MORPHOLOGICAL AND ELEMENTAL CHARACTERIZATION OF POLY[ETHYLENEDIAMINETETRAACETATO-AQUA-DISODIUM-NICKEL(II)] BY SEM–EDS ANALYSIS

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Abstract
Poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] was synthesized and characterized by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS) to investigate its morphology and elemental composition. SEM analysis revealed the formation of highly crystalline elongated prismatic and rod-like crystals with smooth surfaces and well-defined edges. The observed crystal habit indicates anisotropic crystal growth and a high degree of structural organization within the coordination polymer. EDS analysis confirmed the presence of carbon, nitrogen, oxygen, sodium, and nickel as the principal constituents of the synthesized compound. Quantitative elemental analysis showed contents of 49.86 wt.% C, 4.79 wt.% N, 33.01 wt.% O, 3.32 wt.% Na, and 9.02 wt.% Ni. The appearance of characteristic nickel peaks together with the donor atoms of the EDTA ligand confirms successful complex formation. The combined SEM-EDS results demonstrate the formation of a highly crystalline nickel(II)-EDTA coordination polymer with good compositional purity and structural homogeneity.

Keywords: Nickel(II), EDTA, Coordination Polymer, SEM, EDS, Crystal Morphology, Elemental Analysis.

Introduction

Coordination polymers based on transition-metal ions and multidentate organic ligands have attracted considerable attention due to their structural diversity and potential applications in catalysis, ion exchange, environmental remediation, molecular recognition, and advanced functional materials. Among the numerous chelating ligands available, ethylenediaminetetraacetic acid (EDTA) occupies a prominent position because of its strong metal-binding ability arising from four carboxylate oxygen atoms and two amine nitrogen donor atoms. Nickel(II)-EDTA complexes are particularly important because of their remarkable thermodynamic stability and coordination versatility. The incorporation of nickel ions into EDTA-based frameworks leads to the

formation of coordination compounds exhibiting distinct structural, thermal, and physicochemical properties. The performance of such materials is strongly influenced by their crystal morphology, particle size distribution, and elemental composition. Scanning electron microscopy (SEM) is widely used to investigate crystal morphology and surface characteristics, whereas energy-dispersive X-ray spectroscopy (EDS) provides valuable information regarding elemental composition and purity. These techniques are complementary and play a crucial role in confirming the successful synthesis of coordination compounds. Therefore, the present study focuses on the morphological and elemental characterization of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] using SEM and EDS analyses.

Experimental Characterization

The morphology of the synthesized poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] was examined using a scanning electron microscope operating at an accelerating voltage of 5.0 kV. Elemental composition was determined using an integrated energy-dispersive X-ray spectroscopy detector. SEM images were recorded at a magnification of ×300, while elemental quantification was performed using standardless EDS fitting procedures.

Results and Discussion

SEM Morphological Analysis

The SEM micrographs of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] reveal a highly crystalline material composed predominantly of elongated rod-like and prismatic particles (Figure 1). The crystals possess smooth surfaces, sharp edges, and well-developed crystal faces, indicating a high degree of crystallinity and structural order.

The dominant morphology consists of rectangular rods with lengths extending up to several tens of micrometers. Such elongated structures suggest preferential crystal growth along specific crystallographic directions. This phenomenon is commonly observed in coordination polymers where directional metal–ligand interactions govern the crystal growth process. The crystal surfaces appear dense and compact without noticeable porosity, indicating efficient packing of molecular units within the crystal lattice. The absence of extensive amorphous regions suggests that the synthesis conditions favored controlled nucleation and subsequent crystal growth. Furthermore, the crystals exhibit clear geometric boundaries and faceted surfaces, characteristic of well-ordered coordination compounds. Some particles are observed to overlap and intergrow, forming larger crystalline aggregates. These observations indicate strong intermolecular interactions and the formation of an extended coordination framework.

The highly developed crystalline morphology observed in the SEM images demonstrates that the nickel(II)-EDTA coordination network forms a stable and structurally organized material.

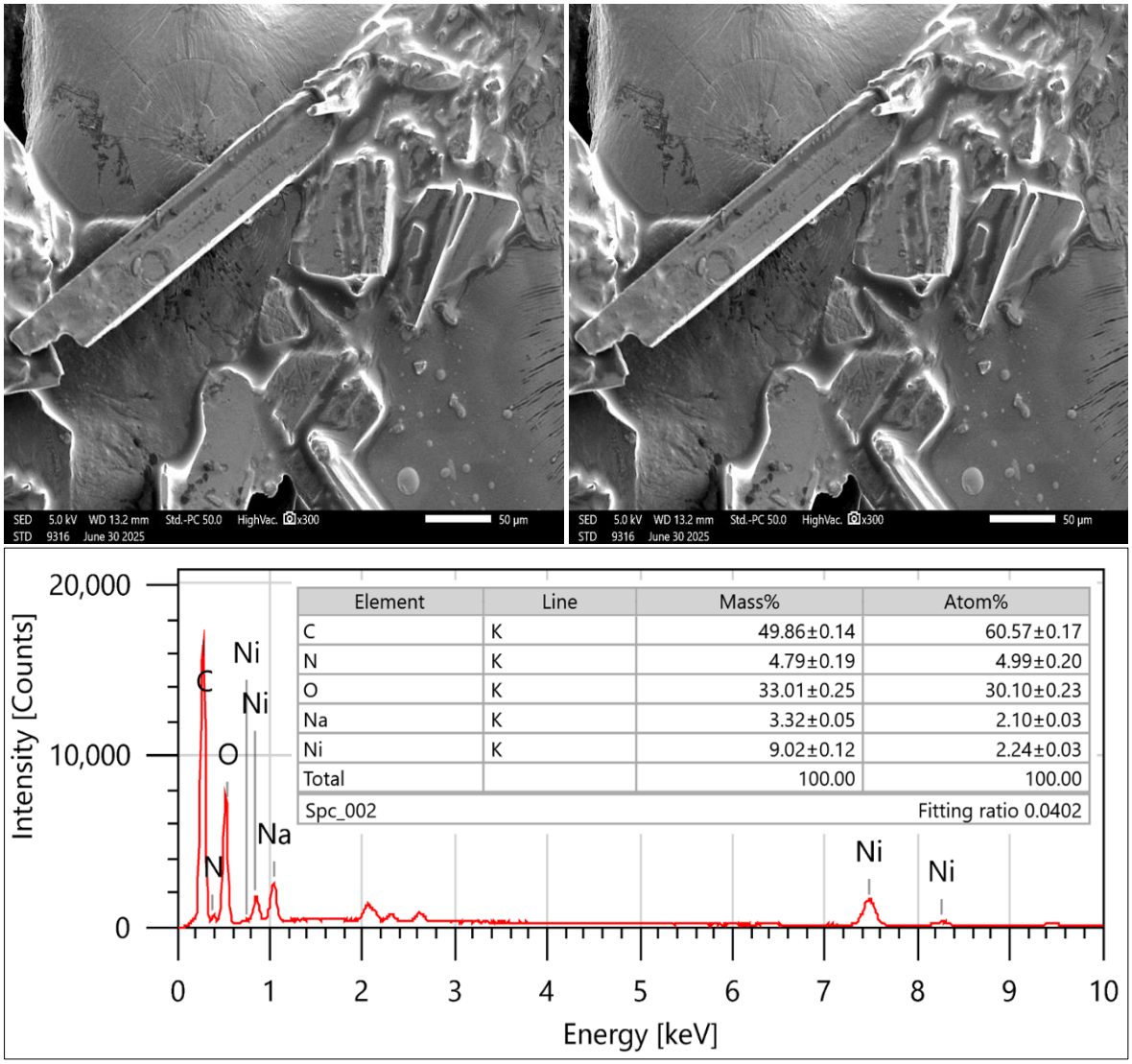

Figure 2. SEM images and EDS elemental analysis of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] showing rod-like crystalline morphology and the presence of C, N, O, Na, and Ni elements.

EDS Elemental Analysis

The elemental composition of the synthesized complex was investigated using EDS spectroscopy. The spectrum displays characteristic peaks corresponding to carbon (C), nitrogen (N), oxygen (O), sodium (Na), and nickel (Ni), confirming the presence of all expected elements in the proposed molecular structure.

Table 1. Elemental Composition of Poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)]

Element	Mass (%)	Atomic (%)
Carbon (C)	49.86 ± 0.14	60.57 ± 0.17
Nitrogen (N)	4.79 ± 0.19	4.99 ± 0.20
Oxygen (O)	33.01 ± 0.25	30.10 ± 0.23
Sodium (Na)	3.32 ± 0.05	2.10 ± 0.03
Nickel (Ni)	9.02 ± 0.12	2.24 ± 0.03
Total	**100.00**	**100.00**

The high carbon content originates from the ethylene backbone and acetate groups of the EDTA ligand. Nitrogen is associated with the amine donor atoms, while oxygen arises from carboxylate groups and coordinated water molecules. Sodium detected in the EDS spectrum confirms the presence of disodium counterions associated with the coordination polymer structure. Most importantly, the strong nickel peaks observed near 7.5 and 8.3 keV correspond to Ni Kα and Ni Kβ emissions, providing direct evidence of nickel incorporation into the coordination framework. The nickel content of approximately 9 wt.% confirms successful metal coordination and is consistent with the proposed nickel-containing EDTA complex. Furthermore, no significant impurity peaks were detected, indicating the high purity of the synthesized material. The agreement between the observed elemental composition and the expected molecular structure strongly supports the successful synthesis of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)].

Structural Implications

The combined SEM and EDS results provide important information regarding the structural organization of the synthesized compound. The rod-like morphology observed by SEM suggests directional crystal growth controlled by strong coordination interactions between nickel (II) ions and EDTA ligands. The presence of carbon, nitrogen, oxygen, sodium, and nickel in the EDS spectrum confirms the integrity of the coordination polymer framework. The absence of foreign elements indicates that the synthesized material is chemically homogeneous and free from significant contamination. The coexistence of high crystallinity and compositional purity suggests that the coordination polymer possesses a well-organized crystal lattice in which nickel centers are effectively coordinated by EDTA ligands, resulting in a stable polymeric structure.

Conclusion

Poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] was successfully characterized by SEM and EDS techniques. SEM analysis revealed highly crystalline

elongated rod-like and prismatic particles with smooth surfaces, sharp edges, and well-developed crystal faces, indicating excellent structural organization. EDS analysis confirmed the presence of carbon, nitrogen, oxygen, sodium, and nickel as the principal constituent elements of the coordination polymer. The quantitative elemental composition agrees well with the proposed molecular structure and demonstrates the successful incorporation of nickel into the EDTA framework. The absence of significant impurity peaks and the presence of highly ordered crystalline particles indicate the high purity and structural stability of the synthesized material. These findings collectively confirm the successful formation of poly[ethylenediaminetetraacetato-aqua-disodium-nickel(II)] and provide a comprehensive understanding of its morphological and elemental characteristics.

References

1. Foreman, M. M., Alessio, M., Krylov, A. I., Johnson, M. A. (2023). *Influence of transition metal electron configuration on the structure of metal-EDTA complexes*. Journal of Physical Chemistry A, 127, 1135–1145. https://doi.org/10.1021/acs.jpca.2c07996

2. Ling, Z., Yuan, Q., Kong, X., Cheng, L., Zhang, H. (2026). *Photoelectron spectroscopy and structural characterization of EDTA complexes (M = Ni, Cu, Zn)*. Journal of Chemical Physics, 164, 054310.

3. Chen, M., Ding, L., Zhu, S., Xiong, X., Yuan, X., Peng, Y. (2023). *Decomplexation of Ni–EDTA enhanced by Fe(III) reduction in the Fenton reaction*. Journal of Environmental Chemical Engineering, 11, 110611.

4. Ma, Y., Wang, H., Bai, M., Li, M., Xu, J., Xie, H., Li, X. (2025). *Mechanistic insights into the removal of metal-EDTA complexes from water*. Journal of Cleaner Production, 503, 145003.

5. González-Varela, D., Araiza, D. G., Díaz, G., Pfeiffer, H. (2022). *$LaNiO_3$ perovskite synthesis through the EDTA–citrate complexing method and its application to CO oxidation*. Catalysts, 12(1), 57.

6. Rabanal-Sanchez, J., Vega-Chacon, J. (2025). *Synthesis and characterization of EDTA coordination compounds and DFT study of EDTA species*. Computational and Theoretical Chemistry, 1253, 114955.

7. Pechinka, D., Martsynko, O. (2025). *Synthesis and comprehensive study of heteronuclear Ge(IV)-Ni(II) complexes with ethylenediaminetetraacetic acid*. Ukrainian Chemistry Journal, 91(2), 45–58.

8. Naresh, K., Sivasankar, B. N., Madhankumar, S. (2026). *Structural elucidation and coordination chemistry of metal-EDTA complexes*. Structural Chemistry, 37, 125–138.

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