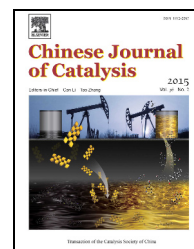


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Minireview

Layered double hydroxide and related catalysts for hydrogen production and a biorefinery

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ABSTRACT

Layered double hydroxides (LDHs) have received much attention for their unique properties. As solid catalysts, LDHs and their derivatives have been widely studied and applied for their excellent catalytic performance. Several synthesis methods of LDHs were briefly introduced, and the applications of LDHs and related catalysts for hydrogen production and a biorefinery were emphasized in this article. The prospects for LDH related compounds in the synthesis of new materials and their catalytic application in green catalysis systems were also presented.

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1. Introduction

Layered double hydroxides (LDHs) are layered anionic clays also known as hydrotalcite-like compounds (HTLCs). They are new inorganic functional materials with positively charged brucite-like lamellas and charge-balancing anions and solvation molecules in the interlayer region. The lamellar structure is constructed with octahedrons by sharing their edges with divalent and trivalent metal cations in the center and six hydroxide ions at vertices [1,2], as shown in Fig. 1.

LDHs have attracted considerable interest in recent years due to their unique structure and properties, including exchangeable lamellar anions, controllable basicity, tunable composition, and uniform deposition of the active metal [3,4]. Homogeneously dispersed mixed metal oxides can be obtained

by a controlled thermal decomposition of LDH precursors. These oxides have many special characteristics, such as high specific surface areas, basic character, high dispersion of the metallic phase, thermal stability against sintering, and capacity to regenerate the original layered structure [5,6]. Therefore, LDHs and their derivatives have been widely used as heterogeneous basic catalysts in many fields, such as clean energy and pollution control [7,8].

LDHs have received much attention for their properties and catalytic performance. Recent progress in the synthesis of LDHs is summarized with a special focus on the application of LDHs and derivatives as catalysts for H₂ production and a biorefinery in this work. We expect that more efforts will be devoted to the special layer structure and properties of LDHs, which are significant for the design of new materials [9,10].

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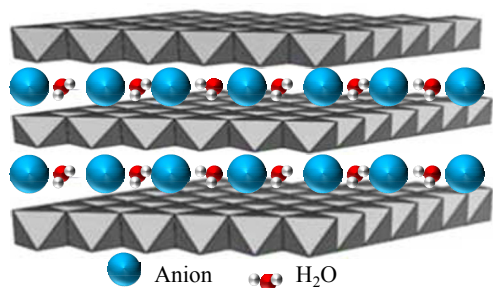


Fig. 1. Structure illustration of LDHs.

2. Synthesis methods of LDHs

The preparation methods of LDHs include co-precipitation method, urea hydrolysis process, anion exchange, and roasting recovery technique. The co-precipitation method is the most widely used process to prepare LDHs [11,12]. For this method, solution A and solution B are prepared first before co-precipitation. Solution A contains different metal nitrate hexahydrate with the proper molar ratio, and solution B is usually a mixture solution of Na₂CO₃ and NaOH. Then, solution A and solution B are simultaneously added dropwise to a vessel containing deionized water with vigorous stirring. During this process, the slurry is kept at a constant pH by controlling the dripping speed of the solution. After aging for a period of time, the precipitate obtained is separated from the mother solution, rinsed with deionized water, and appropriately dried to obtain the LDH [13,14]. Yan et al. [13] successfully synthesized a series of LDH precursors used for the dehydration of biomass-derived fructose into the platform chemical 5-hydroxymethylfurfural by this method. This technology includes the low supersaturation method and high supersaturation method [15]. Usually, the aging process is combined with a hydrothermal or microwave treatment [16,17]. The crystallinity of the LDHs can be improved, but the former is time consuming and the crystals always have a large particle size, while the latter is time saving and the crystals are relatively small with a uniform distribution.

The urea hydrolysis process usually employs urea as precipitant. By controlling the heating rate, the urea decomposition rate can be controlled, and the solution can be kept at a constant pH. Through this way, the sedimentation rate of the catalysts can be controlled [18]. In brief, solid urea is added to an aqueous solution containing metal nitrates with the desired molar ratio, until the urea to metal molar ratio reaches the desired value. The solution obtained is usually kept stirred at a certain temperature in a thermostatic reactor equipped with a coolant. The precipitates are filtered, washed, and then dried at the set temperature, and finally the LDH precursors are obtained [18,19]. Compared to the co-precipitation method, this procedure can be operated more easily and is time saving. Therefore, it has been widely used. In our previous works, Yu et al. [19] successfully synthesized the NiCuMgAl LDH precursors for ethanol steam reforming using this method. The XRD patterns of the Ni_{0.5}Mg_{2.5-x}Cu_xAl precursors are shown in Fig. 2. This method is also combined with a hydrothermal or micro-

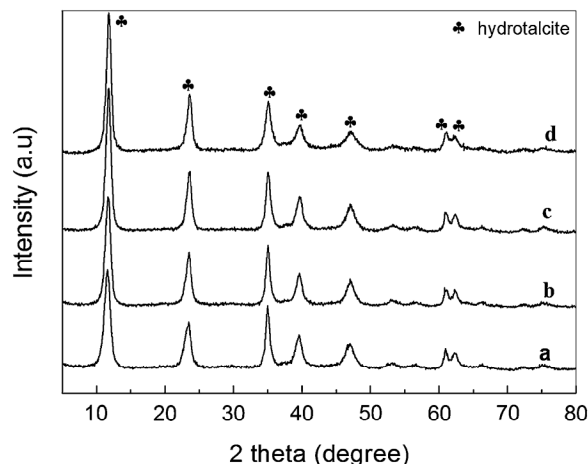


Fig. 2. XRD patterns of LDH precursors. (a) Ni_{0.5}Mg_{2.5}Al; (b) Ni_{0.5}Cu_{0.05}Mg_{2.45}Al; (c) Ni_{0.5}Cu_{0.1}Mg_{2.4}Al; (d) Ni_{0.5}Cu_{0.25}Mg_{2.25}Al. Reproduced from Ref. [19] with kind permission from Springer Science and Business Media.

wave treatment to improve the morphology of the LDHs [20,21]. Recently, Deng et al. [22] synthesized a series of LDH precursors using urea as precipitating agent combined with a microwave-hydrothermal treatment. The resulting material had a narrow particle size distribution. Scanning electron microscopy images of LDH and calcined LDH are shown in Fig. 3.

The anion exchange technique takes advantage of the exchangeability of the interlayer anions to realize the exchange of the interlayer anions with other desired anions. Usually, LDH precursors synthesized by the methods given above are suspended in a solution containing the desired anions under mild conditions. A few hours later, LDHs with the desired anion can be obtained [18,23]. Recently, a binary system of Sb(III), an important alternative for Leishmaniasis chemotherapy, was prepared by Menezes et al. [24] using MgAl LDHs as a nanocarrier via ion exchange. The roasting recovery method makes full use of the “memory effect” of LDHs. When the mixed oxides, calcined LDHs, are put back into a solution containing the required anions with a certain pH, they can reconstruct the original lamellar structure after a period of time [25,26]. Álvarez et al. [5] found that the rehydrated LDHs had many basic Brönsted sites. Therefore, they showed excellent catalytic activity for the transesterification of glycerol. Meanwhile, Sun et al. [26] reported that F⁻ modified CuNiAl LDHs synthesized by this method had high activity for the selective oxidation of toluene to benzyl alcohol.

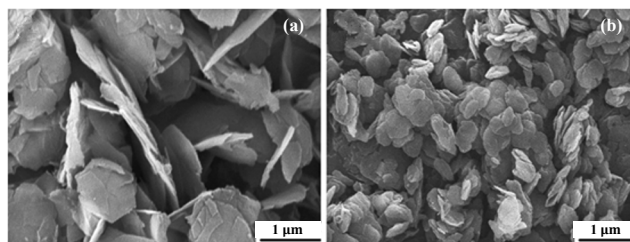


Fig. 3. SEM images of LDH (a) and calcined LDH at 773 K for 6 h (b). Reprinted with permission from Ref. [22]. Copyright 2011 Elsevier.

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