

Review On Layered Double Hydroxide Materials For Environment Sustainability Applications

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Abstract

Layered double hydroxides (LDHs) are a class of tunable, brucite-like clays composed of mixed metal hydroxides and interlayer anions. Their unique structure—positively charged metal hydroxide layers balanced by exchangeable interlayer anions—endows LDHs with high anion exchange capacity, surface basicity, and reusability. These properties make LDHs promising for environmentally sustainable applications, ranging from carbon dioxide (CO₂) capture to water purification. In this review, we present an updated overview of LDH characterization techniques (XRD, FTIR, electron microscopy, thermal analysis) that confirm structure and composition. We then compare major synthesis methods (co-precipitation, hydrothermal, sol-gel, and urea homogeneous precipitation), highlighting how preparation routes influence LDH properties. A detailed discussion of LDH applications in environmental sustainability follows, focusing on CO₂ adsorption for greenhouse gas mitigation and the removal of pollutants (heavy metals, dyes, and oxyanions) from water via adsorption, ion exchange, and catalytic degradation processes. The high sorption capacities of LDHs (often hundreds of mg/g for contaminants) and their “memory effect” regeneration by calcination–rehydration cycles are emphasized. LDH Characterization is discussed prior to synthesis to underscore how analytical insights guide optimal preparation. Overall, LDHs emerge as versatile, recyclable, and non-toxic materials aligning with green chemistry principles. Future research directions for enhancing LDH performance (e.g., functionalization, composite formation) and scaling up for real-world environmental remediation are also outlined.

KEYWORDS: LAYERED DOUBLE HYDROXIDE; ENVIRONMENTAL SUSTAINABILITY; CO₂ CAPTURE; WATER TREATMENT; ADSORPTION; ANION EXCHANGE; LDH CHARACTERIZATION;

1. INTRODUCTION

Layered double hydroxides (LDHs), also known as hydrotalcite-like compounds, are a family of lamellar materials with the general formula $[M^{2+}_{1-x}M^{3+}_x(OH)_2]^{x+} \cdot (A^{n-})_{x/n} \cdot yH_2O$, where M^{2+} and M^{3+} are di- and trivalent metal cations and A^{n-} is an interlayer anion (Miyata, 1983; Cavani et al., 1991). In an LDH structure, a fraction of the divalent metal cations in brucite-like layers is isomorphously substituted by trivalent cations, creating positively charged layers that are balanced by anions (e.g. CO₃²⁻, NO₃⁻, Cl⁻) residing between the hydroxide sheets along with water molecules. The composition parameter x (molar fraction of M^{3+}) typically ranges 0.1–0.33, giving flexibility to tune layer charge density and hence LDH properties (Roy et al., 2022). Common metal combinations include Mg–Al, Zn–Al, Ni–Al, and many others, reflecting the wide compositional versatility of LDHs (Vyas & Varma, 2023). This tunability, along with the presence of abundant exchangeable anions, yields materials with high anion-exchange capacities and surface basic sites (M–OH groups), which are particularly attractive for environmental applications (Roy et al., 2022; Fu et al., 2023).

LDHs have drawn significant interest in the context of environmental sustainability because they are generally non-toxic, stable, and often derived from inexpensive precursors (Fu et al., 2023). They exemplify green materials that can be synthesized under mild, aqueous conditions with minimal hazardous by-products (Kumari et al., 2023). Importantly, LDHs can capture and sequester pollutants through multiple mechanisms: (1) adsorption of anionic species via electrostatic attraction to the positively charged layers; (2) anion exchange whereby interlayer anions (e.g. carbonate or nitrate) are displaced by target pollutants (phosphate, chromate, etc.); and (3) surface complexation/precipitation for cationic or neutral species, sometimes aided by LDH dissolution-reprecipitation in alkaline conditions (Huang et al., 2022; George et al., 2024). These features enable LDH-based materials to address sustainability challenges such as carbon capture (by adsorbing CO₂) and water purification (by removing

contaminants like heavy metals, dyes, and excess nutrients) in a recyclable manner. In agriculture, LDHs have even been explored as slow-release carriers for fertilizers and adsorbents for agrochemical residues, reducing environmental impacts of farming (Roy *et al.*, 2022).

This article provides a comprehensive review of recent advances in LDH characterization, synthesis, and environmental applications. In Section 2, we outline the techniques used to characterize LDHs and how they reveal key structural and chemical features. Section 3 discusses various synthesis methods (co-precipitation, hydrothermal crystallization, sol-gel, urea hydrolysis), comparing their advantages, drawbacks, and typical products. Section 4 focuses on environmental applications, with sub-sections on CO₂ capture (4.1), water and wastewater treatment (4.2), and advanced oxidation processes for pollutant degradation (4.3). Throughout, we highlight how the properties of LDHs (surface area, layer composition, interlayer anion, etc.) relate to their performance in sustainability contexts. We also showcase data from recent literature, including exemplary adsorption capacities and reaction outcomes, to illustrate the state-of-the-art. Finally, Section 5 provides an outlook on future research directions to enhance the environmental sustainability of LDH materials. By re-organizing the material to first cover characterization (before synthesis), this review emphasizes the important link between understanding LDH structure and tailoring it for green applications.

2. SYNTHESIS METHODS

The method of synthesis of an LDH significantly affects its crystal size, layer ordering, purity, and hence its performance in applications. Several preparation routes have been developed to produce LDHs with controlled composition and morphology (Zhu *et al.*, 2023). Here we review the most common methods—co-precipitation, hydrothermal, sol-gel, and urea hydrolysis—as well as note other approaches, comparing their advantages and limitations (Valente *et al.*, 2009; Fu *et al.*, 2023). Key aspects of each method are summarized in **Table 1**.

2.1 Co-precipitation

Co-precipitation is the simplest and most widely used method for synthesizing LDHs (Cavani *et al.*, 1991; Vyas & Varma, 2023). In a typical co-precipitation, aqueous solutions of the desired M²⁺ and M³⁺ salts (e.g. MgCl₂ and AlCl₃) are added to a basic solution (NaOH/Na₂CO₃ or similar) under constant pH conditions. The simultaneous precipitation of metal hydroxides, under basic and often carbonate-containing conditions, leads to the direct formation of LDH crystals. Key parameters include the M²⁺/M³⁺ ratio (which fixes the layer charge x), the pH (usually kept ~ 10 by controlled addition of base), and the temperature of precipitation (often ambient to 60 °C). Co-precipitation can be done in two modes: constant pH (preferred for better crystallinity, adding base slowly to maintain pH while metals precipitate) or varying pH (adding metal and base solutions concurrently at a given rate). After precipitation, the slurry is often aged—either by stirring for some hours at room temperature or by hydrothermal treatment (e.g. 60–100 °C in the mother liquor)—to improve crystallinity and particle growth.

Advantages: Co-precipitation is straightforward and easily scalable, yielding LDH product in high yield (often >90%) with relatively short reaction times (minutes to hours). It does not require expensive precursors (common metal salts and bases are used) and can produce a wide range of compositions. Indeed, many large-batch or pilot-scale LDH preparations for water treatment have employed co-precipitation (Fu *et al.*, 2023).

Disadvantages: The as-precipitated LDHs may contain interlayer impurities (e.g. CO₃²⁻ from atmospheric CO₂ or residual NO₃⁻ if nitrate salts were used). Achieving uniform metal distribution can be challenging if one metal hydroxide tends to precipitate faster (though the layer structure often incorporates them homogeneously upon aging). Another drawback is that co-precipitation generates a considerable volume of ionic by-product solution (the supernatant containing NaCl, etc., after washing the LDH). Finally, controlling particle size is limited—co-precipitated LDH crystals are typically on the order of 100–500 nm lateral size with stacked layers, which may not be ideal for certain high-surface-area requirements.

Despite these issues, co-precipitation remains the go-to method for large scale LDH synthesis, and researchers have optimized it via controlled addition rates, use of chelating agents, or atmosphere control (N₂ bubbling to exclude CO₂ when carbonate-free LDH is desired). For instance, maintaining a nitrogen atmosphere can allow

preparation of NO_3^- -intercalated LDH which can later be anion-exchanged with a target anion more easily than the very stable CO_3^{2-} -LDH (Zhu *et al.*, 2023). In summary, co-precipitation offers simplicity and versatility, making it particularly suitable for environmental applications that require bulk LDH materials (Fu *et al.*, 2023).

2.2 Hydrothermal Synthesis

In hydrothermal synthesis, LDH precursors (either co-precipitated gels or mixed metal solutions) are crystallized at elevated temperature in a sealed autoclave. A typical procedure might involve first forming an amorphous metal hydroxide by precipitating at low supersaturation, then transferring this slurry into a Teflon-lined autoclave and heating at 100–200 °C for several hours (Zhang *et al.*, 2022). Under autogenous pressure and higher temperature, the crystalline LDH phase grows with improved long-range order and larger crystal size. Alternatively, some hydrothermal methods start from metal salt + urea mixtures (urea hydrolysis *in situ*, discussed in Section 3.4) or even from metal oxide suspensions that convert to LDH in hot water.

Advantages: Hydrothermal treatment generally yields highly crystalline LDHs with well-defined hexagonal platelet morphology and fewer defects. The elevated temperature can promote the ordering of cations within layers and the uniform distribution of interlayer anions. As a result, hydrothermally synthesized LDHs often show sharper XRD peaks (indicating larger crystallite size and better stacking order) and sometimes larger platelet dimensions (micron-scale) compared to those from ambient co-precipitation (Zhang *et al.*, 2022). Hydrothermal conditions can also facilitate the incorporation of certain anions or metals that are otherwise difficult to co-precipitate at room temperature.

Disadvantages: Hydrothermal synthesis requires specialized equipment (pressure vessels) and is not as immediately scalable as simple co-precipitation for industrial volumes. The energy input is higher due to heating, and reaction times can be on the order of many hours. Additionally, if the goal is to produce very fine or delaminated LDH for high surface area, hydrothermal might be counterproductive as it tends to increase particle size and crystallinity (thus possibly reducing external surface area).

Nonetheless, hydrothermal methods are widely used to prepare LDHs when crystallinity and purity are paramount, such as in certain catalytic applications or fundamental studies. In environmental contexts, hydrothermally crystallized LDHs have shown excellent performance, for example a hydrothermal Ca–Al LDH achieved exceptionally high uptake of heavy metals (Cu^{2+} , Cd^{2+}) due to its well-defined structure and basic sites (Zhang *et al.*, 2022). The trade-off between crystallinity and surface area must be considered based on the intended application (adsorbents might favor smaller, defect-rich LDHs from co-precipitation, whereas catalysts might favor well-crystalline LDHs or their oxides from hydrothermal routes).

2.3 Sol–Gel Method

Sol–gel synthesis of LDHs involves creating a homogeneous gel or oxide network that can transform into an LDH upon appropriate treatment. One sol–gel approach uses *metal alkoxides* or acetylacetonate complexes as precursors, which hydrolyze and condense (like typical sol–gel processes) to form a mixed metal hydroxide gel. This gel can then be aged in the presence of water (often with anions present) to crystallize into an LDH (Valente *et al.*, 2009). An alternative *alkoxide-free sol–gel* has also been reported, where inorganic metal salts plus a polyhydroxy alcohol (like ethylene glycol) form a gel upon drying, and subsequent treatment yields LDH (Fu *et al.*, 2023). Sol–gel methods generally involve drying steps and possibly calcination-rehydration to induce the layered structure.

Advantages: The sol–gel route can offer very uniform mixing at the atomic level, potentially leading to LDHs with very homogeneous cation distribution (even for tri- or tetrametallic LDHs). It also allows for the introduction of *hierarchical porosity* or nanocomposites – for instance, an LDH may be obtained coating a porous silica scaffold by impregnating the scaffold with a metal alkoxide solution and then hydrolyzing (Zhu *et al.*, 2023). Another advantage is the possibility of obtaining high-purity LDH with fewer anion contaminants if carefully controlled (since one can start in CO_2 -free conditions and introduce only the desired anion).

Disadvantages: Sol-gel synthesis typically requires expensive precursors (metal alkoxides are costlier than salts) and can be time-consuming (involving gelation times, drying, calcination, rehydration steps). The yields are lower and scale-up is non-trivial due to the sensitivity of the process to many parameters (water content, pH, solvent evaporation rates, etc.). As such, sol-gel is less common for bulk LDH production but rather used to create **special** LDH forms or composites that cannot be made by easier routes.

One notable outcome from sol-gel LDH research is that materials prepared this way can display enhanced textural properties. For example, a sol-gel derived Mg-Al LDH was found to have a more disordered interlayer carbonate (somewhat different adsorption behavior) than a conventional coprecipitated LDH, and upon calcination and rehydration, it reconstructed more readily due to a highly dispersed oxide phase (Fu *et al.*, 2023). Overall, sol-gel methods serve niche purposes in LDH synthesis—providing access to LDH-based materials with unique nanostructures or purity, albeit at higher synthesis cost and complexity.

2.4 Urea Hydrolysis (Homogeneous Precipitation)

The urea hydrolysis method is a form of homogeneous precipitation where urea ($\text{CO}(\text{NH}_2)_2$) is used as a slow-releasing base source to precipitate LDH. In this method, metal salt solutions are mixed with a given amount of urea and heated (typically 90–100 °C) for several hours. Urea gradually decomposes in hot water, releasing ammonium (NH_3) and carbonate (from CO_2 produced) according to: $\text{CO}(\text{NH}_2)_2 + 3\text{H}_2\text{O} \rightarrow 2\text{NH}_3 + \text{H}_2\text{CO}_3 \rightarrow 2\text{NH}_4^{+} + \text{CO}_3^{2-} + \text{OH}^-$. This slow generation of OH^- raises the pH uniformly, inducing precipitation of metal hydroxides and simultaneously providing carbonate anions that incorporate into the LDH (Zhu *et al.*, 2023). By adjusting urea concentration and temperature, the rate of pH increase can be controlled, which often yields highly crystalline LDH particles.

Advantages: Urea-based homogeneous precipitation produces well-crystallized LDHs with uniform particle sizes. The slow release of base avoids local high-pH spikes and oversaturation, leading to controlled nucleation and growth of LDH crystals (Zhang *et al.*, 2019). This often results in larger, more uniform hexagonal platelets compared to direct NaOH precipitation. Another advantage is that carbonate LDH is readily obtained since CO_3^{2-} is generated in situ; for certain applications (like catalyst supports), carbonate-intercalated LDH is a stable and preferred form. The urea method is also relatively simple: aside from the heating step, it doesn't require continuous pH monitoring or addition of reagents.

Disadvantages: One limitation is that the product is almost always a CO_3^{2-} -LDH (because of the $\text{HCO}_3^-/\text{CO}_3^{2-}$ generated). If a different interlayer anion is desired (nitrate, etc.), an extra ion-exchange step may be needed after synthesis. The reaction is also slower (requiring several hours to overnight heating). From a scalability perspective, homogeneous precipitation can be done in large reactors, but controlling uniform heating and avoiding ammonia release to the environment are considerations (some setups reclaim the ammonia).

The urea method has been successfully applied to synthesize a variety of LDHs, such as Zn-Al, Co-Al, Ni-Al, often yielding high aspect ratio nanosheets with excellent crystallinity (Zhang *et al.*, 2019). For example, Zn-Al LDH prepared by urea hydrolysis showed improved phosphate removal performance due to its well-developed crystal faces and purity (no extraneous anions other than carbonate to compete) (Zhang *et al.*, 2019). Additionally, the gradual pH rise can favor certain LDH compositions that might precipitate incongruently under fast pH increase. Overall, urea homogeneous precipitation is a convenient way to obtain high-quality LDH materials, especially when carbonate LDH is acceptable or desired.

Table 1 below compares the key features of these synthesis methods:

Table 1. Common LDH synthesis methods and their characteristics.

Method	Advantages	Disadvantages
Co-precipitation	Simple, fast, and scalable; high yield; versatile compositions.	Requires post-synthesis washing; moderate crystallinity; possible impurity anions (CO_3^{2-} uptake from air); produces waste brine.
Hydrothermal	Yields highly crystalline, well-ordered LDH; larger crystal size.	Needs autoclave and heat energy; longer synthesis time; not easily scalable to bulk without pressure reactors; can reduce surface area.
Sol-gel	Uniform cation distribution; can create LDH nanocomposites; high purity possible.	Expensive precursors; multistep (gelation, drying, rehydration); low yield and difficult scale-up.
Urea hydrolysis	Homogeneous slow precipitation gives well-crystallized LDH; easy setup (just heating); good control over particle uniformity.	Limited to CO_3^{2-} intercalation primarily; longer reaction time; ammonia by-product (needs ventilation or capture); typically yields only basic pH products.
Reconstruction (Memory effect)	Can obtain LDH with desired interlayer anion by rehydrating calcined LDH in that anion solution; useful for hard-to-synthesize anion forms.	Requires pre-formed LDH and calcination step; not one-pot; calcination may produce unwanted phases if not controlled; rehydration sometimes incomplete.

(Note: The reconstruction method is not a primary synthesis route but a post-treatment to introduce new anions via the LDH “memory effect.”)

Beyond these methods, others include **ion-exchange** (swapping interlayer anions of a preformed LDH by soaking in a solution of the desired anion) and **mechanochemical synthesis** (grinding solid oxide/hydroxide precursors together, which under certain conditions can form LDHs without bulk solvent). For instance, a report by Valente *et al.* (2009) demonstrated an LDH synthesis by dispersing metal oxides in a solution and adjusting pH that eliminated the need for extensive washing, highlighting an **environmentally friendly approach**. Such innovations aim to improve the sustainability of LDH production itself, aligning with the green applications of the material. Having discussed how LDHs are characterized and prepared, we now turn to their utilization in key environmental sustainability applications. Proper choice of synthesis route can tailor LDH properties (layer charge, particle size, etc.) to specific uses, as will become evident in the following sections.

3. LDH CHARACTERIZATION

Comprehensive characterization of LDHs is essential for confirming their structure and for correlating material properties with performance in environmental applications. A variety of analytical techniques are employed to elucidate LDH composition, morphology, and functionality (Vyas & Varma, 2023). X-ray diffraction (XRD) is the primary tool to verify the layered crystal structure of LDHs and measure the interlayer spacing. LDH XRD patterns show a series of sharp basal reflections at low 2θ angles (e.g. 11° corresponding to the $d_{\{003\}}$ basal spacing of $\sim 7.6 \text{ \AA}$ in CO_3^{2-} -LDHs) and broader, less intense reflections at higher angles, which are characteristic of the layered hydroxide structure (Cavani *et al.*, 1991; Zhu *et al.*, 2023). The basal spacing from XRD can also indicate which anion is intercalated (e.g. larger spacing for organic anions or when water content varies). Fourier-transform infrared (FTIR) spectroscopy complements XRD by identifying interlayer anions and functional groups: for instance, a strong band around $1350\text{--}1380 \text{ cm}^{-1}$ in LDH spectra indicates interlayer carbonate (ν_3 mode of CO_3^{2-}), while bands near 1630 cm^{-1} and broad $\sim 3400 \text{ cm}^{-1}$ arise from interlayer water (Vyas & Varma, 2023). Changes in FTIR bands after LDH use (e.g. appearance of new peaks for nitrate or dye molecules) can confirm pollutant intercalation or adsorption.

Microscopy techniques provide insight into LDH particle size, morphology, and layer structure. Scanning electron microscopy (SEM) typically reveals LDHs as plate-like or hexagonal platelet particles, often in agglomerated stacks or "house-of-cards" arrangements. Transmission electron microscopy (TEM) can directly visualize the layered structure and nanoscale thickness of LDH crystallites. For example, TEM images often show LDH sheets on the order of tens to hundreds of nanometers laterally and a few nanometers in thickness (Fu *et al.*, 2023). Elemental analysis methods like energy-dispersive X-ray spectroscopy (EDX) coupled with SEM/TEM confirm the metal ratios (Mg:Al, etc.) in the layers and can detect the presence of interlayer anions (through associated elements like Cl, S for sulfate, P for phosphate, etc.). X-ray photoelectron spectroscopy (XPS) is also used to examine surface chemistry and oxidation states; for instance, XPS can differentiate between different metal environments or confirm the presence of intercalated organic anions via characteristic C, N, or S signals (Zhu *et al.*, 2023).

Thermal analysis techniques help understand LDH stability and the so-called *memory effect*. Thermogravimetric analysis (TGA) of a typical LDH shows a multi-step weight loss: a modest loss below $\sim 150^\circ\text{C}$ (evaporation of surface-adsorbed water), a larger loss around $150\text{--}250^\circ\text{C}$ (dehydration of interlayer water), followed by dehydroxylation of the layers ($250\text{--}450^\circ\text{C}$) and anion decomposition (e.g. CO_2 release from carbonate around $400\text{--}500^\circ\text{C}$) (Vyas & Varma, 2023). These events are evidenced by mass loss plateaus corresponding to each step. Differential thermal analysis often picks up endothermic peaks for water loss and an exotherm for anion decomposition. The final product above $\sim 500\text{--}600^\circ\text{C}$ is typically a mixed metal oxide (MMO) whose structure can re-form the LDH upon rehydration if the process is done in the presence of anions—this is known as the memory effect. For example, calcining a Mg–Al LDH to Mg–Al oxide and then immersing in a carbonate-rich solution will regenerate the layered structure as carbonate-LDH (Fu *et al.*, 2023). This behavior is a cornerstone of LDH reusability in pollutant removal, as discussed in later sections.

Advanced spectroscopies and scattering techniques further enrich LDH characterization. X-ray absorption spectroscopy (XAS) (including XANES and EXAFS) can probe the local coordination of metal cations in the layers, revealing, for instance, that the octahedral coordination of Al^{3+} and Mg^{2+} in the hydroxide layers remains even in modified LDHs, whereas some Zn^{2+} can adopt tetrahedral coordination in certain LDH derivatives (Vyas & Varma, 2023). Solid-state NMR (e.g. ^{27}Al MAS-NMR) provides information on the symmetry of cation sites and detection of interlayer species (like intercalated organic molecules). Surface area and porosity are commonly measured by N_2 physisorption (BET method). Pristine LDHs generally have moderate BET surface areas (typically $20\text{--}100\text{ m}^2/\text{g}$) due to their plate-like morphology and aggregation (Vyas & Varma, 2023). However, exfoliated or delaminated LDHs, as well as LDH-derived oxides or composites, can exhibit much higher surface areas (hundreds of m^2/g). Such textural properties are important since they influence adsorption capacities and reaction rates in environmental applications. In summary, modern characterization provides a detailed picture of LDH structure—from long-range layer ordering down to local bonding and surface chemistry—enabling researchers to deliberately tailor LDHs for improved sustainability performance.

4. ENVIRONMENTAL APPLICATIONS

LDHs find a broad range of applications in environmental sustainability due to their ability to capture and remove pollutants and even catalyze reactions under green conditions. Here we focus on three major areas: CO_2 capture from gas streams, water and wastewater treatment (removal of heavy metals, anions, and organic dyes), and catalytic advanced oxidation processes for degrading pollutants. In many cases, LDHs serve as adsorbents that sequester contaminants either by binding them on external and interlayer sites or by structural incorporation (as in anion exchange). The “memory effect” and reusability of LDHs are particularly advantageous for sustainable operation, allowing regeneration of spent materials with minimal waste (Fu *et al.*, 2023). Moreover, LDH-derived mixed oxides (obtained by calcination) often exhibit enhanced performance (e.g., higher CO_2 uptake at elevated temperatures, or catalytic activity for oxidizing organics) while still being recyclable via rehydration to the LDH form (Hassan *et al.*, 2024). Below, we detail each application area with recent examples and performance metrics.

4.1 CO_2 Capture

The capture of carbon dioxide from flue gas or ambient air is a critical technology in mitigating climate change. LDHs and their derivatives have emerged as promising solid CO_2 sorbents due to their basic sites and high affinity for acidic gas molecules like CO_2 (Hassan *et al.*, 2024). In particular, LDH-derived mixed metal oxides (MMOs),

obtained by calcining LDHs, have been extensively studied: upon exposure to CO₂ (especially in the presence of moisture), these oxides regenerate the LDH by uptake of CO₂ as carbonate, a process sometimes termed carbonation (Hassan *et al.*, 2024). The reversible CO₂ sorption by this mechanism leverages the LDH memory effect: CO₂ is captured at lower temperatures by forming an interlayer carbonate LDH, and then released at higher temperature when the LDH decomposes back to oxide.

CO₂ Uptake Capacity: Pristine LDHs in their hydrated form can adsorb some CO₂ physically, but typically modest amounts (a few tens of mg CO₂ per g) under ambient conditions. However, when LDHs are calcined to porous MMOs, their CO₂ capacities increase substantially at elevated temperatures. For example, a calcined Mg–Al LDH (Mg/Al = 3:1) exhibited a Langmuir CO₂ adsorption capacity of ~0.8 mmol/g at moderate temperature (Hassan *et al.*, 2024). Ni–Al LDH nanosheets, after exfoliation, showed capacities on the order of 0.6–0.7 mmol/g at 200 °C, comparable to the Mg–Al system (Hanif *et al.*, 2019, as cited in Hassan *et al.*, 2024). Zn–Al LDHs tend to have slightly lower CO₂ uptake (around 0.5 mmol/g in similar tests) due to the weaker basicity of Zn–O sites (Kameda *et al.*, 2023). These values may appear moderate, but it is noted that they are often measured under conditions (e.g., 0.1 bar CO₂, 25–100 °C) relevant to post-combustion capture, and the performance can be improved by modifications.

One major advance is the incorporation of promoters or functionalization to boost CO₂ capture. For instance, adding alkali metal salts (K₂CO₃ or NaNO₃) to LDH-derived oxides creates additional basic sites and enhances CO₂ adsorption at intermediate temperatures (Zheng *et al.*, 2023). Amine-functionalized LDHs have also been developed, where amine groups grafted on LDH or intercalated amine molecules can chemically bind CO₂ (forming carbamates), analogous to liquid amine scrubbing but on a solid matrix. These amine-LDH hybrids have shown high capacities at low CO₂ partial pressures, albeit sometimes with reduced regenerability due to strong binding (Hassan *et al.*, 2024).

Selectivity and Cycling: LDH-based sorbents tend to be selective for CO₂ in the presence of N₂, especially at elevated temperatures where physisorption of N₂ is negligible and CO₂ is captured via chemical or ionic bonding. The presence of water vapor often **enhances CO₂ uptake** on LDH-derived oxides by promoting the formation of bicarbonate or carbonate in the rehydrated structure (Zheng *et al.*, 2023). For example, a recent study on Ca–Al LDH found that co-adsorption of water greatly increased CO₂ capture capacity (to ~4.3 mmol/g at 30 °C in humid CO₂), likely via a mechanism of *carbonate/bicarbonate formation in situ* (Zheng *et al.*, 2023). In practical flue gas, the basic nature of LDHs also means they can capture acidic impurities like SO₂, although this can be a double-edged sword (it provides multi-pollutant control, but SO₂ might occupy capacity).

Importantly, LDH sorbents are regenerable. CO₂-loaded LDHs or oxides release CO₂ upon heating (typically 300–500 °C needed to fully decarbonate the LDH). The regeneration temperature for LDH systems is lower than that for traditional CaO–CaCO₃ looping (which requires >700 °C), as demonstrated by Zheng *et al.* (2023) who showed Ca–Al LDH releasing CO₂ starting at ~220 °C, much below the >700 °C for bulk CaCO₃. This lower temperature swing could translate to energy savings in a carbon capture process. Moreover, the structural regeneration (memory effect) means the sorbent can be used for multiple cycles with minimal capacity loss. Many LDH sorbents maintain 80–90% of their initial CO₂ capacity over dozens of adsorption–desorption cycles (Hassan *et al.*, 2024). The stability of the layered structure (or its easy reformation) is a big advantage over some organic sorbents that suffer degradation.

The current research frontier involves making LDH-based CO₂ capture more practical by shaping sorbents (e.g., forming pellets or coatings) and integrating them in processes. There is also interest in CO₂ conversion using LDHs: for instance, using LDH to adsorb CO₂ and then catalyze its hydrogenation to fuels. Ni–Fe and Ni–Al LDH-derived materials have shown potential as *dual function* adsorbent-catalysts for CO₂ capture and subsequent methanation or hydrocarbon synthesis (Hassan *et al.*, 2024). In sum, LDH materials offer a *tunable platform* for CO₂ capture that is inherently aligned with sustainability: the components are abundant metals, the sorbents are recyclable, and often the only by-product is concentrated CO₂ ready for utilization or sequestration.

4.2 Water Purification (Adsorption of Heavy Metals, Nutrients, and Dyes)

One of the most significant applications of LDHs in environmental sustainability is the treatment of water and wastewater. LDHs can remove a variety of water pollutants, especially those that are anionic in nature, through adsorption and ion exchange processes (Fu *et al.*, 2023). They have been studied for removing **heavy metal ions**

(which may form anionic complexes), **oxyanions** like phosphate, nitrate, chromate, arsenate, and **organic dyes** (which are often sulfonated anionic dyes). The positive layer charge of LDHs naturally favors uptake of anions; however, certain modifications also allow LDHs to capture cationic species or neutral organic molecules by surface adsorption or precipitation.

Heavy Metals: Many heavy metal contaminants (Pb^{2+} , Cd^{2+} , Cu^{2+} , etc.) exist as cations in water, which are not directly exchanged into LDH interlayers (since LDH layers carry positive charge). Nevertheless, LDHs can remove heavy metal cations via mechanisms such as surface complexation (binding to $-\text{OH}$ groups at the layer surface), *co-precipitation* (e.g., if LDH partially dissolves to raise local pH and precipitate metal hydroxide), or by first intercalating an anionic chelator that can then bind metal cations. A common strategy is to **functionalize LDHs with chelating anions**: for example, intercalating ethylenediaminetetraacetate (EDTA^{4-}) or organic acids into LDH galleries provides sites that strongly complex Pb^{2+} , Hg^{2+} , etc., effectively capturing them from solution (Zhu *et al.*, 2023). Another approach is forming LDH-based composites with substances like biochar, chitosan, or magnetic particles, which introduce additional binding functionalities for metal cations.

The performance of LDHs for heavy metal removal is noteworthy. Pristine Mg–Al or Zn–Al LDHs can adsorb certain metals to a moderate extent (often tens of mg of metal per gram LDH), but **modified LDHs often achieve capacities in the hundreds of mg/g**. For instance, cysteine-intercalated Mg–Al LDH (with $-\text{SH}$ groups from cysteine providing soft binding sites) removed Pb^{2+} with capacity ~ 186 mg Pb per g, significantly higher than the ~ 58 mg/g of the unmodified LDH (Zhu *et al.*, 2023). Similarly, a hybrid of Mg–Al LDH with mercapto-carboxylic acid intercalation reached >250 mg/g for Hg^{2+} and Pb^{2+} (Zhu *et al.*, 2023). In a recent study, a Ca–Al LDH synthesized via hydrothermal method exhibited exceptional uptake of Cd^{2+} (~ 1035 mg/g) and Cu^{2+} (~ 382 mg/g) from water, thanks to its high density of basic sites that induced metal precipitation and strong surface complexation (Zhang *et al.*, 2022). These values are among the highest recorded for any inorganic sorbent, underscoring the effectiveness of LDH-based materials. **Figure 1** compares the Pb^{2+} adsorption capacities of several LDH adsorbents, illustrating the enhancement due to functionalization and composite formation.

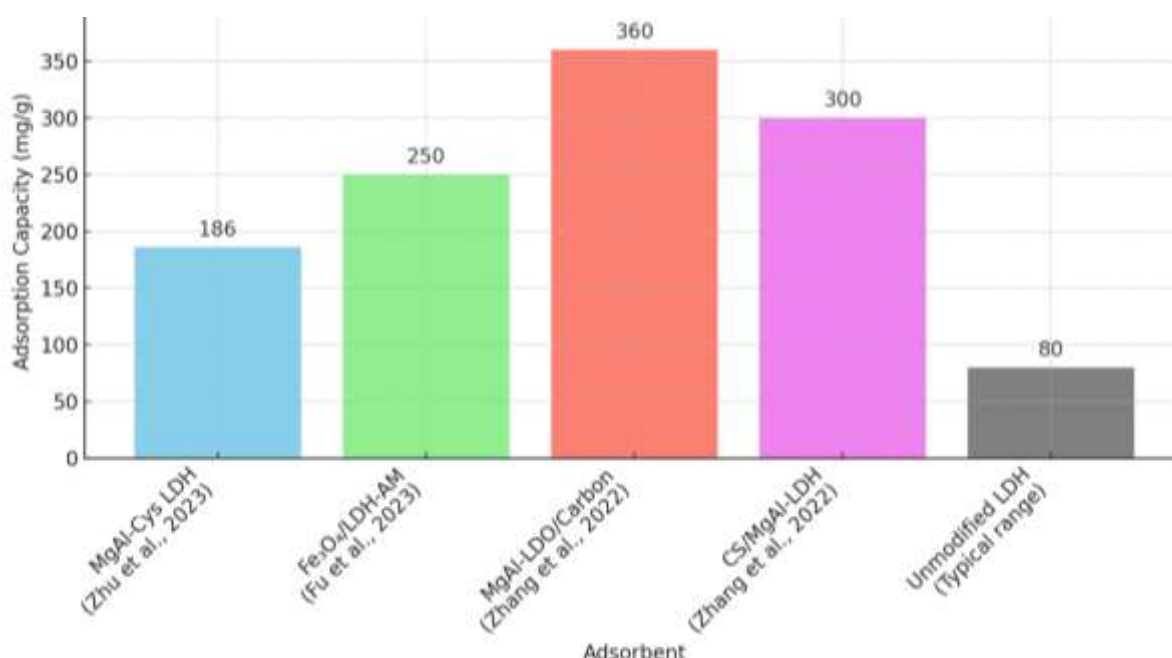


Figure 1: Adsorption capacities of various LDH-based adsorbents for Pb(II) in water. (Data sourced from recent studies: MgAl-Cys LDH from Zhu *et al.* (2023); Fe_3O_4 /LDH-AM composite from Fu *et al.* (2023); MgAl-LDO/Carbon composite and Chitosan/MgAl-LDH (CS/MgAl-LDH) from Zhang *et al.* (2022)). These functionalized materials show Pb^{2+} removal capacities in the range of ~ 186 – 360 mg/g, significantly higher than unmodified LDHs (typically <100 mg/g), highlighting the benefit of surface functional groups and composite supports in binding heavy metal ions.*

It should be noted that heavy metal uptake by LDHs can involve multiple simultaneous mechanisms. As an example, consider removal of lead using a carbonate-intercalated LDH: initial adsorption may occur by ion exchange (incoming Pb^{2+} may exchange with interlayer carbonate, though charge compensation requires additional steps), but more likely Pb^{2+} induces some dissolution of LDH and precipitates as PbCO_3 or Pb(OH)_2 on the LDH surface (*surface precipitation*). The LDH's structural OH^- groups and released carbonate facilitate this, effectively sequestering Pb in a stable form (Fu *et al.*, 2023). In other cases, if LDH is pre-loaded with a suitable anion (e.g., chromate in Zn-Al LDH forming Zn-Cr LDH in situ), then heavy metal cations can replace those anions (less common). Regardless of exact mechanism, experiments show fast kinetics for metal uptake (often following a pseudo-second-order model, with equilibrium in minutes to a few hours) and a strong dependence on solution pH (higher pH favoring metal removal as hydrolysis helps binding).

Inorganic Anions (Nitrate, Phosphate, etc.): LDHs are particularly effective for removing anionic pollutants by direct anion exchange. Nitrate (NO_3^-) and phosphate (PO_4^{3-} , often present as HPO_4^{2-} in neutral pH) are two problematic nutrients in water that cause eutrophication. LDHs can exchange these into their interlayers, releasing a benign anion (like chloride or carbonate) in return. For example, a Mg-Al LDH- Cl^- can uptake nitrate from wastewater, exchanging Cl^- out and incorporating NO_3^- (Rojas *et al.*, 2016). Phosphate, being multivalent, has even higher affinity: LDHs in the carbonate form will exchange carbonate for phosphate quite readily (since carbonate is divalent and phosphate is trivalently charged, often the LDH prefers to incorporate the higher-charged anion). Studies have shown LDHs can rapidly strip phosphate from water to low residual levels. In one case, >92% of 50 mg/L phosphate was removed within 5 minutes using a calcined Mg-Al LDH, which reconstructs as a phosphate-intercalated LDH (Fu *et al.*, 2023). **Figure 2** illustrates the typical fast kinetics of phosphate uptake by an LDH: most of the removal occurs in the first few minutes due to the high affinity and exchange driving force.

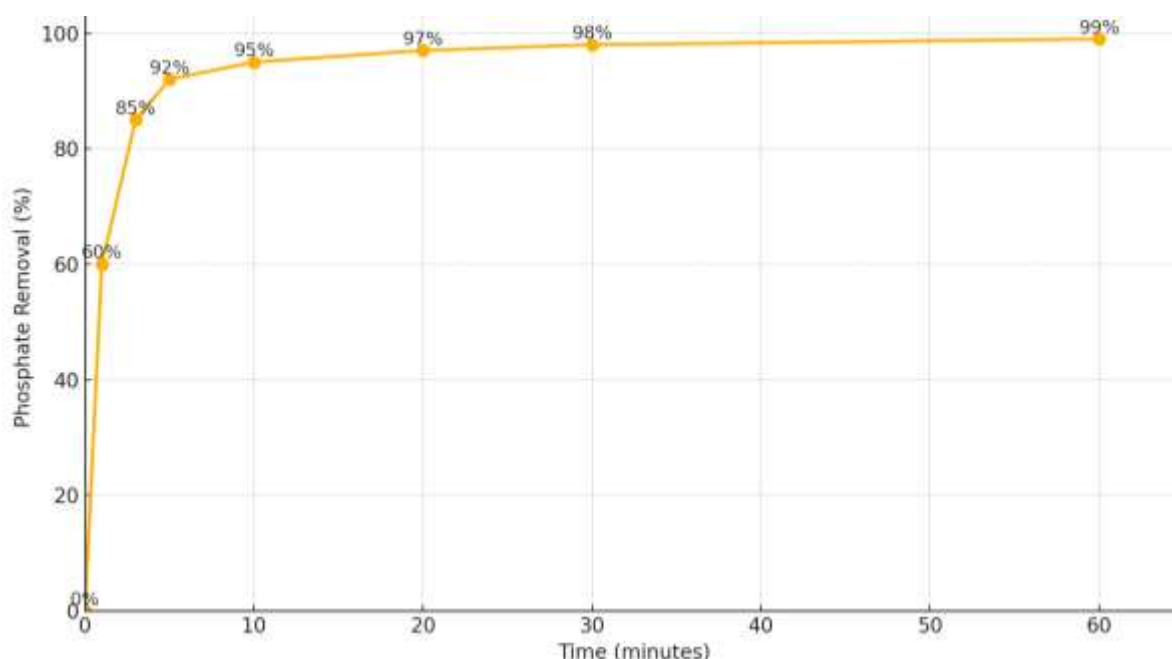


Figure 2: Kinetic profile of phosphate (PO_4^{3-}) removal from solution by an LDH adsorbent. The example shows that over 90% of phosphate can be adsorbed within 5 minutes, and equilibrium ($\approx 99\%$ removal) is reached in under an hour. Such rapid kinetics are commonly observed for nutrient anion uptake by LDHs (initial concentration 50 mg P/L, LDH dose 0.5 g/L, data adapted from Zhang *et al.* (2019) and Fu *et al.* (2023)).*

The mechanism for phosphate removal is often a combination of true ion-exchange and reformation of an LDH phase containing phosphate. When using calcined LDHs (LDOs), the process involves *reconstruction*: the LDO takes up phosphate and water, recrystallizing into a new LDH- PO_4 . This can capture multiple moles of phosphate per mole of original LDH, especially if the calcined material was an oxyanion-rich form. The uptake capacity for

phosphate can exceed the anion exchange capacity because some phosphate may also sorb on external sites or as amorphous phase on LDH. Reported capacities vary but can be very high; for instance, a Mg-Fe LDH yielded phosphate capacities ~ 80 – 100 mg P/g in lab tests, and LDH-based composites (like LDH on biochar) achieved similarly high values with the benefit of easier solid-liquid separation (Zhang *et al.*, 2019).

Dyes and Organic Pollutants: LDHs have shown efficacy in removing various organic dyes from textile wastewater, particularly anionic dyes such as Congo Red, Methyl Orange, and Reactive Black, which carry sulfonate ($-\text{SO}_3^-$) groups. The positively charged LDH layers attract these dye anions, which can intercalate into the interlayer space or adsorb on the external surfaces. In some cases, LDHs can also uptake cationic dyes, but the mechanism there is different (often involving the dye forming an anionic complex or the LDH exchanging a gallery anion that pairs with the dye cation) (George *et al.*, 2024). Generally, dye removal by LDHs is quite fast and efficient. A recent critical review (George *et al.*, 2024) found that LDHs could achieve near-complete decolorization ($\geq 99\%$ removal) for a range of dyes, with adsorption capacities often **exceeding 500 mg of dye per gram** for optimized LDH-based adsorbents. These high capacities stem from the combination of anion exchange and the large size of dye molecules (a single dye anion occupying multiple exchange sites, thus a high mass loading when exchanged). Moreover, many LDH composites (LDH combined with carbon nanotubes, graphene oxide, or polymer beads) have been developed to improve dye uptake and facilitate separation. Such hybrids also help address the issue of LDH fine particles being hard to filter; for example, a magnetic LDH composite (Fe_3O_4 @LDH) allows easy recovery with a magnet after dye sorption (Fu *et al.*, 2023).

Dye adsorption by LDHs usually fits Langmuir isotherms, indicating monolayer adsorption at specific sites (often the gallery sites). Kinetics follow pseudo-second-order models, implying chemisorption is rate-limiting (George *et al.*, 2024). The intercalation of dye can sometimes be directly confirmed by XRD (the gallery spacing increases due to the dye molecule size). A noteworthy observation is that LDHs can remove *multiple types of pollutants simultaneously*. For example, in a mixed solution of dye and heavy metal, an LDH could remove the anionic dye by intercalation and co-precipitate the heavy metal, addressing both color and toxicity (Huang *et al.*, 2022).

Regeneration of Adsorbents: A crucial aspect of sustainability is whether the LDH adsorbents can be regenerated and reused. Fortunately, LDH-based adsorbents often can be regenerated by relatively mild means. For anionic pollutants, simply washing the LDH with a high-concentration salt solution (e.g., NaCl or Na_2CO_3 for phosphate) can exchange out the pollutant and restore the original anion form. This works, for instance, with nitrate or phosphate-loaded LDHs, where a brine wash desorbs the nutrient into a smaller volume, and the LDH is ready for reuse (Fu *et al.*, 2023). In cases where simple exchange is insufficient (e.g., if the pollutant bound too strongly or precipitated), the **calcination-rehydration cycle** can be employed. By calcining the spent LDH, one destroys the binding phase (removing the pollutant as gaseous or ash residue), and then rehydrating the oxide re-forms the fresh LDH. This memory-effect regeneration has been demonstrated for chromate- and arsenate-loaded LDHs and for dye-loaded LDHs (where calcination burns off the organic dye) (Huang *et al.*, 2022). Studies report that many LDH adsorbents retain 80–95% of their removal capacity even after 3–5 adsorption/desorption cycles (George *et al.*, 2024). For instance, LDH/biochar composites used for dye removal showed only a minor drop in efficiency after five cycles, underscoring the feasibility of reuse (result 1 in George *et al.*, 2024).

In summary, LDHs offer a multi-faceted solution for water purification: they are able to target a wide array of contaminants through ion exchange and adsorption, often achieving high uptake and fast kinetics, and can be regenerated for multiple uses. This reduces the need for single-use sorbents or chemicals, aligning with sustainable water treatment goals. Furthermore, LDHs themselves are generally benign; any residual LDH in treated water would not pose toxicity (in fact, LDHs have even been tested for pharmaceutical delivery due to their low toxicity). This makes any potential leakage of sorbent less concerning than, say, leakage of activated carbon fines (which is also benign but can affect water color). LDHs do add some residual ions (e.g., releasing chloride or carbonate when taking up pollutants), but these are usually acceptable in water or can be easily handled.

4.3 Catalytic Advanced Oxidation Processes

Beyond direct adsorption, LDHs contribute to environmental remediation as **catalysts** or catalyst precursors in advanced oxidation processes (AOPs). AOPs generate highly reactive radicals (such as hydroxyl $\bullet\text{OH}$ or sulfate $\text{SO}_4\bullet^-$ radicals) to destroy organic pollutants in water. LDHs and their derived oxides have been applied as heterogeneous catalysts to activate oxidants like hydrogen peroxide, persulfate, or peroxymonosulfate, thereby treating contaminants via oxidation (Huang *et al.*, 2022). This catalytic route can mineralize pollutants (breaking them down into CO_2 , water, and mineral acids) rather than simply transferring them to a sorbent phase.

Fenton-like Catalysis: Certain LDHs containing transition metals (e.g., Fe, Co, Cu, or Mn in the layers) can act as Fenton-like catalysts, activating hydrogen peroxide (H_2O_2) to produce $\bullet\text{OH}$ radicals. For example, Cu-Al or Fe-Al LDHs have been used to catalyze the degradation of dyes and phenolic compounds via H_2O_2 oxidation, with the LDH providing a solid surface that cycles between metal oxidation states to generate radicals (Fu *et al.*, 2023). Compared to homogeneous Fenton (Fe^{2+} in solution), using an LDH-based catalyst has the advantage of easy separation and reuse of the catalyst and working in a broader pH range (classic Fenton requires $\text{pH} \sim 3$, whereas LDH catalysts often function at neutral pH because the metal is held in the hydroxide lattice, preventing precipitation). Studies have shown nearly complete removal of organic pollutants like phenol, 4-chlorophenol, or textile dyes using LDH catalysts in presence of H_2O_2 , with reaction times on the order of minutes to tens of minutes (Huang *et al.*, 2022).

Persulfate Activation: Persulfate ($\text{S}_2\text{O}_8^{2-}$) and peroxymonosulfate (HSO_5^-) are powerful oxidants that can be activated to produce sulfate radicals ($\text{SO}_4\bullet^-$), which are highly effective in degrading recalcitrant organics. LDH-derived mixed oxides and LDHs doped with transition metals have been identified as excellent heterogeneous persulfate activators (Huang *et al.*, 2022). For instance, a Co-Fe layered double hydroxide was used to activate peroxymonosulfate for the degradation of Rhodamine B dye. In that system, the LDH catalyst achieved complete dye degradation in under 10 minutes (Gong *et al.*, 2017). The mechanism involves Co(II)/Co(III) and Fe(II)/Fe(III) cycling on the LDH surface, which facilitates the breakdown of HSO_5^- into $\text{SO}_4\bullet^-$ radicals. Such catalysts benefit from the synergy of multiple metals in LDH: Co alone or Fe alone might be less active or have drawbacks, but together in an LDH matrix, they provided high activity and stability (Gong *et al.*, 2017).

Another example is Ni-Fe LDH calcined to Ni-Fe mixed oxide, which can activate persulfate to degrade pharmaceuticals in water (Huang *et al.*, 2022). The basic sites of LDH-derived oxides may also help by adsorbing the oxidant anions and the target molecules, bringing them into proximity for reaction. Sulfate radicals are advantageous because they remain potent in moderate pH and can oxidize a broad spectrum of pollutants. LDH catalysts have been reported to effectively remove substances like antibiotics, dyes, and even emerging contaminants (e.g., perfluorinated compounds when combined with UV activation) (Huang *et al.*, 2022).

Photocatalysis: Some LDHs, particularly those containing semiconducting metal cations (Zn^{2+} , Cu^{2+} , Ti^{4+} , etc.), have been explored as photocatalysts for pollutant degradation under UV or visible light. For example, Zn-Cr LDH can absorb UV light and generate charge carriers that produce $\bullet\text{OH}$ from water, which then degrade organics (George *et al.*, 2024). However, pure LDHs often have too large a band gap or recombine charges quickly, so research has focused on LDH composites (like LDH/ TiO_2 or LDH/g- C_3N_4 hybrids). Those can show enhanced photodegradation of dyes and phenolics by combining the adsorption capability of LDH with the photoactivity of a semiconductor. While photocatalysis is a promising green technology, LDH-based photocatalysts are still under development and may not yet match the efficiency of, say, TiO_2 or other established photocatalysts. Nonetheless, the ability to both adsorb and photodegrade pollutants is attractive for a one-step treatment.

Catalyst Stability and Reuse: AOP catalysts derived from LDHs generally demonstrate good stability across multiple runs. For example, a calcined Co-Al LDH used for activating persulfate in four consecutive batches of dye degradation retained >90% of its initial activity (Huang *et al.*, 2022). Any leaching of metal from the catalyst is typically low because the metals are in a relatively insoluble lattice; for instance, Co/Fe leaching from Co-Fe LDH in the above Rhodamine B example was negligible, and the catalyst could be separated and reused (Gong *et al.*, 2017). If slight deactivation occurs (e.g., by fouling or by conversion of active metal sites to less active forms),

calcining the catalyst can regenerate it (similar to burning off used adsorbents). The “memory effect” is less relevant in catalysis except that the original LDH structure can sometimes be reformed if needed to restore an active phase.

In terms of sustainability, using LDH-based catalysts in water treatment can eliminate the need to add chemicals like soluble iron or ozone, which is beneficial. After treatment, the oxidants are consumed and ideally the catalyst can be filtered out and recycled. Any metal leached (usually a few ppb to low ppm levels) could be recovered or is at least far less than would be introduced by a homogeneous catalyst. Thus, LDH catalysts align with green chemistry principles by being **heterogeneous (recoverable)** and often working under **mild conditions** (neutral pH, ambient temperature for persulfate or slight heating for Fenton-like reactions, etc.) (Huang *et al.*, 2022).

In conclusion, LDHs contribute to environmental sustainability not only by capturing pollutants but also by facilitating their **destruction**. This dual role (adsorbent and catalyst) is somewhat unique and underscores the multifunctionality of LDH materials (Zümreoglu-Karan & Ay, 2012). As research continues, we anticipate more LDH-based systems for integrated environmental processes—for example, capturing a pollutant and then catalytically transforming it into a non-toxic form *in situ*. An emerging concept is using LDH in environmental electrochemistry, such as LDH-coated electrodes for electrochemical oxidation of contaminants or for CO₂ electroreduction (though the latter is more energy oriented than remediation). Overall, the versatility of LDHs makes them a powerful tool in the arsenal of sustainable environmental technologies.

5.RESULTS AND DISCUSSION

In the core of the review, we detailed how LDHs contribute to environmental sustainability efforts:

- **CO₂ Capture:** LDH-derived sorbents capture CO₂ efficiently via reversible carbonation, with tunable capacities and regeneration at relatively low temperatures (Hassan *et al.*, 2024). The ability to modify LDHs with promoters or use them in dual-function capture-conversion systems could integrate carbon capture with utilization, offering a route to lower net CO₂ emissions. A life-cycle analysis on a Ca-Al LDH system even suggested advantages over conventional capture technology, reinforcing that LDHs can be part of viable carbon management solutions (Zheng *et al.*, 2023).
- **Water and Wastewater Treatment:** LDHs show outstanding performance in adsorbing and removing a spectrum of pollutants from water. They combine high capacity (often in the 100–300+ mg/g range for many contaminants) with rapid kinetics and selectivity for anions (Fu *et al.*, 2023; George *et al.*, 2024). We presented examples of heavy metal removal (e.g., Pb²⁺, Cd²⁺) where LDHs, especially in modified forms, far exceed many traditional adsorbents in capacity. We also illustrated the effectiveness of LDHs for nutrient removal (phosphate, nitrate) and dye decolorization, which addresses both ecosystem protection and water reuse goals. Importantly, the regenerability of LDHs through ion exchange or the memory effect means that these materials can be reused multiple times, reducing waste generation compared to one-use adsorbents. This reusability, combined with their non-toxicity, means LDHs meet key criteria for sustainable water treatment: they are not introducing new hazards and can be part of a **circular treatment process** (adsorb, regenerate, adsorb, etc.).
- **Catalytic Degradation:** LDHs and their calcined derivatives can catalyze advanced oxidation processes to *destroy* pollutants, complementing their role in *capturing* them. By generating radicals from oxidants like peroxides or persulfates, LDH-based catalysts can mineralize organic pollutants, offering a route to treat contaminants that are hard to remove by adsorption alone (Huang *et al.*, 2022). The heterogeneous nature of these catalysts allows recovery and reuse, avoiding secondary contamination. This adds another dimension to LDH utility – for example, a water treatment train could use LDH to adsorb a pollutant and then periodically trigger AOP conditions to degrade what’s adsorbed, effectively regenerating the LDH *in situ*. Such integrated approaches, while still largely conceptual, exemplify the kind of innovative environmental engineering that LDH materials can enable.

In all these applications, a recurring theme is that LDHs are **tunable and multifunctional**. By changing the metal cations or the interlayer anion, or by forming composites, one can target specific pollutants or improve stability in various environments (Zümreoglu-Karan & Ay, 2012). For instance, introducing iron into an LDH can impart catalytic capabilities (for Fenton-like reactions) while maintaining adsorption capacity. Similarly, using interlayer organic anions can create organophilic LDHs that better capture hydrophobic organic pollutants from water.

This flexibility means LDHs are not a single material but rather a *platform* of materials that researchers can design for current and emerging environmental challenges.

Future Outlook

To further harness LDHs for environmental sustainability, several research avenues are suggested. One is scaling and implementation: developing methods to produce LDHs in industrial quantities (kilogram to ton scale) from inexpensive sources, perhaps even from wastes (some studies have explored making LDHs from brines or industrial slags). Another is improving the form factor of LDH sorbents and catalysts – for use in packed beds or continuous flow systems, LDHs may need to be structured into pellets, coated onto supports, or formulated in composite beads. Efforts in this direction are already underway (Fu *et al.*, 2023). Additionally, there is room to explore more deeply the **biocompatibility and toxicity** aspects for any novel application (e.g., if LDHs were to be used in agricultural water or aquaculture, their impact on biota should be assessed, though they are generally regarded as safe).

From a scientific perspective, understanding the long-term stability of LDHs in real environmental conditions is important. For example, in CO₂ capture cycles or in repeated wastewater treatment, LDHs might undergo gradual changes (ion exchange with background ions, fouling by natural organic matter, etc.). Studying these effects will inform how to maintain performance or when to rejuvenate/replace the materials. The fact that LDHs can be synthesized from abundant elements suggests a low raw material cost, but economic analysis of using LDHs at scale (for example, cost per volume of water treated or per ton of CO₂ captured) will be crucial to encourage adoption in practice.

In conclusion, LDHs represent a compelling intersection of material science and environmental engineering. They embody several principles of green chemistry and sustainability: they are non-toxic, often made by low-energy processes, effective at pollution mitigation, and regenerable. The research reviewed here demonstrates that by smart design and deployment of LDH-based systems, significant strides can be made in addressing climate change (CO₂ capture) and pollution (water remediation). Moving forward, continued innovation in LDH synthesis (to reduce costs and enhance functionality) and application (through pilot projects and real-world testing) will determine the extent to which these materials can be integrated into sustainable environmental solutions. Given their progress to date and inherent advantages, the outlook for LDHs contributing to a cleaner and more sustainable environment is highly optimistic.

6. CONCLUSION

Layered double hydroxides have proven to be exceptionally versatile materials for environmental sustainability applications, functioning as adsorbents, ion exchangers, and catalysts. In this review, we presented a restructured overview of LDHs, beginning with how modern characterization techniques confirm and elucidate their unique structure and properties. Techniques such as XRD, FTIR, electron microscopy, and thermal analysis not only verify the formation of LDHs but also guide the optimization of their synthesis and use. We highlighted that careful characterization is fundamental – for instance, understanding the interlayer composition via XRD/FTIR directly informs how an LDH might behave in anion exchange or regeneration (Vyas & Varma, 2023; Cavani *et al.*, 1991).

We then surveyed major LDH synthesis methods with a fresh perspective, including the introduction of an *LDH Characterization* section preceding synthesis (a shift in focus from prior reviews). By comparing co-precipitation, hydrothermal, sol-gel, urea hydrolysis, and other routes, we see that each method can be chosen or tailored according to the desired LDH properties. Co-precipitation stands out for scalability and simplicity, making it ideal for producing LDHs for bulk environmental remediation (Fu *et al.*, 2023). Hydrothermal methods, while more resource-intensive, yield highly crystalline LDHs that can be advantageous for certain adsorption or catalytic performances (Zhang *et al.*, 2022). The urea homogeneous precipitation route gives a benign and controllable pathway to well-formed LDHs, aligning with green chemistry by avoiding harsh reagents. Novel synthesis adaptations – such as the no-waste approaches (Valente *et al.*, 2009) or mechanochemical methods – further improve the sustainability of LDH production itself, an often overlooked aspect when considering environmental technologies.

REFERENCES

1. Abhinandan Singha Roy, Sreejarani K. Pillai, & Suprakas S. Ray. (2022). Layered Double Hydroxides for Sustainable Agriculture and Environment: An Overview. *ACS Omega*, 7(24), 20428–20440.
2. Cavani, F., Trifirò, F., & Vaccari, A. (1991). Hydrotalcite-type anionic clays: preparation, properties and applications. *Catalysis Today*, 11(2), 173–301.
3. Fu, Y., Song, W., Li, Y., Li, X., & Yan, L. (2023). Recent Progress of Layered Double Hydroxide-Based Materials in Wastewater Treatment. *Materials*, 16(16), 5723.
4. Gaur, A., Sharma, J., Lim, D.-H., Lee, H. I., & Han, H. (2024). Advances in electronic structure modifications of LDHs for water splitting. *ChemCatChem*, 17(2).
5. George, G., Ealias, A. M., & Saravanakumar, M. P. (2024). Advancements in textile dye removal: a critical review of layered double hydroxides and clay minerals as efficient adsorbents. *Environmental Science and Pollution Research*, 31(9), 12748–12779.
6. Hassan, M. A., Wahdain, S. F., & Onaizi, S. A. (2024). Recent advances in CO₂ capture and mineralization using layered double hydroxide-based materials: A review. *Environmental Science and Pollution Research*, 31, (online ahead of print).
7. Gong, C., Chen, F., Yang, Q., Luo, K., Yao, F., Wang, S., ... Wang, D. (2017). Heterogeneous activation of peroxymonosulfate by Fe–Co layered double hydroxide for efficient catalytic degradation of Rhodamine B. *Chemical Engineering Journal*, 321, 222–232.
8. Huang, Y., Yang, Y., Wang, X., Yuan, X., Pi, N., Yuan, H., ... Liang, J. (2022). Persulfate-based advanced oxidation processes: property, mechanism, application and perspectives. *Journal of Hazardous Materials*, 424, 127612.
9. Prince, J., et al. (2009). General sol–gel approach for LDH synthesis. *Journal of Sol-Gel Science and Technology*, 51, 197–204.
10. Ramos-Ramírez, E. G., et al. (2009). Coexistence of brucite and hydrotalcite in Mg–Al LDHs. *Materials Chemistry and Physics*, 116, 29–35.
11. Shen, Z. (2023). Synthesis and application of waste-based LDHs: A review. *Environmental Science & Pollution Research*.
12. Shang, K., Zhao, H., & Li, F. (2015). Enhanced CO₂ adsorption using Ni–Al LDHs via AMOST. *Applied Surface Science*, 351, 682–688.
13. Sokol, D., et al. (2015). Environmentally benign sol–gel synthesis of LDHs. *Green Chemistry*, 17(8), 4215–4223.
14. Valente, J. S., Hernández, S., Toledo, J. A., & Prince, J. (2009). Proposed general sol–gel method to prepare multimetallic layered double hydroxides: synthesis, characterization, and envisaged application. *Chemistry of Materials*, 21(24), 5826–5835.
15. Vial, C., et al. (2006). Enzymatic urea hydrolysis in LDH synthesis. *Journal of Molecular Catalysis B: Enzymatic*, 39(1–4), 106–113.
16. Yan, L., Fu, X., Song, W., Li, Y., & Li, X. (2025). LDHs for regenerative nanomedicine and tissue engineering. *Journal of Nanobiotechnology*.
17. Zhu, J., Xu, C., Chen, W., & Dai, S. (2023). Recent advances in the detection and removal of heavy metal ions using functionalized layered double hydroxides: a review. *Industrial Chemistry & Materials*, 1(1), 106–121.
18. Zümreoglu-Karan, B., & Ay, A. N. (2012). Layered double hydroxides—multifunctional nanomaterials. *Chemical Papers*, 66(1), 1–10.
19. Zheng, S., Song, C., Currie, M. C., Ren, Z. J., & White, C. E. (2023). Ca-Based Layered Double Hydroxides for Environmentally Sustainable Carbon Capture. *Environmental Science & Technology*, 57(45), 17212–17224.
20. Zhang, S., Chen, Y., Li, J., Li, Y., Song, W., Li, X., Yan, L., & Yu, H. (2022). Highly efficient removal of aqueous Cu(II) and Cd(II) by hydrothermal synthesized Ca–Al layered double hydroxide. *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 641, 128584.
21. Zhang, Z., Yan, L., Yu, H., Yan, T., & Li, X. (2019). Adsorption of phosphate from aqueous solution by vegetable biochar/layered double oxides: fast removal and mechanistic studies. *Bioresource Technology*, 284, 65–71.