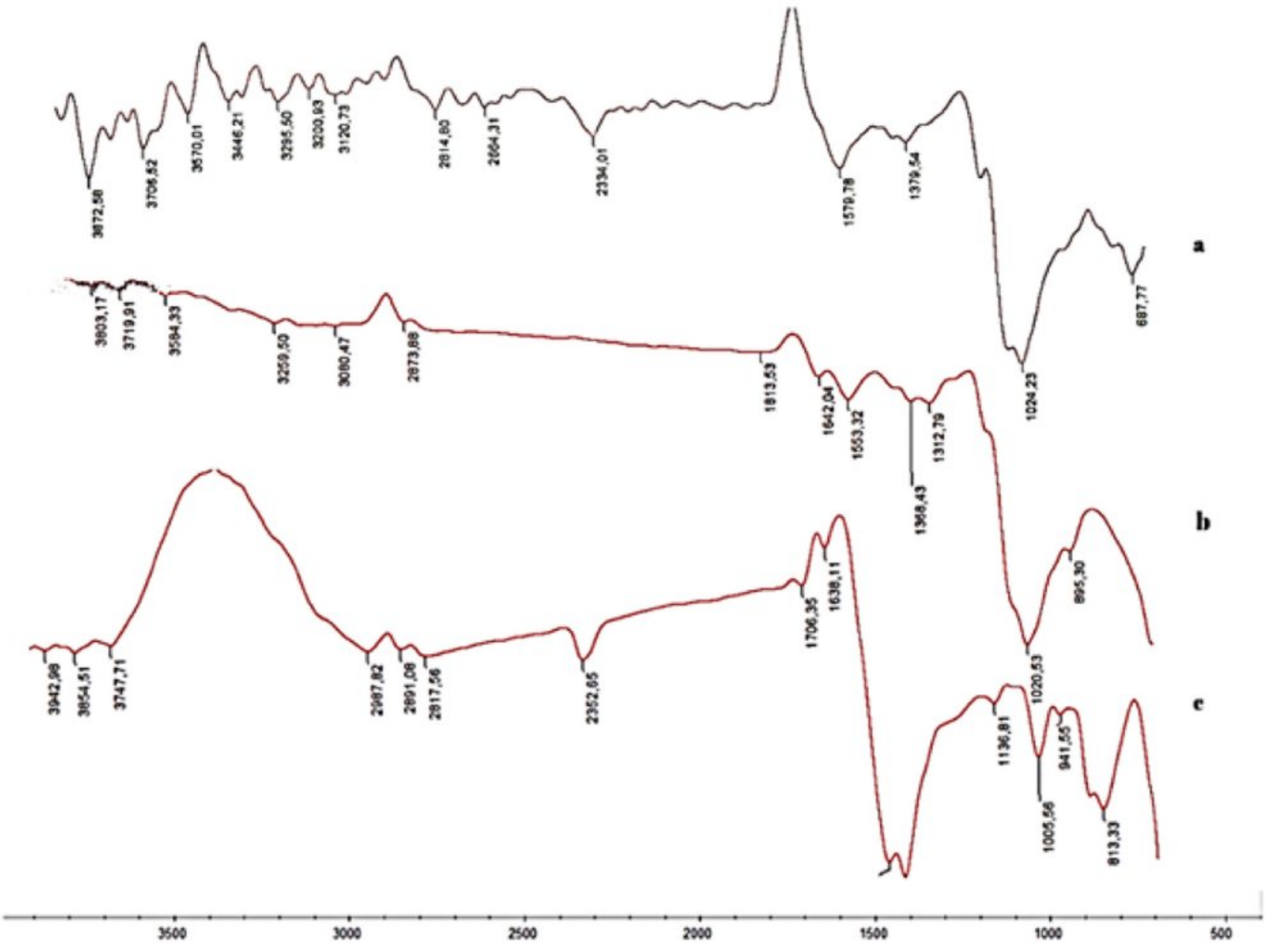

Figure 5. FTIR spectra of (a) pure chitosan; (b) quaternized chitosan; (c) Ni-loaded chitosan nanocomposite.

Table 1. XRD Peak Positions and Structural Assignments for Pristine Chitosan, Benzyl Chloride–quaternized Chitosan, and Nickel-loaded Composites

Sample	2θ (°)	Assignment	Observed Change
Pristine Chitosan	10	(020) plane	-
Pristine Chitosan	20	(110) plane	-
Ni-loaded Composite	37	$Ni(OH)_2$ / Ni complex	Broad, low-intensity peak indicates nanoscale dispersion
Ni-loaded Composite	44	Ni species	Broad, low-intensity peak indicates nanoscale dispersion

the formation of benzyl quaternary ammonium groups, while alterations in the broad O–H/N–H region indicate disruption of hydrogen bonding.[11-19] Subsequent nickel incorporation led to further spectral shifts, including modifications of the O–H stretching region above 3000 cm^{-1}, CH_2 vibrations at 2891 cm^{-1}, C–C at 1399 cm^{-1}, and C–O at 1024 cm^{-1}, consistent with coordination of Ni^{2+} ions through residual amino groups and hydroxyl groups. Although quaternary ammonium centers do not possess a donor lone pair and therefore do not directly participate in metal coordination, they contribute indirectly by enhancing polymer chain flexibility and improving the accessibility of genuine donor sites (Table 2).[39,40] These combined XRD and FTIR results demonstrate that benzyl chloride–quaternized chitosan undergoes substantial structural reorganization, generating a favorable coordination environment for nickel complex formation, with nanoscale dispersion of metal species that is likely to enhance future catalytic and adsorption performance without the need to claim any application in this study.

Table 2. FTIR Spectral Bands and Assignments Indicating Chemical Modifications During Quaternization and Coordination of Ni^{2+} Ions with Chitosan

Sample	Wavenumber (cm^{-1})	Functional assignment	Structural/Functional change
Pristine chitosan	3446	O–H/N–H stretching	-
Pristine chitosan	2920	C–H stretching	-
Pristine chitosan	1646	Amide I (C=O)	-
Pristine chitosan	1579	N–H bending	-
Quaternized chitosan	3000-3080	Aromatic C–H	Benzyl quaternization
Quaternized chitosan	1642	$C–N^+$ stretching	Formation of quaternary ammonium
Ni-loaded composite	2891	CH_2 stretching	Ni^{2+} coordination
Ni-loaded composite	1399	C–C stretching	Ni^{2+} coordination
Ni-loaded composite	1024	C–O stretching	Ni^{2+} coordination

Conclusion

This study demonstrates the successful synthesis of benzyl chloride–quaternized chitosan and its effective utilization as a functional support for nickel(II) complex formation. Quaternization of the primary amine groups introduced benzyl quaternary ammonium functionalities, imparting permanent positive charges, enhancing aqueous solubility, and generating a more flexible, accessible polymer structure conducive to metal coordination. Structural characterization confirmed these modifications: XRD analysis revealed a marked reduction in the crystallinity of native chitosan and the emergence of new diffraction features upon nickel incorporation, indicating successful immobilization of dispersed nickel species. FTIR spectroscopy corroborated the formation of benzyl quaternary ammonium groups and provided clear evidence of coordination interactions involving hydroxyl, amino, and quaternary nitrogen sites with Ni^{2+} ions. Together, these results establish benzyl chloride–quaternized chitosan as a structurally favorable biopolymeric platform for stabilizing nanoscale nickel species. The enhanced accessibility of functional groups and metal-binding capacity highlight the promise of this material as a versatile support for future catalytic or adsorption applications. Further studies may focus on exploring the influence of quaternization degree and metal loading on stability and performance, as well as extending this modification strategy to other transition metals to broaden the applicability of quaternized chitosan as a sustainable and multifunctional support material.

Conflict of Interest: The authors declare that there is no conflict of interest.

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