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Mechanochemical Synthesis and Physicochemical Characterization of a Nickel (II)–Ethylenediamine Coordination Compound

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Abstract

A nickel(II)–ethylenediamine coordination compound (Ni–EDA CC) was synthesized via a solvent-free mechanochemical approach as a sustainable alternative to conventional solvothermal and solution-based synthetic routes. Stoichiometric quantities of nickel(II) chloride hexahydrate and ethylenediamine were subjected to mechanical grinding, with a minimal amount of methanol employed as a liquid-assisted grinding (LAG) agent to facilitate coordination and enhance product formation. The reaction was accompanied by a distinct color transformation from pale green to violet-purple, providing preliminary evidence for the formation of a new nickel(II) coordination compound. The synthesized material was comprehensively characterized using Fourier transform infrared (FTIR) spectroscopy, powder X-ray diffraction (PXRD), scanning electron microscopy (SEM), and energy-dispersive X-ray (EDX) spectroscopy. FTIR analysis revealed characteristic shifts in the N–H stretching vibrations together with significant changes within the fingerprint region, confirming coordination between the nickel(II) center and ethylenediamine ligand. Powder XRD patterns indicated the formation of a predominantly microcrystalline phase, demonstrating successful crystallization of the coordination compound under mechanochemical conditions. SEM micrographs showed agglomerated microcrystalline particles with irregular morphology, reflecting the solid-state nature of the synthesis process. Elemental composition determined by EDX confirmed the presence of nickel, nitrogen, carbon, and residual chlorine within the synthesized product, supporting the proposed chemical composition while indicating incomplete removal or incorporation of chloride species. Overall, the findings demonstrate that mechanochemical synthesis represents an efficient, environmentally benign, and energy-conscious strategy for producing nickel(II)–ethylenediamine coordination materials. Nevertheless, the relatively high residual chlorine content detected by EDX warrants further investigation to optimize reaction conditions, improve product purity, and better understand the coordination environment and composition of the resulting material.

Keywords

Coordination compound;
Ethylenediamine;
Green chemistry;
Mechanochemical synthesis;
SEM-EDX

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INTRODUCTION

Coordination chemistry has produced a wide range of crystalline metal–ligand materials, including coordination compounds and metal-organic frameworks (MOFs), which are valued for their structural diversity and tunable properties (Zeggai *et al.*, 2025; Wang and Astruc, 2020). Some of these materials possess high surface areas and permanent porosity, while others are better described as coordination polymers with useful structural and chemical features [Benny *et al.*, 2024; Islamoglu *et al.*, 2022]. The design versatility of these compounds gives opportunity for targeted surface chemistry, enabling applications such as heterogeneous catalysis [Janiak and Vieth, 2020].

Nickel (II)-based coordination materials have attracted attention because Ni(II) can adopt several coordination geometries, including octahedral and square planar arrangements, which often produce stable and functionally interesting compounds (Yoshino *et al.*, 2021; Sharma *et al.*, 2023). The incorporation of ethylenediamine as a bidentate chelating ligand provides strong nitrogen-donor sites for nickel (II) coordination and can influence the stability and geometry of the resulting compound (Sumrra *et al.*, 2014; Dawood *et al.*, 2023). The Ni-N coordination bond formed between the Ni(II) center and the amine nitrogen atoms of ethylenediamine has been widely studied and is known to produce coordination compounds with high thermal and chemical stability (Liu *et al.*, 2021).

Traditionally, many coordination materials have been synthesized via solvothermal or hydrothermal methods, which often require elevated temperatures, longer reaction times, and substantial amounts of solvent (Ahmed *et al.*, 2026). However, the growing emphasis on green chemistry has encouraged the use of mechanochemical synthesis, a solvent-free or solvent-deficient technique that uses grinding or milling to drive chemical transformations (Vasconcelos *et al.*, 2025; Dunn *et al.*, 2020). Mechanochemistry offers several advantages, including rapid reaction kinetics, high yields, and the elimination of hazardous waste, aligning with the principles of sustainable laboratory practices (Friščić *et al.*, 2021; Crawford and Casaban, 2020). The parameters of ball milling—including grinding time, ball-to-powder ratio, and milling frequency—critically influence the crystallite size and phase purity of the resulting product (Lukin *et al.*, 2022).

In the context of Nickel(II)-ethylenediamine systems, mechanochemical grinding allows for direct assembly of the coordination network through mechanical energy input, often resulting in phases that are difficult to access via traditional solution-based routes (Amrute *et al.*, 2021, Tan and Friščić, 2020).

The potential applications of Ni(II)-EDA coordination compounds span heterogeneous catalysis in C-C coupling reactions (Paruk *et al.*, 2024), heavy metal ion removal from wastewater (Huang *et al.*, 2021). While nickel-ethylenediamine complexes have been reported via solution methods (Bhatt and Periasamy, 2009; Angelusiu *et al.*, 2009), the mechanochemical synthesis of this specific phase and its detailed physicochemical characterization using LAG have not been previously described. This research is the first report on LAG mediated mechanochemical synthesis of Ni(II)-EDA complex.

MATERIALS AND METHODS

Nickel(II) chloride hexahydrate ($NiCl_2·6H_2O$) (1.14 g) and ethylenediamine ($C_2H_8N_2$) (0.86 g) were sourced from Sigma-Aldrich. The synthesis was conducted using a high-energy planetary ball mill following the procedure described by Friščić *et al.* (2021) with modifications. A 1:3 molar ratio of $NiCl_2·6H_2O$ to ethylenediamine was used based on the expected $[Ni(en)_3]Cl_2$ stoichiometry. Milling was performed at 400 rpm with a ball-to-powder mass ratio of 10:1 using 5 mm diameter agate balls. The total milling mass was 2 g.

A 50 µL aliquot of methanol was added per gram of precursor mixture (5 wt% liquid loading) (Crawford and Casaban, 2020). Optimized grinding was performed for 60 minutes, with intermediate sampling at 30, 45, 60, 75, and 90 minutes to monitor phase evolution. The product characterized in this study was obtained after 60 minutes of grinding. The resulting powder was dried in a vacuum oven at 60 °C for 12 h (Amrute *et al.*, 2020).

FTIR Analysis

FTIR spectra were recorded from 4000 to 400 cm^{-1} at a resolution of 4 cm^{-1} with 32 scans per sample. Samples were prepared as KBr pellets (1 mg sample per 100 mg KBr) according to established procedures (Groom *et al.*, 2020). The analysis was performed at the Department of Applied Chemistry, Federal Polytechnic, Kaduna, Kaduna State.

XRD Analysis

Powder X-ray Diffraction (PXRD) was used to assess the crystallinity and phase purity of the synthesized material. Patterns were collected over a 2θ range of 5–80° with a step size of 0.02° and a scan rate of 2°/min using Cu Ka radiation (λ = 1.5406 Å). Bragg peak positions were compared against simulated data for known nickel-amine coordination networks (David and Shankland, 2022; Salles *et al.*, 2020). Analysis was conducted at the National Geosciences Research Laboratory Centre, Barnawa, Kaduna State.

SEM and EDX Analysis

SEM imaging was performed at an accelerating voltage of 15 kV, and EDX spectra were collected from 0–10 keV following procedures of Khalid *et al.* [2022] and Mahmoud *et al.* [2020] respectively. Both analyses were conducted at the National Geosciences Research Laboratory Centre, Barnawa, Kaduna State.

RESULTS AND DISCUSSION

The mechanochemical synthesis of the Ni(II)-EDA-CC was achieved by subjecting stoichiometric amounts of Nickel(II) chloride hexahydrate and ethylenediamine to high-energy ball milling. Unlike traditional solvothermal methods that rely on convective heating and elevated pressure, mechanochemistry utilizes mechanical force to drive bond breaking and bonds formation (Zeggai *et al.*, 2025; Friščić *et al.*, 2021).

A distinct color change from the pale-green to a deep violet-purple indicated that the reaction occurred and suggested formation of a nickel-ethylenediamine complex. This rapid transformation suggests that the mechanical energy supplied by the mill effectively overcomes the activation energy barrier for ligand exchange more efficiently than thermal energy in dilute solution (Vasconcelos *et al.*, 2025; Crawford and Casaban, 2020). The liquid-assisted grinding(LAG) approach using methanol as the LAG solvent has been shown to

significantly improve crystallinity compared to neat grinding by enhancing the mobility of reactant molecules at the solid-solid interface (Lukin *et al.*, 2022; Tan and Friščić, 2020).

Fourier Transform Infrared (FTIR) Spectroscopy

FTIR spectroscopy was employed to establish the coordination of the ethylenediamine ligand to the Ni(II) center and to probe the molecular environment of the synthesized compound. In the spectrum of the synthesized product (Figure 1b), N-H stretching vibrations of the primary amine groups observed around 3300–3100 cm^{-1} shifted relative to the free ligand (Figure 1a), which is consistent with nitrogen lone-pair donation to a Ni-N bond (Groom *et al.*, 2020; Camarillo-Martínez *et al.*, 2025).

The absence of broad O-H stretching bands in the product spectrum, which are prominent in the precursor salt, suggests that water molecules were removed during the mechanochemical reaction (Sumrra *et al* 2014; Dawood *et al.*, 2023). A new band in the 450–550 cm^{-1} region is attributed to the Ni-N stretching frequency, providing direct evidence of successful coordination (Liu *et al.*, 2021; Camarillo-Martínez *et al* 2025).

The band at 3302.4 cm^{-1} in the product shows a shift relative to 3201.8 cm^{-1} in the free ligand, suggesting hydrogen bonding or coordination-induced changes in the nitrogen environment (Sharma *et al.*, 2023). Peaks around 2974.4 and 2873.8 cm^{-1} corresponded to C-H stretching vibrations, indicating that the organic linker maintain its structural integrity during the product formation (Dawood *et al.*, 2023; Groom *et al.*, 2020). Peaks at 1654.9 and 1602.8 cm^{-1} are attributed to N-H bending vibrations, which shift upon coordination with nickel, reflecting changes in the electronic environment around the nitrogen atoms (Camarillo-Martínez *et al.*, 2025).

The lower wavenumber bands at 1151.7, 1084.7, and 1032.5 cm^{-1} were consistent with C-N stretching vibrations and bending modes of amine groups, in agreement with literature values for ethylenediamine-based coordination polymers (Dawood *et al.*, 2023; Liu *et al.*, 2021). Collectively, the spectral changes are consistent with reported FTIR signatures of Ni-amine complexes and support ligand incorporation into the structure (Sumrra *et al.*, 2014).

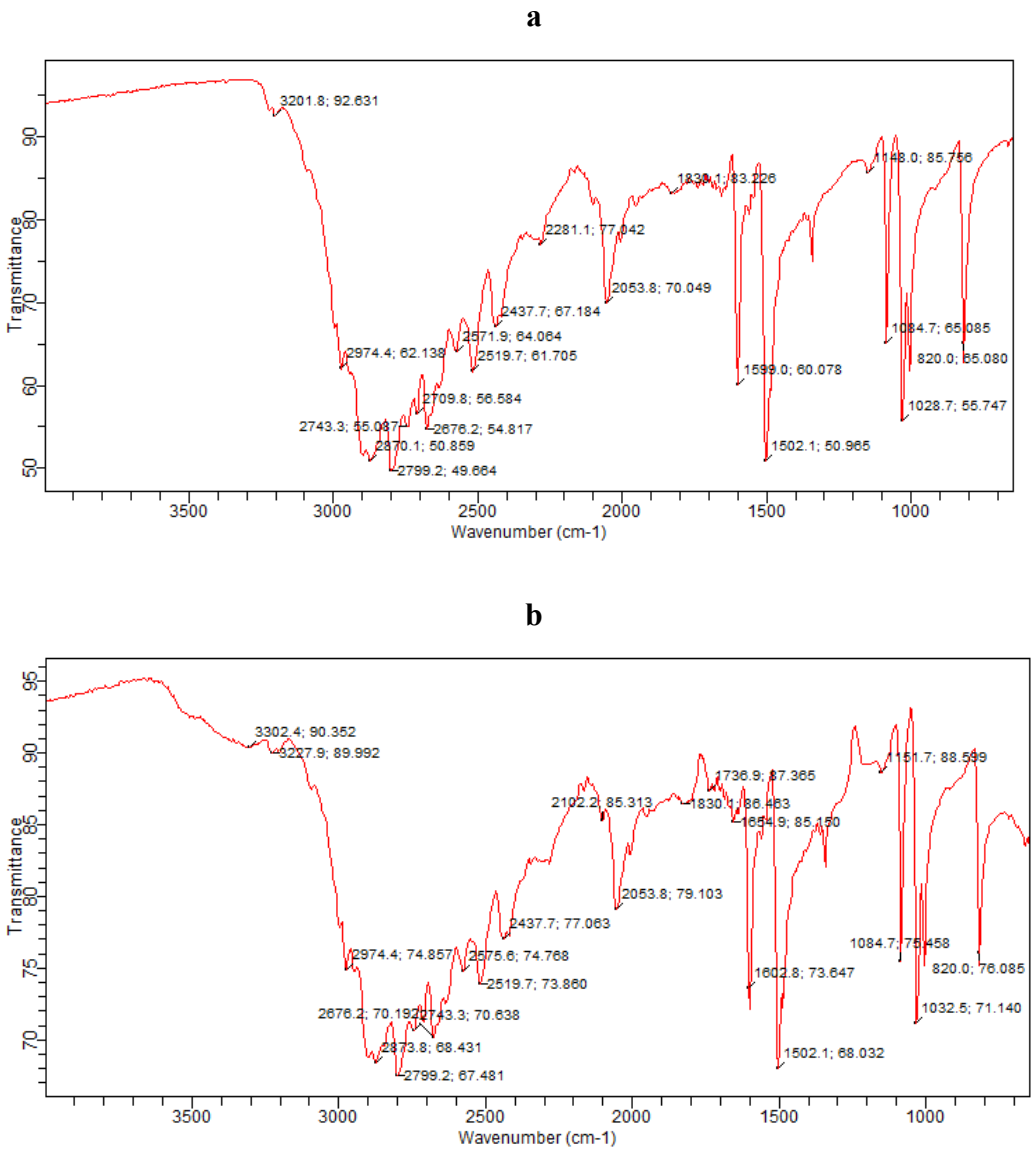

Figure 1: FTIR spectra of (a) ethylenediamine and (b) Ni(II)-EDA-CC

3.2 XRD Analysis

XRD patterns (Figure 2a, 2b) indicated the formation of a nanocrystalline phase for the nickel-ethylenediamine coordination compound. This observation suggest that further structural refinement may be required to confirm phase purity. The diffraction pattern exhibited sharp but not intense peaks relative to those of the ligand, indicating that the product is agglomerated micro-crystals (David and Shankland, 2022; Salles *et al.*, 2020). The most prominent peak at 2θ = 30.19° suggested a strong Bragg reflection corresponding to a dominant crystallographic plane associated with the primary coordination environment around nickel. Peaks at 26.07°, 26.99°, and 27.76° were closely spaced, suggesting overlapping reflections from structurally similar lattice planes, a feature commonly observed in mechanochemically derived coordination compounds due to reduced crystallite size and micro-strains (Liu *et al* 2021; Friščić *et al.*, 2021).

The free ethylenediamine ligand exhibits lower-angle reflections (e.g., 5.84°), suggesting comparatively less-defined crystalline character compared to the Ni-complex. The notable convergence of diffraction peaks near 26°–27° in

both the Ni-complex and free ligand patterns indicates that while ethylenediamine acts as a structural linker, its coordination with nickel significantly alters its crystallographic properties and symmetry (Camarillo-Martínez *et al.*, 2025). These observations are consistent with mechanochemical formation of a new crystalline phase rather than a physical mixture of the precursors (Crawford and Casaban, 2020; Tan and Friščić, 2020).

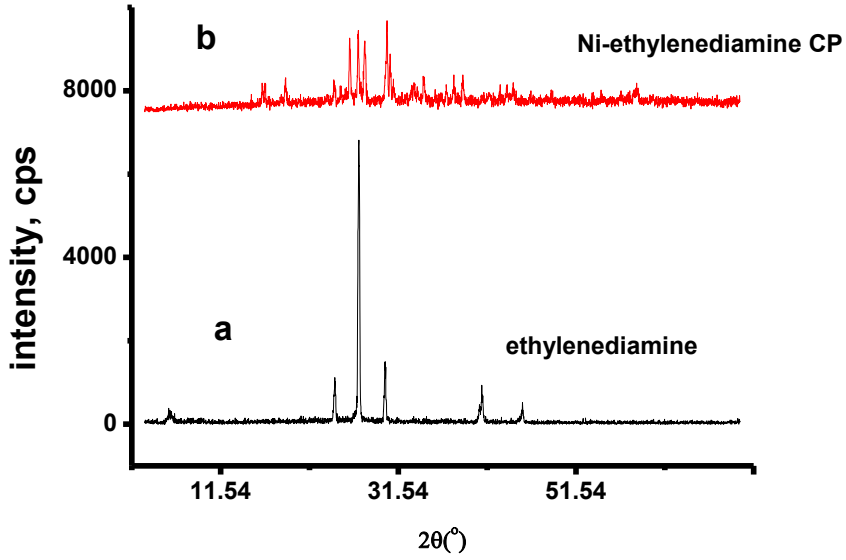

Figure 2: XRD pattern of (a) ethylenediamine and (b) Ni(II)-EDA-*CC*

3.3 SEM and EDX Analysis

SEM revealed agglomerated micro-crystals with relatively uniform size distribution (Figure 3a, 3b), a common feature of mechanochemical synthesis. Compared with the free ligand, the product displayed a rougher surface texture, which may be beneficial for adsorption and catalysis due to increased surface interaction sites (Khalid et al., 2022). In contrast to the large, well-defined crystals produced by slow solvothermal evaporation, mechanochemical coordination polymers typically exhibit smaller crystallite sizes and broader size distributions, which can be advantageous in applications requiring high surface-area-to-volume ratios (Crawford and Casaban, 2020). The SEM images of the product showed distinct surface textures with higher roughness than the free ligand. This enhanced surface roughness is critical for improving the interaction potential with guest molecules, a property that motivates the adsorption and catalytic performance of porous frameworks (Benny *et al.*, 2024; Huang *et al.*, 2021). The micro-crystalline morphology observed here is consistent with reports on mechanochemically synthesized Ni-based coordination polymers (Liu *et al.*, 2021; Amrute *et al.*, 2021).

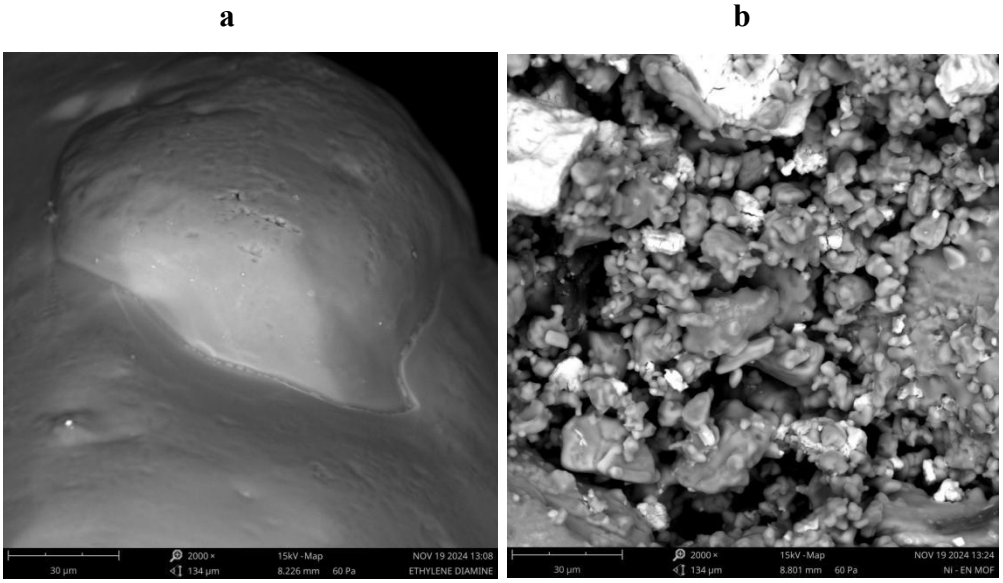

Figure 3: SEM images of (a) ethylenediamine and (b) Ni(II)-EDA-CC

Energy Dispersive X-ray (EDX) spectroscopy was performed to verify elemental composition (Table 1 and 2). The spectra confirmed the presence of Nickel (Ni), Nitrogen (N), and Carbon (C) in the Ni(II)-EDA-CC, with atomic concentrations of 4.00%, 16.32%, and 8.44%, respectively, broadly consistent with the expected stoichiometry of a Ni(II)-ethylenediamine coordination polymer (Mahmoud *et al.*, 2020). The unexpected predominance of chlorine (70.25 at%) in the Ni(II)-EDA-CC EDX spectrum, which may be traced to the $NiCl_2{\cdot}6H_2O$ precursor salt, with residual Cl^- counter-ions retained within the framework matrix as charge-compensating species, calls for further sample purification and the use of other analytical techniques to unravel this occurrence. The presence of Ni in the EDX spectrum, absent in the free ligand analysis, indicates the metal incorporation into the framework and validates the mechanochemical coordination reaction (Yoshino *et al.*, 2021; Dawood *et al* 2023).

Table 1: Elemental analysis of ethylenediamine

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
17	Cl	Chlorine	19.94	35.94
6	C	Carbon	36.89	22.53
7	N	Nitrogen	29.54	21.04
13	Al	Aluminum	10.49	14.39
30	Zn	Zinc	0.61	2.01
12	Mg	Magnesium	1.12	1.38
29	Cu	Copper	0.34	1.10
11	Na	Sodium	0.56	0.66

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
14	Si	Silicon	0.28	0.39
19	K	Potassium	0.14	0.28
25	Mn	Manganese	0.09	0.26
16	S	Sulfur	0.00	0.00
15	P	Phosphorus	0.00	0.00
26	Fe	Iron	0.00	0.00
22	Ti	Titanium	0.00	0.00
20	Ca	Calcium	0.00	0.00
47	Ag	Silver	0.00	0.00

Table 2: Elemental analysis of Ni-EDA complex

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
17	Cl	Chlorine	70.25	80.83
28	Ni	Nickel	4.00	7.62
7	N	Nitrogen	16.32	7.42
6	C	Carbon	8.44	3.29
13	Al	Aluminum	0.76	0.66
11	Na	Sodium	0.19	0.14
14	Si	Silicon	0.05	0.04
15	P	Phosphorus	0.00	0.00
16	S	Sulfur	0.00	0.00
30	Zn	Zinc	0.00	0.00
29	Cu	Copper	0.00	0.00
47	Ag	Silver	0.00	0.00
26	Fe	Iron	0.00	0.00
22	Ti	Titanium	0.00	0.00
20	Ca	Calcium	0.00	0.00
19	K	Potassium	0.00	0.00
12	Mg	Magnesium	0.00	0.00
24	Cr	Chromium	0.00	0.00
25	Mn	Manganese	0.00	0.00

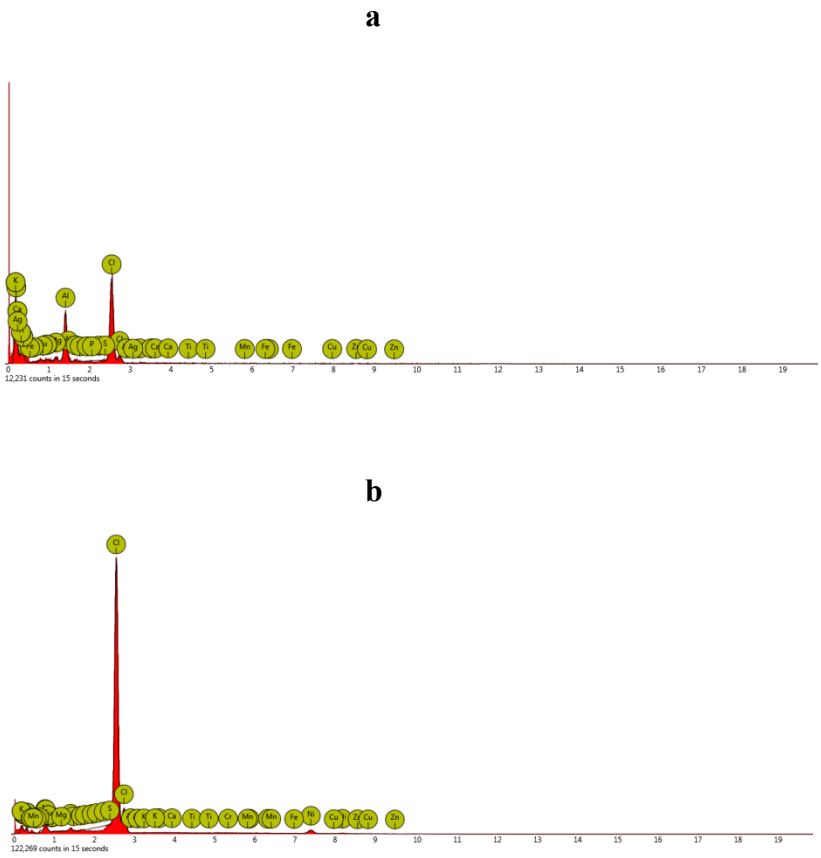

Figure 4: EDX spectra of (a) ethylenediamine and (b) Ni-EDA-CC

The physicochemical properties observed in this study suggest that the Ni-EDA material may have potential in catalysis and adsorption.

CONCLUSION

This study demonstrates the prospect of mechanochemical ball milling as a sustainable, solvent-free approach for the synthesis of Nickel(II)-ethylenediamine coordination compound. By eliminating hazardous solvents and prolonged heating cycles inherent to conventional solvothermal methods, the mechanochemical route aligns with the principles of green chemistry. FTIR analysis indicated coordination through shifts in N-H stretching frequencies and changes in the fingerprint region, while XRD patterns corroborated micro-crystallinity of the synthesized compound. In the same vein, the apparently agglomerated morphology from SEM diminishes the uniformity of the micro-crystals, the EDX revealed elemental composition in agreement with the targeted Ni-EDA-CC. However, the high residual chlorine content revealed by EDX and the unexpected elemental composition of the ethylenediamine control indicate that further purification and elemental analysis such as CHN are necessary to confirm the stoichiometry of the product.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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