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Synthesis and Characterization of Layered Double Hydroxides (LDH): Zn-Al-LDH

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Abstract:

This work focuses on the synthesis and characterization of zinc-aluminum-based layered double hydroxides (Zn-Al-LDH). The coprecipitation method was used at a constant pH. The Zn-Al-HDL was analyzed using various techniques, such as X-ray diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), thermogravimetric analysis (TGA/ATD), scanning electron microscopy (SEM), Brunauer-Emmett-Teller (BET) specific surface area, and zero-charge point pH. The results obtained showed a specific surface area of approximately 19.42 m^2/g, a pore volume of approximately 0.77×10^{-2} cm^3/g, a pore diameter of approximately 24.97 nm, a regular crystalline structure, intercalated carbonate anions, a typical lamellar morphology, good thermal stability, and a zero-charge point pH of 7.4.

1. Introduction

Layered Double Hydroxides (LDH), also known as hydrotalcites, are crystalline materials composed of brucite-type $Mg(OH)_2$ layers in which some of the divalent cations (Zn^{2+}) are replaced by trivalent cations (Al^{3+}) (Cavani *et al.* (1991)). This substitution imparts a positive charge to the layers, which is balanced by intercalated anions (typically CO_3^{2-}) and water molecules (Cavani *et al.* (1991). Thanks to their unique structure and high surface area, HDLs are used in various fields such as catalysis, adsorption, and pollutant treatment (Rives *et al.* (2001). The most common synthesis method is precipitation at constant pH (coprecipitation) (Azzaoui *et al.* (2024)). Zinc- and aluminum-based HDLs are among the most studied due to their excellent adsorption properties, low cost, and ease of synthesis.

Double-layered hydroxides (HDLs), also known as anionic clays, have been known for over 160 years, dating back to the discovery of hydrotalcite in 1842 by the mineralogist Hochstetter (Setti, 2014). HDLs constitute a family of compounds formed by a stacking of brucite-type layers ($Mg(OH)_2$) ((Djebbi *et al.* (2017). They exhibit remarkable properties:

- ✓ two-dimensional structure,
- ✓ positive surface charge,
- ✓ high thermal stability,
- ✓ excellent anion exchange capacity (Bony *et al.* (2018).

The general formula is: $[M^{II}_{1-X}M^{III}_{X}(OH)_2]^{X+}[A^{m-}_{X/m}.nH_2O]^{X-}$ (Setti , 2014)

In synthetic materials, the relative proportions of divalent (M^{II}) and trivalent (M^{III}) cations can vary and range between 0.20 and 0.33 (Setti 2014). The value of x is determined by the formula:

$$x = \frac{[MIII]}{([MIII] + [MII])}$$ (Costantino *et al.*, 1991) **Eqn. 1**

M^{II} and M^{III} are the divalent and trivalent cations of the layer, respectively :

divalent metals: Zn^{2+}, Ni^{2+}, Mn^{2+}, Fe^{2+}, Co^{2+}, Ca

trivalent metals: Al^{3+}, Cr^{3+}, Mn^{3+}, Fe^{3+}, Co^{3+}, V^{3+}… ((Bony *et al.* (2018)

and A is the interfacial anionic species (e.g., CO_3^{2-}) ((Djebbi *et al.* (2017).

LDHs have numerous applications, such as in catalysis, air and water purification, medicine, nuclearwaste treatment, etc. (Cavani *et al.* (1991) ; Rives *et al.* (2001) ; Costantino *et al.* (1998) ; Loni (2018). LDHs have numerous applications, such as in catalysis, air and water purification, medicine, nuclear waste treatment, etc. (Cavani *et al.* (1991) ; Rives *et al.*, 2001 ; Costantino *et al.* 1998) ; Loni 2018). The porous structure of Layered Double Hydroxides (LDH) enables efficient transport of ions and molecules, making them widely used materials of choice for applications in the fields of physics, chemistry, optics, and photocatalysis (Chafiq et *al.* (2023a); Chafiq et *al.* (2023b); Chafiq et *al.* (2023)c; Sadik et *al.* (2026). The overall objective of this study is to synthesize and characterize zinc-aluminum-based layered double hydroxides (Zn-Al-LDH) using the constant-pH coprecipitation method. More specifically, the objectives are:

- ✓ To synthesize Zn-Al-LDH;
- ✓ And to characterize Zn-Al-LDH.

2. Materials and Methods

2.1 Materials

- ✓ aluminum nitrate nonahydrate: $Al(NO_3)_3·9H_2O$;
- ✓ Zinc chloride: $ZnCl_2$;
- ✓ Sodium carbonate: Na_2CO_3;
- ✓ Sodium hydroxide: NaOH;
- ✓ Hydrochloric acid: HCl.

2.2 Methods

2.2.1 Preparation of aqueous solutions A and B:

A 100 mL aqueous solution A containing Zn^{2+} and Al^{3+} ions with a Zn/Al molar ratio of 2 is prepared by dissolving 6.815 g of $ZnCl_2$ and 9.38 g of $Al(NO_3)_3 \cdot 9H_2O$ in distilled water. A 100 mL aqueous solution B, consisting of a mixture of NaOH (2 N) and Na_2CO_3 (1 N), is prepared separately and used as a precipitating solution. This protocol is consistent with the coprecipitation procedures widely used for the synthesis of carbonate HDLs (Miyata 1983; Evans *et al.*, 2006).

2.2.2. Synthesis of Zn-Al-CO_3^{2-} -HDL

The [Zn-Al-CO_3^{2-}] phase is obtained by the coprecipitation method while maintaining the pH constant at 9 (Djezzar, 2019). Aqueous solution A of the metal salts is prepared from a mixture of appropriate proportions of zinc chloride $ZnCl_2$ and $Al(NO_3)_3 \cdot 9H_2O$ with a molar ratio of Zn^{2+}/Al^{3+} = 2:1 in 100 mL of distilled water. Aqueous solution B is added at a constant flow rate to aqueous solution A, with vigorous stirring at room temperature for 2 hours. The solution is then vigorously stirred for 6 hours at 60°C following the protocol used by ((Xiang *et al.* (2008). Finally, the reaction mixture is left to stand (without stirring) overnight at room temperature. The supernatant is removed, and the precipitate is washed thoroughly with distilled water several times until the pH of the supernatant is close to that of distilled water. The solid is dried in an oven set to 70°C for 24 hours, ground, and the resulting powder is stored in a plastic bag (Xiang *et al.* (2008).

2.2.3 X-ray Diffraction (XRD)

XRD is a surface analysis technique that allows for the identification of crystalline species present on the surface of materials. The analysis was conducted at the State Key Laboratory of Chemical Engineering at Beijing University of Chemical Technology in the People's Republic of China.

2.2.4 Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR is based on the absorption of infrared radiation by the material being analyzed. By detecting the characteristic vibrations of chemical bonds, it enables the qualitative analysis of the chemical groups present in the material. The analysis was performed at the State Key Laboratory of Chemical Resource Engineering at Beijing University of Chemical Technology in the People's Republic of China.

2.2.5 Scanning Electron Microscopy (SEM)

SEM is used to characterize morphology. The analysis was performed at the State Key Laboratory of Chemical Resource Engineering at Beijing University of Chemical Technology in the People's Republic of China.

2.2.6 Determination of specific surface area, pore diameter, and pore volume

The specific surface area of Zn-Al-HDL was evaluated using the Brunauer–Emmett–Teller (BET) multipoint method (Kiari *et al.* (2022); Konan *et al.* (2020); Koné *et al.* (2021). Pore volume and diameter were determined using the Barrett, Joyner, and Halenda (BJH) adsorption method.

2.2.7 Determination of the pH of the zero-charge point (pHPCN)

To determine the adsorption properties of Zn-Al-HDL, it is useful to know the pH of the zero-charge point, which corresponds to the state of equilibrium between positive and negative charges on the material's surface. The pH of the zero-charge point is determined using a pH titration procedure (Depa, 2000).

The determination involves the following steps:

- ✓ preparation of 0.1 N NaCl solutions at various initial pH values ranging from 2 to 12 by adding 0.1 N HCl or NaOH;
- ✓ mixing 100 mg samples with 25 mL of the above solutions under stirring for 1 hour;
- ✓ The final pH is measured for each solution ;
- ✓ The pHPCN is obtained by plotting the pHf–pHi curve as a function of pHi. The pH_{PCN} is the point of intersection of the plotted curve with the pHi axis.

2.2.8 Thermogravimetric Analysis

This method involves continuously monitoring changes in a sample's mass as a function of temperature. The analysis was performed using a PerkinElmer thermogravimeter operating simultaneously in thermogravimetric analysis (TGA) and differential thermal analysis (DTA) modes. The analysis was performed at the State Key Laboratory of Chemical Resource Engineering at Beijing University of Chemical Technology in the People's Republic of China.

3. Results and Discussion

3.1. X-ray Diffraction (XRD)

The powder X-ray diffraction (PXRD) patterns were obtained using a Rigaku D/Max-2400 X-ray diffractometer with Cu Kα radiation (λ = 1.54056 Å). The XRD pattern of the synthesized Zn-Al-HDL shown in Figure 1 displays the characteristic peaks of the natural hydrotalcite spectrum, notably featuring very high-intensity peaks at diffraction angles 2θ corresponding to the lattice planes (003), (101), (110), and (113) as indexed in the Powder Diffraction Files (PDF). This confirms that the synthesized Zn-Al-HDL exhibits a crystalline structure. The first reflection (plane (003)) is characteristic of the successive stacking of layers along the c-axis. The c-lattice parameter is easily calculated since it is equal to 3c' (where c' corresponds to the sum of the thickness of a brucite ($Mg(OH)_2$) (4.8 Å)) and the interlamellar region, i.e., the inter-reticular distance between the planes of the (003) family) (Lafi *et al.* (2016). The second reflection (101), in the average angular region around 2θ = 40°, allows the determination of the stacking type of the layers (Lafi *et al.* (2016). The intense line located around 2θ = 60° (dhkl = 1.52 Å) is associated with the (110) plane. This reflection is independent of the stacking type of the layers and is used to calculate the lattice parameter a (Djezzar, 2019). The latter corresponds to the distance between neighboring cations within a brucite-type layer and is defined based on the lattice spacing d110 (a = 2×d110) (Lafi *et al.* 2016).

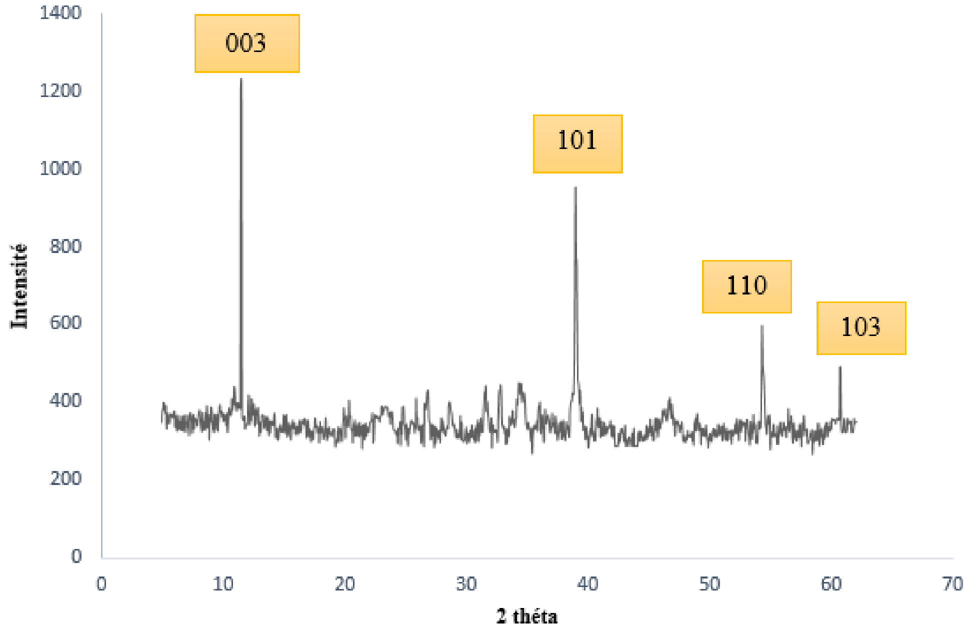

Figure 1. X-ray diffraction of Zn-Al-HDL

3.2. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared (FT-IR) spectra were recorded using KBr pellets with a ThermoFisher 6700 spectrometer. This method allows for the identification of various functional groups, as well as the apparent chemical bonds in Zn-Al-HDL.

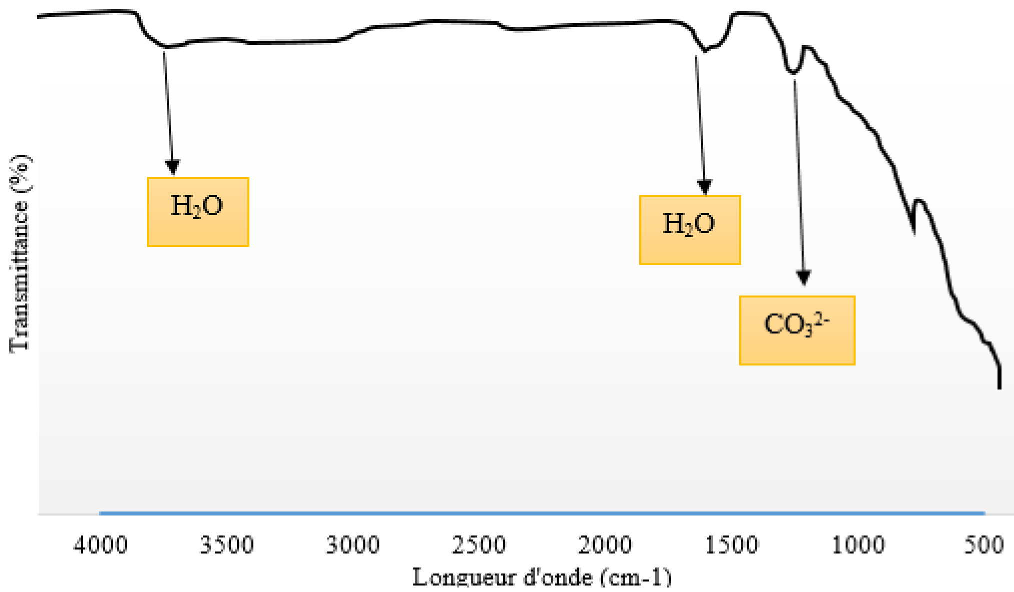

Figure 2. FTIR spectrum of Zn-Al-HDL

Examination of the spectrum reveals the following absorption bands:

- ✓ a broad band around 3600 cm^{-1} is attributed to the valence vibration mode of physisorbed water molecules (Djezzar, 2019).
- ✓ the weak bands centered around 1638–1639 cm^{-1} correspond to the deformation vibration mode of strongly adsorbed water molecules (Djezzar, 2019).

- ✓ very weak bands characteristic of carbonate anions around 1393–1404 cm^{-1} (Djezzar, 2019).
- ✓ The presence of mixed oxides was difficult to detect. Their infrared signature is generally characterized by two broad bands around 1640 cm^{-1} and 1390 cm^{-1}, and a broad region below 500 cm^{-1} with intense bands (υ M–O and M–O–M) (Djezzar, 2019).

3.3. Scanning Electron Microscopy (SEM)

The micrographs obtained by scanning electron microscopy (SEM) were taken using a Hitachi SU8020 microscope, and energy-dispersive X-ray spectroscopy (EDS) analysis of the catalyst surface was performed using a Horiba X-max system. The scanning electron microscopy (SEM) results in Figure 3 clearly show that the synthesized Zn-Al-HDL double-layered hydroxide consists mainly of zinc (yellow), aluminum (red), oxygen (green), and electrons (white).

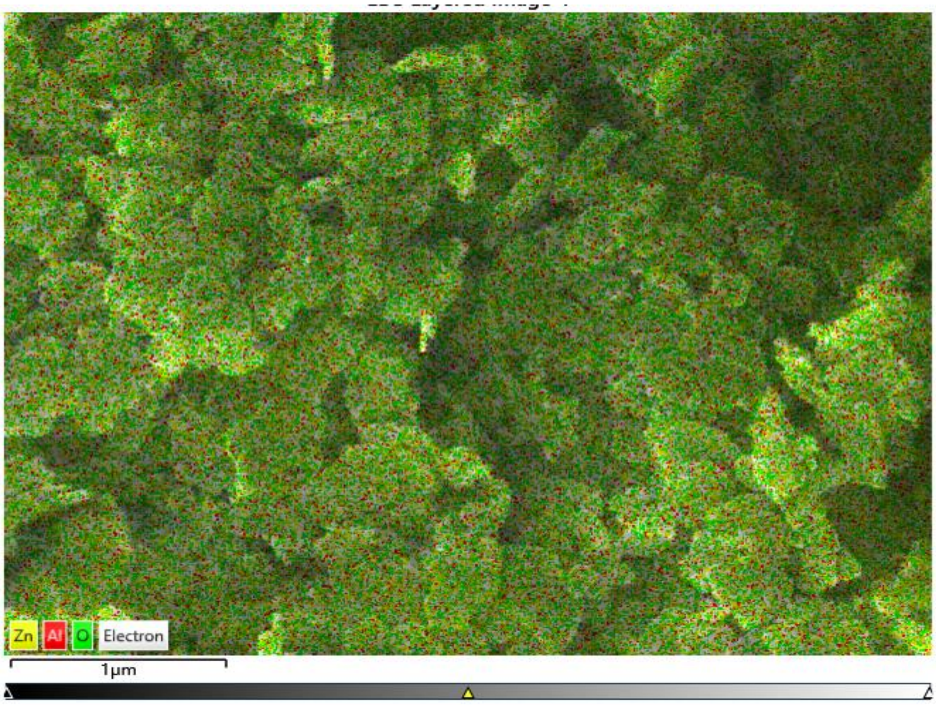

Figure 3. Distribution of Zn, Al, and O in the Zn-Al-HDL material

Figure 4 below shows that the Zn-Al-HDL structure is hexagonal. This confirms the presence of peaks in the XRD pattern. Scanning electron microscopy (SEM) observation in Figure 4 reveals that the resulting material exhibits a predominantly lamellar morphology. The particles appear as small nanoscale platelets (100–500 nm), aggregated into compact clusters. This layered structure is characteristic of lamellar double hydroxides (HDLs), indicating good crystallization of the solid (Bouebdellah , 2022 ; Griffths 2012). The rough and heterogeneous surface, as well as the presence of agglomerates, suggest a large specific surface area and favorable porosity, conferring on the material strong adsorption and catalytic potential (Dussault, 2005; Clark 2018).

3.4. Textural Analysis: Nitrogen Adsorption-Desorption at 77 K (BET Method)

Nitrogen (N_2) adsorption isotherms were recorded using a Micromeritics ASAP 2460 instrument. The pore size distribution was analyzed using the DFT method. The pore characteristics (porosity, specific surface area, etc.) of Zn-Al-HDL were determined by nitrogen adsorption-desorption at 77 K (Figure 5).

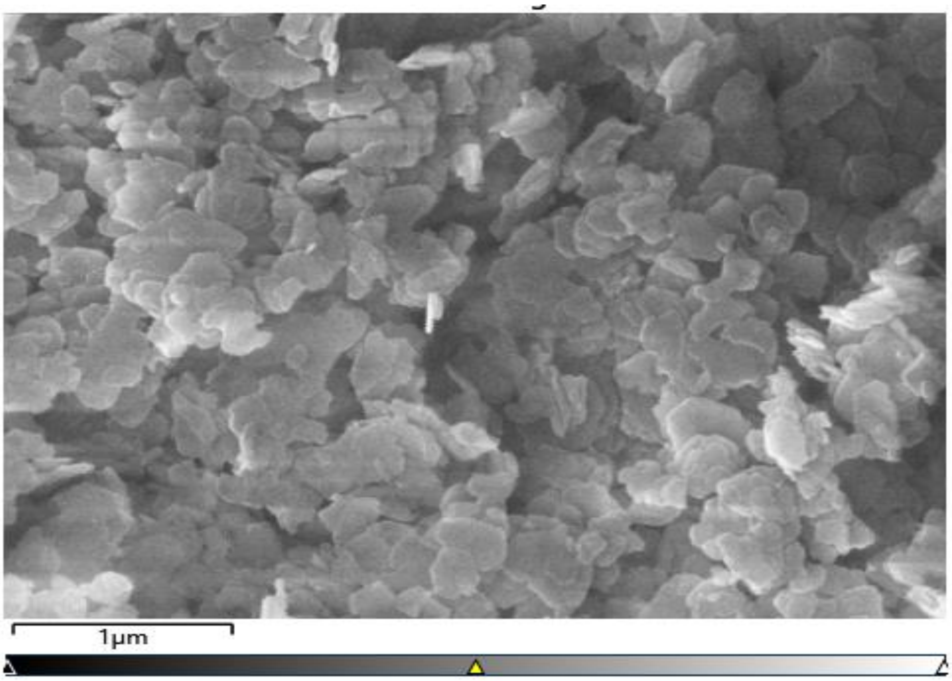

Figure 4. Hexagonal structure of Zn-Al-HDL

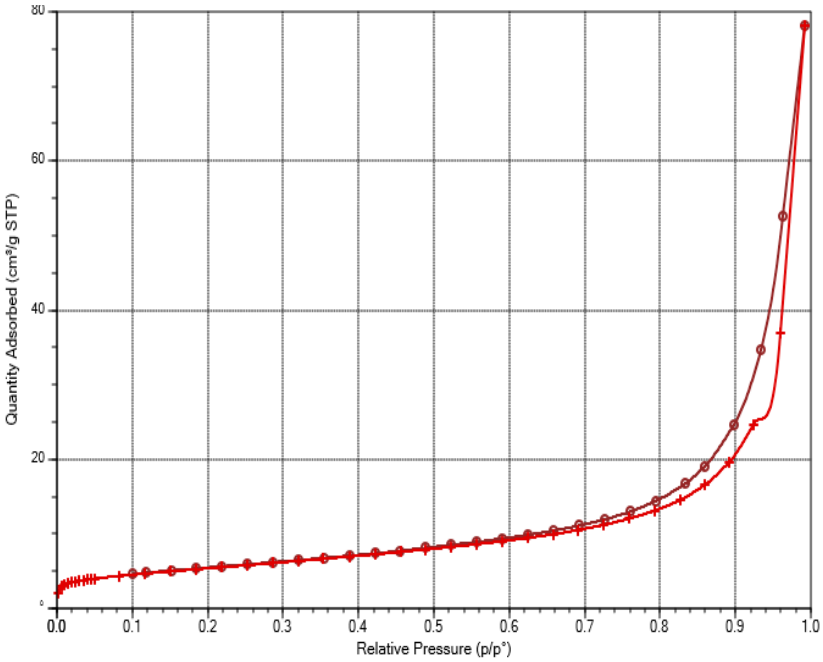

Figure 5. Adsorption-desorption isotherm of Zn-Al-HDL

The shape of the resulting curve is comparable to a Type IV isotherm in the IUPAC (International Union of Pure and Applied Chemistry) classification, with a slight H3-type hysteresis loop. The absence of a plateau at the end of adsorption suggests the presence of platelet- or sheet-shaped particles swelling under the effect of capillary condensation (Lowell *et al.* (2004). These results indicate that the synthesized HDL has a specific surface area of approximately 19.42 m²/g, with the external surface area measured by the so-called t-plot method being on the order of 23.84 m²/g, while the total pore surface area obtained by the Density Functional Theory (DFT) method is 12.77 m²/g (Lowell *et al.* (2004); Thommes *et al.* (2015). These results confirm that adsorption occurs more readily on the first layer than on the surface of the HDL, due to the weak adsorbate-adsorbent interactions compared to the adsorbent-adsorbent (surface-surface) interactions, which reaffirms the macroporous nature of the synthesized HDL (Thommes *et al.* (2015); Brunauer *et al.* (1938). Furthermore, the average adsorption pore diameter, as measured by both the BET and BJH methods, is approximately 24.97 nm and 22.01 nm, respectively, and is greater than the average desorption pore diameter obtained by the two measurement methods (13.67 nm and 21.07 nm, respectively) (Lowell *et al.* (2004); Barrett *et al.*(1951). These values indicate the presence not only of macropores but also of capillary condensation during nitrogen desorption, as evidenced by the fact that in both cases the average desorption diameters are smaller than the adsorption diameters. (Lowell *et al.* (2004)

The total pore volume measured by the DFT method is 12.77 m^2/g, while the maximum volume at P/Po = 0.151236735, obtained by the Horvath-Kawazoe method, is on the order of 0.77×10^{-2} cm^3/g with an average pore size of approximately 0.67 nm (Thommes *et al.* (2015); Horvath *et al.* (1983). These results indicate that the macroporous nature of Zn-Al-HDL is not absolute, since the average diameter of the measured pores ranges from 0.3 to 10 nm, revealing the coexistence of nanopores and macropores (Cavani *et al.* (1991); Rives *et al.* (2001). This can be explained by the fact that the average pore diameter measured by this method corresponds essentially to the pores most accessible to nitrogen. (Lowell *et al.* (2004); Thommes *et al.* (2015)

3.5 Zero-charge point pH : pH_pzc

The zero-charge point pH of Zn–Al–HDL was determined to be pH = 7.4. At pH values below pH_pzc, the material's surface is predominantly positively charged, which promotes the adsorption of anionic species through electrostatic attraction (Figure 6). This behavior explains the effectiveness of Zn–Al–HDL in removing micropollutants present in wastewater, consistent with results reported in the literature (Louni 2018; Dima 2015).

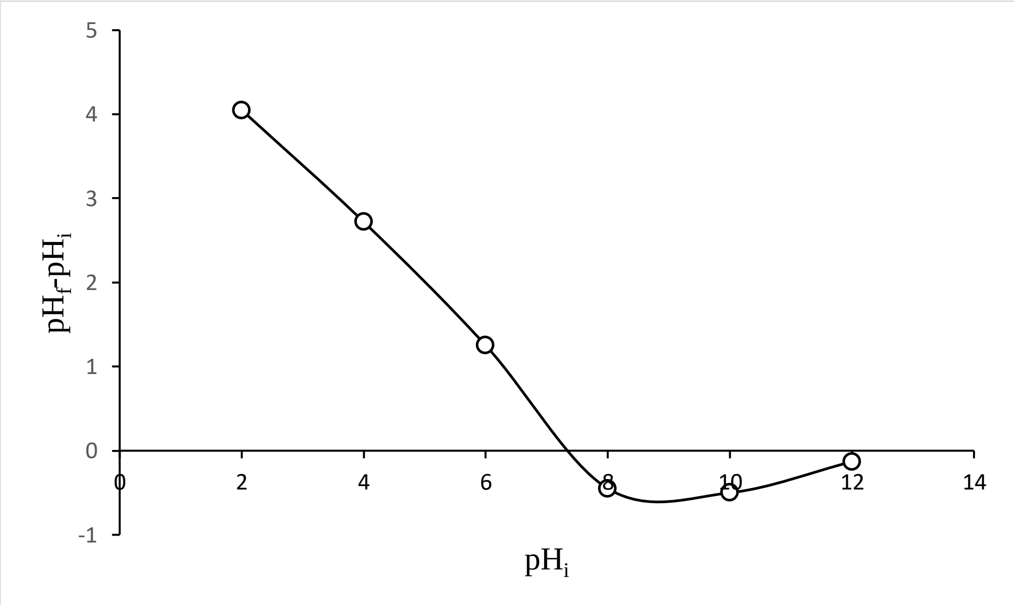

Figure 6. Determination of the zero-load point of Zn-Al-HDL

3.6. ATD/TGA Thermal Analysis

Thermogravimetric analyses (TGA) were performed using an SDTQ600 instrument under an argon (Ar) flow, with a heating rate of 10 °C/min. The ATD/TGA analysis yielded the results shown in Figure 7 below. It reveals five thermal events at 25°C, 150°C, 450°C, 650°C, and 850°C.

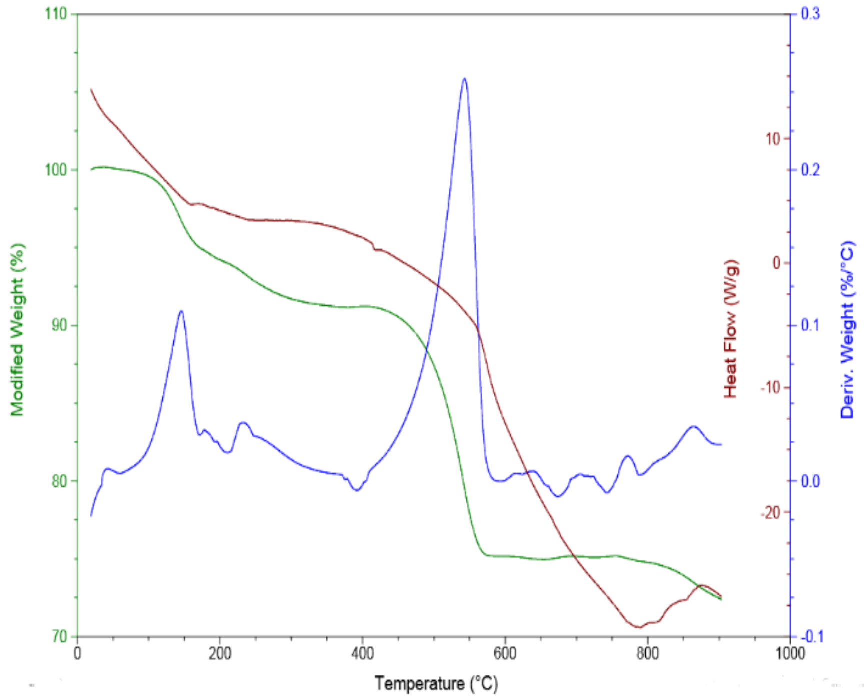

Figure 7. ATD/ATG Analysis Curve

Generally speaking, the change in mass of the HDL phases as a function of temperature is characterized by four (4) stages of mass loss:

- ✓ The first stage, which is endothermic, occurs at low temperatures (25°C to 150°C) and corresponds to the loss of water molecules adsorbed (5%) on the surface and those present in the interlamellar space. This evaporation of water occurs without any collapse of the material's structure (Setti 2014; Garba *et al.* (2026);
- ✓ The second mass loss (5%) occurs at temperatures ranging from 150°C to 450°C and corresponds to the removal of remaining interfacial water and the onset of dehydroxylation, which inevitably leads to the destruction of the structure, resulting in the amorphization of the phase (Setti , 2014);
- ✓ the third stage of mass loss (20%) at temperatures (450°C to 650°C) is due to the decomposition of interlamellar carbonates (Setti, 2014);
- ✓ finally, the mass loss (2%) at temperatures above 650°C results in the material generally decomposing into one or more spinel phases and metal oxides (Setti, 2014).

Conclusion

The aim of this study was to synthesize and characterize zinc-aluminum-based layered double hydroxides (Zn-Al-LDH). The Zn-Al-LDH was characterized using various structural characterization techniques for solids, including XRD, FTIR, SEM, and ATG/ATD. These characterization results show that the Zn-Al-LDH material is highly crystalline with a hydrotalcite structure. The interlamellar space contains carbonate ions and water molecules. The results obtained in this study confirm that the synthesized Zn-Al-LDH layered double hydroxide has a crystalline structure.

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