

Comprehensive Physicochemical Characterization of Gambier Leaf Extract-Modified Layered Double Hydroxide

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Abstract

Layered double hydroxide (LDH) is a promising material; however, its application is still limited by agglomeration and suboptimal physicochemical properties. In this study, Ni–Al LDH was modified with *Uncaria gambir* leaf extract via coprecipitation to improve its properties. Characterization using FT-IR, XRD, BET, SEM/TEM, XPS, and UV–DRS showed that the layered structure of LDH was retained after modification, while significant changes in surface chemistry resulted from the introduction of phenolic groups. Interaction between the extract and LDH occurs primarily at the surface via hydrogen bonding and π – π interactions. Optical analysis revealed a decrease in the bandgap energy from 2.32 eV to 1.32 eV, indicating increased visible-light absorption. Textural properties revealed a decrease in surface area due to pore closure and increased agglomeration. Nevertheless, the presence of active groups from the extract enhances the material's reactivity and interaction potential. These results demonstrate that gambier extract effectively modifies the physicochemical properties of LDH, providing a strategy for developing sustainable biomass-based hybrid materials.

Keywords

LDH, *Uncaria gambir*, Extract, Physicochemical

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1. INTRODUCTION

Layered double hydroxide (LDH) is a layered inorganic material that has attracted widespread attention due to its unique lamellar structure, tunable chemical composition, and diverse physicochemical properties (Dahiya et al., 2026; Theka et al., 2026; Wang et al., 2026). The LDH structure consists of positively charged brucite-like layers balanced by anions in the interlayer space, enabling ion exchange, surface modification, and structural flexibility (De and Kundu, 2026; Mallick et al., 2026; Sadiq et al., 2026). Owing to these characteristics, LDH has been extensively developed for applications in catalysis, environmental remediation, and functional materials (Imran et al., 2026; Koventhan et al., 2026; Mandari et al., 2026; Sharma et al., 2026; Tudorache et al., 2026; Yu et al., 2026). However, pure LDH still has limitations, including relatively low structural stability, a tendency to agglomerate, and suboptimal electronic properties (Chen et al., 2025), necessitating modification strategies to enhance their performance.

In recent years, the integration of natural organic compounds into inorganic matrices has emerged as a promising approach for sustainably modifying material properties. Natural plant extracts are known to be rich in bioactive compounds such as

polyphenols, flavonoids, and tannins, which possess reactive functional groups (Abdou-Allah et al., 2026; Gaikwad et al., 2026; Nath et al., 2026). These compounds can interact with the surfaces of inorganic materials via hydrogen bonding, electrostatic interactions, and π – π interactions, thereby enhancing surface reactivity, modifying electronic structure, and strengthening interfacial interactions.

Recent studies have shown that the incorporation of natural extracts into the structure of layered double hydroxides (LDH) significantly modifies the material's physicochemical properties. A study by Bagherzadeh et al. (2024) reported that Zn–Al LDH composites with thyme extract exhibited strong interactions in the form of hydrogen bonding and electrostatic interactions between phenolic groups and the LDH layers, as indicated by shifts in the FT-IR bands and an increase in specific surface area. Meanwhile, Soleymani et al. (2024) demonstrated that phytochemical compounds from *Matricaria chamomilla* act as reducing agents and stabilizers, resulting in significant changes in the structure and surface properties of LDH, confirmed through FT-IR, XRD, and morphological analysis. Other studies also reveal that modifying LDH with biomass extracts, such as mangosteen peel, can increase surface area and prevent particle agglomeration due to the presence of bioactive compounds, such as flavonoids and

tannins, that enrich surface functional groups (Komis et al., 2026). Similar results were reported by Amri et al. (2026), in which the incorporation of tea extract reduced the band gap and increased visible-light absorption capacity, which is attributed to interactions between polyphenolic compounds and the LDH structure. Additionally, Sayeri et al. (2026) demonstrated that the addition of dragon fruit peel extract can maintain the crystalline structure of LDH while increasing surface area and improving particle dispersion by inhibiting agglomeration. These studies confirm that natural extracts not only act as surface-modifying agents but also significantly alter the structure, morphology, textural properties, and optical characteristics of LDH.

Among various natural biomass sources, gambier leaves (*Uncaria gambir*) show high potential because they are rich in phenolic compounds, particularly catechins, as their main component (Rahmadiawan et al., 2026). Catechins are known to have aromatic structures with abundant hydroxyl groups, enabling them to interact strongly with inorganic materials through hydrogen bonding, electrostatic interactions, $\pi - \pi$ interactions, and complexation with metal cations on the LDH surface (Zhang et al., 2021). The presence of these functional groups not only enhances the material's surface affinity but also can modify the electronic structure and enrich the active sites on LDH. Compared to various other natural biomass sources, gambier leaves are reported to contain high levels of polyphenols, with catechins as the dominant component (Malrianti et al., 2021), which significantly enhances the material's chemical reactivity and other reactive properties. Nevertheless, the utilization of gambier leaf extract in LDH systems has not yet been extensively studied, particularly regarding how these bioactive compounds influence the material's physicochemical properties as a whole. Therefore, a more comprehensive study is needed to elucidate the specific role of gambier extract in modulating LDH characteristics.

Based on this, this study aims to synthesize and investigate Ni–Al LDH composites modified with gambier leaf extract using the coprecipitation method. The resulting materials were characterized using various techniques, including X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), Brunauer–Emmett–Teller (BET) analysis, X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and UV–Vis diffuse reflectance spectroscopy (UV-DRS) to evaluate structural, morphological, surface, and optical properties. This study is expected to provide a more comprehensive understanding of the influence of natural extracts on the physicochemical properties of LDH and open opportunities for the development of more effective and sustainable biomass-based hybrid materials.

2. EXPERIMENTAL SECTION

2.1 Reagents

All reagents employed in this work were of analytical grade and used as received. Metal precursors for LDH synthesis included nickel nitrate hexahydrate and aluminum nitrate nonahydrate, while sodium hydroxide and sodium carbonate were utilized to regulate alkalinity and facilitate precipitation. Hydrochloric acid

was added as needed to adjust pH. Ethyl acetate was selected as the extraction medium for isolating bioactive compounds from gambier leaves. Distilled water was used throughout all experimental procedures. The gambier leaves (Figure 1) were collected from Toman Village, Musi Banyuasin, Indonesia, and served as the natural source for the extract.



Figure 1. Gambier (*Uncaria gambir*) Leaves

2.2 Maceration Extraction of Gambier Leaves

Gambier leaves were processed prior to extraction by drying and size reduction to enhance extraction efficiency. The powdered sample was macerated for 72 hours under ambient conditions in the absence of light (Jefri et al., 2025b). Following extraction, the mixture was separated by filtration, and the resulting solution was concentrated under reduced pressure using a rotary evaporator at 50 °C to remove the solvent. The concentrated extract obtained from this process was directly used as a functional component in composite synthesis.

2.3 Fabrication of LDH–Extract Composites

The Ni–Al LDH matrix was prepared via a co-precipitation approach by combining nickel and aluminum precursors under controlled alkaline conditions (Jefri et al., 2025b). The solution pH was adjusted to 10 by gradual addition of basic solutions, thereby promoting the formation of layered hydroxide structures. After achieving the desired pH, the gambier extract was introduced into the system to enable interaction with the forming LDH layers. The reaction mixture was maintained at an elevated temperature under continuous stirring to ensure proper incorporation of the extract into the LDH framework. The resulting solid product was separated, washed, dried, and ground before further analysis.

2.4 Physicochemical Characterization Techniques

A range of analytical techniques was applied to investigate the physicochemical characteristics of the synthesized materials. FT-IR spectra were collected in the 4000–500 cm^{-1} region using a Shimadzu Prestige-21 instrument, while XRD patterns were recorded on a Rigaku MiniFlex 600 diffractometer over a 2θ range

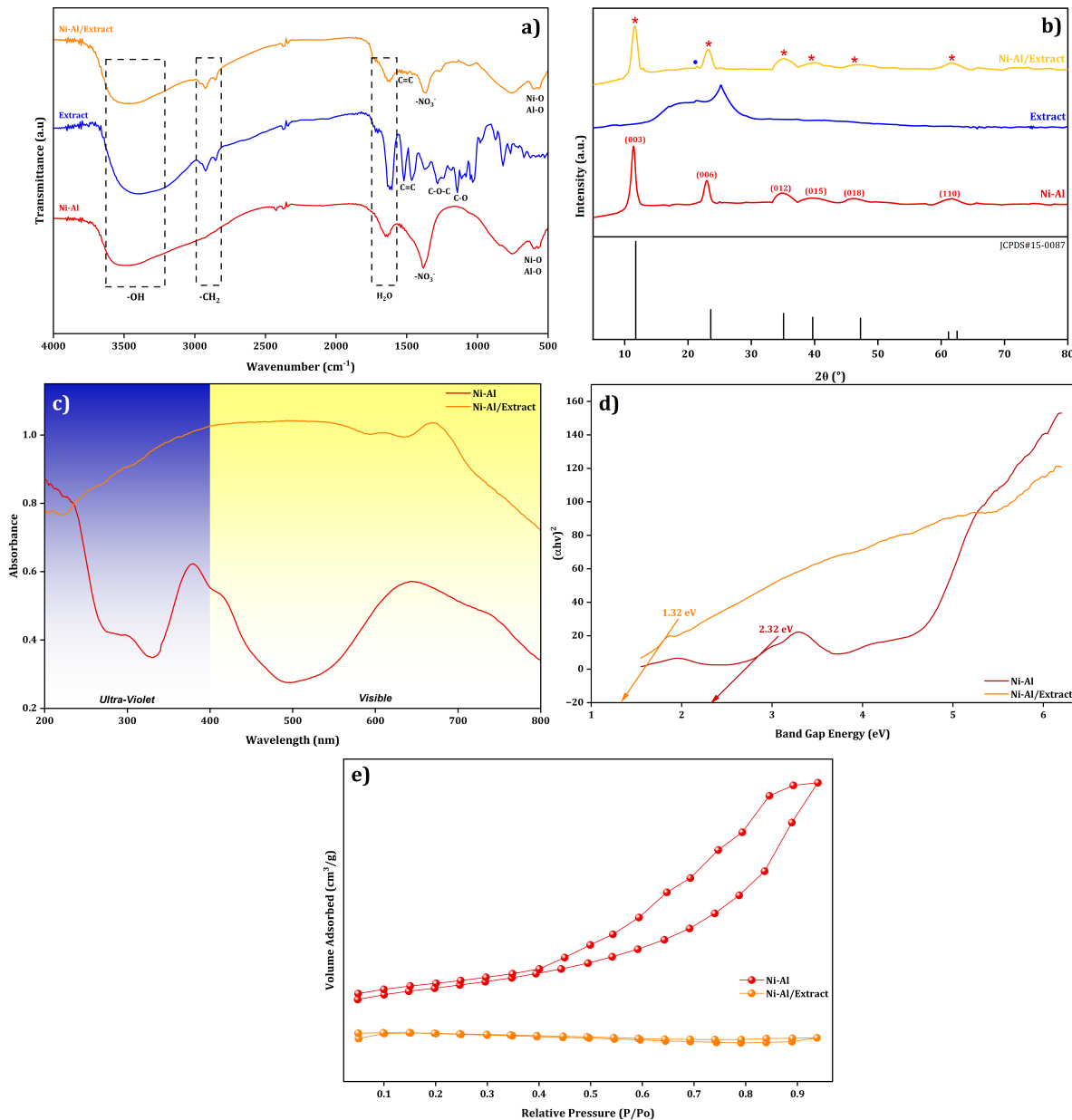


Figure 2. Functional Group Interaction, Crystal Structure, Optical Properties, and Textural Characteristics of Ni-Al LDH and Ni-Al/Extract Composite: (a) FT-IR Spectra, (b) XRD Patterns, (c) UV-DRS Spectra, (d) Tauc Plot for Band Gap Determination, and (e) N₂ Adsorption-Desorption Isotherms

of 5–80°. The optical response was examined using UV-Vis diffuse reflectance spectroscopy (Jasco V-760) over 200–800 nm, and the band gap energy was estimated from the Tauc plot. Textural properties, including surface area and pore structure, were evaluated using BET analysis with a BELSORP-miniX system. Morphology and particle features were observed using a Hitachi SU3500 SEM, supported by particle size analysis via ImageJ, and further detailed by TEM using a Talos F200X instrument. In ad-

dition, surface elemental composition and chemical states were determined by XPS (Kratos AXIS SUPRA PLUS) within a binding energy range of 0–1200 eV.

3. RESULTS AND DISCUSSION

3.1 Functional Group Interaction

The FT-IR spectra of Ni-Al LDH, gambier extract, and the Ni-Al/Extract composite (Figure 2a) reveal characteristic differences

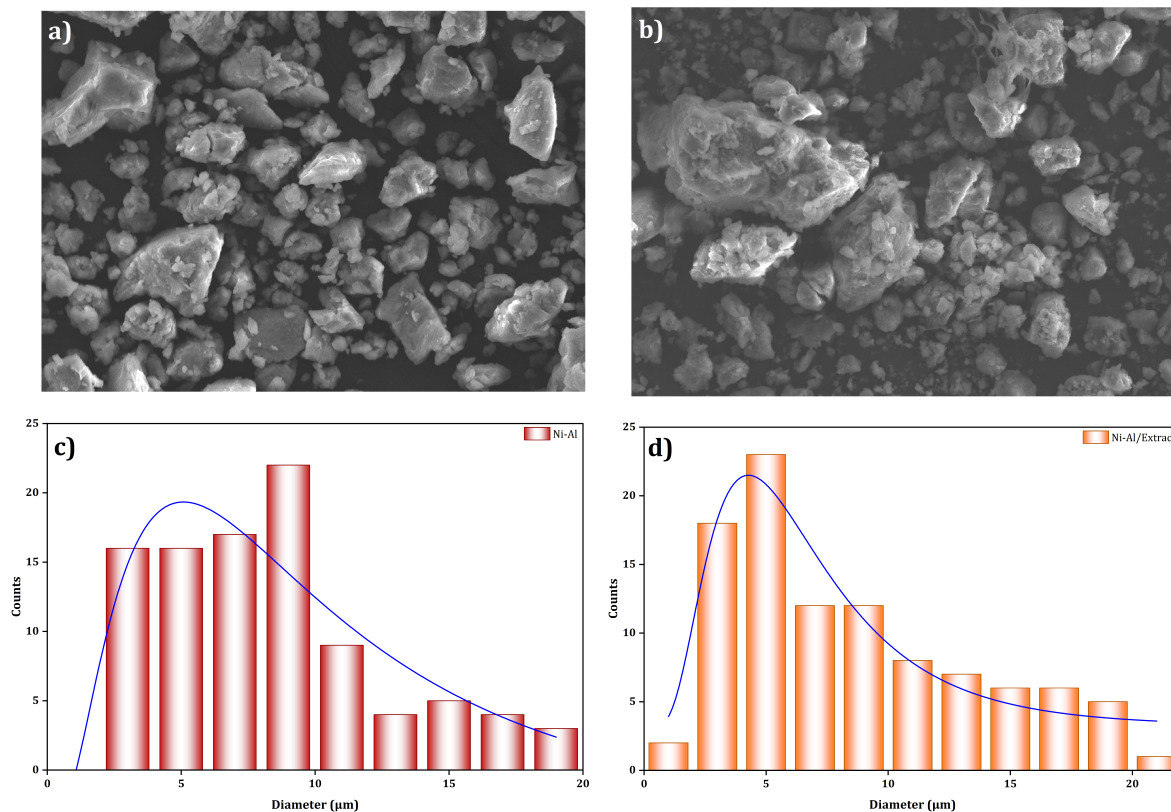


Figure 3. Surface Morphology and Particle Size Distribution of Ni-Al LDH and Ni-Al/Extract Composite: (a,c) SEM Images and (b,d) Particle Size Distribution Profiles

in functional groups, indicating interactions between the components. In pure Ni-Al LDH, the broad band at around 3495.23 cm^{-1} is associated with the $-\text{OH}$ stretching vibration of the hydroxide layer and adsorbed water molecules, while the band at 1638.23 cm^{-1} indicates the H_2O bending vibration (Kaur et al., 2025). The characteristic band of the interlayer anion $-\text{NO}_3^-$ appears in 1374.84 cm^{-1} (Zou et al., 2025), while the bands in the low-frequency region ($500\text{--}800 \text{ cm}^{-1}$) are related to Ni-O and Al-O vibrations (VM et al., 2026), which are characteristic of the LDH structure. On the other hand, gambier extract exhibits a strong $-\text{OH}$ band (3420.53 cm^{-1}), aliphatic C-H vibrations (2917.57 cm^{-1}), aromatic C=C bands (1518.45 cm^{-1}), and C-O/C-O-C bands at $\sim 1000\text{--}1300 \text{ cm}^{-1}$ (Sofyan et al., 2025), indicating the presence of polyphenolic compounds such as catechins.

In the Ni-Al/Extract composite, shifts and intensity changes were observed in several major bands, particularly in the $-\text{OH}$ and aromatic regions, indicating interactions between the extract and the LDH surface. The shift in the $-\text{OH}$ band indicates the formation of hydrogen bonds between the phenolic groups and the LDH hydroxide layers, while the presence and changes in the aromatic C=C bands suggest possible $\pi-\pi$ interactions or the contribution of the aromatic system to the composite structure

(Bagherzadeh et al., 2024). Additionally, changes in the intensity of the $-\text{NO}_3^-$ band indicate interactions or partial exchange of interlayer anions with organic molecules in the extract. Interestingly, the Ni-O and Al-O bands remain observable without significant changes, indicating that the basic LDH structure remains stable after modification. These results confirm that the gambier extract successfully integrates into the LDH system through a combination of physical and chemical interactions, with the potential to enhance the material's surface properties and reactivity.

3.2 Crystal Structure Analysis

The XRD patterns of the gambier extract and the Ni-Al LDH material are shown in Figure 2b. The gambier extract exhibits a broad peak (broad hump) in the 2θ range of approximately $20\text{--}30^\circ$, indicating an amorphous nature due to the absence of long-range crystalline order (Mishra et al., 2025). This characteristic is associated with the predominance of organic compounds such as polyphenols and catechins, which generally do not form crystalline phases. In contrast, Ni-Al LDH exhibits characteristic diffraction peaks at $2\theta = 11.47^\circ$ (003), 23.14° (006), 34.66° (012), 39.76° (015), 46.33° (018), and 64.10° (110), which correspond to JCPDS Standard No. 15-0087 for a hydrotalcite-like structure (Liu et al., 2025; Madhu Malar et al., 2026; Rise et al., 2025). The pres-

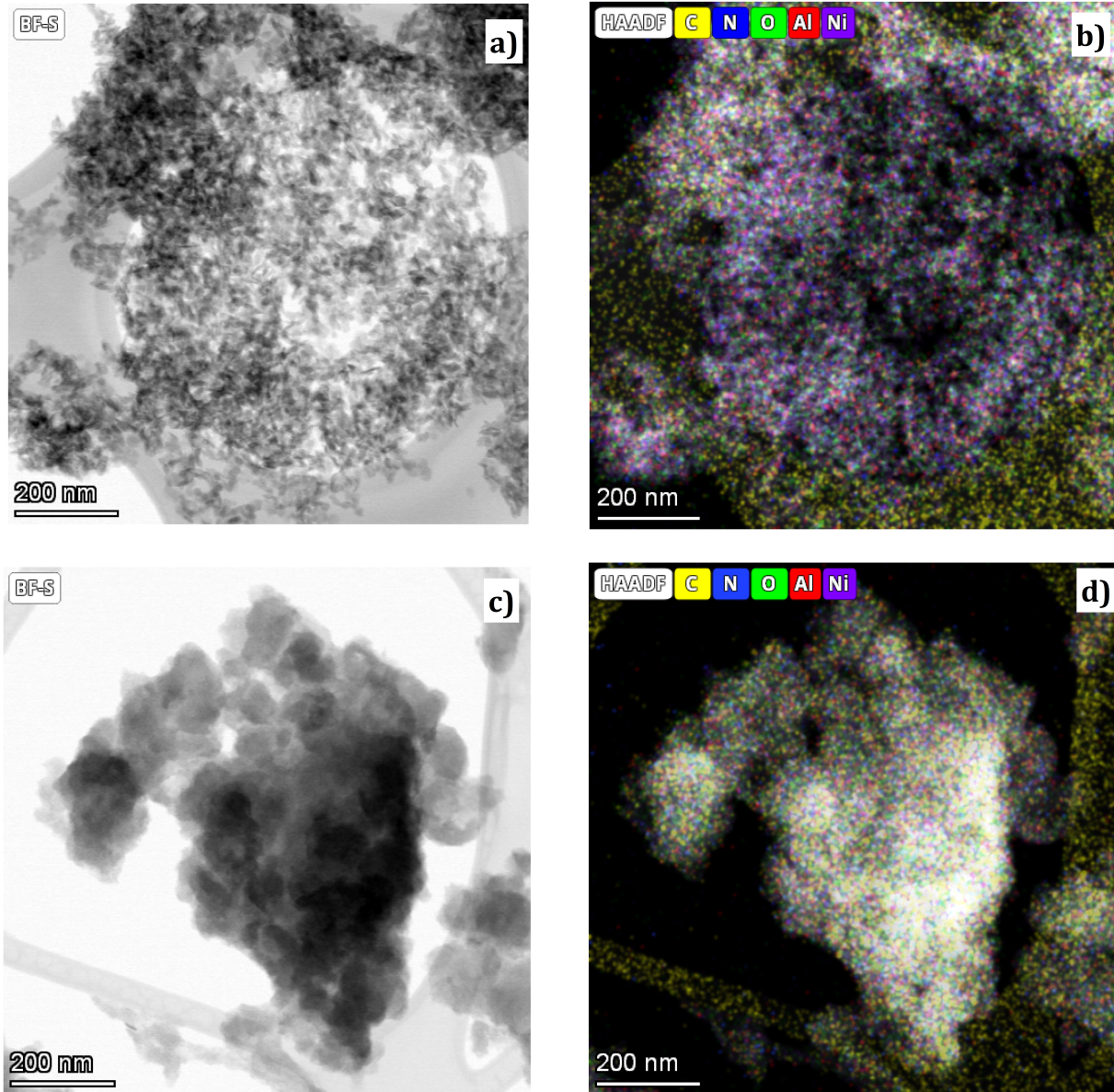


Figure 4. Microstructural and Elemental Distribution Analysis of Ni-Al LDH and Ni-Al/Extract Composite: (a,c) TEM Images and Corresponding Elemental Mapping

ence of these peaks confirms the formation of a characteristic layered LDH structure with good crystal order.

In the composite material, all characteristic LDH peaks remain observable without significant changes, indicating that the primary crystal structure remains stable after modification with gambier extract. However, there was a slight shift in the (003) peak from 11.47° to 11.69° , accompanied by a decrease in the d-spacing value from 7.71 \AA to 7.56 \AA , indicating a reduction in interlayer spacing. This indicates that the interaction between the extract and LDH does not cause significant interlayer expansion, so the extract molecules tend to interact on

the LDH surface (Madhu Malar et al., 2026). Additionally, the appearance of several low-intensity peaks in the composite pattern indicates a contribution from the extract's organic components, suggesting successful integration between the organic and inorganic phases without the formation of a dominant new crystalline phase. These results indicate that the gambier extract is dispersed and interacts with the LDH structure through noncovalent mechanisms, without damaging its primary crystal framework.

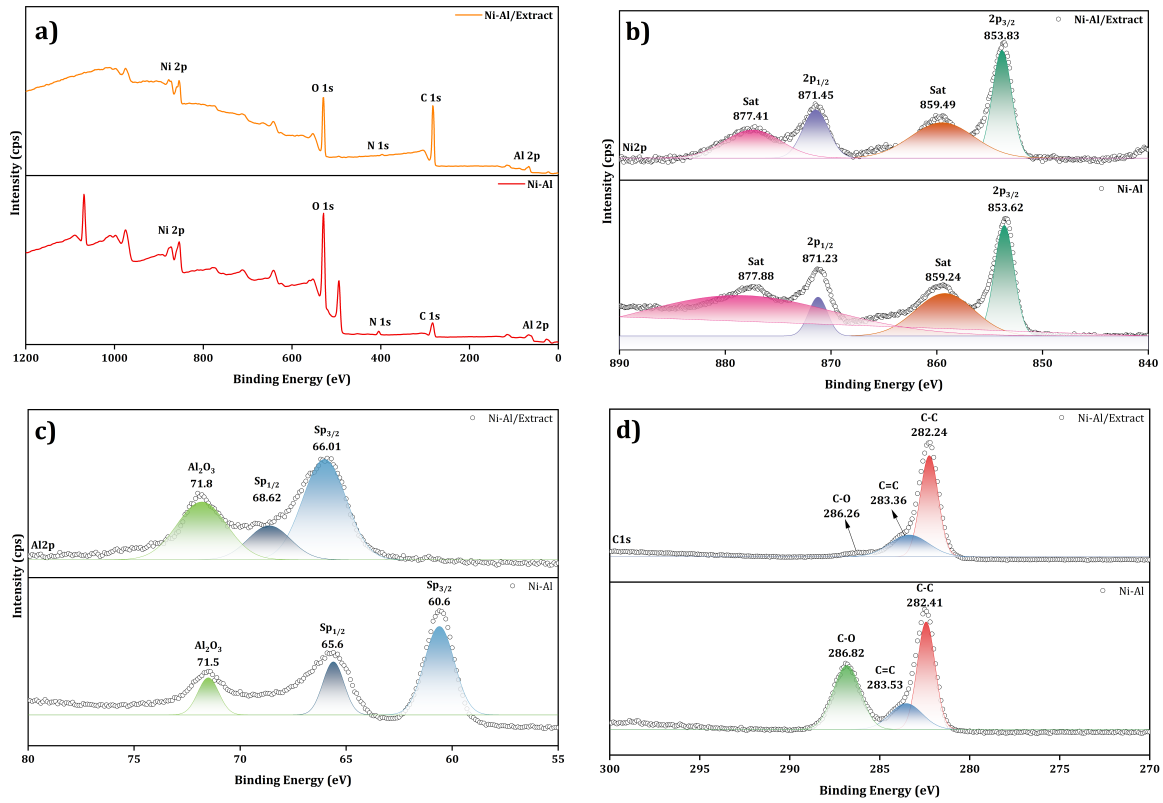


Figure 5. Surface Chemical State Analysis of Ni–Al LDH and Ni–Al/Extract Composite by XPS: (a) Survey Spectra and High-Resolution Spectra of (b) Ni 2p, (c) Al 2p, and (d) C 1s

3.3 Optical Properties and Band Gap Energy

The optical properties of the Ni–Al LDH material and the Ni–Al/Extract composite were analyzed using UV-DRS, as shown in Figure 2c. The Ni–Al LDH exhibits dominant absorption in the ultraviolet region with limited contribution in the visible light region, reflecting a relatively large bandgap. After modification with gambier extract, an increase in absorption intensity was observed in the visible light region. This increase is attributed to the presence of polyphenolic compounds with a π -conjugated system, which enables the material's optical response to extend into lower-energy regions.

The band gap energy was determined using the Tauc method under the assumption of a direct band gap, following the approach reported in previous studies on LDH-based materials (Yekan Motlagh et al., 2026). Based on the analysis results (Figure 2d), the band gap value of Ni–Al LDH, which was 2.32 eV, decreased significantly to 1.32 eV in the composite material. This decrease indicates that the presence of gambier extract modifies the material's electronic structure through the formation of new energy levels or interactions between phenolic groups and metal cations on the LDH surface (Amri et al., 2026). Modification using gambier extract effectively enhances the material's ability

to absorb visible light by reducing the band gap energy.

3.4 Textural Properties

The textural properties of the Ni–Al LDH material and the Ni–Al/Extract composite were analyzed using nitrogen adsorption–desorption isotherms, as shown in Figure 2e. Ni–Al LDH exhibits a gradual increase in adsorption volume followed by a sharper rise at high relative pressures ($P/P_0 > 0.7$), indicating the presence of mesoporous structures and interparticle voids (Waheed et al., 2023). In contrast, the Ni–Al/Extract composite exhibits a significantly lower adsorption capacity across the entire range of relative pressures, indicating a significant change in surface characteristics following the modification process with gambier extract.

The specific surface area determined by the BET method for Ni–Al LDH, which is 31.73 m²/g, decreases drastically to 4.13 m²/g in the composite material. This decrease indicates that organic molecules from the gambier extract cover part of the surface or clog the LDH pores, thereby reducing the number of accessible active sites. The presence of polyphenolic compounds such as catechins is suspected to play a role in this process through strong interactions with the LDH surface (Jefri et al.,

Table 1. Summary of Physicochemical Effects of Biomass Extract-Modified LDHs and Their Applications

LDH-Based	Biomass	Application	Target	Key Findings	Reference
Mg-Cr	<i>Hylocereus costaricensis</i> Peel Extract	Adsorption	Ceftriaxone	Improves surface stability without structural change	Sayeri et al. (2026)
Zn-Al	<i>Camellia sinensis</i> Extract	Photocatalysis	Ceftriaxone	Controls particle growth via phytochemicals	Amri et al. (2026)
Zn-Al	<i>Garcinia mangostana</i> Peel Extract	Adsorption	Levofloxacin	Tunes interlayer spacing and surface functionality	Komis et al. (2026)
Zn-Al	Thyme Extract	Adsorption	Methylene Blue	Slight increase in surface area	Bagherzadeh et al. (2024)
Cu-Al-Zn	<i>Matricaria chamomilla</i> Extract	Antimicrobial	Gram (+/-) Bacteria	Enables surface functionalization via phytochemicals	Soleymani et al. (2024)
Zn-Al	<i>Zingiber montanum</i> Extract	Antimicrobial	<i>S. aureus</i>	Enhances stability and properties	Intachai et al. (2024)
Mg-Al	<i>Sargassum liebmannii</i> and <i>Ulva ohnoi</i> Extracts	Agriculture	Plant Growth	Alters phase aggregation and surface chemistry	Espinosa-Antón et al. (2025)
Mg-Al	<i>Angelica gigas Nakai</i> Extract	Anticancer	Cancer Cells	Modifies crystallinity and surface charge	Kim et al. (2018)
Zn-Al	Callus Leaf Extract	Photocatalysis	MO, Gram (+/-)	Adds functional groups and active sites	Mahious et al. (2025)
Ni-Al/Ni-Fe /Ni-Cr	<i>Uncaria gambir</i> Leaf Extract	Photocatalysis	Tetracycline	Improves optical properties	Jefri et al. (2025a)

2025b). These results are consistent with FT-IR analysis, which revealed functional group interactions, and are supported by XRD, indicating that the extract interacts more with the surface rather than being intercalated into the layers. Although the surface area decreased, the presence of active functional groups in the extract can enhance chemical interactions with target molecules, thereby still contributing positively to the material's adsorption performance.

3.5 Morphological and Microstructural Analysis

Morphological analysis using SEM (Figure 3a,c) with particle size distribution analysis (Figure 3b,d) revealed that Ni-Al LDH exhibits irregular particle aggregates with rough surfaces composed of layered sheets typical of LDH, along with a relatively broad particle size distribution. After modification with gambier extract, the Ni-Al/Extract composite exhibited increased agglomeration, characterized by larger aggregates and a denser

structure, accompanied by a more centralized size distribution. However, a fraction of large particles remained due to agglomeration. A similar phenomenon has been reported in LDH systems modified with natural extracts, where the intercalation of organic compounds and intermolecular interactions lead to the formation of larger aggregates and an increase in structural density (Kechagias et al., 2025).

Further TEM (Figure 4a,c) observations revealed that the Ni-Al LDH consists of nanoparticle aggregates with a relatively uniform distribution and a layered structure that remains visible. In the Ni-Al/Extract composite, an increase in aggregate density and a decrease in nanoscale particle dispersion were observed, confirming the SEM results. Elemental mapping (Figure 4a,c) results showed a homogeneous distribution of Ni, Al, and O, along with the presence of C in the composite, indicating the successful integration of gambier extract. The consistent increase in agglomeration observed in SEM and TEM aligns with

the BET analysis results, which show a decrease in surface area, likely due to pore and surface closure by organic compounds. Modification with gambier extract does not damage the basic LDH structure but significantly affects the morphology, particle size distribution, and microstructure of the material.

3.6 Surface Chemical States Analysis

Analysis of the surface chemical composition was performed using XPS, as shown in Figures 5a–d. The survey spectra confirmed the presence of the main elements Ni, Al, O, N, and C in both samples, with an increased intensity of the C 1s signal in the Ni–Al/Extract composite, indicating the successful immobilization of organic components from the gambier extract onto the material surface (Jefri et al., 2025a). In the high-resolution Ni 2p spectrum, two main peaks were identified in the range of 853.62–853.83 eV (Ni 2p_{3/2}) and 871.23–871.45 eV (Ni 2p_{1/2}), accompanied by satellite peaks around 859.24–859.49 eV and 877.41–877.88 eV, which are characteristic of the Ni²⁺ species; there were no significant changes in peak positions after modification, although a slight shift in binding energy was observed, indicating an interaction between Ni ions and organic functional groups. The Al 2p spectrum shows peaks in the range of 71.5–71.8 eV, consistent with the presence of Al³⁺ in the LDH structure, with no significant changes after modification, thus confirming that the main-layer structure remains stable. Meanwhile, the C 1s spectrum shows main C–C peaks at 282.41 eV (Ni–Al) and 282.24 eV (Ni–Al/Extract), as well as C=C components in the range of 283.53–283.36 eV and C–O at 286.82 eV (Ni–Al) and 286.26 eV (Ni–Al/Extract). Following modification, the relative intensities of the C–C and C=C components increased, indicating the dominance of the aromatic carbon framework in the phenolic compounds of the gambier extract, while the C–O intensity decreased, suggesting a reduced contribution of hydroxyl or ether groups on the surface. These results indicate that the modification process does not damage the basic structure of LDH but alters its composition and surface chemical environment, particularly by increasing the aromatic carbon content, which may influence interparticle interactions and the material's surface properties.

3.7 Correlation Between Structure and Physicochemical Properties

The modification of Ni–Al LDH with gambier extract reveals a clear correlation among the material's structure, surface chemistry, optical properties, and texture. The LDH structure remains stable, with no significant changes, while interactions with the extract occur predominantly at the surface, as evidenced by involvement of –OH groups and aromatic systems, and by changes in carbon composition. This is accompanied by shifts in electronic properties, as indicated by a decrease in the band gap, suggesting modifications in the electronic environment due to the presence of phenolic compounds. On the other hand, textural changes, such as agglomeration and reduced pore accessibility, suggest that the organic components also influence particle arrangement. These modifications not only preserve the material's

basic framework but also produce a more active and responsive surface, thereby opening opportunities for the development of LDH–biomass in various applications such as adsorption, photocatalysis, and interface-based systems. To reinforce these findings, a comparison with various biomass-based LDH systems from previous studies is presented in Table 1.

4. CONCLUSIONS

Modification of Ni–Al LDH with gambier extract successfully improved the material's physicochemical properties without damaging its underlying structure. Interactions occur primarily at the surface through hydrogen bonding and contributions from aromatic groups, leading to changes in optical properties (decreased band gap), surface chemistry, and morphology (agglomeration and reduced surface area). Although the surface area decreases, the presence of active groups from the extract enhances the material's interaction potential, thereby maintaining its application performance.

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