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LAYERED DOUBLE HYDROXIDES: INSIGHTS INTO THE STRUCTURAL REGENERATION BY MEMORY EFFECT

BY

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Abstract. Layered double hydroxides (LDHs) are a unique class of inorganic compounds, presenting positively charged layers arranged in a brucite-like structure. These materials are highly versatile due to their ability to host various anions in the interlayer spaces and accommodate diverse cations in the layers. As a result, LDHs have broad applications in fields like drug delivery, water purification, and catalysis. A key characteristic of LDHs is their structural “memory effect,” which allows them to restore their original structure after calcination. Factors such as temperature, pressure, pH, and the specific anions and cations involved significantly affect the properties of LDHs during regeneration. This work provides an in-depth review of the properties and uses of LDHs regenerated through the memory effect, offering insights into how their physical-chemical features can be tailored.

Keywords: Layered double hydroxide, mixed oxides, structural recovery, memory effect.

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1. Introduction

In recent years, layered double hydroxides (LDHs), a class of inorganic materials characterized by a layered structure, have garnered the attention of researchers due to their intriguing characteristics and compositional diversity.

Also known as hydrotalcites, LDHs are a class of anionic clays consisting of brucite-like layers (Mascolo *et al.*, 2015). Their positively charged layers are composed of $M(OH)_6$ octahedra, which are balanced by anions and water molecules hosted in the interlayer space (Xu *et al.*, 2018). The octahedral structure consists of a central divalent or trivalent metal cation coordinated with six hydroxyl groups (OH^-). Each hydroxyl group is connected to other metal cations (Ma *et al.*, 2012). Adjacent octahedra share edges, forming a continuous two-dimensional layer known as the primary layer of the structure.

These cations, which share the edges of the octahedra along with anions and water molecules in the inter-layer spaces, are in equilibrium, resulting in a unique symmetrical laminar structure (Wang *et al.*, 2023).

The structure of LDHs and the typical octahedral unit is shown in Fig. 1. The general formula of LDHs is $[M^{2+}_{1-x}M^{3+}_x(OH)_2]^{z+}[A^{n-}]_{z/n} \cdot mH_2O$, where:

M^{2+} and M^{3+} are divalent and trivalent metal cations located in the host layers. In this context, the molar ratio between M^{2+}/M^{3+} cations range from 2.0 to 4.0. A^{n-} represents the exchangeable anions located in the interlayer region, that compensate for the positive charge developed in the layers (Xu *et al.*, 2018).

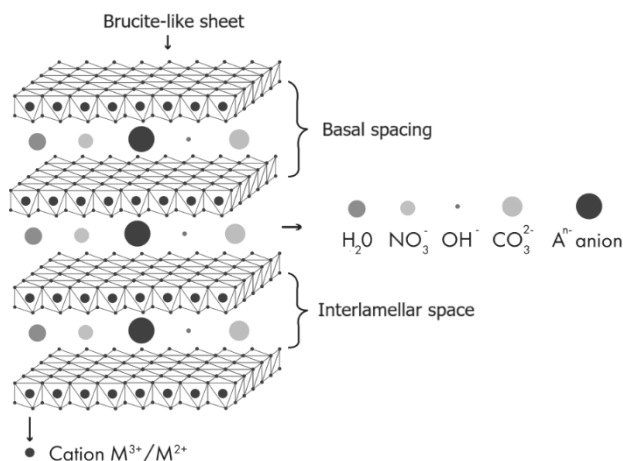


Fig. 1 – The structure of layered double hydroxides.

Moreover, the various combinations of M^{2+} and M^{3+} cations in LDH host sheets demonstrated that a large diversity of $M^{2+} - M^{3+}$ LDHs can be obtained for

the composition of the layers ($M^{2+} = Mg^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Zn^{2+}$ etc.; $M^{3+} = Al^{3+}, Fe^{3+}, Cr^{3+}, Ga^{3+}$ etc.) (Ma *et al.*, 2012).

The structure of LDHs is influenced by the valence state of the metal cations, the type and arrangement of intercalated anions, and the distance between the layers. The chemical properties of LDHs also depend on this structure. Therefore, evaluating the relationship between structure and properties is crucial for the further development of LDHs (Wang *et al.*, 2023).

Due to their ease of preparation, low cost, non-toxic nature, and flexible composition (Lu *et al.*, 2024) LDHs are utilized in various fields, including drug delivery, water decontamination, and catalysis (Peng *et al.*, 2018).

These materials also possess other properties that have been extensively studied by researchers, particularly regarding the incorporation of different species into their structures, especially in the context of drug delivery (Pandey *et al.*, 2022). For instance, the intrinsic properties of LDHs, such as compositional diversity, high stability, large surface area, customizable nanoporous structure, and their 2D layered arrangement make them suitable for use as catalysts. However, limitations related to particle size, morphology, and crystalline orientation can impact the effectiveness of LDHs in absorption processes, highlighting the need for methods to enhance their absorption performance (Lu *et al.*, 2024).

Furthermore, LDHs exhibit further notable characteristics, such as stability, profitability and anion exchange capacity, which stem from their unique 2D layered structure. Due to these attributes LDHs can be utilized in various technologies alone or in hybrid forms with other nanounits, as composites (exfoliated in a polymer matrix) or as modified scaffolds (intercalated with functionalized materials) (Kumari *et al.*, 2023).

This work presents recent findings regarding a notable feature of LDHs, which is the subject of recent research (Jin *et al.*, 2022): their structural regeneration via valorisation of the "structural memory effect" and some of the parameters modulating the restoration of the LDH structures. Further, this study covers essential insights in the future valorisation of the multifunctionality of the regenerated structures of LDH-type materials and their applications.

2. Structural memory effect of LDHs

There are several facile and inexpensive procedures for preparing LDHs. These include the variable and constant pH coprecipitation methods, the calcination-reconstruction process, the sol-gel route, urea hydrolysis, and ion exchange (Benhiti *et al.*, 2023).

During the calcination process, the structural recovery of LDHs is accompanied by the decomposition of anions and water molecules located between the layers. This decomposition results in the transformation of LDHs

into layered double oxides denoted here as LDOs (Ye *et al.*, 2022). The recovery of LDH structure by the “memory effect” involves introducing of LDOs into aqueous solution containing anions or to humid air. This exposure triggers a structural reconstruction reaction, which is crucial for restoring the original layered structure by enabling the absorption of foreign anions and water molecules. This phenomenon is known as the "structural memory effect" of LDHs (Zhang *et al.*, 2022) and it refers to the ability of LDHs to incorporate foreign anions and water molecules into their layers while regenerating their layered structure (see Fig. 2).

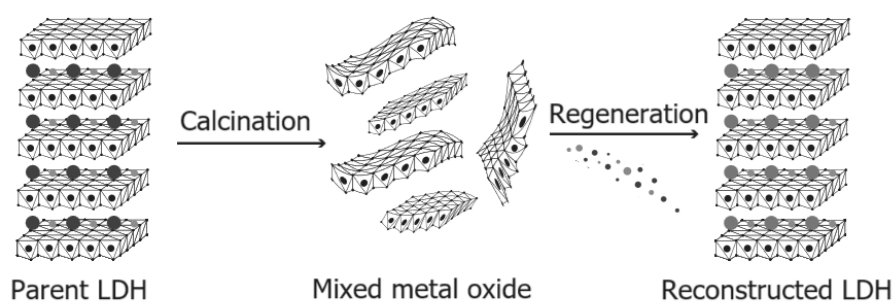


Fig. 2 – Schematic representation of the manifestation of the structural memory effect of LDHs.

Conversely, the "structural memory effect" is considered a significant approach for synthesizing LDHs with incorporated inorganic or large organic anions. However, the inserted anions do not need to be identical to those originally present in the pure LDH (Pandey *et al.*, 2022).

The LDH structures recovered by "structural memory effect" have demonstrated significant potential in various applications, including catalyst activation, structural investigations to pollutant removal and the preparation of new LDHs (Jin *et al.*, 2022).

Recent literature has focused on evaluating the structural memory effect, analysing the fundamental mechanisms behind it, and examining combinations of metal cations for various applications. For instance, Lu *et al.* utilized the memory effect of MgFeLDH to enhance magnetic separation and improve the adsorption performance of methyl orange (Lu *et al.*, 2024). In another study, Benhiti *et al.* proposed an efficient method for removing Cr(VI) from aqueous solutions by leveraging the properties of the memory effect (Benhiti *et al.*, 2023). Additionally, Guo *et al.* investigated the enhanced adsorption of Acid Brown 14 dye on calcined MgFeLDH, focusing on the valorisation of the structural memory effect (Guo *et al.*, 2013). Furthermore, Rodrigues *et al.* explored the influence of high-pressure processing on the structure and memory effect of LDHs (Rodrigues

et al., 2009). Calcination temperature and the properties of the involved species are critical factors to influence the reconstruction process of LDHs. Specifically, a calcination temperature below 600°C is essential for maintaining the structural integrity of LDHs during reconstruction (Pandey *et al.*, 2022). At these lower temperatures, the materials can undergo reversible decomposition, allowing for the potential restoration of their original layered structure upon rehydration. Conversely, when calcination temperatures exceed 900°C, the decomposition of LDHs becomes irreversible, resulting in the formation of stable phases with spinel-like characteristics. In this scenario, the original LDH structure is compromised, leading to a loss of functionality.

Moreover, it is important to emphasize that the memory effect of LDHs is lost at excessively high calcination temperatures. Elevated temperatures facilitate the diffusion of divalent metal ions into tetrahedral sites within the crystal structure. This diffusion results in structural changes that promote the formation of spinels instead of retaining the original layered architecture (Pandey *et al.*, 2022). Therefore, careful control of the calcination temperature is crucial for preserving the desirable characteristics and memory effect of LDHs.

The "memory effect" of LDHs provides a distinctive approach for altering these materials, rendering them appropriate for diverse catalytic applications (Wang *et al.*, 2023). Although additional research is necessary to accurately delineate the manifestation of the structural memory effect in various contexts, numerous studies have demonstrated that modifying LDH catalysts via this effect can generate more active sites. This change improves catalytic stability, functioning, and reusability (Gilea *et al.*, 2024).

The "structural memory effect" of LDHs is related to the topotactic transformation of mixed metal oxides (MMOs) or layered double oxides (LDOs) back into the LDH structure. This change occurs through thermal breakdown via calcination at temperatures between 450°C and 550°C. Throughout this method, stacked double hydroxides with enhanced catalytic and adsorption characteristics were reported (Wang *et al.*, 2023). Furthermore, inter-twinning these characteristics, structural recuperation, and augmented surface area resulted in the LDOs -type materials exhibiting a markedly superior adsorption capability. This enhanced capability enables applications for the efficient elimination of toxins and pollutants from diverse settings, hence expanding their potential applications (Peng *et al.*, 2018).

As a consequence, it was revealed that the "structural memory effect" of LDHs enhances their adsorption properties and catalytic capacities. However, further research is still required in this area to explore additional methods for controlling and optimizing the memory effect valorisation (Wang *et al.*, 2023).

2.1. Parameters influencing the structural regeneration by memory effect

Several factors significantly influence the final characteristics of LDHs obtained through the calcination-reconstruction process, in which the memory effect can be applied. Key parameters include temperature, pressure, pH, metallic composition, and the types of anions and molecules involved in the reconstruction process (Wang *et al.*, 2023).

Given the impact of these factors, researchers emphasize the necessity of developing methods to control and optimize them in order to enhance the applicability and characteristics of both LDHs and the derived LDO-like materials. This optimization involves modifying the composition of LDHs by introducing various cations or anions, which allows for tailored properties to suit specific applications.

Furthermore, it is essential to systematically evaluate the extent of these modifications and to assess the properties of the resulting layered double oxides under various calcination conditions. This comprehensive approach ensures that the multifunctional features of LDH-LDO phases can be effectively harnessed for applications in catalysis, adsorption, and other fields.

2.1.1. Temperature as a parameter in tailoring LDH structural regeneration

According to studies in the literature, temperature plays an important role in the memory effect. Specifically, as the calcination temperature increases, the relative crystallinity decreases, resulting in a corresponding diminishment of the memory effect. Furthermore, if the temperature is too high, the decomposition products will be unable to revert to the layered structure of LDHs.

In the calcination process, temperature significantly influences the properties LDHs/LDOs, specifically with regard to the following aspects:

- the structure of the resulting layered double oxides;
- the presence of the memory effect;
- the reconstructed structure of LDHs (Ye *et al.*, 2022).

Several researchers have demonstrated that the calcination temperature is crucial and significantly affects the memory effect. An appropriate calcination temperature is necessary to obtain the desired materials based on LDHs. For example, a high temperature can lead to the disappearance of the memory effect or unexpectedly affect the reconstruction rate (Kowalik *et al.*, 2013).

The temperature directly impacts the composition of the calcination products, as higher temperatures result in larger amounts of metal oxide. However, the quantity of oxide does not influence the reconstruction conditions of LDO (Ye *et al.*, 2022).

An important aspect to mention is that the repetitive calcination-hydration process affects the characteristics of the material used, specifically the

layer structure, interlayer molecules, the amounts of metal oxide, and its behaviour in relation to the memory effect (Xu *et al.*, 2021).

2.1.2. Pressure as a parameter in tailoring LDH structural regeneration

Pressure is another parameter influencing the memory effect of LDHs; however, it has not been thoroughly studied compared to other parameters. The effects of pressure on the memory effect have been explored by only a limited number of researchers. For instance, one study investigated the impact of high-pressure processing on the structure and memory effect of specific LDH samples that had been calcined at temperatures ranging from 450 to 700°C.

The researchers first demonstrated that LDH samples calcined at 450°C experienced a loss of their clay-like structure, transforming into layered double oxides (LDOs). Notably, when a drop of water was added to the calcined sample, the original LDH structure was regained within a few hours, showcasing the restorative potential of the memory effect. In contrast, when the calcined sample was exposed to air for approximately 72 hours at 6 GPa, the LDH structure could not be regenerated. This finding is indicative how the prolonged exposure to air and applied pressure can hinder the recovery of the original structure. Furthermore, when the calcined sample was compacted at 7.7 GPa without prior exposure to air, the LDH structure remained irreversibly altered, as it could not be restored even after immersion in water (Rodrigues *et al.*, 2009).

These findings underscore the complex interplay between pressure and the memory effect, indicating that higher pressure may inhibit the ability of LDHs to regenerate the original LDH structure. Further, the LDHs were calcined at 700°C and immediately compacted at pressures of 2.5 and 7.7 GPa, without exposure to air. It was observed that these samples did not regain their layered structure after being left in air for 7 days. However, an incipient recovery of the original layered structure was noted when the samples were ground prior to exposure to air, becoming slightly more pronounced when water was added. This recovery process was more evident in samples compacted at lower pressure. Therefore, it can be concluded that the memory effect of the investigated LDHs is, in general, adversely affected by pressure.

2.1.3. pH as a parameter in tailoring LDH structural regeneration

Another parameter that can influence the memory effect of LDHs is the pH value of the recovered aqueous solution. While the impact of pH may be less pronounced compared to other factors, it is still crucial to monitor and evaluate the initial pH when utilizing the memory effect (Ye *et al.*, 2022). Specialized studies indicate that an increase in pH marks the beginning of the reconstruction process for LDHs. Most conclusions regarding the influence of pH have been derived from the adsorption experiments involving LDOs.

The alkalinity of LDHs is largely determined by the characteristics of the divalent metal hydroxides present in the primary layer. Conversely, the presence of trivalent amphoteric metal oxides within the primary layer structure, combined with various anions in the interlayer space, imparts a mild acidity to the LDHs. This dual influence on acidity and alkalinity highlights the complex interactions at play within the LDH structure. As a result, when LDHs undergo heat treatments such as calcination, the material transforms into LDOs, which exhibit significantly increased alkalinity (Wang *et al.*, 2023). This change in alkalinity can enhance the properties and functionalities of the resulting materials, further demonstrating the importance of pH in the context of the memory effect and structural modifications of LDHs.

On the other hand, during reconstruction, pH significantly influences both the surface charge and the hydration process. More specifically, pH affects the charged state of the LDOs surface, which in turn impacts the interaction between anions and the LDO during reconstruction. Consequently, the rates and speed of the structural reconstruction can be modified. Typically, in acidic and neutral reconstruction solutions, LDOs exhibit a higher affinity for intercalating anions due to their protonated surfaces. Conversely, in a strongly acidic medium, the memory effect is irreversibly damaged because the LDOs undergo a decomposition process (Zhang *et al.*, 2020).

2.1.4. The composition of layers as a parameter in tailoring LDH structural regeneration

The composition of metal cations is a crucial factor that can influence the memory effect of LDHs. Discussing this factor is important because it significantly impacts the speed and progression of the reconstruction process. Studies have demonstrated that varying the metal composition of LDHs can lead to different outcomes under the same calcination conditions, including differences in critical temperatures and the timing of spinel structure formation.

A key aspect to consider is that LDHs formed from transition metal cations are generally more challenging to recover than those composed of cations with d0 or d10 electron configurations. This is primarily due to the stability of spinels, which results from the stabilization of the crystalline field surrounding these transition metals (Rives, 2002). The inherent stability of these structures complicates the regeneration of the original LDH form during the reconstruction process. Moreover, during the reconstruction of LDOs, metal hydration occurs more rapidly at lower pKa values, facilitating quicker reconstruction. For example, ZnLDH can be restored more efficiently through the reconstruction process compared to MgLDH (Ye *et al.*, 2022; Marchi *et al.*, 1998).

Another important consideration related to metal composition is the metal ratio within the LDHs. Variations in this ratio can lead to isomorphic substitution within the metallic layers during reconstruction, which ultimately

affects the properties and performance of the reconstructed LDHs when leveraging the memory effect. This interplay between metal cation composition, hydration rates, and structural integrity underscores the complexity of optimizing LDH materials for various applications (Ye *et al.*, 2022).

2.1.5. Nature of inter-layers anions as a parameter in LDH structural regeneration

In the structural reconstruction process of LDHs, it is important to consider the relationship among anions, and the recovered LDH structure. The choice of anions in the reconstruction environment significantly influences the characteristics of the regenerated LDH structure. A wide variety of both organic and inorganic anions can be intercalated between the layers of LDHs when applying the memory effect, resulting in a diverse array of compounds (Ye *et al.*, 2022).

During reconstruction, anions are attracted to the interlayer space due to the positive charge of the LDH layers, facilitating the restoration of the original layered structure. In a single anion system, intercalation via the memory effect is a straightforward method for synthesizing different LDH compounds. However, in a multi-anion system, the process becomes more complex due to the competing intercalation of anions that can occur during reconstruction. Consequently, utilizing the memory effect can help mitigate some limitations related to ion exchange order and competing intercalation sequences in single anion systems (Ye *et al.*, 2022).

Moreover, certain anions can modify the mechanism of the the regeneration by the structural memory. For instance, CO_3^{2-} , which are the most commonly intercalated anions, possess a higher binding capacity for LDHs, leading to more stable structures. As a result, when LDHs are synthesized without a protective gas, they are typically intercalated with carbonate. In aqueous solutions, carbonate can also facilitate the exchange of the original anions within the LDH layers (Meng *et al.*, 2017).

The interplay between the type of anions and structural stability underscores the importance of selecting the appropriate anions during the reconstruction process to enhance the properties and functionalities of LDHs.

2.2. X-ray characterization as a technique to monitor the structural reconstruction by “memory effect”

X-ray diffraction is highly effective for monitoring the structural recovery of LDHs and to analyse the structural changes that occur during the reconstruction process. XRD can elucidate various structural features affected by the memory effect, including the following:

- surface defects and crystallinity: changes in peak intensity provide insights into the degree of crystallinity and the presence of defects in the structure.

- altered intercalation: shifts in peak positions indicate changes in the intercalation of anions between the layers, revealing how the structure adapts during reconstruction.

- layer spacing: variations in peak positions help determine the interlayer spacing, which is crucial for understanding the structural integrity of the LDHs.

The presence of specific diffraction peaks, such as (003), (006), and (009) (see Fig. 3), indicates the existence of an LDH-type structure. For LDHs that have been thermally calcined at temperatures above 300°C, these characteristic peaks exhibit significant changes compared to those of LDHs calcined at lower temperatures. Specifically, the peaks associated with the typical lamellar structure of LDHs diminish or disappear as the calcination temperature increases. As calcination temperature rises, there is a noticeable decrease in the intensity of the (003), (006), and (009) peaks, while the intensity of the (110) and (113) peaks increased. This shift signifies the transformation of LDHs into mixed metal oxides, indicating an almost complete decomposition of the original LDH structure. Such transformations are critical for understanding how calcination conditions impact the properties and functionalities of LDHs in various applications.

For LDH samples calcined at temperatures higher than 500°C, new phases are observed, as evidenced by the presence of the broad reflections, revealing that most of the intercalated ions can be removed at temperatures below 350°C and LDOs start to form.

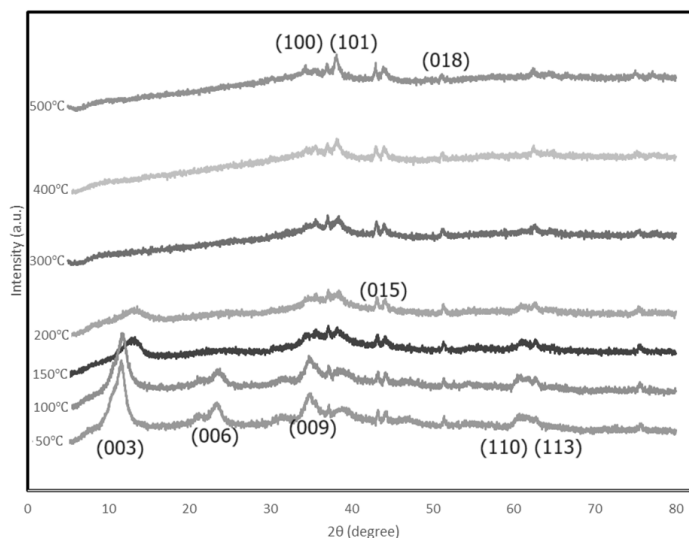


Fig. 3 – X-ray diffraction patterns at different calcination temperatures.

3. About suppression of the memory effect

As has been recently documented in the literature (Jin *et al.*, 2022), LDHs can be used in a wide range of industrial and scientific applications, and the memory effect is particularly useful in the absorption process of layered double oxides. However, there are still several disadvantages regarding the applicability of the memory effect.

For example, one disadvantage is that in practical applications, the memory effect can influence the performance of the materials involved in the process. In this context, we are referring to the suppression of the memory effect. The applicability of the memory effect can be influenced by two factors (Li *et al.*, 2014):

- extreme temperature calcination;
- the inhibition of the memory effect through co-doping.

An important cause for inhibiting the memory effect may be the non-spontaneous hydration reaction involved in the overall LDH reconstruction process. Additionally, it is worth noting that there is still insufficient research offering valuable insights into how composite materials can inhibit the memory effect (Ye *et al.*, 2022).

The suppression of the memory effect is particularly considered when the derived LDOs are involved in catalytic processes. In this context, it is essential to preserve the LDOs large surface area, minimize the formation of hydrogen bonds and the presence of micropores (Marchi *et al.*, 1998).

While suppressing the memory effect can be challenging, it can be overcome by optimizing the factors that influence the intertwined calcination-reconstruction processes.

4. Conclusions

This work highlights recent advancements in the design and fabrication of LDHs with enhanced multifunctional properties, achieved by regenerating their 2D structures through the structural memory effect. The structural memory effect is a key feature of LDH materials and can be carefully controlled by several factors, such as calcination temperature, the pH of the aqueous medium during reconstruction, pressure, and the types of coexisting anions and cations during the process.

During regeneration, the 2D-layered architecture of LDHs undergoes changes due to complex mechanisms that activate and optimize their memory effect. This structural memory effect can impart new functionalities to regenerated LDHs, broadening their applications. These improved LDHs can be used as adsorbents, drug delivery systems, packaging materials, and cosmeceutical agents in the cosmetic industry, making them versatile across various fields.

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HIDROXIZI DUBLU LAMELARI: REGENERAREA STRUCTURALĂ PRIN EXPLORAREA EFECTULUI DE MEMORIE

(Rezumat)

Hidroxiții dublu lamelari reprezintă o clasă de compuși anorganici alcătuiți din straturi încărcate pozitiv ce sunt stivuite unele peste altele într-un aranjament structural

asemănător brucitului. Hidroxizii dublu lamelari (LDH) sunt materiale foarte versatile datorită faptului că pot găzdui în spațiile interlamelare o mare varietate de anioni. Ca urmare a acestei versatilități, LDHs sunt utilizați pe scară largă în diverse domenii, precum stocarea și cedarea de medicamente, depoluarea apei, cataliză etc. O caracteristică aparte a acestor materiale este așa-numitul „efect de memorie structurală”, care se referă la capacitatea de a-și reface structura stratificată, distrusă prin calcinare. Caracteristicile hidroxizilor dublu lamelari obținuți prin acest proces de reconstrucție sunt influențate de factori precum: temperatură, presiune, pH, compoziția metalică, anionii și moleculele de reconstrucție. În acest context, scopul prezentului studiu este de a oferi o perspectivă detaliată asupra proprietăților și aplicațiilor hidroxizilor dublu lamelari regenerați prin efectul de memorie, furnizând informații valoroase asupra manierei în care pot fi exploatate aceste caracteristici.